

Book of Abstracts
E-MRS 2007 Fall Meeting

Book of Abstracts: E-MRS 2007 Fall Meeting

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Welcome

The European Materials Research Society



Fall Meeting Warsaw University of Technology

Warsaw (Poland)

17th - 21st September, 2007

We are pleased to welcome delegates to the 2007 Fall Meeting of the European Materials Research Society (E-MRS).

Organisers

Conference Organisers:

- European Materials Research Society (****)
- Polish Materials Science Society
- Warsaw University of Technology (*)
- Institute of Physics, Polish Academy of Sciences (**)
- Institute of High Pressure Physics, Polish Academy of Sciences (***)

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Sponsors

THE E-MRS FALL MEETING 2007 is supported by:

	European Materials Research Society
	Polish Ministry of Science and Higher Education
	The Polish Materials Science Society
	Faculty of Materials Science and Engineering, Warsaw University of Technology
	Institute of Physics, Polish Academy of Sciences
	Institute of High Pressure Physics, Polish Academy of Sciences
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THE OFFICIAL AIRLINE NETWORK FOR E-MRS 2007 FALL MEETING

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The European Materials Research Society (E-MRS) is a non profit scientific association founded in 1983. It focuses on creating the synergy between interdisciplinary, innovative technologies, spreading and exchanging information and promoting technology transfer from public institutions to industry. The primary objective of E-MRS is to promote and enhance the efficiency of research in Europe in the field of Advanced Materials. E-MRS seeks to quickly inform researchers of the

scientific and technological developments in their field of interest from around the world through the society's links with other MRS societies belonging to the International Union (IUMRS).

E-MRS Opportunities for its Members

The Executive Committee of the European Materials Research Society (E-MRS) has made the decision to diversify and extend the activities of the Society toward a variety of items beyond the organization of the E-MRS Spring and Fall Meetings. The Society's three initiatives are books, short courses, and workshops.

E-MRS seeks authors and editors interested in printing books on topics such as materials science, materials engineering, and materials processing. The book would be published in the frame of the Elsevier/E-MRS Publishing Agreement related to a monograph series of books titled the "E-MRS Monograph Work." Based on the contents of books in the monograph series, E-MRS plans to develop short courses to be held in conjunction with the Spring and Fall Meetings.

For its third initiative, E-MRS plans to establish Advanced Research Workshops on new topics in order to strengthen and develop the relationships between scientists from various European countries. The Society would seek sponsorship from research agencies such as the European Science Foundation and NATO. The output of these workshops is to initiate research activities in Europe, and prepare the ground to develop future collaborative research programs or scientific networks.

For more information, contact E-MRS at 23 Rue du Loess, BP 20, 67037 Strasbourg Cedex 02, France; tel. +33-(0)3-88-10-63-72; fax +33-(0)3-88-10-62-93; e-mail emrs@emrs.c-strasbourg.fr; or access Web site www.emrs-strasbourg.com.

The Polish Materials Science Society

The Polish Materials Science Society (PMSS) was founded in December 2004 and promotes interdisciplinary cooperation between scientists working in different scientific areas - materials science and engineering, chemistry, physics, biology, medicine etc. The PMSS closely collaborates with E-MRS in the organisation of the E-MRS Fall Meeting.

Frame programme

Introduction

The E-MRS Fall Meeting has been organised since 2002 as a twin conference to the Spring Meeting held every year in Strasbourg. During that time the Fall Meeting has grown from 3 symposia and about 200 participants in 2002 to 10 symposia and 600 participants in 2006. What is also important the conference is becoming increasingly multi-national and has representatives from all over the world. The E-MRS 2007 Fall Meeting will consist of two Plenary Sessions, and 10 scientific symposia which will run concurrently. The Opening Ceremony together with the Czochralski Award Ceremony and the Acta Materialia Gold Medal Ceremony will take place on Monday 17th September at 10:00 am. Poster sessions, common for all symposia, will be held from 15.50 - 17.20 on Monday September 17 and Wednesday, September 19.

The detailed timetable and abstracts for papers to be presented in each symposium will be found in the appropriate section.

The Czochralski Award

One of the most important and prestigious events which takes place every year during the Fall Meeting is the Czochralski Award Ceremony. The Award in the name of Jan Czochralski has been instituted by E-MRS willing to commemorate the contribution of Jan Czochralski to materials science, technology transfer to industry, and international collaboration. The Society formally legalised the Czochralski Award for Achievements in Materials Science.

In 2006 – the European Materials Research Society, the Polish Academy of Sciences, the Polish Materials Science Society, and the Polish Society for Crystal Growth jointly established the International Chapter of the Jan Czochralski Award which selects a prize winner. To date three recipients were honoured: in 2004 Professor Dr Walter Heywang, formerly Director of Research of Siemens, for 2005 Professor Dr Boris Paton, the long term President of the Ukraine National Academy of Sciences and in 2006 Professor Thaddeus B. Massalski, Carnegie-Mellon University in Pittsburgh. The beneficiary of the Czochralski Award for 2007 will be **Professor Shuji Nakamura**, the inventor of the Blue Laser Diode, ERATO, JST, UCSB group, Santa Barbara.

The Acta Materialia Gold Medal

This year the E-MRS Fall Meeting has an honour to host the presentation of Acta Materialia Gold Medal to Professor Herbert Gleiter. The Acta Materialia Gold Medal is awarded annually by the Board of Governors of Acta Materialia, with financial support from Elsevier, Ltd. Nominees are solicited each year from the Cooperating Societies and Sponsoring Societies of Acta Materialia, Inc., based on the nominee's demonstrated ability and leadership in materials research. The candidates are named on a ballot for the selection committee (a panel of three judges who serve for three-year terms). A preferential ballot is then conducted and the candidate with the most points is the beneficiary. The Award consists of the gold medal, an inscribed certificate, and a cheque for a sum of money contributed by the Board's. The Gold Medal is awarded each year, unless the judges feel no candidate is sufficiently well qualified. The presentation is made at a meeting selected by the nominating society or at a major technical meeting in the awardee's country. The recipient of the Acta Materialia Gold Medal for 2007 will be **Prof. Herbert Gleiter** of Forschungszentrum Karlsruhe Institute of Nanotechnology

Conference programme

Plenary Sessions:

1. Monday, September 17, 10:00 - 12:30

2. Wednesday, September 19, 09:30 - 10:30

Poster Sessions:

1. Monday, September 17, 15:50 - 17:20
2. Wednesday, September 19, 15:50 - 17:20

Symposia:

SYMPOSIUM A, Room 219:

Chemistry and processes for the design of metal oxide nanoparticles

SYMPOSIUM B, Room 134:

Chemical and electrochemical synthesis of advanced materials and nanostructures on solid surfaces: growth mechanisms, characterizations & applications (CESAM)

SYMPOSIUM C, Room 226:

Atomic Layer Deposition 2007

SYMPOSIUM D, Room 144:

9th international symposium on electrochemical/chemical reactivity of metastable materials

SYMPOSIUM E, Room 231:

Shape memory materials for smart systems

SYMPOSIUM F, Room 213:

The binary ion exchange process in materials science and chemistry: application, characterization and modelling

SYMPOSIUM G, Room 206:

Genetic algorithms in materials science and engineering

SYMPOSIUM H, Room 208:

Current trends in optical and x-ray metrology of advanced materials and devices II

SYMPOSIUM I, Room 309:

New opportunities and challenges in material research using synchrotron and free electron laser sources

SYMPOSIUM J, Room 315:

Microscopy and spectroscopy techniques in advanced materials characterization

Special event:

Acta Materialia Gold Medal Workshop

"Perspectives of nanoscience and nanotechnology"

(17th-21st September, the Conference Venue, room 124)

Satellite events:

Genetic algorithms for beginners

(16th September, Faculty of Materials Science and Engineering, Warsaw University of Technology, 141 Woloska Street, Warsaw, room 305)

COST Action D30 Working Group Meeting

(20th-21st September, in conjunction with Acta Materialia Gold Medal Workshop)

Symposia

Plenary session

Programme

Monday, 17 September

Opening & Plenary Session

Monday morning, 17 September, 10:00

Czochralski Award Ceremony

Monday morning, 17 September, 10:10

10:10

Plenary talk

Recent Performance of Nonpolar/Semipolar/Polar GaN-based Blue LEDs/LDs and Bulk GaN Crystal Growth

Shuji Nakamura¹, S. P. DenBaars¹, J. S. Speck¹, M. C. Schmidt¹, K.-C. Kim¹, R. M. Farrell¹, D. F. Feezell¹, D. A. Cohen¹, M. Saito¹, H. Sato¹, H. Asamizu¹, A. Tyagi¹, H. Zhong¹, H. Masui¹, N. N. Fellows¹, M. Iza¹, T. Hashimoto¹, K. Fujito²

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Charge separation due to spontaneous and piezoelectric polarization inherent to the wurtzite structure has deleterious effects on the performance of most c-axis oriented devices.1) To overcome this problem, Nonpolar GaN, such as a-plane and m-plane GaN or Semipolar GaN substrates have been grown.

We reported the fabrication of violet InGaN/GaN Light Emitting Diodes (LEDs) on semipolar (1011) GaN bulk substrates. The output power and External Quantum Efficiency (EQE) at a driving current of 20 mA were 28 mW and 45 % respectively, with peak electroluminescence (EL) emission wavelength at 411 nm. The LEDs showed minimal shift in peak EL wavelength with increasing drive current indicating an absence of polarization induced electric fields.2) Also, high power and high efficiency nonpolar *m*-plane nitride LEDs were fabricated on low extended defect bulk *m*-plane GaN substrates.3) The first nonpolar *m*-plane (1100) nitride laser diodes (LDs) were realized on low extended defect bulk *m*-plane GaN substrates.4) Broad area lasers without AlGaN cladding layers were fabricated and tested under pulsed conditions. These laser diodes had threshold current densities (J_{th}) as low as 3.7-2.3 kA/cm².5) Stimulated emission was observed around 400 nm. The recent performance of Nonpolar, Semipolar and Polar (c-plane) GaN-based devices are described. Also, I like to talk about the latest result of a GaN bulk crystal growth by anammothermal method at UCSB.

References

1. S. Chichibu, T. Azuhata, T. Sota, and S. Nakamura, Appl. Phys. Lett. 69, 4188 (1996)
2. A. Tyagi, H. Zhong, N. N. Fellows, M. Iza, J. S. Speck, S. P. DenBaars, and S. Nakamura, Jpn. J. Appl. Phys. 46 (2007) L129
3. M. C. Schmidt, K.-C. Kim, H. Sato, N. Fellows, H. Masui, S. Nakamura, S. P. DenBaars, and J. S. Speck, Jpn. J. Appl. Phys. 46 (2007) L126

4. M. C. Schmidt, K.-C. Kim, R. M. Farrell, D. F. Feezell, D. A. Cohen, M. Saito, K. Fujito, J. S. Speck, S. P. DenBaars, and S. Nakamura, Jpn. J. Appl. Phys. L190-L191, Vol 46 (2007).

5. D. F. Feezell, M. C. Schmidt, R. M. Farrell, K.-C. Kim, M. Saito, K. Fujito, D. A. Cohen, J. S. Speck, S. P. DenBaars, and S. Nakamura, Jpn. J. Appl. Phys. L284-L286, Vol 46 (2007).

Acta Gold Medal Ceremony

Monday morning, 17 September, 11:10

11:10

Plenary talk

Our Thoughts Are Ours, Their Ends none of Our Own - Are there Ways to synthesize Materials beyond the Limitations of Today's?

Herbert D. Gleiter

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The methods available today to modify the structure and the properties of crystalline materials may be divided in the following two groups: Modifications resulting from the introduction of lattice defects and modifications resulting from alloying of two or more components.

In materials with grain sizes of 1 mm or more, the introduction of lattice defects modifies the microstructure. However, the modifications of the atomic structure are limited to less than 1 volume % of the material. The way to modify the atomic structure in up to 50 volume % of a material by introducing defects was opened by reducing the crystal size of polycrystalline materials to a few nanometers. Materials of this kind are called nanocrystalline or nanostructured materials. The step towards modifying the entire atomic structure of solid materials seems to be possible by means of nanoglasses. Nanoglasses are glasses that are generated by consolidating nanometer-sized glassy spheres at high pressures (several GPa). The existing structural investigations on metallic nanoglasses as well as studies by means of molecular dynamics suggest that nanoglasses consist - in the as prepared state - of the following two structural components. Glassy regions - resulting from the consolidated spheres - and interfaces between these glassy regions. In these glass/glass interfaces, the free volume is enhanced and the nearest neighbor co-ordinations deviate from the ones in the glassy regions. If these nanoglasses are annealed, the enhanced free volume in the glass/glass interfaces seems to delocalize and, thus, modifies the atomic structure of entire material. In fact, it is found that -after long annealing times- nanoglass specimens consist of a surface region with an enhanced density (due to a high hydrostatic pressure) and a glassy core region with a significantly (up to about 10%) reduced density. In other words, nanoglasses may pave the way to tune the free volume (density) of glasses at constant chemical composition.

The modifications of solid materials by alloying may be divided in the following two groups. Components that can be alloyed by melting followed by solidification, and alloys of components that are immiscible in the solid state, e.g. alloys of metals and ionics such as Au-NaCl. The preparation of alloys of this type seems attractive because they are likely to exhibit new properties. So far, apparently two approaches have been considered for preparing such alloys. In the first approach (applicable to systems with mobile charge carriers), electronic screening effects at interphase boundaries are utilized. If nanocomposites of immiscible components are prepared with a crystal size comparable

to the electronic screening length, the electronic structure of the entire specimen is modified (due to the screening effects). As was shown, this modification may result in the formation of solid solutions of conventionally immiscible components, e.g. of Ag and Fe. In systems without mobile charge carriers, vapor deposition of ions of one of the components on an electrically charged substrate may be used to generate solid solutions.

Lunch break

Monday afternoon, 17 September, 12:30

Wednesday, 19 September

Plenary Session

Wednesday morning, 19 September, 9:30

9:30

Plenary talk

Materials and Technology for Global Carbon Dioxide Recycling -For supply of renewable energy and prevention of global warming

Koji Hashimoto¹, Naokazu Kumagai², Koichi Izumiya², Zenta Kato¹

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According to the data of Department of Energy, USA, the primary energy consumption of the world in 1990-2004 increased by a factor of 1.0181 every year. The primary energy consumption per person in developed countries in 2004 was 6.23 times as high as that in developing countries. Thus the sharp increase in primary energy consumption by developing countries is natural and serious global warming cannot be avoided as long as fossil fuel is used. The increase in the world primary energy consumption at the average rate in 1990-2004 will lead to complete exhaustion of oil, uranium, natural gas and coal reserves in 2034, 2040, 2042 and 2054, respectively. In order to avoid such a miserable situation of no fuel and intolerable warming, we have to establish immediately new technology to use only renewable energy for all people in the whole world. We are proposing global carbon dioxide recycling. The electricity necessary for all people in the whole world will be generated by solar cells in the deserts. 4.71023×10^{20} J corresponding to the world total primary energy consumption in 2004 can be generated in the form of electricity by solar cells with 15 % energy conversion in only 1.85 % of the main deserts on the Earth, if 1000 Wm^{-2} of solar energy are available for 8 hours a day. Because long distance transmission of electricity is not feasible, the electricity will be used for production of hydrogen by seawater electrolysis at nearby desert coasts. Because there are no mass transportation technology and no widely used combustion systems for hydrogen, hydrogen will be converted to methane, that is, the main component of natural gas by the reaction with carbon dioxide at the desert coasts. After combustion of methane carbon dioxide will be recovered and transported back to the desert coasts. Among systems in global carbon dioxide recycling, seawater electrolysis and methane formation by the reaction of carbon dioxide with hydrogen have not been performed industrially. We tailored anodes for oxygen evolution without chlorine formation in seawater electrolysis, energy-saving cathodes for hydrogen production and ideal catalysts for methane formation by the reaction of carbon dioxide with hydrogen with almost 100 % methane selectivity in addition to quite fast conversion. We constructed a prototype plant for

global carbon dioxide recycling in 1995 and an industrial scale pilot plant consisting of seawater electrolysis and carbon dioxide methanation in 2003. The latest advance will be reported.

Prof. Gabriel Crean

Information about IUMRS

Wednesday morning, 19 September, 10:15

Coffee break

Wednesday morning, 19 September, 10:30

Symposium A

Welcome

Aqueous or non-aqueous solution chemistry has proven to be a powerful tool to produce nanoparticles and nanomaterials with finely tuned morphological, compositional and structural characteristics. Control of size, shape, surface and assembly properties of nanoscale materials are fundamental steps towards their study in fundamental research and their implementation in technological devices.

In spite of the immense progress in nanoparticle research, the reported synthesis strategies are often isolated efforts without offering any underlying physical or chemical principles that would allow either the generalization of the processes involved for any kind of nanomaterial or the development of a mechanistic understanding of nanoparticle formation.

The symposium will provide a platform for the presentation of state-of-the-art synthesis procedures to collect and summarize general reaction principles to make first steps towards a rational synthesis design for inorganic nanomaterials. The symposium will focus on chemical methods that make it possible to tune the particle size and/or morphology on the nanoscale. Special attention will be given to works combining original syntheses and mechanistic investigations, elaborating the key parameters for tailor-made nanomaterials. Instrumental techniques to measure in-situ kinetics, as well as nucleation and growth models with a special attention to phenomena at the growing surface will be highly appreciated. Due to their rich chemistry and the variety of possible applications, metal oxides are the key -but not exclusive- materials of the symposium. In addition to nanoparticles, larger objects could enter the topic on condition that they are nanostructured or if the mechanism of their formation involves nano-building blocks.

The symposium aims at covering a wide panel from experimental chemistry to theoretical physics to promote exchange of views and ideas. In this context, communications from various scientific communities will be welcome.

Keywords:

- nucleation, oriented growth, templated growth
- nanoparticles, metal oxides, nanostructures, self-assembly
- precipitation, sol-gel chemistry, solvothermal syntheses, rational synthesis design....
- in-situ measurements, nucleation and growth theory/models

Organisers

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Proceedings

The contributions to the Symposium will be published in a special issue of the European Journal of Inorganic Chemistry entitled "Metal Oxide Nanoparticles" at the beginning of 2008, following a peer reviewing process.

Sponsors

- GIE LVMH Recherche

Programme

Monday, 17 September

Opening & Plenary Session

Czochralski Award Ceremony & Acta Gold Medal Ceremony
Monday morning, 17 September, 10:00

Lunch break

Monday afternoon, 17 September, 12:30

Parallel Session

Monday afternoon, 17 September, 13:30

13:45

Invited

Electric Power Generation from Sunlight by Mesoscopic Solar Cells

Michael Graetzel

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The recently discovered photovoltaic cells based on mesoscopic semiconductors are commonly referred to as "bulk" or interpenetrating network junctions due to their three-dimensional structure. These are formed for example from inorganic oxides, ionic liquids and organic hole conductors or conducting polymers, offering the prospect of low cost fabrication without expensive and energy intensive high temperature and high vacuum steps, compatibility with flexible substrates and a variety of presentations and appearances to facilitate market entry. Contrary to expectation, these new solar cells have shown strikingly high conversion efficiencies, competing with conventional systems. Mesoscopic injection solar cells operate in an entirely different fashion than conventional solar p-n junction devices. Mimicking the principles that natural photosynthesis has used successfully over the last 1.5 billion years in solar energy conversion, they achieve the separation of light harvesting and charge carrier transport. The semiconductors used in conventional cells assume both functions simultaneously imposing stringent demands on purity and entailing high material and production costs. The prototype of this family of devices is the dye-sensitized solar cell (DSC), which accomplishes the optical absorption and the charge separation processes by the association of a sensitizer or quantum dot as light-absorbing material with a wide band-gap oxide semiconductor of nanocrystalline morphology. Conversion efficiencies exceeding 11 % and impressive stability results have fostered first industrial applications. It appears that this type of cell is a viable

contender for large-scale future solar energy conversion systems on the bases of cost, efficiency, stability and availability as well as environmental compatibility.

14:15

Oral

Topologically controlled growth of colloidal semiconductor/magnetic nanocrystal heterostructures: site-selective functionalization of TiO₂ nanorods with either γ -Fe₂O₃ or ϵ -Co spherical domains

Marianna Casavola¹, Raffaella Buonsanti¹, Vincenzo Grillo², Elvio Carlino², Cinzia Giannini³, Miguel Angel Garcia⁴, Fabia Gozzo⁵, Liberato Manna¹, Roberto Cingolani¹, Pantaleo Davide Cozzoli¹

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The recognition of the dimensionality-dependent physical-chemical properties of nanoscale inorganic matter has stimulated efforts toward the fabrication of nanostructured materials in a systematic and controlled manner. Colloidal routes have now advanced to the point of allowing facile access to a variety of finely size- and shape-tailored nanocrystals (NCs) by adjusting thermodynamically and kinetically-limited growth processes in liquid media. While refinement of this synthetic ability is far from being exhausted, the increasing demand for multi-purpose nano-objects has recently oriented efforts of nanochemistry research to envision first prototypes of multimaterial hybrid nanocrystals (HNCs) with a topologically controlled composition.¹ Here, we describe surfactant-assisted seeded-growth approaches to fabricate elaborate noncentrosymmetric HNCs, which comprise semiconductor/magnetic sections fused together through small inorganic interfaces. The nanocrystal heterostructures are fabricated by heterogeneous nucleation of either γ -Fe₂O₃ or ϵ -Co domains onto anatase TiO₂ nanorod seeds.²⁻³ The latter can be decorated at either their tips or longitudinal sidewalls, attaining epitaxial heterointerfaces even in the presence of a large lattice mismatch between the connected crystal structures. Our results illustrate how the surfactant-modulated reactivity of the exposed TiO₂ seed facets, the interfacial strain induced at the relevant junction regions, and the occurrence of local lattice distortion fields interplay with each other at the nanoscale, ultimately dictating the final HNC topology. These studies suggest useful criteria for the rational design of novel generations of nanocrystal heterostructures with high structural complexity and increased functionality.

[1] P. D. Cozzoli, et al *Chem. Soc. Rev.* **2006**, 35, 1195.

[2] R. Buonsanti et al. *J. Am. Chem. Soc.* **2006**, 128, 16953.

[3] M. Casavola et al. *Nano Letters* **2007**, 7, 1386.

14:30

Invited

Flame aerosol synthesis of metal oxide nanoparticles

Sotiris E. Pratsinis

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Gas phase (aerosol) synthesis of ceramic nanoparticles is attractive as it does not require the multiple steps of wet chemistry processes and cleaning of liquid by-products resulting high purity products (e.g. optical fibers) while particle collection is easier from gas than liquid streams. As a result, the aerosol route is preferred in materials manufacture (pigmentary TiO₂ by the chloride versus the sulfate process) or in cleaning of process streams (gas versus wet scrubbing). Today flame aerosol technology, in particular, is used in large scale manufacture of fumed SiO₂, TiO₂ and Al₂O₃ nanoparticles up to several tons/hour.

It will be shown how characteristics (size, morphology and crystallinity) of metal oxide nanoparticles (e.g. SiO₂, TiO₂) can be controlled by flame variables (reactant mixing, composition and flowrate as well as external electric fields). Such understanding guides rational design and operation of aerosol reactors for the above commodities and enables now aerosol technology to develop new processes and products for catalysts, sensors, biomaterials, photonics and even nutritional supplements!

For example, noble metal bearing catalysts (e.g. Pt/Al₂O₃) that were made for eons by multi-step wet impregnation and costly effluent treatment are made now by one-step liquid-fed flame aerosol reactors. Likewise biomaterials for bone replacement and dental prosthetics are made such as transparent but radioopaque Ta₂O₅/SiO₂ nanocomposites. Highly efficient NOx storage-reduction Pt/Ba/Al₂O₃ catalysts are made by stereoscopically arranged twin flame reactors promoting component mixing at the nano rather than at the atomic level. Very recently, it was demonstrated gas-phase synthesis of sensors for organic vapors by one step synthesis of TiO₂ or Pt/SnO₂ nanoparticles and their direct deposition on Pt electrodes resulting in highly porous films. Hence all the demanding, impurity-prone steps of conventional sensor manufacture by wet processes are bypassed.

15:00

Oral

Structural and photocatalytic characterisation of pyrogenic WO_x-TiO₂ nanoparticles.

Katarzyna A. Michalow^{1,2}, Andri Vital¹, Kranthi K. Akurati¹, Katarzyna E. Zakrzewska², Giuseppino Fortunato³, Felix Reifler³, Axel Ritter³, Thomas J. Graule¹, Mieczysław Rękas²

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This work considers synthesis, characterization of structure and morphology of undoped, W-doped TiO₂ and WO_x/TiO₂ composite nanoparticles with focus on photocatalytic application.

Nanoparticles with defined W/Ti ratio in the range of 0.1-10at% W were produced by a flame spray process using diluted metal-organic precursors and metal salt solutions. Specific surface area of the powders was controlled by the precursor flow rate at a constant volume flow of oxygen (atomizing and oxidizing gas). The combustion stoichiometry in the flame influenced the crystallinity of the particles, oxidation state and structural arrangement of the constituents.

The specific surface area of the as-synthesized powders was between 40 and 140 m²/g depending on the process parameters and tungsten concentration. Transmission electron microscopy (TEM) showed that the powders consist of nanoscaled spheres. Anatase is the prominent phase, and X-ray photoelectron spectroscopy (XPS) revealed that the

valence of tungsten is related to the combustion stoichiometry in the flame. The band gap energy of the powders was determined by spectrophotometric measurements. The photocatalytic performance of the nanopowders under visible (400-500nm) and UV-A (365nm) light irradiation was studied for the degradation of methylene blue (MB) and methylene orange (MO) in aqueous suspension as well as decomposition of gas phase pollutants. The influence of W-concentration and specific surface area on the catalytic activity was determined.

We thank to the Polish Ministry of Science and High Education for the financial support, project no. R15 019 02.

15:15

Oral

Thin nanoparticle coatings generated by Liquid Flame Spray method

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In nanoparticle synthesis, gas phase methods, especially flame based methods, are widely used. Liquid Flame Spray (LFS) is one of those, the difference from other flame techniques being mainly the selection of combustion gases providing high temperature, hydrogen and oxygen, and their specific use in liquid atomization. The high temperature allows us to use a great variety of precursor materials. Presently, LFS is used in particle generation for several applications, mostly coating by particle deposition. The particle size in coatings can be adjusted with the process parameters.

One of the applications is fabrication of anti-microbial and self-cleaning surfaces. In this application, we generate TiO₂-Ag particles in one step process and deposit them directly on ceramic tiles. The coatings are then characterized using several analysis methods. Transmission Electron Micrographs show that the composite particles consist of TiO₂ (anatase) particles sized around 20 nm, with metallic Ag particles with the size of few nanometres on top.

Another application of LFS is its use in etching mask fabrication. SiO₂ nanoparticles with size around 100 nm, are deposited from the flame directly on a silicon wafer. Coatings with coverage of less than 100 % can be easily prepared using LFS technique. Here it is essential to have a small coverage and randomly distributed particles for the patterning of the wafer.

Glass colouring is one of the most fascinating applications where LFS method can be used. Metallic and oxide nanoparticles create a specific colour in glass, depending on the particle size. Glass can be coloured in many ways, both in bulk and on surface. The overwhelming property of LFS is that it generates a coloured thin layer on the surface of the glass, creating a visual veil effect. This kind of veil cannot be produced by any other method. The recent studies have concentrated on producing gold particles, commonly known to create red colour with particle size of 50 nm.

Coffee break

Monday afternoon, 17 September, 15:30

Joint Poster Session (Part I)

Monday & Wednesday

Monday afternoon, 17 September, 15:50

Tuesday, 18 September

Parallel Session

Tuesday morning, 18 September, 9:00

9:00

Invited

Ionic Liquid based Approach to Nanoscale Functional Materials

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Ionic liquids (ILs) in recent years have attracted considerable interest due to their exceptional features such as wide liquid range, excellent thermal stability, wide electrochemical window, and low vapor pressure. In between, ILs have gained significant impact on organic synthesis [1]. Aiming at nanoscale materials and advanced nanostructures, exploration towards a beneficial use of ILs has just begun [2].

Here, a microwave-assisted synthesis in ionic liquids is presented as a novel approach to functional nanomaterials. LaPO₄:RE (Ce, Tb, Eu) and In₂O₃:Sn (ITO) have been selected as examples to prove the effectiveness of the synthesis. As a result, non-agglomerated and redispersible particles, 10-15 nm (LaPO₄:RE) and 25-30 nm (In₂O₃:Sn) in diameter were realized. Since heating is performed via microwave irradiation, synthesis is very fast (10 s at 300 °C) and leading to highly crystalline and anhydrous oxide particles. While exploring the material properties, in case of LaPO₄:Ce,Tb (45 mol-%, 15 mol-%) quantum yields of 70 % (Tb-emission) and 90 % (Ce plus Tb emission) were determined [3,4]. With concern to In₂O₃:Sn (7 mol-%) subsequent to ink-jet printing and densification, transparent ITO layers, 500 nm in thickness were formed exhibiting a specific resistance of $6.8 \times 10^{-2} \Omega\text{cm}$. Neither a thermal nor a chemical post-treatment is required to realize these values. With the properties gained the nanocrystals exceed comparable materials realized by other liquid-based methods [5,6].

References

- [1] P. Wasserscheid, T. Welton (Eds.), *Ionic Liquids in Synthesis*, Wiley-VCH, Weinheim 2002.
- [2] M. Antonietti, D. Kuang, B. Smarsly, Y. Zhou, *Angew. Chem. Int. Ed.* **2004**, *43*, 4989.
- [3] G. Bühler, C. Feldmann, *Angew. Chem. Int. Ed.* **2006**, *45*, 4864.
- [4] G. Bühler, C. Feldmann, *Patent Application*, DE 10 2006 001 414.6.
- [5] G. Bühler, C. Feldmann, D. Thölmann, *Patent Application*, DE 10 2006 011 754.9.
- [6] G. Bühler, D. Thölmann, C. Feldmann, *Adv. Mater.* **2007**, in press.

9:30

Oral

Photoluminescence properties of nano-sized BaMgAl₁₀O₁₇:Eu phosphors by liquid phase precursor method

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A new method called the "Liquid Phase Precursor (LPP)" is presented for the synthesis of nano-sized bar shaped BaMgAl₁₀O₁₇:Eu (BAM) blue phosphor. A cellulose pulp is used as a template and a mixed water solution having a predetermined Bax(x=0.80-0.90)Mgy(y=0.91-1.06)Al₁₀O₁₇:Euz(z=0.096-0.115) composition is prepared with barium chloride, magnesium nitrate, aluminum nitrate and europium chloride. The pulp is impregnated by inserting it into the mixed solution. And then pulp including solution fired at 800°C in order to vaporize the pulp. Final firing is conducted at 1300-1450°C for 1 h in a reducing atmosphere (5%H₂ in N₂). Field Emission Scanning Electron Microscopy (FE-SEM), X-ray diffraction (XRD) spectroscopy and Vacuum ultraviolet photoluminescence (VUV-PL) were used to characterize the material. FE-SEM analyses showed that bar shaped homogeneous particles were formed ~60 nm in diameter and 300-500 nm in length. VUV-PL was measured with 147 and 254 nm excitation wavelengths; PL intensities of our specimen and commercial product were obtained at (x=0.14, y=0.52) and (x=0.14, y=0.72) from the CIE chromaticity color coordinates under 254 nm excitation wavelengths, respectively, and it was found that our sample showed a slightly higher normalized intensity (100%) than the commercial product intensity (98.9%). Comparing our product relative to the commercial product at the aforementioned CIE chromaticity color coordinates 80% PL intensity has been obtained.

9:45

Oral

New organic fuels with applications in the direct MgAl₂O₄ low-temperature combustion synthesis

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Starting from our previous results, which have shown that there is a predilection of metal nitrates with respect to certain fuels, the paper presents the exploitation of new organic compounds in the direct magnesium aluminate low temperature combustion synthesis, thus playing an import role in the diversification of the fuel range.

Mg(NO₃)₂·6H₂O and Al(NO₃)₃·9H₂O were used as oxidizing agents. Alongside the already well-known reducing agents, such as urea (U), glycine (Gly) or β-alanine (β-Ala), new organic compounds with nitrogen content have been tested: monoethanolamine (MEA), triethanolamine (TEA), trihydroxymethylaminomethane (THAM) and triethylenetetramine (TETA). The experimental results pointed out that, using a single fuel, the synthesis of a pure, well-crystallized spinel cannot be achieved unless subsequent thermal treatments are applied.

On the other hand, the use of optimum fuel mixtures (U + MEA, U + β-Ala, U + Gly) next to the desired metal nitrates allows the direct synthesis of pure highly crystalline spinel, without any additional calcination phase. In this way, a whole new perspective has been opened concerning the direct combustion synthesis of magnesium aluminate, in particular, and other oxide compounds in general, by eliminating the annealing phase, which is practically indispensable in most of the unconventional synthesis methods.

10:00

Oral

Microwave technique applied on the hydrothermal synthesis and sintering of calcia stabilized zirconia nanoparticles

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Calcia as well as di- or tri-valent oxides stabilized zirconia have received the most attention because of their high oxygen-ion conductivity which combined to their high thermochemical stability makes them as the best candidate for electrochemical devices, such as oxygen sensors, solid oxide fuel cells and oxygen pumps which operate at high temperatures. Main preparation methods of these compounds consist of solid state reaction and also sol-gel route, which require drastic or complex conditions. Hydrothermal treatment appears to be the more simple and less expensive method for the preparation of zirconia nanopowders characterised by high purity and high degree of compositional homogeneity which ensure an appropriate sinterability. The hydrothermal synthesis of calcia zirconia powders has been reported in few papers, never mentioning the possibility to have a microwave-assisted technique. This work is focused on synthesis zirconia nanopowders stabilized by 6 mol% calcium oxide prepared under hydrothermal conditions in combination, for the first time, with microwave technology. Microwave technology is, as diffusely reported, an advantageous tool for the efficient obtainment of inorganic oxides nanoparticles. Hydrolysis of calcium and zirconium nitrates followed by microwave irradiation to reach 200°C for 90 minutes was sufficient to obtain calcia stabilized zirconia as white powders. XRD analysis has evidenced the formation of nanocrystallites of tetragonal symmetry as the main phase with a size of ca 8 nm. The sintering of these nanopowders was performed under microwave irradiation on the sample in the form of a pressed pellet which contains polyvinyl alcohol as binder at a temperature of 1300°C in air. SEM has confirmed that the sample was successfully sintered and the grains have maintained their nanometric size very narrowly dispersed, ranged between 100 and 150 nm.

10:15

Oral

Nanoreactor Engineereng and SPS Consolidation of Nanostructured Multimetal Oxides

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Engineering of multimetal oxide-ceramic nanopowder with precise morphology and homogeneity of the compounds by multiple nanoexplosive technique of preliminary engineered nanoreactors will be de-

scribed. The concept of in situ engineering of nanoreactors has been suggested. Nanoreactors are engineered aggregates of complex intermediate metastable products of synthesis. This allows localization of the reactions within the volume of a single nanoreactor, which provides the heredity of the final structure of the nanosize product. Nanoreactors were produced by morphology optimization technique and loaded by colloidal impregnation of the C₃H₆N₆O₆ into the engineered matrix porous nano-aggregates of intermediate complex compounds. The subsequent initiation of C₃H₆N₆O₆ form gaseous products with a temperature of $\square$ 2500 °C. The short-term high temperature generated during each explosion enhanced the solubility of the components. The rapid evolution of a large volume of gaseous products dissipates the heat of the process and limits temperature increase, reducing the premature local sinterability among the neighbor particles; and leads to the fragmentation of the surrounding agglomerates. Uniformly aggregated nanosize CGO and 8Y-SZ and CGZYOX were synthesized. Low-temperature SPS consolidation of nanosize engineered aggregates of produced CGO, 8Y-SZ and CGZYOX allows production of nanostructured ceramic for electrolyte application. Ionic conductivity data for nano-grained CGO, 8Y-SZ and CGZYOX solid electrolytes with different average grain size and thickness were obtained and attributed.

Coffee break

Tuesday morning, 18 September, 10:30

Parallel Session

Tuesday morning, 18 September, 11:00

11:00

Invited

Bottom up approaches to Inorganic and Hybrid Organic-Inorganic Nanostructured Materials

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Inorganic and hybrid nano-composites materials can be obtained either through hydrolysis and condensation reactions of functional metal alkoxides or chlorides or through the assembly of well defined nanobuilding blocks (NBBs). The properties that can be expected for such hybrid materials of course depend on the chemical nature of their components but also on the extend and the nature of their interface. These hybrid NNBs can be clusters or functional nanoparticles which chemical control of their surface properties by using nucleophilic groups allows to tune their size and functionality. Moreover the use of texturing agents triggers the formation of tailor-made nano-phases. In particular, considerable effort is being currently directed to the synthesis of nanostructured porous transition metal oxides. The use of ordered lyotropic phases as templating agents leading to a mesoscopically ordered hybrid precursors allows to obtain long-range nanostructured hybrid or metal oxide phases shaped as thin films. These nanomaterials can be used as new platforms to develop composite nanolayers with sensing, catalytic or magnetic properties. Some examples concerning the design of inorganic and hybrid nanomaterials made via bottom-up approaches to create nanostructured ordered phases will be presented together with some of their properties.

11:30

Invited

“Soft Epitaxy”: Surfactant-mediated Generation of Iso-oriented Crystalline, Mesoporous Metal Oxide Layers

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Mesoporous metal oxide thin films have attracted significant attention due to their potential use in photocatalysis, redox reactions, sensing etc. The most straightforward preparation method involves sol-gel chemistry in combination with a suitable structure-directing block copolymer (evaporation-induced self-assembly, EISA), and heat-treatment leads to the corresponding crystalline mesoporous oxide. The nanocrystals in the pore walls usually are randomly oriented with respect to the substrate and the mesopores, while it would be desirable to achieve preferred 3D orientation of the crystallites. So far, single-crystalline substrates are generally required to achieve oriented crystallization. Therefore, it is commonly not expected that mesoporous films with crystallographic orientation can be formed from solutions. Here we report a general, surfactant-mediated methodology for the preparation of mesoporous films with uniaxial crystallographical orientation of the nanocrystals (MoO₃, Nb₂O₅, etc.) on non-crystalline substrates (glass), by dip-coating of ethanolic solutions of a metal oxide precursor (e.g. MoCl₅) and block copolymers of the KLE family (poly(ethylene-co-butylene)-*b*-poly(ethylene oxide)) [1]. Subsequently, the films are heated in air to initiate crystallization, and the KLE block copolymers leads to ordered mesoporous, crystalline films with a uniaxial crystallographic orientation of the nanocrystals. For instance, for MoO₃ the [010] planes are orientated parallel to the substrate. Systematic studies revealed that the effect occurs for oxides with highly anisotropic unit cells (MoO₃, etc.), which leads to crystallization perpendicular to the lattice planes of maximum polarizability, and the role of the surfactant is to mediate the polarization induced by the surface (“soft epitaxy”) [1]. We anticipate this approach to provide the basis for the generation of layers of more complex metal oxides.

[1] Smarsly et al. *Adv. Mater.* **2006**, 18, 1827.

12:00

Oral

Facile one-pot aqueous hybrid Inorganic/Organic TiO₂ nanoparticles synthesis by chemical design

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The attachment of organic compound onto the surface of metal oxide nanoparticles is usually the key factor in the design of new materials in the fields such as catalysis, optical devices or in the electronic area.

¹ In this presentation, we will first present syntheses of new well-defined molecular (homo- and hetero-metallic) precursors characterized by elemental analysis, NMR, Fourier transform infrared spectroscopy (FT-IR) and UV-visible spectroscopies. Their uses to crystalline surface-modified titania nanoparticles will then be described in an original one step, simple, efficient, low temperature (100°C) and aqueous sol gel synthetic methodology.^{2,3} This relevant process offers economical advantages and even convenience of being environmental friendly

compared to other chemical or physical methods because: (i) none organic solvent is used and (ii) the initial aqueous solution was used dozens of time. Additional benefits of this process are high and precise control of the organic loadings and a very good chemical stability. Elemental analysis, FT-IR, XDR, TEM, nitrogen adsorption-desorption isotherm (BET), UV-Visible spectroscopy, XPS and neutron scattering data will be addressed in order to fully characterize the nano-objects as well as the chemical interaction of the organic molecule onto the TiO₂ nano-particles. Preliminary results in peptidic coupling reactions, for oxidation catalysis³ and as efficient protection additive against UV-A and -B radiations⁴ will also be demonstrated.

1. See review: C. Sanchez, B. Julian, P. Belleville and M. Popall, *J. Mater. Chem.*, **2005**, *15*, 3559.
2. G. Goutailler, C. Guillard, S. Daniele, L.G. Hubert-Pfalzgraf, *J. Mater. Chem.*, **2003**, *13*, 342-346.
3. M. Beyrouty, S. Daniele, A.B. Sorokin, L.G. Hubert-Pfalzgraf, *New J. Chem.*, **2005**, *29*, 1245-1248.
4. S. Daniele, L.G. Hubert-Pfalzgraf, V. Guyot-Férreol, J.F. Tranchant, Patent FR 0508382.

12:15 Oral

Novel Efficient Routes to Encapsulated Zinc Oxide Quantum Dots and Nanoparticles From Well-Defined Organometallic Precursors

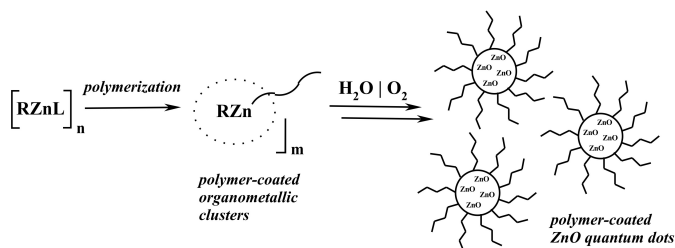
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The unique characteristics of zinc oxide makes it the most diverse inorganic material, with applications covering uses in catalysis and different fields of materials science and solid state physics. It is also widely recognized that the properties of the particles can be significantly different from the bulk materials. Therefore many efforts have been made to stabilize ZnO nanoparticles, including employing different organic ligands and initiating polymerization on nanoparticle surfaces.

In the course of our extensive studies on the modeling active sites in the oxygenation of zinc alkyls[1] as well as in the ring-opening polymerization of heterocyclic monomers,[2] we have developed mild procedures for generating capsules with ZnO quantum dots or its higher aggregates. Our advance involves the straightforward synthesis of an alkylzinc complex supported by a multifunctional ligand followed by its controlled treatment by air, which offers a finely tuned environment for ZnO chemistry. This methodology was extended to the synthesis of polymer-coated ZnO nanosize cages and particles through a three-step approach: the formation of well-defined catalytically active organozinc clusters, the polymerization process of heterocyclic monomers, and the oxygenation of the as-formed nanoparticles. The resulting nanoparticles with intense blue fluorescence can be redissolved by many solvents to form stable colloids that are convenient for further processing.



[1] J. Lewiński, W. Marciniak, I. Justyniak, J. Lipkowski, *J. Am. Chem. Soc.* **2003**, *125*, 12698. J. Lewiński, E. Bojarski, Z. Ochal, I. Justyniak, J. Lipkowski, *Angew. Chem. Int. Ed.* **2003**, *38*, 4643. J. Lewiński, W. Śliwiński, M. Dranka, I. Justyniak, J. Lipkowski, *Angew. Chem. Int. Ed.* **2006**, *45*, 4826.

[2] J. Lewiński, P. Horegląd, K. Wójcik, I. Justyniak, *Organometallics*, **2005**, *24*, 4588. J. Lewiński, P. Horegląd, M. Kościelski, I. Justyniak, J. Lipkowski, *Chem. Eur. J.*, in press.

Lunch break

Tuesday afternoon, 18 September, 12:30

Parallel Session

Tuesday afternoon, 18 September, 14:00

14:00 Invited

Some relevant parameters for the design of nanoparticles by aqueous chemistry

Corinne Chanéac, Jean-Pierre Jolivet, Sophie Cassaignon, Olivier Durupthy, David Chiche

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The chemistry of metallic cations in aqueous solution is a relevant method to the elaboration of nanometric scale metallic oxides. This method is extremely versatile and allows one to control many characteristics of materials like iron, aluminium and titanium oxides, in particular, the crystalline structure for polymorphic compounds, the particles size and shape. The main parameters being more relevant are the acidity of solution, the presence of specific anions, and the temperature. The synthesis process has been optimised in order to study each parameter separately. The acidity of the medium and the temperature govern the first steps of oxide formation namely the hydroxylation of metal complexes in solution involving the nucleation. Species complexing the cations modify the nature of coordination sphere of molecular complexes and play an important role on the size and the shape of solids, and sometime on the crystalline structure for polymorph. We have shown in the case of magnetite and anatase particles that the particles size decreases as the pH of precipitation departs more and more from the point of zero charge, PZC. For others materials such as brucite (Mg(OH)₂) and boehmite (g-AlOOH), the variation of morphology occurs. These examples bring decisive experimental arguments allowing to connect the size and shape of nanoparticles to the acidity of synthesis medium. Results have been interpreted using a thermodynamic approach of equilibrium state taking account the variation of the solubility and the surface energy as a function of acidity of the medium, through a simple semi-quantitative model. In order to improve this approach, we have investigated some experiments using weak complexing molecules and we have shown that the use of small quantity of polyols allowed also to tune surface energy for each crys-

tallite faces involving some change in aspect ratio for a given particle shape.

14:30

Oral

Boehmite Synthesis by Aqueous Precipitation using Organic Complexing Agents to Control Nanoparticle Morphology: Toward Enhanced γ -Alumina Catalyst Supports

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Transition aluminas, and especially the gamma type, are largely used as catalyst supports in refining and petrochemicals. Most studies focus on properties resulting from material texture and casting (specific surface, porous volume, pore shape and diameter). However, surface properties of alumina should be considered as well, as the catalytic activity is tightly related to the structure of exposed crystalline faces. As γ -alumina results from controlled thermal treatment of boehmite γ -AlOOH by a topotactic transformation, the nature of exposed crystalline planes is related to the starting material. Therefore, the synthesis of the oxihydroxide γ -AlOOH, and especially size and shape of these particles, is critical in determining the relevant surface properties.

We present an innovating way to control morphology of boehmite nanoparticles synthesized by soft chemistry routes. The use of organic complexing agents such as polyols, carboxylates, and hydroxy-carboxylates during synthesis causes a modification of usual morphologies observed, specifically resulting in an observed increase of (101) faces (from 45% without complexing agent to 65% in the presence of tartrate). For instance in the case of polyols, we show that the variation of nanoparticles aspect ratio depends on polyol carbon chain length and stereochemistry. Material characterizations were performed using novel methods such as x-ray diffraction powder pattern simulation and zetametry in addition to transmission electron microscopy. Crystal growth mechanisms were investigated through adsorption isotherms, solid-state NMR spectroscopy and molecular simulation. We will also present our first results concerning catalytic properties of γ -alumina synthesized from these boehmite materials. Finally, we expect that this method will provide a promising way for the design of γ -alumina catalyst supports.

14:45

Oral

Structural and morphological control of manganese oxide nanoparticles by aqueous precipitation. Toward improved electrochemical behavior versus lithium.

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Recent researchs in the field of lithium batteries and their performance improvement involve nanoscaled electrode materials [1]. Among them many oxides have been proved to present better behavior upon Li reaction in terms of rate capabilities [2] and reversibility [2, 3] when divided at the nanoscale. Manganese oxide nanomaterials are of particular

interest from a fundamental point of view to investigate structure and size effects on the electrochemical behavior owing to their numerous phases. However, tailoring the properties of such electrodes remains a challenge since it is closely related to the control of the structure and texture. Our group thus develops the synthesis of oxide nanoparticles by precipitation in aqueous medium at low temperature [4].

This contribution addresses a low temperature one-step aqueous route for precipitation of manganese oxides by two different methods: reduction of Mn^{VII} and $\text{Mn}^{\text{VII}}/\text{Mn}^{\text{II}}$ reaction. Pure Mn_3O_4 nanoparticles or γ - MnOOH manganite nanowires are synthesized by the first route. The second method leads to morphology control of MnO_2 type oxides. On one hand the morphology of the lamellar oxide birnessite $\text{K}_{0.19}\text{MnO}_2(\text{H}_2\text{O})_{0.75}$ can be modified. On the other hand, the growth of microporous cryptomelane $\text{K}_{0.11}\text{MnO}_{1.85}(\text{H}_2\text{O})_{0.75}$ nanowires is proved to proceed by coarsening or oriented attachment depending on the growth direction, providing new means to control morphology *via* the growth conditions. Finally, well defined nanoparticles are used to investigate structure and size effects on the electrochemical behavior versus lithium.

[1] Poizot, P.; Laruelle, S.; Grugeon, S.; Dupont, L.; Tarascon, J.-M. *Nature*, **407**, **2000**, 496.

[2] Sudant, G.; Baudrin, E.; Larcher, D.; Dunn, B.; Tarascon, J.-M. *J. Electrochem. Soc.*, **151**, **2004**, A666.

[3] Baudrin, E.; Cassaignon, S.; Koelsch, M.; Jolivet, J.-P.; Dupont, L.; Tarascon, J.-M. *Electrochem. Comm.*, **9**, **2007**, 337.

[4] Jolivet, J.-P.; Chanéac, C.; Tronc, E. *Chem. Commun.*, **2004**, 481.

15:00

Oral

Macroporous morphology of nanostructured Layered Double Hydroxides

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Recently, sacrificial template methods have been shown to offer an efficient way for the fabrication of nanostructured materials. Among all possible templates, self assembled colloidal crystals also called synthetic opals can be used for creating three dimensionally ordered macroporous structures with interconnected macropores.

In this work by using this strategy, we successfully synthesized three dimensionally macroporous Layered Double Hydroxides (LDH), a class of synthetic anionic layered clays, from successive impregnation cycles. Then metal salts precursors impregnated on polystyrene sphere closed packed array is converted by coprecipitation into the solid state LDH structure around the beads. The macroporosity is subsequently liberated by elimination of polymer beads either by dissolution or calcinations. The nanostructured LDH particles obtained display both macro and mesoporosity. The synthetic process has been followed by in situ PXRD, electronic microscopy and ^{27}Al NMR experiments. In varying the nature of the precursors, it is possible to change the composition of the as-prepared macroporous materials, which maintain the own structural and anionic exchange properties of LDH. Nevertheless, the pH conditions and the peculiar crystal growth of some LDH compositions limit the extension to all the compositions, since the precipitation are not under control. All these points will be discussed as well as the thermal decomposition of various macroporous LDH into macroporous mixed oxides.

15:15

Oral

Morphological dependence of electrodeposited ZnO-based semiconductors under different growth conditions

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Control of morphology, orientation and assembly properties of oxide nanostructures are of great importance for their implementation on technological devices. Electrochemical procedures permit the synthesis of ZnO films on different morphologies depending on both the solvent and the substrate used. Aqueous electrodeposition of ZnO produces discontinuous structures under the form of nanocolumns while using a non-aqueous solvent, like dimethylsulfoxide (DMSO), continuous films are mainly obtained. Obviously, both morphologies exhibit different properties. Continuous films are highly transparent due to the flatness of the surface which results in low reflectance values. On the other hand nanocolumnar ZnO structures obtained from aqueous solutions exhibit an intense photoluminescence. The results point out the advantage of using dimethylsulfoxide when uniform, oriented and highly transparent films are required while using aqueous bathes produce perfectly defined hexagonal ZnO columns which can be fully oriented by chosen a suitable substrate.

First results of in-situ transmittance measurements showing the kinetics of the growth mechanisms will be discussed.

Coffee break

Tuesday afternoon, 18 September, 15:30

Parallel Session

Tuesday afternoon, 18 September, 15:50

15:50

Invited

Tailored Nanoparticles and Nanocomposites for Materials and Life Science ApplicationsHorst Weller*University of Hamburg, Institute of Physical Chemistry, Grindelallee 117, Hamburg 20146, Germany**e-mail: weller@chemie.uni-hamburg.de*

This talk describes recent developments in the synthesis and characterization of semiconductor and metal nanoparticles and composite materials. Results on the growth kinetics and the control of size, shape and surface chemistry will be addressed. Examples for 2D and 3D self assembly of nanoparticles are given. We report on the formation of colloidal crystals from semiconductor and magnetic nanoparticles as well as on the formation of ordered films using various coating techniques. We also report on the use of nanoparticles for biological and medical applications which include a robust coating with biocompatible polymers, fluorescence labeling, and embedding nanoparticles in polymer vesicles for directed delivery. Examples on the improvement of magnetic resonance imaging using tailored superparamagnetic nanoparticles are given.

16:20

Invited

Synthesis and Biomedical Applications of Uniform-sized Oxide NanocrystalsTaeghwan Hyeon*Seoul National University, School of Chemical and Biological Engineering (SNU), Seoul 151-744, Korea, South**e-mail: thyeon@snu.ac.kr*

We developed new generalized synthetic procedures to produce monodisperse nanocrystals of many transition metals and their oxides without a size selection process. The synthesized nanocrystals include γ -Fe₂O₃, Fe₃O₄, CoFe₂O₄, MnFe₂O₄, NiO, and MnO. We report the ultra-large-scale (10s of grams) synthesis of monodisperse nanocrystals of magnetite, MnO, CoO, MnFe₂O₄, CoFe₂O₄, cube-shaped iron, ZnO, CeO₂ by the thermolysis of metal-oleate complexes. We synthesized uniform-sized nanocrystals of various oxides via non-hydrolytic sol-gel reactions. We synthesized spherical zirconia nanocrystals with high-temperature tetragonal crystal phase, uniform ZnO nanocrystals with cone, hexagonal cone, and rod shapes, and anatase TiO₂ nanorods with uniform diameter of 3.4 nm, CeO₂ nanowires as thin as 1.2 nm, Sm₂O₃ nanobeams with uniform thickness of 1.4 nm using non-hydrolytic sol-gel reactions. We developed various kinds of nanostructured materials for biomedical applications. We reported on the fabrication of monodisperse nanoparticles embedded in uniform pore-sized mesoporous silica spheres. The magnetic separation (guiding) was demonstrated, and they were also investigated as drug delivery carriers. We developed a simple, reproducible, and general method of preparing multifunctional nanoparticle assembled silica spheres. Magnetite nanoparticles along with other kinds of nanoparticles were simultaneously assembled on uniform silica spheres. We fabricated magnetic gold nanoshells consisting of gold nanoshells (for NIR photothermal therapy) that are embedded with Fe₃O₄ nanoparticles (for MRI contrasting agent), and conjugated them with cancer targeting agent (for targeting). We synthesized Ni/NiO core/shell nanoparticles and applied them to the selective binding and subsequent magnetic separation of histidine-tagged proteins. We developed a new nanoparticle-based T1 MRI contrast agent for *in vivo* molecular and cellular imaging.

16:50

Oral

One-Step Hydrothermal Synthesis of Cubic Nano-Assemblies Comprising Surface-Modified Octahedral Cerium Oxide Nanoparticles.Seiichi Takami¹, Tadafumi Adschiri², Yutaka Wakayama¹, Toyohiro Chikyow¹**1.** *National Institute for Materials Science (NIMS), 1-1 Namiki, Tsukuba 305-0044, Japan* **2.** *Tohoku University, 2-1-1 Katahira, Sendai 980-8577, Japan**e-mail: TAKAMI.Seiichi@nims.go.jp*

Recent progress in the synthetic methods of inorganic nanoparticles facilitates their application as building blocks for miniaturized devices. One of the possible strategies to construct such devices is the controlled preparation of two or three dimensional superlattices, that is, periodic arrays of nanoparticles. Even though most of them comprise simple spherical nanoparticles, a variety of periodic structures have been prepared by using binary components or designed chemical interaction. We believe that the use of polyhedral nanoparticles also widens the world of composed superlattices, because the polyhedral shape possibly enables the sterically restricted inter-particle interaction, leading to the unique coordination number and controlled assembly of nanoparticles.

We have studied hydrothermal synthesis of metal oxide nanoparticles that were chemically modified by carboxylic acids or amines. In this presentation, we report one-step hydrothermal synthesis of cubic nano-assemblies that were composed from surface-modified octahedral CeO₂ nanoparticles. Octahedral coordination of the primary CeO₂ nanoparticles plausibly led to quasi-bcc packing and built the cubic nano-assemblies, which had the different shape as the primary nanoparticles.

17:05

Oral

Synthesis, characterization and surface functionalization of nanoscale transition metal ferrites.

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The preparation of nearly monodisperse and highly crystalline nanoscale spinel-type transition metal ferrites by two novel non-hydrolytic approaches will be reviewed. These synthetic methodologies are based on the complexation of the transition metal ions with polyol molecules and the thermolysis of metal carboxylates in high boiling point organic solvents, respectively. Due to the surface layer of the absorbed organic molecules, the as-prepared nanoparticles are stable against agglomeration and can be easily dispersed in various solvents, either polar and non-polar. The size of the nanocrystals can be varied in a wide range by changing the reaction conditions, such as the nature of the reaction medium, reaction temperature and reaction time. A detailed characterization of the nanoparticles by using a wide variety of techniques, including powder X-Ray diffraction, IR spectroscopy, thermogravimetric analysis (TGA), transmission electron microscopy (TEM), ¹H-NMR spectrometry and X-ray fluorescence spectroscopy (XPS) was performed in order to elucidate the morphology and the properties of the variable-sized Fe₃O₄ nanoparticles. Additionally, the magnetic properties of the variable-sized Fe₃O₄ nanoparticles were investigated at various temperatures and under different magnetic fields. Both finite size and interparticle interaction effects were identified to influence the magnetic behavior of the nanosized particles. Furthermore, the high surface reactivity of the nanocrystals enables the attachment of gold nanograins to their surface with formation of colloidal Fe₃O₄/Au nanocomposites which are highly stable against separation and exhibit magnetic properties similar to those of the parent Fe₃O₄ nanocrystals. By chemically bonding bioactive molecules to the attached Au nanoparticles these novel nano-architectures open up new opportunities for the use of the magnetic nanoparticles as a platform for various biomedical applications.

Wednesday, 19 September

Plenary Session

Wednesday morning, 19 September, 9:30

Coffee break

Wednesday morning, 19 September, 10:30

Parallel Session

Wednesday morning, 19 September, 11:00

11:00

Invited

Producing metal oxide nanoparticles by the sonochemical, microwave and RAPET techniques.

Aharon Gedanken

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Producing metal oxide nanoparticles by the sonochemical, microwave and RAPET techniques.

Aharon Gedanken

Department of Chemistry and Kanbar Laboratory for Nanomaterials

Bar-Ilan University Center for Advanced Materials & Nanotechnology

Bar-Ilan University, Ramat-Gan, 52900, Israel

In the last few years we have employed three techniques for the fabrication of metal oxides nanoparticles. These methods are: Sonochemistry, Microwave dielectric heating, and RAPET (Reaction under Autogenic Pressure at Elevated Temperatures).

An introduction to these three methods will be presented in my presentation and examples will be given.

In the sonochemical method we will emphasize the difference in the products obtained from a precursor which is volatile solute (transition metal carbonyls) and a precursor that is a nonvolatile solute. The first precursor will yield an amorphous product because of the fast cooling rates obtained upon the collapse of the bubble whereas the latter yields a nanocrystalline product.

The Microwave reactions were conducted in a domestic microwave oven that underwent a modification to include a distillation column. We will present the antimicrobial effect of a metal oxide strongly dependent on the particle size. The mechanism of the bacteria killing will be discussed. Another example of doping one metal oxide in another for example doping MnO in ZnO is made easy by the use of microwave radiation.

For the RAPET section we will present results in which transition metal oxides encapsulated in a carbon shell are being produced. The precursors for these reactions are the corresponding alkoxides. The same RAPET reaction under identical conditions was also carried out between the poles of a strong magnetic field (10T). In certain cases the magnetic field has no effect on the products while for others a dramatic change is observed.

11:30

Oral

Silica-alumina mixed oxide nanoparticles with refractive index matched to polyurethane polymer matrix

Elisabeth Barna¹, Andri Vital¹, Christian Weiher¹, Michael Grotklaes², Wolfgang P. Meier³, Thomas J. Graule¹

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Nanoparticles are widely used as reinforcing fillers to optimise polymer properties such as tensile strength, scratch resistance and wear. An especially challenging field is the reinforcement of transparent polymers

without loss of transparency as the particles must not cause any scattering of light and thus have to be very small, monodisperse and/or feature a refractive index close or equal to the polymer matrix. Nanoparticles tend to agglomerate with decreasing size due to increasing Van der Waals forces. Disintegration of the agglomerates and homogeneous dispersion of the single particles in the polymer matrix is a time and energy intensive process. Therefore the approach to minimise loss of transparency by matching of the refractive index of the nanoparticles and the polymer matrix is very promising.

In the present work, the refractive index of $\text{SiO}_2/\text{Al}_2\text{O}_3$ mixed oxide particles was adjusted by variation of the composition. Silica-alumina mixed oxide particles with compositions from 0 to 100 wt% alumina were produced by the flame spray synthesis. Nanoparticles were characterised by means of BET method, XRD and TEM. The refractive index was determined using immersion oils and the Becke Line method. Particles were incorporated in two-component polyurethane clear coating systems and cured at 80 °C for one hour. Transmittance of the cured films was measured by UV/vis spectroscopy.

Nanopowders with refractive indices in the range of 1.460 to 1.760 with an accuracy of 0.002 were produced at rates up to 100 g/h. Up to an alumina content of 60 wt%, corresponding to a refractive index of 1.5080, the nanopowders were amorphous. The target refractive index of the polyurethane matrix of 1.512 was met with a mixed oxide composition comprising 30 wt% alumina. Coatings were prepared and characterised with 10 and 20 wt% mixed oxide particles, respectively, and with SiO_2 (Aerosil OX50) for comparison.

11:45 Oral

Versatility of Laser Pyrolysis for the Synthesis of TiO_2 Nanoparticles

Nathalie C. Herlin-Boime¹, Bruno Pignon¹, Véronique Guyot Ferreol², Thierry Pouget², Jean François Tranchant³, Hicham Maskrot^{1,3}, Monique Gervais³, Cecile Reynaud¹

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Nanomaterials have applications in various fields such as microelectronics, electromagnetism, optoelectronic and chemistry (biomedical, pharmaceutical, cosmetics...). In particular, pure TiO_2 nanoparticles have different properties according to their structure. For example, crystallisation in the anatase form favours photocatalytic activity as compared to the rutile organisation. Doped TiO_2 nanoparticles are also very useful for various catalytic reactions. Moreover, incorporation of heteroatoms may favour photocatalytic activity and absorption of solar light.

Different methods such as sol-gel chemistry and flame spray pyrolysis are often used for the synthesis of TiO_2 nanoparticles. At laboratory scale, the TiO_2 anatase form and various doped TiO_2 nanoparticles have been obtained by these methods. Laser pyrolysis offers some similarities with flame spray pyrolysis and the continuous production makes this method interesting in terms of larger production. It is based on the interaction of a powerful IR laser beam with a mixture of gaseous or liquid precursors, with one of them absorbing the laser radiation. It has been developed up to now mainly for the production of non-oxide nanoparticles (Si , SiC , Si_3N_4 ...).

Pure and doped TiO_2 powders have now been synthesised from laser pyrolysis, using a spray of TTIP (Titanium tetra isopropoxide) as main

precursor. The as-grown powders most often contain an amount of carbon which can be easily eliminated by annealing at low temperature under air. All the crystalline phase which can be identified in the powders can be attributed to the various phases of TiO_2 . This paper shows the possibility to produce in one step nanoparticles in the anatase or in the rutile form or in a mixture between the two phases by tuning the experimental parameters such as the laser power. First results illustrating the consequences of this structural evolution on the properties of nanoparticles will also be presented.

12:00 Oral

Preparation and luminescence properties of $\beta\text{-SiAlON:Eu}^{2+}$ green phosphor for high color gamut

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The structure of $\beta\text{-SiAlON}$ is derived from $\beta\text{-Si}_3\text{N}_4$ by equivalent substitution of Al-O for Si-N, and its chemical composition can be written as $\text{Si}_{6-z}\text{Al}_z\text{O}_z\text{N}_{8-z}$ (z represents the number of Al-O pairs substituting for Si-N pairs and $0 \leq z \leq 4.2$). $\beta\text{-SiAlON}$ has a hexagonal crystal structure and the P6_3 space group. In this structure there are continuous channels parallel to the c direction. During the last few decades SiAlON ceramics have drawn much attention since they can be sintered to full density with low amounts of glassy phase, resulting in good mechanical properties even at high temperature. Recently optical properties have also been studied. It was observed that SiAlONs doped with 3d or 4f transition elements can be used as pigments or as luminescent materials. The luminescence wavelength of an ion in a matrix is strongly influenced by the anion neighbors of the ion.

A green-emitting oxynitride phosphor powder $\beta\text{-SiAlON:Eu}^{2+}$ with composition of $\text{Eu}_{0.013}\text{Si}_{5.769}\text{Al}_{0.231}\text{O}_{0.205}\text{N}_{7.795}$ was prepared from a $\text{EuO-bSi}_3\text{N}_4\text{-cAlN}$ system using a by gas pressure sintering method. The powder mixture was synthesized at 1900 °C for 2 hrs in 0.9 MPa N_2 atmosphere. The prepared phosphor powder exhibits a hexagonally shaped morphology with the length of $\approx 4 \mu\text{m}$ and the diameter of $\approx 0.5 \mu\text{m}$. The crystallinity, surface morphology and chemical composition were presented. And moreover, not only photoluminescence (PL) properties were investigated but also quantum efficiencies and absorption were made an inventory of prepared $\beta\text{-SiAlON:Eu}^{2+}$ phosphor powders in a wavelength range from 300 to 600 nm. It can be excited efficiently over a broad spectral range between 255 and 520 nm, and has an emission peak at 540 nm with a full width at half maximum below 55 nm. It has a superior color chromaticity of $x = 0.32$ and $y = 0.64$. The internal and external quantum efficiencies of phosphor is 64 % and 52 % at $\lambda_{\text{ex}} = 303\text{nm}$, respectively.

Lunch break

Wednesday afternoon, 19 September, 12:30

Parallel Session

Wednesday afternoon, 19 September, 13:30

13:30

Invited

Metal Oxide Nanocrystals by Sol-Gel Transition in a Coordinating Environment: General Principles, Formation Mechanism and Applications

Mauro Epifani

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A metal oxide sol can be prepared very simply by conventional precipitation-peptization procedures. Is it possible to process the oxide species contained in the sol in order to obtain nanocrystals? A positive answer will be given to this question, with the condition that the metal oxide sol contains "small oxide species". It will be shown that the expression "small oxide species" implies a sol processing from simple inorganic precursors in order to avoid the usual precipitation of amorphous, hydrous oxides. When the corresponding sols are injected at 160°C in a coordinating environment, generally constituted by a solution of an amine in tetradecene, competition takes place between the two roles displayed by the amine: catalysis of condensation reactions between metal oxide species and growth hindering by bonding to the surface metal atoms. The result of such competition is the formation of oxide nanoparticles, whose crystalline nature depends on the synthesis temperature and on the particular system under investigation. The corresponding formation mechanism has been determined as a spatially confined, self-arresting sol-to-gel transition, where the metal oxide nanocrystals are formed during the first few minutes after the injection step. It will be shown a careful account of the investigation leading to such conclusion, that was carried out by FTIR, NMR, XRD and HRTEM analysis of the synthesis products obtained in a series of experiments on various systems. The process is extremely general, providing nanocrystals of ZnO, SnO₂, CeO₂, NiO, TiO₂, Ga₂O₃, In₂O₃, magnetic ferrites etc. By a suitable control of the injection environment, completely soluble, stable and processable nanocrystals can also be obtained. As an example of application, it will be discussed the processing of chemoresistive sensors based on SnO₂ and In₂O₃ nanocrystals and their outstanding gas-sensing properties towards a broad range of gases.

14:00

Oral

Multigram synthesis of quasi-monodisperse metal oxide nanoparticles through nonaqueous solution chemistry

Georg Garnweitner, Ninjabgar Tsedev

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In the last years, nonaqueous sol-gel synthesis techniques to metal oxide nanoparticles have been established as a clear alternative to aqueous methods, allowing the synthesis of many metal oxides as small and uniform nanoparticles. Their main advantage is better control over the particle formation, resulting in highly crystalline products already at comparably low reaction temperatures. Additionally, the organic solvent can function as surface modifier, making the addition of surfactants superfluous, which results in improved purity of the products, but also simplifies the synthesis, as the reaction system then consists of only the precursor and a solvent. Moreover, the precursor may be used in quite high concentration, which makes these approaches highly promising for the large-scale production of high-quality nanoparticles.

We show that the nonaqueous synthesis of nanoparticles can be carried out in multigram scale, allowing the synthesis of more than 20 g nanoparticles per batch. Despite the simple reaction system, the particles are practically monodisperse in size and shape, and are highly crystalline directly after the synthesis at moderate temperatures. Through simple and efficient surface modification treatments, they can be functionalized to ensure high stability and compatibility with solvents. Interestingly, low amounts of organic ligands were sufficient to enable the preparation of stable and transparent nanoparticle dispersions. We have investigated the organic reactions in the system during particle synthesis, which mainly involve ether condensation reactions, and studied the surface chemistry of the obtained nanoparticles to gain insight into the particle formation and functionalization processes. Additionally, to demonstrate the high suitability of the obtained nanoparticles even for elaborate and demanding applications, the fabrication of holographic thin film nanocomposites based on zirconia nanoparticles is presented.

14:15

Oral

Novel route for the mass production of nanomaterials for Solid Oxide Fuel Cells

ALEJANDRO MORATA¹, Guilhem Dezanneau², Anthony Chesnaud², Francesca Peiró¹, Joan R. Morante¹

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It is of strong interest in fuel cells fabrication to have a good control of raw materials properties and microstructure. An adequate synthesis method is, for instance, necessary to obtain high performance electrodes with enhanced conduction and gas exchange properties. Here, we present a new method in which cations dissolved in an aqueous solution are trapped inside a 3D chelating gel before fast drying. We prepared nanocrystalline powders of a wide panel of the state-of-the-art fuel cells materials such as: YSZ, CGO, Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_{4-d}, GdBaCo₂O_{5+x}, Nd_{1.9}NiO_{4+d}, La_{0.75}Sr_{0.25}Cr_{0.5}Mn_{0.5}O₃, and BaZr_{0.9}Y_{0.1}O₃. This very fast route allows preparing several tens of grams of nanometric powders in one batch and the feasibility of the process is almost independent from the cations involved in the reaction. The synthesized powders present nanometric crystal size (10–40 nm), narrow size distribution and the possibility of aggregate disagglomeration via soft ball milling, allowing better electrolyte consolidation or control of electrode morphology in fuel cells. We also made this method more attractive from the industrial point of view, by using a non toxic polymer and a non-explosive initiator. Finally, since the process is largely independent from the material formula, this method can be easily extended to other kinds of oxides such as ferroelectrics, CMR compounds or gas sensors...

1. A. Sin, P. Odier, *Advanced Materials*, **12** (2000) 649-652

2. A. Tarancón, G. Dezanneau, J. Arbiol, F. Peiró, J.R. Morante, *J. Power Sources* **118** (2003) 256-264

14:30

Oral

Synthesis and Characterization of Nanostructured Ceria Obtained by the Reverse Microemulsion Method

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Ceria (CeO_2) crystallizes in a very stable fluorite like structure, showing no phase changes up the melting point ($\sim 2700^\circ\text{C}$). Nevertheless, due to many structural defects formed in an activation procedure, it shows anionic conductance and catalytic activity in redox reactions. Doping of the ceria matrixes with Ge or Sm results in formation of very effective anionic conductors applicable as solid electrolytes in the intermediate temperature solid oxide fuel cells (IT-SOFC). Wide application of the cerium oxide materials demands developing of synthesis methods of the nanosized particles being prerequisite for high performance of these materials in their application as well catalysts as anionic conductors, due to participation of nearly whole bulk of such small particles. These attractive properties may be lost during calcinations or even prolonged work at elevated temperatures owing to sintering processes. That is why further searching for new preparation methods are pursued.

In the present work we propose modification of the reverse microemulsion method to obtain a nanosized particles being precursors for the nanostructured ceria crystallites. The parameters of the precursors decomposition were optimized in a series of experiments performed in a thermal analyzer measuring simultaneously mass changes (TG/DTG), thermal effects (DTA) and chemical composition of the evolved gaseous products with quadruple mass spectrometer (MS-EGA). Crystal structure and morphology of the obtained ceria particles were characterized using XRD, BET and SEM techniques. The average grain size obtained from micro-emulsion was estimated as equal to 8 nm, which is an order of magnitude lower than those obtained by the standard precipitation route - about 140 nm. The catalytic activity of the obtained ceria towards volatile organic compounds (VOC's) degradation was tested in benzene and ethylene total oxidation and discussed in terms of ceria samples structure and morphology.

14:45

Oral

Direct Synthesis of Cobalt Incorporated Mesoporous Silica SBA-15: Effects of the Source and Adding Sequence of Cobalt Salts

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Direct Synthesis of Cobalt Incorporated Mesoporous Silica SBA-15: Effects of the Source and Adding Sequence of Cobalt Salts

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Abstract

Highly ordered cobalt incorporated mesoporous silica SBA-15 (CoSBA-15) has been synthesized by single-step direct synthesis method. The degree of cobalt incorporation can be adjusted by variation of Si/Co molar ratio in initial synthesis gels. Mesoporous CoSBA-15 samples were characterized by powder X-ray diffraction (XRD), transmission electron microscopy (TEM), N_2 sorption, infrared spectroscopy (FT-IR), and Inductively coupling plasma with optical emission spectroscopy (ICP-OES) elemental analyses. The effect of cobalt salt adding sequence with respect to the addition of triblock copolymers and siliceous sources was firstly investigated in this study. It is found that most Co ions exist as isolated framework species at low cobalt concentration in synthesis solution ($\text{Si/Co} > 5$). At high cobalt concentration ($\text{Si/Co} \leq 3$), XRD data reveal that there is cobalt oxide nanocrystals exists either within the inorganic walls or within the SBA-15 channels. Cobalt species prefer to grow within the channels and formed nanoparticles with length of 50 ~ 100 nm when cobalt salts are added after addition of siliceous sources. Whereas, cobalt oxides grows within silica walls when cobalt salts are added before addition of siliceous sources. Controlled cobalt loading was possible to a maximum of 7.74 wt% by changing the cobalt source from chlorides to nitrates. No bulk cobalt oxide was detected outside the SBA-15 mesoporous silica particles.

15:00

Oral

Synthesis of nanocrystalline iron oxide particles by the esterification reaction in the iron(III)/acetate/alcohol system

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Among the various synthesis routes, solution-based or “wet” chemistry synthesis offers a very good control over particle size, morphology and phase composition of the final product. Forced hydrolysis, microemulsion technique, hydrothermal, sol-gel, polyol and non-hydrolytic syntheses are often utilized. In an earlier work we have shown that nanosize TiO_2 (*J. Mol. Struct.* 481 (1999) 645), V_2O_5 (*unpublished results*), Bi_2O_3 (*Matt. Lett.* 61 (2007) 709) and Fe- TiO_2 nanoparticle (*J. Sol-Gel Sci. Technol.* 30 (2004) 5) can be successfully synthesized using an esterification reaction between the acetic acid and alcohol. On this route a metal cation was homogeneously hydrolyzed by water molecules generated *in situ* as follows: $\text{CH}_3\text{COOH} + \text{ROH} \rightarrow \text{CH}_3\text{COOR} + \text{H}_2\text{O}$. In this work we are presenting new results relating to the synthesis of nanosize iron oxide particles by using the esterification reaction in the iron(III)/acetic acid/alcohol system. Synthesis started from a clear iron(III) organic solution that was stable at room temperature for months. However, at elevated temperature and under the autoclave pressure, a nanocrystalline iron oxide phase was obtained. Control of the phase composition, morphology, aggregation and particle size of obtained iron oxides was achieved by adjusting experimental parameters such as the type of alcohol, the molar ratio of iron(III)/acetic acid/alcohol and temperature. For instance, nanosize magnetite particles arranged in regular spheres were formed in the iron(III)/acetate/ethanol system in autoclave at 180°C (Fig. 1).

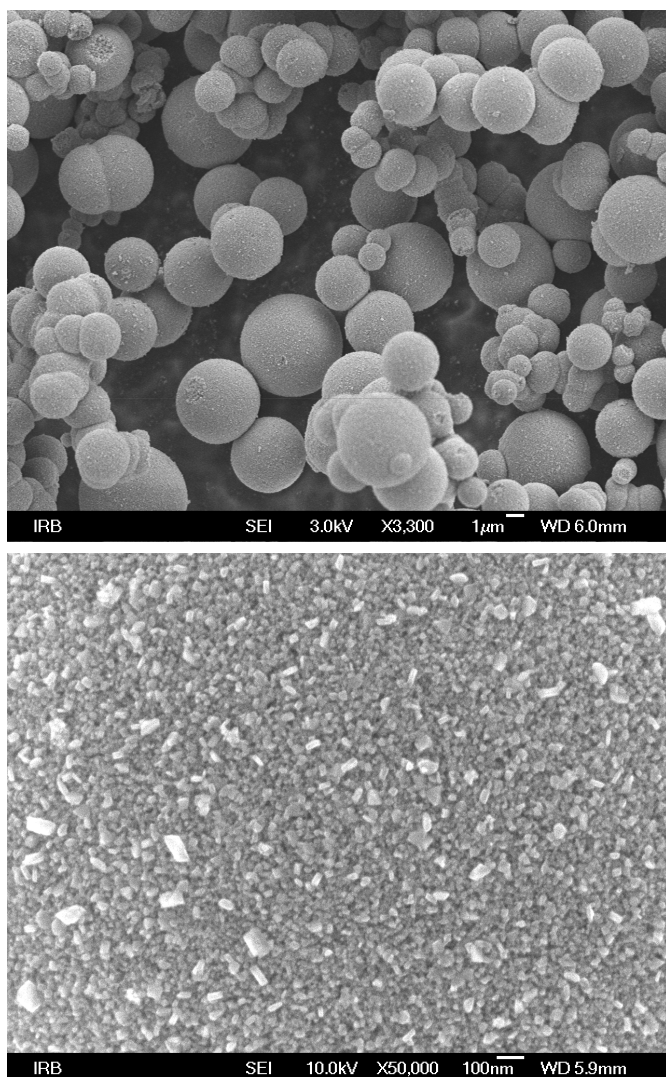


Fig. 1 FE SEM images of nanosize magnetite particles arranged in regular spheres and an enlarged detail showing discrete particles on the surface of one sphere.

Coffee break

Wednesday afternoon, 19 September, 15:30

Joint Poster Session (Part II)

Poster Award Ceremony

Wednesday afternoon, 19 September, 15:50

Thursday, 20 September

Parallel Session

Thursday morning, 20 September, 9:00

9:00

Invited

Doped ZnO: a case study for the influence of solvent and structure

Guyllhaine Clavel¹, Marc-Georg Willinger^{1,3}, David Zitoun², Nicola Pinna^{1,4}

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Transition metal doped ZnO have been theoretically predicted to be ferromagnetic at room temperature. The solution phase synthesis of ZnO usually leads to very anisotropic structures like rods or platelets. We have studied the influence of reactive medium on the oriented growth both in surfactant and solvent directed syntheses. Then, the influence of doping/alloying on the morphology has been studied as a function of the chemical nature and amount of the transition metal (TM) element. Obviously, the results show a very strong effect of the TM on the morphology with sometimes exotic shapes like multipods formed under specific conditions. Nevertheless, the systems surprisingly exhibit a very sensitive variation of the physical properties (in that case magnetic) as a function of the reactive medium.

9:30

Oral

Soft synthesis conditions of original shaped TiO₂ nanoparticles from TiCl₃

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TiO₂ is a popular material with large industrial production. This oxide presents several polymorphs including anatase, brookite and rutile. Each crystallographic phase exhibits physical properties, bandgap, surface states ... [1-2], the particle size is also an important parameter on the nature of the properties [3]. Anatase is commonly synthesized by hydrolysis of TiCl₄ or Ti(OR)₄ [4-6], and hydrothermal way are employed for rutile [9] and micrometric brookite [10]. In literature, few works were reported related to the use of TiCl₃ as a precursor, while it appears as an interesting compound as during the precipitation an oxidation step is necessary to obtain TiO₂. The hydrolysis of TiCl₃ was conducted in aqueous medium while varying synthesis parameters (pH, T, C, ...). The structural and morphological control was realized by the control of synthesis condition. Very original TiO₂ morphologies were obtained with very soft conditions. Brookite and rutile were obtained in the shape of nanometric platelets or rods, and nanorods of anatase was synthesized. This presentation is focused on the synthesis of TiO₂ polymorphs by TiCl₃ precipitation in water and on the influence of the synthesis parameters on the nature of the particles obtained. [1] M. Koelsch, S. Cassaignon, J.-F. Guillemoles, J.-P. Jolivet, thin solid Films, 2002, 403-404, 312 [2] H. Tang, K. Prasad, R. Sanjines, P.E. Schmid, F. Levy, J. Appl. Phys., 1994, 75, 2042 [3] R. Krol, A. Goossens, J. Schoonman, J. Electrochem. Soc., 1997, 144, 1723 [4] E. Matijevic, M. Budnick, L. Meites, J. Colloid Interface Sci., 1977, 61, 302 [5] B.L. Bischoff, M.A. Anderson, Chem. Mater., 1995, 7, 1772 [6] A. Pottier, S. Cassaignon, C. Chanéac, E. Tronc, J.-P. Jolivet, J. Mater. Chem., 2003, 13(4), 877 [7] H. Cheng, J. Ma, Z. Zhao, L.

Qi, Chem. Mater., 1995, 7, 663 [8] I. Keesmann, Z. Anorg. Allgem. Chem., 1966, 346, 30

9:45

Oral

A new chemical route to synthesise TM-doped (TM = Co, Fe) TiO₂ nanoparticles

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Diluted magnetic semiconductors (DMS) exhibiting ferromagnetic behaviour above room temperature are considered key materials for the development of spintronic devices. Among them, TM-doped (TM = Co, Fe) TiO₂ based oxides are considered very promising materials to achieve such goals.

In this work we report a new and swift chemical route to synthesise nanoparticles of such oxides. The synthesis of Ti_{1-x}TM_xO₂ (0 ≤ x ≤ 0.10) anatase phase nanocrystallites was achieved via precipitation of acid solutions of TiCl₃ and TM chlorides with ammonia followed by thermal treatment in autoclave with different suspension media. The Ti_{1-x}TM_xO₂ nanoparticles can be easily stored in suspensions and straightforwardly retrieved by centrifugation whenever necessary. Using such facility, thin films of Ti_{1-x}TM_xO₂ onto Si (100) substrates were prepared by dip- and spin-coating technique.

Both TM-doped TiO₂ nanopowders and films were characterised by X-ray diffraction (XRD), electronic microscopy (TEM and SEM), electronic dispersive spectroscopy (EDS), atomic force microscopy (AFM) and absorption spectroscopy.

Acknowledgments: O.C. Monteiro thanks Fundação para a Ciência e Tecnologia (FCT) for a pos-doc grant (SFRH/BPD/ 14554/2003). This work is supported by FCT, POCI and co-financed by FEDER (POCI/CTM/57019/2004)

10:00

Oral

Spectroscopic Properties of adjoined TiO₂ Nanocrystals

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To include particle attachment and porosity in the discussion of the electronic properties of nanostructured oxide materials is indispensable for deeper insights into electronic conduction across grain boundaries and thus essential to electronics, sensor technology and photovoltaics. We investigated the aggregation of isolated TiO₂-nanocrystals which results from the application of a simple hydration-dehydration cycle. After contact with water and subsequent dehydration and adsorbate removal under high vacuum conditions the powder containing isolated nanocrystals [1] is transformed into a monolithic solid which consists of a mesoporous particle network. These monoliths show significant

changes in the optical absorption properties as investigated by UV-diffuse reflectance spectroscopy. Vacuum annealing at temperatures T > 870 K induces oxygen anion vacancy formation on isolated TiO₂-nanocrystals. The electrons left behind form paramagnetic states which can be tracked by electron paramagnetic resonance (EPR) spectroscopy. On aggregated TiO₂-nanocrystals EPR measurements reveal that vacancy formation occurs at significantly lower temperatures than T = 870 K. In addition, polarizable conduction band electrons [2] have only been observed in the network of adjoined TiO₂-nanocrystals [3]. Changes in the spectroscopic properties resulting from solvent-mediated particle aggregation and particle network formation as well as the facilitated defect formation process will be discussed in the light of the associated structural data.

[1] T. Berger, M. Sterrer, O. Diwald, E. Knözinger, Chem. Phys. Chem. 2005, 6, 2104

[2] E. Serwicka, M. W. Schlierkamp, R. N. Schindler, Z. Naturforschung 1981, 36a, 226

[3] M. J. Elser, T. Berger, D. Brandhuber, J. Bernardi, O. Diwald, E. Knözinger, J. Phys. Chem. B 2006, 110, 7605

10:15

Oral

Chemical Control of Photoexcited States in Titanate Nanostructures

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Layered TiO₂-based nanostructures have attracted much attention due to a broad spectrum of potential photochemical and electrochemical applications including photocatalysis, solar cell technology and gas sensing devices [1]. The photochemical activity of these materials depends on the branching ratio between essentially three pathways photoexcited states can undergo: they can either recombine under photoluminescence emission or generation of heat, or become persistently trapped [2] or undergo interfacial charge transfer at the particle surface. Some of these competitive processes can be tracked by means of electron paramagnetic resonance and photoluminescence spectroscopy.

We synthesized titanate nanowires and -tubes by TiO₂ anatase powder treatment in aqueous alkaline solutions. The resulting structures are composed of layered sheets which consist of edge-sharing [TiO₆] octahedra [3]. A specific photoluminescence process which originates from the deactivation of exciton states trapped in [TiO₆] units was observed. It was found that exchange of intercalated ions provides means to adjust the branching ratio between radiative exciton deactivation, on one hand, and charge separation, on the other. Thus, chemical control over the photoelectronic properties of layered oxide structures has become feasible and the role of various ions in between the layered sheets will be discussed [4].

[1] Mor, G. K.; Shankar, K.; Paulose, M.; Varghese, O. K.; Grimes, C. A.; Nano Lett. **2006**, 6, 215

[2] Berger, T.; Sterrer, M.; Diwald, O.; Knözinger, E.; Panayotov, D.; Thompson, T. L.; Yates, Jr, J.T.; J. Phys. Chem. B **2005**, 109, 6061

[3] Orzali, T.; Casarin, M.; Granozzi, G.; Sami, M.; Vittadini, A.; Phys. Rev. Lett. **2006**, 97, 156101

[4] Riss, A.; Berger, T.; Grothe, H.; Bernardi, J.; Diwald, O.; Knözinger, E.; Nano Lett. **2007**, 7, 433

Coffee break

Thursday morning, 20 September, 10:30

Parallel Session

Thursday morning, 20 September, 11:00

11:00

Invited

Synthesis by sol-gel method of nanocrystalline compounds within $Y_2O_3(Nd_2O_3)-Al_2O_3$ system for optical applicationsLudwika Lipińska¹, Agnieszka Rzepka¹, Detlef Klimm², Witold Ryba-Romanowski³, Anna Pajaczkowska¹

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In the $Y_2O_3(Nd_2O_3)-Al_2O_3$ system there are some compounds, very useful for optical applications. These are as follows: $Y_3Al_5O_{12}$ (YAG), $YAlO_3$ (YAP) and $Y_4Al_2O_9$ (YAM). Also, Y_2O_3 (YO) doped by rare earth cations exhibits luminescent properties. All the above mentioned oxides in the form of nanocrystalline powders may be applied as stable phosphors or materials for laser ceramics. In this work, we have succeeded in preparing of YAG, YAG substituted by Nd, YAM and YO by the modified sol-gel method from aluminum nitrate, rare earth oxides and complexing compounds. All the substrates were carefully solved in acetic acid solutions and then - after the solvent's removing - thermally treated. The obtained nanopowders had crystalline structure confirmed by X-ray diffraction (XRD). The size and morphology of nanopowders were analyzed by scanning electron microscopy (SEM). The mean size of crystallites was evaluated using Scherrer formula and the sizes increased with the calcining temperature from 10 to 75 nm. It's worth mentioning that YAG can be doped by neodymium up to 27,5% without destroying the garnet's structure. This value is considered to be the highest known level of yttrium substitution by neodymium in YAG. Highly doped powders (15, 25, 27,5 % Nd) were examined by thermal analysis of DTA. Luminescence spectra of YAG:Nd powders were recorded using Dongwoo Optron Fluometer system. To conclude, the increase of Nd^{3+} concentration in YAG over 10 at. % causes very strong self-quenching of the Nd emission. Unexpectedly, the lifetime for the powder YAG sample containing 1% of Nd^{3+} is longer than the reported lifetimes for a YAG monocrystal samples. The yttrium oxides doped by neodymium and ytterbium ions were one-phase and of nanocrystalline structures. Spectroscopic examination of these nanopowders revealed occurrence of intense emission in near infrared region.

This work was supported by the Ministry of Education and Science under the research project No. 3T11B00430.

11:30

Oral

Tuning the nanostructuration of Nb_2O_5 oxides for specific optical non linear propertiesAnne Teissier^{1,2}, Valérie Keller Spitzer¹, Jean-Pierre Moeglin², Yves Lutz²

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With the development of high-power laser sources, the study and design of optical limiting systems that protect fine components like eyes and sensors have gained importance. The hazard, due to the narrow divergence of the laser beam and the resulting tightly-focussed spot on the sensor requires a protection in some regions of the spectra, including the visible and infrared domains. This protection could be thought as a material that rapidly interrupts or limits the transmission of the beam to a safe level when reaching very high intensities.

Nanomaterials like nano-oxides should be promising candidates for limiting materials. Recent studies have shown that carbon nanotubes exhibit efficient properties for limiting systems in the near infrared domain. The aim of our study is to develop a protective system in the far infrared domain, using nano-oxides synthesised via sol-gel methods, allowing to obtain advanced and functional powders and coatings of high optical quality. Sol-gel process exhibits the advantages of being an easily modified synthesis of nano-oxides making possible to tune many structural and morphological parameters. Our aim is to correlate the optical response of these nanoparticles at the nanoscale as functions of these various preparation parameters.

Niobium(V) oxide (Nb_2O_5) powders were prepared by the sol-gel procedures from niobium ethoxide, $Nb(OC_2H_5)_5$.

Non linear optical limiting properties of these oxides were investigated using the Z-scan method which enables to measure both the nonlinear refractive index and absorption coefficient.

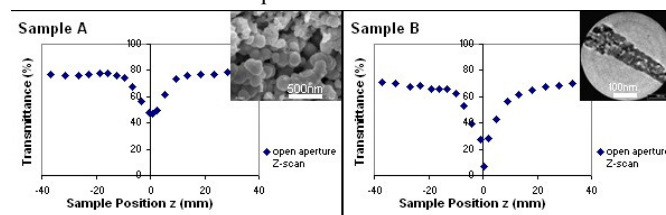


Fig. 1 Sample A : Z-scan results and SEM Fig. 2 Sample B : Z-scan results and TEM

These nano-oxide Nb_2O_5 materials show interesting optical non linear properties (fig 1 and 2) depending on the structural, compositional and morphological nanoscale characteristics, which can be finely tuned using different modifications and variations of the Nb_2O_5 sol-gel synthesis.

11:45

Oral

Hybrid organic-inorganic sol-gel coatings with high laser damage thresholdNicolas Marchet, Philippe Belleville, Philippe Prené

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For fifty years, a considerable effort has been and is still being directed to the production of optical coatings using liquid deposition route. The process investigated at CEA (French Commission for Atomic Energy) is mainly developed to prepare coatings using colloidal oxide-based and/or inorganic/organic hybrid materials in compliance with high power laser requirements.

The CEA megajoule-class laser so-called LMJ, needs 7,000-m² of coated area onto 10,000 large-sized optical components. For use on highly-reflective (HR) component, a specific unstressed multilayer coating has been developed and deposited onto deformable adaptative substrate (Ph. Belleville, J. Sol-Gel Sci. & Tech., 19, 2000). This HR-coating is made of colloidal-based low index and hybrid-based high index thin film coatings with adaptable refractive index depending on the molar ratio of organic and inorganic compounds. Two metallic oxides (AlOOH and ZrO₂) and six organic polymers have been selected and evaluated. The synthesis using sol-gel process of these oxides enables the control of the morphology and size of nanoparticles and suitable organic solvents have been chosen to allow organic polymer dissolution and preparation of uniform coatings. All materials have been selected regarding optical and laser damage properties using tests at 351 nm and 1053 nm wavelength.

Compatibilization of the hydrophilic surface of oxides nanoparticles with the hydrophobic organic polymers has been obtained using adapted functionalization of particles. We showed that it is possible to homogeneously disperse oxide nanoparticles in organic solvent using specific grafting agents like alkoxysilanes or carboxylic acids leading to time-stable sols. And also those high laser damage-resistant coatings can be prepared without any loss of optical properties. To demonstrate optical stacking feasibility, a multilayer coating has been prepared, showing optical reflectivity in good agreement with optical fitting model.

12:00 Oral

Structural Effects of codoping of Nb & Sc on TiO₂ Nanoparticles

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There is a growing interest in the application of nano materials for cleaning and detoxification of surfaces. TiO₂ is one of the oxides that has received considerable attention for organic decontamination owing to its high photocatalytic activity, biological immunity and low cost.

The paper describe the synthesis and characterization of TiO₂ nanosize particles doped with 0.5 at %, 0.75 at % and 1 at % Nb and Sc each, prepared by sol-gel technique. After drying, as synthesized samples were calcined at temperature ranging from 250 to 900 °C. Changes in phase transformation of the doped samples with reference to dopants concentration and calcinations temperature have been studied.

The samples were analyzed by various analytical tools such as X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), X-ray Photoelectron Spectroscopy (XPS) and Energy Dispersive X-ray Spectroscopy (EDS).

I

12:15

Oral

Silica-based sol-gel materials doped with transition metal oxide nanoparticles

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Transition metal oxides are of interest for fabrication of optical and ceramic materials, catalysts, media for magnetic recording, etc. The materials by the type of "nanoparticles-in-a-matrix" provide possibility to control their features through a tuning of chemical composition, particle size, and particle-matrix interaction. The complicated and variable chemical composition may be attained successfully with the sol-gel technique used for fabrication of the oxide-based matrix. However, there is no complete understanding to date for the systems of metal oxide nanoparticles for the elements of variable valence forming compound with complex stoichiometry (e.g., Cu, Ni, Co, Fe, Mn, Cr, V). Phase- and intervalence transitions become more featured in the case of nanoparticles and small clusters. We use the silica matrices possessing both good dielectric properties, low chemical reactivity and optical transparency. Nanoparticles composed from metal oxides within silica matrix were produced by the doping with metal compounds in the course of different steps of sol-gel process followed by a heat treatment in oxidizing/reducing atmosphere. In general case, the oxides formed were not separated from the matrix and incorporated into the silica network either as nanoparticles or as amorphous components. The final result of this interaction strongly depends on the nature of metals and chemical reactivity of oxides with respect to silica. Both thin film materials and bulk glasses have been fabricated those comprised the stabilized metal oxide nanoparticles. We use XRD, XPS, TEM, SEM, UV-Vis-NIR optical spectroscopy and photoluminescence methods to elucidate chemical state and size effects of the nanoparticles. Most featured results are reported for the systems with oxides of Cu, Co, and Fe. They are of importance as non-linear optical elements.

Lunch break

Thursday afternoon, 20 September, 12:30

Parallel Session

Thursday afternoon, 20 September, 13:30

13:30

Invited

Magnetic oxide nanoparticles: Large scale preparation, magnetism, and core/shell architectures

Ram Seshadri

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I will review current high temperature methods of preparing capped magnetic oxide nanoparticles - mostly with the spinel structure - using solvothermal chemistry or high-temperature solvents, and the thermolysis of precursors. The properties of the materials so obtained have been carefully characterized from the viewpoint of structure and magnetism, including using synchrotron and neutron diffraction. The magnetic properties of high quality nanocrystals depend strongly on inter-particle contacts. No evidence for a magnetic "dead-layer" is found in high quality/high temperature preparations, as opposed to aqueous solution preparations. The possibility of core/shell architec-

tures, including exchange biased and exchange spring magnets are explored, as are novel systems for magnetocapacitance studies.

14:00

Oral

The influence of the substitution material on the ferrite nanoparticles prepared by chemical method - Mössbauer studies

Beata Kalska-Szostko¹, Dariusz Satuła², Krzysztof Szymański², Ludwik Dobrzyński^{2,3}

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The nanotechnology is a new branch of science which is one of the most exciting in the world in last decades. The new materials with novel properties are discovered every day. Many scientists are working on the improving useful physical and chemical properties of the nanomaterials. The unique strength, capacitance, tribological and biocompatibility are the driving force for the nanoscience. The tunable composition brings the tunable properties of the nanoobjects. The paper presents changes in the magnetic properties of the ferrite nanoparticles of the size around 15 nm by the changing the Me/Fe ratio in the magnetite based materials. The used materials as a substituents are Mn, Co, Ni.

It is shown that the substitution Me is present in the A and B positions depending on the kind of the metal and also depending on the concentration of the substitution elements. At low concentrations, Co and Ni atoms occupy B position while Mn are found in both, A- and B-sites. In all three cases a doublet shows up in the middle part of the spectra. Its intensity in the case of Co rather smoothly increases with increasing cobalt content. This is not the case of Ni and Mn for which non-monotonic variation is observed. In the case of the nominally 100% substitution by Ni pure superparamagnetic state is observed, while for Co and Mn some of the particles are still below blocking temperature.

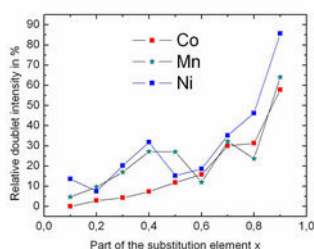


Fig. 1. Plot of the influence of amount of the substitution on the doublet intensity dependence

14:15

Invited

SAXS Characterization of Mesoporous Thin Films: A Brief Review

Aleksandra Turković

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Nanostructured materials are hierarchically structured on different length scales down to the atomic or grain size level. Electromagnetic

radiation can be used to obtain information about materials with dimensions of the same order as the radiation wavelength. Small-angle X-ray scattering (SAXS) experiments generally fulfil these requirements; irradiate a sample with X-rays, measure the resulting scattering pattern, then determine the structure that caused the observed pattern. SAXS is used to explore microstructure on the nanometer scale. Grazing-incidence X-ray reflectivity (GIXR) can provide detailed information about the thickness of single layer, multilayer periodicity and thickness of the individual layers that make up a multilayer. The full potential of these techniques is realised when using a synchrotron source and when patterns are recorded with two-dimensional detectors.

Titanium dioxide, titanium oxide based gels, cerium dioxide and Ce/Sn oxide films, vanadium oxide and V/Ce oxide films on a glass substrate were obtained by sol-gel process. The hydrogenated amorphous silicon and silicon-carbide thin films were deposited by magnetron sputtering and PECVD (Plasma Enhanced Chemical Vapour Deposition) in a wide range of hydrogen and carbon concentration. Mesostructured vanadium-oxo-based films and lamellar structure of silica/organic nanocomposite were prepared by evaporation induced self-assembly. The structure, morphology and phase transitions of these nanostructured and mesoporous films was studied by synchrotron radiation SAXS and grazing-incidence small-angle X-ray scattering (GISAXS). Layer structure in V/Ce oxides was revealed by grazing-incidence X-ray reflectivity (GIXR) method.

14:45

Oral

A Novel Method for the Determination of Nanoparticle Size and Shape based on X-Ray Powder Pattern Simulation: Application to Boehmite AlOOH

David Chiche^{1,2}, Mathieu Digne¹, Renaud Revel¹, Corinne Chanéac², Jean-Pierre Jolivet²

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The control of oxide nanoparticles morphology is of great importance to tailor their physical and chemical properties. As a consequence, routine methods are required to determine the nanoparticles size and shape. A novel method for morphology determination based on X-ray powder diffraction (XRD) is proposed. Morphology-dependent diffraction patterns are simulated using the Debye formula and compared to the experimental pattern. The Debye formula is a general law, allowing to calculate diffraction pattern of any atoms cluster. The most representative particle morphology is thus obtained. The methodology is applied to three different boehmite AlOOH samples: the effect on the synthesis pH value on the particle morphology is clearly evidenced. Further, the method is characterized in term of sensitivity. Extensive comparisons with transmission electron microscopy (TEM) observations are also performed: the results appear as consistent. The XRD method is especially efficient for the smallest nanoparticles (about 3-7 nm): in this case, TEM observations only provide average diameters of the particles, whereas the XRD approach leads to a full and accurate determination of the particles morphology. The developed method can be easily extended to a wide range of nanomaterials.

15:00

Oral

Formation of hierarchical porous structure at mechanochemical treatment of powder vanadium-containing oxide composition

Krystyna Wieczorek-Ciurowa¹, Svitlana Khalameida², Volodymyr Sydorchuk², Jadwiga Skubiszewska-Ziemia³, Valeriy Zazhigalov², Roman Leboda³

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Mechanochemical treatment (MChT) is effective and modern method for synthesis and modification of nanodispersed materials (adsorbents, catalysts e.a.). Increase of specific surface area (SSA) of treated samples takes place during MChT on air as a rule. In our opinion porous structure can be formed at MChT of nonporous matters in liquid mediums, first of all in water. This aspect is not examined in literature at all. High-energy MChT (700 rpm) in water and ethanol was used for milling of coarsely dispersed crystal powders of V-containing oxides. Intensive mechanical influence, higher temperature and pressure promote increased solubility of solid on the one hand and creation of contacts between crystals on the other hand. It results in formation of hard framework of porous material during next drying of treated dispersion. For investigated oxide compositions samples with hierarchically organized porous structure were prepared: primary large crystals form macroporous part and mesopores are created by means of precipitation of dissolved substance in places of contacts of crystals. Porosity of obtained at MChT samples was studied with the help of nitrogen adsorption, mercury porometry, SEM, AFM. Experimental data are following: system V-Mo-O – SSA=2-20 m²/g, pore volume V=0,10-0,60 ml/g, predominating diameter of mesopores – 50 and 100 nm, macropores – 500-600 nm; system V-Mo-Zr-O – SSA=12-40 m²/g, V=0,32-0,53 ml/g; system V-P-O modified by oxides Mo and Zr at MChT – SSA=7-38 m²/g, V=0,30-0,53 ml/g. Oxide compositions prepared by means of MChT in liquid phase with biporous structure are perspective as catalysts.

15:15

Oral

Little is different concerning titanium dioxide

Armin Feldhoff¹, Cecilia Mendive², Thomas Bredow³, Detlef Bahnemann²

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At the heart of photocatalysis lies the recognition that chemical reactions are catalyzed on specific surfaces of catalyst particles. Profound knowledge about the relationship between processing conditions and the abundance of specific crystal facets would allow for tailoring improved catalysts that show predominantly those surfaces that are catalytically most active for specific reactions. Today, titanium dioxide, in the crystalline modifications anatase and rutile, is by far the most important photocatalyst material. There is some evidence that small

crystals prefer to adopt the anatase modification, and large titania crystals are thermodynamically favoured if they have the rutile structure. Therefore, rutile surfaces have been studied extensively on macroscopic crystals, while the knowledge about specific anatase surfaces is less of experimental than of theoretical nature. Here, we present a methodical approach that combines geometric estimations by transmission electron microscopy (TEM) and the elucidation of detailed three-dimensional shapes of anatase nanoparticles by a Wulff-type construction [1]. Relative and absolute surface areas of different crystal facets are estimated, surface-to-volume ratios are deduced and compared to integral measurements of BET surfaces. Facets of the type (101) and (001), which are widely assumed to make up the majority of exposed facets on anatase particle surfaces, were found to be in the minority. Surfaces of commercial powder nanoparticles under investigation are rather dominated by facets of the type (100) or (111), and they show also (110), (112), (102), (103), (104), (106), and (108) facets, depending on the particle shape. The investigations clearly show that the shape of naturally occurring macroscopic anatase is not the key to a better understanding of nanocrystalline materials (i.e. “little is different”).

Reference

[1] A. Feldhoff, C. Mendive, Th. Bredow, D. Bahnemann, *ChemPhysChem* 8, 805 (2007)

Coffee break

Thursday afternoon, 20 September, 15:30

Posters

Monday, 17 September

Joint Poster Session (Part I)

Monday & Wednesday

Monday afternoon, 17 September, 15:50

15:50

Poster

A01

Effects of Applied Voltages on the Electronic Properties of TiO₂ Electrodes and the Photocatalytic Water Splitting

Shin-Tae Bae¹, Jin Young Kim¹, Sangwook Lee¹, Dong Wook Kim¹, Hyunho Shin², Kug Sun Hong¹

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Applied voltage between anode (TiO₂) nanoparticles and cathode (Pt) in a water photolysis reactor drastically increased water photolysis-induced photocurrent and hydrogen production. The amounts of hydrogen production increased by 30 times with a small applied voltage of 1.5 V. The notable increase of hydrogen production was explained in terms of the decreased Schottky contact potential barrier at indium tin oxide (ITO)/TiO₂ by the applied voltage, while at high voltage regime, the alteration of the Helmholtz layer by the applied voltage further modifies the voltage-assisted increase of the photocurrent.

15:50 Poster A02

Hydrothermal synthesis, structural and photocatalytic properties of iron and europium doped TiO₂ nanoparticles

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Iron and Europium doped TiO₂ (titania) nanoparticles powders have been synthesised by a hydrothermal route starting with TiCl₄, FeCl₃·6H₂O and EuCl₃·6H₂O (Aldrich p.a.) as raw materials. A stainless steel Teflon lined autoclave was utilized for the hydrothermal runs at 200 °C. The structure, morphology and optical peculiarities were investigated by means of X-ray diffraction (XRD), transmission electron microscopy (TEM), extended X-ray absorption fine structure (EXAFS), Mössbauer spectroscopy and UV-Vis measurements. The photocatalytic performance was analysed in the photodegradation of salicylic acid and styrene oxidation reactions. Rietveld refinements of XRD patterns reveal that the as resulted samples consist in iron and europium doped TiO₂ in the tetragonal-anatase structural shape, with particle dimensions as low as 16 nm. The doping levels were limited to 1% at. Fe and Eu, correspondingly. By means of Mössbauer spectroscopy on both ⁵⁷Fe and ¹⁵¹Eu isotopes as well as by EXAFS analyses, the presence of Fe³⁺ and/or Eu³⁺ ions in the nanosised powders was evidenced. It was found that iron ions can partly substitute for titanium in the anatase structure, while the europium ions prefer the particles surface. From UV-Vis reflection spectra, by using the transformed Kubelka-Munk [1] functions, the band gap energy (E_g) for all hydrothermal samples has been determined in comparison with that of Degusa photocatalyst. A decrease of E_g from 2.93 eV found for Degusa photocatalyst to 2.83 eV for the titania doped with 1 at. % Fe and 0.5 at. % Eu has been evidenced; this behaviour indicates a valuable absorption shift (~ 20 nm) to the visible light region, for the hydrothermally synthesised titania doped sample. The photocatalytic properties in correlation with the structural and optical peculiarities of the hydrothermal samples are discussed.

1. W. N. Delgass, "Spectroscopy in Heterogeneous Catalysis", Academic Press, New York, 1979.

15:50 Poster A03

Titanium dioxide nanostructured layers for photo electrochemical cells

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Titanium dioxide nanostructured thin layers were deposited by different methods and compared functionally for the use in photo electrochemical cells. Both plasma (DC magnetron sputtering and laser ablation) and chemical (spin coating from titanium butoxide) deposition were

used. Commercially available ITO on glass or on alumina silicate glass were used as substrates. Scanning electron microscopy, X-ray diffraction and positron annihilation were used for structural analysis. As far as the chemical method needed several steps to obtain about 0.5 micron layers, magnetron sputtering with carefully chosen parameters produces such layers in a single step. Layers were used vs. platinum electrode in distilled water to determine photo electrochemical potentials. Essentially, the potentials obtained were only slightly dependent on the deposition method but photo electrochemical currents much more. No significant influence of the titanium dioxide phase (anatase vs. rutile) on the functional features was observed. Instead, the morphological structure influences much fixing of dyes on the titanium dioxide surface.

15:50 Poster A04

Preparation of Brookite-Type TiO₂/Carbon nanocomposite Electrodes for the Application to Li-Ion Batteries

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The increasing demand for portable power has generated significant research interest in nanostructured electrode materials, essentially due to their large interfacial contact area with the electrolyte and short path lengths for Li-ion transport. To date, TiO₂ has been widely investigated as an electroactive Li-insertion host. However, lithium reactivity of brookite-type TiO₂ has rarely been addressed compared to the common polymorphs, anatase and rutile, due to the difficulties encountering in obtaining phase-pure brookite structure. Herein, we report the simple synthesis of monodispersed nanocrystalline brookite-type TiO₂ using titanium trichloride (TiCl₃) and urea ((NH₂)₂CO). The average size of the particles precipitated at 100°C is ~ 10 nm. The brookite structure was stable up to 400°C and was completely transformed to rutile structure at 1000°C in O₂ atmosphere. We evaluated the electrochemical properties of each TiO₂ powder heat-treated at preset temperatures. The hybrid carbon/TiO₂ nanocomposites with high conductivity were also fabricated using stable suspension of 40 nm sized carbon black or multi-walled carbon nanotubes in aqueous suspension with an appropriate surfactant and subsequent precipitation of TiO₂. Incorporation of carbon clearly provides better capacity retention of TiO₂ upon cycling.

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Simple Method Synthesis of Visible Light Active N-Doped TiO₂ Photocatalytic Nanoparticle

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A nitrogen-doped titanium oxide nanoparticle was synthesized via simple sol-gel route and N-doped TiO₂ film was prepared to investigate the photocatalytic properties of the nanoparticle. In order to obtain N-doped anatase nanoparticle crystalline, NH₄OH solution was added dropwise to titanium tetraisopropoxide (TTIP) solution at 80°C and the particulate sol was reacted for 24 hours. The synthesized white

powder leads to pale yellowish powder by following heat-treatment process at 450°C in air for an hour. X-ray photoelectron spectrum and UV-vis spectrum were analyzed to confirm that the nitrogen was doped into TiO₂ nanoparticle successfully. The visible light-induced photocatalysis and hydrophilicity of the N-doped TiO₂ film were enhanced significantly as compared with undoped one. The improved photo activity was attributed to extended absorption range of the TiO₂ nanoparticle from ultraviolet to visible wavelength.

15:50 Poster A06

Synthesis of new TiO₂/Ag₂S nanocomposites and their photoactivity on the degradation of phenol

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TiO₂ nanoparticles have been intensely investigated as photocatalysts for environmental remediation processes, including the degradation of organic pollutants in waters. In this context, a critical factor to consider in using TiO₂ is that due to its wide band-gap (bulk anatase, $E_g = 3.2$ eV), only a small portion of the solar spectrum is absorbed by the photocatalyst. Therefore, several attempts have been made to conjugate TiO₂ nanoparticles with light sensitizers, such as dyes or other semiconducting phases.

Here we present a first example of a TiO₂ based photocatalyst containing Ag₂S nanophases grown from a single-source. We will show, that silver (I) alkylthiocarbamate complexes, in the presence of aliphatic amines yield the respective metal sulphide when subjected to moderate heating; in the presence of inorganic substrates, such as TiO₂ particles, the metal sulphide phase grows over the particles surfaces. This method provides control over nanoparticle growth and substrate coverage in a one-step process, which is clearly advantageous to design new catalysts.

The TiO₂/Ag₂S nanocomposites are composed of a Ag₂S nanophase evenly dispersed as islands that do not completely cover the TiO₂ surface. For nanocomposites prepared for longer reaction times, the Ag₂S islands spreads over the substrate and a less surface area of TiO₂ is available in contact with the solution. As expected, a dramatic decrease of the photocatalytic activity of the TiO₂/Ag₂S nanocomposite was observed for such cases. Our results concerning the photodegradation of phenol using such nanocomposites, under distinct reacting conditions, will be presented and discussed in detail.

Acknowledgments

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15:50 Poster A07

Au/TiO₂ Flowerlike Core-shell Nano Particles: Synthesis and Application in Photocatalysis

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Semiconductor-metal nanocomposites have been widely employed in photo catalysis. In most of the catalytic studies, metal nanoparticles are dispersed on an oxide surface, such a catalyst structure, though effective; result in exposing both to reactants and the surrounding medium. Recently, some metal-core oxide-shell nano structures have been prepared successfully, and a considerable amount of the research has been performed on their crystal structures and morphologies. Yet, the applications such as the application in the photocatalysis have seldom been reported. In the present work, flowerlike Au/TiO₂ core-shell nano composite particles were prepared by hydrothermal synthesis process and characterized by XRD, XPS, TEM, FE-SEM and BET measurement. The Au/TiO₂ flowerlike core-shell nano composites showed drastic improvement in the photocatalytic activity comparing with the nano TiO₂ particles. The authors explained in detail the reason that the flower-like structure plays an important role in the activity increase. In addition, the effects of the Au-core size on the photocatalytic activity were also studied. The higher photocatalytic activity was obtained in the composite catalyst with smaller Au-core size than that composed of larger Au particles.

15:50 Poster A08

An Effect of Different pH Solution of TiO₂ on properties and photocatalytic activity

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Since the properties of the TiO₂ powder are influenced mainly by synthesis and processing methods, various synthesis methods of TiO₂ powders such as sol-gel, hydrothermal and hydrolysis of inorganic salt have been proposed with the aim to obtain the suitable TiO₂ which yield good photocatalytic activity. In this work, TiO₂ solutions were prepared using titanium halide as precursor via precipitation method and adjusted pH with ammonium solution. Then the solution was calcined at various temperature to obtain TiO₂ nanopowder and investigated the effect of pH on crystal structure, morphology and photocatalytic activity of the synthesized TiO₂. Crystal structure and phase transformation of the prepared samples were studied by XRD technique. Morphology and crystallite size of the TiO₂ powders were investigated using SEM and TEM. Photocatalytic activity of TiO₂ was evaluated by investigating degradation of methylene blue after UV irradiation. Degradation of the solution was then determined by measuring absorbance of the methylene blue of each decanted solution using UV-VIS spectrophotometer. We expected that different synthetic conditions such as different pH, different calcination temperature would result in different crystal phase, crystallite size and especially in photocatalytic activity of TiO₂. Results showed that with increasing calcination temperature, the crystallite size was increased. Comparing crystal structures obtained from different pH values in synthesis, at same calcination temperature, the lower pH values resulted in anatase and rutile phases while the higher pH values showed only anatase phase. An affect of such properties on the photocatalytic activity of synthesized TiO₂ will be presented.

15:50 Poster A09

Thermal decomposition of alkoxo-organometallic precursors for metal oxide materialsŁukasz John¹, Józef Utko¹, Leszek Kępiński², Lucjan B. Jerzykiewicz¹, Piotr Sobota¹**1.** University of Wrocław, Faculty of Chemistry, Joliot-Curie 14, Wrocław 50-383, Poland **2.** Polish Academy of Sciences, Institute of Low Temperature and Structure Research (INTiBS), Okólna 2, Wrocław 50-422, Poland

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Alkoxides and aryloxides of group 2 metals can be easily modified by organometallics giving crystalline binary metallic complexes. Such compounds are efficient single-source precursors (SSP) for metal oxide materials. Here we report syntheses, structures and thermal decomposition of mixed metal complexes: $[M\{\mu\text{-ddbfo}\}_2M'R_x]_2$ (ddbfoH = 2,3-dihydro-2,2-dimethylbenzofuran-7-ol; $M/M'/R/x = \text{Ba/Al/Me/2}$, Ba/Al/Et/2 , Ba/Ga/Me/2 , Ba/In/Me/2 , Ba/Zn/Et/1 , Sr/Al/Me/2), $[\text{Ca}(\mu\text{-ddbfo})\{\mu\text{-ddbfo}(\mu\text{-Me})\text{AlMe}_2\}]_2$, $[\text{Ca}\{\mu\text{-OR}(\mu\text{-Me})\text{AlMe}_2\}]_2$, $[\text{Ca}\{\mu\text{-OR}\text{AlMe}_3\}_2(\text{THF})_2]$ ($\text{ROH} = 2\text{-methoxyethanol}$). The SSP species possesses desired stoichiometry which is achieved on the molecular level and the resulting molecules subsequently thermolyzed yield oxide products. The chemical composition and surface morphology of heterometallic oxides were analyzed by SEM-EDS and X-ray powder diffraction (XRD) that revealed the formation of highly pure oxide materials with μm particle sizes.

15:50 Poster A10

Microwave-assisted non-aqueous synthesis of metal oxides nanoparticles

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The development of new synthetic routes for inorganic nanomaterials should take into account the necessity of being fast, energy efficient and environmentally friendly. The low-temperature solvothermal synthesis of metal oxides in benzyl alcohol has been successfully applied for the preparation of a broad variety of metal oxides. This non aqueous sol gel route is based on the reaction of metal organic precursors with benzyl alcohol and offers the possibility to tailor the particles size and shape by choosing the appropriate reaction conditions. Another technique that has been developed during the last ten years is microwave-assisted synthetic organic chemistry.

This non-classical heating technique is well known to dramatically reduce the reaction time from hours to minutes or even seconds. The objective of this work is to combine both advantages, the robust solvothermal synthesis and the rapid and efficient microwave heating, for the fast preparation of various nanocrystalline metal oxides. The as-obtained particles are characterized by powder X-ray diffraction, TEM and HRTEM.

We present here a one minute synthesis of highly crystalline nanoparticles of iron oxide, cobalt oxide and zinc oxide. These nanocrystalline particles could be synthesized by choosing carefully the metal organic precursor and the organic solvent. A complete microwave heating protocol was developed with specific temperature, pressure and microwave power. In addition to the short reaction times, the microwave-assisted solvothermal synthesis method is characterized by

high yields and the possibility to control the crystal size through the reaction time. These advantages make microwave-assisted non-aqueous synthesis a highly efficient and promising technique for the production of metal oxide nanoparticles for chemical, pharmaceutical and biological applications.

15:50 Poster A11

Nanosized ZnO clusters from Organometallic One-Component PrecursorsJanusz Lewiński¹, Wojciech Bury¹, Marek Kościelski¹, Iwona Justyniak²**1.** Warsaw University of Technology, Faculty of Chemistry, Noakowskiego 3, Warszawa 00-664, Poland **2.** Polish Academy of Sciences, Institute of Physical Chemistry, Kasprzaka 44/52, Warszawa 01-224, Poland

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Zinc oxide can be regarded as one of the most important metal oxide semiconductors for future applications. A great deal of effort has been made to synthesize nanostructured ZnO and understand the resulting properties. The control of both the physical and chemical properties of the nanoparticles is crucial and it depends on several factors, namely the particle size, the shape of the particle, and their dispersability. The challenge is also in stabilizing as prepared nanoparticles to avoid further agglomeration and preserving their shape and size. Most of preparative methods for ZnO nanoparticles are based on an inorganic sol-gel method. This route needs a precise control of pH of the reaction media and the use of alkali metal hydroxides as precipitants. Only few reports concern the preparation of nanosized particles from organometallic one-component precursors, which are readily available in a pure form.

In our novel approach we have utilized well-defined alkylzinc alkoxides or carboxylates as efficient ZnO precursors. These classes of compounds may be smoothly transformed into zinc oxide nanosize clusters and particles by the treatment with O_2 or $\text{O}_2/\text{H}_2\text{O}$ system. We will also demonstrate that the stabilization of nanosized ZnO clusters is viable by coating them with polycarbonates. Finally, different $[\text{RZnL}]_n$ (where $\text{L} = \text{O}, \text{O}'$ -chelating ligand) complexes have been tested and the influence of the precursor structure and its aggregation on the structure and properties of ZnO nanoscaled clusters will be discussed.

15:50 Poster A12

Solvo-thermal synthesis of $\text{Zn}_{1-x}\text{Li}_x\text{O}$ ($x = 0.0 \sim 0.20$) nanoparticlesByung-Chun Choi¹, Jung Hyun Jeong¹, Byung Kee Moon¹, Byeong-Eog Jun¹, Jong-Seong Bae², Haeyoung Choi³, Ill-Min Kwon¹, Yu-Sung Kim¹**1.** Department of Physics, Pukyong National University, 599-1 Daeyeon-Dong Nam-Gu, Busan 608-737, Korea, South **2.** Busan Center, Korea Basic Science Institute, San 30, Jangjeon-Dong, Geumjeong-Gu, Busan 609-735, Korea, South **3.** Department of Physics, Dong-Eui University, 995 Eomgwangno, Jin-Gu, Busan 617-714, Korea, South

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The Li-substituted zinc oxides $\text{Zn}_{1-x}\text{Li}_x\text{O}$ ($x = 0 \sim 0.20$; ZLO) nanoparticles were synthesized by the wet chemical methods. The precursors for the synthesis of ZLO nanoparticles were zinc acetylacetonate hydrate and lithium acetates di-hydrate, which were resolved in the mixed solvent of toluene : 2-propanol = 7 : 1. ZLO nanoparticles were confirmed by the transmission electron microscopy and X-ray diffraction.

tion. The size of ZLO nano-particles does not change with Li contents. The hexagonal lattice constants a and c increases with Li contents increases at $0.05 < x < 0.20$. The X-ray photoelectron spectroscopy reveals that there is a slight over-lapping of the shallow core levels O 2p and Zn 3d. The correlation between the Li contents and the d-p coupling is examined by the shifts of the core levels Zn 2p, O 1s, etc. We characterize the photoluminescence of ZLO nano-particles with regard to the morphology change and/or the d-p coupling. We examine the effective dielectric constants and effective electrical conductivities of ZLO nano-particles.

15:50 Poster A13

Magnetic Properties of Cobalt and Manganese Doped ZnO Nanocrystals

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Diluted Magnetic Semiconductors (DMSs) are considered promising materials for spintronic applications. Since they were predicted ferromagnetic at room temperature, research efforts focus their attention in system such as transition-metal doped zinc oxide¹. However, the origin of magnetism of these compounds is not entirely understood and it is still debated. Indeed, experimental reports reveal a great disparity on the magnetic behavior of doped ZnO; going from paramagnetic to ferromagnetic behavior for the same transition-metal dopant. Though, for homogeneous systems, magnetism seems to be strongly dependent on dopant/matrix interaction, carrier concentration, defects, and thus on synthetic condition^{2,3}.

We present the synthesis, characterization and magnetic behavior of $\text{Zn}_{1-x}\text{M}_x\text{O}$ ($\text{M} = \text{Mn}$ or Co) systems grown in different organic solutions. Doped ZnO were synthesized by both, thermal decomposition in coordinating solvent such as trioctylamine^{4,5} and solvothermal reaction with Benzyl alcohol⁶. In both syntheses we used identical precursors in order to determine the role of solvent on the magnetic and optical properties and, on the control of morphology as well. Moreover, these solution routes lead to the synthesis of gram quantities of pure phase nanostructures with high reproducibility and allow the incorporation of several transition metal dopants.

References:

- [1] T. Dietl, H. Ohno, F. Matsukura, J. Cibert, D. Ferrand, *Science*, 287, 1019-1022, 2000.
- [2] A. Fouchet, W. Prellier, and B. Mercey, *Journal of Applied Physics*, 100, 013901, 2006.
- [3] M.H.F. Sluiter, Y. Kawazoe, P. Sharma, A. Inoue, A.R. Raju, C. Rout, U.V. Waghmare, *Physical Review Letter*, 94, 187204, 2005.
- [4] B.D. Yuhas, D. O. Zitoun, P. J. Pauzauskie, R. He, P. Yang, *Angewandte Chemie-International Edition*, 45, 420-423, 2006.
- [5] G. Clavel, N. Pinna, D. Zitoun, *Physica status solidi A*, 204, 118, 2007.
- [6] G. Clavel, M. G. Willinger, D. Zitoun, N. Pinna, *Advanced Functional Materials*, 2007, in print.

15:50 Poster A14

The synthesis and structural characterization of $\text{Zn}_x\text{Cd}_{1-x}\text{S}$ luminescent nanoparticles

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In this work, $\text{Zn}_x\text{Cd}_{1-x}\text{S}$ nanocrystalline particles synthesis from aqueous solution is presented. As starting reagents, $\text{CdSO}_4/\text{Cd}(\text{NO}_3)_2$, $\text{ZnSO}_4/\text{Zn}(\text{NO}_3)_2$ and $\text{Na}_2\text{S}_2\text{O}_3/\text{Na}_2\text{S}$ were used. A chemical stabilizer was added into the solution aiming to provide a colloidal state of the synthesized nanoparticles, preventing $\text{Zn}_x\text{Cd}_{1-x}\text{S}$ to form clusters. The particles size distribution from the final powders was investigated by laser analysis and by transmission electronic microscopy (TEM), which revealed crystallites of 1.5 – 10 nm diameter. The shape and the crystalline structure of the particles were investigated by transmission electronic microscopy and scanning electronic microscopy (SEM). Thin films containing nanocrystalline cadmium and zinc sulphide are used in photo and electroluminescent cells. Different methods to obtain thin films have been investigated. A high efficient method is the digital deposition (bubble jet), which enables to perform complex configurations with low costs, based on nanocrystalline $\text{Zn}_x\text{Cd}_{1-x}\text{S}$ particles. Other compounds as phosphors $\text{ZnS}:\text{Cu}$ and $\text{ZnS}:\text{Cu},\text{Ag}$ exhibit a good UV conversion efficiency into fluorescence emission, applied also in photo and electroluminescent cells.

15:50 Poster A15

Preparation and characterization of CdSe nanoparticles stabilized by PVA using sonochemistry method

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Semiconductor CdSe nanoparticles were prepared using cadmium acetate ($\text{C}_4\text{H}_6\text{CdO}_4 \cdot 2\text{H}_2\text{O}$) as a Cd^{2+} ion source and Sodiumselenosulfate (Na_2SSeO_3) as a Se^{2-} ion source by inducement ultrasonic irradiation at room temperature (27°C) and normal laboratory conditions and ambient pressure. Polyvinyl alcohol (PVA) was used as a matrix and stabilizer. The obtained particles were characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive X-ray analysis (EDAX) and UV-Vis spectrophotometer. SEM images show that the CdSe nanoparticles formed uniformly in polymer matrix; consist of spherical nanocrystallites aggregated in the form of polydisperse nanoclusters of about 100nm in dimension. The nanoparticles in PVA polymer matrix prepared by ultrasonic waves were more monodispersed than prepared particles in aqueous media by other methods in the same technology conditions. XRD pattern of as-prepared nanoparticles show that the nanoparticles are pure and crystalline in nature. From UV-Vis absorption spectra a great blue shift of about 3eV in band gap was observed due to quantum confinement effect. Elemental analysis of as-prepared samples from EDAX show that the atomic percent of Cd to Se was 50 to 50, are exactly stoichiometric. The size of particles from observed SEM images and calculated from XRD pattern and UV-Vis absorption spectra confirm each other.

15:50 Poster A16

The affect of reactant purity and specimen post-processing on the cathodoluminescence of ZnO nano-powdersKatie E. McBean¹, Matthew R. Phillips¹, Dominique Drouin²**1.** University of Technology (UTS), PO Box 123, Broadway, NSW 2007, Sydney, Australia **2.** Université de Sherbrooke, Sherbrooke J1K 2R1, Canada

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ZnO nano-powders were synthesised by mixing equal volumes of 0.05 M zinc chloride in ethanol and 0.20 M sodium hydroxide (either 97+% or semiconductor grade) in ethanol, for six hours at room temperature. The resulting cloudy solutions were centrifuged and the resulting powders rinsed before being dried under vacuum. The synthesised ZnO powders are referred to as: HP ZnO; made with the semiconductor grade NaOH, and ZnO; made with the 97+% NaOH.

Cathodoluminescence (CL) spectra and images were collected at 5 K of these as-prepared specimens, as well as specimens annealed at 700°C for 10 minutes in an atmosphere of 5% H₂ / 95% Ar. The HP ZnO displayed a much more intense UV near band edge (NBE) emission compared with ZnO, and lacked the broad green defect related emission observed in the specimen prepared with 97+% NaOH. Annealing in H₂ increased the intensity of the NBE emission in both powders although the HP ZnO NBE emission remained by far the most intense, while the intensity of the green defect emission of the ZnO was greatly reduced.

The NBE emission from H₂ annealed HP ZnO contained recombination centres at 3.36 eV; attributed to I_{6/6a} Aluminium donor and 3.32 eV; an unidentified acceptor-bound exciton emission with LO phonon replicas at 3.25 eV and 3.18 eV. The H₂ annealed ZnO displayed weaker intensity emissions at the same energies in addition to a weak green defect emission at 2.40 eV.

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Photoluminescence of cadmium sulfides nanoparticles doped with rare-earth elements

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Cadmium sulfide nanoparticles are one of the perspective materials for application in optoelectronics, in high-speed computing systems developing, biomarkers. One of the main properties of these systems are photoluminescence(PL) properties. In the given work it was investigated PL of nanoparticles doped with rare-earth elements. Doping process conducted as follows. After CdS nanoparticles growth by SILAR it was realized sorption of ions of rare-elements. The results of investigations show that doping with chrome ions intensity of PL decreases. During doping with praseodymium in excitation spectrum the maximum of excitation and curve behavior don't changed ($\lambda = 470$ nm). During Pr doping in 2 growth cycles in the long-wave region of emission spectrum appear a new line with maximum. In 4 growth cycle after alloying intensity of peak with $E_{\max} = 1.81$ eV increases. Intensity of emission spectrum during Pr doping increases to ~ 1.5 time. In 12 growth cycle samples emission spectrum has complicated character on interphase interaction. In all samples character of temperature dependence identical and by increasing of temperature PL intensity increases sharply.

15:50 Poster A18

Monodisperse ZnO and ZnSe nanoparticles with extremely strong UV-photoluminescenceAlexey S. Shaporev¹, Vladimir K. Ivanov^{1,2}, Andrey A. Eliseev²**1.** Kurnakov Institute of General and Inorganic Chemistry RAS (IGIC), Leninsky prospect, 31, Moscow 119991, Russian Federation **2.** Faculty of Material Science, Moscow State University (MSU), Lenin Hills, Moscow 119992, Russian Federation

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A^{II}B^{IV} semiconductor nanoparticles attract close attention of the researchers due to their size-dependent optical and electrical properties. They possess effective luminescence in UV region of spectra, and are highly prospective for transparent electrodes for solar cells, electroluminescent devices, chemical sensors etc. Here, we describe a new route to prepare ZnO and ZnSe nanoparticles with ultra narrow size distribution. The method was based on usual TOP/TOPO preparation procedure. ZnO and ZnSe were synthesized from diisopropyl zinc, with TOPO or TOPSe as B^{IV} element source (for ZnO and ZnSe, correspondingly) and oleic acid as a surface active compound. The reactions were carried out at 180°C under argon flow (5 min). ZnO and ZnSe colloidal solutions were subjected to size selective precipitation procedure and redispersed in heptane.

Synthesized materials were studied by HRTEM, UV-Vis and luminescent spectroscopy. According to TEM, they consist of 3-5 nm nanoparticles with rather narrow size distribution. The particles are well separated and no aggregates were observed. Obtained colloidal solutions showed strong excitonic absorption peaks in UV region. The band edges are blue-shifted compared to bulk materials, and particle sizes calculated from UV-Vis absorption spectra are in a good agreement with those observed by TEM. It should be noted that no absorbance in visible region was found at all. Obtained materials demonstrate good photoluminescent properties: ZnO has an intense luminescence in UV region (346 nm, excitonic emission) and low-intensity wide green line with maximum at 538 nm (defect). Variation of synthetic conditions allowed us to form ZnO nanostructures with UV/Vis emission ratio up to 50 at room temperature. Emission from ZnSe nanostructures was found much less intensive than that of ZnO. However nearly the same UV/Vis emission ratio was observed.

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Controlled synthesis of nanocrystalline ceria particles

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Nanocrystalline ceria is a promising oxide material, which become widely used as an abradant for mechanochemical polishing of silicon wafers, protective anti-UV radiation coating and catalyst.

Here we have proposed two methods of ceria synthesis based on precipitating CeO₂ by ammonia from cerium nitrate aqueous alcoholic solutions of different concentrations (50, 65, 75, 85, 95 vol.%) and homogeneous hydrolysis of cerium nitrate in the presence of hexamethylenetetramine (HMTA).

According to heat flux calorimetry data, CeO₂ formation process is accompanied by a considerable heat effect (-73.2 kJ/mol); kinetic

analysis indicates that the process runs by the mechanism of autocatalysis – the hydroxocomplexes decomposition proceeds on the surface of the already formed CeO_2 particles, leading to their growth. This was confirmed by the UV-visible spectroscopy: the absorption band edge drifts towards the longer wavelength region, so that the band gap energy changes from 3.35 to 3.15–3.20 eV. We can conclude that in the aqueous alcoholic CeO_2 suspensions a gradual particle growth takes place, which is accompanied by the decrease of the band gap energy and the oxidation of the residuary Ce(III) ions. The micromorphology of the samples was studied by XRD, TEM, SANS and low temperature nitrogen adsorption. It was found that the surface of nanopowders possesses fractal properties, and the value of fractal dimension depends both on concentration of initial solutions and conditions of subsequent thermal treatment.

It was also established that synthesis of ceria via hydrolysis of cerium nitrate in the presence of HMTA proceeds through formation of unstable cerium hydroxocarbonate. It was shown that CeO_2 mean particle size and the band gap value can be finely tuned by variation of synthesis duration, temperature, concentration of reactants and their molar ratio.

15:50	Poster	A20

Effect of iron addition on the properties of ZnO obtained by precipitation

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New ferromagnetic semi-conductors are interesting because of the expectations connected with their use in magnetoelectronic or spintronic devices. Potential candidates are semi-conductive oxides of metals doped with ions or oxides of transition metals, as for example zinc oxide doped with iron oxide. Such samples were prepared by wet chemical method – precipitation from nitrate solutions using ammonia. The obtained hydroxides were filtered, dried and calcined at 300 °C during 1 hour. A series of samples containing 5 to 95 wt.% of Fe_2O_3 was obtained. The prepared samples are ferromagnetic. The phase composition of the samples was determined using XRD. Depending on the chemical composition the phases of hexagonal ZnO, and/or rhomboedric Fe_2O_3 and/or cubic ZnFe_2O_4 were identified. The mean crystallite size of these phases, determined using Scherrer's formula, varied from 8 to 30 nm. The real chemical composition of the samples determined using ICP AES method was close to the assumed one. The measurements of density and surface area showed a good quality of nanopowders. The morphology of the samples was investigated using scanning electron microscopy. Spherical or elongated nano-grains can be observed. The measurements of magnetization were also carried out.

15:50	Poster	A21

Electrochemical synthesis of ZnO nanoparticles

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The anodic behaviour of zinc was investigated in ethanol solution containing LiCl, and water. Zinc was polarised using cyclic voltammetry and chronoamperometry techniques. In a course of the anodic zinc dissolution, a colloidal suspension of ZnO nanoparticles has been obtained in the 0.1 M LiCl – ethanol solutions containing 3% of H_2O . The presence of ZnO nanoparticles was confirmed by mean of transmission electron microscope with EDX and X-ray diffraction analysis. The suspension is formed only if water is present and a rate of the process depends on water content. Polarisation of zinc in the given conditions is a very simple method of producing ZnO nanoparticles.

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15:50	Poster	A22

Kinetic Study on the Hydrothermal Synthesis of Surface-Modified Cerium Oxide Nanoparticles Using a Flow-Type Reactor

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We report kinetic study on the hydrothermal synthesis of cerium oxide nanoparticles using a flow-type reactor. The synthesis was performed in the presence of organic molecules to modify the surface of the cerium oxide nanoparticles during their synthesis. The amount of reacted cerium ion was evaluated using inductively coupled plasma (ICP) method. Arrhenius plots assuming first-order reaction showed that the presence of organic molecules slowed the consumption of cerium ion, which was plausibly caused by the capping of the growing surface of the metal oxide nanoparticles; the size of synthesized nanoparticles in the presence of the surface modifiers was much smaller than that of the products without surface modifiers. Based on these kinetic studies, we can optimize the configuration of flow-type reactors and synthetic conditions for desired products. For example, we have successfully synthesized cerium oxide nanoparticles whose diameter was about 4 nm using the flow-type reactor. Their surface was capped by organic molecules that increased the affinity with dispersion media and prevented agglomeration between them.

15:50	Poster	A23

Synthesis and characterization of nanocrystalline Y_2O_3 powder

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Interests in nanoarchitected materials and design grows academically and industrially and nanomaterials are of high interest in a wide range of technical applications. In this work, Solution Combustion Synthesis (SCS) is used to prepare nanocrystalline rare-earth ceramic oxides

Re_2O_3 with $\text{Re} = \text{Y}$. Nanosized Y_2O_3 oxides are used in the production of Oxides Dispersion Strengthened (ODS) steels. ODS steels are envisaged as structural materials for first wall and breeding blanket applications in future fusion reactor concepts. Nanocrystalline oxides can be synthesized by a variety of sophisticated methods e.g. oxidation of metal nanoparticles, laser ablation, sol gel methods SCS in contrary, is a successful and relative simple inorganic reaction system for the production of small oxides particle size, crystalline homogeneous ceramics and this at low reaction temperatures and within reduced reaction time. By modification and control of the reaction chemistry it is possible to vary the yield, particle size and crystallinity/amorphous content of the as-prepared powder. Size and crystallinity of the as-produced powder can be modified by post production heat treatments.

A primary characterization of the nanoscaled rare-earth oxides powder is performed with X-ray diffraction (XRD). Based on the obtained diffraction data, crystallinity and particle size has been determined using conventional and more sophisticated calculation techniques. Transmission Electron Microscopy (TEM) is the most important technique used as observation technique to prove the calculated particle size and crystallinity of the obtained powders, before and after re-irradiation.

15:50 Poster A24

Theoretical analysis of oxygen adsorption at SnO_2 grains with slab geometry

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Operation of SnO_2 resistive gas sensors is mainly governed by the surface space charge region, which is formed due to both adsorption of gas molecules and ionization of surface states related to the oxygen vacancies at the surface. The surface-related phenomena become particularly important for nanostructures, which exhibit a much higher surface-to-volume ratio compared to SnO_2 thick films composed of micro-grains. Therefore, the understanding and control of the surface and near-surface electronic properties and their relationship with materials ones are key factors for technology optimization, and thus improvement of the sensitivity and selectivity of SnO_2 – based sensor devices. In addition, the modeling of sensor structures based on nanolayers needs to consider size and shape of grains, which decide about the layer conductance.

The purpose of this work is a computer analysis of an influence of oxygen adsorption at the SnO_2 surface on the electronic parameters of the induced depletion layer. The surface potential value and in-depth potential profiles were obtained by solving the Poisson-Boltzmann equation in the case of finite grains with slab geometry. The SnO_2 grain size was in the range from 15 nm to 150 nm. The surface energy barrier dependence on grain size was determined. The mobile singly or double ionised bulk oxygen vacancies (donors) were assumed. The effect of donor mobility and degree of donor ionisation on the electrical potential inside of grains with different thickness was analysed. Furthermore, the dependence of conductance per square on temperature (from 400 to 900 K) was rigorously computed. The temperature variations of the carrier mobility were also taken into account. It was found that the development of near-surface depletion region and thus modification of the layer electronic parameters are strongly dependent on the layer thickness, bulk donor concentration, and temperature.

15:50 Poster A25

Low temperature synthesis of nanocrystalline α -alumina by unconventional methods

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Three unconventional synthesis methods have been successfully applied for the preparation of $\alpha\text{-Al}_2\text{O}_3$ powders as follows: thermal decomposition of the complex combination resulted from the oxidation reaction of 1,2-ethanediol and glycerol by the aluminium nitrates; exothermal redox reaction of the aluminum nitrates with an adequate fuel; respectively acid catalyzed sol-gel method using aluminium triisopropylate precursor. The synthesis temperature, purity and morphology of the prepared alumina powders are highly affected by the used synthesis method. Nanocrystalline α -Alumina were obtained by: heating aluminium nitrate and an organic fuel mixture at 500°C; using aluminium glyoxilate at 1100°C and using sol-gel method at 1200°C. The formation of α -alumina phase was investigated by X-ray diffraction (XRD) studies and thermal analysis (TG, DTG and DTA).

15:50 Poster A26

Peculiarities of $\text{CaO}\cdot 6\text{Al}_2\text{O}_3$ formation using low temperature combustion synthesis

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The paper presents for the first time a new version of the solution combustion synthesis with applications in the $\text{CaO}\cdot 6\text{Al}_2\text{O}_3$ direct formation, without any additional thermal treatments. The starting point was the premise - confirmed in our previous results - that there is a predilection of metal nitrates with respect to certain fuels.

The used raw materials were: $\text{Ca}(\text{NO}_3)_2\cdot 4\text{H}_2\text{O}$, $\text{Al}(\text{NO}_3)_3\cdot 9\text{H}_2\text{O}$, urea, glycine and β -alanine. The experimental determinations evidenced that glycine or β -alanine lead to the formation of an amorphous reaction product, whereas urea leads to the formation of a phase mixture consisting of $\text{CaO}\cdot 6\text{Al}_2\text{O}_3$ and $\alpha\text{-Al}_2\text{O}_3$.

On the other hand, the use of a stoichiometric fuel mixture containing urea and β -alanine alongside the desired metal nitrates ensures the straight formation of a pure, well-crystallized $\text{CaO}\cdot 6\text{Al}_2\text{O}_3$, without any subsequent calcination step. The experimental results prove that the version we suggested - the use of a fuel mixture - is definitely better than the traditional version of the combustion synthesis, based on using a single fuel.

15:50 Poster A27

Genesis of Al-doped apatite-type nanocrystalline lanthanum silicates prepared by mechanochemical synthesis: Phase, structural and microstructural study

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Apatite-type lanthanum silicates (ATLS), especially doped ones, have recently attracted a considerable attention due to their high ionic conductivity at intermediate temperatures but their usual synthesis requires too long firing cycles at ~1500°C. To promote ATLS application, the development of low-temperature preparation methods is necessary. Earlier, mechanochemical treatment has been successfully used for a low-temperature synthesis of undoped ATLS.

In this work, the results on the formation of Al-doped ATLS in the course of mechanochemical activation (MA) in a high-power planetary ball mill at room temperature are presented. Phase and structure evolution during 5-35 min MA of a stoichiometric mixture containing La₂O₃, Al(OH)₃ and amorphous SiO₂ were investigated using XRD, IR, NMR and TEM methods. At the first MA stage, a strong structure disorder of the initial La₂O₃ and SiO₂ occurs with a formation of an amorphous aluminosilicate phase as a result of interaction of SiO₂ and Al(OH)₃ and, at the same time, an apatite phase starts to form. At increasing MA time, further interaction of highly disordered La₂O₃ and amorphous aluminum silicate which cover La₂O₃ particles suggests the formation of highly crystalline ATLS with a particle size of 10-60 nm. Subsequent MA of formed ATLS leads to distortion of ATLS anion sublattice and to destruction of some share of ATLS resulted in the formation of LaAlO₃. The latter fact shows a restriction of Al-substitution on Si-sites into ATLS.

15:50 Poster A28

Surface chemistry of Pr-doped nanocrystalline zirconia

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Nanopowders of zirconia doped with praseodymium were obtained by microwave assisted hydrothermal synthesis. The standard wet precipitation method was used as well to study the precipitation process. The XRD spectra of samples prepared using both methods were compared. Zirconium hydroxide precipitates at lower pH than praseodymium one, than a single nanoparticle of powder should be composed of a zirconia core covered by a shell of praseodymium oxide. This result was con-

firmed by the XPS measurements, showing the surface enrichment in praseodymium. According to the EPR studies of the nanopowders performed after the heat treatment under hydrogen, the creation of ZrO₂⁻ⁿ radicals can be suggested.

15:50 Poster A29

Surface state and structure zirconia nanoparticles under high pressure

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Nanosized zirconia (ZrO₂) is a well-know nanostructured oxide material with excellent electrical, optical and catalysis related properties. It is known that the properties of nanosize particles are determined by their phase composition, morphological features, the nature of inter-particle interaction and the level of aggregation.

In the present work the peculiarities of formation of a nanopowder hydroxide system and connection between the state of hydrate shell of hydroxide nanoparticles and their aggregation were studied. The wet gel hydroxide system ZrO₂-3 mol% Y₂O₃ was obtained by the method of chemical precipitation of the corresponding ammonium hydrate salts. Various physical influence (high pressure, microwave heating) were used to form nanoparticles with the defined state of hydrate shell. The structure of zirconia and evolution of the surface hydroxyl groups on amorphous nanosize powders was examined by FTIR spectral analysis, XRD, small-angle X-Ray scattering and by rheology methods.

The anomalies of physical properties (density, specific surface, rheological properties and other) are observed under the high pressure influence at 6 kbar. The physical model is offered which considers the nanoparticle as a heterogeneous system containing the closed and opened pores. It is shown, that the aggregation of nanoparticles is determined by correlation of the external pressure *P* and pressure inside closed pore *P_p*. A rapprochement of particles at *P* < 6 kbar promotes to a communications between them and results in formation of big conglomerates of particles with decreased density of material. When *P* > 6 kbar the pressure inside closed pores increases, the "misfit" stresses appeared because of substantial distinction in compressibility increase that leads to destruction of agglomerates on more small fragments. This research was performed with the framework of complex program basic research NAS Ukraine "Nanosystems, Nanomaterials, Nanotechnology".

15:50 Poster A30

Magnetic properties of nanocomposite manganites p-La_{0.78}Mn_{0.99}O_{3.5} and p-La_{0.8}Mn_{1.04}O_{3.5}

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En experimental investigations of the structure and magnetic properties of nanocomposite manganites p-La_{0.78}Mn_{0.99}O_{3.5} and p-La_{0.8}Mn_{1.04}O_{3.5}

are presented. The semiconducting composite nanomaterials p-La_{0.78}Mn_{0.99}O_{3.5} and p-La_{0.80}Mn_{1.04}O_{3.5} p-type of conduction was produced by high pressure technique at 7.5 GPa.

The X-ray diffraction measurements show, that samples are single-phase with the cubic perovskite crystal structure and with unit cell parameters: $a = 3.860 \pm 0.003$ Å, for p-La_{0.78}Mn_{0.99}O_{3.5} sample and $a = 3.8762 \pm 0.0051$ Å, for p-La_{0.80}Mn_{1.04}O_{3.5}. The nanostructure of these manganites were investigated by scanning atom-force microscopy method. The most characteristic value of nanograins diameter is near 70 nm.

The temperature dependence of magnetic susceptibility in temperature range 4.2 – 300 K for both samples was studied in small external magnetic field 25 Oe. There is clear-cut distinguishes between samples with different composition and thermal annealing. At temperature near 260 K for both specimens there are anomalies typical for the phase transition to a ferromagnetically ordered state. At temperatures above T_c the susceptibility behaviors are described by Curie-Weiss law quite well. But the difference in the curves of the temperature dependence of magnetic susceptibility in ZFC and FC regimes argues for phase separation and spin cluster phase existence.

The magnetization curves $M(H)$ in the wide range of temperatures from 4.2 up to 260 K was also measured. They demonstrate typical for ferromagnets behavior with temperature depended hysteresis. The temperature dependence of saturation moment are different for both samples, nevertheless both of them can be described with Brillouin function.

15:50 Poster A31

Nanotechnologies, structure and properties of magnetoresistive nanopowder pressings, mesostructured ceramics and nanostructured thin film prepared from La_{0.6}Sr_{0.3}Mn_{1.1}O₃

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The investigation is urgent due to the urgency of preparation and study of nanostructured metal oxides where unique magnetic and transport properties are combined. Manganite-lanthanum perovskites possessing colossal magnetoresistance are among them [1, 2].

Different nanotechnologies have been used: 1- mechanical mixing and grinding of powder oxides and salt of respective metals; 2 – combined precipitation of their nitrate salts; 3 – hydrolytic sputtering.

Methods of X-ray diffraction, low-temperature adsorption of argon, resistive, magnetic (including the ⁵⁵Mn NMR) ones [3] have been applied to investigate phase composition of powder, their dispersity and presence of defects in the structure, as well as density (γ), resistivity (ρ), coercive force (H_c), magnetic susceptibility (χ) and magnetoresistance ($MR = \Delta\rho/\rho_0$) of nanopowder pressings, sintered ceramics and thin film prepared from nonstoichiometric La_{0.6}Sr_{0.3}Mn_{1.1}O₃ [4].

Differences in physical and chemical properties of nanopowder produced by different technologies have been investigated. It is shown that with the increase of high hydrostatic pressure (HHP) to $P=1.6$ GPa γ , H_c , MR are increasing and ρ is decreasing.

Regularities have been determined for temperature and pressure dependences of χ and ρ ; the tunnel character of conductivity and mag-

netoresistance at interparticle contacts, and for the relationship between MR , particle size and pressure. A comparative analysis of magnetoresistance mechanism and differences for nanopowder pressings, sintered ceramics and thin film has been performed.

1. Nagaev E.L. Phys. Rep. 346, 387 (2001).
2. Mandal P., Ghosh B. Phys. Rev. B 69, 014422 (2003).
3. Pashchenko V.P. et al. Low Temp. Phys. 29, 910 (2003).
4. Pashchenko V.P. et al. Technical Physics 50, 1497 (2005).

15:50 Poster A32

Nanostructure clusterization, phase transitions and magnetoresistive properties of nonstoichiometrical perovskites La_{0.6}Sr_{0.2}Mn_{1.2-x}Co_xO₃ ($x=0-0.3$)

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Among nanostructured metal-oxides an increased attention is given to rare earth manganites and cobaltites, in which a colossal magnetoresistive effect (MR) has been revealed. Practical interest is caused by manganite-lanthanum perovskites, containing surplus superstoichiometric manganese.

By the X-ray, resistive, magnetic and NMR of ⁵⁵Mn methods the phase composition, defectiveness of perovskite structure, metal-semiconductor (T_{ms}) and ferro-paramagnetic (T_c) phase transitions, magnetoresistance effect (T_p), local magnetic and charge states of manganese in nonstoichiometric manganite-lanthanum-strontium perovskites La_{0.6}Sr_{0.2}Mn_{1.2-x}Co_xO₃ have been investigated.

Phase composition of ceramic samples is specified. It is shown that perovskite structure (R-3c) contains Mn³⁺_B, Mn⁴⁺_B, Mn²⁺_A ions, anionic and cationic vacancies (in A- and B-sublattice) and which are in high-frequency electron-hole exchange slim nanostructure clusters with Mn²⁺ in deformed A-sublattice. Such character of defectiveness of structure is confirmed by computer decomposition of NMR ⁵⁵Mn spectra and their analysis taking into account the irregular surroundings of manganese ions by both other ions (La³⁺, Sr²⁺, Mn²⁺, Co³⁺) and defects with cluster type nanostructure.

Co³⁺ ions, substituting Mn³⁺ ions, lower magnetization (M) and temperatures T_c , T_p and less distinctly T_{ms} . Clusterization, related to the surplus manganese was confirmed by hysteresis of temperature dependences for M below 45 K.

The tendency of MR enhancement by T_p , which is connect with defective crystal structure, and of MR lowering by 77 K, caused by tunneling along intercrystallite zones, that is (i.e.) mesoscopic inhomogeneity of ceramic samples has been determined.

On the basis of analysis of the results a conclusion is made on regularities of Co effect on nano- and mesostructures inhomogeneity, on the phase transitions, transport and magnetic properties of doped manganite-lanthanum perovskites.

15:50 Poster A33

Synthesis and Magnetic Properties of the $\text{La}_{0.50}\text{Ba}_{0.50}\text{MnO}_3$ NanomanganitesAleksey Trukhanov¹, Svyatoslav Stepin, Sergei Trukhanov², Cristian E. Botez³, Henryk Szymczak⁴

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$\text{La}_{0.50}\text{Ba}_{0.50}\text{MnO}_3$ nanosample has been synthesised using modified sol-gel method. The $\text{C}_3\text{H}_8\text{O}_3$ glycerin has been used as organic matrix. Parent nanosample has been annealed in air at $T_{\text{synth}} = 500^\circ\text{C}$, 700°C , 900°C и 1500°C . Average size of crystallite increases with annealing temperature from $D \sim 30$ nm to ~ 7 μm . The stoichiometry was correct. All the samples had cubic perovskite-like unit cell with increasing parameter from $a \sim 3.787$ Å to ~ 3.904 Å as average size of crystallite increases [1]. Pressure effect is maximum for the sample with $D \sim 30$ nm. Temperature Curie and spontaneous magnetic moment have non-monotone behavior and increases from $T_C \sim 264$ K to ~ 331 K and from $\sigma_S \sim 1.52$ $\mu_B/\text{f.u.}$ to ~ 3.31 $\mu_B/\text{f.u.}$, respectively. Anomalous magnetic properties of the $\text{La}_{0.50}\text{Ba}_{0.50}\text{MnO}_3$ is explained by frustration of Mn^{3+} -O-Mn⁴⁺ exchange interactions [2] and compression of the crystal lattice as a result of nanograin formation [3]. This study has been partly supported by the BFBR (F06R-078).

References:

- 1.S.V. Trukhanov, L.S. Lobanovski et al., J. Phys.: Condens. Matter 15, 1783 (2003).
- 2.S.V. Trukhanov, J. Mater. Chem. 13, 347 (2003).
- 3.K.S. Shankar, S. Kar, G.N. Subbanna et al., Solid State Commun. 129, 479 (2004).

15:50 Poster A34

Organic precursor synthesis of mesoporous Al-Mg mixed oxides and hydrotalcites

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Among the different types of basic oxides studied, hydrotalcites (HT) have been reported as promising catalysts for the fine-chemicals industry. The known methods of HT synthesis demand complex equipment, include time-consuming stages of precipitation, washing and drying and cause ecological problems due to the contamination of waste water with environmentally unfriendly ions.

The new route for Al-Mg oxides synthesis on the basis of Mg and Al organic salts is presented in this communication. Mixed Al-Mg oxides were synthesized by thermal decomposition (600°C) of Al- and Mg-nitrates in presence of citric acid (citrate method) and Al- and Mg-formates (formate method). The HT sample prepared by traditional co-precipitation method was used for a reference. The prepared Al-Mg

oxide samples possess mesoporous structure with different pore-size distribution (determined by nitrogen adsorption method).

The hydration of Al-Mg oxides by its immersion in water have been performed to obtain HT samples. The formation of high ordered HT lamellar structure was confirmed by XRD analysis.

Significant decreasing of surface area (S_{BET}) was observed for the re-constructed HT samples. The applying of the ultrasonic treatment under hydration of the Al-Mg mixed oxides has resulted in increasing of S_{BET} to 220 m^2/g which one is comparable with S_{BET} values for starting mixed oxides.

The catalytic activity of prepared samples was tested in the thermo programmed reaction of 2-methyl-3-butyn-2-ol decomposition with evaluation of acetone and acetylene. Among all the samples tested, HT prepared by hydration of mixed oxides with ultrasonic treatment shows the higher catalytic activity in this reaction.

15:50 Poster A35

Study of synthesis and formation mechanisms of nanometric magnesium alumina spinel powdersAnna Wajler¹, Henryk Tomaszewski¹, Ewa Drożdż-Cieśla², Helena Węglarz¹, Zbigniew A. Kaszkur³, Czesława Paluszkievicz², Jolanta Borysiuk¹

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Synthesis and thermal decomposition of magnesium aluminum spinel powder precursor composed of ammonium aluminum carbonate hydroxide and Mg-Al- CO_3 layered double hydroxide (hydrotalcite) was studied. The precursor was prepared by co-precipitation technique using ammonium bicarbonate as the precipitant. The morphology of precursor and calcined nanometric spinel powders were observed by SEM and TEM. To enable accurate monitoring of the decomposition process, the thermal evolution of structure, nature and stability of the resulting phases several in-situ techniques (DTA and TG coupled with MS of evolved gases, FTIR and HT-XRD) were used. Based on the results obtained a model describing spinel formation in this system has been proposed.

15:50 Poster A36

Anodic dissolution: an interesting way to fabricate small particles?Abdelhafed Taleb¹, Christine Vautrin-UI², Henri Mendy², Janusz Stafiej³, Annie Chaussé², Jean Pierre Badiali¹

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During the last century physicists and chemists succeed to fabricate materials in nanometer scale, they showed that these materials exhibit unexpected properties, which differ from those of the bulk materials. Different methods were proposed to achieve such materials which can

be classified into three main categories: Physical, Chemical and Mechanical methods. Among the chemical fabrication ways, the electrochemical methods are very attractive. The electrode potential allows the rate and temporal profile of the nanostructure growth and dissolution to be controlled with high accuracy.

The method we intend to develop can not be classified in the categories given above. It consists in the combination of two processes: electrochemical reaction which dissolves the anode and produces a given surface roughness with peninsulas and a mechanical one which provokes detachment of pieces of metal and thus the production of particles injected into the solution. Previously, some authors showed that during the beryllium dissolution¹, disintegration of the anode and the detachment of pieces of metal (particles) are observed. Their properties in terms of shape and size distribution remain controversial. Few works in the literature give a size varying from few atoms to microns, but no evidence is given about the relevant parameters which control these particles properties.

In the present work, we show using the cellular automaton model that the surface roughness during metal dissolution can be enough to induce small metal particles detachment. Additional to the surface roughness we show that the properties of these particles depend also on the material crystalline defects. In parallel to our theoretical study, we perform some electrochemical measurements in which we observe Faraday law deviation. Particles characterisations by using light scattering and TEM experiments are in progress.

References

[1] H. Aida, I. Epelboin, M. Garreau, J. Electrochem. Soc. 107, 960, (1971).

15:50 Poster A37

Oxide nanoparticles produced by laser ablation

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Oxide nanoparticles have been synthesized by pulsed laser ablation method.

Titanium, aluminum, tin, indium, nickel, germanium and zinc oxides nanoparticles were produced in oxygen atmosphere using a Nd:YAG laser at 355 nm, 60 mJ, 10 Hz, 5 J/cm². The oxygen flow rate was maintained at 200 mbar.

The influence of environmental and laser beam parameters on the nanoparticles properties were studied. The nanoparticles morphology, structure, composition, surface composition, size, size distribution and production rate were investigated. The particles were characterized by X ray diffraction, high resolution transmission electron microscopy, scanning electron microscopy, X ray photoelectron spectroscopy and laser granulometry measurements.

The bimodal size distribution was in the range 2-50 nm (75-85 % vol.) and 0.1-2 μ m (15-25%) and nanoparticles production rate was between 0.7 and 3 mg/kJ.

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Morphology and structure of collapsed carbon nanoparticles

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Carbon's ability to hybridize as sp , sp^2 and sp^3 bonds results in the formation of structures, such as fullerenes, nanotubes and nanofibers, nanooxions, nanocapsules, carbon nanospheres and nanoparticles (CNPs). Carbon nanoparticles have been of considerable research attention due to their intrinsic chemical and physical properties CNPs are different structures comparing to fullerenes or carbon nanooxions, because they have unclosed graphene layers. The semi-crystalline structure of carbon nanospheres reflects their unique properties: (i) low density, (ii) high porosity and surface area, and (iii) relatively high chemical and thermal stability. Carbon nanospheres may find many prospective applications, e.g. as catalysts supports, lubricating agents, electrode materials, in hydrogen storage and in construction of advanced sensors.

In this paper we present detailed morphological and structural studies of carbon nanoparticles obtained from combustion synthesis of $\text{NaN}_3/\text{C}_6\text{Cl}_6$ mixtures. High resolution scanning and electron microscopy as well as atomic force microscopy were applied for the morphology investigations. Raman spectroscopy and XRD diffraction revealed the structure of carbon nanoparticles. The results shows that carbon nanoparticles are hollow and 10-40 nm in diameter. Surprisingly, the height of CNPs does not exceed 5 nm. It strictly suggests the very collapsed nature of the nanoparticles. Carbon nanoparticles exhibit the semi-crystalline character with local ordering between the unclosed graphene layers. The collapse structure of CNPs was also discussed in the terms of obtained structural parameters from Raman spectroscopy and XRD.

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15:50 Poster A39

A Low Temperature Synthesis of CoFe_2O_4 Ferrite

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Low temperature synthesis of such materials has been found to yield to ultrafine particles with unique and unusual particle size by means of chemical stoichiometry reaction. The aim of this work was to prepare ultrafine cobalt ferrite by a wet chemical method. Ammonium oxalate and a solution of hydrazine hydrate were mixed and stirred under argon atmosphere at 80 °C to provide ammonium oxalate hydrazinate. A saturated mixture of cobalt and ferrous chloride solution was added slowly to the ammonium oxalate hydrazinate complex to obtain a precipitate of

cobalt and ferrous oxalate hydrazinate complex with chemical formula $\text{CoFe}_2 \cdot 3\text{N}_2\text{H}_4 \cdot 6\text{H}_2\text{O}$. The precipitate was washed, filtered and dried at 70 °C and then, heated in air at 260 °C for decomposes the precursor and producing a powder of oxide material. This material was analysed after a thermal treatment at 500 °C in air. For characterization, we have used chemical analyses, X-ray diffraction (XRD), UV-Vis absorbance spectroscopy, IR spectroscopy, thermal analyses TG-DTG, Mossbauer spectroscopy, and electron microscopy. The optimum composition of oxalate hydrazinate precursors from the point of view of the number of hydrazine molecules is the results of the compromise between the decomposition temperature and the violence of decomposition. It is desired a low decomposition temperature but also a control of the decomposition reaction is necessary. The presence of water is necessary because it is a moderator for thermal decomposition and it favours the yielding of oxide materials. The analyses show the presence of a homogenous compound with characteristic properties and well crystallised spinel structure, with formula CoFe_2O_4 .

15:50 Poster A40

Synthesis and Characterization of Nanocrystalline Iron Oxides for Nanocomposites with Biomedical Applications

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The properties of iron oxides (hematite $\alpha\text{-Fe}_2\text{O}_3$, magnetite Fe_3O_4 and maghemite $\gamma\text{-Fe}_2\text{O}_3$) make them important materials for biomedical applications. Magnetite and maghemite are a spinel but hematite is organized like a corindon. These iron oxides can be obtained by direct coprecipitation or physical methods, like nanopowders or thin films. This work aims to establish and to optimize the conditions for chemical synthesis of nano-sized magnetic iron oxide powders with desired magnetic properties. We have obtained iron oxides by chemical coprecipitation using mixed ferric and ferrous salts, because is enabling synthesis at low-temperature, low costs, high material purity and nanostructure control. For biomedical applications like magnetic bio-functional material vectors to target tissues, the iron oxide particles obtained have to be spherical with 10 nm average diameter. Magnetic properties obtained (saturation magnetization M_s and coercivity field H_c) are proper for superparamagnetic character of these nanoparticles. After synthesis, we have used the best iron oxide nanoparticles obtained to produce some nanocomposite systems with polyvinilic alcohol (PVA) and polypyrrole (PPy). The structure of nanoparticles obtained was investigated by X-ray diffraction (XRD), fluorescence X-ray spectrometry (XRF), transmission electron microscopy (TEM) and atomic force microscopy (AFM). Magnetic properties were determined with vibrating sample magnetometer (VSM). Nanocomposite systems with iron oxides were characterized with UV-Vis absorbance spectroscopy, AFM or scanning electron microscopy (SEM) techniques.

15:50 Poster A41

Continuous flow synthesis of magnetic nanoparticles using spinning disk processing

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Superparamagnetic magnetite (Fe_3O_4) nanoparticles are prime candidates for a wide range of applications such as magnetic storage media, magnetic resonance imaging, targeted drug delivery and magnetic separation. Various methods have been reported for the synthesis of magnetite nanoparticles. Co-precipitation is the most common and convenient way to synthesize magnetite by oxidation of ferrous (Fe^{2+}) and ferric (Fe^{3+}) salt solution by a base, such as NaOH or NH_4OH in an aqueous solution. However, the size distribution of the magnetite nanoparticles produced using this method is normally very broad. Furthermore, scale-up of this method using the batch processing can be problematic as it results in inhomogeneous agitation and areas of localized pH which in turn results precipitation of non-magnetic iron oxides. In this study, we demonstrate the successful synthesis of magnetite using NH_3 gas as base source via process intensification on a rotating surface (PIRS) under scalable and continuous flow conditions. Spinning disc processing (SDP) is a milestone in process intensification modernization which in turn ensures uniform reaction conditions on the rotating disc in contrast to traditional batch technology where conditions can vary across the dimensions of the vessel. The rapid mixing and high contact area between the fluid and the atmosphere above it ensure effective mass transfer and efficient mixing. In addition, the high throughput of the system, low changeover times and wastage make this a very solvent efficient method of synthesis. The geometry and key elements of a SDP are illustrated in Figure 1.

15:50 Poster A42

Structural and magnetic properties of nanosized barium hexaferrite powders obtained by microemulsion techniques

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The magnetic properties of ferrites are strongly dependent on the chemical composition, the cations distribution and on the preparation method in general, as well as on the structure in particular. It is well known that water-in-oil reverse micelles can be used as microreactors to produce monodispersed nanosized particles. The present work aims to prepare barium hexaferrite ($\text{BaFe}_{12}\text{O}_{19}$) powders of nanometer particles size via two types of the microemulsion technique – single microemulsion and double microemulsion. We use a water-in-oil microemulsion system with cetyltrimethylammonium bromide (CTAB) as a cationic surfactant, n-butanol as a co-surfactant, n-hexanol as a continuous oil phase, and an aqueous phase. The single microemulsion technique, in which the aqueous phase contains only the Fe^{3+} and Ba^{2+}

cations is less complicated and much less expensive than the double microemulsion one. The co-precipitation was performed by concentrated solution of NaOH at pH = 12. In the second step of the synthesis, the hydroxide precursor was calcined at 800°C temperature. We were examined the influence of the type of microemulsion techniques on the microstructure, the magneto-crystalline structure, and on the magnetic properties of the barium hexaferrite powders. The microscopy investigations show a narrower grain size distribution and the average particle size is 280 nm, well below the critical mono-domain diameter for BaFe₁₂O₁₉. The magnetic properties of the powder were investigated at 4.2 K and at room temperature. The saturation magnetization values (M_s) were obtained from the magnetization curves in high magnetic fields up to 130 kOe. The M_s were determined 43 emu/g and 48.86 emu/g for single and double microemulsion techniques, respectively. The coercive force for the two temperatures was 4.3 kOe and 3 kOe for single and double microemulsion techniques, respectively. The magnetic properties might be related to the mono-domain structure.

15:50	Poster	A43

Kinetics of the formation of nanocrystalline magnetite in a carbon matrix

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Preparation method of superfine particles of magnetite distributed in a carbon matrix was developed. A nanocrystalline iron catalyst was used as the substrate. The formation of iron carbide and subsequently iron oxides in a carbon matrix were performed in differential reactor with ideal mixing. Sample mass changes were monitored by means of a thermogravimeter. It enabled the studies of process kinetics. The carburization process was performed in a stream of pure methane in the temperature range 600 – 660°C. The processes were carried out as long as the assumed degree of iron carburization from 0 to 2gC/gFe was reached. Next the samples were cooled down under nitrogen flow to different temperatures from the range 300 - 500°C and then oxidized in the flow of nitrogen/water vapor mixture, where water vapor mole fraction ranged from 0,006 to 0,043. The phase composition and the average crystallite size of the products were determined using X-ray diffraction. The average crystallite size was calculated using the Scherrer's equation.

The rate of iron carburization degree increases linearly with the degree of iron carburization. Only higher reaction temperature, at least 450°C gives reasonable rate of oxidation, for example $r=1,3 \times 10^{-4} \text{ s}^{-1}$ for $p_{\text{H}_2\text{O}}=0,024$ and iron carburization degree equaled 1gC/gFe. The apparent activation energy was calculated as $E_a=50 \text{ kJ/mol}$. The only product of iron oxidation under these condition is magnetite. Obtained magnetite particles in carbon matrix had an average crystallites size close to 40 nm. For oxidation temperatures lower than 450°C and the mole fraction of water vapor lower than 3%vol. a mixture of magnetite and hematite was obtained.

15:50	Poster	A44

The effect of the promoters on the structural properties of nanocrystalline cobalt oxides

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Nanocrystalline cobalt oxides and cobalt are important materials for recent technologies. These materials find applications in various fields of modern research. This include various types of sensors, catalysis, electrical, electrochromic etc.. Properties of these materials depend on their preparation.

In this work influence of the promoters as aluminium and manganese on the specific surface area and average crystallite's size of the cobalt oxides was determined. It was determined how these parameters are changing after oxide phase reduction.

The influence of the aluminium content on CoO reduction was studied. The recrystallization process of the fine cobalt oxide Co₃O₄ and oxide promoted with aluminium has been studied.

In the study techniques as thermogravimetry, XRD with high-temperature cell HTK-16 and thermal desorption were used.

15:50	Poster	A45

Synthesis and Characterization of Magnetic Nano-structural Materials.

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I have prepared the nano-structural materials. The structural, magnetic and transport behavior were studied using different experimental behavior. Present work is concerned with the study of size effects on magnetic and electrical properties of Ca-based La_{0.7}Ca_{0.3}MnO₃. Size effects on magnetization and coercivity have been analyzed. A.C Susceptibility of these particles as a function of temperature has also been studied. Surface contribution was further confirmed through the measurements of Banerjee Plots and Magnetocaloric effects.

15:50	Poster	A46

Surface modification of Iron Oxide nanoparticles by the chemical and thermal treatment

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Iron oxide nanoparticles can be synthesized by sol-gel technique [1] in aqueous media or in organic solvents. It was obtained that the nanoparticles' properties strong depend on their surface characteristics. The aim of a present report was to investigate influence of heat treatment of the iron oxides nanoparticles aqueous solutions on their structure and physical properties.

Magnetite Fe₃O₄ nanoparticles were synthesized by co-precipitation from 0,01 M Fe²⁺ and 0,02 M Fe³⁺ solutions, added to ammonium hydroxide and stabilized by oleic acid. The obtained solutions were heated in 50-100°C range during 1 - 10 h.

It was shown that the nanoparticles of magemite and magnetite can be obtained by this method varying amount of the precursors. Increasing amount of stabilizer, leads to decreasing of magnetic susceptibility of the nanoparticles. Prolonged heat treatment result in the nanoparticles growth and forming of spherical aggregates with size about 40-50 nm.

The TEM analysis showed that iron oxide nanoparticles sizes can be regulated in 5-50 nm range by the amount of stabilizer. Magnetic properties of obtained solutions were investigated by the depth of solution meniscus in electromagnetic field. Peaks, at 1050 nm, were observed in the infrared absorption spectra of magnetite due charge transfer transitions Fe^{2+} - Fe^{3+} are in agreement with [2], where synthesis based on the decomposition of presynthesized iron oleate was used.

References

1. Berger, P.; Adelman, N. B.; Beckman, K. J.; Campbell, D. J.; Ellis, A. B.; Lisensky, G. C. *Journal of Chemical Education* 1999, 76, 943-8.
2. Park, J.; An, K.; Hwang, Y.; Park, J.-G.; Noh, H.-J.; Kim, J.-Y.; Park, J.-H.; Hwang, N.-M.; Hyeon, T. *Nature Mater.* 2004, 3, 891-895.

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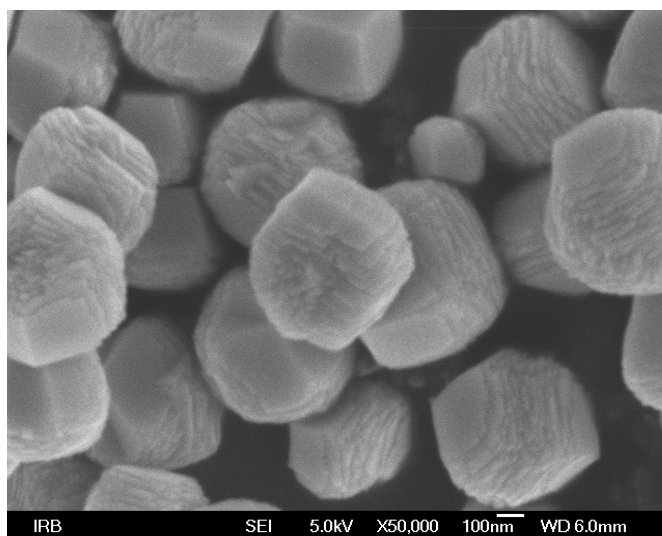
Precipitation by forced hydrolysis of Fe^{3+} ions in perchlorate-containing aqueous solutions

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Slow and forced hydrolyses of Fe(III)-salts in aqueous solutions were extensively investigated because of the importance of these reactions in natural aquatic or sediment systems and living systems on one side, and chemical syntheses and technology (corrosion problems) on the other. The hydrolyses of Fe(III)-salts in aqueous solutions are often used as models with a view to better understanding the mechanism of hydrolyses of metal cations. The kinetics, the phase composition of the precipitates, as well as the size and morphology of particles depend on experimental parameters such as temperature, pH, concentration of Fe^{3+} salts, the nature of the anion present, or some other «foreign» substance, for example surfactants. In the present work we have focused on the process of forced hydrolysis of Fe^{3+} ions in a perchlorate-containing aqueous solution. Forced hydrolyses were conducted at 90 or 160 °C. The influence of the concentration of $\text{Fe}(\text{ClO}_4)_3$, HClO_4 , NaClO_4 as well as selected surfactants, were investigated. The precipitates, as the solid products of Fe^{3+} ion hydrolysis, were analysed with XRD, Mössbauer, FT-IR and FE-SEM, and EDS techniques. Hematite ($\alpha\text{-Fe}_2\text{O}_3$) was the principal product of hydrolysis of solutions at 90 °C. However, very small amounts of goethite ($\alpha\text{-FeOOH}$) were found in the precipitates. FT-IR spectroscopy in particular was found to be very sensitive to small amounts of goethite present. Hematite was found as a single phase in the precipitates obtained at 160 °C. However, the size and morphology of hematite particles obtained at this temperature depended on the absence/presence of HClO_4 . The presence of SDS or AOT drastically changed the phase composition and microstructure of the precipitates. Hollow goethite particles were formed by the aggregation mechanism.



FE SEM image of $\alpha\text{-Fe}_2\text{O}_3$ particles obtained by forced hydrolysis of Fe^{3+} ions in perchlorate containing aqueous solution. $\alpha\text{-Fe}_2\text{O}_3$ particles are composed from nanostructured subunits.

15:50 Poster A48

Synthesis, structure and adsorption properties of SBA-15 materials functionalized with bridged and surface groups

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Ordered mesoporous organosilicas are very attractive materials due to their high surface areas, large volumes of ordered mesopores and diverse morphology what makes them attractive potential catalysts and adsorbents. Possibility of introduction of the organic and inorganic groups into the ordered structure of the final material during the synthesis is an additional invaluable advantage of the sol-gel processing of organosilicas. In the present work channel-like mesoporous organosilicas were synthesized via co-condensation of tetraethyl orthosilicate (TEOS) with bridged polysilsesquioxane monomers bearing $-\text{C}_2\text{H}_4-$, $-\text{C}_6\text{H}_4-$ bridges and alkoxysilanes bearing aminopropyl and mercaptopropyl groups. Obtained materials were characterized by IR spectroscopy, powder X-ray diffraction (XRD), transmission electron spectroscopy (TEM) thermogravimetry, elemental analysis and nitrogen sorption measurements.

Obtained materials have well-developed ordered mesoporous structure - values of specific surface area are in the range 700-1000 m^2/g and the size of the cylindrical mesopores is in the range 6.5-9.4 nm. It was determined that size of the mesopores strongly depends on small amounts of monomers co-condensing with TEOS. The XRD and TEM data shows high degree of the order of the final structures.

15:50 Poster A49

Size controlled nanostructures by PLD for nanoparticle-biomolecule hybrid systemsKenji Ebihara¹, Fumiaki Mitsugi², Tomoaki Ikegami², Takayuki Ushijima**1.** Dojindo Laboratories, Kamimashikigun, mashiki machi, Tabaru, 2025, Kumamoto 861-2202, Japan **2.** Kumamoto University, Kur-okami, Kumamoto 860-8555, Japan*e-mail: k-shrimp@road.ocn.ne.jp*

The similar dimension of biomolecules and metallic or semiconductor nanoparticles (NPs) enables the synthesis of biomolecule and nanoparticle hybrid systems. The functions of biomolecule-nanoparticle hybrid devices have been studied in many applications such as the electrical conducting of redox enzymes with electrodes, photoexcitation of semiconductor nanoparticles, and biocatalytic metallic nanoparticles. Furthermore, size-controlled magnetic NPs provides functionalization of carbon nanotubes and attractive DDS (Drug Delivery System) technology. The electrical, chemical (catalytic), biological properties of these hybrid systems can be optimized by controlling the size of the NPs. We have proposed the metallic doping technique to control the grain size or particle size of oxide materials such as ZnO and LiMn₂O₄. The pulse laser deposition using KrF excimer laser (248nm) was used to prepare the nanostructured materials on several substrates. It was shown that the additive aluminum dopant level has strong effect on size of grains, crystallinity and electrical properties of LiMn₂O₄. The grain size of LiMn₂O₄ gradually decreases with increase of aluminum dopant amount from 231nm to 96nm. The crystal size of ZnO thin films determined from Scherrer's formula is in the range of 10nm-34nm depending on atmosphere oxygen pressure at fixed dopant condition (ZnO with 2%Al₂O₃). This doping size controlling technique will be applied to preparation of Fe₃O₄ magnetic nanoparticles and luminescent nanoparticles (ZnS, CdS, CdTe).

15:50 Poster A50

Influencing the morphology of three dimensional LDH nanostructures based on amino-acid complex precursorsVanessa Prevot, Nathalie Gaillard-Caperaa, Claude Forano*BLAISE PASCAL UNIVERSITY, 24 Av des landais, Aubiere 63177, France**e-mail: vanessa.prevot@univ-bpclermont.fr*

Layered Double Hydroxides (LDHs) are very attractive materials due to their high versatility and wide range of composition. They are developed for a large number of applications as anionic exchangers, adsorbants, catalysts and catalyst precursors or as filler for polymers. These materials are described by general formula $[M^{II}_{1-x}M^{III}_x(OH)_2][A^{m-}_{x/m}H_2O]$ with $M^{II} = Mg^{2+}, Zn^{2+}, Ni^{2+}, Co^{2+}, Cu^{2+}, \dots$, $M^{III} = Al^{3+}, Cr^{3+}, Fe^{3+}, Ga^{3+}, \dots$ and $A =$ inorganic or organic anions. The great variety of metal and interlayer species combinations allows to tailor their physical (magnetic, optical, electrochemical) and chemical properties (redox and acido-basic properties) for different specific applications. LDH are usually obtained prepared by the constant pH coprecipitation method. Moreover, in order to tune and improve the textural properties of these compounds (surface area, morphology, particle size), other synthesis methods have to be proposed.

In this work, we have synthesized three dimensional nanostructures of NiAl-based LDH (5-10nm) via a novel approach using a strong alkaline solution of amino acid. Under hydrothermal treatment, the as formed complex is thermolysed into LDH matrix. Reaction parameters

such as reaction time, nature of the amino acid and reaction temperature are found to be crucial in controlling the final morphology. Scanning electron microscopy, FTIR spectroscopy and X-Ray diffraction are adopted to investigate the evolution process of Ni precursor. The morphology variation are interpreted based on the amino-acid precursor nature and its decomposition pathways. Details of the synthesis and characterization of the nanomaterials generated therefrom will be presented.

15:50 Poster A51

Synthesis of mesoporous supported materials on base of H3PMo12O40 and silicaVolodymyr Sydoruk, Valeriy Zazhigalov, Svitlana Khalameida*Institute of Sorption and Problems of Endoecology NAS of Ukraine, 13 Gen Naumova Str., Kyiv 03164, Ukraine**e-mail: bilychi@online.com.ua*

Solid tugstophosphoric acid (TPA) is very strong Brønsted acid. It is applied both in acid catalysis and as solid electrolytes. Often it is necessary to deposit it on suitable support with developed porous structure. We have used homogeneously-mesoporous silica which was prepared on basis its pyrogenic form as support. TPA is fixed chemically on silica surface. For achievement this silica was undergone pre-activation by interaction with γ -aminopropyltriethoxysilan: extent of grafting 0,34 (support 1) and 0,68 mmol/g (2). Reaction TPA with modified silica was carried out in aqueous or acetone solutions either at 20°C or under high pressure and temperature in autoclave. Some results are presented in the table. TPA is connected chemically with silica surface: absorption bands 805, 895 and 983 cm⁻¹ indicate at presence of Keggin-structure on surface, DTA-TGA at its high thermostability. The surface organic rests are also sufficiently stable. It is interesting that both hydrophobic and hydrophilic groups are on surface. Thus hybrid materials are obtained. General acidity of prepared samples which was determined by adsorption of pyridine decreases at deposition of TPA on surface. Preliminary chemical activation of support permits to obtain samples with higher concentration of acid groups than at impregnation of initial silica. Geometrical modification of support also occurs: surface area, volume and pore size change in wide limits. Support Conditions of treatment S, m²/g V, ml/g d, nm General acidity, μ mol/m² Bulk TPA 5 0,780 Initial silica 303 1,10 14,5 0,009 Initial silica Impregnation by TPA 192 0,96 20,0 0,035 Support 1 Acetone solution, 20°C 118 0,51 17,5 0,023 150°C 96 0,41 17,0 0,034 Support 2 Acetone solution, 20°C 80 0,39 19,5 0,039 150°C 49 0,40 32,5 0,065 Water solution, 200°C 89 0,39 17,5 0,052

15:50 Poster A52

Retarding effects of cellulose ethers on early Portland cement hydration studied with X-raysHeinz J. Weyer¹, Ingo Mueller², Dirk Bosbach³, B. Schmitt¹, Andrew Putnis⁴**1.** Paul-Scherrer-Institut, Villigen CH-5232, Switzerland **2.** Hercules, Düsseldorf 40599, Germany **3.** Institut fuer Mineralogie, Universitaet Muenster, Münster 48149, Germany **4.** Forschungszentrum Karlsruhe (FZK), Postfach 3640, Karlsruhe D-76021, Germany*e-mail: heinz-josef.weyer@psi.ch*

By means of a novel micro-strip detector the influence of Cellulose ethers (CEs) on cement hydration has been measured by powder diffraction at the Swiss Light Source (SLS). It has been found that these CEs have phase-specific retarding effects. The retardation is correlated with the CE adsorption on the mineral surfaces and depends on the

degree of substitution (DS) of the CE. The lower the DS, the higher the amount of adsorbed CE and the stronger the retardation. With the fast micro-strip detector the following phase reactions could be monitored in situ:

1. $C_3A + Cs^- / Cs_{0.5}^- + H \rightarrow A Ft$ (1st Ettringite formation)
2. $Cs^- / Cs_{0.5}^- + H \rightarrow Cs_2^-$ (gypsum crystallization)
3. $C_3A + Cs_2^- + H \rightarrow AFt$ (2nd Ettringite formation)
4. $C_3S / C_2S + H \rightarrow CSH + CH$ (Portlandite precipitation)

The C_3S hydration is strongly and DS-specific retarded. The retardation of gypsum crystallization is much weaker but distinguished, depending of the DS. The First Ettringite formation is not affected by the CE. This agrees with the adsorption rates, e.g. for a CE with a DS of 1.7 : 540 microgram/m² on Portlandite., 240 microgram/m² on gypsum and no adsorption on Ettringite. The second Ettringite formation does not fit into the scheme. It is strongly, DS-dependent retarded although no CE adsorbs on Ettringite. In the presence of silicates the second Ettringite formation takes place always simultaneously with Portlandite precipitation. The kinetics of the second Ettringite formation in cement might, therefore, be influenced by the Ca concentration in the pore water. The hydration of pure-phase mixtures of C_3A and sulfates without silicates was studied in experiments using water and $Cs(OH)_2$ -saturated solution. It turns out that the Ettringite crystallization is strongly inhibited in a $Ca(OH)_2$ -saturated solution.

15:50	Poster	A53

Morphology evolution, optical properties of $Cu_2PO_4(OH)$ crystals and their application to photocatalysts.

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In the synthesis of copper phosphate hydroxide, $Cu_2PO_4(OH)$, crystals via simple wet chemical route, a pH-dependent morphology evolution was observed. By changing the pH value from 2.8 to 4.0, the morphology of crystals was varied from micro-sized rod to nano-sized dendritic rods. Furthermore, the reaction time and temperature were controlled to varying the size of the copper phosphate hydroxide crystals. The crystal structure, morphology and optical properties were investigated by XRD, SEM and UV-vis spectroscopy, respectively. Photocatalytic properties were evaluated using FT-IR spectroscopy via the disappearance of stearic acid (SA). The deference of the decomposition rate with morphology was discussed in a view of the specific surface area.

15:50	Poster	A54

Structure of nanocrystalline powders of MgO prepared by sol-gel method

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Wet gels obtained by sol-gel method were dried at different conditions. Calcination at 723 K under vacuum yielded nanocrystalline MgO powder. Structure studies were performed by X-ray diffraction, scanning and transmission electron microcopies. Electron microscopy images reveal rough, unfolded and ramified structure of solid skeleton. X-ray patterns reveal the broadened diffraction lines of periclase, the only crystalline form of magnesium oxide. The gamma crystallite size distributions were determined using $FW(1/5)/(4/5)M$ method proposed by R. Pielaszek. The obtained values of $\langle R \rangle$ and σ (measure of polydispersity) of particle size parameters are about 7 nm and 2 nm, respectively, whereas the average crystallite size estimated by Williamson-Hall procedure was equal to 6.5 nm. The obtained at Rietveld refinement R_{wp} , and S fitting parameters are the order 7.0% and 1.8, respectively, seem to be satisfactory due to the nanosize of MgO crystallites and because of the presence of amorphous phase.

15:50	Poster	A55

Oxide Islands on MgO Nanocubes

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Limitations for light-emitting devices, the application of which range from full colour displays to optical communication systems, arise from the lack of materials that efficiently emit photons of required energy. Usually semiconductors where discrete electronic transitions can be reached by spatial confinement of the bulk are discussed as components for such devices. The respective situation is different for nanometer-sized alkaline earth oxides which are prototypical ionic insulators. Surface excitons with molecular-like properties can exclusively be generated at low coordinated surface sites¹ and give rise to photoluminescence emission in the range between 200 and 600 nm.

High thermal stability, a sharp size distribution and a well-defined particle morphology² make MgO nanocubes a well-suited model system for investigating the surface electronic structure of oxide particles. Unlike MgO nanocubes CaO, SrO and BaO nanoparticles agglomerate in the course of vacuum annealing and produce large grains of ill-defined morphology³. In this contribution, we present a new approach for the production of thermally stable CaO, SrO and BaO thin films supported on MgO nanocubes. UV-Diffuse Reflectance and photoluminescence measurements reveal novel optical properties compared to those of the pure oxide components. These properties will be discussed in terms of low coordinated surface elements that act as excitation and emission sites. Furthermore, chemical surface reactivity changes induced by the second metal oxide component are evidenced by the IR spectroscopic characterization of H₂ activation processes.

¹ Garrone et al. *Philos. Mag.* 1980, 42B, 683

² Stankic et al. *Angew. Chem. Int. Ed.* 2005, 44, 4917

³ Stankic et al. *J. Phys. Chem. B* 2006, 110, 13866

15:50 Poster A56

Molecular precursor routes to vanadate hosts and luminescent lanthanide-doped vanadates

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Luminescent materials are used in a wide range of devices like phosphor lamps, display monitors and for medical imaging. Typically, an inorganic luminescent material consists of the luminescent ion or activator embedded in a host lattice. In particular, lanthanides (Ln) are very attractive as activators because of their high emission efficiency and the appropriate wavelength of their f-f transitions in the visible range. The most widely used host lattices are based on oxoanions of group 5 and namely yttrium and lanthanide vanadates. Up to now, research was mainly focused on the development of new chemical compositions of phosphors prepared by conventional solid state methods but few efforts have been made to develop alternative routes that could improve their physico-chemical properties.

This work aims at developing a soft chemistry approach to prepare vanadate hosts as well as luminescent Ln-doped vanadates, and focuses on the consequences of the preparation method on the properties. The method consists in using, for the different elements to be incorporated, molecular precursors which are coordination compounds. The multi-metallic oxide materials are obtained by an appropriate thermal treatment in air of a solid homogeneous mixture of the different complexes.

For that purpose, EDTA-based complexes of general formula $(\text{NH}_4)[\text{Ln}^{\text{III}}(\text{EDTA})]\cdot x\text{H}_2\text{O}$ and $(\text{NH}_4)_3[\text{V}^{\text{V}}(\text{O})_2(\text{EDTA})]\cdot \text{H}_2\text{O}$ are synthesized, characterized and used as precursors for the preparation of zircon-type MVO_4 hosts at a moderate temperature of 800°C , as evidenced by XRD and Raman analyses. Solid solutions such as $\text{Y}_{1-x}\text{Pr}_x\text{VO}_4$ and $\text{Y}_{1-x}\text{Gd}_x\text{VO}_4$ are also obtained. Linear correlations between chemical compositions and lattice parameters or Raman shifts are observed. SEM is used to characterize the particle size and morphology. A slightly disordered intergranular porosity is observed. Finally, Ln-doped YVO_4 are prepared and their emission spectra are recorded.

15:50 Poster A57

Low temperature Synthesis of nanoparticle $\text{GdVO}_4\text{:Eu}^{3+}$ by solvothermal methodJong Won Chung¹, Hyun Kyoung Yang¹, Byung Kee Moon¹, Byung Chun Choi¹, Jung Hyun Jeong¹, Soung Soo Yi², Jung Hwan Kim³

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Gadolinium orthovanadate (GdVO_4) is an attractive host lattice for several lanthanide ions to produce efficient phosphors emitting a variety of colors. Recently, $\text{GdVO}_4\text{:Eu}^{3+}$ phosphor has been considered as a candidate phosphor for flat panel displays (FPDs) because of its good color purity. $\text{GdVO}_4\text{:Eu}^{3+}$ is an interesting red-emitting material; its most important application would be as a phosphor of flat panel displays. Because of a strong absorption to ultraviolet light by GdVO_4 , an effective of Eu^{3+} by the 450 nm strong emission of VO_4^{3-} ,

$\text{GdVO}_4\text{:Eu}^{3+}$ is a highly efficient red-emitting material. Eu^{3+} doped GdVO_4 phosphors were prepared solvothermal reaction method. This method which offers molecular or nano-level mixing of constituents leading to chemical homogeneity is very useful method to synthesize novel nano-size compounds with high crystallinity. The doping concentration of Eu^{3+} was 3 mol% and $\text{Gd}(\text{AcAc})_3$, $\text{Eu}(\text{AcAc})_3$, and $\text{VO}(\text{OR})_3$ were used as starting materials and they were dissolved in 2-Propanol. Stoichiometric amounts of above solutions were mixed and the solution was initially heated to 230°C in air and continuously stirred in a mixture for 10h. The samples were calcined at 570°C in autoclave for 5h and then sintered at various sintering temperatures in autoclave for 3h. The room temperature PL spectra of $\text{GdVO}_4\text{:Eu}^{3+}$ nanophosphors were four emissions at 611, 615, 619 and 595 nm of Eu^{3+} ion when excited by 312 nm light. Their crystalline structures, surface morphologies and phase transitions were investigated for the samples prepared at various annealing processes by using X-ray diffraction (XRD), scanning electron microscopy (SEM), and transmission electron microscopy (TEM), respectively. And luminescent characteristics were studied by measuring photoluminescence (PL) and cathodoluminescence (CL). The purpose of this study is to compare the luminescence characteristics of $\text{GdVO}_4\text{:Eu}^{3+}$ phosphors which were synthesized at different sintering temperatures.

15:50 Poster A58

Synthesis of luminescent $\text{YVO}_4\text{:Eu}$ nanocrystals in ionic liquids.

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Inorganic luminescent materials have found many practical applications ranging from full colour displays to optical communication systems. Recently, the synthesis and the spectroscopic properties of nanocrystals and nanocrystalline layers of these compounds have attracted considerable interest due to their unique physical or chemical properties and their potential application in new optoelectronic devices and in biomedical areas.

Europium doped yttrium vanadate ($\text{YVO}_4\text{:Eu}$) is an efficient red emitting phosphor and is widely used in colour television and fluorescent lamps. Here, we present a novel strategy for the synthesis of luminescent $\text{YVO}_4\text{:Eu}$ nanocrystals based on the precipitation in ionic liquids (ILs) as reaction media. The solvent is able to limit the growth of the particles and to form a stabilizing shell on the surface thus preventing particle aggregation. Due to the weakly coordinating properties of ILs, a complete removal after the synthesis should be much easier compared to standard coordinating solvents. Electron diffraction and X-ray powder diffraction patterns of the prepared nanocrystals can be identified as a tetragonal wakefieldite phase. Morphology and size of the nanoparticles were examined by electron microscopy and dynamic light scattering. The results evidence primary particles, 15 nm in diameter and a low degree of agglomeration. Photoluminescence spectra of the samples show strong emission of red light under UV-excitation.

15:50 Poster A59

Yttrium-Aluminum Garnet Synthesized in the Medium of Supercritical FluidsTadeusz Chudoba¹, Ed Lester², Martyn Poliakoff, Witold Lojkowski¹, Jun Lee, Ewa Grzanka¹, Adam Presz¹

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The pure powder of the yttrium-aluminum garnet (YAG) was obtained in a one step process utilizing supercritical (SCR) liquids in a flow reactor. Acetates (and acetyloacetates) of Yttrium³⁺ and Aluminum³⁺ in aqueous-alcoholic solutions as a starting materials were used. The obtaining product was characterized using XRD, SEM, TG, BET and helium pycnometry. Manufactured in this way YAG has high regular microstructure and very low level of agglomeration. High-Pressure-Low-Temperature-Sintering of this powder was investigated.

15:50 Poster A60

Luminescent Properties of Tb³⁺:Y₃Al₅O₁₂ – Coated Sub-micron SiO₂ ParticlesPiotr Psuja¹, Paweł Głuchowski¹, Dariusz Hreniak¹, Łukasz Marciniak², Magdalena Pietrzak², Wiesław Stręk¹

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The SiO₂ submicron particles coated with an yttrium aluminum garnet (Y₃Al₅O₁₂, YAG) layer doped with Tb³⁺ were prepared by the sol-gel method. The morphology and structure of samples determined by the X-ray powder diffraction (XRD) measurements and transmission electron microscope (TEM) images, respectively, indicated that well-crystallized nanocrystallites were form the evolution of luminescence properties. The effect of coating was analyzed by comparing properties of the Tb³⁺:Y₃Al₅O₁₂ nanocrystalline powders and composite Tb³⁺:Y₃Al₅O₁₂/SiO₂ materials. A possible applications of obtained structures is discussed in this work.

15:50 Poster A61

Synthesis and photoluminescent characteristics of red phosphors (Y,Gd)₂O₃:Eu³⁺ for VUV excitationWoo-Jung Park¹, Mong-Kwon Jung¹, Ayse Dulda², Takaki Masaki¹, Dae Ho Yoon^{1,2}

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Recently, an improved performance of displays and lamps requires high quality of phosphors for sufficient brightness and long-term stability. To improve the luminescent characteristics of the phosphors, extensive research has been carried out on rare earth-activated oxide-based phosphors because of good luminescence and chemical stability.

Among these phosphors, Y₂O₃:Eu³⁺ and Gd₂O₃:Eu³⁺ exhibit strong ultraviolet (UV) and cathode ray excited luminescence. Y₂O₃:Eu³⁺ and Gd₂O₃:Eu³⁺ are also used as composite compositions to improve the luminescent properties.

In this work, the red phosphors (Y,Gd)₂O₃:Eu³⁺ were synthesized by liquid phase precursor methods. We investigated luminescent properties of the samples by the effect of firing temperature. The samples were investigated by analyzing their particle size and crystal structure with scanning electron microscopy (SEM) and X-ray diffractometer (XRD). Also the excitation and emission spectra were measured at a room temperature using photoluminescence (PL) under UV and VUV range.

15:50 Poster A62

Synthesis of SiAlON-type Phosphors and Their Photoluminescence Property

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Rare-earth doped oxynitride or nitride phosphors have been found to have longer excitation and emission wavelengths compared to their oxidic counterparts. The luminescence is attributable to the strong nephelauxetic effect and large crystal field splitting as the activator ions are coordinated to nitrogen. Besides this oxynitride or nitride phosphors are expected to have high thermal and chemical stabilities because the crystal structure of the host lattice is built on stiff frameworks consisting of Si-N or Al-N tetrahedron.

We report yellow and green oxynitride phosphors which are suitable for use in white LEDs: Eu²⁺ activated α/β -SiAlON. α -SiAlON, isostructural with α -Si₃N₄, has a solid solution composition $M_{m/n}Si_{12-(m+n)}Al_{m+n}O_nN_{16-n}$. In this structure, $m+n$ (Si-N) bonds are replaced by m (Al-N) bonds and n (Al-O) bonds; the charge discrepancy caused by the substitution is compensated by the introduction of a modifying cation M including Li, Mg, Ca, Y and most lanthanides. The M cations occupy the interstitial sites in the crystal lattice, and are coordinated by seven (O,N) anions at three different M -(O,N) distances. The structure of β -SiAlON is derived from β -Si₃N₄ by equivalent substitution of Al-O for Si-N, and its chemical composition can be written as Si_{6-z}Al_zO_zN_{8-z} (z represents the number of Al-O pairs substituting for Si-N pairs and $0 < z < 4.2$). β -SiAlON has a hexagonal crystal structure and the P₆₃ space group.

In this structure there are continuous channels parallel to the c direction. In this work, fine SiAlON:Eu²⁺ phosphors were synthesized using a gas pressure sintering (GPS) equipment. The crystallinity, surface morphology and chemical composition were presented. And moreover, temperature dependent photoluminescence (PL) properties of the prepared SiAlON-type phosphors were investigated in a temperature range from 30 to 150 °C.

15:50 Poster A63

Microwave-assisted Synthesis of CaWO₄ Nanocrystals in Ionic Liquids

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CaWO₄ is highly interesting because of its luminescent properties [1]. The title compound has been extensively used as blue emitting phosphor (433 nm) in oscilloscopes and X-ray-detection [2]. Nanoscale

CaWO₄ may play an essential role in future luminescent thin film devices.

Different methods are known to synthesize luminescent CaWO₄ nanocrystals, e.g. hydrothermal and microemulsion techniques [3] or microwave heating in aqueous solutions [4, 5]. The demerit of these methods is the need for a strong stabilizing agent.

We have firstly developed a microwave-assisted synthesis of CaWO₄ nanocrystals in Ionic Liquids (ILs). Aim of this novel synthetic approach is to combine the advantages of ILs and microwave heating, to realise nanoscale phosphors with a high crystallinity and quantum yield [6]. Due to the polarity of ILs, intense absorption of microwaves leads to instantaneous heating. This measure results in very high reaction rates as well as in a rapid nucleation and crystallisation.

Here, non-agglomerated and resuspendable CaWO₄ nanocrystals with an average diameter of 60 nm were synthesized. The factors affecting the size, morphology and crystallinity of the product, such as reaction temperature, concentration of the reactants and type of tungstate precursor, were studied. It was possible to recycle the Ionic Liquid after completion of the reaction. Additionally, the quantum efficiency was measured and compared with bulk material [7].

[1] S.H. Yu, M. Antonietti, H. Cölfen, M. Giersig, *Angew. Chem. Int. Ed.* 41 (2002), 2356.

[2] G. Blasse, B.C. Grabmaier, *Luminescent materials*, 1994, Springer Verlag Berlin.

[3] L. Sun, M. Cao, Y. Wang, G. Sun, C. Hu, *J. Crystal Growth* 289 (2006) 231.

[4] J.T. Klopogge, M.L. Meier, L.V. Duong, R.L. Frost, *Mater. Chem. Phys.* 88 (2004) 438.

[5] J.H. Ryu, J.W. Yoon, K.B. Shim, *Electrochem. Sol. Stat. Let.* 8 (2005) D15.

[6] G. Bühler, C. Feldmann, *Angew. Chem. Int. Ed.*, 118, (2006) 1.

[7] M. Mai, C. Feldmann, in progress.

15:50	Poster	A64

Growth of silica nanoparticles in two step condensation of silicon tetramethylorthosilicate

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Hydrolysis and condensation of metal-organic precursors is an interesting process for the design of Nanomaterials, in particular de fabrication of nanoparticles for emerging applications. One issue of this process is the control of the chemical stability of the solutions and the minimization of the condensation-nucleation processes at the desired stage of particle formation. For the fabrication of nanoparticles, process parameters have to be adjusted in order to avoid gelation or to stop particle growth in the solution. Common approaches to control the chemical reactions in the "sol" state have been applied by using complexing agents in order to control precursor reactivity toward the hydrolysis and condensation. Once the nanoparticles are formed, particle growth or network formation needs to be controlled in order to avoid the formation of clusters, agglomerates or gelation. Two ways commonly used to limit particle growth or cluster formation has been by limiting solution concentration and by surface treating the nanoparticles. In this work, a process to stabilize sol-gel solutions without additives is presented. The process allows significant reduction of the kinetics of condensation resulting in controlled nanoparticles size in relatively

high concentration solution which is stable over the time. The finding is also interesting for practical purposes. TMOS was used to produce silica nanoparticles in the two step thermal condensation process. Stable silica solutions with colloids of 70 nm were produced. The solutions were stable for more than six month. The solutions were characterized by laser granulometry, turbidity and viscosity techniques.

15:50	Poster	A65

Sol-gel derived silica particles doped with metals

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Chemical modification of the silica surface by thiol groups allows to obtain functionalized silica materials. It has been observed that the presence of thiol groups improves the adsorption of several metal ions to the silica [1]. Therefore, silica can effectively anchor metal complexes and metal ions. SiO₂ particles, modified in this way, could be used in order to explore some of practical applications such as ion exchange, preconcentrations of metals from dilute solutions, catalysis of enzymatic reactions and metal-supported reactions [2].

The silica particles with different size (400-600 nm or less) and modified with thiol groups have been obtained by the sol-gel method. As a silica precursors, tetraethoxysilane (TEOS), 3-mercaptopropyltrimethoxysilane (MPTMS) and 3-mercaptopropyltriethoxysilane (MPTES) have been used. Gold, silver, platinum or other metal ions have been doped to colloidal solution of SiO₂ particles and then, they have been reduced. The silica particles doped with metallic elements have been obtained and characterized by scanning and transmission electron microscopy (SEM and TEM), EDAX and IR spectroscopy.

Keywords: silica particles, metallic elements, sol-gel, thiol group

[1] G. Hernández, R. Rodríguez, *J. Non-Cryst. Solids*, 246 (1999) 209-215.

[2] A.R. Cestari, C. Airolidi, *J. Colloid Interface Sci.*, 195 (1997) 338-342.

15:50	Poster	A66

Optical and structural characterization of zirconia-titania-silicate glasses

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The paper presents a study on obtaining and characterization of silicate glasses from the oxide systems: R₂O (R: Na, K) – R'O (R': Ca, Mg, Zn) – ZrO₂ - SiO₂. A supplementary amount of titanium oxide was added, in order to improve the refractive index, average dispersion and Abbe coefficient of the final glass. These oxide systems are investigated aiming to replace the conventional lead crystal glass, where harmful compounds as PbO and BaO are replaced by ZrO₂ and TiO₂. Ware glass, stained glass, decorative products and technical applications are the main uses of these vitreous materials.

The samples were optical and structural characterized by UV-VIS, FTIR and Raman spectroscopy. FTIR transmission spectra in the range 4000-400 cm^{-1} , put in evidence ZnO_4 , ZnO_6 , TiO_4 , TiO_6 , ZrO_6 , SiO_4 groups as well as Si-O-Ti and Ti-O-Ti groups. This confirms that TiO_2 is a network former together with SiO_2 , providing a mixed oxide network.

Raman spectra of TiO_2 -containing glass samples show vibration peaks characteristic for silicon oxide and new ones, attributed to Si-O-Ti bonds. The samples containing ZrO_2 , reveal a shift of TiO_2 Raman peaks to low energy, which also reveals the formation of Zr-O-Ti vitreous mixed network.

15:50 Poster A67

Formation mechanism of Zn_2SiO_4 nanoparticles depending on the Zn/Si ratio of Zn-Si-O films

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Low dimensional materials have attracted much interest due to potential to optoelectronic and/or electronic device applications using their unique properties. From the viewpoint of practical applications, fabrication of low dimensional materials directly on silicon substrates, which are mostly used for various devices, is important issue. In this work, Zn-Si-O films with various compositions were sputter-deposited on Si (100) substrates and post-annealed by using rapid thermal process at 700 °C for 3 minutes in a nitrogen atmosphere. The composition of Zn-Si-O films ranging from Zn/Si=0.4 to Zn/Si=11.2, measured by EDS equipped with high-resolution TEM (HRTEM), could be obtained by controlling the area ratio of ZnO to Si target. As a result of HRTEM analysis, the microstructures of Zn-Si-O films were strongly dependent on the Zn/Si ratio. Three types of microstructure were evolved depending on the Zn/Si ratio. When the Zn/Si ratio was below ~1.6, nanoparticles were distributed in an amorphous matrix of SiO_x . In this case, nanoparticles exist almost independently. When Zn/Si ratio was ~4.0, nanoparticles are almost interconnected and nanoparticle-network structure was developed. When Zn/Si ratio was ~11.2, ZnO film structure was formed, in which ZnO film was grown in a preferred orientation of [002] normal to substrate surface. From the HRTEM analysis, the nanoparticles could be identified as a Zn_2SiO_4 phase. Consequently, Zn_2SiO_4 nanoparticles could be simply synthesized by controlling the film composition and post-annealing conditions, which show strong UV-blue cathodoluminescent emission leading to optoelectronic device application. The mechanism of formation of a Zn_2SiO_4 nanoparticle will be systematically discussed based on the HRTEM analysis of Zn-Si-O films and the XRD results of Zn_2SiO_4 bulk annealed at various temperatures.

15:50 Poster A68

Optical properties of Zn_2SiO_4 nanoparticles embedded in SiO_x amorphous matrix

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Optoelectronic materials compatible with silicon substrates are important from the viewpoint of practical applications for silicon photonics. In this work, we have fabricated Zn_2SiO_4 nanoparticles embedded in SiO_x amorphous matrix showing a strong cathodoluminescent emission in the UV-blue region by adjusting the composition of Zn-Si-O film and post-annealing temperature. The Zn-Si-O films with various compositions of Zn/Si ratio=0.4~11.2 were sputter-deposited on Si (100) substrates by changing area ratio of ZnO to Si target and were post-annealed at the temperatures of 400~1000 °C for 3 minutes in a nitrogen atmosphere by rapid thermal annealing process. Among them, the film with Zn/Si ratio=~1.6, which was annealed at 700 °C, showed a strong and broad cathodoluminescence (CL) emission ranging from 350 to 550 nm. Especially, the UV-blue peak around ~400 nm was remarkable. Weaker UV-blue emission peak was also observed from the film with smaller Zn/Si ratio. However, this UV-blue emission peak disappeared as the Zn/Si ratio increased. The high-resolution TEM analysis showed that the films with Zn/Si ratio below ~1.6 contained Zn_2SiO_4 nanoparticles with a size of ~5 nm which were distributed in the SiO_x amorphous matrix. However, the Zn/Si ratio larger than the optimized value for the formation of Zn_2SiO_4 nanoparticle caused a formation of nanoparticle-network structure and further increase of Zn/Si ratio resulted in a formation of ZnO film structure. Therefore, the UV-blue emission peak around ~400 nm is thought to be attributed to Zn_2SiO_4 nanoparticles, which is supported by the fact that the intensity of this UV-blue emission peak increased with increasing the number of Zn_2SiO_4 nanoparticle. The origin of the strong and broad CL emission containing the UV-blue emission from the Zn-Si-O film with Zn/Si ratio=~1.6, leading to a possible application to Si-based optoelectronic devices, will be discussed analytically.

15:50 Poster A69

X-ray reflectivity analysis of SnO_2 thin films used as gas sensors and biochip components

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Semiconducting SnO_2 thin films are often used as an active element in gas sensors technologies¹. An emerging application is the use as electrode or sensitive element in biochip technologies². These two applications require a good control of the composition, microstructure and physical properties of the film. The films have been fabricated from metal-organic precursors by using a submicroscopic aerosol pyrolysis deposition method. This elaboration method allows obtaining films with small amounts of catalytic metallic aggregates such as Pt or Pd by codeposition. This incorporation can drastically improve the films sensitivity to polluting gas. The best sensitivity is obtained from a low concentration of the metal in the film. A previous local microstructural analysis of Pt-doped SnO_2 thin films prepared by this deposition method have shown that the porosity of the films is significantly affected by finely varying the metal amounts³. To correlate these local structural evolutions with statistical information on the film porosity, X-ray reflectivity analysis has been performed from a series of films loaded with various contents of Pt (0 at. % to 6 at. % in the precursor solution calculated as $[\text{Pt}]/([\text{Sn}] + [\text{Pt}] + [\text{O}])$). To obtain a reliable X-ray reflectivity data, larger samples (e.g. 2 to 4 inch Si wafers) have been prepared by using a specific large area deposition method. The results indicate that the film density increases with increasing loading (from ~75% to 95% of SnO_2 bulk value for used range of Pt contents in the precursor solution). The films analysis is combined with Trans-

mission Electron Microscopy and Atomic Force Microscopy microstructural analysis. Results will be discussed in terms of potential improvement for applications in chemical sensor technologies.

¹ M. Gaidi et al., J. Electrochem. Soc. 147 (8) (2000) 3131

² V. Stambouli et al. Sensors and Actuators B 113 (2006) 1025

³ I. Matko et al., J. Electrochem. Soc. 149 (8) (2002) H153.

15:50 Poster A70

2D GISAXS Analysis of Morphology in Nanostructured Cerium/ Vanadium Oxide Thin Films

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Thin films of mixed cerium and vanadium oxides were prepared by sol-gel process and dip-coating on glass substrates. The applications of these films as electrodes in electrochromic devices make specific demands upon their morphology and structural properties. The information about the film performance quality was obtained by electrochemical measurements performed under lithium intercalation. The aim of this work was to investigate which particular properties of structure and film morphology are characteristic for the films with best electrochemical measurement results and how the processing of the film material, as well as the intercalation of small ions, affects its properties. As the films were deposited on glass substrates, a non-destructive technique for examination was required. One of such techniques is the grazing-incidence small-angle X-ray scattering (GISAXS). It is particularly suitable for porous nanostructured films as ours. The GISAXS measurements were performed at the synchrotron ELETTRA in Trieste, Italy. Two-dimensional GISAXS spectra for each film were measured at different grazing angles. By varying the grazing-incidence angle, different regions of the film can be probed. The surface and bulk contributions to scattering, as different functions of sample to scattering angle orientation, can be clearly distinguished. Scattering measurements with two-dimensional detectors also allow a specific film depth-structural profiling. Besides the estimation of the primary morphological characteristics - the average nano-size of the scatterers and the properties of the inner scatterer/pore surface, such measurements can give information about the orientation of the scatterers, which depends on their position in film. The results of the described scattering measurements for thermally processed films of mixed cerium and vanadium oxides with different morphologies, before and after intercalation of lithium ions, are presented.

15:50 Poster A71

Fabrication and properties of nanocrystalline $\text{Eu}^{3+}:\text{SnO}_2$ and $\text{Eu}^{3+}:\text{SnO}_2/\text{In}_2\text{O}_3$ thin film

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The europium doped SnO_2 nanocrystallites were obtained by modified Pechini method. Next, the powders were added to $\text{SnCl}_4 \cdot 5\text{H}_2\text{O}$ and dissolved in isopropyl alcohol (IPA). The mixtures with different $\text{Eu}^{3+}:\text{SnO}_2$ to SnCl_4 ratio were prepared. After few hours of mixing suspension was deposited on glass slides by spin-coating method and treated in 550 °C. The same procedures have been done for $\text{Eu}^{3+}:\text{SnO}_2/\text{In}(\text{NO}_3)_3/\text{SnCl}_4$. Optical and electrical properties of obtained layers have been determined and discussed.

15:50 Poster A72

Growth and characterization of ITO/ZnO nanofiber thin films deposited by spray pyrolysis

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Transparent conducting oxide (TCO) thin films have received extensive attention because of their diverse applications as transparent electrodes for optoelectronic devices such as solar cells and flat panel displays. Recently, this search has been oriented toward zinc oxide nanoporous in order to fabricate the low cost of new generation organic semiconductor based photovoltaic devices. In this work, transparent conducting ZnO thin films were prepared by spray pyrolysis technique using $\text{ZnCl}_2 (2\text{H}_2\text{O})$ as a precursor aqueous solution with concentration of 0.1M/l. The layers were deposited onto Sn-doped In_2O_3 (ITO) thin film using soda lime glass (SLG) substrate heated at 350°C. The ZnO films were annealed in room air, two hours, at 300°C. These films are crystallized in the hexagonal structure and are stoichiometric. The samples are semiconductors with n-type conductivity. Scanning electron microscope (SEM) shows that the films are highly porous with small grain size and their diameter f is smaller than 100 nm. A cross section visualisation of ZnO film shows a thickness around 200 nm. X-ray diffraction (XRD) exhibits a strong evidence of ZnO crystallites orientation at $2\theta = 34.28^\circ$ according to the line (002). Transmittance measurements at 300 K varies versus substrate temperature (T_s) and yielded band gap values of 2.5 and 3.34 eV for $T_s = 300^\circ\text{C}$ and 400°C , respectively. Here we discuss also the fabrication of such nanostructures and present results from ZnO nanofiber/polymer composite photovoltaic devices.

Key words: Nanoporous ZnO, Transparent Conductive Oxide, Spray Pyrolysis, Hybrid solar cells.

15:50 Poster A73

Fabrication of Metal Nano Dots Using Block Copolymer Thin Film

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In general, feature size greater than 500nm is routinely produced by photolithography techniques and for feature size between 500nm and 30nm, electron beam lithography is commonly used. However, feature size less than 30nm is hard to achieved with the above mentioned standard semiconductor lithography techniques. The technical trend of nanolithography shows some novel techniques, such as nano patterning by using diblock copolymer can be a promising candidate.

In this work, we have fabricated dense and uniformly distributed arrays of nano dots on SOI (silicon/silicon dioxide/silicon) surface. SOI substrates were prepared by low pressure chemical vapor deposition

(LPCVD) on Si substrates, and cleaned by Pirhana cleaning to remove organic chemicals. Si surfaces with balanced interfacial interactions of polystyrene (PS) and polymethylmethacrylate (PMMA) were prepared by anchoring 3-(p-methoxyphenyl) propyltrichlorosilane (MPTS, $\text{CH}_3\text{OPh}(\text{CH}_2)_3\text{SiCl}_3$, > 95%) to the native silicon oxide layer.

To obtain nano size patterns, self-assembling resists were used to produce a layer of uniformly distributed parallel cylinders of PMMA in a PS matrix. The PMMA cylinders were degraded and removed by acetic acid rinsing, forming a PS mask to transfer the pattern. The patterned holes of PS template were approximately 20 nm wide, 40 nm deep, and 50 nm apart. 100 Å-thick Au, 30 Å-thick Cr, Ni, and Al thin film was deposited by using e-beam evaporator. PS template was removed by lift-off process using N-formyldimethylamine (DMF). As a result, the nano template of PS was analyzed with field emission scanning electron microscope (FESEM) and the sizes of Cr, Ni, Au and Al nano dots left on Si surface were measured by atomic force microscope (AFM). The sizes of metal nano dots were in the range of 17–22 nm.

15:50 Poster A74

Porphyrim controlled anisotropic and dendritic growth of thallium chloride crystals

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Fabrication of nano- to microscopic-scale inorganic materials with special size and morphology are of great interest for materials chemistry due to their importance in basic scientific research and potential technological applications. In the past few decades, there has been an increasing number of reports on the synthesis of inorganic crystals, such as rods, wires, plates, tubules, dendrites. Of these shapes, hierarchical dendrites have attracted much attention stimulated by the practical importance related to some fractal growth phenomena. Studies on the shape control of inorganic crystals will to some extent give insights into the crystallization behavior in a nano- or micro-sized scale owing to the traditional lack of understanding of the growth history and shape evolution process. In this work, the morphology of TlCl crystals grown at the liquid-liquid interface between TlSO₄ and HCl solutions was investigated in the presence of various porphyrin molecules. The addition of TPPS porphyrin molecules changed the morphology from spherical to single-crystalline wires with a width of 100–2000 nm. The addition of TPPyP changed morphology of TlCl crystals into branching dendrites. The presence of different porphyrins molecules in the crystallization medium significantly influenced the morphology of the crystals. Crystals of TlCl with elongated rod-type or branching superstructures were nucleated with high aspect ratios depending on porphyrin type was used. The composition and microstructure of dendrites was investigated by X-ray technique, SEM and AFM. The wires were produced by elongation and stacking of planar TlCl crystals exhibiting {100} faces. The anisotropic growth behavior causing the wire formation is tentatively ascribed to stepwise capping of the specific surfaces of TlCl crystals by porphyrin molecules.

15:50 Poster A75

Synthesis and Electrochemical Properties of the Misfit-Layered Cobaltite Nanomaterials

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We report on a preparation of nano-sized calcium cobaltite materials with good homogeneity and their detailed lithium reactivity for the application to Li-ion battery electrodes. Derived from Na_xCoO_2 , the cobaltite, $\text{Ca}_3\text{Co}_4\text{O}_9$ has a misfit structure alternating $[\text{CoO}_2]$ and $[\text{Ca}_2\text{CoO}_3]$ with CdI_2 - and rocksalt-type layers, respectively. Compared to the carbon-based anode, transition metal oxides have an extremely large reversible capacity arising from their displacement reactions. Accordingly, we designed a series of $\text{Ca}_3(\text{Co}_3M)\text{O}_9$ compounds in order to substitute cation sites in this layered structure with transition-metal, M ($M = \text{Co}, \text{Cu}, \text{and Fe}$). After the intermediate formation of polybasic chelates using citric acid, $\text{HOC}(\text{CH}_2\text{CO}_2\text{H})_2 \times \text{CO}_2\text{H}$ with metallic ions, each powder was successfully produced at 700°C for 2 hours in O_2 atmosphere. The obtained powders show a two-dimensional morphology consisting of very thin mica-like nano-platelets. We found that these cobaltite nanomaterials show excellent lithium reactivity, which results in a high reversible specific capacity. In addition, the formation of nanocomposite composed of highly dispersed transition-metal nano grains embedded into a Li_2O and CaO matrix with pseudo-amorphous character is also observed during the charge/discharge process, using a high resolution transmission electron microscopy.

15:50 Poster A76

Optical Properties In Mechanically Alloyed ZnO-Based Ternary Oxides

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Cation substitution modifies the band width of ZnO leading in consequence to a change in optical and electronic properties for its potential use in many demanding technological applications. Mechanical milling has proved to be an effective and simple technique to produce nano-crystalline powders and the possibility of obtain large quantities of materials. In this work we present a structural and optical characterization of M-alloyed ZnO (M : Mg and Cd) powders obtained by mechanical milling. The starting materials were commercially ZnO, MgO and CdO powders. $\text{Zn}_{1-x}\text{M}_x\text{O}$ samples with different concentrations of M-element (x between 5–30%) were prepared by mechanical milling in a vibratory horizontal ball mill. In a previous work [1] we analyzed the evolution with milling time of cation substitution by X-ray diffraction and positron annihilation spectroscopy. As milling proceeds an unambiguous reduction of grain size and homogenization are observed. It is the aim of this work the Raman and optical characterization of the ternary alloys. The energy position of the fundamental absorption edge shifts to UV or IR depending of the guest element. From the analysis of Raman spectra the incorporation of the M-element in the ZnO lattice

can be followed. The lattice disorder introduced by the mechanical work is also evidenced in the Raman features.

[1] "Composition influence on positron annihilation parameters in ZnO-based nanocrystal semiconductor powders", L.C. Damonte, M.A. Hernández Fenollosa, V. Donderis and B. Marí, *Physica (C)* (2007) in press.

15:50

Poster

A77

Spectroscopic studies of magnon excitations in nanosized NiO

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Magnon excitations in pure nickel oxide (NiO) were successfully probed in the past by Raman spectroscopy [1,2]. Recently we have extended these studies to $\text{Ni}_x\text{Mg}_{1-x}\text{O}$ solid solutions [3,4], where unpredicted behavior of temperature and composition dependences of one-magnon excitation energies has been observed.

In this work, we present the Raman scattering in nanosized NiO powders, produced by the plasma synthesis [5]. Single-crystal NiO, grown by the method of chemical transport reactions on MgO(100) substrate, was also used for comparison. Raman experiments were conducted in a wide range of temperatures from 10 to 300 K.

The Raman spectrum of single-crystal NiO shows several bands in the probed region above 400 cm^{-1} . The first four bands have vibrational origin and, according to assignment in [3], correspond to one-phonon (1P) TO and LO modes (at $\sim 570\text{ cm}^{-1}$), two-phonon (2P) 2TO modes (at $\sim 730\text{ cm}^{-1}$), TO + LO (at $\sim 906\text{ cm}^{-1}$) and 2LO (at $\sim 1090\text{ cm}^{-1}$) modes. The last strongest band at 1490 cm^{-1} is due to a two-magnon (2M) scattering. It was observed that the intensity of two-magnon band decreases rapidly in nano-sized NiO. This effect is attributed to a decrease of antiferromagnetic spin correlations and leads to the antiferromagnetic-to-paramagnetic phase transition.

References:

- [1] R. E. Dietz, G. I. Parisot, A. E. Meixner, *Phys. Rev. B* 4, 2302 (1971).
- [2] M. Grimsditch, L. E. McNeil, D. J. Lockwood, *Phys. Rev. B* 58, 14462 (1998).
- [3] E. Cazzanelli, A. Kuzmin, G. Mariotto, N. Mironova-Ulmane, J. *Phys.: Condens. Matter* 15, 2045 (2003).
- [4] E. Cazzanelli, A. Kuzmin, G. Mariotto, N. Mironova-Ulmane, *Phys. Rev. B* 71, 1344151 (2005).
- [5] J. Grabis, I. Steins, D. Rasmane, G. Heidemane, J. *European Ceramic Soc.* 17, 1437 (1997).

Symposium B

Programme

Monday, 17 September

Opening & Plenary Session

Czochralski Award Ceremony & Acta Gold Medal Ceremony
Monday morning, 17 September, 10:00

Lunch break

Monday afternoon, 17 September, 12:30

Introductory session

Monday afternoon, 17 September, 14:00

14:00 Invited

Magnetic anisotropy of ultrathin layers grown by electrochemistry on Au(111)

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Anisotropy is one extremely important property of magnetic material, thin films and nanostructures because it determines device functioning and performances. Magnetic anisotropy is sensitive to the atomic scale environment as well as to long range crystallographic structure. For instance, the magnetic moment of magnetic ultrathin films may be out of plane while the 2D shape would impose an in-plane orientation. This phenomenon, called perpendicular anisotropy (PMA) is a manifestation of the strong influence of material crystallographic structure, elastic strains, hybridization at interface. PMA is of considerable interest for magnetic data storage. We have recently demonstrated that Co/Au(111) layers grown by electrochemistry from a modified Watts bath [1] present a strong PMA after capping the cobalt layer with a Cu film since M is out of plane for a cobalt thickness ~ 5 ML [1]. We also obtained epitaxial Co/Au multilayers in which each individual Co layer presents a perpendicular magnetization [2].

In this presentation, we will first review data coupling *in situ* STM observations and real time *in situ* Polar Magneto optical Kerr Effect (PMOKE) measurements to discuss the magnetic properties of iron group metal layers electrodeposited on Au(111) layers. In the case of cobalt we will discuss the unique behaviour observed at submonolayer surface coverage. New investigations concerning the influence of surface chemical modification will be also presented.

References

- [1] L. Cagnon et al. Phys. Rev. B 63(2001) 104419
- [2] P. Prodhomme et al., J. Magn. and Magn Mat, 315 (2007) 26

14:30

Invited

Electrochemistry-based fabrication of semiconductor nanotemplates and two-dimensional semiconductor-metal ordered networks for electronic and photonic applications

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Nowadays nanotemplates based on porous alumina are commercially available. However, the high resistivity nanomatrix of porous alumina plays only a passive role in nanofabrication. At the same time it is well known that the properties of semiconductor materials can be easily modified by external illumination, applied electric and/or magnetic fields etc., thus implying that semiconductor nanotemplates may in fact play an active role in nanofabrication. We report on the fabrication of semiconductor nanotemplates based on III-V and II-VI semiconductor compounds, namely on InP ($E_g = 1.3$ eV at 300 K), CdSe ($E_g = 1.7$ eV), GaP ($E_g = 2.3$ eV), and ZnSe ($E_g = 2.7$ eV), using electrochemical and chemical wet etching techniques. Growth of ordered two-dimensional hexagonal arrays of nanochannels is achieved thanks to self-arrangement phenomena. Membranes with mirror-like surfaces and pore diameters ranging from about 50 nm to several hundreds of nanometers were fabricated using the proposed technological approaches. We demonstrate the possibility to grow ordered arrays of metal nanowires and nanotubes using pulsed electrochemical deposition of Pt, Cu and Ag from solutions of relevant salts. A strong charging effect is observed in metal nanowires and nanotubes due to the potential barrier at the metal-semiconductor interface. Characteristics of novel varicap structures based on metal-semiconductor interpenetrating networks are presented. Possible applications of the developed two-dimensional metallo-dielectric periodic networks as photonic band-gap structures are discussed.

15:00

Invited

Facile synthesis of uniform NH_4TiOF_3 mesocrystals and their conversion to TiO_2 mesocrystals by topotactic transformation

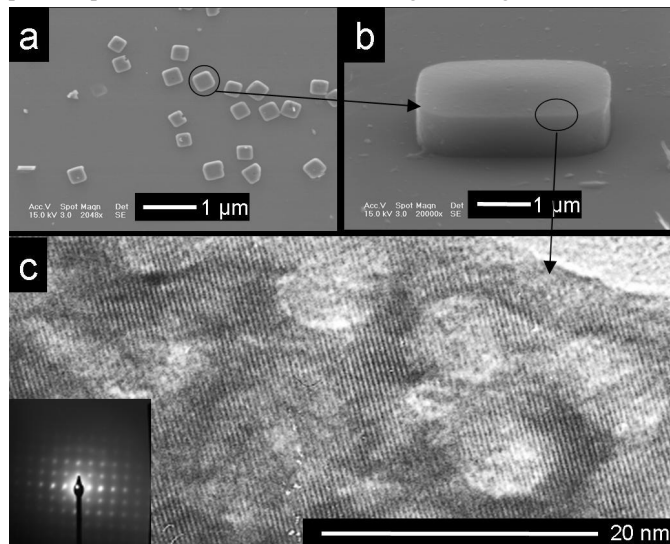
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Mesocrystals are an interesting class of nanomaterials, similar to colloidal crystal assemblies but differing by processing superstructures of vectorially (rather than randomly aligned) non-spherical nanocrystals. The nearly perfectly-ordered nanocrystalline subunits results in single-crystal-like electron diffraction patterns. Mesocrystals and their syntheses also provide a challenge to accepted views on crystal growth as is typically described by a process of nucleation and growth; and/or Ostwald ripening. Uniform mesocrystals of anatase TiO_2 have been prepared by a topotactic transformation from NH_4TiOF_3 . Mesocrystals of NH_4TiOF_3 were synthesised from an aqueous solution containing $(\text{NH}_4)_2\text{TiF}_6$ and H_3BO_3 in presence of Brij series surfactants at room temperature. The exterior shapes of NH_4TiOF_3 mesocrystals can be

tuned by adjusting the reaction parameters including: reagent concentration, reaction time, reaction temperature and rate of stirring. The formation of the mesocrystals of NH_4TiOF_3 proceeds *via* self-assembly process involving non-classical kinetically driven processes and amorphous intermediates. By sintering in air at 450 °C or washing with H_3BO_3 solution at ambient temperatures, the NH_4TiOF_3 mesocrystals can be converted to anatase TiO_2 mesocrystals by self-templated topotaxial conversion, thus retaining their original architecture.



SEM (a & b) and TEM (c) images of NH_4TiOF_3 mesocrystals

Coffee break

Monday afternoon, 17 September, 15:30

Joint Poster Session (Part I)

Monday & Wednesday

Monday afternoon, 17 September, 15:50

Tuesday, 18 September

Electrodeposition of semiconductors and nanowires

Tuesday morning, 18 September, 9:00

9:00

Invited

Electrodeposition and Combustion Synthesis of Oxide Semiconductors for Fun and Profit

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Semiconductors are generally prepared by high temperature methods or by the use of vapor phase and ultra-high vacuum environments. Milder temperatures coupled with the use of condensed media, using water or other solvents (organic compounds or ionic liquids) represent interesting and economic alternatives for the growth of semiconductors in either bulk or thin film form. Electrodeposition is one such preparative methodology in this category, which already has seen widespread acceptance in the metallurgical and microelectronics sectors. This method is attractive from both fundamental and practical perspectives in terms of applicability to large and irregular device areas and the

ability to prepare composite (e.g., metal/semiconductor) structures and unique semiconductor morphologies (e.g., nanotubes, nanodots). Anodic and cathodic electrosynthesis of metal oxide films and nanoporous/nanotubular morphologies will be briefly reviewed in terms of the underlying chemistry/electrochemistry and the mechanistic aspects. A new pulse anodization approach will be described along with data on titanium dioxide and tungsten trioxide nanotube and nanoporous films.

Another attractive method for the preparation of oxide semiconductors is combustion synthesis. This method offers an energy-efficient alternative to the high-temperature, energy intensive routes that are popular in the ceramics community. One attractive feature of this approach is the possibility it offers of in situ doping of oxide semiconductor hosts by simply tuning the precursor chemistry. New data on Nb-, Cr- and S-doped titanium dioxide will be presented as well as results on the enhanced visible light response of these materials relative to benchmark titania samples (e.g., Degussa P-25).

9:30

Invited

Electrodeposited metal nanowires on glass

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Electron beam lithography (EBL), invented in the early 70's, provides a means for patterning polycrystalline metal nanowires as small as 20 nm in diameter onto surfaces. The applicability of EBL, however, has been limited to research and development applications because it is a serial patterning technology. In 1990, a parallel version of EBL was described by Berger and Gibson, but space charge "blurring" has prevented this technique from approaching the resolution of direct-write EBL. Heath and coworkers recently described a novel method for depositing high density arrays of linear, 10 nm diameter metal nanowires onto etched semiconductor superlattices prepared by molecular beam epitaxy. We have demonstrated that ensembles of 30 nm diameter metal nanowires can be prepared by localizing the electrodeposition of metal on the step edges present on a graphite surface, but no control of nanowire position or inter-wire pitch is possible using this method. Lithographically Patterned Nanowire Electrodeposition (LPNE) is a new method for synthesizing noble metal nanowires on glass or oxidized silicon surfaces. LPNE involves the preparation of a sacrificial nickel nanoband electrode using optical lithography and the subsequent electrodeposition of a metal (Au, Pt, Pd, or Bi) nanowire at this electrode. However there is an important wrinkle: The nickel nanoband electrode is recessed into a horizontal trench defined by the insulator surface and photoresist. The electrodeposition of a nanowire into this "nanoforn" produces a wire with a rectangular cross-section where the height and width are independently controllable with ≈ 5 nm precision, down to 20 nm in width and 6 nm in height. Since optical lithography is used to define the position of the nanowires on the surface, the spacing between nanowires on the surface is constrained by the optical diffraction limit.

10:00

Oral

Influence of pulsed electroplating conditions on the nucleation, morphology and properties of bismuth telluride films

Virginie Richoux, Sebastien Diliberto, Clotilde Boulanger, Jean-Marie Lecuire

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Continuous advances in electronics and micro–nanotechnologies require the use of generators, local coolers and temperature stabilizers with a significantly reduced size, a light weight and long life duration. The thermoelectric technology is a good alternative to fabricate such devices. This technology can be easily miniaturized in the opposite of compressors and battery in reason of the absence of moving parts. Most Peltier devices for cooling and power generation applications are made with bismuth telluride-based compounds, the best materials to date for room-temperature. These materials were already synthesized using the continuous electrodeposition [i]. However, the thermoelectric properties of the electroplated films are lower than the single crystal due to the presence of defaults and cracks.

In order to optimize the morphology and the physical properties of these materials, it is possible to have recourse to pulsed electrodeposition. This advanced form of electrodeposition offers better control over deposit properties by controlling the interfacial supply and electrochemical reaction. A high number of variables offers effective ways to control macroscopic properties [ii].

This study presents the synthesis of Bi₂Te₃ films by pulsed electrodeposition on gold substrate. The influence of the cathodic potential or current density and the electrolyte concentration over stoichiometry and nucleation mode was studied. In order to measure the thermoelectric parameters of these films, the gold substrate was replaced by a 30 nm Bi₂Te₃ film deposited by an electroless method [iii]. Then, the power factor of pulsed electrodeposited films (5 µm) on this substrate was determined.

[i] P. Magri, C. Boulanger, J.M. Lecuire, *J. Mater. Chem.* **6** (1996) 773.

[ii] D. Del Frari, S. Diliberto, N. Stein, C. Boulanger, J.M. Lecuire, *J. Appl. Electrochem.* **36** (2006) 449.

[iii] L. Scidone, S. Diliberto, N. Stein, C. Boulanger, J.M. Lecuire, *Materials Letters* **59** (2005) 746.

10:15

Oral

One-step electrochemical synthesis of polypyrrole nanostructures, without using a template.

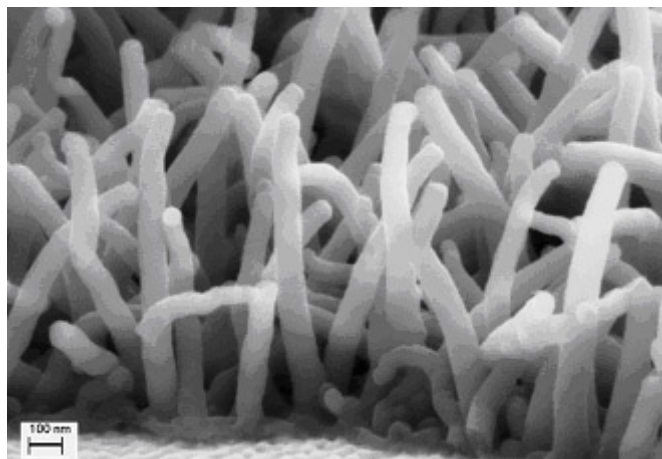
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Nanostructured conductive polymers have attracted growing attention due to their potential applications. Up to now, conducting polymer nanowires were primarily obtained with porous membrane as template. In this communication, the electrosynthesis of wire/tube-like polypyrrole nanostructures without using a template will be presented. These structures are obtained by electrochemical oxidation of pyrrole in the presence of jointly weak-acid and non-acid anions. The growth mechanism of these nanostructures will be discussed. Notably, it will

be highlighted that the presence, at the electrode-solution interface, of weak-acid anions is essential.



Coffee break

Tuesday morning, 18 September, 10:30

Electrodeposition of semiconductors

Tuesday morning, 18 September, 11:00

11:00

Oral

Deposition of nano-crystalline lead chalcogenide thin films using a simple electrochemical technique

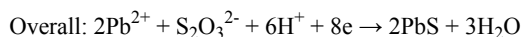
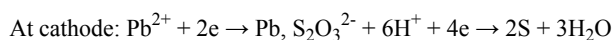
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Nano-crystalline thin films of lead chalcogenides (PbS, PbSe and PbTe) were deposited on transparent conducting oxide (TCO) coated glass substrates using a simple galvanic technique. The basic electrochemical cell consisted of a Pb strip as a self-decaying anode, with TCO coated glass substrate as the cathode and a Pb(CH₃COO)₂ solution. Na₂S₂O₃, H₂SeO₃ and HTeO₂⁺ were introduced in the cell for PbS, PbSe and PbTe thin film depositions, respectively. No external bias was applied for the depositions; instead, the two electrodes were short circuited externally by a copper wire. The deposition potential was achieved from the difference in the electrode potentials. The Pb²⁺ was complexed to Pb-EDTA in all the cases, so that, no other Pb salts are precipitated through chemical reactions. A pH of 3 for the working solution was found to be optimum for all the depositions. Depositions were carried out at room temperature, without stirring of the solution. The following are the cathode reactions for the three electrochemical processes:

For PbS:



For PbSe:



Overall: $\text{Pb}^{2+} + \text{SeO}_3^{2-} + 6\text{H}^+ + 6\text{e}^- \rightarrow \text{PbSe} + 3\text{H}_2\text{O}$

For PbTe:

At cathode: $\text{Pb}^{2+} + 2\text{e}^- \rightarrow \text{Pb}$, $\text{HTeO}_2^+ + 3\text{H}^+ + 4\text{e}^- \rightarrow \text{Te} + 2\text{H}_2\text{O}$

Overall: $\text{HTeO}_2^+ + \text{Pb}^{2+} + 3\text{H}^+ + 6\text{e}^- \rightarrow \text{PbTe} + 2\text{H}_2\text{O}$

The anodic reaction $\text{Pb} \rightarrow \text{Pb}^{2+} + 2\text{e}^-$ is the same for all the three cases.

X-ray characterization revealed that PbS, PbSe and PbTe thin films were all of cubic phase. The formation of nano-crystallites with dense and compact surface morphology was detected from FESEM and AFM measurements. EDX analysis showed metal deficient compositions, indicating p-type nature of the films. The nano-crystalline films showed 'blue shifted' IR absorption, which changed to bulk values when the crystallites were allowed to grow large. The nature of the n-TCO/p-PbX (X = S, Se, Te) hetero-junction has been studied.

11:15

Oral

Direct electrochemical deposition of CuInSe_2 absorber layers

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Large scale PV (Photovoltaic) energy production needs large-area solar-cell installations. Thin-film solar cell deposition on soda-lime glass, stainless steel, or polyamide substrates is a cheap technology, which would make these solar cells truly lightweight and large area. The benefits electrodeposition can provide are convenient and inexpensive production of large-area solar cells, utilizing a technology readily adaptable to industrial production.

The report presents a new modified approach to the thermodynamics and kinetics constituent of the electrodeposition mechanism of CuInSe_2 . It will be shown that the mechanism CuInSe_2 phase formation goes through a number of consecutive reactions. The formation of Cu + Se phases precedes the assimilation of indium and CuInSe_2 compound formation. Under the conditions of electrodeposition all three elements in the one-step electrochemical deposition process, the formation of Cu+Se phases are controlled by the diffusion of selenium and copper species, the insertion of indium into the film depends on the applied potential.

The report describes developed associated model used to determine the fluxes of copper, indium and selenium species to the cathode and relative concentrations of deposited solid phases. The results of computations enabled us to build the distribution diagram of the final products.

It will be shown that the morphology of electrodeposited films is determined by the nucleation rate of crystals (is the function of overpotential) and the rate of mass transport of solute species.

11:30

Oral

New insights in the electrodeposition mechanism of CuInSe_2 thin films for solar cells applications

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Optimizing the electrodeposition of multinary compounds is of key importance for tomorrow technologies. Cu-In-Se system represents a case example, with a great potential in the field of photovoltaics. At IRDEP we achieved efficiencies up to 11.5% based on electrodeposited Cu-In-Se material.

The electrochemical deposition of this ternary compound, whose components exhibit very different redox potentials and several oxidation states, is a complex process.

In the present work, the electrocrystallization mechanism of CuInSe_2 is investigated, in dilute acidic solutions, by polarization and impedance spectroscopy using a rotating disc electrode. The bulk composition is determined by x-ray fluorescence. The morphology is characterized by SEM and the structure by X-ray diffraction.

At low polarizations, no indium is deposited, binary Cu-Se phases are obtained. These phases catalyse the reduction of Se(IV) species into elemental selenium, which, in turn, enables the incorporation of indium beyond a potential threshold close to $-0.6\text{V}/\text{MSE}$. At higher overpotentials, CuInSe_2 chalcopyrite phase is formed, together with elemental selenium and additional Cu_xSe phases. In the intermediate potential zone corresponding to indium incorporation, the polarization curve exhibits a current peak similar to a passivation process. A specific impedance response with a large capacitive feature with a negative polarization resistance is observed. This indicates the adsorption of a blocking, poorly conducting compound.

A reaction path is proposed which accounts for the main experimental features. This leads to a better understanding of key parameters controlling the composition and the structure of the layers. The proportion between different phases and their organization at the nanoscale in the film are governing the recrystallization processes during post-thermal annealing and finally the photovoltaic properties.

11:45

Oral

A green approach towards deposition of CdS thin films by an electrochemical technique

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The galvanic technique for CdS thin film deposition has been modified, so that the spent solution hardly contains free toxic Cd^{2+} ions. In electrochemical deposition of CdS, even the spent solution contains sufficient Cd^{2+} ions, disposal of which is a problem. The typical galvanic deposition of CdS involves an electrolyte solution containing Cd^{2+} and $\text{S}_2\text{O}_3^{2-}$ ions. Two electrodes immersed in the solution, one

TCO (transparent conducting oxide) coated glass, acting as the cathode and a Cd strip, acting as a sacrificial anode, completes the cell. No external bias is applied for deposition; instead, the two electrodes are short circuited externally by a Cu wire, the difference in the electrode potential serves as the deposition potential.

We have shown that CdS thin films can still be deposited without the prior presence of Cd^{2+} ions in solution. It contained only 0.01M $\text{Na}_2\text{S}_2\text{O}_3$, pH of which was adjusted to around 3. Deposition was carried at 85°C with stirring. After constituting the cell, Cd dissociates leading to release of Cd^{2+} ions in solution ($\text{Cd} = \text{Cd}^{2+} + 2\text{e}^-$, $E^0 = 0.403\text{ V}$). The two electrons move via the external circuit to the TCO cathode, where the released Cd^{2+} ions again get reduced to Cd ($\text{Cd}^{2+} + 2\text{e}^- = \text{Cd}$); the $\text{S}_2\text{O}_3^{2-}$ ions simultaneously get reduced to S ($\text{S}_2\text{O}_3^{2-} + 6\text{H}^+ + 4\text{e}^- = 2\text{S} + 3\text{H}_2\text{O}$, $E^0 = +0.50\text{ V}$); the two reduced elements react to produce CdS on the TCO surface.

X-ray data showed the deposited CdS film to be of cubic phase. EDX analysis yielded Cd:S atomic percentage about 1:1.03. SEM image revealed a uniform and compact surface morphology. Optical absorption study gave band gap energy around 2.36 eV. Resistivity of the films was in the range of $10^5 - 10^6\ \Omega\cdot\text{cm}$.

Hence, the presence of a cadmium solution in the galvanic cell is not necessary for CdS film deposition, the Cd electrode decays to provide the Cd^{2+} ions for film deposition, keeping the solution practically clean of Cd^{2+} ions after the deposition, and thus enabling the process to be green.

12:00 Oral

Synthesis and structural study of Diluted Magnetic Semiconductors by electrodeposition

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Spintronics has attracted great attention since the discovery of a giant magneto-resistance in magnetic multilayers. Spintronic devices combine the advantages of magnetic materials and semiconductors and are expected to be non-volatile, versatile, fast and capable of simultaneous data storage and processing, while at the same time, consuming less energy. Among the different classes of material systems the diluted magnetic semiconductors (DMS) have received much attention because of the great advantage of integrating with conventional semiconductor materials and devices and the challenge is to increase their Curie temperatures for room temperature operation.

DMS are semiconductor compounds in which a few fractions of host elements are replaced by magnetic ions. Different methods for synthesizing DMS thin films have been reported in literature to obtain these magnetic materials. In this work we will present how electrodeposition is an effective method to obtain magnetic ternary oxide thin films. This technique presents some interesting advantages as the deposition occurs at low temperature and at atmospheric pressure, it is a low-cost method with requires only simple apparatus, the film thickness can be directly monitored by the charge consumed during the deposition process. The unique restriction is that conducting substrates can be used.

For some oxides, such as ZnO and the magnetic materials derived from it, $\text{Zn}_{1-x}\text{Co}_x\text{O}$ and $\text{Zn}_{1-x}\text{Mn}_x\text{O}$ the compound is directly electrodeposited in a crystalline form and present high crystal quality. In this work, $\text{Zn}_{1-x}\text{Co}_x\text{O}$ and $\text{Zn}_{1-x}\text{Mn}_x\text{O}$ thin films were synthesized by means of the cathodic electrodeposition technique and the influences of Co and Mn contents on the structural and optical characterizations of

$\text{Zn}_{1-x}\text{Co}_x\text{O}$ and $\text{Zn}_{1-x}\text{Mn}_x\text{O}$ thin films were discussed and compared in detail.

12:15 Oral

Optical properties of nanostructured mesoporous semiconductor films

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Nanostructuring of semiconductors and metals is now a clear route to optimise and elicit new functional properties, e.g. allowing quantum engineering of electron and photon states. Hence materials with mesoporous nanoarchitectures (2–50 nm) exhibit novel electronic, optical and magnetic properties in comparison to their non-mesoporous counterparts. Recently we have devised an elegant and highly versatile route to the production of a wide range of nanostructured materials with well-defined mesoporous architectures of extended spatial periodicities. This method utilises lyotropic liquid crystalline (LC) mesophases of non-ionic surfactants as templates for electrochemical growth. Here we report the fabrication of adherent nanostructured semiconductor films such as cadmium telluride and lead telluride that have been fabricated by this method. Our results indicate that nanostructuring produces strong birefringence for wavelength both above and below the bandgap of the mesoporous semiconductor films. These exciting results show that electrodeposition from LC phases can be used to access a wide range of Group VI, II-VI and related semiconductor films (e.g. CdSe, CdS, PbSe). Such materials would be of considerable fundamental interest because they are entirely composed of surface atoms (no atom in the structure is further than 1.5 nm from a surface). As a consequence we expect that these materials should exhibit unusual charge carrier dynamics, improved collection of the photogenerated carriers and hence solar conversion efficiencies. Nanostructured semiconductors will show quantum size effects by virtue of their reduced dimensionality and nanometre lengthscales (from 2 to 15nm). The ability to electrodeposit mesoporous nanostructured semiconductors highlights the possibility of creating a new class of materials that would exhibit unusual electronic and optical properties.

Lunch break

Tuesday afternoon, 18 September, 12:30

ZnO topical session I

Tuesday afternoon, 18 September, 14:00

14:00

Invited

Electrodeposition of Porous Crystalline ZnO/Dye Hybrid Thin Films and Development of Plastic Solar Cells

Tsukasa Yoshida^{1,2}, Daisuke Komatsu^{1,2}, Jingbo Zhang¹, Keigo Ichinose^{1,2}, Torsten Oekermann³, Kazumasa Funabiki⁴, Masaki Matsui⁴, Daniel Lincot⁵

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Porous crystalline ZnO/dye hybrid thin films are self-assembled by cathodic electrodeposition from aqueous mixed solutions containing Zn salt, dye and O₂. These materials exhibit high performance as photoelectrodes in dye-sensitized solar cells (DSCs) because of the high surface area due to three dimensionally interconnected nanopores formed inside of ZnO grains. At the same time, they are well crystallized unlike the conventional electrodes based on sintering of TiO₂ nanoparticles to achieve fast transport of electrons. Because the process requires neither high temperature nor aggressive chemicals, it permits use of plastic electrodes.

We have carried out extensive electrochemical and chemical analysis on ZnO/eosinY hybrid system. We have also carried out structural characterizations that elucidated the internal hybrid nanostructure of the film. Switching of electrocatalytic behavior of eosinY to O₂ reduction was found to be the key mechanism for the evolution of the hybrid materials.

The highest conversion efficiency of 5.6% (AM 1.5) has been achieved when D149 dye (Mitsubishi Paper Mills) is used as a sensitizer. For further improvement, development of sensitizers specially suited for ZnO is necessary. We are developing new organic sensitizer dyes especially for near IR region that could not be utilized by D149.

These solar cells are light weight, flexible in shape and colors. Recent progress in application of colorful plastic solar cells by industrial partners will also be presented.

14:30

Invited

Electrodeposition of ZnO nanowire arrays

Ramon Tena-Zaera¹, Jamil Elias¹, Claude Lévy-Clément¹, Ivan Mora-Sero², Yan Luo², Juan Bisquert²

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Single-crystal ZnO nanowire arrays have emerged as promising building blocks for a new generation of devices in different technological domains such as optoelectronics, solar cells, gas sensing, field emission and piezoelectrics. Among the various deposition techniques, electrochemical deposition appears as a versatile method well suited to synthesize ZnO with a large variety of morphologies as well as epitaxial thin films. It is a low temperature method, well suited for

cheap large-scale production. After reviewing the state of the art of the electrodeposition of ZnO, this presentation will focus on the deposition of arrays of ZnO nanowires (NWs). I will show, using more precisely the electroreduction of molecular oxygen method, that vertically aligned ZnO nanowires with tailored dimensions and electrical properties were obtained. The role of the composition and concentration of the electrolyte on the ZnO growth mechanisms will be emphasized. The anions from the zinc precursor and supporting electrolyte were found to play a major role in the synthesis of ZnO, opening new routes to tailor the dimensions of the nanowires. ZnO NWs with a diameter between 25 and 500 nm and a length up to 10 µm were successfully attained, resulting in high aspect ratios up to 50. The electrical properties of the ZnO NW arrays were analysed by electrochemical impedance spectroscopy. A cylindrical model which takes into account the geometry of ZnO NWs was developed, allowing to determine the carrier density of the ZnO NWs. Donor densities between 10¹⁷ and 10²⁰ cm⁻³ were obtained as a function of the deposition conditions and thermal treatments. Those results showed that playing the electrolyte composition and concentration allows not only to monitor the dimensions of the ZnO NWs, but also their electrical properties. In conclusion, the application of electrodeposited ZnO NW arrays in nanostructured ETA-solar cells will be discussed.

15:00

Invited

Electrodeposition of ZnO hybrid layers – role of molecular adsorbates and substrate structure on a way towards efficient photoelectrodes

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The preparation of porous ZnO films is of interest to utilize the sensitization of wide-bandgap semiconductors for a photoelectrochemical photovoltaic cell. Electrochemical deposition of ZnO films from aqueous Zn salt solutions is an attractive deposition method because of a wide accessible range of crystal size and film texture. If molecular adsorbates are added to the deposition bath, films of high porosity but still maintained high crystallinity can be prepared. Eosin Y is a very suitable molecule to serve this purpose. The influence of the molecular adsorbates represents an important optimization parameter. The reactions are compatible with a large number of conductive substrates, even textile fibers.

In this study, ZnO/Eosin Y hybrid materials were electrodeposited on (0001) GaN and on (0001) ZnO to allow a detailed investigation of the structure and texture of such electrodeposited films. Scanning electron microscopy (SEM) revealed the formation of domains with different crystal sizes pointing at a varying density of nucleation sites on the substrate. X-ray diffraction showed the expected preferential orientation with the c- plane of ZnO parallel to GaN (0001) at good in-plane orientation as seen in rocking curves. The peak positions of the diffractogram were shifted corresponding to a lattice expansion by 3.6 % in the c- direction. This clearly indicated the strong influence of the Eosin Y molecules adsorbed during the growth of ZnO.

Conductively coated textiles on the other hand, represent a substrate of high technical interest since textile- integrated electronics, sensing

elements and also solar cells will offer almost perfect opportunities to utilize portable electronics. Textiles are characterized by a very rugged nature, light weight and extreme flexibility. Depositions on textiles on the other hand ask for an optimized control of deposition conditions, hence a detailed kinetic analysis appears necessary.

Coffee break

Tuesday afternoon, 18 September, 15:30

ZnO Topical session II

Tuesday afternoon, 18 September, 15:50

15:50

Oral

Tuning the pore size in electrodeposited ZnO films using surfactants as structure-directing additives

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Dye-sensitized p-n solar cells are a concept to avoid the leakage of the liquid electrolyte used in conventional dye-sensitized solar cells by replacing it by a p-type semiconductor. CuSCN (p-type) electrodeposited into porous electrodeposited ZnO (n-type) is a promising combination due to the usually good charge carrier transport in electrodeposited semiconductors. Porous ZnO films can be obtained by co-deposition with large organic molecules, e.g. dye molecules with functional groups that interact with the growing ZnO film. However, a systematic tuning of the pore size, which will be important in order to find the optimum pore size for CuSCN deposition, has not been achieved yet with these additives. We have now explored the possibilities of surfactants, which are often used as additives in sol-gel methods, in the electrodeposition of ZnO. With anionic surfactants such as sodium dodecyl sulfate lamellar ZnO structures are deposited if the surfactant concentration in electrodeposition bath is above half the critical micelle concentration (cmc), i.e. when surface micelles start to form on the growing ZnO film. The distance between the ZnO layers can be tuned by changing the chain length of the surfactant. However, an easily accessible porosity like that formed by micelles in sol-gel films could not be observed. Larger pores compared to those in ZnO films deposited with dye molecules or surfactants alone could be obtained by a combination of both kinds of additives. The anionic dye eosin Y and the cationic surfactant n-dodecylpyridinium chloride in concentrations above the cmc were found to form mixed aggregates. SEM images show pore diameters in the range of several tens of nanometers, compared to only 10 nm if only eosin Y is used. Preliminary results of CuSCN deposition in ZnO films with different pore sizes will also be reported.

16:05

Oral

Electrochemical synthesis of ZnO in the presence of Dye or polymer additives.

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In recent years, electrodeposition has emerged as a versatile process for the preparation of zinc oxide thin films. Mesoporous thin films of ZnO were prepared by an electrochemical method using eosin Y (EY) as a structure directing agent (SDA). In the hybrid ZnO based film, EY is incorporated after a two electron reduction process [1] and forms a continuous fibrous structure [2]. Interestingly, EY can be almost completely removed from the film and a transparent porous ZnO films is released [3]. The specific surface area and mesoporosity of films prepared at various EY concentration and deposition time have been characterized in detail by physical adsorption using krypton as the adsorptive [4]. The preparation conditions have been optimized to obtain films with both the largest possible surface area and good mechanical properties. The preparation of ZnO/polymer hybrid films will be also illustrated [5]. Due to the complexing properties of the polymer for Zn^{2+} , this compound can be incorporated in a large amount in ZnO films and strengthening their mechanical properties for applications in flexible devices. It has been found that the polymer controls the crystallographic properties of ZnO. The strong film orientation can be varied between [002], [101] and [100] with increasing polymer concentration. Furthermore, higher polymer film content leads to a better crystallographic quality of the compound as shown by the increase in the intensity ratio between near band edge (NBE) UV emission and deep level green emission in room-temperature photoluminescence spectra.

References :

[1] A. Goux, T. Pauporté, D. Lincot, L. Dunsh, *ChemPhysChem*, **2007**, 8, 926-931.

[2] A. Goux, T. Pauporté, D. Lincot, *Langmuir*, **2006**, 22, 10545.

[3] Yoshida T., Iwaya M., Ando H., Oekermann T, Nonomura K, Schlettwein D., Wöhrle D., Minoura H., *Chem. Commun.* **2004**, 4, 400.

[4] Rathousky J., Pauporté T. *J. Phys. Chem. C*, DOI: 10.1021/jp071465f

[5] Pauporté T, *Crystal Growth Design*, submitted.

16:20

Oral

Templated electrodeposition and hydrothermal growth of nanostructured arrays of zinc oxide: similarities and differences

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Electrochemistry has been used to deposit zinc oxide in track etched polymer membranes and to monitor the composition of the reaction solution during hydrothermal growth of zinc oxide nanostructures on seeded substrates. This presentation will consider the advantages and disadvantages of the two techniques.

Electrodeposition in a track etched polymer membrane involves two steps; (i) electroreduction of either hydrogen peroxide or nitrate ions to alter the local pH within the pores, (ii) precipitation of the metal oxide within the pores. Synthesis at 25°C via the reduction of hydrogen peroxide yielded polycrystalline zinc oxide nanorods. When deposition was performed at 90°C, using the reduction of nitrate to control the local pH, zinc oxide nanorods which displayed the same growth direction along their entire length were obtained. The length of all as-grown rods increased with the integrated charge passed. The growth direction

of the ZnO rods obtained at the higher temperature was unusual, being perpendicular to the (1,0,1(bar), 1) plane.

The immersion of a material, seeded with ZnO nanoparticles, in an aqueous solution of $\text{Zn}(\text{NO}_3)_2$ and hexamethylenetetramine (HMT) at 90°C yielded an extended array of one-dimensional ZnO on the substrate surface. The structure of the ZnO evolves with reaction time. Initially nanorods are formed. At longer times the rods are tipped with nanotubes. Here we report a series of experiments in which both the composition of the reaction solution; concentrations of H^+ , Zn^{2+} and HMT; and the structure of ZnO deposited on the substrate are monitored as a function of reaction time. It is shown that the change from ZnO rod to tube growth arises when the solution composition is such that it is no longer thermodynamically favorable to precipitate $\text{Zn}(\text{OH})_2$.

16:35 Oral

In-situ Synchrotron Experiments and Modelling of the Growth of Electrochemically Deposited ZnO Nanostructures

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ZnO is known to produce a wide variety of nanostructures that have enormous scope for optoelectronic applications. Using an aqueous electrochemical deposition technique, it is possible to tightly control a wide range of deposition parameters (Zn^{2+} concentration, temperature, potential, time) and hence the resultant deposit morphology. By simultaneously conducting synchrotron x-ray absorption spectroscopy (XAS) experiments during the deposition, we are able to directly monitor the growth rates of the nanostructures, as well as providing direct chemical speciation. X-ray scattering provides information on texture evolution during film growth. *In-situ* experiments such as these are critical to understanding the nucleation and growth processes of such nanostructures.

We present recent results from *in-situ* synchrotron experiments demonstrating the growth behaviour as a function of potential and Zn^{2+} concentration. These are compared with the electrochemical current density recorded during the deposition, and the final morphology revealed through *ex-situ* high resolution electron microscopy. The results are indicative of two distinct growth regimes, and simultaneous changes in the morphology are observed.

These experiments are complemented by modelling the growth of the nanostructures in the transport-limited case, using the Nernst-Planck equations in 2 dimensions, to yield the growth rate of the volume, length, and radius as a function of time.

17:05 Oral

Electrowetting study of redox Self assembled layers on ZnO nanostructures prepared by electrodeposition

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Oxides are known as the basis of smart devices. Among the group of oxide, we were interested in Zinc Oxide (ZnO) synthesis for its potential applications in many fields [1]. In our laboratory, a variety of nanostructures were fabricated including nanorods and nanowires of ZnO by electrodeposition from a ZnCl_2 solution with O_2 bubbling [2].

Self assembled monolayers on nanorods and nanowires of ZnO were prepared from the attachment of a ferrocene derivative: N-(3-trimethoxysilyl)-propylferrocenecarboxamide. The different steps of this synthesis were characterised by RMN, FTIR and ESI.

The reversible one-electron wave of the ferrocene/ferricinium was observed at a potential close to the one in solution. The system was found very stable in both organic and aqueous media. Electrowetting study (contact angle variation within the potential applied on a sessile drop) was carried out on these monolayers. We observed a decreasing in contact angle while increasing the potential. This was attributed to the ferrocene oxidation at the interface.

These redox devices have many applications in photovoltaic and chemical detection of organic and inorganic ions.

References

- [1] a) M. H. Huang, Y. Wu, H. Feick, N. Tran, E. Weber, P. Yang, *Adv. Mater.* **2001**, *13*, 113. b) Z. R. Dai, Z. Pan, Z. L. Wang, *Adv. Funct. Mater.* **2003**, *13*, 9.
[2] a) C. Badre, T. Pauporté, M. Turmine, D. Lincot, *Superlattices and Microstructures*, in press. b) A. Goux, T. Pauporté, J. Chivot, D. Lincot, *Electrochim. Acta* **2005**, *50*, 2239.

Discussion session

Tuesday afternoon, 18 September, 17:35

Wednesday, 19 September

Plenary Session

Wednesday morning, 19 September, 9:30

Coffee break

Wednesday morning, 19 September, 10:30

Chemical bath deposition I

Wednesday morning, 19 September, 11:00

11:00 Invited

Chemical bath deposited Zn-compound buffer layers as heterojunction partner for Cu-chalcopyrite thin film solar cell devices

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Chemical bath deposition (CBD) techniques can offer convenient, low cost and environmentally compatible tools to address a number of manufacturing thin films for different application especially in photovoltaic. CBD has played a crucial role in thin film solar cells based on the Cu-chalcopyrite material system. At present, device efficiencies up to 19.5 % for low-gap $\text{Cu}(\text{In,Ga})\text{Se}_2$ and around 12 % for high-gap CuInS_2 have been demonstrated. All these high efficiency values so

far have been reported for solar cell devices with CBD-CdS buffer layers between the well-conducting n^+ -ZnO/i-ZnO window bi-layer and the Cu-chalcopryrite thin film absorber. CBD-CdS has also proven successful in the industrial pilot line and the technology of chalcopryrite solar modules with the conventional CdS buffer as hetero-junction partner is close to commercialization. However many material issues are not fully understood: a) why is the CdS buffer necessary?, b) is Cd diffusion into the absorber and n-type doping of the absorber surface creating a buried junction? c) is a Cu-deficiency phase present at the chalcopryrite surface?, d) What is the role of the band offsets at the buffer/absorber interface?, etc. In addition CdS as a heavy metal compound should be avoided in the final solar modules as well as in their production processes from an ecological as well as an economical point of view. Therefore a large number of alternative materials have been developed for the deposition by CBD. We report on the progress done for understanding the role of CBD and the recent development on alternative buffers in chalcopryrite solar cells. Particular interest will be given to the development of ZnS/Zn(S,O) bi-layer as alternative to CdS. Analysis of CuInS₂/ZnS/Zn(S,O) interfaces, the diffusion/intermixing processes affecting the absorber/buffer hetero-interface and the resulting device performances are discussed.

11:30 Invited

From Nanocrystalline to Monocrystalline Thin Films: Microstructure, Morphology Evolution and Epitaxy in Chemically Deposited Semiconductor Films

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Chemical deposition from solution (CD) offers a simple and cost-effective route for the fabrication of high quality semiconductor thin films, and is useful for the deposition of a large variety of semiconductors. Lead selenide and lead sulfide semiconductor films deposited using CD can be obtained in a wide range of morphologies, from nanocrystalline films, through microcrystalline films with columnar grains, to epitaxial monocrystalline films. This is achieved by effectively controlling the active deposition mechanism during growth: The cluster mechanism, in which clusters of the semiconductor material are formed in solution and migrate onto a solid support to form a film, or the ion-by-ion mechanism, in which the reaction occurs (and the film forms) directly on the substrate surface. Strategies for improving the material quality and for controlling orientation of the films will be discussed. In addition to conventional in-house characterization techniques, we will highlight *in-situ* techniques such as laser light scattering and synchrotron grazing incidence x-ray diffraction for monitoring film growth and morphology evolution in real time.

12:00 Invited

Chemical bath deposition of CdSe and CdS nanocrystalline films: tailoring of morphology, optical properties and carrier dynamics

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Chemical bath deposition (CBD) has been used for decades to deposit thin films of various semiconductors [1]. Recently, there is a new interest in the CBD which is motivated by its successful use in depositing buffer layers of CdS (and similar materials) in thin-film photovoltaic cells based on CdTe and CuInSe₂ [1]. We review the results of our research towards tailoring the CBD-deposited films morphology, composition and optical properties for foreseen applications in photonics, spintronics, etc. We have been concentrating on the films consisting of closely-spaced nanocrystals of CdSe and CdS with radius from 2 to more than 10 nm [2-4]. We describe the effect of chemical (e.g., the concentration of individual components, the solution pH, ...) and physical (e.g., illumination, deposition temperature, ...) parameters of the deposition on the properties of the films. These were studied by TEM, XRD, EMP, optical absorption, photoluminescence and Raman spectroscopy and ultrafast laser spectroscopy. On basis of the obtained results, in particular of photoexcited carrier dynamics [5], we propose a microscopic model describing well the optical properties of nanocrystalline films. This work was supported by Ministry of Education of the Czech Republic in the framework of the research plan MSM 0021620834.

References

- [1] G. Hodes, Chemical Solution Deposition of Semiconductor Films, Marcel Dekker, Inc, New York, Basel, 2003.
- [2] F. Trojaneck, R. Cingolani, D. Cannoletta, D. Mikes, P. Nemec, E. Uhlířová, J. Rohovec, P. Maly, J. Crystal Growth 209, 695 - 700 (2000).
- [3] P. Nemec, I. Nemec, P. Nahalkova, K. Knižek, P. Maly, J. Cryst. Growth **240**, 484-488 (2002).
- [4] P. Nemec, M. Simurda, I. Nemec, D. Sprinzl, P. Formanek, P. Maly, J. Cryst. Growth **292**, 78-86 (2006).
- [5] M. Simurda, P. Nemec, F. Trojaneck, K. Neudert, T. Miyoshi, K. Kasatani, P. Maly, Physica E 36, 205-210 (2007).

Lunch break

Wednesday afternoon, 19 September, 12:30

Chemical Bath Deposition II

Wednesday afternoon, 19 September, 14:00

14:00 Oral

Chemical bath deposition of single-phase Pb_{1-x}Cd_xS films and their optical properties

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According to the phase diagram, at room temperature, CdS is almost insoluble in PbS. However, metastable $\text{Pb}_{1-x}\text{Cd}_x\text{S}$ films have been described using both vacuum evaporation and chemical bath deposition (CBD). While there are some reports implying solid solution over a wide range, values of x up to nearly 0.4 (for evaporated films) and 0.2 (for CBD films) have been supported by experimental evidence. For the CBD samples, mixed phases are sometimes reported, and even where this was not the case, it is not clear that only one phase was deposited.

Our experience in repeating previous recipes for $\text{Pb}_{1-x}\text{Cd}_x\text{S}$ is that CdS was also deposited along with the solid solution, but that this CdS is often not seen in XRD (hence it would be easily overlooked if XRD were the main characterization method).

We describe a bath which gives single-phase $\text{Pb}_{1-x}\text{Cd}_x\text{S}$ films with x varying up to 0.15. Because of the small variations in lattice parameters with varying x , we use not only accurate XRD, but also atomic absorption chemical analysis for characterization.

Few studies have been made on the optical absorption (transmission) spectra of these solid solution films and none on CBD films. We correlate these optical spectra with the film composition and find, as did earlier studies using evaporated films, that the optical bandgap is a strong function of the Cd concentration, with a bandgap of ca. 0.9 eV obtained for the highest Cd contents (compared to 0.4 eV for pure PbS). This increase in bandgap is much stronger than expected based on a simple linear relationship between PbS and (rocksalt) CdS with a bandgap of ca. 1.7 eV.

14:15 Oral

Zn(S,O,OH) Chemical Bath Deposited buffer layers for high efficiencies Cu(In,Ga)(S,Se)₂ solar cells : a better understanding of the growth mechanism

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In the next few years, one of the major objectives in the field of CuInS_2 and Cu(In,Ga)Se_2 PV technology is the development and rapid implementation in production line of Cd-free alternative for the currently used CdS buffer layer. Zinc-based buffer layers, prepared by Chemical Bath Deposition (CBD), have already demonstrated their potential to obtain high efficiencies solar cells. However, the understanding of both deposition mechanism and film properties is by far lower than what is the current status of CBD of CdS. We report here a study of the synthesis of zinc-oxy-sulfide films by CBD in a wide and rigorous experimental parameter window.

For industrial applications, the priority is to achieve scalable CBD ZnS buffer layer with efficiencies comparable to CdS using a simple and short deposition time process (lower than 10 min), with minimum of chemicals and which can be easily extended to stable modules.

In this paper first a theoretical approach, with thermodynamic calculations of ZnS, ZnO and Zn(OH)_2 solubility are presented taking into account in particular the effect of temperature and the complexing agent such as ammonia over a wide pH range. In parallel in-situ growth studies by quartz crystal microbalance has been made in order to have

a better understanding of the growth mechanism and to establish a general equation concerning the deposition growth. Correlations between film properties obtained by XPS, XRF and SEM with deposition conditions are established. Optimized conditions lead to high efficiency devices, competing very well with classical CdS.

14:30 Oral

Effects of different Zn precursors on Zn(S,O) buffer layers deposited by chemical bath for chalcopyrite based Cd-free thin film solar cells

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The effect of different Zn salt precursors on solar cell device performance is investigated using low band Cu(In,Ga)(S,Se)_2 (CIGSSe) and large band gap CuInS_2 (CIS) absorbers provided by AVANCIS and SULFURCELL Solartechnik respectively. The deposition of a Zn(S,O) layer by chemical bath deposition (CBD) process is described. This process has been successfully developed for the deposition of Zn(S,O) buffer layers on laboratory scale samples using CIS absorbers from the technology department (SE3) at the Hahn-Meitner-Institut [1,2] and is currently being up-scaled to substrates up to 30cm x 30cm. During the first minutes of the process the solution stays relatively clear. After some time, which depends on the CBD conditions, there is an increase of the turbidity probably due to the formation of Zn(OH)_2 . This turbidity has been monitored using an optical setup. Light from a laser source is scattered by the particles formed in the solution. The transmitted light is detected at a Si-sensor and plotted vs. time. Transmission electron micrographs of glass/Mo/CIS/Zn(S,O) structures show a conformal coverage of the absorber by a Zn(S,O) layer of 15-25 nm consisting of nanocrystals with radii of ~5 nm. Scanning electron micrographs (SEM) reveal different crystal structures depending on the zinc precursor used and on the temperature of the CBD.

The results for small area (0.5 cm²) single cells as well as large area modules (10x10 cm² and 30x30 cm²) with interconnected cells are also discussed. The new process shows similar results compared to CdS base line and can be successful in an industrial pilot line.

[1] German Patent. International Publication Number: WO 2006/0180013 A1

[2] A. Ennaoui, M. Bär, J. Klaer, T. Kropp, R. Sáez-Araoz and M. Ch. Lux-Steiner

Highly-efficient Cd-free CuInS_2 Thin-film Solar Cells and Mini-modules with Zn(S,O) Buffer Layers Prepared by an Alternative Chemical Bath Process

Prog. Photovolt: Res. Appl. 2006; 14:499–511

15:15 Oral

Hydrothermal epitaxy of ferromagnetic TiO₂ films

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TiO₂ films are important as self-cleaning and anti-fog optical coatings and as switches and interferometers in optical communication devices

because of its large birefringence and favorable photocatalytic properties. More recently, epitaxial TiO₂ films doped with ferromagnetic ions have received considerable attention due to their potential as dilute magnetic semiconductors in the field of spin electronics.

In this report, transition metal doped epitaxial anatase films are grown on (001) oriented single crystal SrTiO₃ substrates by liquid phase deposition at temperatures as low as 50°C. Besides being a simple and cost effective method of film deposition, the precursor solution can be tailored to provide an oxidizing environment, thus precluding the formation of metal clusters. This is important as it has been argued that the presence of metal clusters in transition metal doped TiO₂ films formed by conventional physical vapor deposition techniques is actually responsible for the observed ferromagnetism and not carrier induced.

The epitaxial nature of the as-grown films was confirmed by pole figures while capillary stress induced cracking indicates that the film is nanoporous. XPS and SIMS show the presence of the dopant ions throughout the films and absorption measurements suggest that the dopants have substituted Ti⁴⁺ from its octahedral environment. The films are ferromagnetic, as determined by AGM and SQUID, and the effect of heat treatments on the chemistry of the film and its ferromagnetism is reported.

15:15 Oral

Characterization of nanostructured manganese dioxide thin films prepared by a novel self-assembly process.

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Nanostructured manganese dioxide thin films were deposited on metalized plastic supporting substrates by a horizontal submersion self-assembly process at ambient temperature and pressure. Films with the desired thicknesses were prepared by repeating the submersion process after each prior deposited layer had been air-dried completely. The self-assembly process entailed the initial adsorption of manganese dioxide nanoparticles onto surface active sites, and subsequent particle growth, clusters formation, and aggregation or self-organization of clusters which eventually led to the deposition of nanostructured thin film on the supporting substrate. As such, thin films with tailored microstructure could be prepared by optimizing deposition conditions such as pH, duration of submersion, temperature, suspension concentration, and ionic strength of suspension, as well as the post-deposition calcination temperature. Scanning Electron Microscope (SEM) micrographs showed that self-assembled manganese dioxide thin films were nanoparticulate and highly porous in nature. These films exhibited excellent capacitive behavior as evidenced by the almost perfectly rectangular shape of cyclic voltammograms within the potential range of 0.0 to 1.0 V (versus SCE) in mild aqueous Na₂SO₄ electrolyte. Besides, these films showed high cycling stability and reversibility upon prolonged cycling of exceeding 2,000 cycles. The high pseudocapacitance could be attributed to redox reactions involving the homogenous intercalation and deintercalation of protons during charge and discharge cycling. Self-assembled manganese dioxide thin films therefore possessed high potential utility for the fabrication of electrochemical devices, in particular thin-film electrochemical capacitors.

Key words: Manganese dioxide, nanoparticles, self-assembly, nanostructures, thin films, electrochemical capacitors.

Coffee break

Wednesday afternoon, 19 September, 15:30

Joint Poster Session (Part II)

Poster Award Ceremony

Wednesday afternoon, 19 September, 15:50

Thursday, 20 September

Nanostructures I

Thursday morning, 20 September, 9:00

9:00

Invited

Medical and nutritional applications of nanoscale silicon : from the concept to clinical use

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Very high surface area silicon powders can be realized by high energy milling or electrochemical etching techniques. The biodegradability of nanostructured silicon (1) is receiving increasing attention for medical applications such as drug delivery (2). The use of semiconducting silicon in brachytherapy (highly targeted radiotherapy) is now under clinical evaluation for treating primary liver cancer (3) and more recently, pancreatic cancer. This talk will give an overview of progress in medical therapy and conclude by describing recent experiments relating to food and drink technology (4).

- (1) L.T.Canham. Adv.Mater.7,1033 (1995)
- (2) C.Prestidge et al. Expert Opinion in Drug Delivery 4, 101 (2007)
- (3) A.Goh et al. Int.J.Radiat.Oncol.Biol.Phys. 67, 786 (2007)
- (4) L.T.Canham. Nanotechnology 18, 185704 (2007)

9:30

Invited

Highly Ordered Anodic Porous Alumina for Functional Nanodevices

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The use of materials with highly ordered structures is effective for the fabrication of various types of functional nanodevices. Anodic porous alumina prepared by the anodization of Al in acidic solutions is a typical material with a self-organizing structure of nanometer dimensions. This material is one of promising candidates for the starting structure of nanofabrication due to its unique fine geometrical structures. In the report, processes for highly ordered nanohole arrays in anodic porous alumina and several recent examples of application to functional nanodevices will be presented.

10:00

Invited

Nanofilm Formation Using Electrochemical Atomic Layer Deposition (ALD)

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ALD describes methodologies for the growth of materials an atomic layer at a time, using surface limited reactions. Surface limited reactions such as underpotential deposition (UPD) are well known in electrochemistry. The work to be presented involves the use of electrochemical surface limited reactions to deposit metals and semiconductors using ALD. Electrochemical ALD promotes layer by layer growth and atomic layer control of thickness. The work being pursued in this group concerns development of electrochemical ALD, and studies of the interface chemistry needed to form nanofilms of materials electrochemically. To date, deposition cycles for Cu, Ag, Pd, Au, Ru and Pt, have been developed, as well as numerous cycles for compound semiconductors. The advantages of an electrochemical deposition methodology include: conformal deposition, room temperature deposition, less costly tooling, through mask plating, selective plating (only on areas in electrical contact), and lower particle formation. Using surface limited reactions and ALD, smooth morphologies along with deposit thicknesses control at the atom layer level, are had. Results presented here will include some discussion of compound semiconductor formation, and the deposition of elements: Pt, Cu, and Ru. These deposits are being formed using surface limited redox replacement (SLRR), a methodology traceable to the work of Brankovic, Wang and Adzic. SLRR involves use of a sacrificial element, one that will form a UPD layer on the surface and that is less noble than the desired element. Exposure of the an atomic layer deposit of the sacrificial element to a solution containing ions of a more noble element results in redox replacement by the more noble element. As the amount of deposition is limited by the amount of the sacrificial element deposited at an underpotential. This process can frequently be repeated to form elemental nanofilms layer by layer using and electrochemical ALD.

Coffee break

Thursday morning, 20 September, 10:30

Nanostructures II

Thursday morning, 20 September, 11:00

11:00

Oral

TiO₂ nanotubes: Formation, Properties, Applications

Patrik Schmuki¹, Andrei Ghicov¹, Jan M. Macak¹, Hiroaki Tsuchiya¹, Sebastian Bauer¹, Sergiu P. Albu¹, Robert Hahn¹, Jung Park², Klaus Von der Mark²

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We report approaches to grow self-organized high aspect ratio titanium oxide nanotubes. Key to obtain highly defined tubes by simple anodization is an optimized and controlled treatment of Ti in fluoride containing solutions. In general, the morphology of porous TiO₂ and of the tubular layers is affected strongly by the electrochemical parameters such as solution pH and anodization voltage. By optimizing the local

electrochemical conditions within the tubes layers consisting of highly ordered TiO₂ nanotubes with a length of several 100 micrometers can be grown on Ti surfaces. The diameters that can be obtained range from 20 nm to 200nm - typical wall thicknesses are in the range of 10-20 nm (1-3). Titanium dioxide is a highly functional material that has numerous interesting properties, for example, in solar energy conversion, catalysis of the decomposition of organic compounds (self-cleaning), wettability and biocompatibility. Therefore this simple approach to produce this highly defined nanoscopic form of TiO₂ bears a high potential for technological exploitation. Due to the wide range of applications the feasibility to produce self-organized TiO₂ nanotubular nanostructures have received huge scientific and also significant technological interest. We have recently shown that these nanotubular structures show significant conversion efficiencies, when dye-sensitized (4), can be used as a matrix for hydroxyapatite formation to achieve osseointegration with bones (5), show extremely enhanced visible photoresponse after N-doping (6), can be used as a highly efficient photocatalyst (7) as a catalyst carrier for Pt/Ru nanoparticles for methanol electrooxidation (8) or as a template for Cu deposition (9). Further, they show ability to change their hydrophobic/hydrophilic behaviour (10). The presentation will address particularly the fabrication of free standing nanotube membrane (11) and size sensitive interactions of biological cells with the nanotubes surface (12).

11:15

Oral

Formation of oxide nanotube layers on titanium alloys

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We have examined the electrochemical formation of self-organized titanium oxide nanotubes. Key to achieve highly ordered tubes with high aspect ratio is an optimized electrochemical anodization in fluoride containing electrolytes. The morphology of resulting nanotube layers is strongly affected by anodization parameters such as electrolyte pH, viscosity and anodization voltage. Layers consisting of highly ordered titanium oxide nanotubes with a length of over 100 micrometers can be formed by optimizing the local electrochemistry within the tube. The diameters can be also tuned from 10 nm to 200 nm by controlling the anodization conditions. Due to the wide range of applications of titanium oxides, such as solar energy conversion, photocatalysts, wettability and biocompatibility, the nanotubular structures have attracted much scientific interests as well as significant technological interests. We have reported that titanium oxide nanotube layers show higher conversion efficiency than flat titanium oxide layer, can be used as coating for enhanced hydroxyapatite formation to achieve osseointegration to living bone and show extremely enhanced visible photoreponse when nitrogen-doped.

Recently novel nanotubular structure with two discrete sizes and geometries has been reported on titanium alloys – self-organization on two size scales can be achieved by simple anodization. The two-size-scale self-ordering is achieved on single b-type Ti₂₉Nb₁₃Ta_{4.6}Zr alloy where nanotubes with a diameter of approximately 50 nm are satellited by 30 nm nanotubes. The present work will report the formation process of layers consisting of two discrete tubes on the alloy and key factors to achieve the two-size-scale self-ordering will be briefly addressed.

11:30

Oral

Anodic titanium oxide nanotube coating for biomedical applications

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Titanium and its alloys are widely used in biomedical applications, for example, for artificial hip, orthopaedic, or dental implants due to their good mechanical properties and biocompatibility. The superior biocompatibility of these materials can be attributed to the high corrosion resistance as well as to the good binding ability to living bone. These materials form a biological apatite layer on their surfaces in body and bind to living bone via the apatite layer. Therefore, the apatite formation on these materials can be an essential step for the binding to bone. To enhance the biocompatibility of titanium alloys, various surface treatments have been proposed.

Recently we have reported the electrochemical formation of nanotube oxide layers on a whole range of valve metals (Al, Zr, Hf, W, Nb, Ta and Ti) in fluoride-containing electrolytes. In particular, the formation of titanium oxide nanotube layers has been extensively examined due to a variety of practical applications of titanium oxides. With specific electrochemical parameters, highly ordered TiO₂ nanotube formation is achieved. Layers formed in HF-containing acidic electrolytes essentially consist of TiO₂ nanotubes with a diameter of approximately 100 nm and a length of 500 nm. The geometry can be modified by tailoring the electrochemical parameters, for example, anodization in neutral electrolytes containing a small amount of fluoride ions results in thicker TiO₂ nanotube layers (several micrometers).

In the present work, we report the formation of oxide nanotube layers on titanium alloys and the application of anodic titanium oxide nanotube layers as a biological surface treatment.

11:45

Oral

Stress-Driven Transport in Ordered Porous Anodic Films

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Porous anodic alumina (PAA) films are grown by anodizing in acidic baths. Procedures to grow films with highly ordered geometry have led to the extensive use of PAA films as templates for fabrication of nanostructured devices. The morphology of PAA consists of evenly spaced and mutually parallel pores, oriented perpendicular to the metal/film interface. The ordered PAA morphology has never been fully explained.

A significant body of experimental evidence supports the occurrence of creep of the oxide during anodic film growth. In the present work, we have adapted a continuum theory in which viscous creep is coupled to ion migration through the stress field. The driving force for high-field migration is the gradient of the chemical potential of aluminum and oxygen ions, which includes contributions from stress as well as electric potential. These ions are transported independently, by their generalized migration flux and by convective flow. The model enforces

conservation equations for both electrical charge and volume, and the Cauchy momentum equations of fluid flow.

One-dimensional model calculations in a concave spherical shell, simulating the scalloped geometry of the oxide film at the pore base, demonstrate the origin of the experimentally observed flow during growth of PAA. Because of the nonlinearity of high-field migration, metal ions dominate the migration flux where the current density is high (near the solution) while oxygen ion transport is most significant at lower current densities (near the metal). Since metal ions are small compared to oxygen ions, the resulting volume flow due to migration is nonuniform. A stress distribution is produced which generates inward convective flow of oxide near the solution. The flow pattern is very similar to that inferred from recent experimental tracer studies. Further results of two-dimensional calculations will be presented, relating creep directly to the mechanism of ordering during PAA growth.

12:00

Oral

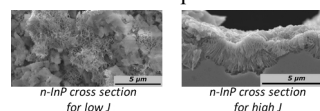
Morphologies and photoluminescence properties of porous n-InP in acidic liquid ammonia

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Electrochemical pore formation on n-InP has been firstly been evidenced in HCl by P. Kohl in 1983 [1]. Since then, numerous studies have been carried out and have revealed a strong influence of the electrolyte in the porosification process [2]. Liquid ammonia, due to its low viscosity, low dielectric constant and its strong basic feature, appears to be relevant to investigate the porosification of semiconductors under true water-free conditions [3]. In this work, anodic pore formation of n-InP was performed in acidic liquid ammonia (NH₃+NH₄Br) by galvanostatic treatments (*U-t*). Morphologies were characterized by scanning electron microscopy (SEM) and the evolution of optical properties was analysed by *ex-situ* photoluminescence (PL) with four different wavelengths (442 nm; 488 nm; 514 nm and 633 nm). According to the current density (*J*) range, two different morphologies have been observed. For low *J* (<1 mA/cm²) the morphology is comparable to a "sponge", whereas for high *J* (>1 mA/cm²) to a "coral". The "sponge" morphology is anisotropic; there are only few aperiodic irregularities on the *U-t*. The pores of the "coral" structure grow tortuously and perpendicularly to the surface in hemispherical depressions. For high *J*, the periodic lift off of the porous layer is correlated to voltage oscillations and can form multilayered structures [3]. PL experiments combined with SEM characterisation demonstrate that the porosified surfaces exhibit a dead layer behaviour [4]. From these results, the porosification process in this solvent will be explained and compared with those observed in aqueous media.



[1] P.A. Kohl, *et al.*, J. Electrochem. Soc., **130**, 2288 (1983)

[2] P. Schmuki, *et al.*, Phys. Status Solidi A, **182**, 51 (2000); C. O'Dwyer, *et al.*, J. Electrochem. Soc., **153**, G1039 (2006)

[3] A.M. Gonçalves, *et al.*, Electrochem. Solid State Lett., **10**, D35 (2007)

[4] D.B. Wittry, *et al.*, J. Appl. Phys., **38**, 375 (1967)

12:15

Oral

Electrochemical formation of InP porous nanostructures and its application to amperometric chemical sensors

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High-density formation of semiconductor nanostructures has been intensely researched for applications such as photonic crystals, quantum and optoelectronic devices, and chemical and biochemical sensors. In this study, we report a high-density array of size-controlled InP porous nanostructures formed by a two-step electrochemical process and their feasibility for the application to chemical sensors. Our process consists of the pore formation on a (001) n-type InP substrate and the subsequent etching of pore walls caused by changing the polarity of the InP electrode in a HCl-based electrolyte. By applying the anodic bias to the InP electrode, the high-density array of uniform pores was formed on the surface. The pores orderly aligned in a triangular lattice with a period of 130 nm and a wall thickness of 30 nm. In the depth direction, the pores were straightly formed over 80 nm with keeping good uniformity. These features are very similar to the Al₂O₃ porous structures. Next, the cathodic bias was applied to the porous sample to reduce the wall thickness by cathodic decomposition of InP. From the analysis using a scanning electron microscope (SEM), we found that the thickness of InP nanowall was precisely controlled in a range from 30 to 15 nm by changing cathodic bias and time. This resulted in the formation of unique quantum nanostructures with a high aspect ratio that are not obtainable by other methods. From the electrochemical measurements on the InP porous nanostructure, we found that the efficiency of some electrochemical reactions increased at the porous surface having the extremely large surface area. For example, the current-voltage characteristics of the porous sample showed larger response to the addition of a H₂O₂, as compared with the planar InP. This result indicates that InP porous nanostructures formed by the present process is very promising for application to amperometric chemical sensors.

Lunch break

Thursday afternoon, 20 September, 12:30

Nanostructures III

Thursday afternoon, 20 September, 14:00

14:00

Oral

Porous silicon for field emission display applications

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We have obtained cathodoluminescence (CL) emission from porous silicon (PSi) films by excitation with electrons with kinetic energies below 2 KeV. We observed two types of CL emissions, a stable one in well oxidized samples and a non-stable one in hydrogen-passivated samples. The stable emission is characterized by a CL peak that is red shifted with respect to its photoluminescence (PL) peak. The other CL emission is the most promising in terms of its application to field emission display technology. We were able to tune this CL emission

by controlling the average size of the nanoparticles. This shows that the mechanism for this type of CL emission can be associated to the quantum confinement and surface chemistry effects that are known to exist in the porous silicon system. To further prove this point we were able to correlate this CL emission with its PL counterpart. We also found that the electron bombardment modifies the PSi films at the nanoscale level which is manifested by an increase in the density of nanoparticles and appears to be the reason behind the PL enhancement after electron bombardment. In addition, we present our preliminary results for the beginning of a prototype pixel for a field emission display using carbon nanotubes as the electron emitter component and a "PSi paste" as the display component.

14:15

Oral

Development and characterization of electrochemically enlarged silicon electrodes for energy storage microcapacitors

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The objective of this work is to develop a silicon integrated microcapacitor with an enlarged electrode area, suitable for high energy storage in powering advanced microsystems. That enlargement has been obtained by anodizing the semiconductor, which results in the formation of macroporous structures with an aspect ratio between 10 and 100, in both P and N type silicon.

An electrochemically etched <100> P type substrate ensures a vertical growth of silicon macropores, due to its crystalline orientation. The pore density increases with the anodization current density, which also has an increasing effect over the growth rate, in a certain electrolyte. The relationship between the mentioned parameters has been analyzed by the use of image processing software and Scanning Electron Microscopy (SEM).

Starting from a N type silicon wafer, back side illumination is required to ensure the desirable vertical growth of the macropores, according to the <100> crystalline orientation and towards the source of electric holes. An Indium Tin Oxide (ITO) thin film has been deposited on the back side, as an ohmic contact, allowing the wafer being illuminated. A high transmittance of the deposited film and a resistivity as low as 3×10^{-4} ohm.cm have been obtained. The properties of the ITO layer grown under different process parameters have been characterized by a spectrometer, X-ray Diffraction (XRD) and a Sheet Resistance Monitoring System.

The local formation of porous silicon has been delimited by regions highly doped with phosphorous, which endure against the electrochemical etching. A dry thermal oxidation of the substrate constitutes the dielectric layer of the capacitor. The top electrode has been built by a homogenous polysilicon layer grown by the Low Pressure Chemical Vapor Deposition (LPCVD) technique, which has been characterized by Field Emission Gun SEM and RXD. A doping process with phosphorous at 1000 °C reduces the electrode sheet resistance up to 3 ohm/sq.

14:30

Oral

Fabrication and properties of multisegment metal-semiconductor-metal nanowires

Ionut Marius Enculescu¹, Elena Matei¹, Marian Sima¹, Monica Enculescu¹, Cornel Ghica¹, Lucian Ion², Stefan Antohe², Reinhard Neumann³

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Over the last decade the research focusing on the fabrication of new devices based on semiconductor nanowires increased strongly. New methods for preparing semiconductor nanowires were extensively developed during this period. It is an interesting fact that the weight center of the research shifted in the last few years from methods of preparation of nanowires towards methods of fabrication of nanowire based devices.

Electrochemical replication of nanoporous membranes, the so called template method, represents a flexible approach towards the fabrication of semiconductor based nanowires. In this paper we will present our results in using this method in fabricating several types of metal – semiconductor – metal nanowires, with the semiconductor segment from the II – VI group. Besides the morphological and structural characterization, transport properties of such complex nanowires will be presented.

14:45

Oral

Electrodeposition of ZnTe nanowires into porous anodic alumina membranes

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Template synthesis, a simple and versatile method for preparing nanostructured materials, entails synthesizing the desired material within the pores of a template membrane. Among various templates, the anodic alumina membrane (AAM) is a promising template for its uniform, nearly parallel, and ordered pore structure. Moreover, electrodeposition of nanowires inside nanoporous alumina templates has emerged as an easy, fast, and economic approach to form this type of nanostructured materials.

Zinc telluride is an important semiconductor material with a direct optical band gap of 2.2-2.3 eV at room temperature, which has potential applications in optoelectronic and thermoelectric devices. A detailed electrochemical study aimed to obtain thin films of this compound from a plating solution containing cadmium sulfate, tellurium dioxide and citric acid was conducted prior to the fabrication of ZnTe nanowires by electrodeposition into AAM pores. From this study it was possible to establish the optimal experimental conditions for the preparation of

as grown ZnTe films with an almost stoichiometric atomic composition (50.40% Zn, 49.60 % Te). The ZnTe nanowires on the AAM membranes with 50 nm of pore diameter were then prepared through potentiostatic electrodeposition under similar conditions. The electrodeposited ZnTe nanowires were characterized by SEM and XRD analysis. The latter showed that they presented a preferential crystalline growth along the 200 direction, different to that reported in the scarce literature available on this subject.

Acknowledgements. This work was supported by FONDECYT-Chile (Grant N° 1040650). Special acknowledgements to the ECOS-CONICYT program (projet N°C05E07) between France and Chile governments.

15:00

Oral

Multicolour Photochromic Behaviour of Silver Nanoparticles in Titanium Dioxide Matrix

Jana Preclikova, Frantisek Trojanek, Petr Nemec, Petr Maly

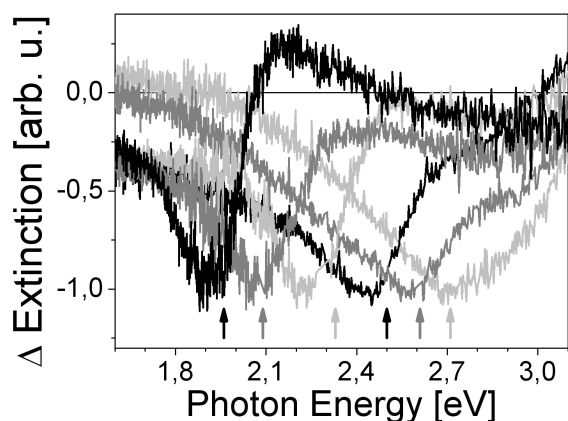
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Recent remarkable progress in nanomaterial research led to the development of new materials with unusual features. We report on the properties of a composite material made of silver nanoparticles embedded in nanoporous TiO₂ film in which unique multicolour photochromic behaviour was observed [1, 2]. The colour of the material can be changed by light irradiation. The original hue gray film surface appears to change its colour in accord with that of the illuminating light. We prepared our samples by UV photocatalytic reduction of Ag⁺ ions to Ag nanoparticles in nanoporous TiO₂ films. Different shapes and sizes of nanoparticles result in a wide inhomogeneous broadening of their extinction peak over the whole visible spectral region. Spectrally narrow light interacts selectively with some of the nanoparticles leading to a reduction of the film extinction in the spectral region of illuminating light ("hole-burning"), see Fig. 1. We report on the optimization of preparation of photochromic Ag- TiO₂ films and on their properties as studied by optical spectroscopy. We discuss also the microscopic model of the observed behaviour. The authors acknowledge the financial support of the Grant Agency of Charles University in Prague (grant No. 130607) and of the Ministry of Education of the Czech Republic under the research plan MSM 0021620834.

[1] Ohko Y., Tatsuma T., Fujii T., Naoi K., Niwa C., Kubota Y., Fujishima A., Nat. Mater. **2** 29 (2003)

[2] Okumu J., Dahmen C., Sprafke A. N., Luysberg M., von Plessen G., Wuttig M., J. Appl. Phys. **97**, 094305 (2005)



1 – Changes in the extinction spectra (normalised) of nanocrystalline Ag-TiO₂ films after irradiation by six different laser lines indicated by arrows.

15:15 Oral

Dynamics of PBLG Polypeptide Nanorods Embedded into Nanoporous Alumina Templates

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In this work, N-carboxy anhydride (NCA) monomer molecules were polymerized on the pore walls of an initiator coated nanoporous alumina template, leading to the formation of polypeptide poly(γ -benzyl-L-glutamate), known as PBLG nanorods. The structure and the associated dynamics have been investigated for a series of PBLG using DSC, XRD, FTIR, and dielectric spectroscopy (DS), respectively. High molecular weight bulk PBLG exhibits a glass transition temperature (T_g) at about 286 K and a broad endothermic peak at around 333-353 K during the first heating run. The dynamic investigation by DS revealed a glass temperature at about 281 ± 2 K for the PBLG nanorods, i.e., slightly lower than the bulk T_g . Studies as a function of pore size are in progress. In addition, DS revealed a local process below T_g , associated with the localized fluctuations of the side-group and a slower process associated with the relaxation of the secondary structure. The intensity of this mode suggested "broken" helices. An "ultra slow" process was also detected that arises from the inhomogeneous medium (Maxwell-Wagner-Sillars polarization). The structural characterization of the pure PBLG showed a strong peak of the lyotropic liquid crystalline phase at $2\theta = 6.7^\circ$. This reflection corresponds to an intermolecular spacing of α -helical chains with a spacing of 13.2 Å. XRD patterns of PBLG nanorods had only one- albeit very broad- reflection at $2\theta = 6.5^\circ$. The broader reflection and the absence of higher order reflections are in agreement with the DS study revealing broken helices.

Coffee break

Thursday afternoon, 20 September, 15:30

16:05

Oral

Zinc oxide nanostructured layers by spray of zinc acetate solutions

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The spray pyrolysis process is an attractive low cost technique for the deposition of metal oxide films. Zinc oxide thin films were deposited from zinc acetate aqueous and alcohol solutions at growth temperature ranging from 350 °C to 540 °C on glass and ITO substrates using continuous and pulsed spray modes. The deposited layers were characterised by optical spectra in the wavelength range of 300-800 nm, structural properties by XRD, surface morphology by SEM. Photoluminescence (PL) and wettability measurements were conducted as well. The use of continuous spray gave nanostructured ZnO layers which surface comprises conical needle-shape crystals with diameter of 30-70 nm and flower-like crystals. The diameter of the needles increased with growth of deposition temperature until they lose their shape at ~500 °C. Deposition of ZnO layers by pulsed spray or doping by indium results in dense and smooth films with no nanoneedles. The films with structured surface are more hydrophilic (CA=17°) than the flat ones obtained by pulsed spray or by doping (CA=55 and 32°). The reflectance and the transmittance of the films depend on the morphology. The band gap of the undoped films did not depend on the morphology and was $E_g = 3.28$ eV, with indium doped films it was $E_g = 3.33$ eV. Undoped films show the intensive near band edge (NBE) PL emission at room temperature indicating high purity of the film. PL emission did not depend on the film morphology.

16:20

Oral

Synthesis of porphyrin nanotubes by ionic self-assembly and properties characterization.

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In this work the porphyrin nanotubes were built by ionic self-assembly in aqueous solutions of two oppositely charged porphyrins- anionic Meso-tetra(4-sulfonatophenyl)porphine dihydrochloride (TPPS4) and cationic Meso-tetra(4-pyridyl)porphine (T4MPyP). The electrostatic forces between these porphyrin blocks contribute to the formation of porphyrin aggregates in the form of nanotubes, enhance the structural stability of these nanostructures. The porphyrin nanotubes obtained are hollow structures with the length of 300 nm and diameter 50 nm. Photocatalytic porphyrins are used to reduce metal complexes from aqueous solution and to control the deposition of Au from AuHCl₄ onto porphyrin nanotubes. Porphyrin nanotubes are shown to reduce metal complexes and deposit gold selectively onto the outer surface

of the tubes. The colloidal solution of gold nanoparticles lead to gold strip inside nanotube composite structures. Solution and monolayer spectra for the UV-vis experiments were obtained on an Ocean Optics diode array spectrophotometer to confirm the *J*-aggregates and nanotubes formation. The porphyrin nanotubes structure was investigated by Hitachi S-4800 electron microscope in STEM mode and by AFM imaging. The electrical properties of porphyrin nanotubes and nanotubes functionalized by gold were investigated by conductivity imaging using Scanning Probe Microscopy. It was demonstrated the possibility to build porphyrin nanotubes from the anionic Meso-tetra(4-sulfonatophenyl)porphine dihydrochloride and cationic HematoporphyrinIX dihydrochloride porphyrins and to deposit gold on the nanotubes from gold salt solution and inside the porphyrin tubes from gold nanoparticles (3-15 nm) colloidal suspensions. The results obtained in the work open a possibilities to build a new kind of nanodevices for photovoltaics, nanophotonics and sensing.

Discussion Session

Thursday afternoon, 20 September, 16:35

Friday, 21 September

Metals and ceramics

Friday morning, 21 September, 9:00

9:00 Oral

Microstructure and mechanical properties of nanocrystalline copper prepared by electrodeposition

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Nanocrystalline thin films have been widely used for many applications and their microstructure and mechanical property studies have been a focal point for the developments in the field of materials science and engineering. Recent literature shows that nanocrystalline copper can have good combination of mechanical and electric properties. This work as an attempt to systematically study and then understand the processing-microstructure-property relationship of nanocrystalline metallic thin films. In the present work, nanocrystalline copper thin films have been prepared using electrodeposition method. A high purity copper was set as anode and the cathode a steel. Both constant current and pulsed current at different levels have been used in order to evaluate the effect of current density on the microstructure and hence mechanical performance of the coatings. SEM and X-ray diffraction (XRD) technique have been used to characterise the deposited thin films. Preferential orientations of the films were studied using XRD. The orientation index, as evaluated using Harris method, shows a strong {220} texture with orientation index ~2, which differs from the preferred {111} texture reported on copper thin films deposited in a similar procedure in the literature. The grain sizes of the nanocrystalline copper have been evaluated using both the Scherrer method as well as the direct measurement using TEM. The relationship between processing (deposition parameters) and microstructure will be discussed and this serves to explain the mechanical properties measured using depth-sensing nanoindentation tests.

9:15

Oral

Copper deposition using wet chemistry. A new way : Organometallic chemistry for microelectronics

Clément Barrière^{1,2}, Pierre Fau¹, Olivier Margeat¹, Vincent Collière¹, Gilles Alcaraz¹, Christine Anceau², Jean-baptiste Quoirin², Bruno Chaudret¹

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Metallic Copper is currently one of the most used materials in microelectronics for the preparation of conducting lines. This interest comes from its good electric conductivity and its good resistance to electromigration¹. Several chemical and physical methods are used to create these deposits. Physical methods used are mostly PVD² techniques (Physical Vapor Deposition) as sputtering or vacuum evaporation. The most employed chemical ones are electrolysis and electroless^{3,4}, two methods using mild chemical conditions. However, both require a pre-metalized substrate to initiate the reaction. Objective of our work is to form homogeneous and adherent layers of copper on silicon wafer substrates with an oxide layer on surface. We have chosen two main ways. First one uses nanoparticles in solution coming from an organometallic synthesis. We will detail here synthetic roads leading to copper nanoparticles of controlled size and shape. These particles can be deposited on a substrate by spin-coating or ink-jet. The second way is to realize the deposit on the substrate in situ during the decomposition reaction of the organometallic complexes. Thin films are obtained after this first step, which can be followed by an electroless deposit to increase the thickness. All deposits are characterized by scanning electron microscopy, X-ray diffraction and profilers. An electric characterization is also done.

¹J. Li, T.E. Seidel and J.W. Mayer, Copper based metallization in ULSI structures. *MRS Bull.* **XIX** 8 (1994), p. 15.

²R. Jagannathan and M. Krishnan, Electroless plating of copper at a low pH level, *IBM J. RES. DEVELOP.*, 37, (1993), 117-123.

³S.H.Y. Lo, Y.-Y. Wang, C.-C. Wan, Synthesis of PVP stabilized Cu/Pd nanoparticles with citrate complexing agent and its application as an activator for electroless copper deposition, *J. Coll. Int. Sci.* **310** (2007), 190-195.

9:30

Oral

Electrodeposited Amorphous Bronze (CuSn) Alloy Coatings as replacement for Ni

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Electrodeposited amorphous alloy coatings present a promising alternative to single metal coatings. Alloy coatings with an amorphous or nanocrystalline structure combine properties, like high hardness and increased wear and corrosion properties, not obtained by coatings of the single alloy components. Apart from the unique properties increasingly strict environmental and safety legislation is an important driver to replace existing single metal plating technology.

Amorphous white-bronze, CuSn(Zn), electroplating was investigated as alternative to Ni plating as undercoat for noble metals in jewellery and PVD coatings on tools. A methanesulphonic acid based bath for

CuSn alloy deposition was developed. The relation between alloy composition and the bath composition and the process conditions was established and supported by basic electrochemical and chemical speciation data. The coating composition was optimized to the desired coating properties and the resulting amorphous/crystalline structure can roughly be explained from the Cu-Sn binary phase diagram.

The optimal CuSn composition was determined as 50 wt Cu / 50 wt% Sn giving bright white coatings with attractive appearance. The hardness of the amorphous bronze coatings, 500 HV, is higher than that of Ni, but also that of single Cu or Sn coatings. The corrosion and tarnishing resistance (< 200h NSS) of the white bronze coating outperforms Ni and even white bronze coatings from traditional cyanide based baths (Table 1). Upscaling at an industrial facility shows promising results for application.

Table 1: Comparison of the corrosion and tarnishing resistance of the acid CuSn to standard nickel and cyanide based CuSn. (5 µm coatings on brass as undercoat to 1 µm gold finish)

Test / Coatings	Nickel	Acid CuSn	Cyanide based CuSn
Neutral salt spray test	4	200	110
Thioacetamide(tarnishing)	> 200	> 200	4

9:45

Oral

Electrochemical Deposition of Ni-Based Thin Film Cermets Using Polymeric Additives

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Solar coatings with high absorptance in the visible part of the solar spectrum and low IR emittance represent the active part of the solar-thermal panels. This property combination is ideally reached by cermets. The coatings must supplementary show good stability in extreme conditions of temperature, humidity and corrosive media.

Thin layers of black nickel are good candidates for IR absorbers used as active solar-thermal coatings. The absorptance/emittance properties combination depends on the cermet composition, thin layer thickness and, especially, on morphology. Optimized morphology can be reached, in electrochemical deposition processes, based on the precursors' solution characteristics (nickel precursor salt and concentration, solvent and additives).

The deposition of Ni/NiOx cermets (on copper substrates) is reported in this study, using NiCl₂ aqueous solutions with different concentrations. Maleic anhydride/acid copolymers, with controlled water solubility, are used as additives in the electrolytic bath for morphology control. The addition of NaCl in the electrolytic bath is discussed both in terms of effects on the ionic strength and polymer dissociation and conformation.

The optimal electrochemical deposition parameters are obtained in voltammetry studies. The AFM images are used for morphology characterisation and UV-VIS-IR spectroscopy is used for the optical measurements.

The stability of the polymer – nickel ion structures represents a control possibility of the thin layer morphology, based on the different nucle-

ation and growth reaction rates. The structural properties of the ion metal-polymer species are correlated with the absorbance and emittance values of the cermets.

10:00

Oral

Synthesis and characterisation of cerium oxide by electrochemical methods

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Ceria-based materials have a lot of potential uses in many applications such as catalysts in gas sensors, electrolyte and catalytic interfacial layer in solid oxide fuel cells, etc. There are many ways to synthesize ceria compounds: physical techniques (Atomic Layer Deposition, Sputtering), chemical methods (chemical bath deposition, hydrothermal, sol-gels processes) and electrochemical process.

Electrodeposition is an interesting cheap method which can be done at ambient pressure and rather low temperature (less than 100°C). Moreover, it allows the control of the film thickness, *in situ*. Electrodeposition of ceria is based on the reduction of an oxygen precursor to form hydroxide ions nearby the electrode. Consecutively higher is the pH in the vicinity of the electrode. In the presence of Ce(III) ions in the solution, cerium oxide is thus directly formed on the electrode by precipitation with hydroxide ions.

Depending on the nature of the electrolyte (aqueous or organic), the temperature (from room temperature to 60°C), ranging the solution acidity level (4<pH<8), the imposed potential value (0.7 to 1 V/SCE) and the duration of deposition (20min<t<2h), different ceria thin films features such as thickness, colour, crystallinity, were obtained. This work focused on finding the best conditions (especially pH and oxygen sources) to synthesize adherent, thin and homogeneous films of ceria onto stainless steel for high temperature fuel cell applications.

The optimized deposition conditions lead to homogeneous and covering films. The microstructure and the crystallinity of the ceria thin layers were characterized by SEM and XRD measurements. Electrochemical microscopy (SECM) was used as a unique tool to study the morphology and homogeneity of the films as well as their electronic characteristics. Finally, electrochemical characterisations such as impedance spectroscopy were performed and more precisely impedance spectroscopy under air atmosphere.

10:15

Oral

An EQCM study of ultrasonically assisted electrodeposition of Co/CeO₂ and Ni/CeO₂ composites for fuel cell applications

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Ni is widely used as cathode material in molten carbonate fuel cells. Under the extreme conditions the cathode material undergoes dissolution. Addition of LiCoO₂ on the surface of the Ni cathode is known to reduce the precipitation of Ni in the bath [1]. It is also reported that cerium oxide layers reduce nickel oxide dissolution, and catalyse the Li incorporation into the nickel oxide cathode [2]. Therefore there is

a strong interest in the electrodeposition of Co/CeO₂ composites for protection of cathodes in MCFC fuel cells.

The application of ultrasound in electrochemistry has become increasingly popular in the past decade, especially in the field of materials science because of its potential in the production of nanomaterials, metals, and metal / oxide composites with superior quality, hardness, and homogeneity [3].

In this context the applicability of the electrochemical quartz crystal microbalance technique (EQCM) in a sonic field created by an ultrasound probe is first time demonstrated for the electrocodeposition of cobalt and nanoscaled cerium oxide particles. For comparison nickel was electrocodeposited with cerium oxide as well. EQCM is based on the measurement of the change in resonance frequency Δf of a thin single-crystalline quartz disc coated with keyhole-shaped gold electrodes on each side in parallel to the electrodeposition. One of the Au electrodes is in contact with the electrolyte and serves as the working electrode. From Δf , mass changes at its surface and therefore deposition rate can be measured with a very high sensitivity.

[1] T. Brenscheidt, F. Nitschke, O. Söllner, and H. Wendt, *Electrochim. Acta*, 46, 783 (2001)

[2] J. Hoffmann, C.-Y. Yuh and A. Godula Jopek, in Handbook of Fuels Cells, Fundamentals and applications, ed. W. Vielstich, H.A. Gasteiger, Arnold Lamm, Vol. 4, p. 921ff, 2003 John Wiley & Sons

[3] R. G. Cornpton, J. C. Eklund, F. Marken: *Electroanal.*, 9, 509 (1997).

Coffee break

Friday morning, 21 September, 10:30

Oxide nanostructures

Friday morning, 21 September, 11:00

11:00 Oral

Well-controlled, uniform dimension as well as vertical-alignment growth oxide nanostructures for potential optoelectronics and field emission applications

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For any future vast applications of oxide nanostructures in particular photovoltaic cell and field emission applications, well-controlled, uniform dimension as well as vertical-alignment growth is essential. The aims of this work are to demonstrate the ability to synthesize and characterise a range of single crystals oxide nanostructures of controllable aspect ratios as well as to measure their optical and field emission properties. The highlight of this synthesis procedure can be characterized by the success of controlled growth of single-crystal oxide nanostructures using mild conditions. The variations of the solute concentration, temperature, surfactant additives, solvent etc. afford effective and simple way to control the dimensions and orientation of the oxide nanostructures. The oxide nanostructures are characterized using a range of techniques such as X-ray diffraction, electron microscopy, X-ray spectroscopy and other characterisation tools. Structural analyses revealed these nanostructures to be highly crystalline.

11:15

Oral

Electrochemical Synthesis of Macroporous Zinc Oxide by Employing Hydrogen Peroxide like Oxygen Precursor

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Through Macroporous zinc oxide (MPZnO) synthesis in both two and three-dimensional colloidal crystal templates should be possible to get photonic behaviour or enhance sensing characteristics as well as classical properties of ZnO bulk.¹ Successful results have been obtained by atomic layer deposition, sol-gel, spray pyrolysis, infiltration and electrodeposition (ED).¹⁻⁴ However by ED only nitrate ions have been employed as oxygen precursor. Our goal was to consider new electrochemical routes and optimize the MPZnO ED by employing hydrogen peroxide (H₂O₂), an oxygen precursor never employed for this case and concentrated zinc ions electrolyte.

In current work we use polystyrene spheres templates (350 and 900 nm in diameter) prepared by Ghanem's method on Indium tin oxide (ITO).⁵ We could compare our results with those obtained by Sumida et al who have found the overpotential effect in the quality of macroporous films with nitrate ions.² In our case, we found concentrations effects being better those films obtained at H₂O₂ concentrations above 20 mM on ITO. Below that, the structure was disordered and doesn't follow the geometry of the template. These observations are supported by analysis of X-Ray Diffraction data, where the mean size of crystallites is close to 15 nm, even smaller than results obtained by Pauporte-Lincot.⁶ These results will be discussed in term of competition between grain growth habits and complete pore filling.

References

1. S.M. Abrarova, S.U. Yuldasheva, S.B. Leeb and T.W. Kang; *J. Lumin.* 109 (2004) 25.
2. T. Sumida, Y. Wada, T. Kitamura and S. Yanagida; *Chem. Lett.* (2001) 38.
3. M. Scharrer, X. Wu, A. Yamilov, H. Cao, and R. P. H. Changa; *Appl. Phys. Lett.* 86 (2005) 151113.
4. Y. Yang, B. Yang, Z. Fu, H. Yan, W. Zhen, W. Dong, L. Xia, W. Liu, Z. Jian and F. Li; *J. Phys.: Condens. Matter.* 16 (2004) 7277.

5. M. A. Ghanem, P. N. Bartlett, P. de Groot, A. Zhukov; *Electrochem. Comm.* 6 (2004) 447.
 6. T. Pauporté and D. Lincot; *J. Electrochem. Soc.*, 148 (2001) C310.

11:30

Oral

Mapping dopants effects in the High pressure synthesis of nanostructured zinc oxide

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Nanostructured zinc oxide is a material of increasing interest for both scientific and industrial fields due to its large spectrum of properties due to both quantum confining effects (such as giant magnetoresistance, luminescence and photoluminescence, UV – VIS light transmission) or very high surface to bulk ratio (chemical activity, photo catalysis, etc). Consequently important improvements may be achieved using nanomaterials and nanotechnologies in the production of materials for spintronics applications. The paper presents the state of the art in the synthesis of OD, 1D and 2D ZnO nanomaterials doped with different main and transition elements and a SWOT analysis for the procedures used for their production. Original results in the synthesis of Al, Co and Mn-doped ZnO nanomaterials are finally presented. The doped ZnO nanopowders were synthesised by a controlled nucleation process under high pressures in aqueous solutions. Electrochemical deposition was used for growing doped ZnO thin films on metallic substrates. Instrumental chemical analysis, XRD, BET surface area and RES have been used to explain the correlation between dopants nature and concentration and materials microstructure. The results are compared with the thermodynamic prediction using a special thermodynamic software.

Acknowledgement: Research supported by contract CEEX 69/2006 SINAPS and COST Action D 30.

11:45

Oral

Electrical and Gas Sensing Properties of Doped and Undoped ZnO Thin Films by Sol-Gel Process

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The electrical and gas sensing properties of both doped and undoped ZnO thin films are investigated in a dark chamber. The electrical measurements were carried out in ambient air, in vacuum and under nitrogen (N₂) flow at the room temperature and also in the temperature range of 290–425 K. The effect of volatile organic compounds such as alcohol and acetic acid on electrical conductivity of thin films was investigated. Clear, homogeneous and stable ZnO solution was prepared by sol-gel synthesis using zinc acetate (ZnAc) precursor. Methanol was selected as a solvent and acetyl acetone was used as chelating agents. In order to improve adhesion of ZnO on the interdigital transducer (IDT), triethanolamine was used in the solution. Yttrium 2, 4 pentanedionate was used as the dopant. ZnO and ZnO doped with 1-5 wt% Y thin films were coated on the IDT using Spin coater Model P6700 Series and preheating at 250 °C for 2 min after each coating.

After that all films were post-annealed at 600 °C for 30 min. The IDT consisted of 10 interdigital pairs of gold fingers, evaporated on microscope slides in a UNIVEX 450 coater system with a thickness of 150 nm, 100 µm wide and spaced 100 µm from the next finger. The dc and ac conductivity measurements were carried out on the thin films using Keithley 6517A Electrometer/High-Resistance Meter and Agilent 4294A Precision Impedance Analyzer, respectively. The effect of the VOCs vapors on the conductivity of the thin films was measured in a home made chamber and dry nitrogen was used as carrier gas at the room temperature. It was found that the conductivity of doped ZnO increased approximately 5 times than the case of undoped ZnO. This conductivity increase of the thin films depends on the concentration of VOCs which is between 500ppm-5000ppm. The details of the conduction and sensing mechanism of the samples will be discussed.

12:00

Oral

Surfactant-based nanostructured tungsten oxide thin films by sol-gel

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This work is focalized on the definition of a low-cost, low-impact, surfactant-based sol-gel route suitable to fabricate nanostructured thin films of tungsten oxide, in view of the development of electrochemical and sensing devices. Besides its low cost and ease of application to wide areas, the sol-gel process offers an effective possibility to direct the structure of the products. To gain major control over both the kinetics of crystallization and the architecture of the metal oxide networks, various templating agents (Brij[®]76, Triton X-100, Tween-60 and CTAB) were added to the sols. Their effect on the final structure has been evaluated by XRD, SEM, and micro-Raman spectroscopy.

The films were deposited by spin-coating on ITO-coated glass substrates to enable future electrochemical analyses, being ITO a transparent conducting oxide. The structure-tailoring effects of surfactants were then made evident, justifying the chosen approach.

At low temperatures (100-200 °C), the presence of a medium range ordered phase (mesophase) of hybrid organic/inorganic nature is found by XRD. During the annealing, the short and medium range ordering in the oxide network at intermediate temperatures (250-300 °C), as revealed by Raman analysis, takes place even in presence of residual surfactant and before complete crystallization (XRD). Around 300-350 °C the monoclinic γ-WO₃ phase crystallizes and evolves under thermal treatment at higher temperatures. The sintering process, however, keeps memory of the organic-template-induced mesophase.

Being tungsten oxide characterized by an isotropic perovskite-related structure, in absence of specific anisotropic growth requirements, spherical particles are formed, condensing into an interconnected fractal-like network. The formation of spherical particles takes place in the initial steps of the spin-coating/evaporation process, while the random arrangement of particles appears before the complete solvent removal.

Conclusion Session

Friday afternoon, 21 September, 12:15

Lunch break

Friday afternoon, 21 September, 12:30

Posters**Monday, 17 September****Joint Poster Session (Part I)**

Monday & Wednesday

Monday afternoon, 17 September, 15:50

15:50 Poster B1

Properties of In₂S₃ buffer layers deposited by CBD method: Quartz crystal microbalance studyBegoña Asenjo, Antonio M. Chaparro, Jose Herrero, M^aTeresa Gutiérrez*Centro de Investigaciones Energéticas, Medioambientales y Tecnológicas (CIEMAT), Avda. Complutense 22, Madrid 28040, Spain**e-mail: mbegona.asenjo@ciemat.es*

Indium sulphide (In₂S₃) is an important material for optoelectronic and photovoltaic applications [1, 2]. It can be used as substitute for cadmium sulphide (CdS) in Cu(InGa)(SSe)₂ (CIGS) based solar cells to avoid toxic cadmium and obtain more environmentally friendly photovoltaic technology. A recent paper report the technical progress that has occurred in the last years in the development of Cd-free buffer layers for CIGS thin-film solar cells, in different laboratories, deposited by different methods [3]. Among them, In₂S₃, with optimal physical properties can meet the requirements to be used as window material or buffer layer for these photovoltaic structures.

In this work the In₂S₃ films have been grown on soda lime glass with different composition, temperature, stirring and time of deposition. The kinetics growth of layers deposited by CBD is studied with a quartz crystal microbalance. Different physical, chemical and morphological properties of the films have been obtained. Analysis of surface and bulk composition is performed with XPS showing mixed composition.

The absorption coefficient spectra of the films prepared showed a variation depending on the CBD conditions. The absorption edges vary between 2.2 and 3.45 eV.

Surface morphology of the films, observed with scanning electron microscopy, reveals changes with the temperature and time of deposition.

References

- [1] T. Asikainen, M. Ritola and M. Leskela, Appl. Surf. Sci. 82, (1994)122
- [2] N. Naghavi, S. Spiering, M. Powalla, B. Cavana, D. Lincot, Prog. Photovolt. Res. Appl 11 (2003) 437.
- [3] D. Hariskos, S. Spiering and M. Powalla, Thin Solid Films, 480-481 (2005) 99

15:50 Poster B2

Synthesis and characterization of nitrogen doped TiO₂ aerogelsVirginia V. Danciu¹, Zsolt Pap¹, Lucian Baia², Florin Vasiliu³, Lucian Diamandescu³, Veronica Cosoveanu¹, Monica Baia²

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Titania based aerogels are a special class of porous solid materials with high surface area, ultra-low density and high homogeneity that make them attractive for various applications in solar energy conversion, pigments and electronic devices as well as in (photo)catalysis [1].

Nitrogen doped TiO₂ aerogels prepared by different chemical routes are studied by various physical investigation techniques (Brunauer–Emmet–Teller (BET), Transmission Electron Microscopy (TEM), Selected Area Electron Diffraction (SAED), X-ray diffraction (XRD) and Raman spectroscopy) in order to get valuable information concerning the morphology and the structure of the newly prepared porous materials. The catalytic activity of the nitrogen doped TiO₂ aerogels was determined by measuring the degradation rate of the salicylic acid used as reference organic material. The correlations between the morphological and structural parameters and those obtained from the photocatalytic activity assessment are discussed

References

- [1]. Z. Zhu, L. Y. Tsung, M. Tomkiewicz, J. Phys. Chem., **99**, 1995, 15945-15949.

15:50 Poster B3

Kinetics of induced electrodeposition of coatings containing Mo from citrate solutionsEwa Bełtowska-Lehman*Polish Academy of Sciences, Institute of Metallurgy and Materials Sciences (IMIM PAN), Reymonta 25, Kraków 30-059, Poland**e-mail: nmbeltow@imim-pan.krakow.pl*

Alloys containing molybdenum are characterised by a high hardness, wear, corrosion resistance and good catalytic properties for hydrogen evolution.. They also offer an important alternative to toxic, hard chromium coatings. It is known that molybdenum cannot be electrodeposited in the pure state from aqueous solution, but it can readily be co-deposited with iron-group metals such as nickel to form an alloy. The phenomenon has been called induced co-deposition. Binary Ni-Mo coatings can be electrodeposited at room temperature from aqueous electrolytes despite of the large difference in melting points of these metals and limited their mutual solubility. In the present work, the effect of the citrate electrolyte composition (pH 8) on the partial polarisation behaviours was investigated under potentiostatic conditions using a rotating disk electrode system (RDE). The kinetic correlations in co-discharge process of Mo(VI) and Ni(II) complex ions have been established. The relationship between electrodeposition parameters, structural properties and functionality has also been determined.

15:50 Poster B4

Electrodeposited ZnO / tetrasulfophthalocyanatonickel (TSPcNi) films: an inorganic / organic hybrid system with infinitely variable composition

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Electrodeposition is a low-cost and convenient method for the preparation of inorganic / organic hybrid materials for optoelectronic applications such as dye-sensitized solar cells in which high efficiencies were recently achieved with electrodeposited nanostructured ZnO / dye films. In view of various other optoelectronic applications, the variation of the dye content would be desirable in order to tune properties such as conductivity and photoconductivity of the hybrid material. However, all ZnO / dye hybrid systems investigated so far were based on a ZnO framework, which became instable at higher dye concentrations. We now found that electrodeposited ZnO / TSPcNi hybrid films represent a system which can be infinitely varied in its ZnO and dye contents. With increasing dye concentration in the deposition bath, a transition from films based on crystalline ZnO, which appear green due to the presence of TSPcNi momomers, over blue films with dye dimers or aggregates to deep blue films based on an amorphous dye framework is observed. Independent from the composition, all films are highly transparent. Structural changes were analysed by means of XRD, SEM and TEM coupled with SAED. The electrical and photoelectrical properties are dominated by the respective framework of each film. Consequently, films with a balanced composition show the lowest conductivity, while their photoconductivity is the highest, indicating that both phases take part in the photogeneration of charge carriers and/or transport of photogenerated charge carriers.

15:50 Poster B5

Synthesis of single-walled carbon nanotubes from aliphatic alcohols by CCVD method

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The catalytic chemical vapor deposition (CCVD) and carbon arc plasma sublimation of graphite are at present the most exploited ways of CNTs production on a large scale. On a proper choice of catalysts and process conditions depends whether the products are single- or multi-walled carbon nanotubes (SWCNTs and MWCNTs, respectively). In the case of the CCVD method also starting carbon bearing materials play crucial role for the final product. There are large variety of liquids, as well, gaseous hydrocarbons that can be used for efficient CNTs synthesis.

The objective of this work was the synthesis of the SWCNTs from ferrocene aliphatic alcohol solutions by the CCVD method. The influence of alkyl chain length on the SWCNTs synthesis was investigated. The alcohols were injected to the reactor, a quartz tube placed in a furnace, in the liquid or aerosol. The carrier gas was Ar or Ar/H₂ gas mixture. The feeding rate, digitally controlled, was varied between 0.1 and 3 ml s⁻¹. Also the temperature distribution along the furnace was the variable parameter.

The morphology of the obtained products were investigated by scanning and transmission electron microscopy (SEM and TEM, respectively). The quality of the CNTs were also analyzed by using the Raman spectroscopy.

This work was supported by the Ministry of Science and Education through the Dept. of Chemistry, Warsaw University, under Grant No. N204 096 31/2160. Author (M.B.) thanks the Foundation for Polish Science (FNP) for the financial support.

15:50 Poster B6

The Novel Diphenylamines - Basic Electroconducting MaterialsJoanna Cabaj¹, Krzysztof Idzik¹, Jadwiga Sołoducho¹, Mieczysław Łapkowski², Sylwia Golba²

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Conjugated polymers semiconductors with high charge carrier mobility air stability are of enormous interest for applications in organic electronics, particularly thin film transistors or photovoltaic cells. Although a large number of structurally diverse π -conjugated polymers have been investigated, only a small number have field effect mobility of holes or electrons.

Nearly all current p-type polymer semiconductors for OFETs with moderate to high hole mobility are poly(3-alkylthiophene)s and various thiophene-containing copolymers, that materials require, most notably good π -electron delocalization along the chain and a high degree of intramolecular order, good intramolecular π -stacking and important, low ionization potential.¹

Polymers constructed from diphenylamine building blocks are versatile materials that exhibit a variety of advanced functional properties such as high hole-transporting and light-emitting efficiencies large two-photon absorption cross sections, high photorefractivity and photoconductivity, and large stabilization effect on high-spin polyradicals in organic magnets.²

In this study, a novel series of multi-diphenylamine substituted derivatives bearing an bisarylene as a functional core were synthesized. The key step in this type of synthesis³ has been the Stille type coupling reaction between the *N*-alkyldibromodiphenylamine and tin derivatives of arylene as the best way for preparation of conjugated *N*-alkyldiphenylamine derivatives.

For this group of compounds were also investigated an electrochemical polymerization effect and widely adopted approach to prepare structurally ordered thin, electroconducting films by Langmuir – Blodgett technique.

1. Zhu, Y., Babel, A., Jenekhe, S. A., *Macromolecules*, **2005**, 38, 7983-7991
2. Velusamy, M., Justin Thomas, K. R., Lin, J. T., Wen, Y. S., *Tetrahedron Lett.*, **2005**, 46, 7647-7651
3. Sołoducho J, Idzik K., Cabaj J., Doskocz J., Łapkowski M., Plewa S., *ARKIVOC* (Gainesville, FL, United States) **2007**(vi) 75-89

15:50 Poster B7

Reversibly UV-light-induced hydrophobic/oleophilic to amphiphilic surface transition in thin films of organic-capped TiO₂ nanorods

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Nanocrystalline TiO₂ is among the most studied semiconductor oxides, owing to its low-cost widespread applications in environmental clean-up, in sensing, and in the photodynamic deactivation of bacteria and malignant cells. More recently, it has been discovered that UV-irradiation of TiO₂ surfaces rapidly converts them from an initially hydrophobic state to a highly amphiphilic one, followed by a slow recovery of the starting properties under dark storage. These findings have stimulated the fabrication and the study of inorganic coatings which exhibit simultaneous self-cleaning and antifogging behaviour. However, the understanding the mechanism leading to light-induced wettability modification on TiO₂ surfaces remains elusive yet and the ability to control them has to be developed further. In this contribution we will present a novel approach to engineer TiO₂ surfaces with UV-switchable wettability, which relies on the use of organic-capped TiO₂ nanorods (NRs) for the fabrication of thin films made of close-packed laterally aligned arrays of TiO₂ crystalline domains exposing well-defined light-active crystal facets. As opposed to conventional polycrystalline or single-crystal TiO₂ surfaces, such NR-based films exhibit a reversible surface transition from a highly hydrophobic/oleophilic state (water and octadecene contact angles of 110° and 8°, respectively) to a highly amphiphilic one (water and octadecene contact angles of 20° and 3°, respectively) under remarkably milder UV-irradiation conditions (energy density as low as 1-2 mJ/cm²). By using a combination of techniques (XRD, HRTEM, SEM, FTIR and Raman spectroscopy, and contact angle measurements), we will demonstrate that the observed light-driven wettability changes are accomplished by progressive hydroxylation of the TiO₂ surface irrespective of the presence of surfactant molecules, which simultaneously undergo conformational changes without suffering for significant photocatalytic degradation.

15:50 Poster B8

In situ synthesis and Biocompatibility of nano-potassium titanate-rutile /Ti alloy bio-composite

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A K₂(Ti₈O₁₇)/TiO₂ composite bio-ceramic coating on the surface of titanium was prepared successfully by an *in situ* electrochemical technique. During emersion in simulated body liquid the surface topography, microstructure, composition-of-phases, and the bioactivity of the coating were evaluated. The test results indicated that the K₂(Ti₈O₁₇)/TiO₂ bio-ceramic composite coating has fine biocompatibility and bioactivity.

15:50 Poster B9

Electrodeposited CuIn(S,Se)₂ based solar cells: electrical properties

Arouna Darga¹, Denis Mencaraglia¹, Anne migan Dubois¹, Zakaria Djebbour¹, Jean-Francois Guillemoles², James P. Connolly², Veronica Bermudez², Daniel Lincot³

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The effects of the Cadmium Sulphide (CdS) buffer layer thickness (d_b) on the electrodeposited CuIn(S,Se)₂ (e-CISSe)/CdS heterojunction properties are investigated. For this purpose, five samples of e-CISSe/CdS solar cells, with different buffer layer thicknesses ranging from 25nm to 63nm have been studied using current-voltage characteristics measurements as a function of the temperature ($I(V, T)$) and admittance spectroscopy ($C(w, T)$). For all devices, admittance spectroscopy reveals two relative shallow defect levels located at 0.1eV and 0.2eV, respectively. From the Meyer-Neldel behaviour observed on the pre-exponential factor of defects emission frequencies; we observed that the electronic properties of defects such as capture cross section are not dependent on the buffer layer thickness. In addition, analysis of the $I(V, T)$ characteristics reveals blocking current-voltage behaviour as an abnormal serial resistance at high forward bias voltage. All samples show this abnormal serial resistance which is characterized by activation energy of 0.2eV. In this contribution we discuss the $C(w, T)$ and $I(V, T)$ results in order to give an insight about electrical properties of e-CISSe based solar cells.

15:50 Poster B10

Modelling of the III-V thin films wettability

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The simulation have been performed using a density functional theory (DFT) in a periodic supercell approach, based on pseudopotentials and numerical localized atomic orbitals as basis sets, as implemented in the SIESTA code. Group III nitrides surfaces of different polarity were modeled using (2x2) surface unit cell.

Comparison to experiment is done based on direct calculation of surface energy as well as molecule-surface interaction modeling for different liquids. Calculated surface energies agree well with published data, however the absence of knowledge of the exact chemical potential values brings quite high uncertainty to the results. In contrast, analysis of molecule-surface interaction allowed separation of different components, namely disperse, polar and hydrogen bonding and their direct comparison with experimental results based on extended Fowkes method.

15:50 Poster B11

Microstructures Characterization of Protective Surface on Electrochemical Treated Nuclear Steels

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In the primary and secondary circuits of CANDU nuclear power plant, austenitic and martensitic steels are used as structural materials. In such environmental conditions these materials exposed to water results in corrosion. The improvement of the surfaces corrosion resistance is very important from nuclear safety point of view. Thermo electrochemical treatments of bath carburizing and nitriding represent a good choice for these improvements. Because the mechanical and anticorrosive properties are related to the microstructure of surfaces it is important to make good microstructures characterization of the protective surface layer.

The austenitic and martensitic steels samples thermo electrochemical treated were analyzed by X-ray diffraction (XRD) techniques. The acquisitions of diffraction patterns were performed in symmetrical Bragg-Brentano focusing geometry and asymmetrical geometry using a conventional powder diffractometer. The depth profile phases identification of modified surfaces is obtained in the asymmetrical arrangement. The line profile analyses were applied in order to determine the mean crystallites size and the micro-strains of the surface layer. XRD data acquired were analysed by integral methods for micro strains and crystallites size determination of external Fe₃O₄ layer. The Williamson-Hall plot and integral breadth method using first and second order of a particular reflexion were used.

By low angle XRD we found that Fe₃O₄ appear as external layer both on austenitic and martensitic steel. On to the martensitic steel the surface layer contains also CrC and CrN for carburizing respectively nitriding treatment. The results obtaining by low angle XRD were confirmed by SEM-EDX measurements. The surface layer microstructures do not differ too much for austenitic and martensitic steel after carburizing treatments. By nitriding treatments, the microstructures of martensitic steel layer is different of austenitic steel surface layer.

15:50 Poster B12

Mechanistic Study of ZnO Nanowires Electrodeposition

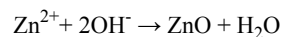
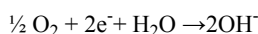
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Zinc oxide Nanowires (ZnO NWs) are a very promising oxide semiconductor nanostructure because of their good optical, electrical, and piezoelectric characteristics with possible use for field-emission displays, solar cells, laser¹ and electroluminescent devices.

The synthesis of ZnO NWs is possible by electrodeposition from aqueous solution, a simple and cheap method working at low temperature. Its principle is based on an increase of the interfacial pH by electrochemical formation of OH⁻ from the reduction of dissolved oxygen. This leads to the precipitation of the zinc oxide at the electrode following the reactions^{2,3}:



ZnO NWs are formed in presence of low concentration of zinc². Many papers are now published on the cathodic deposition of ZnO NWs by this method, but mechanistic aspects have not been explored in detail.

The aim of this study was to understand the mechanism of electrodeposition of ZnO NWs. The influence of different parameters such as pH, bath composition, time of deposition and potential of deposition on the growth was investigated by means of in situ techniques: chronoamperometry, pH-metry and Electrochemical impedance spectroscopy (EIS). The morphology and structure of the ZnO NWs were studied by field emission scanning electron microscope (FE-SEM). The optical properties of ZnO NWs were also studied by means of specular transmission and photoluminescence measurements. From the results we derive a general analysis of electrochemical reactions taking place during the formation with respect to the pH of the solution as a key parameter. We will show that ZnO NWs formation and properties correspond to very specific solution chemistry and substrate effect criteria.

References

1. T. Pauporté, D. Lincot, B. Viana and F. Pellé, Appl. Phys. Lett, 89, 233112 (2006)
2. S. Peulon, D. Lincot, Adv. Mater, 8, 166 (1996)
3. S. Peulon, D. Lincot, J. Electrochem. Soc, 145, 864 (1998)

15:50 Poster B13

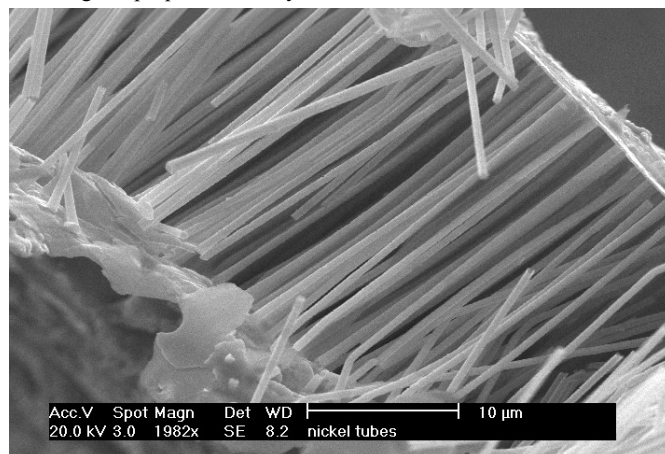
Nickel nanotubes prepared by electroless deposition in ion track templates

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Nickel nano- and micro- tubes were prepared by electroless deposition in ion track template membranes. By choosing the appropriate etching conditions, membranes with cylindrical or conical pores were obtained, allowing the preparation of cylindrical or conical tubules.



A two step activation process was employed for membrane activation. The bath used for deposition was an acidic one. Typically for acidic bath Ni deposition, by energy dispersive X ray analysis a phosphorous content of up to 10 % was found in the deposit.

15:50 Poster B14

Tailoring WO₃ Thin Layers Using Spray Pyrolysis Technique

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Thin layer properties can be tailored based on the optimization of the precursor system and the deposition technique and parameters. For large surface – area applications, appropriate reproducible deposition must be chosen.

Tungsten trioxide presents remarkable optoelectric properties making it an ideal candidate as photoanode in a photo-electrochemical cell (PECC), [1-3].

This paper provide a comparative approach on WO₃ thin layers obtained via spray pyrolysis deposition (SPD) and other state-of-art deposition techniques. The influence of the SPD parameters (precursor system and temperature) on the thin WO₃ films with different crystal-line properties, morphology and surface tension were considered.

References:

- [1] P.S. Patil, S.B. Nikam, L.D. Kadam, Materials Chemistry and Physics 69 (2001) 77;
- [2] D. Barreca, S. Bozza, G. Carta, G. Rossetto, E. Tondello, P. Zanella, Surface Science 532 – 535 (2003) 439;
- [3] A.A. Akl, H. Kamal, K. Abdel-Hady, Physica B 325 (2003) 65.

15:50 Poster B15

A dipcoating technique for isotropic surface structuration of metallic surfacesChristian Boyko¹, Raphaël Foschia², Stefan Hengsberger¹

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An important number of surface structuration methods allows for regular, periodic and uniform structures on metallic surfaces that consequently show a certain anisotropy. However, for purposes such as medical implants, random surface structures equivalent to the size of the cells (a few μm) promote their adsorption on the implant and therefore the integration of the implant in the human body. This abstract presents an isotropic and inexpensive structuration method for metallic surfaces of any geometry.

A polished sample of stainless steel is heated up to 200°C at ambient pressure. The piece then cools down under vacuum conditions (10^{-5} bar) to avoid the generation of a water vapour layer and is then coated with gold. The sample is then placed in a 50%:50% 11-mercaptoundecanol & 1-dodecantiol solution for 20 hours. The stoichiometry of these two solutions allows for regulating the surface energy. The sample is dip coated under controlled 33% humidity at 1mm/sec in a 50%:50% solution of PMMA and P2VP. These non-miscible polymers show a phase separation that depends on the surface energy of the substrate. Their phase separation leads to a random formation of P2VP isles with a mean size of several micrometers. Rinsing with ethanol removes the P2VP isles, and a treatment with potassium iodide dissolves the under-

lying gold layer and thiols. The remaining porous polymer structure serves as a mask for surface etching.

For this issue two alternative methods were employed, chemical etching with concentrated HCL and electrochemical etching in a buffered NaCl bath applying repeated short current pulses.

This combined dip-coating and etching method has shown to generate micrometer sized isotropic structures on stainless steel pieces of different geometries. In future work this technique will be adapted to industrial applications in the biomedical sector.

15:50 Poster B16

The influence of the solution composition includes additives on the structure and composition of electrochemical coatings Ni-B

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The structure and internal stress was investigated for d.c. – plated Ni composites deposits prepared from low - concentrated electrolyte Watts, without or with additive agents and dispersed particles: B. The weight percentage of the dispersed phase in the coatings was determined gravimetrically. Diffractometer Siemens D500 with Si[Li] radiation was used to examine the texture and grain – size determination. Internal stresses of deposited coatings Ni-B have been measured with dilatometric method using IS-metru of OMI International Company. The microhardness of the obtained coatings was measured by the Vickers method using a Hanneman microhardness tester at a load of 0.01 kG. The roughness of the obtained nickel layers was measured by a TR 100 profilograph (surface analyzer) from Elcometr Instruments Ltd.

Dispersion particles and additives used in the bath were shown to affect the composition and structure of the electrodeposited composite layer. An increase in the content of B particles in the coating and in the size of nickel [111] crystallites led to increased roughness of Ni-B coatings. The microhardness of the deposited Ni-B layers increased as the size of nickel [111] crystallites decreased. The internal stress of the deposited composite coatings depended to a large degree on the additive in the bath.

Ni-B coatings had a (100) texture, with texture (111) and (311) also frequently occurring. Orientation (100) developed even more strongly than in coatings deposited without additives. This shows that in the presence of 3-(1- pyridinio) – propanesulfonate inner salt (PPS), the development of orientation (110), (111) and (311) is blocked. When the additives were used together, the dimensions of the nickel [111] crystallites in all coatings decreased to the smallest size in any of the studied coatings.

The results suggest that the effect used of the organic compounds was the development of the nanostructure of the nickel matrix.

15:50 Poster B17

LPD deposition of TiO₂ films on mica substrate for hydrogen sensing

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Liquid phase deposition (LPD) also known as chemical bath deposition (LPD) was first reported by Nagayana et al. In this process metal oxide thin films are formed on the substrate by means of ligand-exchange hydrolysis of a metal-fluoro complex and the F^- consumption reaction by boric acid. Since aqueous solutions are used for deposition of oxide film in the LPD method, it is very sensitive to the properties of substrate in particular hydrophilicity. In this regard mica is much desired because of the presence of intrinsic OH groups on its surface. In addition mica is electrically insulator, thermally stable at high temperatures and has low weight. These properties identify mica as a suitable substrate for gas sensors instead of alumina which usually is used.

In the present work, we prepared anatase titania films on mica substrate from aqueous solutions of $(NH_4)_2TiF_6$ and H_3BO_3 , by LPD method in order to make H_2 gas sensor. The deposition temperature was set at $50^\circ C$ and defined the optimum preparation conditions for obtaining thin films with good morphology. A small difference in solution concentration, pH and deposition can lead to different solution super-saturation which obviously affects the growth rate and finally on film thickness and morphology. XRD, AFM, SEM and Uv-Vis spectroscopy were used to determine the degree of crystallinity, surface morphology, electronic characteristics and film thickness. XRD analysis showed TiO_2 nanocrystalline parcels in anatase phase with good crystallinity in spite of low temperature of deposition. The films thickness, electronic band gap and porosity of films were estimated by simulation of the optical transmission spectrum. These results together with AFM and SEM imply that concentration and pH of solutions has substantial influences in the morphological aspects of the films: grain size, porosity and thickness. Finally H_2 gas sensing properties of the films are reported.

15:50 Poster B19

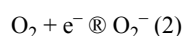
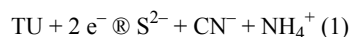
Electrodeposition of Polyphasic $[(ZnO)_a(ZnO_2)_b(ZnS)_c]$ Thin Films in DMSO Solution. Study of Their Morphological, Optic and Crystalline Properties.

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In this work the structural and optic properties of the polyphasic $[(ZnO)_A(ZnO_2)_B(ZnS)_C]$ thin films was studied. The synthesis of the films was performed by electrodeposition (ED) at controlled potential in dimethylsulfoxide (DMSO) solution. Compared with aqueous solution, the election of the organic solvent was done considering the wider potential window for electrochemical processes and the possibility to dissolve chalcogenide precursors. The cathodic potential applied to the working electrode allowed the electrochemical reduction of molecular oxygen (O_2) and thiourea ($(NH_2)_2CS$) as source of sulfide ions, at 373 K according to the following reactions:



Which were analyzed by cyclic voltammetry. The films were formed on two different substrates: tin oxide fluorine doped glass ($SnO_2:F$, FTO) and a mono-crystalline n-type InP substrate (1,0,0) and (1,1,1) with crystallographic plane in the phosphorous face. These deposits showed a particular morphology like honeycomb, which were observed by SEM. The morphology was independent of the electrodeposition

potential employed. This morphology possesses a marked polycrystalline character, which was confirmed by the RHEED patterns. In these patterns, numerous rings could be observed and was assigned to the three phases present in the film. However, the presence of supplementary reflections (dots) in the patterns, confirm that ZnS phase (ZnS/InP mismatch $\sim 7\%$) showed an epitaxial growth onto the mono-crystalline substrate.

The study of the transmission spectra of the deposits formed on FTO shows that the band gap energy was 3.46 eV. Due to these results, it is possible to postulate that these phases can be employed as substitution of CdS as layer window in photovoltaic devices with $Cu(In,Ga)Se_2$ (CIGS) as absorbent material.

15:50 Poster B20

Characterization of Preferred Orientated Vanadium Dioxide Film on Muscovite (001) Substrate

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Substrate plays an important role in the microstructure and switching performance of the thermochromic VO_2 films. In this paper, natural orientated muscovite (001) slices were used as the substrate for deposition of the vanadium dioxide films. Muscovite has layer aluminum silicate structure linked by electrostatic force of potassium ions, so it is easily cleaved to expose the (001) cleavage plane as surface. Muscovite is cheap, readily available in large sheets which may be cleaved to very thin and flexible plates and suitable for applications in optical and electrical devices.

The vanadium oxide films were fabricated on a muscovite (001) cleavage plane by aqueous sol-gel method, and then were crystallized and reduced to vanadium dioxide films by annealing in static N_2 atmosphere. The surface XPS analysis demonstrated the obtained films were stoichiometric vanadium dioxide. The AFM morphology picture indicated the vanadium dioxide film was composed of grains in nano scale (30nm~50nm). The X-Ray patterns of the films have the unique diffraction peak of monoclinic VO_2 (011) plane. It indicated that the films were (011) orientated on muscovite (001) plane, conforming a monoclinic VO_2 (011) $\square$ muscovite (001) structure at room temperature. When heating temperature increased to above the metal-to-insulator phase transition temperature (T_c) of VO_2 $\square$ structural phase transition (SPT) from monoclinic (011) to tetragonal (110) was detected by using X-Ray diffraction with an in-situ heating. The electrical and optical properties of the thermochromic VO_2 films on muscovite (001) were also investigated below and above T_c , it exhibited changes in electrical resistivity more than three orders of magnitude and optic transmittance switching more than 70 percent in the range of wavenumber 4000 cm^{-1} - 1000 cm^{-1} .

15:50 Poster B21

Epitaxial Electrodeposition of ZnO Thin Film on GaN(002) Bulk Single Crystal Substrate

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Electrodeposition of compound thin films can result in high quality materials as it was demonstrated by epitaxial electrodeposition of single crystalline thin films. For example, electrodeposition of ZnO onto Au(100) bulky single crystal and GaN(002) single crystal thin film grown by MOCVD on sapphire lead to the growth of ZnO(002) thin films owing to the small lattice mismatch. In this work, we have employed heavily Si-doped conductive bulky GaN(002) single crystal as an electrode to attempt electrodeposition of single crystalline ZnO from O₂ saturated aqueous solutions of ZnCl₂.

The (002) cut and polished GaN was purchased from Furukawa, Co., Ltd. and soaked in 0.1 M HCl for 2 min before use. When O₂ reduction current was measured in a KCl solution, cathodic current gradually increased below ca. -0.5 V vs. Ag/AgCl but never reached a plateau down to -1.5 V. Potentiostatic electrolysis at -1.3 V, however, indicated slow increase of the current that finally reached a constant value. Electrochemical analysis was carried out for such fully activated electrodes. The plateau current was confirmed as O₂ diffusion limited from Levich analysis. Koutecky-Levich analysis in the potential range between -1.15 and -1.3 V revealed the standard charge transfer rate constant (k^0) of 1.9×10^{-9} cm s⁻¹ and the transfer coefficient (α) of 0.089 for O₂ reduction, indicating that the GaN electrode we used is fairly inactive.

Electrodeposition of ZnO was performed by potentiostatic electrolysis at -1.0 V after addition of ZnCl₂. Formation of single crystal phase was confirmed by ω scan of XRD. In-plane alignment of the hexagonally shaped deposits was also observed by SEM. The SEM observation, however, found presence of randomly orientated domains, so that the present conditions are not well established for epitaxial electrodeposition. The influence of surface condition to the structure of the deposited layer is studied in detail and will be presented.

15:50 Poster B22

Tailoring Alumina Thin Films Properties Using Hydrophile/Hydrophobe Polymer Additives

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Micro- and nanostructured-cermet, with high absorbance in the visible region of the solar spectrum and low IR emittance, are widely used as heat absorbers in solar selective coatings. Thin films of alumina (Al₂O₃), with high refractive index ($n = 1.54 - 1.76$), good adhesion to different substrates, high thermal and chemical stability, are promising materials for ceramic matrix in cermet, IR coatings. The alumina based cermet properties strongly depend on Al₂O₃ layer properties, such as thickness and morphology. These properties can be tailored by varying the precursor solution composition (aluminium ions concentration, solvents and additives) and the deposition parameters (temperature, number of spraying sequences etc.).

In the present study, thin films of Al₂O₃ are deposited onto microglass and aluminium substrates by spray pyrolysis technique. As precursors, solutions with different AlCl₃ concentrations in water:ethanol (1:1) mixture are used. As morphology controlling agents, acetylacetone and maleic acid based co-polymers are added in spraying solutions. The deposition temperature is varied from 150 to 350 °C, depending on the substrate type. The surface morphology (AFM) and composition (XRD) are correlated with optical properties (UV-VIS, FTIR), for op-

timizing the deposition parameters of Al₂O₃ thin film required for an alumina based cermet (Ni/Al₂O₃). In addition, a model of the alumina film formation (nucleation and growth) from precursors' solution containing macromolecular compounds as additives is proposed.

15:50 Poster B23

Crystallinity of Nanostructured Cu₂S Thin Films Deposited by Spray Pyrolysis. A Defect Chemistry Approach

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Copper sulfides (Cu_xS, $x = 1.8-2$) thin films are recognized as attractive materials for Solid State Solar Cells (SSSC). The optimum photovoltaic efficiency is obtained when chalcocite (Cu₂S) is used as p-type semiconductor and/or visible-light absorbing layer in a junction with TiO₂. Depending on the deposition conditions, Cu₂S shows mixed electronic-ionic conduction, with predominant electronic conductivity. The p-type conduction is the result of crystalline point defects (free holes from acceptors levels of copper vacancies), while ionic conduction is due to the Cu⁺ ions mobility within the crystal lattice.

Chalcocite thin films were deposited onto TCO (SnO₂:F) glass by chemical spray pyrolysis. As precursors, aqueous and mixtures of water:alcohol solutions of copper(II) chloride and thiourea with Cu:S molar ratio in the range 0.32-0.35 have been used. Depositions were done at 235 °C, respectively 285 °C. The X-ray diffraction analysis of as-deposited thin films showed that Cu₂S may have one of the tetragonal, orthorhombic or monoclinic crystal structure. The films crystallinity can be controlled by varying the deposition parameters: precursors' ratio and concentration, solvent and deposition temperature. According to crystallographic data (distance between lattice planes, d , lattice parameters, a , b and c , average crystallite sizes, D , average lattice strain, ϵ), the crystalline point defects in Cu₂S crystal lattices are identified. The defect chemical equations, using the Kröger-Vink notation, are proposed, explaining the defect formation and the conduction mechanisms in Cu₂S thin films.

15:50 Poster B24

Structure studies on Ni-Mo electrodeposited alloys with polymers by X-ray diffraction and Auger electron spectroscopy

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The aim of this paper is the presentation of the structure and Auger electron spectroscopy studies on the electrodeposited Ni-Mo alloys with different polymers. Composites based on Ni-Mo alloys with polythiophen, polypyrrole and polyethylene were obtained by electrochemical method. Depending on the potential and current density of electrodeposition and electropolymerization processes. The structural analysis made by X-ray diffraction shows that the solid solution of molybdenum in nickel is forming. The unit cell parameters of solid solution are slightly changing with the increasing of molybdenum contents in the alloy. The analysis of the diffraction line broadening indicates presence of the Ni-Mo solution nanocrystallites in the deposited layers. The Auger electron spectroscopy verifies both the presence of the solid solution of molybdenum in nickel and presence of polymers in composites. The SEM images show the general microstructure typ-

ical for the grain structure. Composites obtained by electrochemical method studied in this paper are unique in that the electrochemical methods can be used for processing ceramics, polymers, metals, composites and hybrid materials. The co deposition of alloys with polymers or polymerization with alloy decomposition have created new electrode materials for a number of applications including rechargeable batteries, electrochemical sensors etc.

15:50 Poster B26

Surface potential of conversible self assembled monolayer

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Organosilane self assembled monolayer have been widely applied to the artificial control of surface chemical functionalities. These chemical functionalities is very important to promote or minimize the biomolecular adhesion because these SAM surfaces are modified with such materials or molecules, the resulting surface modification chemistries are governed by intermolecular interactions between the SAM surfaces and the adsorbing molecular species [1-4]. In addition to chemical functionalities, SAM provide the ability to form micro pattern. By patterning aminosilane SAMs, microtemplates have been fabricated and successfully applied to the spatial arrangement of biomolecules. In order to prepare the terminal functional group, aminosilane self assembled monolayers (SAMs) are formed onto the photochemically cleaned SiO₂/Si substrates by the chemical vapor deposition method. Each of the samples are sonicated for 20 min in absolute ethanol, toluene, 1 mM NaOH and 1 mM HNO₃ aqueous solutions. The surface chemical functionalities of amino-terminated SAMs are analyzed with an atomic force microscope and Kelvin-probe force microscopy. The molecular functionalities can be switched on and off by changing their polarization states while applying various bias voltages ranging from -6 to + 6 V to the SAM. Depending on the bias voltage, surface potential of the SAM is 150 mV at -6V and - 50 mV at 6 V. The NO-terminated SAM surface is to be negatively polarized since the NO groups attract electrons from the aromatic rings that consist of the SAM. Therefore, the surface potential of the SAM shifts toward the negative direction. In contrast, the NH-terminated surface is positively polarized since the NH groups supply electrons to the aromatic rings. These conversible reduction and oxidation processes are very useful for memory device writing and erasing an information data depending on differences in the surface potential.

15:50 Poster B28

Influence of surface pre-treatment on the pore growth of nanoporous alumina at room temperature

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Nanoporous templates, such as anodic porous alumina, have gained much importance in the last 20 years due to their potencial industrial and technological applications for nanometric device fabrication. In fact, because the existent nano-lithographic techniques (e-beam. X-ray, focused ion beam) are expensive and have a low throughput, new

assembling methods are needed for the large scale inexpensive production of nanodevices. Nanoporous anodic alumina templates, which can be readily obtained by common electrochemical processes, may be one such method for achieving the required nanodimensions. These templates can be used to obtain different nanomaterials such as nanodots, nanochannels or nanowires, showing their versatility of use. Furthermore, the fact that different ordered structures (hexagonal, square or triangular lattices) have been obtained by two step anodization and pre-patterning techniques, opens new possibilities for the applications of nanoporous anodic alumina.

In this work we study how different pre-treatments (as-rolled, etanol degreasing, immersion in 1M NaOH for native oxide removal, diamond paste polishing, electropolishing and ion-milling etching) of high purity (99.997%) aluminum foils (prior to the first anodization step), influence pore growth and organization. Anodizations were carried out in 1.2 M H₂SO₄ for 1 hour, at room temperature. The current transients were monitored during anodization, allowing us to probe pore formation. After the first anodization we observed, using Scanning Electron Microscopy (SEM), the top of the anodized foil (alumina surface). The removal of this alumina layer allowed us to observe the pattern formed at the underlying aluminum surface. SEM images after the second anodization (under the same conditions as the first) were also performed. A comparative study of the influence in the porous structure of all pre-treatments will be shown and discussed.

15:50 Poster B30

Contributions Regarding the Research of Some New Nanostructured Coating Materials

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Organic-inorganic hybrid materials are very interesting for several applications thanks to their properties based on the synergistic combination of different components.

The combination, on a nanometric scale, of inorganic moiety with organic polymers has high potential for future applications, by combining the flexibility and usability of organic polymers with the durability and chemical stability of inorganic materials, to create high performance coatings that are easily incorporated into the traditional process.

Among the large number of methodologies for obtaining organic and inorganic building blocks in one material, the sol-gel process represents one of the preferred ways due to the mild operative conditions.

The paper presents combinations of alumina nanoparticles with functionalized acrylic polymers, that lead to the enhancement of coating properties, such as biocide activity. Our preliminary results with acrylic polymeric matrices can be explained by assuming a core-shell-like structure for the acrylic polymers and alumina nanoparticles.

15:50 Poster B31

Ni doped ZnO nanowires

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Ion track nanoporous membranes were used as templates for electrochemical fabrication of Ni doped ZnO nanowire arrays. The approach allows a good control of wires morphology, composition and structure. In this report we will present our results regarding the influence of deposition conditions such as deposition voltage and bath composition (concentration of the metallic ions and presence of wetting additives) on the preparation of ZnO nanowires doped with nickel.

Scanning and transmission electron microscopy were employed for the study of morphology and structure of the nanowires. Energy dispersive X-ray analysis was used for compositional studies.

15:50	Poster	B32

Functional Nanostructured Coatings by Molecule-based PECVD Techniques

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Thin film deposition by CVD techniques plays an important role in the development of both decorative and functional coatings, important for their technological implications. Commonly, multi-component materials are prepared from a mixture of precursors; however the efficiency of such processes is hampered by the mismatch of chemical reactivity such as thermal stability, vapour pressure etc. among the precursor species. Given the inherent differences in the physico-chemical properties phase separation and elemental segregation is commonly observed in CVD deposited materials. The de-mixing of elements in multi-component systems is thermodynamically driven and sensitive to the chemical behaviour of the precursors. Transformation of precursor compounds possessing bonding features inherent to the solid-state lowers the need of diffusion and counterbalances the thermodynamic impediments. Recently, we have designed several new metal-organic systems and demonstrated their suitability to deposit *decorative and functional coatings* on metallic and non-metallic substrates. For instance, we have investigated the decomposition of single molecular sources like $\text{Hf}(\text{O}^i\text{Bu})_4$, $\text{Ti}(\text{O}^i\text{Pr})_4$ and hexamethyldisiloxane (HMDSO) in plasma-enhanced CVD techniques to deposit hafnium oxide (HfO_2), titanium oxide (TiO_2) and silicon (SiO_x) coatings on different substrates as single-layer or multi-layer systems, and have investigated their application as anti-reflective and scratch-resistant surfaces. The plasma-activation process enhances the deposition rates and is suitable for deposition at low substrate temperature like polycarbonate, the film attributes are sensitive to the deposition conditions and a small variation can be detrimental to the film properties. In addition, we will address the role of precursor chemistry in plasma-enhanced deposition of nanostructured coatings.

15:50	Poster	B33

Microstructural analysis of ZnO powders with different concentrations of Al dopant obtained by means of hydrothermal synthesis

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The ZnO powders were obtained by means of hydrothermal synthesis using ZnNO_3 as precursor and NaOH as hydrolysis agent. The hydrothermal synthesis was performed in a Cortest autoclave at the following parameters: temperature of 200°C, pressure of 4.5 bars and time of 120 minutes. The precipitates have been filtered, washed with distillate water and dried at 110°C for few hours. The samples obtained by means of hydrothermal synthesis have been used as precursors in a vapour-condensation treatment in a solar reactor. Three types of samples were investigated: pure ZnO, ZnO doped with 0.05wt% Al and ZnO doped with 2.5wt% Al. The powder samples have been analyzed using different X-ray diffraction methods: Williamson Hall plot, Warren-Averbach, double-Voigt and Rietveld refinement. Qualitative phase analysis confirmed the existence of the ZnO hexagonal phase. No Al phase has been identified in the X-ray diffraction pattern. In all the cases nanocrystalline ZnO was obtained. The vapour-condensation treatment has enhanced the diminution of microstrain. Scanning electron microscopy was performed and the results were used for completing the X-ray diffraction analysis.

15:50	Poster	B34

Rhenium electrodeposition process onto p-Si(100) and electrochemical behaviour of the hydrogen evolution reaction onto p-Si/Re/0.1 M H_2SO_4 interface.

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In this study the rhenium electrodeposition process onto p-Si(100) and the photoelectrochemical behaviour of the hydrogen evolution reaction (HER) on the pSi/Re electrode system were examined. The study of the formation of p-Si/Re system was carried out by means of cyclic voltammetry (CV), and the potential-steps method from which the corresponding nucleation and growth mechanism (NGM) were determined. Both methods were performed under illumination for the electrons photogeneration in the conduction band of the p-Si. Likewise, a morphologic analysis of the deposits obtained at different potential values, it was realized by means of Atomic Force Microscopy (AFM). The HER study onto the p-Si modified by rhenium, was carried out by means CV using 0.1 M H_2SO_4 electrolyte.

The results obtained indicated that ReO_4^- ions are reducing in the surface of the semiconductor. The NGM obtained through adimensional analysis of the j_{ph}/t transients was 3D progressive nucleation diffusional controlled which was confirmed by AFM. The analysis of the HER using the p-Si/Re electrochemical system in the acidic media indicated that the rate of this process increases approximately in two orders of magnitude, fact that can be attributed to the photoelectrocatalytic effect of the p-Si/Re system on the HER.

15:50	Poster	B35

Dielectric properties of nanocomposites on the basis copper sulfide and porous silicon oxide

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Purpose of offered work is to study electrical properties of nanostructured composites on the basis of copper sulphide and porous silicon oxide in variable field. Porous structures were formed by method of sol-gel technology. Tetraethylorthosilicate [TEOS] was exposed hydrolysis in water, and added silasorb- 600, 10 mkm, and as a result was obtained sol, which is after drying it turned out into the gel, and after annealing at 1000°C turned out into the xerogel. After annealing sol of polysilicon acid turned out into O-Si-O- silicon-oxide lattice. In the volume of obtained porous structures were formed copper sulphide nanostructures with the help of successful ion layered adsorption and reaction (SILAR). Researches of samples capacity dependences on frequencies (at 1V) showed that, in small frequencies ($\nu=265$ Hz-5 kHz) capacity of samples changes mirror. The situation changes by increasing of frequency (5 kHz-1 MHz). Strong dispersion of dielectric conductivity from the frequency is observed in xerogel. Copper sulphide including to the volume of xerogel results increasing of permittivity and decreasing of frequency dispersion. Increase of permittivity related with copper sulphide filling of porous. Investigation of dielectric loss dependence on frequency showed that, maximum loss after including copper sulphide is shifted in high frequency diapason decrease half-width absorption line. Peak value loss tangent is increased after including copper sulphide. Conducted investigations showed that, in high frequencies ($\nu=10$ kHz-1 MHz) complex resistance of samples decrease after including copper sulphide. Thus nanocomposites have been formed on the basis of CuS: xerogel silicon oxide. It was shown that, with the help of including copper sulphide into the matrix it is possible to change electrical properties of obtained structure. This work carried out by STCU financial support, grant № 3486.

15:50 Poster B36

Wet pre-treatment of poly(butylene) terephthalate-poly(ethylene) terephthalate-polycarbonate co-polymer and subsequent metallization by electroplating

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Surface modification of poly(butylene) terephthalate-poly(ethylene) terephthalate- polycarbonate, PBT-PET-PC blend, using sodium hydroxide pre-treatment and subsequent Cu deposition through electroplating technique is described. By pre-treatment with sodium hydroxide, a rough with carboxylate ions surface is obtained compared to the control as is shown by the etching rate. The molecular fragments generated during the pre-treatment process are analyzed by Fourier transform infrared spectroscopy and X-ray photoelectron spectroscopy. The roughness and the cross-section morphologies of the blend is observed by scanning electron microscopy. After the pre-treatment, the substrate surface is activated by using Sn/ Pd colloidal activating bath, and then a thin Ni layer is deposited by electroless deposition for 6.5 min. Finally, Cu layer is obtained by 30 min electroplating. Adhesion of Cu layer evaluated by the Scotch tape test mainly depends on the surface roughness and the palladium content of the activating bath used.

15:50 Poster B37

Ketone based electrolytes for 3d-metallic layers deposition.

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Electrodeposition of metallic layers are important technological processes because of their low cost and wide availability. Nonaqueous electrolytes are widely used for deposition of Rare Earths or alkaline metals. We have found that these types of electrolytes could also be used for 3d elements deposition. Aim of this work is demonstration of new type of almost nonaqueous electrolyte. Unlike commonly used solutions, the electrolyte based on ketone and inorganic acid has property of universality. Under similar conditions metallic layers of few different 3d elements (Fe, Co, Ni, Cu, Zn) could be deposited. Possibilities of multicomponent deposition are discussed. Properties of the deposited layers are reported and the method of deposition is presented.

15:50 Poster B38

Textured-nanostructured zinc oxide films deposited by the spray pyrolysis process.

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Zinc oxide films have been deposited by the pneumatic spray pyrolysis process using a 0.1M zinc acetate dissolved in deionized water start solution. Three kinds of films were prepared a) without surfactant and without doping, b) with surfactant and without doping and c) with surfactant and with doping. Polyvinylpyrrolidone, 360k molecular weight, was used as surfactant, in a proportion of 1.02 g per 70mL of solution. The doping of solution was achieved adding 0.1g iridium acetylacetonate (IrAAC) per 70 mL. All kind of films were deposited at the same deposition conditions: substrate temperature 330 °C, gas flow rate of 10 L/min being air the carrier gas, solution flow rate of 2 cm³/min. All these films were submitted to an annealing process at 500 °C during one hour in air atmosphere. The thickness of the films depends on the spraying solution and it takes values from 415 up to 1150 nm. From optical absorbance measurements the calculated grain size has values between 7 and 13 nm, with the peak of excitonic absorption located at wavelengths of the order of 360 nm. However, the grain size determined from x-ray diffraction measurements and atomic force microscopy is around 30 nm, with maximum rms roughness of 3.95 nm. High resolution transmission electron microscopy analyses are being carried out in order to clear these results. On the other hand, films of the three kinds were deposited onto substrates of alumina with inter-digit gold electrodes, previously deposited by vacuum thermal evaporation, to form hydrogen sensors. Some sensors with type b zinc oxide films were surface doped with an alcoholic solution of IrAAC by impregnation. Dynamic measurements of the sensitivity to 10⁵ ppm

of hydrogen, as test gas, in an air flow were achieved. The temperature for maximum sensitivity depends of the active layer. Meanwhile, the maximum sensitivity takes values from 12 for sensors with type a zinc oxide film up to 8280 for iridium surface doped sensors.

15:50 Poster B39

Introduction of metal dopants and/or Ag nanoparticles into direct-patternable ZnO thin film formed by photo-chemical solution deposition

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Zinc oxide (ZnO) has drawn much interest as a potential transparent conducting oxide (TCO) for applying to solar cell and front electrode of electro-luminescent devices due to non-toxicity, low cost, and high abundance and stability in hydrogen plasma. For the enhancement of electrical property of TCOs, dopant introduction and hybridization with conductive nanoparticles have been investigated. Finally metal dopants and nanoparticles have been used in the electronic industry for the manufacture of conductive circuits and internal electrodes. An introduction of metal dopants and nanoparticles into TCOs influences its conductivity and also transparency but their influences are not always prospective.

In this work, the optical and electrical characteristics of ZnO films with the introduction of metal dopants with/without Ag nanoparticles have been investigated to check whether there is a combined effect between metal dopants and Ag nanoparticles on the characteristics of ZnO film. ZnO films were formed on glass substrate by using photo-chemical solution deposition of Ag nanoparticles dispersed or various metal (Ag, Cd, In, or Sn) contained photosensitive ZnO solutions. The usage of photosensitive solution permits us to obtain a micron-sized direct patterning of ZnO film without using conventional dry etching procedure. The phase formation and crystallinity of ZnO film were monitored with x-ray diffractometer. The optical transmittance measurement was carried out using UV-VIS-NIR spectrometer and the electrical properties such as sheet resistance and conductivity were observed by using four-point probe. X-ray photoelectron spectroscopy was used to investigate whether there is a chemical interaction between additives and matrix ZnO film.

15:50 Poster B40

Electrochemical mechanistic study of ZnO Nanowires Growth by Electrodeposition Inside Alumina Templates

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Zinc oxide nanowires (ZnO NWs) made by potentiostatic electrodeposition (ED) inside both anodic alumina membranes (AAM) and polycarbonate membranes have been widely reported.¹⁻² Unlike to metallic NWs, here it's not possible to see clearly the growing behaviour in current-time transients (i.e. NWs growing out the pore mouth) probably due to resistive nature of semiconducting materials. Our goal

is to address these aspects by means of in situ method and then to optimize the growth of ZnO NWs. For metallic reference, we employed electrochemical parameters obtained for zinc nanowires (Zn NWs) which also is an interesting indirect way to form ZnO NWs after post-treatment.³

AAM were prepared by anodization in Oxalic Acid. Zn NWs were grown in order to determine the critical time to fill the AAM pores with Zn NWs completely. We compare these results with those obtained in ZnO NWs growth and the electrical charge calculated for both Zn and ZnO NWs. Different electrochemical techniques allowed us to determine critical parameters in order to understand the growth mechanism: By Potentiodynamic measurements we found different current behaviour at different nanowires lengths and conventional Impedance together with Mott Schottky measurements allowed to get information about the charge transfer resistance and characterize the semiconducting properties. The current-time transients gave information about diffusive behaviour of electroactive species in each case. We will discuss the effect of key parameters like applied potential and pore length in correlation with ex situ characterization (Field emission scanning electronic microscopy with X-Ray microanalysis and X-Ray Diffraction)

References

1. M.J. Zheng, L.D. Zhang, G.H. Li, W.Z. Shen; Chem. Phys. Lett. 363 (2002) 123–128. (68-02)
2. Y. Leprince-Wang, A. Yacoubi-Ouslim and G.Y. Wang; Microelectronics Journal 36 (2005) 625–628. (290-81)
3. J. Wang, M. Tian, N. Kumar and T. E. Mallouk; Nano Lett. 5 (2005) 1247.

15:50 Poster B41

Iron oxide and oxyhydroxides thin films obtained by electrodeposition

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Iron oxides-oxyhydroxides are abundant materials in nature, and play a relevant role in many scientific and technical applications, participating in a number of environmentally and industrially important processes.

There are 15 iron oxides, oxyhydroxides, and hydroxides known to date. They show a variety of physical and chemical properties depending on their chemical composition and crystal structure. Among the more important ones, we have magnetite (Fe₃O₄), goethite (FeOOH), and lepidocrocite (α-FeOOH).

Different iron oxides and iron oxyhydroxides thin films were obtained by potentiostatic electrodeposition on a titanium foil substrate, from an aqueous solution 0.01M. FeSO₄(NH₄)₂SO₄·6H₂O + 0.04 M CH₃COOK and pH 6.0. Changes in the deposition potential allow to obtain different crystalline pure phases of the iron oxide-oxyhydroxides as magnetite, goethite, and lepidocrocite thin films with different structural and morphological characteristics. The films were characterized by XPS, XRD, SEM and TEM.

15:50 Poster B42

p-type silicon electrode modification with ferrocene. Effect of the distance electrode-molecule in its electric properties.Gonzalo Riveros¹, Sebastián Meneses²

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The formation of a hybrid semiconductor-molecule is a feasible alternative to extend silicon properties to a molecular scale so it can be applied to the development of nanoelectronic devices. In this field, there are many studies that exhibit the union of different organic and organo-metallic molecules to the silicon surface by way of different routes (UV activation, UHV). Moreover, the union of active redox molecules, specifically porphyrins, to the silicon surface is a complicated procedure, mainly due to previous synthesis and characterization of these molecules. On the other hand, the use of derived molecules of ferrocene is limited to a few compounds commercially acquired, thus restricting potential research of these systems. Therefore, this study shows an alternative route for the modification of silicon using derived molecules of ferrocene anchored by an alkyl chain of variable length. The modification is carried out by adding a double bond of an alkene halide to the H-terminated silicon surface activated by white light and its subsequent reaction with monolithio ferrocene. Of this way, only the ferrocene molecule is linked to the carbon chain previously anchored to the silicon, thus avoiding the forming of by-products onto the surface. This method paves the way for a systematic study of the effect that the length of the chain will have on the electric properties of these hybrid materials. Chains with 3 and 5 carbon chains equipped with ferrocene terminals were linked to (100) p-type silicon. AFM images and contact angle measurement were put into effect in order to characterize the modified surface. The electric properties of both samples were determined by cyclic voltammetry, AC voltammetry, open circuit potential amperometry and electrochemical impedance spectroscopy measurement. All of them exhibited variations related with the length of the carbon chain.

Acknowledgements. This work was supported by FONDECYT-Chile (Grant N° 1050700).

15:50 Poster B43

The polymorphic modifications of CdS prepared from coordination compounds of thiourea

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The well-directed synthesis of cadmium sulphide from aqueous solutions of its coordination compounds with a thiourea (SC(NH₂)₂, thio) allows to prepare the thin films, which have consisted of one or two polymorphic modifications (zinc blende or wurtzite). The technique of thermal destruction of thiourea coordination compounds by sputtering of aqueous solutions on the hot substrates has been employed in this work. The main purpose of this study is to discuss the experimental results, which have been received during the examination of the real crystal structure of CdS thin films. CdS films were fabricated from aqueous solutions of cationic [Cd(thio)₄]²⁺ and neutral [Cd(thio)₂X₂] (X – Cl-, Br-, I-, and CH₃COO-) complexes. The X-ray analysis data

show that CdS films prepared from aqueous solutions of [Cd(thio)₂(CH₃COO)₂], [Cd(thio)₄](NO₃)₂, and [Cd(thio)₄]SO₄ complexes are characterized by zinc blende crystal structure. Under the same experimental conditions (concentration of solution and temperature of substrate) CdS layers fabricated from aqueous solutions of [Cd(thio)₂Cl₂] and [Cd(thio)₂Br₂] coordination compounds are crystallized in wurtzite structure. Cadmium sulphide samples prepared from [Cd(thio)₂I₂] complexes consist of two phase mixture. Together with the main wurtzite modification the phase with zinc blende crystal structure has been observed in these CdS layers. The values of tetrahedral radii of Cl and Br are close to radius of sulfur atom (0,104 nm). During the crystallization process the iodine atoms (r = 0,128 nm) create the essential distortions in crystal structure, which have led to formation of zinc blende phase.

15:50 Poster B44

The function of thiourea coordination compounds composition in synthesis of CdS thin films

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The synthesis of metal sulphide thin films from aqueous solutions of its coordination compounds with a thiourea (thio) has some advantages. First of all, this technique allows controlling the functional properties of metal sulphides thin films by designing of coordination compounds with the different structure and composition. The technique of thermal destruction of thiourea coordination compounds by sputtering of aqueous solutions on the hot substrates has been employed in this work. The main purpose of this study is to discuss the experimental results, which have been received during the examination of the optical properties and the real crystal structure of CdS thin films. CdS films were fabricated from aqueous solutions of [Cd(thio)₄-nXn]⁽²⁻ⁿ⁾⁺ (where n = 0, 1, 2; X – Cl-, Br-, I-, and CH₃COO-). The presence of halogen and oxygen atoms results in a capability of CdS films doping by impurities in anion sublattice. It has been established that for CdS thin films prepared from aqueous solutions of neutral thiourea complexes with halogen anions the intensity of a photoluminescence increases approximately in 40 - 100 times. Besides, the optical band gap decreases in CdS (Hal – Cl, Br, I) layers. In CdS films the shift of the optical absorption edge demonstrates that energy spectrum changes have intensified with increasing of atomic weight of halogen impurity atoms. At the same time the electrically inactive isovalent substituents OS are delivered in CdS films by the thermal destruction of [Cd(thio)₄-nXn]⁽²⁻ⁿ⁾⁺ with acetate ligands. The mechanism of photoluminescence stimulation under influence of halogen impurities is discussed in this work.

15:50 Poster B45

Semiconductor properties of α -Fe₂O₃ (hematite) thin films prepared on FTO electrodes by hydrogen peroxide electroreduction. Influence of the E/t perturbation program used during electrosynthesis of iron oxyhydroxide precursor.

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In this work the semiconductor properties of α -Fe₂O₃ (hematite) with different carriers density, N_D , were studied. This oxide was obtained from iron oxyhydroxide thin films (FeOOH) prepared using the H₂O₂ reduction reaction on a conductor glass FTO (SnO₂:F). This reaction takes place in the interface: FTO / 0.1 M H₂O₂ + 0.05 M FeCl₃ + 0.05 M NaF at 50°C. To obtain α -Fe₂O₃ with different N_D , the oxy-hydroxide precursor was synthesized by the repetitive potential pulses technique. This E/t program consisted to apply a cathodic potential $E_C = -0.9$ V during a time $0.5 \text{ s} \leq t_C \leq 5 \text{ s}$ and after, an anodic potential $0.0 \text{ V} \leq E_A \leq 0.5 \text{ V}$ was applied during a time $t_A = 5 \text{ s}$. At E_C , onto surface the OH⁻ electrogeneration and iron oxy-hydroxide precipitation take place. At E_A , it is procured that Fe(III) deposited maintains its oxidation state. The electrodeposition process was stopped when the total time at E_C was 30 min. Then, FeOOH thin film was transformed into α -Fe₂O₃ upon heat treatment at 525°C by 30 min. This phase was characterized morphological and structurally by SEM and XRD, respectively. By Orange II method, a roughness factor 17 ± 2 was determined. This is in agree with dendritic structure observed by SEM. From Mott-Schottky plot obtained at 10 kHz in 0.1 M NaOH, the flat band potential ($E_{FB} = -0.63$ V vs HNE) and the donors density, N_D , were determined. N_D was dependent of anodic potential, E_A , that was used during the oxyhydroxide precursor formation. In general, N_D decreased from $1.2 \cdot 10^{19} \text{ cm}^{-3}$ at $E_A = 0.0$ V until $2.2 \cdot 10^{17} \text{ cm}^{-3}$ at $E_A = 0.5$ V. Photocurrent transients (PCT) and cyclic photovoltammetry in an 0.05 M NaI + 0.1 M NaOH solution were measured. These results are discussed considering the processes associated to the electron-hole pair generation, charge transfer in the interface, hole diffusion and electron-hole pair recombination.

15:50 Poster B46

The effect of P on the Seebeck coefficient of Bi₂Te₃ thick film

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Phosphorous doped Bi₂Te₃ thick films have been prepared by electrochemical deposition. The average grain size of the films was calculated to be 14-26 nm based on Scherrer's equation. The effect of P on the Seebeck coefficient of thermoelectric Bi₂Te₃ thick film was investigated. The results show that P-doped thick film has n-type conductivity with the Seebeck coefficient of -35 μ V/K. The correlation between P site occupancy in the crystal and the Seebeck coefficient was discussed.

15:50 Poster B47

Microelectrode surface modification with carbonaceous nanostructured hybrid composite sensing layers

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Hydrogen peroxide sensing composite layers were prepared on Pt microwires by different approaches. A number of alternative preparation procedures were explored regarding not only changes in material and reagent composition but also different processing steps such as pretreatment, synthesis, polymerization and deposition of the sensing system constituents. Multiple-wall carbon nanotubes (CNT) and carbon black nanoparticles were used in this study. After optional oxidative pretreatment they were combined with ionic liquid, dispersion promoters and/or polymers and - essentially - with the Prussian Blue (PB) redox system, thus completing a hybrid layered structure. Various deposition techniques were employed: chemical, electrochemical (electropolymerization) or physical coating. Electroanalytical properties of the obtained microelectrodes were assessed by comparison of the cyclic voltammetry (CV) scans performed in physiological buffer solution (PBS) alone or with hydrogen peroxide added as the analyte.

The experimental results, showing correlations between observable electrocatalytic effects and structural properties of the composite layers resulting from their fabrication process, are comparatively displayed and briefly discussed. Cyclic voltammetric measurements confirmed that carbon nanomaterials improved and stabilized electrochemical parameters of the produced surface layers. The influence of CNT form (length, diameter) and surface modification resulting from covalent or non-covalent functionalization on their dispersion or interaction with the polymer matrix, metal substrate and the PB system could be also estimated. The structural effect is related to the facilitation of electron transport between the PB reaction centres and Pt substrate and stabilization of PB integrity within the layer.

The presented results have helped to select the promising microelectrodes for the assembly of the complete sensor for monitoring hydrogen peroxide level in biological systems.

15:50 Poster B48

Influence of treatment conditions on chalcopyrite films deposited at atmospheric pressure

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Spin-coating technique was used to deposit precursor layers for chalcopyrite films of the series CuInX₂ and Cu(In,Ga)X₂ where X=S or Se or (S,Se). The influence of different parameters of the process, such as solution composition, air pretreatment and chalcogenation treatment is discussed with respect of film applicability in photovoltaic devices. Layer morphology and stoichiometry varied widely with the different treatments.

15:50 Poster B49

Preparation, structural and mechanical properties of electrodeposited Co/Cu multilayers

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In present study, Co/Cu multilayers with bilayer period thickness less than 100 nm were grown by electrodeposition. By changing deposition potentials alternating Co and Cu layers were deposited from single solution on (001)-oriented Si wafers and polycrystalline copper plates.

The structure of Co/Cu multilayer was investigated by X-ray diffraction, scanning electron microscopy (SEM) and transmission electron microscopy (TEM). It was found that the multilayered deposits have columnar structure, with various column diameters. The column diameters and the preferential crystallographic orientation depend on the total thickness of the deposit (number of Co/Cu bilayer repetitions).

The mechanical properties of Co/Cu multilayers were investigated by hardness measurements on Knoop microhardness tester (10 g load) and tribological tests on T-05 tester. For these investigations the series of Co/Cu multilayers with the bilayer periods in the range from 2 nm to 40 nm (with constant total thickness of the deposit equals 3 μm) were electrodeposited on copper plates. It was found that hardness of the Co/Cu multilayers depend on the bilayer period. The maximum hardness, 500 HK_{0.01}, could be obtained for the nanoscale multilayered coating with the bilayer period of 10 nm, which is much higher than those of Co and Cu single layer coatings. We also observed completely different wear mechanism of Co/Cu multilayers for bilayer period 6, 10 and 40 nm.

15:50 Poster B50

Nanocomposite Field Effect Transistors based on Zinc oxide/polymer blends

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Significant progress is being made in the realization of thin-film transistors (TFTs) for application in various electronic devices and circuits. Currently, one of the important challenges in this area is to design low-cost and stable organic semiconductors that possess high field-effect mobilities for constructing low-power high-speed transistor devices. However, there are only limited stable and cheap organic semiconductors that are applicable for OTFT applications. Here, we report the work in our laboratory that focus on stable, inexpensive and high field-effect mobility nano-composite materials for the potential application in OTFT technologies. Solution processed polymer based nano-composite field effect transistors with wide band gap semi-conducting ZnO nano-tetrapods and nano-crystals dispersed in the polymer matrix were utilized to study the field effect behaviour. The electrical characteristics of polymer based wide band gap nano-crystal or nano-tetrapod composite devices exhibit an increase in the hole mobility up to two orders of magnitude higher than the pristine polymer.

Symposium C

Programme

Monday, 17 September

Opening & Plenary Session

Czochralski Award Ceremony & Acta Gold Medal Ceremony
Monday morning, 17 September, 10:00

Lunch break

Monday afternoon, 17 September, 12:30

Opening

Opening
Monday afternoon, 17 September, 14:15

Parallel Session 1

Monday afternoon, 17 September, 14:30

14:30 Invited

ALD of TiO₂ based photocatalysts

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Titanium dioxide is the most widely studied and used photocatalyst. TiO₂ based photocatalysts are used for air and water purification, and as self-cleaning and sterilizing coatings. A lot of methods exist for preparation of TiO₂ photocatalysts in the form of both powders and coatings. Among these methods atomic layer deposition (ALD) can distinguish itself mostly based on its excellent conformality and possibility to tailor the composition at atomic layer level. This paper summarises our work on ALD of TiO₂ based photocatalysts. Areas to be covered include comparison of various TiO₂ ALD processes available, doping and modification of ALD-TiO₂ in an effort to make it active also under visible wavelengths, preparation of three-dimensional photocatalyst structures exploiting the unique features of ALD, and enhancement of the photocatalytic process by an external bias voltage (photoelectrocatalysis).

15:00 Invited

Ab initio precursor design for rare earth oxide ALD

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At the atomic scale, the success of atomic layer deposition (ALD) depends on the efficient elimination of precursor ligands from the surface of the growing film. For the ALD of high- κ rare earth oxide films, we consider alkoxide, cyclopentadienyl, beta-diketonate and amide pre-

cursors of La and Sc, as representative rare earths. We use density functional theory to compute their reactivity with respect to ligand elimination during ALD with water vapour, neglecting surface effects so that intrinsic precursor chemistry comes to the fore. The energetic data are correlated with electronic properties and steric demand, allowing recommendations for precursor design to be made. Ligands that are weaker Lewis bases than Brønsted bases are shown to be more successful as components of ALD precursors.

Coffee break

Monday afternoon, 17 September, 15:30

Joint Poster Session (Part I)

Monday & Wednesday
Monday afternoon, 17 September, 15:50

Tuesday, 18 September

Parallel Session 2

Tuesday morning, 18 September, 9:00

9:00 Invited

ALD of High- κ Dielectrics and Metals

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Selection of an appropriate precursor is a critical first step in developing ALD processes. Many precursor properties are required: sufficient vapor pressure at a temperature at which decomposition is negligible, self-limiting deposition reactions that produce a pure product at a high enough rate on suitable substrates, a synthesis procedure that produces a sufficiently pure precursor at a cost that is low enough for applications, and long shelf life. Several other precursor properties are desirable: low melting points (preferably below room temperature), low toxicity, and non-corrosive precursors and reaction byproducts. Case studies of precursor design will be discussed: ALD of high- κ dielectrics (hafnium oxide, zirconium oxide and multicomponent lanthanide oxides), and ALD and CVD of transition metals (iron, cobalt, nickel, copper and ruthenium) and their nitrides and oxides. The connections between precursor structures and the desired properties will be emphasized.

9:30 Invited

Recent development in the design of cyclopentadienyl-based precursors for the ALD of high- κ thin films

Paul R. Chalker¹, Anthony C. Jones², Helen C. Aspinall², Peter N. Heys³, Rajesh Odedra³

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The introduction of hafnium oxide-based gate dielectrics for the 45-nm CMOS technology node made by leading semiconductor device manufacturers was a necessary instrument in downscaling of transistor dimensions in order to match consumer demand for increased computing power without additional cost. For future nodes, the ITRS roadmap predicts the need for equivalent oxide thicknesses (EOT's) down to 5 Å which will only be possible using materials with even higher κ -values ($\kappa > 30$), while at the same time sufficiently large band offsets will be needed to control gate leakage. New dielectric materials based on doped ZrO_2 and HfO_2 , or lanthanide oxides have been considered as potential candidates in this respect. In order to deposit these materials via ALD a key requirement is the availability of precursors with appropriate deposition characteristics. This requires the molecular design of ALD precursors tailored to optimise process parameters such as evaporation temperature, oxide deposition temperature, layer purity and uniformity. For example, careful selection and design of ligands in complexes such as $[(\text{RCp})_2\text{MMe}(\text{OR})]$ (R = alkyl, Cp = cyclopentadienyl, M = Zr, Hf) leads to precursors with improved physical properties and significantly enhanced ALD performance. Even with careful precursor selection and design it can be difficult to obtain precursors with the desired properties, which has led to the increased use of liquid injection ALD techniques. Using this approach, a precursor is held at room temperature in solution until required at "point of use" in the ALD process allowing the use of higher evaporation temperatures while minimising precursor decomposition. In this paper, we describe the deposition of gate dielectric thin films by liquid injection ALD using the precursors $[(\text{MeCp})_2\text{MR}(\text{OR}')]]$ (M = Zr, Hf; R, R' = alkyl) combined with lanthanum and titanium Cp-based precursors.

10:00

Invited

Electrical properties of high-k materials prepared by ALD

Kaupo Kukli

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Atomic layer deposition has been recognized as a prospective, in several cases an appropriate, method for the fabrication of contemporary and advanced electronic devices - insulating layers in head gap devices, field effect transistors, three-dimensional dynamic random access memory structures. Attempts have been made to adopt several metal precursors in the deposition process of high-permittivity oxide thin films. Suitable chemical precursors have been searched among a list of compounds, such as metal halides, alkoxides, alkylamides, cyclopentadienyls. Accommodation of different precursors with the deposition process has resulted in a variety of physical and chemical properties, characterizing the resulting thin solid films. For instance, hafnium and zirconium alkylamides and cyclopentadienyls have been considered as promising precursor enabling the conformal atomic layer deposition. At the same time, some of these precursors suffer from the ability to decompose thermally, whereas other precursor produce films possessing significant crystallographic phase heterogeneity. The films grown in different processes show noticeable differences in the leakage current densities, conduction mechanisms, capacitance-voltage behaviour and defect densities. Some of these issues will be described in the present work.

Coffee break

Tuesday morning, 18 September, 10:30

Parrallel Session 3

Tuesday morning, 18 September, 11:00

11:00

Invited

In-situ investigation of the early stages of the growth by ALD of high-k dielectrics on silicon and high mobility-substrates

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The scaling down of modern nano-electronic devices, based on a MOS structure, has motivated an intense activity on high-k dielectrics grown on silicon as well as on high mobility substrates, such as strained-silicon, Ge, and GaAs. Among the most important deposition methods we find atomic layer deposition (ALD) and molecular beam epitaxy (MBE).

Defects at the semiconductor/high-k interface and in the dielectric layers play a crucial role in determining the device performance. There is a need to identify the microscopic structure of electrically active point defects in the dielectric and at its interface with the semiconductor.

The investigation of the early stages of growth is an important step towards the understanding of the growth mechanisms and interface formation. While in UHV deposition techniques, such as MBE, this study can be pursued with a variety of surface characterization methods, the ALD growth environment prevents the use of most of these powerful tools and limits these studies.

I will report on the *in-situ* characterization by electrically detected magnetic resonance spectroscopy (EDMR) and spectroscopic ellipsometry (SE), of the early stages of ALD growth of selected oxides on silicon, strained-silicon, and germanium. These investigations have been carried out using a novel home-made system where a small ALD chamber has been connected to an electron spin resonance (ESR) spectrometer and an ALD growth chamber equipped with an ellipsometer.

The *in-situ* characterization is compared with the *ex-situ* investigation of the high-k/semiconductor interface based on various techniques.

11:30

Oral

Atomic layer deposition of crystalline Al_2O_3 thin films

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Atomic layer deposition of Al_2O_3 from vapors of AlCl_3 and H_2O was studied in the substrate temperature range of 200–900°C in order to explore possibilities for growing crystalline films. On single crystal silicon and amorphous silica substrates, the films started to crystallize at temperatures exceeding 600°C being, however, of relatively poor crystallinity even when grown at temperatures as high as 800–900°C. Nevertheless, a marked increase of density and refractive index was observed with appearance of crystalline phase in the films. On single crystal sapphire substrates as well as on epitaxial TiO_2 and SnO_2 , epitaxial Al_2O_3 films were successfully obtained at 700°C and higher

temperatures. On epitaxial Cr_2O_3 , by contrast, the substrate temperature as low as 500°C was sufficient to grow epitaxial Al_2O_3 .

11:45

Oral

Conduction mechanisms in nanolaminates and mixture films grown by atomic layer deposition

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Interest in high-permittivity oxide films has grown mainly due to the replacement of silicon oxide in transistors and memory capacitors. Besides single metal oxides, attempts have also been made to tailor useful properties of different oxides, combined as mixed materials of variable stoichiometry, or by deposition of stacked layers of different oxides in the form of nanolaminates. Oxides exhibiting good insulating properties, such as Al_2O_3 , may be combined with oxides of high polarizability, such as TiO_2 . ALD processes are established, enabling conformal growth of dense oxide layers. However, combination of different materials induces additional amounts of bulk defects as well as multiple interfaces, possibly degrading the electrical properties of these insulators. In addition, different materials exhibit variable stability in contact to semiconductor substrates and metal electrodes, affecting the quality of contact charge barriers.

The $\text{Al}_{1-x}\text{Ti}_x\text{O}_y$ mixtures and Al_2O_3 - TiO_2 - Al_2O_3 nanolaminates with variable composition and thicknesses were grown to investigate the electrical quality of such films. Several samples were annealed in N_2 or O_2 at 700°C to study the effect of thermal post-deposition treatment. At similar capacitance values, films containing TiO_2 remained inferior to Al_2O_3 films in terms of insulating properties. The analysis of current-voltage characteristics revealed strong influence on defects and impurities. However, the modeling of electron emission into layered high-k stacks has first suggested the preferred exploitation of insulating materials with wider band gap even in defect-free cases. Further, the modeling suggested superiority of higher k films (TiO_2) when electrodes with higher work function were used, especially when EOT value of stack became smaller than 2 nm. In such films, the leakage in stack containing higher-k film (TiO_2) remained smaller even when a discrete impurity level was included to take into account trap assisted currents.

12:00

Oral

Characterization of the initial growth of hafnium silicate films in dependence of process parameters and substrate materials

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Hafnium based oxides, grown by atomic layer deposition, are the most prominent candidates for future high-k dielectrics in next generation DRAM technology. In order to temperature stabilize these materials and to achieve the crystalline phases with the desired high dielectric

constant k in the range of 25 - 30, doping, e.g. by Al or Si has drawn considerable interest over the last years.

Although much work was done in examination of the initial growth of HfO_2 -films from hafnium tetrachloride and water, there is a lack of information regarding the usage of metal-organic precursors and ozone, especially in the case of ternary films like $\text{Hf}_x\text{Si}_{(1-x)}\text{O}_2$. Therefore we have in our work examined the initial growth of $\text{Hf}_x\text{Si}_{(1-x)}\text{O}_2$ -films using tetrakis(ethylmethylamino)hafnium, tetrakis(dimethylamino)silane and ozone in dependence of process parameters (deposition temperature and chamber pressure) as well as substrate materials (SiO_2 , Si_3N_4 , Al_2O_3 , TiN). Most measurements were done by angle resolved X-ray photoelectron spectroscopy and interpreted with a model proposed by Mack et al [1]. With the model it is possible to determine the coverage and/or thickness of submonolayers/layers of $\text{Hf}_x\text{Si}_{(1-x)}\text{O}_2$ in dependence of process conditions.

In general the film growth is inhibited during the first 5 - 10 cycles, but nevertheless is 2-dimensional. It also does not show significant difference depending on the substrate material except for TiN. In this case the growth rate is nearly constant from the first cycle on, which is attributed to TiN-substrate roughness. It will be shown, that with increased deposition temperature and chamber pressure there is an increase in the growth per cycle value together with a change in Si/Hf-ratio to higher values. This can be explained by decomposition of the precursors at higher deposition temperatures along with lower impurity content.

[1] Mack et al, Applied Surfaces Science, Volume 252, Issue 23, 2006, Pages 8270-8276

12:15

Oral

ALD of ZrO_2 Thin Films Exploiting Novel Mixed Alkylamido-Cyclopentadienyl Precursors

Jaakko Niinistö¹, Kaupo Kukli^{1,2}, Mikko Ritala¹, Markku Leskela¹, Nicolas Blasco³, Audrey Pinchart³, Christophe Lachaud³, Nadia Laaroussi³, Christian Dussarrat⁴

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ALD has high potential in microelectronics for the growth of high permittivity (high-k) films to replace SiO_2 -based solutions as insulators for complementary metal oxide semiconductor devices and dynamic random access memory capacitors. For alternative high-k materials the leading solutions include the oxides of Hf and Zr. For ALD of these materials, alkylamides have gained wide interest as precursors. The suitable atomic layer deposition (ALD) growth temperature range for the Zr- alkylamide processes, e.g. TEMAZ ($\text{Zr}(\text{NETMe})_4$) is rather limited to obtain excellent film properties. Thermal decomposition of TEMAZ begins to play a major role in growth mechanism when the applied growth temperature exceeds 275°C , thus preventing the ALD-type growth mode and resulting in poor film uniformity. However, the relatively high growth rate favors the alkylamide precursors over many other options, such as dicyclopentadienyl (Cp)-type precursors that have been investigated.

In this presentation we introduce a novel class of zirconium precursors, combining the beneficial characteristics of alkylamides (physical state, volatility, reactivity) with the enhanced thermal stability of the cyclopentadienyl precursors. We compare several precursors from this

novel class and TEMAZ thermal properties (TGA/DT/DSC, vapor pressure) and ALD behavior. All precursors reported in this work provide comparable volatility and higher thermal stability than TEMAZ. In addition, one of the precursors reported in this work, namely $\text{Zr}(\text{EtCp})(\text{NMe}_2)_3$ provides comparable growth rate (0.9 Å/cycle with ozone as the oxygen source) and film properties with TEMAZ. Properties of films obtained with other similar mixed Cp-alkylamido-precursors are also discussed.

Lunch break

Tuesday afternoon, 18 September, 12:30

Parallel Session 4

Tuesday afternoon, 18 September, 14:00

14:00 Invited

Atomic Layer Deposited Al_2O_3 on semiconducting polymers for plastic electronics

Sandro Ferrari

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The strong sensitivity of organic/polymeric semiconductors to the exposure to O_2 and H_2O atmospheres makes the use of capping layers mandatory for the realization of stable devices based on such materials. Atomic Layer Deposition (ALD) provides smooth and pinhole-free films with a great potential as passivation layer for organic based devices. The use of Al_2O_3 grown by ALD on polymers has been explored by different groups and it was demonstrated that the growth mechanism depends on the nature of the polymer. When ALD oxides are employed to cap organic semiconductor based devices the control of the polymer/oxide interface is critical, since any diffusion of the oxide into the organic semiconductor leads to a degradation of the electrical properties of the device. Moreover the condition for the deposition of the capping layer might cause device degradation.

We show that the deposition of Al_2O_3 on transistors based on poly-3 hexylthiophene (P3HT) allows to obtain air stable devices. Nevertheless the growth of Al_2O_3 directly on the P3HT layer leads to a rough interface and significant intermixing between the oxide and the polymer, resulting in a deterioration of transistor performances. By using polymeric interlayers between the P3HT and the Al_2O_3 it is possible to prevent oxide interdiffusion. The choice of the interlayer is critical, and if a polymer with an high filling factor and proper functional groups is used it is possible to prevent oxide interdiffusion with a thickness of just 10 nm. We have considered particularly a poly-alcohol such as Poly-vinylphenol (PVP). We have studied in detail the growth mechanism of Al_2O_3 on the PVP. Transistors capped with Al_2O_3 /PVP are very stable in air, with no appreciable differences in the electrical characteristics when measured in vacuum or in air. In addition no significant degradation of the transistors electrical properties was detected even after one month of air exposure.

14:30 Oral

Influence of substrates on the growth of $\text{Ge}_2\text{Sb}_2\text{Te}_5$ films by combined atomic-layer- and chemical-vapor-deposition

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Stoichiometric $\text{Ge}_2\text{Sb}_2\text{Te}_5$ (GST) films were deposited on TiN/Si, chemically or thermally grown SiO_2/Si , atomic-layer-deposited TiO_2/Si and $\text{Al}_2\text{O}_3/\text{Si}$ substrates using a shower-head type 8-inch-scale plasma-enhanced ALD reactor at a wafer temperature of 200°C. $\text{Ge}(\text{i-C}_4\text{H}_9)_4$, $\text{Sb}(\text{i-C}_3\text{H}_7)_3$, and $\text{Te}(\text{i-C}_3\text{H}_7)_2$ were used as the Ge, Sb and Te precursors, respectively. In order to confirm whether the ALD or CVD reaction took place, the precursor purge and precursor injection times were independently varied. It appeared that the incorporation of Ge was ALD-like whereas those of Sb and Te were of CVD nature at 200°C. For investigating the nucleation and growth behaviors on each substrate, chemical composition and area layer density of the film were investigated by an X-ray fluorescence analyzer. The structure, cross-section and surface morphology of GST film were investigated by X-ray diffraction, field-emission scanning electron microscopy and atomic force microscopy. The X-ray diffraction showed that the as-deposited GST films on the TiN and TiO_2 substrates have an fcc structure with (200) preferred growth directions. They also have fcc structure but with (111) preferred directions on SiO_2 and Al_2O_3 substrates. There was negligible incubation period with high nucleation density for the growth on the TiN and TiO_2 substrates. On the contrary, GST films on SiO_2 and Al_2O_3 substrates showed island-type growth behavior and appears to have a rather long incubation period. Among the three components, Sb showed the best nucleation behavior on the various substrates. Ge and Te incorporation on the pre-deposited Sb layer was more efficient so that uniform and stoichiometric GST films were obtained when Sb layer was used as the nucleation layer. However, the films growth still showed the strongly substrate surface dependent behaviors. Step coverage on 3-dimensional hole structure was also investigated.

14:45 Oral

Plasma enhanced atomic layer deposition of titanium oxide passivation layers on polymer substrates

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Organic light-emitting diodes (OLED) have been intensively studied, but the organic and cathode materials of OLED devices are easily degraded by water vapor and high deposition temperature. It is widely reported that the upper limit requirement for a water vapor transmission rate (WVTR) for an OLED is 1×10^{-6} g/m²·day. In this work, we demonstrate the ability to form a passivation film that obtains a good titanium dioxide WVTR value. Titanium oxide thin films was conducted using Tetrakis dimethylamino titanium (TDMATi) and an oxygen plasma on a polyethersulfon (PES) substrate at a deposition temperature of 90 °C by plasma enhanced atomic layer deposition (PEALD). In

order to investigate structural behaviors, morphology and chemical composition of deposited films, we used X-ray photoelectron spectroscopy (XPS), Rutherford backscattering spectrometry (RBS) and atomic force microscopy (AFM). WVTR measurements on titanium oxide films were performed using a MOCON Corporation system. The titanium oxide film thicknesses with number of cycles were measured using cross sectional transmission electron microscopy (TEM) images and ellipsometer. The effects of the induced plasma power on passivation properties were investigated according to film thickness. The growth rate of the titanium oxide film was $0.8 \text{ \AA/cycle}$, and the water vapor transition rate (WTVR) for a 80 nm titanium oxide film was $0.023 \text{ g/m}^2 \text{ day}$.

15:00

Oral

Molecular organic - inorganic hybrid materials by atomic layer deposition

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The atomic layer deposition (ALD) technique has been used to deposit thin films of molecular type organic- inorganic hybrid materials. The growth has been exemplified by construction of thin films of Alq_3 , Znq_3 and Tiq_4 ($q = 8\text{-hydroxyquinoline}$), as well as growth of aluminium benzoate. Traditionally the ALD technique has been used for deposition of materials that form three dimensional networks. However, we have now also demonstrated growth of molecular type of compounds by the ALD technique. The Alq_3 and Znq_2 material are potential light emitting materials in OLED structures.

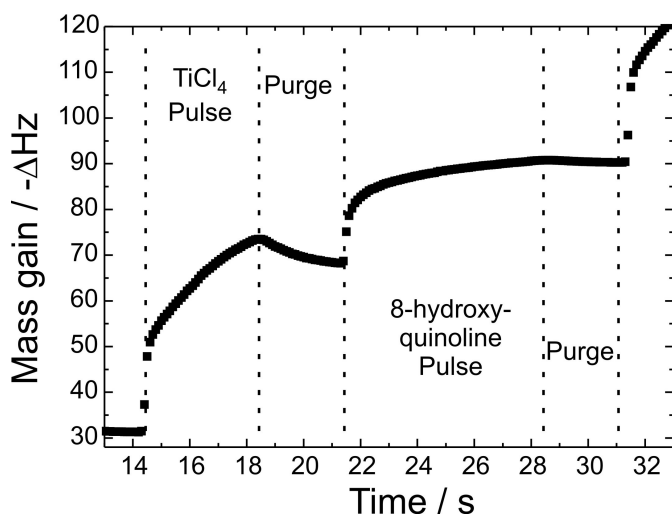


Fig. 1. In situ measurement of mass change by quartz crystal microbalance during deposition of titanium tetra 8-hydroxyquinoline at a reactor temperature of 85°C .

15:15

Oral

Al-Oxynitride interfacial layer for PrO_x on SiC and Si

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PrO_x is one of the candidates for both the realizing of further shrinking of equivalent gate oxide thickness (EOT) in microelectronic devices and electric field scaling at the interface between semiconductor and insulator in high power applications. However, the chemical reactivity of the PrO_x/SiC and PrO_x/Si interfaces causes a destructive interaction yielding silicate and graphite formation after direct deposition of PrO_x onto SiC and Si, respectively. This leads to high leakage current values as well as to a limitation of the reduction of the EOT due to an interfacial layer with small permittivity values. Therefore we introduced an additional chemically inert layer. In previous studies we reported already on spectroscopic investigations of Al-oxynitride (AlON), and we recognized a stable AlON/3C-SiC interface even for annealing steps up to 900°C .

In this contribution we will focus on the results of electrical characterizations of $\text{PrO}_x/\text{AlON}/\text{Si}$ stacks. We find a strong improvement in the leakage current by several orders of magnitude down to values of 10^{-7} A/cm^2 at an EOT of 4nm and interface state densities of mean values of $5 \cdot 10^{11} \text{ eVcm}^{-2}$. We will also show comparisons of spectroscopic (XPS) investigations of thickness dependent PrO_x series with and without AlON buffer layer, and will further discuss, if this buffer layer can improve the above mentioned EOT limitation. We also report on our ongoing electrical characterization of such stacks on SiC substrates.

Coffee break

Tuesday afternoon, 18 September, 15:30

Parallel Session 5

Tuesday afternoon, 18 September, 15:50

15:50

Invited

Wide band-gap II-VI semiconductors for optoelectronic applications

Elżbieta Guzewicz¹, Marek Godlewski^{1,2}, Krzysztof Kopalko¹, Andrzej Szczerbakow¹, Iwona A. Kowalik¹, Aleksandra M. Wojcik¹, S. Yatsunenko¹, Wojciech Paszkowicz¹, Elżbieta Lusakowska¹

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II-VI semiconductors are prospective candidates for optoelectronic applications like white light emitters, elements of solar cells, transparent conductive layers or buffer thin films.

We discuss properties of selected II-VI thin films obtained by the method of Atomic Layer Deposition (ALD) for above mentioned applications.

We show results for monocrystalline films of sphalerite-type ZnSe grown on GaAs(100) substrates from elemental Zn and Se precursors. Due to color mixing of band edge and deep defect-related emissions these layers emit intensive white light. Depth-profiling Cathodoluminescence (CL) investigations indicate that green and red emissions come from disordered regions of the films, close to the ZnSe/GaAs interface. The chromaticity parameters (color perception) of light emitted from ZnSe/GaAs layers can be tuned by either regulating the appropriate accelerating voltage of electrons or current density of primary electrons in CL investigations. These make ZnSe thin films good candidates as white light sources.

Zinc oxide thin films may be used for solar cells as transparent electrodes as well as for blue/UV light emitting devices. We achieved extra-low temperature growth of this material using diethylzinc (DEZn), which is a volatile and reactive zinc precursor. We obtained polycrystalline ZnO layers of good quality as confirmed by X-ray diffraction. Photoluminescence (PL) of these films show strong emission of excitonic origin, which is observed even at room temperature.

Wurzite- and sphalerite-type ALD CdS films show crystalline structure, flat surfaces and good optical properties. Room temperature CL spectra are dominated by strong edge emission at 2.4 eV. Low temperature PL of wurzite-type CdS show a strong edge luminescence with decay times similar to those typically found for the best quality bulk CdS crystal.

The work was partly financed by grant number 1 P03B 015 29 of Ministry of Education and Science, Poland

16:20

Oral

ALD metal fluoride thin films for UV optics

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Optical components and their coatings are being forced to operate at wavelengths in the deep-ultraviolet (DUV) region because the push for higher density semiconductor devices requires smaller circuit features. These can only be obtained by reducing the wavelength in the photolithographic process used to create the circuits. One of the most anticipated applications is antireflection coating for ArF lithography which is said to be the next main process for the fabrication of semiconductors. Highly damage-resistant dielectric optical coatings are also essential for high output UV lasers. Metal fluorides like CaF_2 , LaF_3 and MgF_2 are interesting materials due to their good light transparency in UV range. The problem in depositing fluorides by ALD has been lack of a good fluoride source. The previously used fluoride precursor HF etches silicates and also otherwise is not ideal for ALD.

This paper introduces an Atomic Layer Deposition (ALD) process for preparation of selected fluoride thin films using TiF_4 as a fluoride precursor at temperature range of 225–450 °C. $\text{Mg}(\text{thd})_2$, $\text{Ca}(\text{thd})_2$ and $\text{La}(\text{thd})_3$ (thd = 2,2,6,6-tetramethyl-3,5-heptanedionato = $\text{C}_{11}\text{H}_{19}\text{O}_2$) were used as metal precursors. The crystallinity, densities, and roughness of the films were measured by x-ray diffraction and x-ray reflection. Thicknesses and refractive indices of the films were determined from reflection and transmission spectra obtained with a spectrophotometer in the wavelength range of 190–1100 nm. Film morphology was studied with scanning electron microscopy (SEM). Composition and impurity levels of the films were analyzed by time-of-flight elastic recoil detection analysis TOF-ERDA.

The films were polycrystalline and MgF_2 grew more columnar than LaF_3 and CaF_2 on silicon. Low impurity levels were obtained at 350–400 °C with good stoichiometry. Refractive indices of 1.34–1.42 for MgF_2 , 1.43 for CaF_2 and 1.57–1.61 for LaF_3 films were obtained.

16:35

Oral

Characterisation and properties of low temperature ALD TiO_2 films

Gerry Triani¹, Peter J. Evans¹, Jonathan Campbell², Bruno Latella¹, Armand Atanacio¹, Darren Attard¹, Robert P. Burford²

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The atomic layer deposition of films under conditions outside the ALD window involves additional processes that have to be accounted for in order to achieve good quality films. [1] In the present study, the growth of ALD TiO_2 films on silicon and polycarbonate in the temperature range 80 – 120°C has been investigated in detail for two combinations of pulsing times. Furthermore, both substrate materials were exposed to a low-pressure water plasma to investigate the effect of pre-treatment on the deposited films. A suite of characterisation techniques including XRD, SIMS, RBS, AFM, XTEM and spectroscopic ellipsometry was used to probe the physical and chemical properties of the films. In addition, microtensile testing of the films enable the interface energy and toughness to be determined. These measurements showed water plasma treatment prior to deposition increased the interface energy and interface toughness from 11 to 26 Jm⁻² and 1.24 to 1.96 MPa.m^{1/2} respectively. The contact angle of the TiO_2 films was measured to assess their wettability. These tests involved subjecting the films to single and cumulative exposures of UV radiation followed by measurement of the contact angle. For an 85 nm film on polycarbonate, the contact angle decreased from 60° for the as-deposited surface to 10° following a 15 minute exposure. A 25 nm film yielded a similar decrease though this was only achieved after a 50 minute exposure. I.M. Ritala and M. Leskelä, in Handbook of Thin Film Materials, Volume 1: Deposition and Processing of Thin Films, H.S. Halwa (ed.), Chap. 2, Academic, NY, 2002.

16:50

Oral

Atomic-layer-deposition and local ferroelectric properties of PbTiO_3 and $\text{Pb}(\text{Zr,Ti})\text{O}_3$ thin films

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Atomic layer deposition of ferroelectric PbTiO_3 (PTO), $\text{Pb}(\text{Zr,Ti})\text{O}_3$ (PZT) thin films and its component oxide films were attempted using the $\text{Pb}(\text{DMAMP})_2$, $\text{Ti}(\text{Oi-Pr})_4$ or $\text{Ti}(\text{Ot-Bu})_4$, and $\text{Zr}(\text{Ot-Bu})_2(\text{DMAMP})_2$ as the Pb-, Ti- and Zr-precursors, respectively, and H_2O as oxidant at a wafer temperature of 200 °C on $\text{Ir}/\text{IrO}_2/\text{SiO}_2/\text{Si}$ substrate. The ALD characteristics of the component oxides, PbO , TiO_2 , and ZrO_2 films were investigated in detail for determining the ALD conditions of PTO and PZT thin films. The stoichiometric PTO thin films were grown by a proper control of the cycle ratio of the PbO and TiO_2 cycles. The increase of the PTO film growth rate due to the catalytic effect was observed compared to its component oxide films for both processes using $\text{Ti}(\text{Oi-Pr})_4$ or $\text{Ti}(\text{Ot-Bu})_4$. Two different post-deposition annealing method, i.e. slow and fast furnace annealing at 600 °C, were used to crystallize the as-grown amorphous PTO films. The ferroelectric switching behaviors of the crystallized films were

confirmed. The surface morphologies, local ferroelectric properties and leakage current behaviors of PbTiO_3 thin films, which were annealed using fast and slow furnace annealing methods, were investigated by scanning electron microscopy (SEM), piezo-force microscopy (PFM) and conductive atomic-force microscopy (CAFM). The higher growth rate of PTO film grown using $\text{Ti}(\text{O}i\text{-Pr})_4$ due to the higher growth rate of TiO_2 film compared to the case using $\text{Ti}(\text{O}t\text{-Bu})_4$ resulted in less dense PTO film with the higher density of micro-pores inside as well as surface of film after the fast furnace annealing. Slow furnace annealing improved the surface morphology of PTO film and reduced the micro-pore density. In case of PTO film grown using $\text{Ti}(\text{O}i\text{-Pr})_4$, slow-furnace annealed film showed lower local-leakage current compared to fast-furnace annealed film, whereas PTO films grown using $\text{Ti}(\text{O}t\text{-Bu})_4$ showed the opposite results. The local ferroelectric polarization switching was observed under the DC bias of 5V.

Wednesday, 19 September

Plenary Session

Wednesday morning, 19 September, 9:30

Coffee break

Wednesday morning, 19 September, 10:30

Parallel Session 6

Wednesday morning, 19 September, 11:00

11:00

Invited

Low temperature ZnMnO by ALD

Aleksandra M. Wojcik¹, Marek Godlewski^{1,2}, Krzysztof Kopalko¹, Elżbieta Guzewicz¹, Rafał Jakiela¹, Michał Kiećana¹, Roman Minikayev¹, Wojciech Paszkowicz¹, Matti Putkonen^{3,4}, Lauri Niinistö⁴

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Spintronics is one of the most promising directions of nano-electronics. To realize predicted devices we should master technology of ferromagnetic semiconductors, which is necessary step for high efficiency of injection of spin polarized carriers. Ferromagnetism (FM) above room temperature has been predicted for heavily doped p-type ZnMnO [1], which is thus considered as a promising material for spintronics. It has been found, however, that the often observed ferromagnetism of ZnMnO relates not to alloy properties, but to the presence of Mn precipitates in ZnMnO and also to the formation of foreign phase inclusions of various Mn_xO_y oxides. These oxides show strong magnetic ordering as anti-, ferro-, and ferrimagnetic oxides are formed [2].

We used Atomic Layer Deposition (ALD) to grow ZnMnO films. Very low growth temperatures, from 160°C up to 400°C, were crucial to block formation of foreign phases. We used two types of Mn precursors: $\text{Mn}(\text{thd})_3$ and $\text{Mn}(\text{acac})_3$, and two types of Zn precursors, viz. zinc acetate ($\text{Zn}(\text{OAc})_2$) and diethyl zinc (DEZn). These films exhibit controlled magnetic properties. Properties of obtained materials will be discussed based on structural and magnetic investigations.

[1] T. Dietl, H. Ohno, and F. Matsukura, *Science* **287**, 1019 (2000)

[2] C. Liu, F. Yun, H. Mokoc, J. Mater. Sci.: Mater. Electron. **16**, 555 (2005).

Microchemistry F-120 ALE reactor was financed by Foundation for the Polish Science within the Sezam program.

11:30

Invited

Ferromagnetic Nanostructures by Atomic Layer Deposition: From Thin Films towards Core-shell Nanotubes

Kornelius Nielsch^{1,2}, Julien Bachmann¹, Mihaela Daub¹, Jing Jing¹, Mato Knez¹, Ulrich Goesele¹, Sven Barth³, Sanjay Mathur³, Juan Escrig⁴, Dora Altbir⁴

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A broad range of applications of magnetic nanostructures have been developed in recent years. Magnetic data storage, microelectronics, or biomedical uses such as cell separation or biosensing present a special interest among these applications. Atomic layer deposition (ALD) is a very suitable method for the conformal deposition of magnetic films in pore structures of high aspect ratio, while offering the precise tuning of the layer thickness and high uniformity.

For the ALD of Co and Ni, a metal oxide film has been grown initially by the reaction of CoCp_2 (cobaltocene) or NiCp_2 (nickelocene) with ozone. Subsequently, the metal oxide film has been reduced in hydrogen atmosphere after the ALD process and converted to the metallic ferromagnetic phase with low-degree of surface roughness. In a similar manner, Fe_3O_4 films have been grown by the ALD deposition of Fe_2O_3 films based on the reaction of water and iron(III) *tert*-butoxide ($\text{Fe}_2(\text{O}t\text{Bu})_6$) and followed by a hydrogen reduction. On the other hand, we have observed that direct reaction of a metal-organic precursor and hydrogen during the ALD cycle always yields very granular films with ill-defined magnetic properties and low deposition rates ($<0.1 \text{ Å/cycle}$).

By conformal coating of self-ordered Al_2O_3 membranes, arrays of magnetic nanotubes with diameters down to 20 nm and wall thicknesses of $<3 \text{ nm}$ have been achieved. The magnetic properties of the nanotube arrays as a function of wall thickness and tube diameter have been studied by using SQUID magnetometry and compared with magnetic simulations. As an outlook we will discuss the properties of magnetic multi-layer nanotubes (core-shell) and the application of conformal magnetic coatings of other template systems and 3D nanostructures.

12:00

Oral

High performance Transparent Thin Film Transistors from N doped atomic layer deposition ZnO

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Recently, ZnO based thin film transistor is attracting great interests for transparent and flexible displays. So far, ZnO active layers have been mostly deposited by physical vapor deposition such as sputtering or pulsed layer deposition. However, atomic layer deposition (ALD)

can be a good deposition technique for this purpose since it can produce films with good uniformity over large area at relatively low growth temperature. Additionally, other components of TFT such as gate insulator and source/drain/gate can be also deposited by ALD. However, the ALD ZnO film from diethyl Zn for precursor and water or ozone for reactant, respectively, has been reported to have intrinsically high carrier concentration ($> 10^{17} / \text{cm}^3$). This is a significant drawback for the application to TFT channel material, since large off current is expected. In this research, we developed an efficient way to control the carrier concentration in the ALD ZnO film. By using NH_4OH as a source for reactants, nitrogen doping has been achieved *in situ* during ZnO ALD. The nitrogen concentrations of the films were controlled by changing the ammonia concentrations in NH_4OH solution. By nitrogen doping, the microstructures of the ZnO films were changed to amorphous at high nitrogen concentration and, more importantly, the carrier concentrations were reduced to $< 10^{15} / \text{cm}^3$. Inverted staggered type TFTs were fabricated with N:ZnO as a channel at different ammonia concentrations from 0 to 29 % and ALD Al_2O_3 as a gate insulator at low process temperature below 150°C . By this process, very good electrical properties were achieved; $m_{\text{sat}} = 7 \text{ cm}^2/\text{Vs}$ and $I_{\text{off}} < 3 \times 10^{-12} \text{ A}$, $I_{\text{on}}/I_{\text{off}} > 10^8$. Also, the hysteresis and subthreshold swing of the TFTs were low, about 0.5 V and 0.7 V/dec, respectively.

12:15

Oral

Atomic layer deposition as a tool for tailoring adhesion properties of interfaces.

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Adhesion properties of inorganic-biological and inorganic-inorganic interfaces play an important role in modern technology applications. ALD is a unique method due to its controllability even at nanometer scale level. We will present results from three different material combinations where surface properties were altered by ALD.

Ca-P-O mimicking hydroxyapatite ($\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$) were deposited onto silicon, glass or sapphire substrates. The phosphorus content was controlled by changing the pulsing ratio between $\text{Ca}(\text{thd})_2/\text{O}_3$ and $(\text{CH}_3\text{O})_3\text{PO}/\text{H}_2\text{O}$. Ca-P-O films were crystallized at $500\text{--}800^\circ\text{C}$ by RTA treatment in a moist N_2 atmosphere. According to the bioactivity test carried out by mouse pre-osteoblast MC-3T3-cells it was observed that stoichiometric and crystallized films enhanced the proliferation and adhesion of cells at the surface.

$\text{TiCl}_4/\text{H}_2\text{O}$ and $\text{ZrCl}_4/\text{H}_2\text{O}$ processes were utilized for the deposition of TiO_2 and ZrO_2 films onto titanium substrates for tailoring metallo-

ceramic interface layers of dental applications. After thin film deposition, commercial dental porcelain (DucerinPlus) was applied and fired at 780°C . Three-point bending tests were executed according to DIN 13927. It was observed that a thin oxide layer restrains the reactions between silicon based ceramics and metallic titanium to inhibit formation of a weak silicide layer.

TiO_2 and Al_2O_3 thin films were used for improving the adhesion between sputtered Ti or TiC and polymeric PMMA or PC substrates. Tetrakisdimethylamidotitanium (TDMAT) and TMA were used as metal precursors and ozone as an oxygen source. As the ALD film thickness was increased there was a significant effect on the adhesion properties. The most prominent increase in adhesion was obtained by a 30-50 nm thick layer between PMMA and TiC and with optimal film thickness the adhesion force exceeded the cohesion force of the polymeric substrate and led to disruption deep within the bulk of the polymer, not between the substrate and coating.

Lunch break

Wednesday afternoon, 19 September, 12:30

Parallel Session 7

Wednesday afternoon, 19 September, 14:00

14:00

Invited

Remote plasma ALD of oxides and nitrides: fundamentals and applications

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The extension of the atomic layer deposition (ALD) technique with plasma processes (i.e., plasma-assisted ALD) is actively being researched as it can provide several potential advantages over thermal ALD for selected applications such as an enhanced growth rate, improved material properties, and lower deposition temperature (down to room temperature). In this contribution, an overview is presented of the deposition of several oxides (Al_2O_3 , HfO_2 , TiO_2) and nitrides (TiN , TaN) by plasma-assisted ALD. Results are presented as obtained by remote plasma ALD, a plasma configuration that distinguishes itself from direct plasma ALD and radical enhanced ALD by a high degree of reactivity combined with virtually no surface damage due to plasma ions. Remote plasma ALD processes based on halide and metalorganic precursors and plasmas of H_2 , N_2 , O_2 , NH_3 and combinations thereof will be described and the resulting material properties will be presented for wide substrate temperature ranges. The versatility of the plasma-assisted ALD process will be illustrated by several applications ranging from the semiconductor industry (capacitor stacks) to emerging applications in the field of 3D-integration in microelectronics (Cu diffusion barriers), photovoltaics (Si surface passivation), energy storage (Li diffusion barriers) and flexible electronics (ultrahigh moisture barrier coatings). Generic insight into the plasma-assisted ALD surface reactions will be presented from mechanistic studies carried out by a variety of *in situ* techniques: spectroscopic ellipsometry (film thickness, optical properties and crystal phases), transmission infrared spectroscopy (surface species and reaction products), mass spectrometry (reaction products), quartz crystal microbalance measurements (surface mass uptake), and optical emission spectroscopy (plasma emission and reaction products).

14:30

Invited

Dielectric and electrode thin films for stack-cell structured DRAM capacitors with sub 50-nm design rules

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Dynamic random access memory (DRAM) is used as the main memory of every modern computer, due to its high density, high speed and efficient memory function. For successful operation of DRAM, a large cell capacitance (~ 25 fF) and low leakage current (10^{-7} A/cm² or 1 fA/cell) are required. In a traditional Si-based capacitor, the target cell capacitance has been achieved by increasing the surface area of the capacitor. More recently, innovations have been made in the component materials. A metal electrode, TiN or Ru, and a dielectric material with a higher- k value (k is the relative dielectric constant) than that of the SiO₂/Si₃N₄ layer ($k \sim 6 - 7$), such as HfO₂ ($k \sim 25$) and ZrO₂ ($k \sim 40$), are being explored in giga-bit scale DRAMs. The minimum achievable t_{ox} is ~ 0.7 nm for HfO₂ and ZrO₂ which are currently being used in the DRAM industry. However, the technology road map for memory devices states that t_{ox} less than 0.5 nm is necessary for the DRAMs with a design rule of < 40 nm. Perovskite-based dielectric films such as SrTiO₃ and (Ba,Sr)TiO₃ were reported to exhibit k values of several hundreds. However, growth of these films is extremely difficult with the atomic-layer-deposition (ALD) which is a method of choice for the growth of the dielectric films in microelectronic devices. In this presentation, the authors report Al-doped TiO₂ (ATO) dielectric thin films grown by an ALD method for application to next-generation DRAM capacitors. TiO₂ in rutile phase exhibits a k value ~ 100 and therefore can be used for DRAMs with a design rule of < 40 nm. However, the small energy gap of 3.1 eV implies that the control of leakage currents is not trivial. We achieve the specifications on the leakage currents, i.e., 10^{-7} A/cm² at 0.8V, through elaborate doping of TiO₂ films with Al atoms. In addition, it was found that this specific thin film material and process can meet all of the critical requirements.

15:00

Oral

A study for interface of ruthenium deposited by rf-direct plasma enhanced atomic layer deposition

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ALD ruthenium thin film using the metal electrode for trench DRAM capacitor has been researched because the film has not only excellent uniformity also high work function energy of deposited films. Although ALD ruthenium has the good properties, growth of ruthenium has some problems. Especially, in the cause of limitation of ALD ruthenium initial growth with incubation time, thicknesses of deposited ruthenium films showed no linear relation with cyclic numbers. This type of problem is becoming a significant subject in ultra-thin films for metal electrode or gate oxide.

In this work, we examined the correlation of initial growth with interface of deposited ruthenium films. Ruthenium films were prepared by Ru(EtCp)₂ as a ruthenium precursor gas and ammonia as a reactant gas alternately. Ruthenium films were deposited as various source precursor injection in order to be unlike adsorption of ruthenium precursor onto substrate. This study deals with composition and structural

behaviors of interface between ruthenium and pure silicon substrate. To investigate surface/interface physical property and impurity of deposited films, EDX, HRTEM, AFM, XPS, Rs measurement and RBS were used. Two deposited ruthenium films were compared including differential initial ruthenium source injection replaced commercial 1 with 10 second for investigation of initial ALD growth. Figure 1 shows HRTEM images of ruthenium films with the above process and the ruthenium films were deposited by ALD cyclic number of totally 100.

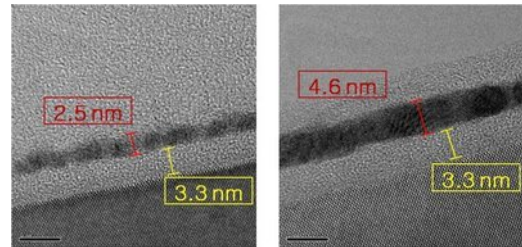


Figure 1 HRTEM images of ALD ruthenium films with initial injection source pulse cycle (left) including 25 and (right) including 50

15:15

Oral

Thin NiO films grown by atomic layer deposition using cyclopentadienyl-type of precursors and ozone

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We discuss the effects of growth temperature (T_g) on the structural and chemical properties of thin NiO films deposited by atomic layer deposition (ALD). Thin NiO films were grown on Si(100) using Ni(Cp)₂ (Cp=cyclopentadienyl, C₅H₅) or Ni(EtCp)₂ [EtCp=ethylcyclopentadienyl, (C₂H₅)C₅H₄] and ozone as precursors in the 150-300 °C temperature range. Neither Ni(Cp)₂ nor Ni(EtCp)₂ combined with water as oxygen source generates any film. The prepared films were characterized using X-ray reflectivity (XRR), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), and time of flight-secondary ion mass spectroscopy (ToF-SIMS). The behavior of films deposited using Ni(Cp)₂ and Ni(EtCp)₂ has been compared and discussed. NiO films with good composition are obtained at $T_g = 250$ °C or above. At a fixed T_g , the growth rate for NiO films deposited using Ni(Cp)₂ is higher than for those deposited using Ni(EtCp)₂. The electronic density of NiO films deposited at 300 °C is close to the one of bulk NiO. According to XRD and FTIR, films deposited at $T_g \geq 200$ °C have a cubic polycrystalline structure. Impurities in NiO films decrease with increasing T_g , as detected by ToF-SIMS. Based on the structural and chemical properties, we selected Ni(Cp)₂ to grow NiO films on metal substrates. Metal-NiO-metal devices were fabricated to explore the resistive switching properties of this material.

Coffee break

Wednesday afternoon, 19 September, 15:30

Joint Poster Session (Part II)

Poster Award Ceremony

Wednesday afternoon, 19 September, 15:50

Thursday, 20 September

Parallel Session 8

Thursday morning, 20 September, 9:00

9:00 Invited

Template-based synthesis of single- and multi-layered metal oxide nanotubes using ALD

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A very interesting field within the world of nanoscience is the nanotube area. One route to obtain well-ordered arrays of nanotubes is to use a template and transfer the pattern to a desired material. A suitable template for this method is anodic aluminium oxide (AAO), which can be electrochemically grown and exhibits a high degree of order over large areas. A problem is that few deposition techniques are able to conformally cover the pore-walls of the AAO. However, ALD is a very well suited technique for the template deposition method.

In the present paper, different metal oxide nanotubes were manufactured by ALD using AAO templates, both commercial and in-house made. An overview of the obtained results will be presented for synthesis of single-layered Nb₂O₅, Fe₂O₃ and TiO₂ nanotubes. In addition, some preliminary results for fabrication of multi-layered TiO₂/Fe₂O₃ nanotubes will be briefly mentioned. The precursor combinations used were NbI₅-O₂, Fe(Cp)₂-O₂ and TiI₄-H₂O, with pulse lengths of 30 s for both precursors and purging pulses. These large values for the pulse lengths are necessary to reach saturation of the precursor species on the surface of the pore walls and also to suppress any possible gas phase reactions in the pores.

For all metal oxides, well-ordered and free-standing nanotubes with aspect ratios up to 35 could be fabricated in the in-house made AAO templates. In commercial templates, aspect ratios of 200 was achieved. From SEM and TEM studies it could be seen that the pore-walls of the AAO were conformally covered, with the same oxide thickness regardless of the depth. This indicates that the duration of the pulses were sufficient to saturate the entire surface of the pore walls.

9:30 Invited

(Bio)organic-Inorganic Hybrid Nanostructures by ALD

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In nanotechnology biological systems play an increasingly important role, since some of the naturally occurring biological macromolecules show perfect order on the nanoscale.

The perfection of the natural organization of molecules is, in particular for the nanotechnology, often a target for imitation, but can almost never be obtained in a similar quality. An alternative way to obtain nano- or microstructures in close similarity to natural ones is their structural replication from inorganic materials or the modification of the chemical or physical properties of such systems by attachment of inorganic materials. Some of the methods applied recently rely on wet-

chemistry which frequently leads to non-uniformity or bad quality of the attached materials.

The ALD is one method-of-choice for performing such replication and modification experiments, since it offers the unique possibility to cover biological structures with inorganic films as thin as few Angstroms or nanometers.

In this way a number of novel nanostructures with interesting properties can be synthesized, however, limited with the deposition processes which can be performed at temperatures below the stability limit of the biological or organic molecular units.

10:00 Oral

Electrochemical Atomic Layer Deposition

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Electrochemical ALD, developed in the Authors group, has been used to form a number of elemental deposits, including Cu, Pt, and Ag, by this group and others. ALD is a methodology where surface limited reactions are used in a cycle to form nanofilms of compounds or elements, one atomic layer at a time. Underpotential deposition (UPD) is generally used for surface limited reactions in the electrochemical form of ALD. However, in the formation of a pure element, it might be assumed that UPD could be used. However, use UPD of an element on itself is by definition impossible. The definition of UPD is that an atomic layer of one element can be formed on a second element, due to the formation of a stable surface compound or alloy, at a potential prior to, under, that used to deposit the element on itself. Because of this issue, surface limited redox replacement (SLRR) has been used to form elemental deposits. The SLRR involves UPD to form an atomic layer of a sacrificial element on the desired element. This atomic layer is then exchanged for the desired element by redox replacement of the sacrificial atomic layer in a solution of a precursor of the desired element. This process can then be repeated to form nanofilms of the desired element of a thickness dependent on the number of replacement cycles performed. Nanofilm growth of Cu, Ru, and Pt will be discussed, as a function of the deposition conditions: the sacrificial metal, the UPD potential used, and the coverage of the sacrificial metal atomic layer. Film growth has been characterized using in-situ STM, LEED and Auger, as well as EQCM and XRD. Deposits of Cu appear to grow in a layer by layer format. However, some problems have been encountered with the growth of Pt films, in that they appear to roughen quickly under some conditions.

10:15 Oral

Atomic Layer Deposition of Yttria Doped Zirconia as Solid Oxide Fuel Cell Electrolyte

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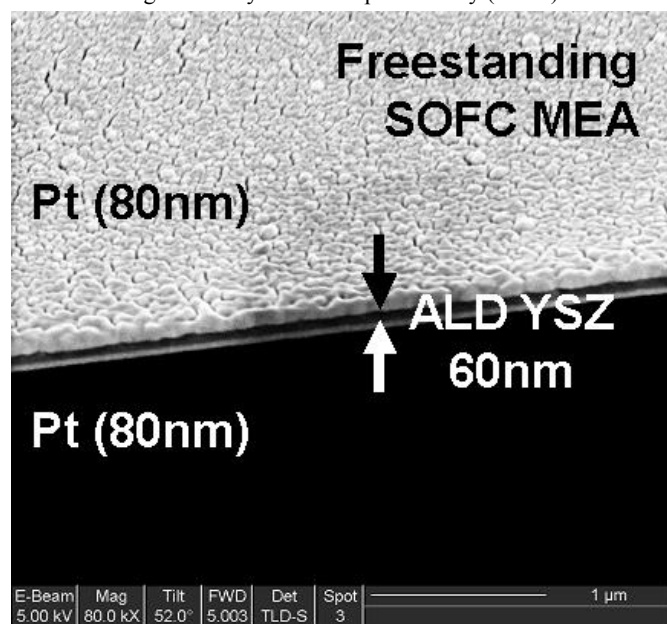
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Abstract

Yttria-stabilized zirconia (YSZ) membranes have been widely used as solid-state electrolytes for solid oxide fuel cells (SOFC) because of their high oxide ion conductivity and good chemical stability. Traditionally, high operating temperatures have been required for sufficient ionic conduction, in turn limiting applications of SOFCs due to high thermal stresses and accelerated system degradation. A number of

studies have been performed to enhance ionic conduction at reduced temperatures by lowering the thickness of the electrolyte to minimize Ohmic losses [1]. Production of thin YSZ has been demonstrated using various conventional thin film fabrication techniques such as radio frequency (RF) sputtering [2], pulsed laser deposition (PLD) [3] and chemical vapor deposition (CVD) [4].

In this work, we built thin film SOFCs with YSZ electrolytes with thickness of tens of nanometers by atomic layer deposition (ALD). Tetrakis-(dimethylamido) zirconium and tris-(methylcyclopentadienyl) yttrium were used as ALD precursors with distilled water as oxidant. X-ray photoelectron spectroscopy (XPS) compositional analysis was applied to confirm relationship between film composition and ALD precursor cycle ratios. Crystal and grain structures of ALD YSZ films grown on silicon nitride-coated silicon wafers were analyzed by X-ray diffraction (XRD) and atomic force microscopy (AFM). We evaluated our SOFC with ALD YSZ by measuring current-voltage (I-V) curves. In addition diffusion profiles of oxide ions in YSZ membrane are established using secondary ion mass spectrometry (SIMS).



References

- [1] de Souza, S.; Visco, S. J.; DeJonghe, L. C. *Solid State Ionics, Diffusion & Reactions* **1997**, 98, 57-61.
- [2] Huang, H.; Nakamura, M.; Su, P.; Fasching, R.; Saito, Y.; Prinz, F. B. *J. Electrochem. Soc.* **2007**, 154, b20-4.
- [3] Kokai, F.; Amano, K.; Ota, H.; Ochiai, Y.; Umemura, F. *J. Appl. Phys.* **1992**, 72, 699-704.
- [4] Chour, K. W.; Chen, J.; Xu, R. *Thin Solid Films* **1997**, 304, 106-112.

Coffee break

Thursday morning, 20 September, 10:30

Parallel Session 9

Thursday morning, 20 September, 11:00

11:00

Invited

Exploiting novel precursor chemistry for the growth of oxide thin films by Atomic Layer Deposition

Lauri Niinistö

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Traditionally, simple and volatile metal compounds, such as halides, alkoxides, and β -diketonates, have been used to grow oxide films by ALD.¹ However, these precursors exhibit a number of shortcomings such as low reactivity towards O sources as well as introducing Cl or C impurities into the films. To avoid these and other potential difficulties, it is necessary to explore and develop new precursor chemistry.

In recent years, cyclopentadienyl (Cp)-compounds and amido complexes have emerged as ALD precursors.² Among these, we recently reported novel Cp-compounds to grow HfO_2 at high temperatures of 350-500°C.³ Additionally, the ALD of Ga_2O_3 and Er_2O_3 using dialkyl-amido-based and amidinate-containing precursors, respectively, is discussed.^{4,5} Based on this, the growth of $\text{Er}_x\text{Ga}_{2-x}\text{O}_3$ is demonstrated from 2 systems, the first one using β -diketonates and O_3 while the 2nd one uses $\text{Er}(\text{CpMe})_3$, $\text{Ga}_2(\text{NMe}_2)_6$ and H_2O .⁶ Finally, we have an example of a dialkylamido-based dimer for oxidation state control in W oxide films, where the reaction between $\text{W}_2(\text{NMe}_2)_6$ and H_2O maintains W in its +3 state producing W_2O_3 .⁷

Ta_2O_5 and BaTiO_3 are important materials for microelectronics. Discovery of new ALD precursors could prove useful for these materials, especially for Ba, which lacks stable sources. The new pyrazolate-based precursor for Ta exhibits high thermal stability and produces films of low impurity levels, whereas the novel pyrazolyl-based Ba precursor shows promise towards developing a feasible ALD process for BaTiO_3 .^{8,9}

1. L. Niinistö *et al. Phys. Stat. Solidi a* 201 (2004) 1443.
2. M. Putkonen and L. Niinistö, *Top. Organomet. Chem.* 9 (2005) 125.
3. J. Niinistö *et al. Chem. Mater.* 19 (2007). In press.
4. C.L. Dezelah IV *et al. Chem. Mater.* 18 (2006) 471.
5. J. Päiväsäari *et al. J. Mater. Chem.* 15 (2005) 4224.
6. C.L. Dezelah IV *et al. Ibid.* 17 (2007) 1308.
7. C.L. Dezelah IV *et al. J. Am. Chem. Soc.* 128 (2006) 9638.
8. C.L. Dezelah IV *et al. Ibid.* (2007). Submitted.
9. C.L. Dezelah IV *et al.* To be published.

Olympus Presentation

Thursday morning, 20 September, 11:30

Closing

Thursday afternoon, 20 September, 12:00

Lunch break

Thursday afternoon, 20 September, 12:30

Posters

Monday, 17 September

Joint Poster Session (Part I)

Monday & Wednesday

Monday afternoon, 17 September, 15:50

15:50	Poster	C-1

Passivation of GaAs surface by using ALD technology

Markus Bosund, Abuduwayiti Aierken, Jouni Tiilikainen, Teppo Hakkarainen, Jaakko Sormunen, Harri Lipsanen

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The suitability of atomic-layer-deposited (ALD) Ti(Al)N GaAs surface passivation and protection has been investigated. Ti(Al)N-film were fabricated by using titanium tetrachloride (TiCl₄), trimethyl aluminum (TMA) as a reducing agent and ammonia (NH₃) at 275 °C. The

passivation effect was studied by using GaAs/InGaAs near surface quantum well (NSQW) structures, where photoluminescence (PL) intensity measurement was used as an indicator for the effectivity of passivation. Passivation reduces surface states which can be recombination center for the carriers. Radiative recombination decreases due to tunneling of carriers from quantum well to surface when surface states in energy bandgap is increased. The number of surface states can be reduced by depositing appropriate material on the surface. In this work Ti(Al)N was deposited on NSQW resulting eight times higher PL intensity compared to PL intensity of uncovered reference sample. Ti(Al)N passivated NSQW protected by aluminum oxide (Al₂O₃) and titanium oxide (TiO₂) layers was also studied. PL intensity diminished when oxide layers were used but protective effect was still observable when compared to NSQW samples which were covered only with Al₂O₃ or TiO₂. The temperature

stability of Ti(Al)N-covered NSQW was tested. It came out that Ti(Al)N-covered NSQW doesn't luminesce after 400 °C annealing. Thermal properties of the film were studied by using X-ray reflectivity. Titanium nitride layers manufactured on Si (100) substrates. XRR-measurement was made before and after 400 °C annealing and preliminary test showed that the thermal treatment didn't influence the film significantly. This study shows that

low temperature ALD Ti(Al)N is promising material for GaAs surface passivation and protection when post deposition temperature is limited well below 400 °C.

15:50	Poster	C-2

Investigation of non-monotonous dependencies of the surface roughness on the thin film deposition parameters

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It is observed that the surface roughness varies non-monotonously with different deposition parameters. The aim of this work is to analyze and explain the non-monotonous dependencies of the surface roughness on the substrate temperature and on the deposition flux. For this purpose the kinetic model has been proposed. The proposed kinetic model is based on the rate equations and includes various steps (surface diffusion of adsorbed atoms, nucleation, growth and coalescence of the islands) of island film growth. Analysis of experimental results done by modeling explains non-monotonous dependencies of surface roughness on the substrate temperature and deposition flux which is resulted by size of the islands and diffusivity of adatoms.

Keywords: surface roughness; island films deposition; surface diffusion; kinetic modeling

15:50	Poster	C-3

Atomic layer deposition of HfO₂ thin films on In_{0.53}Ga_{0.47}As/InP substrates prepared using both aqueous and in-situ sulfur surface passivation methods

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The properties of high-k dielectric layers of materials such as HfO₂ on III-V surfaces are interest for the development of III-V MOSFETs since such devices will require high-k/metal gate stacks designed to minimize gate leakage. In a study of potentially high-mobility InGaAs-based structures, we have focused on the growth of HfO₂ overlayers on high indium content (53% In) lattice-matched In_{0.53}Ga_{0.47}As layers grown by MOVPE on InP substrates. In an attempt to address the problems associated with native oxides we have explored the use of both a wet chemical *ex-situ* pre-growth treatment and a novel *in-situ* surface treatment performed at the end of the InGaAs MOVPE growth cycle. The former *ex-situ* treatment involves the use of aqueous (NH₄)₂S solution while the latter *in-situ* treatment involves introducing H₂S to the environment of the MOVPE growth reactor following completion of the growth itself.

XPS analysis reveals that as compared to an untreated surface, both passivation approaches significantly inhibit the formation of native oxides on the InGaAs surface upon exposure to ambient conditions. The electrical characteristics of gate stacks prepared after ALD growth of HfO₂ from tetrakis dimethylamido hafnium (Hf(NCH₃)₂)₄ and water were evaluated using Pd/HfO₂/In_{0.53}Ga_{0.47}As/InP MOS structures. While both passivation methods yielded good results, the use of the *in-situ* H₂S passivation of the InGaAs at 50°C prior to HfO₂ deposition was found to be particularly effective in terms of the maximum accumulation capacitance, dielectric leakage and interface state densities. These results were achieved without post deposition annealing of the overall Pd/HfO₂/In_{0.53}Ga_{0.47}As/InP MOS structure. The results indicate that the interfacial Fermi-level is unpinned for both *in-situ* and *ex-situ* passivation approaches. We discuss these results in terms of possible models for the passivated interface and the subsequent mechanisms for the initial growth of the HfO₂ layer.

15:50 Poster C-4

Synthesis and Characterization of Multilayer Thin Films ZnO-TiO₂ on Patterned Surfaces by Atomic Layer Deposition

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ZnO is a key technological material for different applications: such as piezoelectric transducers, optical waveguides, surface acoustic wave devices, phosphors, transparent conductive oxides, chemical and gas sensors, spin functional devices, and UV-light emitters. ZnO also exhibits the most different nanostructures types (nanowires, nanobelts, nanobow, etc.). It is biocompatible.

Titanium dioxide (TiO₂) thin films have recently found several applications in optics, biomaterials and photocatalysis because of their appealing physical and chemical properties.

ZnO and TiO₂ thin films can be synthesized through various chemical and physical methods; among these Atomic Layer Deposition (ALD) is one of the most promising because it allows to obtain single atomic layers with excellent uniformity and conformality.

In this work we present an innovative method to coat nanopatterned surfaces with multilayers of ZnO and TiO₂ synthesized by means of ALD. The nanopatterned surfaces were obtained by nanosphere lithography on silicon substrates.

The structure and the microstructure of the thin films were analyzed through different characterization techniques such as X-Ray Reflectivity (XRR), X-Ray Diffraction (XRD and XRD²), microRaman Spectroscopy, Scanning Electron Microscopy (SEM) and Atomic Force Microscopy (AFM).

15:50 Poster C-5

Study of CIS based solar cells with a ALCVD ZnO/ZnO:Al as window layers

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Classical CIS thin films solar cells contains a thin film of CdS. To avoid its toxicity and make cadmium-free buffer layers solar cells, we tried to make some surface modification of CIS. The electrochemical approach was defined for wet treatments and ALD was chosen from dry interface engineering. Electrochemical modifications were already investigated successfully, and also ALD work led us to achieved the world record with vapor phase cadmium free processed CIS cells, with 16.4 % in 2003^[1].

In a second time, we would to complete the CIS cell with ZnO/ZnO:Al as window layer. The window layer has to respect two major parameters, the sheet resistivity and the optical transmittance. ALD ZnO films and doped ZnO films were optimized for CIS absorber devices. This process is well known method to prepare high quality multilayer films

of compound materials like II-VI semiconductors, and provided a good way for the fabrication of photovoltaic devices. This is a soft method with a good control of the thickness, which allows obtaining homogeneous films with a good covering. We obtained films with good homogeneity and good covering properties as shown by the SEM views. By varying the number of pulses of Trimethyl aluminium we have been able to change the concentration of aluminium into the films and improved the conductivity of the films we used for complete the CIS cells. The resistivity of the films can be tuned from about 10⁻² to about 2.10⁻³ W.cm. The films we obtained are highly transparent shown by transmission curves. This will allow us to complete devices.

Electrical performances of the cell were characterized by current-voltage measurements and Quantum efficiency (QE) characteristics. Complete cells with CdS and sputtered ZnO gives efficiencies of 12 - 13%. The first surface modified cadmium free results (basic treatment of CIS absorber) are lower (about 10%).

[1]: N. Naghavi, S. Spiering, M. Powalla, B. Canava, D. Lincot, Prog. Photovoltaics 11 (7), 2003, 437.

15:50 Poster C-6

SiO₂ and Si₃N₄ Films by Remote Plasma Enhanced Atomic Layer Deposition

Qi Fang, Chris Hodson

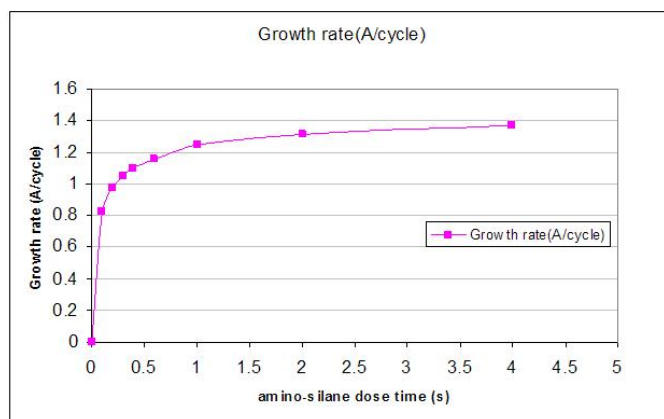
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Increasingly ultra thin film processing is required for MEMs, nanostructure devices such as bio-sensors, detector-arrays, quantum devices and plastic electronics. Although Atomic Layer Deposition (ALD) is capable of depositing many different materials, the more traditional oxides and nitrides of silicon are still of considerable interest due to their many applications and long established material properties. This contribution focuses on SiO₂ and Si₃N₄ thin films grown by remote plasma enhanced ALD at low temperatures. The SiO_x and SiN_x layers were prepared using various amino-silane sources, which will be discussed. The SiO_x layers were deposited using oxygen plasma as the oxidant and deposition rates of around 1.4 Å/cycle at a temperature of 300°C have been obtained, see figure 1. The effect of the processing parameters, such as deposition temperature, the dose time of the precursors, ratio of nitrogen and hydrogen (for SiN_x), plasma power and plasma time, on the deposition rate and optical and electrical properties of the SiO_x and SiN_x thin films are also discussed.

Spectroscopic Ellipsometer (SE), FTIR, AES, SEM, hydrofluoric acid etch rates and electrical measurement were used for characterisation of the physical, electrical, chemical and optical properties of the as-deposited SiO_x and SiN_x thin films. Effect of post-annealing on the ALD SiO_x and SiN_x films will be discussed as well.

Figure 1, growth rate of SiO_x using an aminosilane source and oxygen plasma at 300°C.



15:50 Poster C-7

Crystalline Bi₂O₃ nanofibers fabricated on sapphire substrates using a trimethylbismuth and oxygen mixture

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The bismuth oxide (Bi₂O₃) has attracted great attention due to their applications in several technological fields, such as oxide-ion conductors, piezo-optic materials, solar cells, gas sensors, and ceramic glass manufacturing. Additionally, Bi₂O₃ is a component of various important electroceramic oxides including Bi-Sr-Ca-Cu-O superconductors, and Sr-Bi-Ta-O/Sr-Bi-Nb-Ta-O ferroelectric oxides, which have applications in non-volatile computer memories. In the present work, we have synthesized the rod-like nanostructures of bismuth oxide (Bi₂O₃) on sapphire substrates by a reaction of a trimethylbismuth (TMBi) and oxygen (O₂) mixture. The produced nanorods had diameters in the range of 40-200 nm. The prepared nanorods had a crystalline Bi₂O₃ structure. The X-ray energy dispersive spectroscopy revealed that the materials contained elements of Bi and O. Photoluminescence measurements revealed that the Bi₂O₃ nanorods had visible emission bands.

15:50 Poster C-8

Atomic layer deposition of W-C-N diffusion barrier and its thermal stability for Cu interconnects

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For the past decade, an adequate diffusion barrier material has been an issue for the Cu interconnects. As a solution, atomic layer deposition (ALD) has been intensively studied for depositing transition metal-nitride materials such as TiN, TaN, W-N, W-C and W-C-N. Among the these, the resistivity of ALD-TaN is about 350-900 mΩ-□ and then, it performs not so good as a barrier against the Cu diffusion [1,2]. ALD-W_{1.5}N films are deposited with WF₆-NH₃-C₂H₄-SiH₄-NH₃ gas system and it has low resistivity (480 mΩ-□). But, the barrier performance is failed at 500 °C [3]. ALD-WN, WC, W-C-N films have been prepared by several researchers with metal organic precursors such as (t-BuN)₂(Me₂N)₂W and B(C₂H₅)₃ and the resistivity is in the range of 300-2000 mΩ-□ [4-6]. On the other hand, we had deposited W₂N films with PECVD and ALD using WF₆ and NH₃ gases [7,8]. Then, resistivity of the ALD-W-N is as high as 3000 mΩ-□ although the

PECVD-W-N is very low (200 mΩ-□). Therefore, in this work we have prepared the ALD-W-C-N film using WF₆-N₂-CH₄ gas system for low resistivity and good thermal stability. As a result, we have successfully deposited the ALD W-C-N films using only simple halid gases without any metal organic precursors and pulse plasma synchronized with the exposure cycles of N₂-CH₄. This pulse plasma assisted ALD (PPALD) method offers a simple and easy deposition process since metal organic precursors are not used and low resistivity (~300 mΩ-□) and superior thermal stability till 800 °C. The W-C-N films are prepared with the ALD process using WF₆-N₂-CH₄ gas system. Experimental results suggest that the H₂/N₂ plasma pretreatment and N₂ source gas instead of NH₃ gas lead to higher W and lower N concentrations. The deposition of the W-C-N film is governed by self-limiting ALD mechanism, easily controlling the film thickness.

15:50 Poster C-9

Using carboxylic acids to grow organic-inorganic hybrid materials by ALD

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Thin films of organic-inorganic hybrid materials where grown by ALD, using TMA (TMA = trimethylaluminium) and different types of carboxylic acids as precursors. In-situ analysis by quartz crystal microbalance was used to establish growth parameters for the different systems. Measurements of growth rate as function of temperature show that most systems do possess ALD-windows. X-ray diffraction studies of the as-deposited films indicate that most films are of an amorphous character, which is also supported by very low surface roughness as measured by atomic force microscopy. The growth rates of these hybrid materials are high when compared to traditional materials deposited by ALD, and growth rates as high as 4.32 nm/cycle has been found for the TMA and pimelic acid system by X-ray reflectivity, Fig. 1. As-deposited films were investigated by Fourier Transform Infrared Spectroscopy (FT-IR).

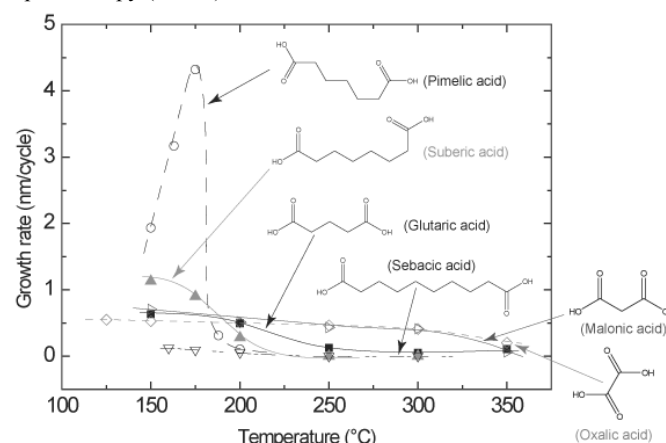


Fig.1: Growth rates of hybrid films deposited with linear carboxylic acids and TMA.

15:50

Poster

C-10

Characterization of ruthenium titanium nitride thin films deposited by rf-direct plasma atomic layer deposition

Myoung-Gyun Ko¹, Woong-Sun Kim¹, Tae-Sub Kim¹, You-Kee Lee², Young-Ki Lee², Jong-Wan Park¹

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As the silicon process migrates to small device geometries, new deposition process technology will be required to solve the problems obtained with high trench capacitor and ultra thin gate oxide. Attempts to deposit material with atomic level control, atomic layer deposition as a technique which can be available good film uniformity and excellent step coverage have been reported. Ruthenium is a rare element has low electrical resistivity, high chemical inertness, thermal stability and hardness. Especially, ALD of ruthenium thin films using the metal electrode for trench DRAM capacitor have been researched because the films have not only excellent uniformity also high work function energy of deposited films. Moreover, ALD titanium nitride film has thermal stability below 900 degree C. In this work, we have studied radio-frequency direct plasma enhanced atomic layer deposition (PEALD) growth of ruthenium titanium nitride (RuTiN) film. Ruthenium thin films were prepared by Ru(EtCp)₂ as a ruthenium precursor gas and ammonia as a reactant gas. Titanium nitride films growth was using Ti[N(CH₃)₂]₄ as a titanium precursor gas and ammonia as a reactant gas. RuTiN thin films were prepared with various ratio of ruthenium and titanium nitride process window. In order to investigate physical, electrical properties and impurity of deposited films HRXRD, HRTEM, XPS, Rs measurement and RBS were used. According to formations of agglomeration sites, electrical behavior changed with RTA treatment with ambient O₂. The thermal stability of RuTiN thin films deposited by ALD was affected by structural changes such as carbon contamination in the

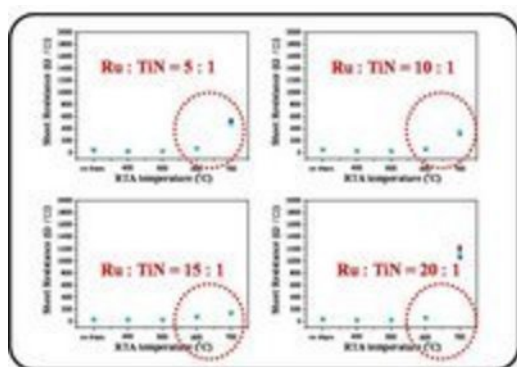


Fig. 1 Sheet resistance of RuTiN as various with RTA temperatures.

15:50

Poster

C-11

Investigating New Precursors for Silicon Dioxide

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Silicon dioxide is a widely used material in semiconductor industry. It is used for instance as a gate oxide and as a dielectric layer in dynam-

ic random access memories (DRAM). Recently, other possible applications have evolved that would require good step coverage and good SiO₂ filling capabilities, for example shallow trench isolation (STI). ALD has been proposed as a possible technique to meet these more stringent requirements. Unfortunately, the growth rate per cycle has traditionally been low for ALD SiO₂ processes making the processing economically non-viable. New precursors with higher reactivity enabling higher growth rate are therefore needed.

In this study, two silicon precursors were tested: tris(dimethylamino)silane (trisDMAS) and SAM.24 from Air Liquide. Ozone was used as the oxygen source. The depositions were done in ASM Pulsar[®]2000 ALCVD[™] reactor on 200 mm silicon wafers. The film thickness and uniformity were determined with a spectroscopic ellipsometer.

TrisDMAS was tested at 250 – 325 °C. Only very low growth rates were obtained when a 0.2-s trisDMAS pulse was used, the growth rate was increased from 0.07 to 0.09 Å/cycle with increasing deposition temperature. The film non-uniformity was also increased with increasing temperature from 5.4 to 10% (Uf 1σ). The highest growth rate was achieved with 0.5-s precursor pulses at 275 °C: 0.15 Å/cycle. The non-uniformity% was 4.8.

Depositions using SAM.24 were done at 250 and 300 °C. When 0.5-s precursor pulses were used, the growth rate and non-uniformity% were 0.15 Å/cycle and 4.8% at 250 °C, and 0.35 Å/cycle and 4.7% at 300 °C. A higher growth rate of 0.4 Å/cycle was achieved both at 250 and 300 °C using 1.0-s precursor pulses.

In this paper, the effect of deposition parameters on the growth of SiO₂ will be presented.

15:50

Poster

C-12

Molybdenum nitride nanotubes

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Since the discovery of carbon nanotubes in 1991¹, nanometer-scale 2-D structures have been studied increasingly and various materials, in nanotubular and in nanowire form, have been described. Nanotubular form of nitrides is a new area of inorganic nanostructures. Preparation of boron nitride nanotubes has been reported², but nanotubes of transition metal nitrides have gained less attention. Like other nitrides of transition metals, molybdenum nitride is a hard material. It is stable against mechanical and chemical stress, and it has a low coefficient of friction³. Molybdenum nitride has been widely applied in catalysis.

In this study, molybdenum nitride nanotubes were prepared by depositing nitride film on anodized aluminum oxide (AAO) template by atomic layer deposition. The precursors were bis(*tert*-butylimido)-bis(dimethylamido)molybdenum and ammonia. The AAO template was then etched away with sodium hydroxide solution. The effect of deposition parameters on film growth and the properties of the nanotubes were investigated.

The maximum intrusion depth of the molybdenum nitride film into the AAO pores, 20 μm, was achieved with 7-second precursor pulses. Three different architectures of molybdenum nitride nanotubes were isolated. Separated nanotubes were found when the template was etched in an ultrasonic bath, while bundling dominated when template etching was conducted without ultrasound (Fig. 1.). The nanotubes remained on the surface with the tips strongly intertwined when the nitride-coated AAO template was mounted onto a steel plate before etching.

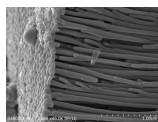


Fig. 1. Bundle of molybdenum nitride nanotubes.

References

1. Ijima, S. *Nature***1991**, 354, 56.
2. Bengu, E., Marks, L. D. *Phys. Rev. Lett.***2001**, 86, 2385.
3. Lengauer, W. *Transition Metal Carbides, Nitrides and Carbonitrides*; Riedel, R., Ed.; Handbook of Ceramic Hard Materials, Vol. 1; Wiley: Weinheim, 2000.

15:50 Poster C-13

A comparison of thermal and plasma enhanced ALD of TiO₂ and TiN

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Several authors have reported successful ALD growth of TiO₂ or TiN using TiCl₄ as a precursor and H₂O or N₂/H₂ as the reactant. However, the corrosive nature of the HCl reaction by-product and/or the presence of small amounts of chlorine in the film can be a drawback for certain applications. In this work, the metal organic precursor tetrakis-dimethyl-amido titanium (Ti(N(CH₃)₂)₄, usually abbreviated as TDMAT) was used for ALD growth of TiO₂ and TiN films. Oxygen, water vapor, nitrogen or ammonia were used as reactants. The reactant was flowing either directly into the ALD chamber (for thermal ALD) or through a home-made inductively coupled RF plasma source for plasma enhanced ALD (PE-ALD). The kinetics of both thermal and plasma enhanced ALD were studied systematically as a function of deposition temperature in the range of 50 °C to 350 °C. X-ray reflectivity (XRR), x-ray diffraction (XRD) and x-ray photo-electron spectroscopy (XPS) were used to characterize the thickness, crystallinity and chemical composition of the films after growth. Thermal ALD with H₂O and PE-ALD with O₂ or H₂O resulted in the deposition of TiO₂ films. The as deposited films were amorphous, but could be crystallized into the anatase phase after post-annealing at about 450 °C. Surprisingly, the growth rate of thermal ALD with TDMAT and H₂O was observed to increase for decreasing substrate temperatures, with a maximum rate of 0.14nm/cycle at a substrate temperature of 50 °C.

Plasma enhanced ALD with N₂ or NH₃ resulted in the growth of TiN films at a rate between 0.5 and 1 Angstrom per cycle. TiN films with a resistivity of about 1100 μOhm cm could be grown at temperatures between 150 and 300 °C

15:50 Poster C-14

Growth of 3D core-shell nanostructures by ALD

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Nanocrystals of Co₃O₄ have been made by a hydrothermal process utilizing microwaves, resulting in cube shaped crystals with dimensions in the range of 10 – 90 nm. The Co₃O₄ cubes have further been used as core material for 3D layered growth of Al₂O₃ and Fe₂O₃ by ALD, see Figure 1. These are the first steps towards multilayered core-shell materials by ALD. Such core-shell materials may show unique properties not found in their components alone^[1]. The challenge has been to construct means to cover the nanocubes equally on all sides in the ALD reactor. Different approaches include deposition on cubes placed on different flat substrates and the use of a powder cell, as well as direct synthesis on TEM grids.

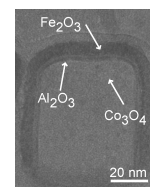


Figure 1: Multilayers of Fe₂O₃ and Al₂O₃ on a Co₃O₄ nanocube

[1] Lloyd, S. J.; Molina-Aldareguia, J. M. *Philos. Trans. R. Soc. Lond. A***2003**, 361, 2931-2949.

15:50 Poster C-15

Raman characterization of strain in epitaxial Cr₂O₃ films on sapphire

Ahti Niilisk¹, Martti Pärs¹, Ilmo Sildos¹, Rainer Pärna^{1,2}, Aivar Tarre¹, Arnold Rosental^{1,2}, Jaan Aarik¹

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By using atomic layer deposition from CrO₂Cl₂ and CH₃OH at 420 °C, chromia (eskolaita α-Cr₂O₃) was epitaxially grown on c- and r-cut sapphire. Also, the material was grown in the polycrystalline form on fused silica. Micro-Raman spectra of the films excited with 532 nm laser radiation were measured. Five Raman bands are expected in the spectrum of α-Cr₂O₃, including the most intensive one (A_{1g}) at about 550 cm⁻¹. We demonstrated that the position of the latter band depended on the substrate: in the sapphire case the band was greatly blue shifted with respect to its position in the silica case. The shift was used for the evaluation of the strain in the epitaxial chromia. The evaluation gave a hydrostatic equivalent stress of 5.9 GPa for the thinnest (about 4 nm) film on c-cut sapphire. With the thickness increase up to 210 nm, the stress in the films decreased down to 1.9 GPa. The compressive stress was observed also in the films grown on r-cut sapphire.

15:50

Poster

C-16

In situ heat analysis of ALD growth by usage of thermopile

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We have constructed a thermopile capable of detecting the heat evolved in each reaction taking part in the cycles of atomic layer deposition growth. The sensor is tested on the growth of aluminiumoxide from trimethylaluminium (TMA) and water. This show that both half reactions of the TMA and water process are exothermic while excess reactants apply a cooling effect on the substrate. The outcome for analysis of similar processes will also be presented. The thermopile may be used to asses whether reactions are taking place, to indicate the necessary pulsing parameters for growth, and to provide important insight into the reaction process.

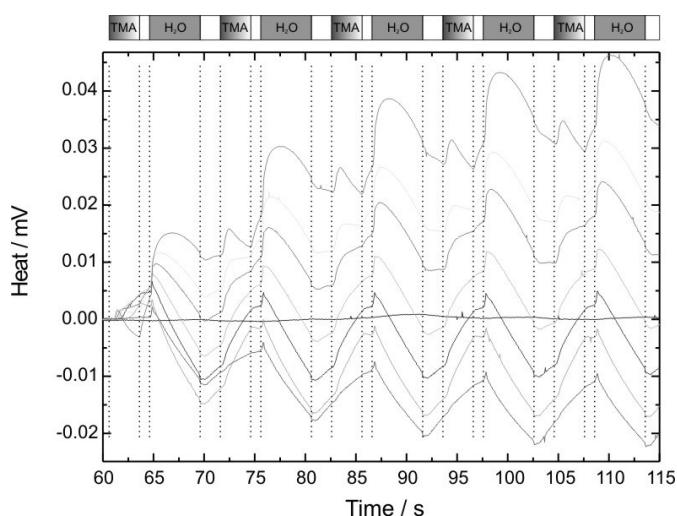


Fig. 1: The evolution of heat as measured by a thermopile during alterations of TMA flow for the pulsing sequence: 5s H₂O, 2s purge, 3s TMA, 1s purge. The flow is increased gradually on going from the bottom to the upper line.

15:50

Poster

C-17

X-ray absorption spectroscopy as a method for structural study of HfO₂ and ZrO₂ thin filmsRaul Rammula^{1,2}, Jaan Aarik¹, Arvo Kikas¹, Tanel Käämbre¹, Hugo Mändar³, Teet Uustare¹, Väino Sammelselg^{1,2}

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Methods based on recording of photoelectrons excited by X-rays have frequently been used for composition studies of ultrathin films because of high surface sensitivity of the methods. It is not so well known, however, that a similar technique can be applied for structural characterization of thin films. This work continues our recent study of crystal phase effects on X-ray absorption spectra of HfO₂ and ZrO₂. HfO₂ and ZrO₂ films with different phases were deposited on Si (100) substrates in a flow-type hot-wall ALD reactor at temperatures of 180–600 °C using halide-based processes. X-ray absorption near edge structure

(XANES) and X-ray photoelectron spectroscopy measurements were performed at beamline D1011 of MAX-II storage ring (Lund, Sweden). For the reference measurements of the phase composition RHEED and XRD methods were used.

In the XANES studies of the amorphous, cubic, monoclinic and tetragonal phases, the O 1s edge of HfO₂ and ZrO₂ was demonstrated to be more sensitive to the phase composition. The main bands in the spectra peaked at 532, 537, 541 and 543 eV. In case of amorphous sample, the peaks were not well resolved probably due to non-periodicity of local environment of oxygen atoms. In cubic and especially monoclinic phases the peak at 532 eV was split into two sub-peaks. Tetragonal phase gave very intense and well-resolved peak at 532 eV, but the second maximum at 537 eV was less intense and consisted of two overlapping maxima. XANES spectra of several cubic samples showed no significant differences, although the film thicknesses as well as precursors and growth temperatures differed markedly.

15:50

Poster

C-18

Atomic Layer deposition of iron oxide nanotubesMårten Rooth¹, Anders Hårsta¹, Anders Johansson, Mats Boman, Kaupo Kukli, Jaan Aarik²

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Iron oxide is a material of great interest in numerous scientific fields and applications. Amongst the many applications and properties of this material photo catalysis, gas sensors and the interesting magnetic behaviour can be mentioned. In many applications a large surface area is desired. Surface enhancements can be done in several ways; one way is to deposit the desired material on a template with a high surface area. Anodic aluminium oxide (AAO) is a suitable template, since it has a high surface area and it also possesses a high degree of order. The porous structure of the AAO puts high demands on the deposition method, especially when depositing a non-conducting material. One technique that meets these demands is atomic layer deposition (ALD).

In this study thin films and nanotubes of iron oxide have been successfully deposited using atomic layer deposition (ALD) with the precursor combination ferrocene Fe(Cp)₂ and oxygen. Thin films and nanotubes have been grown on Si (100) and anodic aluminium oxide (AAO), respectively. Depositions were carried out in the temperature region of 350 °C to 600 °C on Si (100) and at 400 °C on AAO. The growth per cycle values were around 0.14 nm on Si (100) and 0.06 nm on AAO. Quartz crystal micro balance (QCM) showed that the process exhibited ALD characteristics. GIXRD showed that below 500 °C the iron oxide crystallised as a phase mixture consisting of the rhombohedral Fe₂O₃ phase (hematite) and a second phase that could not be unambiguously identified. Above 500 °C only phase pure hematite was detected. XPS analysis showed a carbon contamination level of around 1-3 at-%. For deposition of nanotubes both commercial and in-house made AAO membranes were used, having aspect ratios of 200 and 35, respectively. By etching of the in-house made AAO membranes after deposition, free-standing nanotubes retaining the order of the AAO template could be fabricated. The nanotubes were supported by either silver rods or glue.

15:50 Poster C-19

TEM Observation of ZnO Thin Films Grown by Atomic Layer DepositionKoki Saito¹, Yuya Hiratsuka¹, Atsushi Omata¹, Hisao Makino², Seiichi Kishimoto², Tetsuya Yamamoto², Michiya Yamaguchi³**1.** Teikyo University of Science and Technology, 2525, Uenohara, Yamanashi 409-0193, Japan **2.** Kochi University of Technology, Research Institute, Materials Design Center, Miyanokuchi, Tosayamada-cho, Kami-city, Kochi 782-8502, Japan **3.** CASIO Computer, 2951-5, Ishikawa-cho, Hachioji, Tokyo 192-8556, Japan

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ZnO has become attractive as a new candidate for short wavelength optical devices and transparent conductive oxides because of its wide bandgap of 3.37 eV and a large exciton binding energy of 60 meV. The growth technique of atomic layer deposition (ALD) is effective to realize precise control of crystal growth and large area uniformity at low growth temperature. In our previous work, we have achieved the atomic layer growth of ZnO thin films on sapphire substrates. However, the crystal quality of those films was insufficient for the application to optical devices because of the large lattice mismatch of 18 percent between ZnO and c-plane sapphire substrate. In this work, in order to improve the crystal quality, we tried to grow ZnO thin films on ZnO substrate by ALD and crystal quality was evaluated by X-ray diffraction (XRD) measurements and transmission electron microscopy (TEM) observations.

ZnO thin films were grown on glass, c-plane sapphire and ZnO substrates by ALD. Diethylzinc (DEZ) and H₂O were used as Zn and O precursors, respectively, and these precursors were alternately introduced into the growth chamber. It was found that the atomic layer growth was achieved at the growth temperatures ranging from 175 to 250 °C, which was independent of the types of the substrates. By XRD measurements, both the (0 0 0 2) and (1 0 -1 1) orientations were observed for ZnO thin films grown on glass and sapphire substrates, while only the (0 0 0 2) orientation was observed for ZnO thin films grown on ZnO substrates. The defect configuration of ZnO thin films was evaluated by TEM. It was found that the defect density was reduced by increasing the growth temperature from 200 to 250 °C for all types of the substrates. In addition, it was clearly observed that the crystal quality of ZnO thin films was greatly improved by using ZnO substrate instead of glass or sapphire substrates.

15:50 Poster C-20

ZnO homoepitaxial growth by Atomic Layer Epitaxy technique.Paweł Skupiński¹, Krzysztof Kopalko¹, Krzysztof Grasa^{1,2}, Elżbieta Łusakowska¹, Wiktor Domuchowski¹, Andrzej Mycielski¹**1.** Polish Academy of Sciences, Institute of Physics, al. Lotników 32/46, Warszawa 02-668, Poland **2.** Institute of Electronic Materials Technology (ITME), Wólczyńska 133, Warszawa 01-919, Poland

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ZnO is a wide bandgap semiconductor (3.37 eV) with large exciton binding energy (60 meV). For these reasons, it is a good material for applications in ultraviolet light-emitters and laser diodes. However, the UV applications are being impeded because obtaining p-type conductivity ZnO is still difficult. There are some papers, which report on successful preparation of ZnO p-type layers with sufficient electrical quality. This fact and unquestionable advantages of homoepitaxial growth, such as lack of stress in epitaxial layer and, in turn, low number

of structural defects, motivated us to grow ZnO using the Atomic Layer Epitaxy technique. The easy access to ZnO substrates produced by CVT method also influenced our decision. ALE grown samples were analyzed using Atomic Force Microscopy. On the first obtained homoepitaxial ZnO layers we found regions of several square micrometers with good crystalline quality. This success encouraged us to continue growth and characterization experiments, which are presented in this work.

This work was supported by the Ministry of Science and Information Technologies, Poland, through the grant No. 3 T08A 051 28.

15:50 Poster C-22

ALD of Cr₂O₃ on aluminaAivar Tarre^{1,2}, Rainer Pärna^{1,2}, Alar Gerst¹, Ahti Niilisk¹, Teet Uustare¹, Arnold Rosental^{1,2}**1.** Institute of Physics, University of Tartu, Riia str. 142, Tartu 51014, Estonia **2.** Estonian Nanotechnology Competence Center, Riia str. 142, Tartu 51014, Estonia

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Since the 1960-s, chromia has been the subject of much experimentation as a functional material for semiconductor gas sensors. In general, the material has a p-type gas response, distinguished, e.g., by the decrease of conduction under the influence of reducing gases. Such behavior contrasts with the n-type response typical of metal oxides conventional in this field of application. The two cases differ in the surface chemistry. However, the details of the chemistry and the dependence of conduction of the chromia layers on the partial pressure of different gases are still poorly understood. It seems useful to tackle the problems by examining what happens in the compact thin films. Here we describe atomic layer deposition (ALD) of such films and give a brief survey of their properties.

The films were grown at 420 °C on ceramic-phase and (012) and (001) cut single-crystalline alumina substrates. Chromyl chloride and methanol served as precursors. 25–900 ALD cycles were applied. The growth per cycle was, on average, 0.08 nm. The samples were provided with Pt electrodes covering the films or being underneath the films. The current through the samples was measured at 450 °C in the flowing synthetic air and in the air with hydrogen, methane, or carbon monoxide admixtures.

According to RHEED data, the films grown on single-crystalline substrates were epitaxial. The films on ceramic substrates were polycrystalline without any preferred orientation. Micro-Raman analysis did not show the presence of phases other than chromia. All the films were well-conductive. In a thickness range of tens of nanometers, the thickness of the films had only a slight effect on their conduction. The films were responsive to 30 ppm of hydrogen and carbon monoxide, being unable to discriminate between them, and almost unresponsive to methane. Transition times approached seconds and minutes. The epitaxial films had better sensor properties than their polycrystalline counterparts.

15:50 Poster C-23

Low Temperature Deposition of Tantalum CarbideEva Tois, Raija Matero, Suvi Haukka, Marko Tuominen

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For decades polysilicon has been the gate material of choice for CMOS devices. Easy processing and tuning of the work function by doping are the advantages of polysilicon gate, while the disadvantage is the formation of depletion layer that leads to unwanted increase of EOT (equivalent oxide thickness). Metal gate materials do not form any notable depletion layer, which enables further EOT down scaling. These must, however, have a suitable work function to meet either the requirements of NMOS or PMOS devices. Also good thermal stability at high temperature is preferred. Tantalum carbide (TaC) has shown to be one promising candidate for n-type gate metal electrode material. In this paper we report a low temperature TaC deposition process in which two metal compound precursors are alternatively pulsed to the surface in an ALD like mode.

Tantalum carbide films were deposited in ASM Pulsar[®]2000 AL-CVD[™] reactor at 325 °C from tantalum pentachloride (TaCl₅) and trimethylaluminum, TMA. Substrates were 200mm silicon wafers either with 20 nm thermal oxide or 3 nm ALD HfO₂. Film thickness was determined by spectroscopic ellipsometry and X-ray reflectometry (XRR). Crystallinity and composition were analyzed by X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS) and Rutherford Backscattering Spectrometry (RBS). Sheet resistance was measured with four-point probe. For thermal stability study, the films were annealed at 400-1000 °C in ASM Levitor[®]4200.

TaC growth rate was as high as 2.3 Å/c, which could be a sign of some other growth mode than only pure ALD. The films were smooth, XRR roughness was < 1 nm, and only slightly crystalline which did not change during high temperature annealing. Density of as deposited film was ~7 g/cm³ and resistivity was ~1200 μΩcm, while thermal annealing in He atmosphere 30s at 1000°C decreased the resistivity to ~600 μΩcm. In this paper the effect of deposition parameters on growth of TaC and on the film properties will be presented.

15:50	Poster	C-24

Thermal Stability of thin ALD ZrO₂ Layers as Dielectrics in Deep Trench DRAM Devices Annealed in N₂ and NH₃

Wenke Weinreich¹, Martin Lemberger², Elke Erben³, Johannes Heitmann³, Uwe Schröder³, Velislava A. Ignatova¹, Lutz Wilde¹, Steffen Teichert³, Anton J. Bauer², Heiner Ryssel², Peter Kücher¹

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The evolution of deep trench DRAM capacitors necessitates innovations in material selection as well as in process technology. For the 36 nm generation, crystalline ZrO₂ is intended as high-k dielectric in order to meet the electrical targets. Additionally, the development of suitable ALD processes for the deposition of the dielectric in high aspect ratio trenches is a second essential issue. Therefore, material evaluation adjusted to the process conditions used in trench DRAM production with a capacitor-before-transistor process flow is inevitable, especially, as the thermal stability during the post deposition anneal (PDA) up to 1000°C is one of the most challenging requirements to the dielectric. Growth and crystallization of ALD-ZrO₂ on a SiO₂-interface depend on both deposition temperature and layer thickness. Un-patterned wafers with as deposited amorphous and after various PDA's ZrO₂ layers (5 nm physical thickness) were electrically characterized by the non-contact corona charge / Kelvin-probe technique. Physical properties were analyzed by Ellipsometry, AFM, XRD/XRR, ARXPS and SIMS. For the PDA in N₂ up to 400°C, the layers remain amorphous showing

the same CET (1.55 nm), thickness and density. PDA's up to 700°C, however, crystallize the material to the tetragonal phase with an increased density and a reduced layer thickness. Crystallization and nitrogen incorporation induce a CET decrease (1.25 nm). After 800°C PDA, an increased physical thickness and CET (1.35 nm) is observed due to an interfacial layer growth. For the more reactive NH₃ PDA, all above effects especially the changes in CET are more pronounced. Leakage current densities do not change significantly after PDA in both atmospheres up to 700°C, but severe degradations occur for temperatures higher than 800°C. In conclusion, the NH₃ treatment is more promising than N₂ to reduce the CET of ZrO₂ although no improvement in leakage current densities at high temperatures compared to N₂ could be found.

15:50	Poster	C-25

Application of interval analysis for determination of the parameters of the Schottky contacts

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Due to its relatively wide band gap, ZnO is attractive material for various optoelectronic applications in the short wavelength region. For such devices good Ohmic and Schottky contacts are required. In particular, the achievement of high quality Schottky diodes with large barrier heights and low leakage currents is still very challenging.

An important part of the production process of the metal-semiconductor junction is its final characterization in terms of current-voltage and capacitance-voltage dependencies. From these characteristics various parameters like ideality factor, effective barrier height of the diode, series resistance and saturation current density can be extracted. This is usually done with fitting of some theoretical formulae to experimental data. Because in many cases measured quantities depend on values of above parameters in a nonlinear way, this fitting process may be seriously disturbed by the well known difficulties. For example, if there are multiple solutions to the problem and have all been located? Do local minima (if any) differ substantially from a global one? What is the sensitivity of the solution on the uncertainties of the fitting and model parameters? Traditional methods do not answer for above questions. On the other hand interval analysis provide tools needed to resolve these problems with mathematical and computational certainty. In this work we present the results of interval computations of the ZnO based Schottky diodes. It was found that interval analysis offers interesting alternative for other fitting procedures.

Symposium D

Welcome

This Symposium is aimed at getting together specialists in the related areas and is organized to follow the tradition already established by the previous Symposia in Warsaw (1993, 1996, 2003), Szeged (1995, 2005), Dresden (1997), Sendai (1998), and in Mt. Tremblant (2001).

The Symposia focus on chemical properties of nonequilibrium materials of disordered or nanocrystalline structure. Both fundamental and applied aspects of research are discussed. It shall be emphasized that to meet extreme demands of the modern technology functionalized materials of enhanced specific properties are needed. Moreover, high resolution methods of characterization of new materials are necessary to identify factors responsible for their unique properties. Nonequilibrium processing techniques provide potential for producing compositionally and structurally graded materials with optimized properties.

In order to extend the necessary collaboration of chemists and physicists with material scientists, the Scientific Committee of these cyclic Symposia has decided to join the Annual European Materials Research Society Fall Meetings and to organize the next Symposium as a part of this Meeting.

The scope of this Symposium belongs to the general topic "Chemistry for Material Science", and it is aimed at promoting various opportunities for interdisciplinary collaboration of scientists from around the world in addition to presentation of new results, ideas and technologies in the field of chemical properties of novel materials including (but not limited to) amorphous and nanocrystalline alloys obtained by rapid quenching, sputter deposition, mechanical alloying, electrodeposition and other chemical and physical methods.

Due to their almost infinite compositional flexibility, novel amorphous or nanomaterial alloys can be tailored to have a number of properties optimized for the specific requirements of modern technologies (including corrosion protection, charge storage or electrocatalysis)

Low reactivity with respect to degradation reactions (localized/general corrosion and dissolution, hydrogen embrittlement) is required to improve the stability of usable parts made from these materials.

High reactivity and selectivity are required for efficient electrocatalysts and catalysts. To develop such materials from composition-controlled nonstructural and amorphous alloys by exploiting mechanical, electrochemical, and chemical activation, fundamental and applied research are required.

Discussion among specialists from the areas of material science, physics, chemistry and electrochemistry should contribute to:

*characterizing the decisive factors, including, local changes introduced during modification and /or activation procedures,

*finding efficient, interdisciplinary ways of tailoring novel functional materials for new processes including electrocatalysis; this also includes new materials characterized by a high hydrogen uptake and their application in hydrogen storage.

List of scientific committee members

Anett Gebert , IFW Dresden, Germany

Hiroki Habazaki , Hokkaido University, Sapporo, Japan

Koji Hashimoto , Institute for Materials Research, Tohoku University, Sendai, Japan

Maria Janik-Czachor , Institute of Physical Chemistry, Polish Academy of Sciences, Warsaw, Poland

Stanisław M. Filipek , Institute of Physical Chemistry, Polish Academy of Sciences, Warsaw, Poland

Paweł Kulesza , Faculty of Chemistry, Warsaw University, Poland

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Proceedings

***The post conference materials will be published in Journal of Solid State Electrochemistry (selected papers).**

***Authors presenting papers not dealing with electrochemistry may wish to publish their work in "Materials Science Poland".**

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Programme

Monday, 17 September

Lunch break

Monday afternoon, 17 September, 12:30

Opening Symposium D

A. Jabłoński, Director, Institute of Physical Chemistry, PAS; G. Chalasinski, Dean, Faculty of Chemistry, Warsaw University; K. Sikorski, Vice-dean, Faculty of Materials Science and Engineering, WUT

Monday afternoon, 17 September, 13:45

14:10 Invited

Structure and electrochemical reactivity of oxide nanolayers on metal and alloy surfacesPhillipe Marcus*Ecole Nationale Supérieure de chimie de Paris (ENSCP), 11 rue P. et M. Curie, Paris 75005, France**e-mail: philippe-marcus@enscp.fr*

Oxide layers on metal and alloy surfaces play a role in various areas including corrosion protection, electrocatalysis, energy conversion and storage. They often exist under conditions that are not equilibrium conditions, but rather stationary or even non stationary conditions.

Their electrochemical reactivity is a key factor, and it is largely governed by their nanostructure.

In this lecture, the following aspects will be addressed :

- mechanisms of growth of oxide nanolayers on metals and alloys under controlled electrochemical potential
- nanostructure of oxide nanolayers under stationary conditions
- oxide surface/electrolyte interface : electrochemical reactivity at the atomic scale.

The materials selected to illustrate the above aspects are Cu₂O/Cu, NiO/Ni, Cr₂O₃/Cr and stainless steels, V₂O₅/V.

The experimental techniques used for surface imaging and spectroscopy are : scanning tunnelling microscopy (STM), atomic force microscopy (AFM) and X-ray photoelectron spectroscopy (XPS).

14:50 Invited

Polyoxometalate chemistry for electrochemical material scienceGalina Tsirlina*M.V. Lomonosov Moscow State University, Vorobyevy gory, Moscow 119992, Russian Federation**e-mail: tsir@elch.chem.msu.ru*

Various functional materials designed on the basis of polyoxometalates (POMs) inherit some structural and electronic features of their molecular precursors. Surprisingly, 99% of groups using POMs limit themselves by less than 1% of POMs described in the literature, namely Keggin and Dawson anions with non-metallic heteroatoms. Much wider variety of materials can be obtained if less popular and even exotic POMs are involved.

The following groups of POMs are considered: (i) less known heteropoly compounds, namely salts of Anderson and Dexter anions and decatungstates; (ii) difficult-to-synthesize keggins with metallic heteroatoms; (iii) isopoly compounds tending to complex pH-dependent equilibria in aqueous solutions, i.e. always coexisting with each other; (iv) mixed (W-Mo or W-V) isopoly compounds. Various routes to use these precursors for fabricating rechargeable materials and metal-oxide nanostructures are discussed and illustrated using the original experimental data on cathodic electrodeposition.

The following phenomena important for obtaining some certain material anions are accentuated: (i) adsorption of POMs on various electrode materials; (ii) association of multicharged POM and supporting cations; (iii) protonation of POMs; (iv) the specific features of electrochemical kinetics known for electroreduction of POMs. Carbon, platinum, gold and ITO supports are the most typical, but some data are related to mercury electrode involved for better understanding of kinetic details.

The techniques applied to materials characterization are XRD, SEM, STM, IR spectroscopy, XPS and voltammetry.

This work is supported by RFBR, project No 05-03-32520a.

15:20 Oral

Interface stability and composition profile of electrodeposited nanolayersLászló Péter¹, Imre Bakonyi¹, Enikő Tóth-Kádár¹, Kálmán Vad², Gábor L. Katona²

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The sharpness of layer interfaces and a homogeneous component distribution within each layer are a key issue in the electrodeposition of nanostructures from magnetic alloy films to multilayers.

When alloys are used to build up multilayers with a thickness in the nanometer range, the codeposition of the constituent metals usually lead to a composition gradient within the layer along the growth direction. The composition change is a result of the mixed control of deposition by diffusion and activation. This change in the local composition can have a significant impact on the magnetic properties of the layer. The local composition evolution can be calculated from the change in the total composition as a function of the layer thickness. Also, it can be followed by direct depth profiling methods. We used secondary neutral mass spectrometry to observe the depth profile of various metallic multilayers. A comparison of the indirect and direct local composition analysis results will be presented.

In metallic multilayers, the composition of the individual layers may change already during the electrodeposition process due to the dissolution of the more active material. In this case, component intermixing occurs at the layer interfaces, and the layer thicknesses will significantly deviate from the nominal ones. This can be clearly observed both from direct imaging methods like TEM and indirect methods like compositional analysis and magnetic properties. An easy-to-follow electrochemical optimisation process is suggested to identify the optimum conditions for the electrodeposition of metallic multilayered materials.

Coffee break

Monday afternoon, 17 September, 15:30

Joint Poster Session (Part I)*Monday & Wednesday*

Monday afternoon, 17 September, 15:50

Session 2

Monday afternoon, 17 September, 17:00

17:00 Invited

Structural control of anodic titanium oxide using non-equilibrium titanium alloysHiroki Habazaki, Muhammad T. Tanvir, Yuki Oikawa, Koji Fushimi
*Hokkaido University, Sapporo 060-8628, Japan**e-mail: habazaki@eng.hokudai.ac.jp*

Anodic films formed on a range of valve metals are classified into two types, i.e., barrier-type and porous-type. The barrier-type anodic titanium oxide is a potential dielectric material for electrolytic capacitors, while the porous-type anodic titanium oxide has potential applications in photocatalysis, photoelectrolysis, photovoltaics and as sensors.

The barrier-type anodic films are formed on high purity titanium in acidic and neutral aqueous electrolytes, but an amorphous-to-crystalline transition occurs at low voltages of less than 10 V. After the transition, the film growth proceeds, accompanying oxygen gas evolution on the crystalline oxide, which is located at the inner part of the anodic film. Thus, the films developed contain high density of flaws, and are not suitable for the capacitor application. In this paper we have demonstrated the effective suppression of the amorphous-to-crystalline transition of anodic titanium oxide by alloying of titanium. Magnetron sputtering is used to form homogeneous solid solutions exceeding the solubility limits of alloying elements at equilibrium

The most effective element in suppressing the amorphous-to-crystalline transition is silicon. By the addition of only 6 at% silicon to titanium, the amorphous film growth proceeds to more than 100 V at high current efficiency of ~100%. The contents of the alloying elements required to suppress the crystallization are strongly dependent upon the element. For instance, more than 25 at% aluminium is necessary to grow the anodic film without crystallization. It has been found that there is a good correlation between the content of the alloying elements for crystallization suppression and mobility of the alloying element species in growing anodic oxide; more slowly migration species are more effective in suppressing the crystallization.

17:30 Oral

On some peculiarities of electrochemical oxidation of Ti and Ti6Al6V alloy

Andrzej Sadkowski¹, Janusz Kamiński², Malgorzata Lewandowska², Magdalena I. Walczak¹

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Electrochemical oxidation is commonly used in industrial practice, e.g. as a method for coloring of the metal surface and modification of its properties. However, the process of oxide film formation is still not fully understood. Ti and Ti6Al4V electrodes were examined with various electrochemical methods in conditions of anodic film formation in 1M H₂SO₄. The influence of metal microstructure on the process of oxidation was found. Good conditions for anodizing were available only after etching electrodes surface in HNO₃/HF solutions revealing fresh, active metal surface ready to hydrogen intake. Etched microcrystalline Ti electrodes presented different interference colors at various crystallites seen under optical microscope while nanocrystalline anodized surfaces were colored uniformly. Rest potential of both etched and anodized electrodes drifted in long runs to reversible hydrogen electrode potential.

The process of anodic film formation in the voltage range of 10V – 95V examined with cyclic voltammetry (CV) was clearly stepwise with oxidation state attained at lower voltage preserved and only super-added in subsequent scan to higher voltage limit. CV tests in broad voltage range disclosed sharp current spikes possibly related to phase transition in interfacial region. There was not clear difference in electrochemical characteristics of anodized Ti and Ti6Al4V electrodes.

Electrochemical impedance (0.1 Hz – 100 kHz) showed that the electrodes with “native oxide” presented ohmic resistance component of several ohms typical for metal/electrolyte interface and oxide layer resistance of the order of 10⁷ Ohm while oxides formed electrochemically presented properties typical for semiconductor/electrolyte interface without clearly distinct ohmic resistance and at least three relaxation times. This character was preserved for many days for electrodes stored in laboratory atmosphere.

18:00 Invited

Electrochemical property and surface composition of metallic biomaterials under the in vivo and in vitro environments

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Biocompatibility and corrosion of metallic biomaterials is dominated by interaction between the material surface and tissue (cells). In this study, the interaction of metallic biomaterials with cells was examined by electrochemical measurement and surface analysis of the materials retrieved from the patients or immersed in medium with and without cells.

Polarisation test of pure Ti and 316L stainless steel was performed in medium with and without fibroblast cells. Influence of the cells on cathodic reaction depended on cell density and cathodic current density corresponding to the reduction of dissolved oxygen decreased with the increase of cell density. Protectiveness of passive film on pure Ti was not significantly influenced with the cells whereas that on 316L steel decreased with the cells.

On the surface of Ti alloy orthopaedic devices retrieved from the patients, calcium and phosphor always precipitated. In the case on 316L steel devices, these elements precipitated on the surface formed in hard tissue more frequently than that in soft tissue. Similar results were obtained on the surfaces formed in the medium with and without the cells. The amount of precipitated calcium and phosphor on pure Ti was larger than that on 316L steel. Moreover, the presence of cells caused the decrease of these elements. Characteristic influence of the cells appeared on the chemical state of sulphur precipitated. The binding energy of S2p XPS spectra on the surfaces formed in the human body and with the cells was rather lower than that on the surface formed without the cells.

Consequently, interaction between metallic biomaterials and cells forms a different interface environment from that in the absence of cells. In order to accurately evaluate the biocompatibility and corrosion of the materials, the dominant factor derived from cells should be elucidated.

18:30 Oral

Morphological and surface analytical characterization of chemically pretreated Ti surfaces intended for biomedical applications

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Titanium is known as a biocompatible metal characterized by biological and corrosion immunity and good mechanical properties, including a high fracture toughness. In variety of environments this metal undergoes "natural" oxidation which determine its resistance to corrosion. It can also be exposed to chemical treatments in acidic or alkaline solutions which "enforces" chemical and morphological changes of Ti surface. Those methods, if well controlled, may increase Ti surface thus making it more biocompatible. However, the morphological and chemical factors responsible for their interactions with biological cells are still not well known.

The aim of this work was to compare surface chemical and morphological changes introduced by commonly used NaOH aq pretreatment with those occurring in a new "piranha" acidic solution. Special care was exercised to identify possible changes which may be decisive for the biocompatibility of the Ti-elements subjected to these surface modifications.

Surface analytical techniques such as AES or XPS combined with Ar⁺ ion sputtering allowed to investigate in detail the chemical composition of Ti oxide layers. SEM examinations provided morphological characterization of the surface of Ti samples. The results revealed large differences in morphology of Ti surfaces pretreated with different procedures whereas only minor differences in the chemistry of the surfaces were detected.

All tested materials appeared to be biocompatible in vitro. "Piranha" treated samples seem to be a good support for osteoblast-like cells to proliferate and differentiate. Cells seeded on modified titanium surface could synthesize extracellular proteins, especially collagen.

Keywords: biomaterials, surface chemical treatment, Auger electron spectroscopy (AES), X-ray photo-electron spectroscopy (XPS)

Tuesday, 18 September

Session 3

Tuesday morning, 18 September, 9:00

9:00 Oral

Effects of severe plastic deformation on the corrosion behavior of aluminum alloys

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Effects of severe plastic deformation on the corrosion behaviors of aluminum-based binary alloys containing precipitates were investigated. The aluminum alloys were severely deformed by equal-channel angular pressing (ECAP) processes and the corrosion resistance of the aluminum alloys was evaluated by means of potentiodynamic polarization measurements in a neutral buffer solution containing chloride ions at a concentration of 0.002 M. The pitting potential of Al-5.4wt%Ni alloy whose microstructure has a-Al phase and Al/Al₃Ni eutectic region was increased by the introduction of strain by ECAP. ECAP treatment of Al-5wt%Cu alloy also resulted in increase in pitting potential. The pit initiation sites of the Al-Ni alloy without ECAP were in the relatively large a-Al phase and the pit growth was suppressed in the Al/Al₃Ni eutectic region. By applying ECAP, the microstructure of the Al-Ni alloy became more homogeneous and relatively large a-Al phase dis-

appeared. Presumably, the more homogeneous microstructure is attributable to the higher pitting resistance of ECAPed Al-Ni alloys. In the case of the Al-Cu alloy, pit formed selectively in Cu-depleted zones around Al₂Cu particles which seemed to be acting as cathodic sites. The improvement of the pitting resistance seems to be due to diminution of Cu-depleted zone and the refinement of Al₂Cu particles caused by huge plastic strain during ECAP.

In contrast, ECAP treatment of Al with 4N purity resulted in a decrease in open circuit potential, slight increase of passive current and shift of pitting potential to the negative direction. Consequently, the improvement of pitting resistance of Al binary alloys is not because of introduction of dislocations and defects by severe plastic deformation but because of the change in microstructure such as homogenization and refinement of precipitates.

9:20

Oral

The Effect of Hydrostatic Extrusion on the Corrosion Stability of Austenitic Stainless Steels

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The stability of passive film formed on austenitic 303 and 316 stainless steels in the as-received state and after a heavy deformation by hydrostatic extrusion (HE) was investigated in an aggressive environment containing Cl⁻ ions.

TEM and stereological image analysis were used to examine structural changes introduced by HE treatment. The effects of surface oxide film on pit nucleation in stainless steel was investigated by anodic polarization in borate buffer + 0.1 M NaCl. Surface analytical techniques such as light microscopy, SEM, AES and SAM were employed to characterize morphology, grain size, and chemistry of the surface, including local characterization of nonmetallic inclusions and their surface before and after HE modification. The results confirm a drastic reduction of grain size, due to HE process and a distinct reduction of the size of sulphide inclusions occurring in the 303 matrix. Moreover a discontinuity of the surface oxide film on MnS inclusions was found using a local SAM analysis. These factors apparently result in a lowering of resistance of 303 HE to pit nucleation as compared to the as-received material. On the contrary, 316 stainless steel, where only scarce nonmetallic inclusions occurred, has shown a higher resistance against passivity breakdown after HE process. A tentative role of the above differences in stability of the passive oxide film on these materials is discussed.

This work was financially supported by the Polish Ministry of Education and Science through grant 3T08C 013 29. Surface characterizations were performed using a Microlab 350 located at the Physical Chemistry of Materials Center of the Institute of Physical Chemistry, PAS and of the Faculty of Materials Science and Engineering, WUT.

9:40

Oral

Spontaneous passivity of bulk amorphous Ni-Cr-Ta-Mo-Nb-P alloys in concentrated hydrochloric acids

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Some amorphous alloys possess extremely high corrosion resistance to aggressive environments such as concentrated HCl where conventional metallic materials cannot be used. Copper mold casting is the easiest and simplest method to form amorphous bulk alloy rods, and if copper-mold cast 1 mm diameter rods become amorphous, desirable shapes of amorphous bulk alloys can be processed in the super-cooled liquid state of the amorphous alloy powders. The present work applying copper-mold casting aimed to tailor amorphous Ni-Cr-Mo-Ta-Nb-P alloys resistant to concentrated hydrochloric acids. The roles of corrosion-resistant alloying elements were studied mostly by XPS.

Ni-(1-4)Cr-1Mo-22Ta-14Nb-7P and Ni-1Cr-(1-8)Mo-22Ta-14Nb-7P alloy rods of 1 mm diameter were identified as the amorphous single phase, while Ni-5Cr-1Mo-22Ta-16Nb-7P and Ni-1Cr-10Mo-22Ta-16Nb-7P alloy rods in addition to Ni-0Cr-1Mo-22Ta-16Nb-7P and Ni-1Cr-0Mo-22Ta-16Nb-7P alloys were composed of major amorphous and minor nanocrystalline phases. The single amorphous phase alloys were immune to corrosion in 12 M HCl at 30°C but the alloys with a nanocrystalline phase showed the detectable corrosion loss. All alloys including those of phase mixtures were immune to corrosion in 6 M HCl at 30°C. They were all spontaneously passive. The presence of molybdenum was prerequisite for immunity to corrosion but the excess increase in molybdenum resulted in the increase in the corrosion loss.

According to XPS the air-formed film contained Mo^{4+} , Mo^{5+} and Mo^{6+} in addition to concentrated Ta^{5+} , Cr^{3+} and Nb^{5+} , consisting of the outer triple oxyhydroxide of Ta^{5+} , Cr^{3+} and Nb^{5+} and the inner Mo^{4+} oxide. The high stability of the alloys with these air-formed films were responsible for spontaneous passivity in concentrated hydrochloric acids. The outer triple oxide seemed to be stable in the concentrated hydrochloric acids and the inner Mo^{4+} oxide was assumed to act as the barrier for diffusion of matters through the film.

10:00

Oral

Investigation of aqueous surface processes and corrosion mechanisms of Al-(Cu)-Fe-Cr quasicrystalline approximant

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Because of the unique properties of quasicrystals and approximants in terms of microstructure, adhesion and inherent hardness, they are promising materials for coatings of aluminium alloys when good corrosion resistance is needed. However until now, no comprehensive studies on their corrosion mechanisms have been performed. In this work, two stable approximant phases - a gamma-brass phase (γ -AlCrFe) and a mixture of two orthorhombic approximants of the decagonal phase (O-AlCuFeCr) - have been investigated.

The uniform corrosion rate, electrochemical reactions and localized attack susceptibility have been studied in aqueous environments (0.1 M Na_2SO_4 of pH 2, pH 5.5 and pH 12, 0.1 M NaCl and 1 M NaCl) using Electrochemical Impedance Spectroscopy (EIS), potentiodynamic polarization and Microcapillary Electrochemistry techniques. Polarization measurements in sulphate solutions show that the quasicrystalline approximant are particularly resistant to uniform corrosion and their electrochemical behaviour is comparable to stainless steel. A passive oxide layer showing low current density and stabilizing with ageing time (EIS characterization) is forming in a wide pH range. The presence of a transpassive dissolution is also typical for chromium containing stainless steel. The used sintered samples contain a number of weak points inducing a high susceptibility to localized attack. By characterizing micrometer scale areas with the microcapillaries, the material specific localized corrosion susceptibility can be assessed and is then similar to Stainless Steel.

Auger Electron Spectroscopy (AES) surface composition investigation and in particular depth profiling experiments have shown that the oxide layer is still nearly pure aluminium hydroxide. Mott-Schottky analyses indicate semi-conductive properties of the oxide layer on approximant phases in contradiction with the insulating nature of Al hydroxide. All these results point out on very special properties for these surfaces.

Coffee break

Tuesday morning, 18 September, 10:30

Session 4

Tuesday morning, 18 September, 11:00

11:00

Invited

SiO₂-supported heteropoly acids and Nafion-H prepared by ball-milling for catalytic application

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Silica-supported dodecatungstophosphoric acid (HPW) and Nafion-H were prepared using the ball-milling technique. The samples were characterized and used in catalytic transformations and their properties were compared to those of catalysts made by conventional methods.

Catalyst characterization were performed by physical (BET surface, SEM, DSC, XRD, DRIFT and Raman spectroscopy) and chemical (acid-base titration) methods. Catalytic properties were studied in a range of chemical transformations, which can be induced by electrophilic catalysts including Friedel-Crafts reactions (alkylation of toluene with benzyl alcohol, acylation of anisole with acetic anhydride) and the dimerization of alpha-methylstyrene.

Milling conditions (stainless steel or polystyrene apparatus, milling time), loading of the active components, and the type of support were found to have profound effects on the physical characteristics and catalytic performance of the samples. Ball-milled HPW-on-silica samples prepared under appropriately selected conditions exhibit catalytic properties similar to catalysts made by impregnation. HPW in ball-milled samples, in turn, proved to be more resistant to extraction with polar solvents (hot methanol) and these catalysts display higher stability in recycling studies. Supported Nafion-H samples prepared by ball-milling show high activities in test reactions. The best catalyst sample exceeds the performance of SAC-13, a commercial Nafion-silica nanocomposite catalyst in the selective synthesis of 1,1,3-tri-

methyl-3-phenylindane in the dimerization of 2-phenylpropene. The preparation of catalysts of high catalytic performance requires the use of optimal milling time, because particle coalescence and the resulting decrease in BET surface area hinder the accessibility of the active sites.

11:30

Invited

Selective hydrogenation of isophorone in the presence of catalysts containing nanostructured metals

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Nanostructures of metals (especially from 10 to 13 group of periodic table of elements) can play an important role as hydrogenation catalysts. In this lecture convenient methods have been presented for the selective hydrogenation of isophorone (3,5,5-trimethylcyclohex-2-enone) to: a. isophorol (3,5,5-trimethylcyclohex-2-enol), b. homomenthol (3,3,5-trimethylcyclohexanol) and c. dihydroisophorone (3,3,5-trimethylcyclohexanone). All of the reactions were performed under the atmospheric pressure using gaseous hydrogen and propan-2-ol as a solvent.

Isophorol synthesis (yield *ca.* 40 %, selectivity > 90 %) was performed in the flow system with the fixed catalyst's bed at 423 or 473 K. Au nanoparticles deposited on MgO were used as catalysts. The catalysts were characterized by means of XPS, SEM, TEM and SIMS. A size and shape of Au particles depended on the method and conditions of the catalyst preparation.

Homomenthol was obtained by quantitative hydrogenation of isophorone in the batch system at 353 K over Ni-Al and Ni-Al-Co alloys pretreated with NaOH_{aq}.

Dihydroisophorone synthesis (yield 97 %, selectivity *ca.* 95 %) was also carried out in a batch system at 315 K over Ni-Al-Cr alloy pretreated with NaOH_{aq}. The surfaces of alloys have been examined by SEM and Auger spectroscopies.

The mechanism of C=O and C=C bonds hydrogenation over above mentioned catalysts has been proposed and discussed in respect to the surface species responsible for the reaction selectivity. The role of solvent has been also discussed.

12:00

Invited

Nanostructured electrode materials for low temperature fuel cells and Li ion batteries: mechanochemical synthesis and electrochemical properties

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The development of advanced materials for energy storage and conversion has been receiving wide attention to meet the rising demand of clean energy technologies. In particular a great effort is being devoted towards the improvement of electrochemical power sources perform-

ance, in particular rechargeable lithium ion batteries and low temperature fuel cells, through the investigation of nanostructured bi or multicomponent alloys for electrodes and electrocatalysts respectively. Besides the enhanced electrochemical reactivity related to the thermodynamic metastability, their structural features could offer several advantages as, for instance, short path length for electronic and cation transport, higher electrodes or electrocatalysts active specific area, which can result in higher capacity and power density.

Mechanochemical synthesis by ball milling is recognised as a powerful technique for the preparation of a variety of metastable materials including nanostructured as well as amorphous and quasicrystalline phases. Along this line in this contribution we present the synthesis by mechanical processing and the electrochemical properties of different nanocrystalline alloys: tin based alloys and LiFePO₄ were studied as Li ion battery anode and cathode materials, while PtCo systems were taken into account as electrocatalysts in fuel cells electrode processes. Through a detailed structural and thermodynamic characterization, we try to optimize the microstructural features to improve the electrochemical performances.

Lunch break

Tuesday afternoon, 18 September, 12:30

Session 5

Tuesday afternoon, 18 September, 14:00

14:00

Invited

In situ AFM studies of solid state electrochemical conversions

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In situ AFM allows studying the electrochemical conversion of sub-micrometer size solid particles immobilized on the surface of electrodes. The reaction pathways considerably differ depending on the reaction and on the chemical composition of the solid particle. We have studied the *electrochemical* reduction of PbO to Pb [1], the reductive dissolution of FeOOH [2], the oxidation of Ag nanoparticles to silver halide particles [3], the reduction of Ag(I) and Hg(I) halide crystals [4], and the reduction of Ag₂S crystals [5]. Additionally we have studied the chemical oxidation of Ag to particles to AgBr [6]. Whereas the reduction of PbO to Pb is a topotactic reaction starting at the three-phase boundary solution|solid|electrode, the reduction of Ag₂S to Ag proceeds via a very soft amorphous particle, and the oxidation of silver particles to silver halides proceeds via oversaturated silver halide solutions. The latter reactions exhibit features of double nucleation-growth reactions [7].

[1] U. Hasse, F. Scholz: *Electrochem. Commun.* 3 (2001) 429-434.

[2] U. Hasse, J. Nießen, F. Scholz: *J. Electroanal. Chem.*, 556 (2003) 13-22.

[3] U. Hasse, F. Scholz: *Electrochem. Commun.* 6 (2004) 409-412.

[4] U. Hasse, K. Wagner, F. Scholz: *J. Solid State Electrochem.* 8 (2004) 842-853.

[5] U. Hasse, F. Scholz: *Electrochem. Commun.* 7 (2005) 173-176.

[6] U. Hasse, F. Scholz, *Electrochem. Commun.* 8 (2006) 1005-1010.

[7] U. Hasse, S. Fletcher, F. Scholz: *J. Solid State Electrochem.* 10 (2006)

833-840

14:30

Invited

Two-Phase Synthesis of Charged Metal Nanoparticles and Their Assembly on Electrode Surfaces

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Monolayer-protected metal nanoparticles are commonly prepared by a two-phase synthesis in which metal complexes in aqueous solution are reduced in the presence of a nonpolar solvent that contains an alkanethiol. This approach, often termed the Brust-Schiffrin method, is extended to the preparation of polyoxometalate-protected nanoparticles in aqueous solution. Initially, hexanethiol-protected gold nanoparticles were prepared in toluene. After purifying and fractionating to obtain monodisperse nanoparticles, transfer of the metal center to an aqueous solution was accomplished by ligand exchange involving replacement of the alkanethiolate with a polyoxometalate (POM), phosphomolybdate or phosphotungstate. Evidence for phase transfer of the gold nanoparticle core without a change in size will be presented. These ca. 5-nm anionic moieties are amenable to electrostatic assembly of multilayers on electrodes such as indium tin oxide modified with a monolayer of 3-aminopropyl triethoxysilane. The ability of the POM to catalyze electrochemical reductions is enhanced by the gold nanoparticle core. This synergism will be demonstrated for the reduction of bromate and of dioxygen. Fabrication of multilayered composites of POM-protected nanoparticles and conducting polymers also will be described. For example, alternating layers of anionic POMs and aniline, which subsequently forms a conducting polymer, increases the population of catalytic centers; communication between centers is promoted by the conducting polymer. Control of the quantity of deposition of the conducting polymer by assembly of polyaniline nanoparticles rather than the aniline precursor will be discussed.

15:00

Invited

Conducting discotic polyaromatic material with two unpaired electrons at oxidized state: Synthesis and properties.

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Polyaromatic molecules having symmetry axis equal or larger than three, possess degenerate HOMO's and LUMO's. This opens the possibility to obtain radical anions or cations in a high spin state. Organic molecules having high spin state are attractive in the search of electrical conductivity, ferromagnetism and even superconductivity [1]. Polyaromatic hydrocarbons are of interest as high spin-systems, because many of the monoradicals and diradicals are relatively stable systems. [2]

We report the electrochemical oxidation in acetonitrile of hexapyrrolyl benzene (HPyB) a polyaromatic heterocyclic compound. This oxidation leads to a discotic **polyaromatic heterocyclic** material (HPyB6a) and not to a linear polymer. [3] The electroactivity of this material shows two successive reversible redox waves corresponding to the transfer

of two electrons and esr measurements at oxidized state shows paramagnetic behaviour at room temperature.

[1] F. Demol, F. X. Sauvage, A. Devos, M.G. De Backer, *Synth. Met.*, 99 (1999) 155.

O. Lemaire, M. De Backer, A. Devos, F. X. Sauvage, *Synth. Met.*, 123 (2001) 61.

[2] F. Dietz, N. Tuytuykov, G. Madjarova, K. Mullen, *J. Phys. Chem. B*, 104 (2000) 1746.

N. Tuytuykov, G. Madjarova, F. Dietz, K. Mullen, *J. Phys. Chem. B*, 102 (1998) 10183.

[3] M. Lazerges, M. Jouini, P. Hapiot, P. Guiriec, P.-C. Lacaze, *J. Phys. Chem. B*, 107 (2003) 5042.

Coffee break

Tuesday afternoon, 18 September, 15:30

Session 6

Tuesday afternoon, 18 September, 15:50

15:50

Invited

Preparation strategies and structure – activity relationship of platinum-free electrocatalysts

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Up to now, platinum and its alloys are dominantly used as catalysts for the electro-reduction of oxygen because of its high chemical stability and activity in acidic media. Development of cost-saving alternatives has to face the requirement that the new materials have to be oxidatively stable in acids.

The lecture introduces two promising material systems, based on molecular cobalt – iron – nitrogen centres integrated in a graphite matrix as well as stabilized ruthenium particles.

Catalytically active cobalt and iron centres can be protected from oxidation by anchorage of the metal ions in a chelate-type structure. In the case of ruthenium a decoration of the surfaces of the nanoparticles using chalcogen such as sulphur or selenium leads to a stabilisation and a significant enhancement of reactivity.

Furthermore, In contrast to carbon-supported platinum catalysts where nano-sized particles coalesce after short operation time under fuel cell conditions, surface stabilized ruthenium particles remain separated.

In the talk, the following topics will be addressed:

- preparation strategies and synthesis of non-platinum electrocatalysts;
- elucidation of structure and of electronic state of the catalysts using
 - a) synchrotron radiation (in-situ X-ray Absorption Fine Structure XAFS and Anomalous Small Angle X-ray Scattering SAXS analyses),
 - b) Mößbauer spectroscopy and c) Electron Paramagnetic Resonance EPR;
- structure–activity relationship combining electrochemical and structural findings.

16:20

Oral

Activation of Methanol-Tolerant Carbon-Supported RuSe_x Electrocatalytic Nanoparticles Towards More Efficient Oxygen Reduction

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Although platinum based electrocatalysts are still the most commonly used systems for the reduction of oxygen in acid media, there have been many attempts to synthesize different catalytic materials. Bi-component ruthenium-selenium (RuSe_x) nanoparticles can be viewed as a promising alternative to Pt and Pt-alloy based cathode materials.

In our recent preliminary communication [2], we have reported that modification of carbon-supported RuSe_x nanoparticles with ultra-thin films of tungsten oxide tends to shift the oxygen reduction potential towards more positive values. An electrocatalytic system, that utilizes tungsten oxide modified carbon-supported RuSe_x nanoparticles, is developed and characterized here using transmission electron microscopy and such electrochemical diagnostic techniques as cyclic voltammetry and rotating ring-disk voltammetry and upon introduction (as cathode) to the low-temperature hydrogen-oxygen fuel cell. Following modification of RuSe_x catalytic centers with ultra-thin films of WO₃, the potential of oxygen reduction in 0.5 mol dm⁻³ H₂SO₄ (in the absence and presence of methanol) is shifted ca. 80 mV (under rotating disk voltammetric conditions) towards more positive values, and the percent formation (at ring) of the undesirable hydrogen peroxide has decreased approximately twice when compared to the WO₃-free system. Further, the WO₃-modified RuSe_x (carbon-free) samples have been immobilized on the gold electrode surface for Synchrotron X-ray Photoelectron Spectroscopy (XPS) examination. Relative to bare electrocatalyst, an increase of power density over 10 mW cm⁻² (to 100 mW cm⁻² at 80°C with 300 mA cm⁻²) has been observed upon deposition of WO₃-modified RuSe_x.

References:

- [1] D. Cao, A. Wieckowski, J. Inukai, N. Alonso-Vante, J. Electrochem. Soc., 153 (2006) A869.
[2] P. J. Kulesza, K. Miecznikowski, B. Baranowska, M. Skutnik, S. Fiechter, P. Bogdanoff, I. Dorbandt, Electrochem. Comm., 8 (2006) 904.

16:40

Oral

Modification of electrodeposited cobalt alloys for hydrogen evolution by superimposed external magnetic field

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The electrocatalytic activity towards the hydrogen evolution reaction is most likely related to the active surface area of the coatings (accounting for the porosity of the coatings and the presence of some nodules at the coatings surface). It is known that magnetic field (B) influence

the electrodeposition process of metals. The different magnetic force generates an additional convection, which can affect the morphology of the deposit.

In principle, the grain size is a function of the nucleation rate and the growth of the nuclei: the more the nuclei the smaller is the resulting grain size. It is known that the nucleation rate increases with decreasing nucleation work. Magnetic field applied parallel to the electrode generates an additional convection in the electrolyte caused by the Lorentz force, which acts on the ions. A laminar flow of ions in the electrolyte close to the electrode surface takes place. The magneto hydrodynamic effect (MHD) reduces the thickness of the diffusion layer and leads to an increasing concentration gradient, which should have an effect on the grain size. Superimposed B can influence the texture and the phase formation of electrodeposits. This can be explained by two different effects: Firstly, the layer growth can be influenced directly by B, if B induces a grain growth in the direction of the easiest magnetization. Secondly, the whole electrodeposition process is influenced by the additional convection.

The work aims to obtain Co-W and Co-P alloys by electrodeposition having high cathodic activity for hydrogen evolution in 8 M NaOH at 90°C. External magnetic field generated by permanent magnet was used in order to modify composition and morphology of alloy. The changes in morphology and composition of alloys was searched in order to determine influence of magnetic field on alloys' electrodeposition.

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17:00

Oral

Energy-saving seawater electrolysis for hydrogen production

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For the use of renewable energy, fluctuating power generated in remote deserts or sea will be converted first to hydrogen by seawater electrolysis. The anodes for oxygen evolution without chlorine formation have been tailored for seawater electrolysis. In the electrolysis H₂ and O₂ formation induces high and low pH on the cathode and anode. The difference of pH 10 on the two electrodes leads to 0.6 V increase in the cell voltage. If seawater is flowed in a series first from the anode compartment and then to the cathode compartment, the pH of seawater decreases at the anode compartment inlet and becomes neutral at the cathode compartment outlet, and the pH in the most parts of the cell will be kept to the low value without neutralization because penetration of Na⁺ through the membrane prevents 100% H⁺ penetration. In the electrolysis flowing seawater from the bottom of the anode compartment of 1 m high cell in 1 h consuming 10 % of water from seawater, the cell voltage will be kept at the theoretical one in the most parts of the cell if the H⁺ penetration through the membrane is 98 %.

Necessary conditions for realization of this idea were examined. If pH of 0.5 M NaCl was 1.03, pH on the cathode surface did not increase by electrolysis at 1000 Am⁻², but if pH was 1.34 and 1.98, pH on the cathode increased immediately to 10 by electrolysis at 400 and 30 Am⁻², respectively.

When a proton selective membrane was used, the low cell voltage was kept at 1000 Am^{-2} from the bottom to 60 cm height in 0.25 M Na_2SO_4 due to 93-97% H^+ penetration, although in 0.5 M NaCl the low voltage was kept till about 10 cm height due to 87-92% H^+ penetration. This difference is based on higher degree of dissociation of NaCl than Na_2SO_4 . When 0.025 M Na_2SO_4 was used, the H^+ penetration was 96-97.5% and the cell voltage in the whole cell was 2.5-2.7 V in which 0.5 V were based on the membrane resistance. Thus the membrane through which the H^+ penetration is 98% or higher is required.

Wednesday, 19 September

Plenary Session

Wednesday morning, 19 September, 9:30

Coffee break

Wednesday morning, 19 September, 10:30

Session 7

Wednesday morning, 19 September, 11:00

11:00 Invited

Self Organized nanotopographies for Photovoltaic and Proteomic Applications

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Nanotopographies originating from self organized processes at semiconductors are used for development of a Schottky-type nanoemitter solar cell and for immobilization of proteins on semiconductor surfaces. Using metal electrodeposition into nanopores formed in silicon oxide during oscillatory processes at silicon electrodes in fluoride containing solutions, photoelectrochemical solar cells that operate in the photovoltaic and photoelectrocatalytic mode have been realized. Presently, solar-to-electric conversion efficiencies are above 7%. Improvement strategies and development of solid state solar cells will be discussed.

Applications of electrochemically prepared nanotopographies on silicon for protein immobilization and subsequent detection using scanning probe microscopy will be presented. Also, scanning tunnelling microscopy images of enzymes such as reverse transcriptases deposited on nanostructures onto the layered semiconductor MoTe_2 will be presented and the charge transfer into and through the biological molecules will be discussed based on an energetically resonant injection mechanism and the hypothesis of hydration-induced detrapping of electrons along the energetic landscape of polypeptides.

11:30

Invited

STM studies of surface properties of conducting polymers

Robert A. Nowakowski¹, Tomasz Jaroch¹, Marek Knor¹, Rafał Pokrop², Małgorzata Zagórska², Adam Pron³

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Since 1997, when Heeger, Shirakawa and MacDiarmid et al have discovered high electrical conductivity of doped polyacetylene, organic conjugated systems have been subject of extensive interest. Nowadays, conjugated polymers are applied in organic electronic devices, like light emitting diodes (OLED) or field effect transistors (OFET), to give few examples. Principal advantages of organic electronics consist of facile processibility of organic semiconductors and of the possibility of precise tuning of their electronic and/or optoelectronic properties by the modification of the molecular structure. However, the next important factor which has to be taken into account is their supramolecular organization. Low dimensional character of conjugated macromolecules combined with their specific supramolecular organization may result in anisotropy of the charge carriers mobility.

Recent results concerning STM studies of the supramolecular organization of poly(4,4''-diocetyl-2,2':-5',2''-terthiophene) - a very promising polymer for the construction of OFETs will be presented. The post-reaction mixture was fractionated into fractions of low polydispersity. Then, the monolayers prepared for each fraction were imaged in molecular resolution. By this approach, the transformation of the film structure, caused by the increase of molecular weight (corresponding to the elongation of conjugated chain) could be observed. Shorter macromolecules form well defined two-dimensional islands. Interactions between longer macromolecules become anisotropic. As a consequence the supramolecular organization is columnar and consists of more or less separated columns of ordered macromolecules. The conductivity of organic films is a compromise between two counteracting factors: extension of the conjugation length and significantly more difficult supramolecular ordering expected for longer macromolecules. The presented discussion based on STM results is therefore of interest for potential applications.

12:00

Oral

Synthesis, characterization and hydrogen storage properties of Mg-Ti-Ni system, produced by different ball milling techniques

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Magnesium is considered as one of the most promising material for hydrogen storage, because of its high theoretical hydrogen capacity (7.6wt.% in MgH_2), low price and low specific energy. The poor sorption kinetics, fast oxidation as well as high thermodynamic stability of MgH_2 ($\Delta H = 75 \text{ kJ/mol}$), decrease its maximum capacity, cycle life and therefore make practical utilization very difficult. Improvement in hydrogen absorption properties have been achieved by different approaches, mainly by (i) surface modification of Mg by fluoride treatment, nanocrystalline/amorphous microstructures, (ii) addition of

alloying elements and (iii) formation of composite materials with different catalyst such as metals and oxides or another hydride forming alloy or intermetallic compound. Hydrogen storage properties of composite materials mainly depend on the interaction between the different components present in the system. Mechanical milling leads to improvement of the sorption kinetics and thermodynamic properties by reducing the crystalline size. Hydrogen storage in the Mg-Ti-Ni system is presented here. Low temperature desorption and high hydrogen capacity have been found and reported for this system. Here $(\text{Mg}_{0.8}\text{Ti}_{0.2})_2\text{Ni}$ powders were ball milled under low and high hydrogen pressures using different milling conditions as duration of the milling process and ball to powder ratio. Cryomilling was performed in argon atmosphere as well. Powder X-ray diffraction analysis was carried out to investigate the structural changes as a function of the milling time. The microstructure, particle size and chemical composition were characterized with SEM and EDX analysis. Hydrogen absorption/desorption behavior as well as the cycling stability of the composites were examined using the Sieverts method at different temperatures. Financial support from the NANOMAT program in the Research Council of Norway is acknowledged.

Lunch break

Wednesday afternoon, 19 September, 12:30

Session 8

Wednesday afternoon, 19 September, 14:00

14:00 Invited

Local monitoring of surface chemistry with Raman spectroscopy

Andrzej Kudelski

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In this talk some examples of local Raman studies of molecules adsorbed on various catalysts and metal electrodes will be presented. First, examples of standard Raman microscopic measurements with spatial resolution of about one micrometer will be shown. In many experiments we are, however, able to record spectrum dominated by the contribution from molecules adsorbed on the area significantly smaller than the area actually illuminated by the laser beam. Such measurements are possible, for example, on surfaces covered with the metal nano-resonators. Theoretical calculations of the efficiency of Raman scattering for molecules adsorbed at the system of metal nano-resonators showed that the efficiency of Raman scattering for molecules adsorbed at so-called "hot spots" can be even ten orders of magnitude higher than the efficiency of Raman scattering for molecules adsorbed at "cold zones". Extremely large Raman enhancements are accomplished with a rather high spatial confinement. Therefore, although there is a very large number of adsorbed molecules in the focal area of the Raman microscope (about one square micrometer), in many cases it is possible to measure Raman spectrum dominated by the contribution from only a few semi-randomly chosen molecules (clusters). Some practical applications of "a few molecules" surface-enhanced Raman scattering (SERS) measurements will be presented. The mechanism of very large efficiency of Raman scattering for some molecules (clusters) will be also analysed. In the last part of the talk the combination of Raman spectroscopy and scanning probe microscopy (STM or AFM) - so called tip-enhanced Raman spectroscopy (TERS) - will be described. In this technique only the Raman scattering

of the molecules which are very close to the tip is enhanced. Therefore, TERS has both very large spectroscopic and spatial sensitivity.

14:30 Invited

K-Alpha, a new approach to X-ray photoelectron spectroscopy (XPS).

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X-ray Photoelectron Spectroscopy (XPS) is a powerful surface analysis technique. It provides quantitative information about the elemental and chemical state composition of the first few monolayers of a material. When combined with ion beam sputtering, compositional depth profiles can be obtained from a few micrometers. The method has applications in many fields, including polymers, biomaterials, glasses, metals catalysts, and semiconductors.

Until now, the instrumentation has been expensive to acquire and has needed an expert to determine the most appropriate analytical conditions. These two factors make the cost of ownership very high. Many scientists, who would benefit from this type of analysis, are deterred from using the technique.

In this symposium, it will be shown that, by the use of modern manufacturing techniques, novel technology and advanced software design, XPS has become available to all analysts. High sensitivity combined with extensive automation maximises throughput. Automation in data processing and reporting allows the analyst to obtain maximum use of the instrument, avoiding the need for repetitive and time-consuming activities. XPS has become a multi-user analytical method.

15:00 Oral

Electrodeposited tungsten and molybdenum amorphous/nanocrystalline alloys. Deposition and properties.

Mikolaj Donten, Zbigniew J. Stojek

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Amorphous and nanocrystalline materials are frequently used in contemporary engineering. Glassy metals offer unique properties such as high hardness, small abrasive wear, and increased corrosion resistance. Amorphous / nanocrystalline alloys containing tungsten and molybdenum are especially interesting, because in addition to the mentioned properties they exhibit useful magnetic properties. Finally they are extensively investigated.

The physical and chemical properties of amorphous alloys of either tungsten or molybdenum and one or more iron-group metals [1-3] make those alloys an alternative for the chromium plating. If the protective coatings are obtained by the electroplating processes, and contain a relatively high percentage of tungsten or molybdenum they will outperform hard chrome deposits. Those deposits also exhibit a good resistance to strong oxidizing acids such as nitric and hydrochloric acids, unlike the hard chrome which corrodes rapidly when it is in contact with these corrodents.

The obtained X-ray diffractograms and STM micrographs indicate that the tungsten amorphous alloys consist of closely packed nanofibers oriented perpendicularly to the substrate surface. For the molybdenum alloys, the amorphous character (grain size in the range of tens of nanometers) is obtained for its content higher than circa 20 at %. The

amorphous Ni-Mo alloys never formed nanofibers, what was detected for the Ni-W alloys.

For a complete evaluation of this kind of alloys a reliable corrosion study were added to its characterization.

References

1. M. Donten, Z. Stojek, H. Cesiulis, J. Electrochem. Soc., **150** (2003) C95
2. Donten M., Cesiulis H., Stojek Z., Electrochim. Acta, **45** (2000) 3389-3396.
3. Donten M, Cesiulis H., Stojek Z., Electrochimica Acta, **50** (2005) 1405-1412.

Coffee break

Wednesday afternoon, 19 September, 15:30

Joint Poster Session (Part II)

Poster Award Ceremony

Wednesday afternoon, 19 September, 15:50

Thursday, 20 September

Session 9

Thursday morning, 20 September, 9:30

Coffee break

Thursday morning, 20 September, 10:30

Session 10

Thursday morning, 20 September, 11:00

11:00 Oral

Self-organization in nonlinear dynamical systems and its relation to the material science

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Dissipative structures, i.e. oscillations, multistability and related phenomena occurring under non-equilibrium conditions are a result of the loss of stability by the steady-states, caused by the feedback loops in the processes determining the dynamics of such systems. Such self-organization can manifest itself as a function of either only time coordinate (simultaneous oscillations of the entire system's state), or only spatial coordinate (e.g. Turing patterns), or both coordinates (traveling chemical waves). The universality of such phenomena in various systems, at the level of their mathematical description, is remarkable.

Following the basic characteristics of such nonlinear dynamic phenomena their selected examples, studied in our lab for recent years, will be presented. The electroreduction processes of complex compounds of nickel(II) and of mercury ions at Hg electrode will serve as the examples of the oscillatory changes of state, bistability and (rarely experimentally reported) tristability. The universal self-organization scheme of the convective cells into hexagonal and fingerprints-like patterns

will be shown for the electrohydrodynamic convection in the thin-layer electrolytic cells, with the luminescence distribution showing the geometry of the fluid flows. Finally, the catalytic oxidation of thiocyanates with hydrogen peroxide will be shown as a source of traveling chemical waves, here visualized by the spatial distribution of luminescence of luminol.

The relation of such type of nonlinear phenomena to the material science will be further outlined based on selected examples. The examples will include, among others, the effect of the self-organized convection on the morphology of the surface of cathodically deposited copper, the formation of other surface (micro)patterns, as well as polymerization reactions.

11:30

Oral

Catalysts for carbon dioxide methanation

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In global carbon dioxide recycling for prevention of global warming and supply of renewable energy, fluctuating power generated by solar cells in remote deserts will be converted to H₂ by seawater electrolysis and then to methane by the reaction of H₂ and CO₂ at desert coasts, so as to use currently existing infrastructures for natural gas for transportation and combustion. One of the authors (KH) and his coworkers found effective catalysts derived from amorphous Ni-Zr-rare earth element alloys for hydrogenation of CO₂ with extremely high reaction rate and almost 100% selectivity. The high activity was attributed to the formation of nickel supported by tetragonal zirconia in contrast to the low activity of nickel supported by monoclinic zirconia. Although the stable polymorph is monoclinic, tetragonal zirconia is stabilized by doping of rare earth elements in zirconia lattice. Because amorphous alloy precursors are not suitable for mass production, we developed conventional method for preparation of the catalysts consisting of nickel supported by tetragonal zirconia and evidenced the effectiveness of such catalysts. The present work aimed to determine suitable composition and preparation method for catalysts composed of nickel supported by samarium-stabilized tetragonal zirconia. Zirconia sol with samarium nitrate and nickel nitrate were calcined at 500°C. The oxide mixtures were treated in hydrogen stream at 300°C for 5 h to form the catalysts. X-ray diffraction revealed that the catalysts consisted of metallic nickel supported by tetragonal zirconia. The methanation performance of the catalysts was examined passing a gas mixture of CO₂ and H₂ (1:4 volume ratio) at an atmospheric pressure through the catalyst. After reaction the gas was analyzed using a gas chromatograph equipped with a thermal conductivity detector. The catalyst with the best performance was determined by changing the ratio of nickel to oxide and that of zirconium to samarium in the oxide.

11:50

Oral

Acetylene Hydrochlorination Over Mechanochemically Pre-Activated Metal Complex Catalysts

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Solid phase mechanochemical synthesis and mechanoactivated reactions can be considered as one of the rapidly developing trends in the framework of green chemistry [1]. Mechanochemical methods open up high possibilities in heterogeneous catalysis: in particular, compounds regarded as catalytically inert could be involved in catalytic reactions in such a way [1, 2]. Active sites of mechanoactivated catalysts are usually associated [1] with defects in solids. Such states are metastable due to retarded diffusive mobility in solids, and their surface concentrations can essentially exceed the equilibrium concentration as well as that which can be obtained by conventional methods of heterogeneous catalysts activation. However, despite obvious advantages, a progress in this field is strictly restrained by limited data on pathways of mechanical energy transformation into structural defects of different pattern lattice. Most papers dealing with mechanoactivated catalysis are restricted to phenomenological description of the observed effects. In order to clarify the question it is convenient to use relatively clear catalytic systems. For such a model reaction we chose the catalytic acetylene hydrochlorination. The results of reactions mechanisms studies for heterogeneous catalytic acetylene transformations over mechanically activated platinum and palladium complexes will be presented and discussed. The main attention will be paid to the nature of active sites formed by mechanical pre-activation of the catalysts. References: 1. Butyagin P.Yu., Russian Chem. Rev. 63 (12) (1994) 1031. 2. Mitchenko S.A., Khomutov E.V., Shubin A.A., Shul'ga Yu.M.// J. Mol. Catal. A: Chemical. 2004. V.212. P.345-352.

12:10 Oral

Combined SRPES and SPM on selected Nanostructures on Silicon

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The use of synchrotron radiation photoelectron spectroscopy (SRPES) in combination with scanning probe microscopy (SPM) allows chemical, electronic as well as structural analyses of (photo)electrochemically modified Si surfaces. Chemical analysis by SRPES demands high quality processing to reduce ambient air contaminations which is done using a specifically designed transfer vessel which allows *in-system* investigation of electrochemically treated samples. We describe this system and apply it to the investigation of the surface transformation of Si(111) from H-termination to step bunching in alkaline electrolytes. Spectral deconvolution of the Si 2p core level lines allows assessment of the predominant dissolution route of Si in alkaline solution. In an extension of these experiments, electrodeposition of Pt onto the H-terminated and the step bunched surfaces is investigated with both, SRPES and AFM. Whereas the H-terminated surfaces are in flatband situation, the step bunched structures are in accumulation with a carrier density of the order of $3 \times 10^{18} \text{ cm}^{-3}$ on a background doping of $\sim 10^{15} \text{ cm}^{-3}$. Pt electrodeposition is accompanied by silicon oxide formation, evidenced by SRPES. On step bunched surfaces, Pt dendrites are found at the step edges of the nanostructure and similarly sized dendrites are found on H-terminated surfaces. Due to the oxide growth, the underlying atomic step structure can not be resolved anymore and it is not clear whether these Pt islands grow on specific sites at the H-terminated surface. The simultaneous oxidation of the surface during Pt deposition is interpreted by hole injection into the valence band from Pt species.

Koji Hashimoto: Concluding Remarks

Thursday afternoon, 20 September, 12:30

Closing Symposium D

Thursday afternoon, 20 September, 12:50

Lunch

Thursday afternoon, 20 September, 13:00

Posters

Monday, 17 September

Joint Poster Session (Part I)

Monday & Wednesday

Monday afternoon, 17 September, 15:50

15:50 Poster D-1

Corrosion Behaviour of Iron-based Bulk Metallic Glasses

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Great effort has been devoted for the last two decades to the development of Fe-based BMGs and for the last three years successful preparation of alloys with high glass forming ability (critical diameter higher than 10mm) and interesting combination of properties were reported. An important aspect for the applicability of these alloys is their chemical stability in different aggressive media. In this study the $(\text{Fe}_{44.3}\text{Cr}_5\text{Co}_5\text{Mo}_{12.8}\text{Mn}_{11.2}\text{C}_{15.8}\text{B}_{5.9})_{98.5}\text{Y}_{1.5}$ alloy was characterized from this perspective. Amorphous samples of this composition were obtained by copper-mould casting in an argon atmosphere and analyzed regarding their microstructure and thermal behaviour. Their anodization behaviour in aqueous environments with varying pH values was studied by electrochemical polarization techniques in combination with surface analytical investigations, i.e. HRSEM, EDX, AFM. For a more complete understanding electrochemical impedance spectroscopy was also used. The results are discussed in terms of chemical composition and structure influence on dissolution/passivation mechanisms and, more generally, on corrosion behaviour of these alloys.

15:50 Poster D-2

Improvement effects by solution heat treatment of 5083F alloy

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The FRP ships, including small fishing boats, have many environmental and recycling problems, and there is no method to decommission an FRP ship. If aluminum were used as a substitute for FRP, the result would be more environmentally friendly vessels. Therefore, the Al alloy was carried out to prevent corrosion with solution heat treatment. The optimal heat treatment involved heating specimens for 120 minutes at 420 °C. In addition, the optimal aging condition involved aging specimens for 240 min. at 180 °C. The SSRT in seawater revealed that

heat treatment under optimal conditions produced improved elongation, time-to-fracture and amount of dimples.

This research was financially supported by the Ministry of Commerce, Industry and Energy (MOCIE) and Korea Industry Technology Foundation (KOTEF) through the Human Resource Training Project for Regional Innovation.

15:50 Poster D-3

Corrosion protection characteristics in application of various thermal spray coating materials for STS304

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The optimum conditions for various thermal spray methods used to protect ship and marine structures in a seawater environment were investigated from the viewpoint of microstructure and electrochemical methods. This study investigated the corrosion protection characteristics of Zn, Al, and Zn-15%Al spray coatings for STS 304. If STS 304 is exposed to seawater owing to a defect such as porosity or an oxide layer, the STS 304 and the thermal spray coating materials act as an anode and cathode, respectively. Tafel analysis showed that the corrosion current densities of STS 304 and with Zn, Al, and Zn-15%Al thermal spray coatings were 7×10^{-7} , 2.4×10^{-5} , 7×10^{-6} , and 1×10^{-5} A/cm² respectively. The corrosion current densities recorded in the Tafel experiment were lowest in Al-coated specimens. In the galvanic cell that is formed by the thermal spray coating layer and STS304, the corrosion protection characteristics and the lifetime of the thermal spray coating layer were estimated, as the corrosion potential decreases with increasing corrosion current density.

15:50 Poster D-4

The investigation on optimum corrosion protection potential determination of Al alloy in marine environment

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The Al alloy is environmental friendly, easy to recycle, and provides a high added value to fishing boats. We report a number of electrochemical experiments undertaken to determine the optimum conditions for protecting 5083 Al alloy in seawater. The current density in the cathodic polarization increased with the potential shifts to a more negative value. Current densities in $-1.8 \sim -2.4$ V increased abruptly with any shift toward the active direction. On shifting in more anodic and cathodic directions outside of the $-1.4 \sim -0.7$ V, the current density increased suddenly. It was concluded that this potential range offered optimal protection potential. This research was financially supported by the Ministry of Commerce, Industry and Energy (MOCIE) and Korea Industry Technology Foundation (KOTEF) through the Human Resource Training Project for Regional Innovation.

15:50 Poster D-5

Characteristics evaluation for stress corrosion cracking and hydrogen embrittlement of Al-Mg alloy in seawater

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The fiber-reinforced plastic (FRP) ships, including small fishing boats, have many environmental and recycling problems, and there is no method to decommission an FRP ship. If aluminum were used as a substitute for FRP in ships and boats, the result would be more environmentally friendly vessels that are easy to recycle and have the added value of reduced fuel consumption, higher speed, and increased load capacity. In this study, we investigated the mechanical and electrochemical properties of aluminum alloy. Aluminum alloy do not corrode due to the formation of an anti-corrosive passive film, such as Al₂O₃ or Al₂O₃·3H₂O, which resists corrosion in neutral solutions. In seawater, however, Cl⁻ ions destroy this passive film. The optimum protection potential range with regards to hydrogen embrittlement and stress corrosion cracking was determined to lie between -1.1 and -0.7 V (vs. Ag/AgCl). These results can be used as reference data for ship design.

This research was financially supported by the Ministry of Commerce, Industry and Energy (MOCIE) and Korea Industry Technology Foundation (KOTEF) through the Human Resource Training Project for Regional Innovation.

15:50 Poster D-6

Characteristics evaluation with spray distance in Al thermal spray coating for stainless steel

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The cathodic protection has been widely adopted as the more effective protection technique in a marine environment. Thermal spray coating technology is a kind of sacrificial-anode cathodic protection method, since differences in the electrical potential of materials are employed. If a base metal (STS304) is exposed to seawater owing to a defect in the thermal spray coating, the base metal is protected by the sacrificial anodic characteristics of the coating layer. This is because a micro-cell is formed by the base metal and the thermal spray coating layer that respectively act as the cathode and anode electrochemically. As regards optimal spray distance, surface morphology was not found to make a great difference, but a spray distance of 80 mm seemed to give a dense coating. Clear differences in the thickness of the coating were observed in cross-sections, which verified that with increased spray distance there was more porosity in the coating layer resulting from oxygen and impurities in the air.

15:50 Poster D-7

The Effect of Hydrostatic Extrusion on Resistance of 316 Austenitic Stainless Steel to Pit NucleationMarcin Pisarek^{1,2}, Piotr Kędzierzawski², Maria Janik-Czachor², Krzysztof J. Kurzydłowski¹**1.** *Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland***2.** *Polish Academy of Sciences, Institute of Physical Chemistry, Kasprzaka 44/52, Warszawa 01-224, Poland**e-mail: marcinp@ichf.edu.pl*

The resistance to pit nucleation of an austenitic 316 stainless steel was investigated in an aggressive environment containing Cl⁻ ions in the as-received state, and after a heavy deformation by hydrostatic extrusion (HE) leading to nanostructurisation.

XRD and TEM were used to examine structural changes introduced by HE treatment. The corrosion resistance of 316 stainless steel was investigated by potentiodynamic polarization in a borate buffer + 0.1 M NaCl. The results show that the HE processed material exhibits an extended stable passivity region by about 0.4 V or more, as compared to that of the as received one.

This work was financially supported by the Polish Ministry of Education and Science through grant 3T08C 013 29. Surface characterizations were performed using a Microlab 350 located at the Physical Chemistry of Materials Center of the Institute of Physical Chemistry, PAS and of the Faculty of Materials Science and Engineering, WUT.

15:50 Poster D-8

Dissolution-precipitation of the secondary phase particles in copper-rich Cu-Cr alloys during high-pressure torsionSvetlana N. Faizova¹, Ruslan Z. Valiev², Natalya V. Balabanova²**1.** *Institute of Mechanics, Ufa Scientific Center, 71, Prospect Oktyabrya, Ufa 450000, Russian Federation* **2.** *Institute of Physics of Advanced Materials, Ufa State Aviation Technical University (IPAM USATU), 12 K. Marx st., Ufa 450000, Russian Federation**e-mail: snfaiz@mail.ru*

There exists growing experimental evidence that the SPD may accelerate diffusion and cause decay of a solid solution and precipitation of particles in its process. In their turn, the particles influence the plastic flow of the material. Such an interdependence may have an important effect on the resulting structure refining.

In the present work high-copper Cu-Cr alloy samples were treated with the high-pressure torsion (HPT) at the room temperature. After 5 turns the average grain size become ~ 90 nm and microhardness increases up to 2100 MPa from 620 MPa in the initial state. The decay of a solid solution has been observed. At the early stages of deformation the alignment of precipitation particles along the localized slip planes is evident. With the further development of the plastic flow relatively large (~1-2 μm) particles remaining in the material totally disappear and the number of nanometer sized ones (~10..20 nm) increases. This change may be caused by mechanical fragmentation or by the concurrent process of the particle dissolution. An evidence for the latter case is the significant increase of the microhardness value after post-deformation ageing. It rises from 2100MPa to 2700 MPa, i.e. by 600 MPa. For comparison, the same treatment of the alloy sample without the HPT provides an increase of the microhardness only by 300 MPa. Dissolution of precipitation particles is incompatible with the equilibrium state diagram for the alloy under investigation and may be linked

to the local dynamical distortions of crystalline lattice in the process of the intensive deformation.

This work was supported by RBRF grants (projects #06-08-00971 and #07-08-00567.)

15:50 Poster D-9

The optimum selection and protection characteristics with using impressed current cathodic protection for ship's shaft materialsSeong-Jong Kim¹, Seokcheol Ko²**1.** *Mokpo Maritime University, Chukkyo Dong, Mokpo 530-729, Korea, South* **2.** *Jeonnam Regional Innovation Agency (JINA), 1000 Namak-Ri Samhyang-Myun, Muan-Gun Jeonnam 534-700, Korea, South**e-mail: suntrac@jina.re.kr*

Stainless steels such as STS 304, 316 and 630 are frequently used as shaft materials in small FRP fishing boats. If the shaft material is exposed to a severely corrosive environment such as seawater it is protected using appropriate methods. We investigated the use of impressed current cathodic protection to inhibit corrosion in shaft materials. In anodic polarization, passivity was remarkably more evident in STS316 stainless steel than in STS304 and STS630. The pitting potentials of 304, 316, and 630 stainless steels were 0.30, 0.323, and 0.260 V, respectively. The concentration polarization due to oxygen reduction and activation polarization due to hydrogen generation were evident in the cathodic polarization trends of all three. STS316 had the lowest current densities in all potential ranges, and STS630 had the highest. Tafel analysis showed that STS316 was the most noble of the three. In addition, the corrosion current density was lowest in STS316.

15:50 Poster D-10

Characterization and modelling of a superconducting flux flow transistor with a three-channel structure by an atomic force microscope methodSeokcheol Ko¹, Seong-Jong Kim², Min-Su Han²**1.** *Jeonnam Regional Innovation Agency (JINA), 1000 Namak-Ri Samhyang-Myun, Muan-Gun Jeonnam 534-700, Korea, South* **2.** *Mokpo Maritime University, Chukkyo Dong, Mokpo 530-729, Korea, South**e-mail: suntrac@jina.re.kr*

The superconducting flux flow transistor (SFFT) with a three-channel was successfully fabricated by an atomic force microscope (AFM) technique. The current-voltage measurements were performed on drain current line with a three-channel, under the gate currents up to 6 mA. From the calculated and measured results, the current-voltage characteristics of an SFFT with a three-channel are discussed. The transresistance value was 0.017 Ω for I_d=221mA at I_g=1 mA.

15:50 Poster D-11

Amorphous Co-Mo cathodes for hydrogen evolutionWojciech Gagatek, Remigiusz Kowalik, Piotr Zabinski*AGH University of Science and Technology (AGH), al. Mickiewicza 30, Kraków 30-059, Poland**e-mail: zabinski@agh.edu.pl*

Tailoring of active cobalt alloy cathodes for hydrogen evolution in hot concentrated sodium hydroxide solution was attempted by electrodeposition. Enhancement of cathodic activity of cobalt for electrolytic hydrogen evolution has been carried out by the formation of cobalt alloys containing molybdenum.

The present work aims to prepare the Co-Mo alloys with high cathodic activity for hydrogen evolution by electrodeposition. Also we try to clarify the effect of the molybdenum addition in enhancing the high activity for hydrogen evolution and changes of electrodeposits morphology.

The hydrogen evolution activity of the electrodes was examined in 8M NaOH solutions at 90°C by the galvanostatic polarization curves measurement. The structure of the deposited alloys was identified by XRD. According to X-ray diffraction patterns electrodeposited of Co-Mo alloys with high molybdenum content are amorphous.

When the molybdenum contents in the deposit are low, the linear dependence between logarithms of current density and potential is observed (Tafel dependence). It suggests that the rate-determining step was proton discharge on the cathode surface. The hydrogen evolution is mainly the same as for pure cobalt. If the alloys contain high molybdenum content and the surface is mostly amorphous alloy and the activity for hydrogen evolution is much higher. For those alloys we do not observed Tafel dependence. It means that proton discharging is so fast that rate-determining step seems to be desorption of hydrogen from the surface of the electrode after recombination of two discharged protons.

The authors express their acknowledgment for financial support under grant No. 3 T08B 016 29.

15:50	Poster	D-12

Studies of H₂ uptake by aluminum carbide (Al₄C₃) using volumetric absorption and elemental combustion analyses and FT-IR

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Aluminum carbide (Al₄C₃) is a low atomic mass (144 u) and low density (2.36 g/cm³) compound and its hydrogen absorption ability has not yet been studied. Al₄C₃ consists of elements which form hydrides of large gravimetric hydrogen capacity: AlH₃ (10.1 wt. %) and CH₄ (25 wt. %). It was therefore presumed that heterolytic addition of the dihydrogen molecule (H₂) to Al₄C₃ (with the concomitant formation of the Al-H and C-H bonds) might be possible. Thermodynamic analysis of various possible reactions showed that full degradation of Al₄C₃ to CH₄ and AlH₃ (or metallic Al) is not thermodynamically privileged.

Here we present our experimental studies on the ability of Al₄C₃ for the absorption of H₂ in a broad range of [p, T] conditions, with or without of the hydrogenation catalysts (Ti, TiH₂). Hydrogenations have been carried out at various conditions, from mild (T = 30°C, p = 50 bar) to very aggressive (T = 450°C, p = 100 bar). Doping with catalysts (~10 mol. %) was achieved by high-energy disc-milling (5 min, tungsten carbide mill) under an inert gas atmosphere. Hydrogen uptake has been scrutinized by volumetric absorption and elemental combustion analyses, supplemented by infrared spectroscopy (FT-IR).

None of the samples studied have shown any significant uptake of hydrogen. Elemental analysis suggests that the samples are virtually H-free (method's sensitivity is 0,2 wt. %). IR spectra do not exhibit bands attributable to the Al-H, C-H and Ti-H stretching modes. Ti and TiH₂ do not serve as H₂-transfer catalysts to Al₄C₃. Aluminum carbide does not absorb any significant quantities of hydrogen (even under harsh [p, T] conditions) and therefore it could serve as a lightweight protective shield or seal against compressed H₂.

15:50	Poster	D-13

Synthesis of Micro- and Nano-Sized LiNi_{0.5}Mn_{1.5}O₄ and LiNi_{0.5}Mn_{0.5}O₂ Materials and Studies of Their Electrode Behavior in Li-Ion Cells

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In the present work, we compare the electrochemical behavior of LiNi_{0.5}Mn_{1.5}O₄ and LiNi_{0.5}Mn_{0.5}O₂ electrodes prepared from micro- or nano-particles of the active material, in Li-ion cells. The electrochemical performance in terms of capacity, rate capability, electrode impedance, stability at elevated temperature, and ageing of the nano-materials in solutions, was compared to that of micro-particles. Nano-particles of the layered LiNi_{0.5}Mn_{0.5}O₂ and spinel LiNi_{0.5}Mn_{1.5}O₄ materials were synthesized by a modified self-combustion reaction, and micron sized particles were prepared via a solid-state reaction method.

It was established that stable electrochemical behavior could be obtained during prolonged cycling of these electrodes even at elevated temperatures (60°C) in LiPF₆/alkyl-carbonates solutions. We have found that nano-LiNi_{0.5}Mn_{0.5}O₂ and nano-LiNi_{0.5}Mn_{1.5}O₄ electrodes demonstrate a faster kinetics, more reversible electrochemical behavior, and superior capacity retention at high rates compared to their micro-sized counterparts.

We proposed that a stable electrochemical behavior of nano-LiNi_{0.5}Mn_{0.5}O₂ electrodes under conditions of high temperature cycling/ageing and high oxidation potentials (> 4.5 V), may be related, in part, to stabilization of the electrode/solution interphase by developing surface films. The latter contain polycarbonates, LiF, Ni and Mn oxides and fluorides. An important finding is nearly constant impedance of nano- and micro-LiNi_{0.5}Mn_{0.5}O₂ electrodes during ageing and cycling after these electrodes being stabilized at elevated temperatures. It was demonstrated that ageing of nano-particles proceeded with a partial Ni and Mn dissolution from LiNi_{0.5}Mn_{1.5}O₄, as a result of which the complex domain structure of nano-particles is changed in terms of a more homogeneous Ni²⁺-to-Mn⁴⁺ distribution. For micro-particles, the partial dissolution of Ni and Mn was found to be limited.

15:50 Poster D-14

AgF₂ as a 2D scaffold of the organic-inorganic hybrid materials with tailored magnetic properties? Probing reactivity of silver(II) fluoride towards a variety of inorganic and organic salts.Przemysław Malinowski¹, Wojciech Grochala^{1,2}

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High-temperature superconductivity in fluoroargentates(II) has been postulated in 2001 [1]. AgF₂, a fascinating binary fluoride of Ag(II) and a powerful oxidizer, contains puckered [AgF₂] sheets and it exhibits ferromagnetic ordering below 165 K. Unfortunately, ferromagnetism is usually detrimental to the appearance of superconductivity. In this account we describe attempts of the formation of the inorganic-inorganic and inorganic-organic hybrid materials with well-defined electronic two-dimensionality. We target materials with flat [AgF₂] sheets, to promote antiferromagnetic ordering, which is beneficial for superconductivity.

We have studied experimentally the solid-solid reactions between AgF₂ and a variety of organic (salts of perfluorinated carboxylic acids and perfluorinated alcohols) and inorganic compounds (including oxides and chlorides of selected transition metals). For many systems we observe redox reactions with the concomitant oxidation of respective anions even at quite low temperatures (100 °C). The potential of organic and inorganic compounds to form a hybrid with AgF₂ is therefore limited by their redox properties.

We do not observe the formation of hybrid materials at the conditions of experiments performed. We therefore propose an alternative wet approach based on generation of a metastable beta form of AgF₂, i.e. a diamagnetic Ag(I)[Ag(III)F₄], and its in-situ exothermic transformation to AgF₂ in the presence of the hybrid-forming component. Such approach could hopefully lead to novel 2D fluoroargentates with tailored magnetic properties.

[1] W. Grochala, R. Hoffmann, *Angew. Chem. Int. Ed. Engl.* 40 (15): 2743-2781 **2001**.

15:50 Poster D-15

Electrochemical Synthesis of 5-Aminosalicylic Acid from 5-Nitrosalicylic Acid in Alkaline Media

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5-Aminosalicylic acid (5-ASA) is currently prepared by electrochemical reduction of 5-nitrosalicylic acid (5-NSA) in acidic media. In this work, electrolytic reduction of 5-NSA in alkaline media is presented. Reaction conditions and parameters such as anode and cathode electrodes, electrolyte type, reaction time, temperature, current, and the pH for isolation of product are investigated. The yield of the reaction was 90.9% and the melting point was 280 °C with decomposition. The UV-Vis and FT-IR spectra of the product was in good agreement with the standard material. The elemental analysis of the product was almost the same as the standard.

Key Words: 5-aminosalicylic acid, 5-nitrosalicylic acid, electrochemical synthesis, elemental analysis, reduction.

15:50 Poster D-16

Structure and properties of the multilayers produced on Inconel 600 by PACVD method with the participation of trimethylaluminum vapoursRyszard Sitek¹, Krzysztof Sikorski¹, Janusz W. Sobczak², Tadeusz Wierchoń¹

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The paper presents the results of examinations of the structure and properties of the Al₂O₃+(NiAl+Ni₃Al)+Ni(Cr,Fe,Al)+(Cr-Ni+Cr₇C₃) layers produced on the nickel alloys by the PACVD method with the participation of trimethylaluminum vapours in gaseous atmosphere. The layers have a high surface hardness of about 1300 HV0.05, and high heat and frictional wear resistance. In order to find the correlation between the structure of the multilayers and their properties, the following examinations have been executed: phase analysis by XRD and XPS, analysis of microstructure by optical and scanning electron microscopes as well as X-ray microanalyser, micro-hardness measurements, corrosion and frictional wear resistance testing.

15:50 Poster D-17

Development and characterization of bioelectrocatalytic systems for oxygen reduction

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One of the most frequently considered enzymes for use as the cathodic biocatalyst in biofuel cells is laccase. It belongs to the multi-copper group of oxidases, and drives the four-electron reduction of the co-substrate O₂ to H₂O effectively, while it is quite unspecific with respect to the electron donating substrate. The dianion ABTS²⁻ (2,2' - Azinobis (3 - ethylbenzothiazoline - 6 - sulfonate)) undergoes a reversible oxidation - reduction reaction and is commonly used with oxygen reducing enzymes as a mediator. ABTS was mostly considered as a diffusional mediator but recently attempts have been made to immobilize ABTS together with laccase on the electrode surface. Immobilization of the enzyme and mediator eliminates the need to separate the anode and cathode compartments with a membrane.

In order to improve the effectiveness of the bio-electrocatalytic system for the electro-reduction of oxygen, we have employed carbon nanotubes (CNTs). Carbon nanotubes exhibit very good mechanical and electronic properties and they facilitate electron transfer in bioelectrocatalytic systems. We have prepared colloidal suspensions of carbon nanotubes modified with ABTS utilizing the procedure proposed by us for the fabrication of phosphomolybdate-stabilized carbon nanotubes [1]. The bioelectrocatalytic film based on CNTs_ABTS and laccase exhibits very good electrocatalytic activity towards oxygen reduction. The combination of CNTs and ABTS gives a highly effective mediation system able to propagate charge between the active center of the enzyme and the electrode surface.

15:50 Poster D-18

Structural and Electric Field-Induced Properties of the narrow-Gap Semiconductor ZnCdHgTe Thin FilmsHalyna M. Khlyap¹, Petro G. Sydoruk, Jacek Polit², Olha M. Pihur³

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Narrow-gap semiconductor solid solutions ZnCdHgTe are of particular interest as an alternative to worldwide known compound CdHgTe. The abstract presents some experimental results on structural and electric field-induced properties of the compound.

The samples were grown on insulator substrates by the pulse laser deposition technology. X-ray diffraction analysis showed a good quality of the deposited films. Transmission line method was applied to measure the surface resistance of the grown layers. Transmission line method gives the value of contact resistance $3 \cdot 10^4$ Ohm, the surface resistance is $2,5 \cdot 10^5$ Ohm, the value of contact line $l_c = 2.5$ mm. Ni contacts are performed by physical vapor deposition technique.

Electric field-induced properties were investigated for as-grown films (at the room temperature and under the applied bias from 0 to 1.0 V) and for the samples underwent a heat treatment in hydrogen during 1 h at $T = 300$ K. In the first case experimental current-voltage characteristics revealed space-charge-limited currents under all range of applied voltage. In the second case the current-voltage dependencies were changed. Some parameters of the investigated structures as a whole as well as that for the films are calculated. The nature of this behavior is also discussed.

15:50 Poster D-20

Non-equilibrium phase composition of ZnNi alloys prepared by electrocrystallizationMarek S. Michalec¹, Zbigniew Świątek², Piotr Ozga²

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Electrodeposited zinc coatings are widely used for corrosion protection of steel. Apart from chemical composition, the corrosion resistance of Zn-Ni coatings depends mainly on their microstructure, phase composition and structural parameters (texture and crystal lattice imperfections). The zinc-nickel coatings were electrodeposited on steel substrates from sulfate solutions with the addition of sodium citrate Na_3Hcit ($\text{cit}=\text{C}_6\text{H}_4\text{O}_7$) and ammonium sulfate. The influence of electrolyte composition (different $[\text{Ni(II)}]/[\text{Zn(II)}]$ ratios, pH, buffer), cathode current density and hydrodynamic conditions on the chemical and phase compositions of coatings were determined.

From X-ray diffraction studies, it is evident that the composition of the phase occurring in as-deposited Zn-Ni alloys varies greatly with the deposition conditions and do not corresponds to those obtained at thermodynamic equilibrium. Moreover, various Zn-Ni phases appear with the same mean Ni content. For the deposits with Ni content up to 13% in Zn-Ni alloys electrodeposited in presence of acetate ions,

the meta-stable and distorted h-phase crystallizing hexagonally is dominating, whereas alloys with the highest Ni content consist mainly of the cubic g-phase. On the contrary in citrate solution practically single-phase (hexagonal h) deposits were obtained in whole range of Ni content. Deposited alloys are not in thermodynamic equilibrium and such phase situation changes with time, leading the whole system to the equilibrium state.

The aim of this paper is the structural characterization of the electrodeposited Zn-Ni phases in stable and meta-stable state. The investigations were carried out on the as-deposited, 4-years natural aged and annealed alloys. Structural changes of the hexagonal Ni-rich h-phase and Zn-rich cubic g-phase caused by crystallization and/or recrystallization processes are analyzed. The phase diagram of electrodeposited Zn-Ni alloys is proposed.

Symposium E

Welcome

Recent progress in research and fabrication of shape memory materials has opened the opportunity to develop novel smart systems, which are capable of sensing and actuating and thus may respond to their thermal, mechanical and magnetic environment in a controllable way. Meeting the demands of smart systems requires the development of new multifunctional materials, detailed material characterization, the development of models of material behavior and technologies for implementation.

The symposium intends to continue the tradition of a previous Symposium at E-MRS Fall Meeting 2005 emphasizing the basic physics, materials research and early engineering development in this rapidly evolving interdisciplinary field. Experts from the materials science, mechanics and application communities are encouraged to participate.

One focus will be on magnetic shape memory alloys and systems, which are currently a hot topic in materials science. These materials experienced a widespread research boom in the last decade as they exhibit a unique combination of thermoelastic, magnetoelastic and ferromagnetic properties. One example are the Heusler ferromagnetic shape memory alloys Ni-Mn-Ga, which reveal in bulk single crystalline form, e.g., the giant magnetostrain effect, the inverse effect of strain-induced change of magnetization and a large magnetocaloric effect. It is expected that the multiple transducing effects being present in ferromagnetic shape memory alloy materials will enable various new solutions for actuation and sensing applications.

In order to stimulate the dialogue between basic research and engineering, further sessions focus on the modeling of material behavior and the engineering of practical devices and systems. For many applications, the design and simulation of three-dimensional structures is of prime importance. This requires the development of suitable models for predicting the thermo-mechanical, magneto-mechanical or the fully coupled thermo-magneto-mechanical response of the material. Numerical field simulations using, e.g., the finite element method are demanded. Further emphasis is on the combined response of shape memory materials with other functional materials taking into account the constraints of state-of-the-art technologies. New trends in the application of shape memory alloys will be presented ranging from the medical field to the maturing field of microsystems technology.

Welcome to this interdisciplinary event covering material science, mechanics and applications.

Organisers

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Proceedings

Selected camera-ready manuscripts will be published in a Special Issue of the European Physics Journal - Special Topics. the manuscript length is limited to 6 printed pages for contributed and invited papers including

Figures and Tables. Details on the format can be found in the Internet: <http://www.epjst-journal.org>.

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Programme

Monday, 17 September

Opening & Plenary Session

Czochralski Award Ceremony & Acta Gold Medal Ceremony
Monday morning, 17 September, 10:00

Lunch break

Monday afternoon, 17 September, 12:30

Parallel Session

Shape Memory Alloys I
Monday afternoon, 17 September, 14:00

14:00	Invited

Nano-domained Martensite: Strain glass

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Martensitic transformation has attracted continual interest for a century, owing to its key role in hardening steels and in the functional properties of shape memory alloys. Martensitic transformation can be viewed as a long-range ordering of lattice strain (distortion of the parent lattice) below M_s , with strain being the order parameter. This is analogous to the ordering of magnetic moments in a ferromagnet or electric dipoles in a ferroelectric. The strain-ordering transition creates a low symmetry martensite phase characterized by a micron-sized hierarchical twin/domain microstructure.

The above “normal” martensite has been the research subject of our field to date. Here we show that, when doping point defect into a martensitic system beyond a critical value, there appears a hitherto unrecognized wide composition range in which an “abnormal martensitic state” comes into being. As the first example, Ni-rich $\text{Ti}_{50-x}\text{Ni}_{50+x}$ alloys for $x>1$ (here the excess Ni is regarded as point defect), which have been regarded as non-transforming so far, are shown to undergo a “strain glass transition” below a critical temperature T_g . Such a transition is characterized by the formation of *nano-sized* martensite domains instead of micron-sized hierarchical twins as the case of normal martensite. The strain glass transition is not accompanied by a change in the average structure, or a thermal peak in the DSC curves. It is a freezing of the nano-martensite domains. We show that the seemingly “non-martensitic” strain glass exhibits unexpected properties: shape memory effect and superelasticity, like a normal martensitic alloy, although there is no sign of a spontaneous martensitic transformation in such a system.

Being parallel to cluster-spin glass and relaxor, strain glass may provide a new horizon for martensite research.

1. S. Shampa, X. Ren, and K. Otsuka, *Phys. Rev. Lett.*, 95, 205702 (2005)

2. Y. Wang, X. Ren, K. Otsuka, *Phys. Rev. Lett.* □97□225703 (2006)

14:30

Oral

Localization Phenomena in NiTi Shape Memory Alloys - Experimental Analysis of Lueders-like Shear Bands in One- and Two-dimensional Loading Scenarios

Andreas Schäfer^{1,2}, Christian Großmann^{1,2}, Gunther Eggeler¹, Martin Wagner^{1,2}

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Deformation of pseudo elastic NiTi shape memory alloys is often associated with the formation of macroscopically observable Lueders-like shear/transformation bands. Within these bands, the stress-induced martensitic transformation occurs almost completely. Simultaneously, the adjacent material remains in the austenitic state. Macroscopic straining of a specimen proceeds via the movement of the martensite/austenite interfaces between transformation bands and (yet) untransformed regions. During cyclic loading, functional fatigue (i.e., microstructural changes that alter macroscopic mechanical behavior) occurs only in those regions subjected to the forward and reverse transformation. In consequence, deformation associated with transformation bands leads to a localization of functional fatigue and partitions a specimen (or an engineering component) into regions of different mechanical properties.

While these phenomena are in principle well-documented, the fundamental mechanisms during the formation and growth of Lueders-like transformation bands are not understood in detail. In this contribution, we present experimental results on the deformation behavior of pseudo elastic NiTi wires, ribbons and sheet material during quasi-static and cyclic loading. The formation and propagation of transformation regions is characterized by infrared thermal imaging, in-situ optical microscopy and Speckle interferometry for different specimen geometries (one-dimensional loading: tensile specimens; two-dimensional deformation: plate specimens with and without notches/holes). The combination of these experimental approaches, supplemented by detailed microstructural characterization of initial and deformed microstructures (grain size distributions, textures) allows a detailed analysis of microstructural, mesoscopic and macroscopic factors contributing to the formation of transformation bands with a particular emphasis on non-uniaxial load cases.

14:45

Oral

Origin of diffuse scattering appearing in iron-doped Ti-Ni shape memory alloys

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We have investigated diffuse scattering appearing in Ti-Ni based shape memory alloys to understand the origin of instability of the B2-type structure in the system. Transmission electron microscope observation and a first-principle electronic structure calculation have been made for the alloys with Ti-(50-x)Ni-xFe ($x = 6, 7, 8, 10$). Obvious first order

R-phase transformation is suppressed in the examined alloys. Still, they exhibit diffuse scattering in the electron diffraction pattern at incommensurate positions of $\langle \zeta \zeta 0 \rangle$ on cooling. The value ζ increases with decreasing temperature for all the alloys. It locks into the exact 1/3 for the alloy with $x = 6$, while does not for the alloys with $x = 7$ and more down to about 20 K (the lowest limit of the present study). The temperature at which the incommensurate scattering start to appear, T_1 , is close to the one at which the electrical resistivity exhibits a local minimum for all the alloys and the value ζ at T_1 decreases with increasing Fe content. For all the alloys, the value of ζ at T_1 is nearly the same as the length of the Fermi surface nesting vector, which is obtained by calculating generalized susceptibility of the B2-type structure along the [110] direction. From these results, it is most likely that Fermi surface nesting is responsible for the instability of B2-type structure in Ti-Ni based shape memory alloys.

15:00

Oral

The effects of the control variable on the stress-strain hysteresis loops in martensitic transitions.

Lluís Manosa¹, Erell Bonnot¹, Eduard Vives¹, Antoni Planes¹, Ricardo Romero²

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Ferromagnetic materials exhibit hysteresis loops when driven across a first order phase transition by sweeping an external control variable. While the equilibrium trajectories in the generalized force-displacement phase space are independent of the external control variable, there is no reason to expect that out-of-equilibrium hysteresis loops obtained when controlling the generalized force will be similar to those obtained when controlling the generalized displacement. To address such an issue, we have studied the hysteresis loops obtained for a Cu-Zn-Al single crystal which has been driven across its martensitic transition. Experiments have been performed using two different experimental devices: a commercial tensile machine, which controls the displacement while the load is monitored (strain-driven), and a specifically designed dead-load device which enables a fine control of the applied load while the displacement is continuously monitored (stress-driven). Results have revealed significant differences in the hysteresis loops depending on which control variable used. The strain-driven curves show re-entrant behaviour which is not observed in the stress-driven case and the dissipated energy in the stress-driven loops is larger than in the strain-driven ones. In situ optical microscopy has also revealed differences in the transformation mechanisms. The obtained results conform to the recent theoretical predictions proposed within the general framework of athermal systems.

15:15

Oral

Phase formation upon crystallization of the melt-spun high temperature shape memory alloys along ZrCu-Zr₂CuNi quasibinary cross-section

Georgiy S. Firstov¹, Yuri Koval¹, Philippe Vermaut², Richard Portier², Patrick Ochin³, Jan Van Humbeeck⁴

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It is known that Ni replacement of Cu in ZrCu high temperature shape memory alloy (HTSMA) results in the increase of the temperatures of the martensitic transformation (MT) [1]. Once the Ni content reaches 20 at.%, the temperatures of MT are becoming higher than the temperature of crystallization of the amorphous alloy with the same composition [2] and crystallization from the amorphous state in this case takes place through formation of B19' phase without any visible signs of the nanoscale phase separation.

Transmission electron microscopy (TEM) in the present study revealed that once the Ni content is not sufficient to increase the MT temperatures above crystallization one, the nanoscale phase separation plays important role. The increase of MT above crystallization like in [2], indeed, results in the preferential formation of B19' upon crystallization. DSC and X-ray diffraction were used to find a correspondence between kinetics of the crystallization and structural phase formation qualitatively detected by TEM.

References

- [1] Koval Yu.N., Firstov G.S., Delaey L., Van Humbeeck J., *Scr. Met. et Mat.*, v.31, No. 7, 1994, pp.799-802.
[2] Firstov G.S., Koval Yu.N., Van Humbeeck J., Portier R., Vermaut P., Ochin P., *Materials Science and Engineering: A, Volumes 438-440*, 25 November 2006, Pages 816-820

Coffee break

Monday afternoon, 17 September, 15:30

Joint Poster Session (Part I)

Monday & Wednesday

Monday afternoon, 17 September, 15:50

Tuesday, 18 September

Parallel Session

Ferromagnetic Shape Memory Alloys I

Tuesday morning, 18 September, 9:00

9:00

Invited

Recent topics in microstructure control by magnetic field in Ni₂MnGa and CoPt and new transformation behavior in Ti-Ni-Fe alloys

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Three topics related to solid-solid phase transformations will be presented. The first topic is related to ferromagnetic Ni₂MnGa. The condition for rearrangement of martensite variants by magnetic field in this alloy will be discussed quantitatively. In addition, we will show that a new phase (X-phase) appears in Ni₂MnGa prior to the formation of the intermediate phase on cooling. The parent to X-phase transformation is close to second order while the X to intermediate phase transformation is obvious first order. The second topic is related to microstructure control using magnetic field. In the topic, we will show that single variant state of ordered CoPt (tetragonal L1₀-type structure) can be obtained by an ordering heat-treatment under magnetic field. We also show that magnetic field is especially effective at the early stage of ordering for obtaining single variant state. The third topic is related to the so-called precursor phenomena in Ti-Ni-Fe shape memory alloys. In the topic we will show the existence of a commensurate phase, which inherits the microstructure of the incommensurate phase and is probably different from the R-phase. Furthermore, we will discuss how the anomalies observed in Ti-Ni alloys are related to the instability of the B2-phase.

9:30

Oral

Dynamic observation of field induced twin boundary and magnetic domain wall motion in bulk NiMnGa

Ryan Yiu Wai Lai, Jeffrey McCord, Nils Scheerbaum, Oliver Gutfleisch, Rudolf Schäfer, Ludwig Schultz

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A study of twin boundary movement in NiMnGa shape memory single crystals in connection with in-situ magnetic domain observation is presented. Time-resolved polarization microscopy, using a magneto-optical indicator film technique, was applied to record the magnetic domain pattern's reorganization during the process. No magnetic domain wall movement is observed during twin boundary motion, which is different from previously proposed model. Moreover, a dynamic actuation experimental setup with the ability to apply mechanical stress and alternating magnetic field perpendicularly is developed in cooperation with time-resolved microscopy. Reversible twin boundary motion activated in the frequency range up to 200Hz is directly observed. Hysteresis curves of magnetic field induced strain in different frequencies show the dependence of twin boundary motion with excitation field frequency. Funding through the DFG priority program SPP1239 is gratefully acknowledged.

10:00

Oral

Preparation of textured Ni₄₈Mn₃₀Ga₂₂

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NiMnGa alloys have gained large research interest because of their possible application as magnetic shape memory materials. This effect is caused by the movement of twin boundaries in a magnetic field. So far this effect has only been shown in single crystals. The preparation of single crystals is a long time and cost intensive process and there can be compositional changes along the crystal and segregations. That is why for technical applications there is a great interest in polycrystals. To expand this effect to polycrystals, directional solidification was applied in order to get a coarse grained, textured sample. The technique of stationary casting in a preheated ceramic mold mounted on a copper plate was chosen in order to have a heat flow from the sample to the bottom and therefore a directional solidification in the opposite direction. To achieve a higher mold temperature at cast an additional heater was built and used for the casting process. The transformation temperature was checked by DSC. Since the transformation temperature strongly depends on composition, chemical homogeneity along the sample axis was approved by using this method. The preferred growth direction was determined by EBSD. Further annealing, which is necessary for chemical homogeneity, coarsening of grains and stress relaxation, influences the texture. Investigations on the texture development during annealing were done.

10:00

Oral

Training, constraints, and high-cycle magneto-mechanical properties of Ni-Mn-Ga magnetic shape-memory alloys

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Magneto-mechanical experiments with a rotating magnetic field of 0.97 T were performed with a Ni-Mn-Ga single crystal. Periodic strains exceeding 1% were recorded over a hundred million magneto-mechanical cycles. The twin-microstructure of the cycled crystal was characterized using atomic force microscopy and magnetic force microscopy. In the center of the sample, no twin boundaries were found. At the sample edges, the microstructure consists of a dense twin pattern. The results are compared with previous experiments of differently trained crystals. It is distinguished between "less efficient training" which results in a nearly self-accommodated martensite and "efficient training" which results in a nearly single variant state. The evolution of twin-microstructure is discussed in terms of training, magneto-mechanical cycling, and extrinsic constraints imposed by the experimental setting. It is concluded that the response of a magnetic shape-memory alloy to an alternating excitation depends strongly on the initial twin-microstructure established through training. In particular, a less efficient

training results in a twin-microstructure which can adapt to extrinsic constraints resulting in continued large periodic magnetic-field-induced deformation. In contrast, the twin-microstructure of an efficiently trained crystal cannot adapt to extrinsic constraints resulting in early failure by fracture.

10:15

Oral

Hexagonal Co-rich and L₁₂ ordered precipitates in annealed Co-Ni-Al shape memory alloy

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Ferromagnetic shape memory alloys (FSMAs) are presently intensively studied because of their potential applications as smart materials. Martensitic transformations and lattice reorientation processes in FSMAs can be triggered not only by changes in temperature and stress, as in conventional paramagnetic SMAs, but also by applying a magnetic field. Recently, Co-Ni-Al alloys have attracted increasing interest as new FSMAs [1,2].

Conventional transmission electron microscopy and electron diffraction studies together with advanced microscopy techniques (HRTEM, EFTEM) were carried out in order to characterize the microstructure of annealed (1275°C/4h and 1100°C/72h) Co₃₈Ni₃₃Al₂₉. In addition to the major constituents (B2 type ordered β -phase and f.c.c. ordered γ -phase), rod shaped precipitates ranging from 20 to 75 nm of h.c.p. ϵ -Co were observed in the 1275°C/4h sample (Fig.1). EFTEM measurements confirm that the precipitates are Co-rich (Fig.2). The orientation relationship between the nanosized precipitates and the B2 matrix is found to be the Burgers orientation relation. Martensitic transformation to the tetragonal L₁₀ structure was observed at -40°C which was confirmed by in-situ experiment and DSC measurements. Large precipitates (Co,Ni)₃Al with size up to several microns were found in specimen annealed at 1100°C/72h. These precipitates show the L₁₂ ordered structure while a majority contain a single twin boundary (Fig.3).

References:

- [1] Karaca H.E. et al., 2004, Scripta Mater. 51, 261-266
- [2] Kainuma R. et al., 1996, Intermetallics 4, S151-S158

Acknowledgements: B. Bartova and S. Ignacova like to thank MUL-TIMAT "Multi-scale modeling and characterization for phase transformations in advanced materials", a Marie Curie Research Training Network (MRTN-CT-2004-505226) for supporting this work.

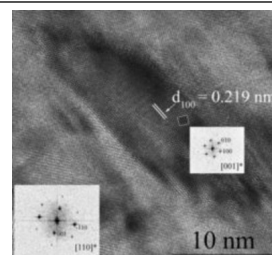


Fig.1

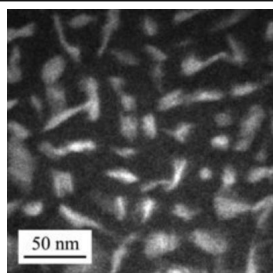


Fig.2

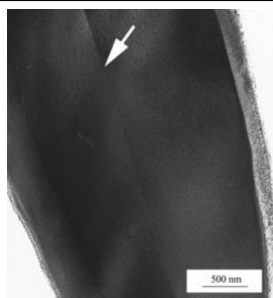


Fig.3

Coffee break

Tuesday morning, 18 September, 10:30

Parallel Session

Ferromagnetic Shape Memory Alloys II

Tuesday morning, 18 September, 11:00

11:00 Oral

Crystal structure of martensite and intermediate phases in Ni_2MnGa studied by neutron diffraction

Hiroaki Kushida¹, Koudai Fukuda¹, Tomoyuki Terai¹, Takashi Fukuda¹, Tomoyuki Kakeshita¹, Takuya Ohba², Toyotaka Osakabe³, Kazuhisa Kakurai³, Ken-ichi Kato⁴

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Ferromagnetic shape memory alloy Ni_2MnGa transforms from the L2_1 parent phase to a martensite (M-) phase via an intermediate (I-) phase. We have investigated crystal structures of M- and I-phases in stoichiometric Ni_2MnGa using neutron diffraction measurement with a single crystal specimen and synchrotron X-ray diffraction measurement with powder specimen. The neutron diffraction profile of the M-phase at $T = 100$ K exhibits four satellite reflections between $(0\ 2\ 0)_\text{P}$ and $(2\ 0\ 0)_\text{P}$ fundamental reflections (index is given on the basis of the parent lattice). They appear at incommensurate positions of $(h\ 2-h\ 0)_\text{P}$ with $h = 0.427, 0.861, 1.139$ and 1.573 , which are near $3/7, 6/7, 8/7$ and $11/7$, respectively. Intensities of four satellite reflections relative to the fundamental ones are approximately $1/10, 1/1000, 1/1000$ and $1/10$,

respectively. They move toward the fundamental reflection with increasing temperature. The neutron diffraction profile of the I-phase at $T = 210$ K exhibits two satellite reflections between $(0\ 2\ 0)_\text{P}$ and $(2\ 0\ 0)_\text{P}$ fundamental reflections. They appear also at incommensurate positions of $(h\ 2-h\ 0)_\text{P}$ with $h = 0.343$ and 1.657 , which are near $1/3$ and $5/3$, respectively. Intensities of these satellite reflections relative to the fundamental ones are approximately $1/100$. Temperature dependence of the satellite positions is negligibly small, while their intensities decrease with increasing temperature and disappear at $T = 250$ K. To determine atomic positions of Ni, Mn and Ga in M- and I-phase, powder synchrotron X-ray diffraction has been carried out. Rietveld analysis of the result revealed that atoms in both phases displace along $[1\ -1\ 0]_\text{P}$ from the positions in L2_1 structure. The displacements are represented by a sine wave whose propagation vector is parallel to $[1\ 1\ 0]_\text{P}$.

11:15

Oral

Crystal structures of “nM” modulated martensitic phases of MSM Ni-Mn-Ga Heusler alloys: precise determination and new viewpoints

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Ferromagnetic Ni-Mn-Ga Heusler alloys have recently attracted a great deal of interest due to the giant magnetic field-induced strain effect. The composition of Ni-Mn-Ga alloys determines both the temperature of martensitic transformations and the structure type of the product phase. Martensitic structural transformation consists in an uniform lattice distortion (the so-called Bain deformation) of the parent phase showing a cubic L2_1 -ordered structure. In addition, a periodic shuffling of the close-packed atomic planes, frequently called as a structural modulation can be observed. Depending on the number of satellites, s , between the main reflections of diffraction patterns, martensitic phases in Ni-Mn-Ga alloys have been associated to superstructures, involving 5, 7, 10 or even 12 lattice modulation periodicity and indicated as 5M, 7M, 10M and 12M, where $n\text{M}$ is determined by the relation $n=s+1$.

In the present work, the so-called “5M” and “7M” martensitic modulated structures occurred in Ni-Mn-Ga alloy system are thoroughly studied by a powder X-ray diffraction. The results made possible a detailed description of these structures in terms of symmetry, atomic coordinates, occupancy factors and thermal parameters. Our findings indicate the presence of incommensurate and commensurate modulation properly described within superspace approach. Moreover, the presented results clarify a series of critical points and discrepancies emerging from the literature dealing with the structural studies of Ni-Mn-Ga martensitic phases. A broadly used classification of martensitic modulated structures based on the number of observed satellites must be reconsidered. In fact, it will be demonstrated that the number of satellites does not always determine the type of possible martensitic superstructures.

11:30

Oral

Characterization of textured Ni-Mn-Ga polycrystals after plastic deformation

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Single crystals of Ni-Mn-Ga are reported to show magnetic-field-induced strain (MFIS) due to twin boundary motion (TBM). This mechanism is substantially different from the phase transformations responsible for the large strains in the conventional shape-memory alloys. A large uniaxial magnetic anisotropy, a low barrier against twin boundary motion, and an appropriate orientation of the twins are prerequisites to enable field-induced twin boundary motion. Both features depend not only on composition, but also on crystal structure and microstructure. In the last time different routes (hot forming process, bar extrusion) were studied to prepare textured polycrystals. The goal is to establish the conditions under which MFIS can be achieved in Ni-Mn-Ga polycrystals. The plastic deformation experiments of Ni-Mn-Ga samples encased by steel plates were successful above the ordering temperature up to $T = 1000^\circ\text{C}$. The hot deformed samples were characterized by means of DSC, XRD, electron microscopy using EDX and EBSD. The final thickness reduction of Ni-Mn-Ga samples was about 60 %. The influence of Fe-diffusion processes on the Ni-Mn-Ga samples was very small. The presence of martensitic twins can be clearly seen within the recrystallized grains. Modulated martensitic phases (crystal structure 7M or 5M) were found. The optimization of microstructure using different hot-rolling steps was performed. As result a strong grain aspect ratio in rolling-direction was found. Further, large grain growth can be obtained by heat treatments. The resulted grain size was approximately 500 μm . The measurements of orientational dependence of the magnetization have shown, that the perpendicular direction is the easy axis of magnetization. Further magneto-mechanical treatments ("training") were performed and are essential to obtain MFIS. The results will be used to prepare improved magnetically driven actuators by efficient preparation routes.

11:45

Oral

Magnetic Field-Induced Phase Transformation in NiMnGa and NiMnCoIn Ferromagnetic Shape Memory Alloys

Ibrahim Karaman¹, Haluk E. Karaca¹, Burak Basaran¹, Yuri I. Chumlyakov², Hans J. Maier³

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In recent years, magnetic shape memory alloys (MSMAs) have attracted interest because of the ability to obtain one order of magnitude higher magnetic field induced strain (MFIS) than magnetostrictive materials and few orders of magnitude faster dynamic response than conventional shape memory alloys. They can also be used for sensing and passive power generation due to significant changes in magnetization upon the application of fluctuating mechanical forces or displacements making them truly multifunctional. Previous studies have mainly fo-

cused on field-induced martensite variant reorientation being the governing mechanism for the magnetic field-induced shape change, especially in NiMnGa alloys, which has resulted in low actuation stress levels. We have recently shown that field-induced phase transition is possible in NiMnGa alloys and it can be reversible or irreversible depending on the magnitudes of stress hysteresis, magnetocrystalline anisotropy and saturation magnetization. Utilizing field-induced phase transformation in NiMnGa alloys instead of variant reorientation, more than one order of magnitude increase in actuation stress is achieved. An extensive experimental program is undertaken on NiMnGa and NiMnCoIn single crystals in quest for identifying physical and microstructural parameters critical in field-induced phase transformation phenomena. A thermodynamical framework is constructed and will be discussed to identify the effects of magnetic field on martensitic phase transformations. Using the thermodynamical model, guidelines to increase the actuation stress levels and possible future directions for research on magnetic shape memory alloys will be presented.

12:00

Oral

Acoustic-Assisted, Magnetic-Field-Induced Strain and Stress Output of Ni-Mn-Ga

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The effect of acoustic energy input from a piezoelectric actuator on the magnetic-field-induced strain (MFIS) of Ni-Mn-Ga ferromagnetic shape memory alloys (FSMAs) is observed to enhance the MFIS performance [1]. The threshold field required to induce twin boundary motion is reduced by this new technique by up to 1 kOe. The twinning-yield stress is also reduced by the technique by up to 0.5 MPa. The significant reduction in the twinning stress or the threshold field by the acoustic-assistance is beneficial to reduce the size of a FSMA actuator with a given output. To appreciate the benefit of the acoustic-assistance better, the stress and strain output of the FSMA actuation under acoustic-assist is studied.

In this work, the stress-biased, field-induced strain of a Ni₂MnGa single crystal (100-cut measuring 15 x 5 x 2 mm) is measured. A cyclic transverse field, driven at 0.01 Hz from 0 to +/- 5 kOe, is applied along the sample's width direction. A constant compressive load is applied to one end of the sample (perpendicular to the field direction). A piezo stack is attached at the other end to generate a longitudinal vibration along its length. The sample displacement under varying constant biased stress is measured with and without piezo assist.

Both operating stress and strain outputs of the FSMA actuation is clearly enhanced by the piezo-assisted effect. Without the piezo-assist, the maximum reversible strain is 3% and appears only in a limited external stress range between 0.7 and 1 MPa. With the piezo-assist, the maximum reversible strain increases to 4.5% and appears in a broader stress output ranging between 0.4 and 1.2 MPa. Smaller magnetic energy is required for twin motion as the twinning stress is reduced by the acoustic assist; the unused magnetic energy can be utilized to work against a larger external stress.

[1] B. Peterson et al., J. of Appl. Phys., **95**, 6963-6964 (2004).

12:15

Oral

The acoustical investigations of a $\text{Ni}_{53}\text{Mn}_{25}\text{Ga}_{22}$ Heysler alloy ferromagnet crystal

Vasiliy D. Buchelnikov¹, V V. Koledov², Dmitriy A. Kostiuk³, Yuri A. Kuzavko⁴, Vladimir V. Khovailo², Vladimir G. Shavrov²

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Some metal alloys have reversible thermoelastic martensite transitions with shape memory effects (SME). SME coupled with a reversible transitions from a deformed martensite phase into the high-temperature austenitic ones. Ferromagnetic crystal of $\text{Ni}_{53}\text{Mn}_{25}\text{Ga}_{22}$ Heysler alloy (austenite starting temperature $A_s=116^\circ\text{C}$ and finishing one $A_f=134^\circ\text{C}$, martensite starting temperature $M_s=109^\circ\text{C}$ and finishing one $M_f=97^\circ\text{C}$, Curie temperature $T_c=134^\circ\text{C}$) with a coupled magnetostructural transitions [1] allows to get the decrease of a magnetic field magnitude, to control the reversible change of the sample dimensions (4x4x10 mm sample in our case). For our crystal of $\text{Ni}_{53}\text{Mn}_{25}\text{Ga}_{22}$ the lattice parameters $a=b=0.7753$ nm, $c=0.6598$ nm, as for Ni_2MnGa $a=b=0.5920$ nm, $c=0.5566$ nm at D_{4h} phase, $a=0.5825$ nm at O_h . The investigations of SME are aimed to combine rapid response with large reversible deformations. The decrease of a magnetic field magnitude for a controlled SME is a common problem for a wide engineering applications of such materials.

The presence of a soft acoustical transverse mode TA_2 with $k=(\frac{1}{3}, \frac{1}{3}, 0)$ wave vector and $[110]$ polarization takes place for Heysler alloys. It is related with accession to zero of the elastic constant $c'=(c_{11}-c_{12})/2$ [2].

The sample was temperature-controlled in experiment. The accuracy of measuring was 0.025 K, the minimal speed of heating - 1 K / 3 min., the sample was placed in constant magnet of 2 kOe at necessity. All five characterizing temperatures of crystal were definable from acoustical measuring.

Authors are grateful for BRFFI and RFFI (F04R-080, 04-02-81058 grants) and ME RB (grant 05-505) for their financial support of carried out researches.

1. C. Jiang, G. Feng, H. Xu. Appl. Phys. Lett. 2002. V.80. pp. 1619-1621.

2. T.E. Stenger, J. Trivisonno. Physics. Rev.B 1998. V. 57. pp. 2735-2739.

Lunch break

Tuesday afternoon, 18 September, 12:30

Parallel Session

Modeling and Simulation I

Tuesday afternoon, 18 September, 14:00

14:00

Invited

Thermodynamics of one-way shape memory effect at complex stress state and unrestricted strains

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The early phenomenological thermodynamic theory of pseudoelasticity (R_L model) is generalized to account for one-way shape memory effect under single thermomechanical cycle (arbitrary path in the temperature-stress space). The thermoelasticity of austenite (high temperatures), pseudoelasticity (moderately high temperatures), R - plastic flow (low temperatures), and the complex deformation behaviour consisting in combined reorientation and phase transition - p.t. (moderately low temperatures) are encompassed into the single theoretical framework.

The basic concepts:

- In micro-scale the phases are thermoelastic solids having the same thermal properties, the motion of interfaces is the only source of the entropy production,

- The macroscopic RVE is a single component, three-phase system (austenite and two compounds: self-accommodated martensite - M_{sa} , and oriented martensite - M_{or}) being in constrained equilibrium with respect to the global amounts of M_{sa} and M_{or} at each instant of a homogeneous process,

- the concept of optimal internal rearrangement of martensite objects (RVE is in unconstrained equilibrium with respect to all possible microscopic phase orientation variables) leading to the notion of macroscopic phase strain energy,

- the second law of thermodynamics acts as the exclusion principle. It indicates which p.t. processes are admissible and it establishes their onset (macroscopic phase strain energy reaches the critical value),

- the multiplicative decomposition of the deformation tensor is applied to extend the infinitesimal theory into the range of unrestricted strains.

The preliminary verification procedure showed that at simple tension, the theoretical isothermal stress-strain ($212\text{K} < T < 336\text{K}$) and isostress strain-temperature hystereses ($30\text{MPa} < \text{Sig} = \text{const} < 400\text{MPa}$) follow 15 experimental plots (TiNi alloy) with an error acceptable in the structural engineering.

14:30

Oral

A phenomenological 1D model describing main and secondary effects of stress-induced solid phase transformations.

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The diffusion and the use of shape memory alloys (SMAs) in many different engineering applications is resulting in an always increasing research effort toward a reliable and complete modeling of their macroscopic behavior.

Starting from some robust phenomenological models proposed in the recent past and capable of describing main SMA macroscopic effects [1,2], we present here a more flexible model, able to include many other well-known SMA effects, such as tension-compression asymmet-

ric behaviors (e.g. asymmetric yield stress level, loop width and length) and elastic properties depending on the reached transformation strain level.

Several numerical experiments are performed in order to prove the capabilities of the model.

We finally show how this model can be modified in order to obtain a good tool for describing a new, very promising, class of shape memory materials: the ferromagnetic SMAs.

References:

1. F. Auricchio, L. Petrini, "A three-dimensional model describing stress-temperature induced solid phase transformations: solution algorithm and boundary value problems", *International Journal for Numerical Methods in Engineering*, 61: 807-836 (2004).
2. F. Auricchio, A. Reali, U. Stefanelli, "A three-dimensional model describing stress-induced solid phase transformation with permanent inelasticity", *International Journal of Plasticity*, 23: 207-226 (2007).

14:45

Oral

A phenomenological model for superelasticity in NiTi Alloys

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Nickel-Titanium (NiTi) alloys have seen growing interest in many engineering applications in the last decades due to their special functional properties namely shape memory effect (SME) and superelastic effect (SE). To better understand the potentialities of NiTi alloys many researches have been focused on the numerical modeling of their mechanical and functional behavior in the last few years [1]. In this regard, the contributions made by Tanaka et al. [2] and Brinson [3] are of great significance; more recently Auricchio et al. developed other models based on the elastoplasticity theory [4-5]. In a previous work [6], the authors proposed a phenomenological approach to simulate the two way shape memory effect (TWSME) in NiTi alloys, by a Prandtl-Ishlinskii hysteresis operator. In this work, a new phenomenological model, based on the aforementioned hysteresis operator, is developed to simulate the superelastic effect. The parameters of the phenomenological model are identified, from experimental measurements, by simple and efficient numerical procedures, developed in the commercial software package Matlab® and Simulink®. As an example Figure 1 shows a preliminary result of the proposed model, together with a comparison with experimental measurements.

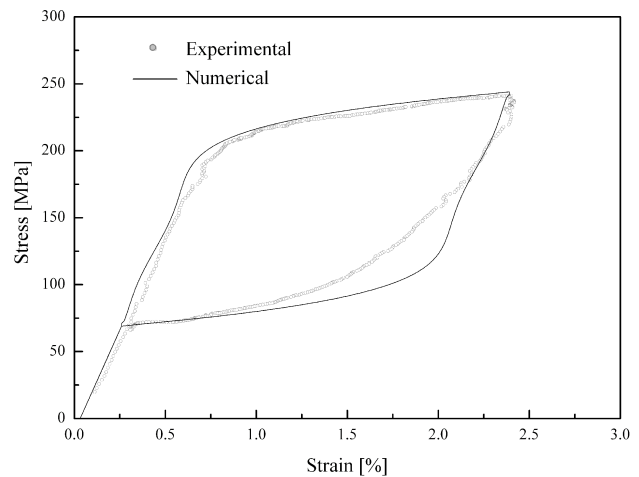


Figure 1: Comparison between numerical simulation and experimental measurements

References

- [1] Paiva A., Savi M. A., *Mathematical Problems in Engineering*, art. no. 56876 (2006), pp. 1-30.
- [2] Tanaka K., Nagaki S., *Ingenieur Archive*, 51 (1982), pp. 287-299.
- [3] Brinson L. C., *Journal of Intelligent Material Systems and Structures*, 4 (1993), pp. 229-242.
- [4] Auricchio F., Sacco E., *International Journal of Non-Linear Mechanics*, 32(6) (1997), pp. 1101-1114.
- [5] Auricchio F., Reali A., *Mechanics of Advanced Materials and Structures*, 14 (2007), pp. 43-55.
- [6] Falvo A., Furguele F., Maletta C., *Smart Materials and Structures*, 16 (2007), pp. 771-778.

15:00

Oral

Modeling superelastic NiTi wire structures

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Research on the possibility to produce textile structures using superelastic NiTi wires is currently in progress. NiTi knitted self-expanding stents can be mentioned as a well-known example. Less known is that thin NiTi wires are considered for production of knitted and woven smart textiles with unique thermomechanical properties promising a wide range of new engineering applications. Optimal design of NiTi textiles requires reliable knowledge and modeling of superelastic behaviour of NiTi wire structures, composed from not just straight wires but generally from curved shape set wires. This appears to be a new area for SMA experimentation and modeling.

In the present contribution, we describe a newly proposed SMA model describing superelastic behavior of NiTi wires in tension and compression at constant temperature developed particularly for the purpose of simulating superelastic behaviour of NiTi 2D wire structures. The model describes the internal state with three internal variables: the martensite volume fraction, R-phase volume fraction and return points. The proposed model fulfils four basic requirements given on it:

- It captures the strains due to multiple deformation mechanisms taking place in NiTi transforming through B2-R-B19' sequences (the presence

of R-phase dramatically affects mechanical behaviour of shape set NiTi structures).

·It reliably describes the closed shapes of entire and partial superelastic loops in stress – strain space including the return-point memory phenomenon (reliable modeling of partial loops is of key importance for shape set NiTi structures) .

·It captures asymmetric behavior in tension and compression (essential to simulate bending).

·It can be easily implemented into finite element models (essential to simulate nonhomogeneous deformations in shape set NiTi structures).

For demonstration, the experimental and simulated superelastic behavior of knitted self-expanding stent and NiTi “snake yarn” is presented and discussed.

15:15

Oral

Simulation of Discrete Actuators using Magnetic Shape Memory Alloys

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For further miniaturizing micro actuators, new motor drive schemes are desirable. One novel approach is the use of new materials like Magnetic Shape Memory (MSM) alloys. These materials achieve an elongation of up to 10% when exposed by a sufficiently strong magnetic field. But only the area exposed to a magnetic field strong enough to cause crystal reorientation are being elongated. For the design of discrete MSM-actuators, simulations were conducted by the Finite-Element Method (FEM). The goal of the simulation was to find a setup for an optimized system which exposes an as high percentage as possible of the MSM alloy to the required field. Such a system requires three components: (1) an excitation coil to create a magnetic field which is great enough to exceed the required field strength, (2) a soft magnetic flux guide with a magnetic permeability as high as possible, and (3) a strip of MSM alloy with an appropriate crystalline direction allowing the crystalline transformation resulting in an elongation. Investigations revealed that before the crystalline transformation the relative permeability μ_r of the material is 7, while after the transformation it is 12. Based on the magnetic data of the MSM material and the soft magnetic alloy (NiFe45/55), a design was modeled and simulated.

So far, we discussed the elongation of the material. For creating a switchable actuator, we also have to look into its contraction. The easiest way to accomplish one is to using two stripes working against each other. For creating a motion in one direction, a first MSM element is extended, to return it to the starting point, a second MSM element compresses it by being extended itself, and vice versa. As a result, a system was created with separate magnetization capabilities for the two stripes. For the design chosen, up to 35% of each MSM stripe is exposed to a sufficiently strong magnetic field for causing an actuation.

Coffee break

Tuesday afternoon, 18 September, 15:30

Parallel Session

Shape Memory Alloys II

Tuesday afternoon, 18 September, 15:50

15:50

Oral

Forming and controlling of pores by capsule-free hot isostatic pressing in NiTi shape memory alloys

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The pore evolution process of porous NiTi shape memory alloys (SMAs) fabricated by capsule free hot isostatic pressing (CF-HIP) was investigated by adopting different fabricating parameters in this study. It has been demonstrated that porous NiTi SMAs with homogeneous pore distribution and nearly rounded pore shape can be prepared by CF-HIP under suitable condition. In addition, two special kinds of porous NiTi SMAs with novel pore structure were fabricated by adjusting CF-HIP parameter. One of them has a special sandwich structure distribution of pore along radial section. This structure was formed owing to the balance between hot pressure in the HIP furnace and gas pressure produced by gas expansion in the green sample. Another one shows gradient distribution of pore size along radial direction, increased from outside to the center, which is related to the formation of air bubble in the melting NiTi alloy.

16:05

Oral

Static and dynamical functional properties of ultra thin NiTi wires

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2D/3D structures made from ultra thin (<100 µm) NiTi wires are currently considered in engineering applications in textile, medical or robotics field. Thin NiTi wires have several advantages over bulk elements – they are stronger, show enhanced functional properties, they are less prone to failure and able to be actuated with higher frequencies.

The development and design of such novel applications require the knowledge of the thermomechanical behaviour of NiTi ultra thin wires. First, since the material state of the thin NiTi wires is different from that of the bulk NiTi (texture, cold work etc.), their tensile properties cannot be obtained by simply scaling down the results measured on thicker wires but have to be determined experimentally. Second, since the shape set thin NiTi wires in 2D/3D structures deform by combined bending-tension-torsion, such behaviors have to be determined experimentally as well. Third, since many of the introduced applications attempt to take advantage of the unique damping capabilities of NiTi in a form of thin NiTi wires, it is of utmost interest to investigate their dynamical behavior, particularly the influence of temperature, prestress and vibration amplitude on the damping capacity.

In this paper, we will introduce various novel approaches towards thermomechanical testing of ultra thin commercially available NiTi wires and simple shape set NiTi structures as helical or snake springs. Basic testing procedures include thermomechanical tensile testing, combined tension-torsion testing realized on a self-made experimental apparatus and forced tensile vibration testing. The experiments involve in-situ measurement of electrical resistance providing feedback information

on the micro structural processes in the activated wires. The collected data provide a basis upon which FEM implementable models (related talk by Sedlak et. al.) describing functional thermomechanical behaviors of 2D/3D structures made of thin NiTi wires are built.

16:20 Oral

Direct casting and rapid solidification of CuAl SMA wires and characterisation of the microstructure

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A rapid solidification technique was used to produce thin wires of Cu-Al SMA alloys directly from the melt. In-rotating-liquid-spinning (INROLISP) is an economical and straightforward process to obtain wires in a single-step. In contrast to previous publications a horizontal arrangement of the INROLISP process was used for the direct casting of thin wires. A computer controlled facility was designed and installed to determine and control the near-net-shape casting process and the process parameters over an extended time period. The main process parameters, such as the velocity of the melt jet, the stable free flight length, the nozzle geometry and the cooling rate, were examined. In particular, the meltflow was simultaneously observed by a high speed camera and theoretically described using fluid dynamic equations. After optimization of the process parameters, thin wires of approximately 100 μm in diameter with a reduced grain size were produced. The reduction of the grain size and additions of Ni will depress the martensitic transformation temperature indicating the structural transformation of the high temperature cubic β -phase to martensite. The shape memory properties as the characteristic transformation temperatures of the bulk alloys-ingot samples and of the as cast wires were measured by using differential scanning calorimetry (DSC) and by performing tension tests. The quality of the wires varies with different process parameters and is determined by optical microscopy and scanning electron microscopy (SEM). The effect of rapid solidification on grain size and on the crystalline microstructure of the martensite fraction is demonstrated by EBSD mapping in combination with an EDX scan and XRD analysis based on the Debye-Scherrer technique.

16:35 Oral

Localization Phenomena in NiTi Shape Memory Alloys - Finite Element Modeling of Lüders-like Shear Bands during quasi-static and cyclic loading

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Several reasons for the occurrence of localized deformation (as opposed to an evenly spaced "homogeneous" deformation) of pseudo elastic NiTi shape memory alloys have been proposed in the literature. Grain size and texture may well favour localized deformation (microstructural scenarios). Stress and strain states at the martensite/austenite interface are envisioned to promote the transformation in adjacent regions (mesoscopic and micromechanical models). The specimen shape, overall stress state and heat transfer to ambient media may well have an important effect on the deformation behavior (macroscopic ap-

proaches). While there exists a vast body of experimental data on the mechanical behavior of NiTi shape memory alloys, a clear understanding of the individual contributions of these effects is yet to be developed.

In the present contribution, we use a simple Finite Element model in order to study the macroscopic mechanical aspects of the formation and propagation of martensitic transformation regions in thin tensile specimens (wires and ribbons) and in plate specimens with and without notches during quasi-static and cyclic loading. Based on detailed experimental data (also presented in a companion paper in these Proceedings), we include the effect of localized functional fatigue (i.e., changes of transformation behavior due to repeated cycling through the forward and reverse transformation). We study the effect of different specimen geometries and uniaxial versus bi-axial load cases on localization of the stress-induced transformation. Finally, we discuss how uniaxial tensile deformation differs from the more general stress and strain states in plate specimens, and how these subtle differences have a distinct effect on localization of transformation, deformation, and functional fatigue.

16:50 Oral

Study of the tensile constrained recovery behaviour of a Fe-Mn-Si shape memory alloy

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The microstructural evolution of Fe-Mn-Si Shape Memory Alloys (SMAs) was monitored during the main stages of the classical technological flow: (1) casting; (2) homogenizing and (3) hot rolling. With increasing the plastic deformation degree, including by additionally elongating the hot rolled specimens, the increase of the amount of ϵ stress induced martensite (SIM) plates in the detriment of α' SIM was revealed by means of optical and scanning electron microscopy. The tensile-stress variation was analysed during complex cycling by constrained recovery of a high-plasticity Fe-23.27 Mn-2.76 Si (wt.%) SMA, with a tensile strength about 670 MPa and an ultimate strain of 8 %. The cycles were composed of: (i) room temperature (RT) loading; (ii) RT partial unloading; (iii) constant strain heating; (iv) constant strain cooling and (v) RT unloading. During the cycles, characterized by average maximum stresses above 460 MPa and recovery stresses above 240 MPa, the observed stress-temperature evolution confirmed the four stage stress variation reported by literature [1]. In order to maintain the above stress levels, constrained-recovery strain had to be increased with an average of 0.5 % per cycle, between 2 and 4 %.

[1] Z.Z.Dong, S.Kajiwar, T.Kikuchi, T.Sawaguchi, Acta mater. 53 (2005) 4009-4018

17:05

Oral

Martensitic transformations and mechanical behavior of amorphous-crystalline Ti-Ni-Cu and Ti-Hf-Ni-Cu thin ribbons

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Martensitic transformations and strain variation in amorphous-crystalline thin ribbons of $\text{Ti}_{50}\text{Ni}_{25}\text{Cu}_{25}$, $\text{Ti}_{40.7}\text{Hf}_{9.5}\text{Ni}_{44.8}\text{Cu}_5$ and $\text{Ti}_{40.7}\text{Hf}_{9.5}\text{Ni}_{39.8}\text{Cu}_{10}$ alloys were investigated. The amorphous-crystalline state in the samples was produced by an interruption of the isothermal crystallization in DSC apparatus. The volume fraction of the crystalline phase (V_{cr}) in the samples was varied from 10 to 100 %. Transformation temperatures in amorphous-crystalline thin ribbons of $\text{Ti}_{50}\text{Ni}_{25}\text{Cu}_{25}$ alloy depended only slightly on the crystalline volume fraction. On the contrary the strong dependence of the martensitic transformation temperatures on the volume of crystalline phase was obtained in $\text{Ti}_{40.7}\text{Hf}_{9.5}\text{Ni}_{44.8}\text{Cu}_5$ and $\text{Ti}_{40.7}\text{Hf}_{9.5}\text{Ni}_{39.8}\text{Cu}_{10}$ alloys. Unusual difference between the transformation intervals of the direct transition and the reverse one was found. The interval of the direct transformation was 3 °C whereas the interval of the reverse transition was 40 °C. It was shown that as the crystalline volume raised the transformation temperatures increased and the interval of the reverse phase transition reduced. Probably the main reason of the observed phenomena consisted in a various phase composition of the samples after crystallization.

The shape memory effect was observed in the amorphous-crystalline thin ribbons. Increase of the volume fraction of crystalline phase resulted in the growth of the strain recovery. The perfect shape memory effect was observed in the samples with the volume fraction of the crystalline phase exceeded 60%. It was concluded that the unelastic deformation realized in the amorphous phase as well as in the crystalline one in the case of the $V_{\text{cr}} < 60\%$, otherwise the strain occurred in the crystalline grains only.

Wednesday, 19 September

Plenary Session

Wednesday morning, 19 September, 9:30

Coffee break

Wednesday morning, 19 September, 10:30

Parallel Session

Ferromagnetic Shape Memory Alloys III

Wednesday morning, 19 September, 11:00

11:00

Invited

Selected fundamental and technical advances in magnetic shape memory alloys

Robert O'Handley¹, Samuel M. Allen, Ratchatee Techapiesancharoenkij², Jari T. Kostamo^{3,4}, Marc Richard⁵, Jesse Simon⁶

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11:30

Oral

Characterization of frequency dependent magneto-elastic properties of magnetic shape memory actuators

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The recent developments with magnetic shape memory alloys have made FSMA's interesting option for producing mechanical actuation up to 2 kHz frequencies. It has been shown that Ni-Mn-Ga crystals can respond to external magnetic field pulses rapidly and response times of less than 0.25 ms have been recorded [1]. The short response time combined with high energy density of FSMA make these actuators an interesting candidate for applications where compact actuators are needed for producing larger strains than piezoelectric or magnetostriuctive devices can typically produce.

However, to understand better the behavior of this material at different excitation frequencies, the AC properties of this material should be studied more closely. The understanding of the frequency dependent behavior of this material could be used to facilitate the design of efficient actuators. This paper will concentrate on developing a dynamical model for Ni-Mn-Ga actuator and the model will be validated with experiments in both static and dynamic cases.

The modeling of the actuators will be started from the equation describing the dependence between magnetic anisotropy of the Ni-Mn-Ga and the MFI stress. The result will be validated by static measurements to get the maximum magnetic field induced force and strain. To measure the dynamical behavior of the FSMA, a test device based on a tunable resonator has been developed and it has been used to estimate the frequency dependent parameters. The dynamical behavior of damped harmonic oscillator is described by well known differential equation and the characteristics of the FSMA actuator will be extracted from the frequency responses by comparing the measurements with the theoretical model.

[1] Pulsed magnetic field actuation of single-crystalline ferromagnetic shape memory alloy Ni-Mn-Ga, Miguel A. Marioni et.al. Proc. SPIE Int. Soc. Opt. Eng. 4699, 191 (2002)

11:45

Oral

Structure and mechanical training of 5M and 7M Ni-Mn-Ga magnetic shape memory alloys

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Magnetic shape memory materials gained a large research interest owing to their capability to deform by some percent via twin boundary motion under the influence of a magnetic field. Concurrently, they are supposed to react faster than conventional shape memory materials because neither heating nor cooling are involved. The predominant material system is Ni-Mn-Ga with compositions around Ni₂MnGa. In our case Ni₅₀Mn₃₀Ga₂₀ and Ni₅₀Mn₂₉Ga₂₁ alloy samples are used to produce polycrystalline textured samples. Upon cooling these alloys transform martensitically at 100 °C and 55 °C, respectively. The evolving martensitic structure is either orthorhombic (7M) or tetragonal (5M, NM) and depends on the thermomechanical history of the sample as well as the composition. Since only two of the three possible structures are suitable of providing the mandatory highly mobile twin boundaries, it is important to understand and control the phase formation process by appropriate thermal and mechanical treatment. Once the sample is given a suitable structure, samples for training are hot mold cast for directional solidification. X-ray diffraction techniques are applied to investigate the texture in these samples. After being cut erosively they are trained thermomechanically and in successive compression tests to lower the (de)twinning stress. The stresses in the training process have to be kept as low as possible to avoid brittle fracture of the samples during the cyclic compression process. An Instron testing machine has been modified in order to allow for magnetomechanical tests in magnetic fields of up to 0.8 Tesla.

12:00

Oral

Effect of deformation on structure and mechanical behavior of polycrystalline Ni-Mn-Ga alloys

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The purpose of this work was to investigate the stress-strain behavior of the Ni-Mn-Ga alloys with nickel excess. The deformation was introduced in prismatic specimens by compression test for both parent and martensitic phase. The effect of the deformation on structure was studied by means of X-ray diffraction, transmission and electron scanning microscopy (TEM and SEM).

The martensitic structure, dependently on chemical composition of the alloys, was 10M or non-modulated tetragonal. The non-modulated martensite was observed in the alloys with higher nickel content. The deformation of the martensitic samples up to the fracture does not change their structure. The stress-strain curve shows that the initial elastic deformation stage terminates at a strain of about 2% and critical stress 70 MPa, which corresponds to the start of martensite reorientation.

Several of studied alloys, obtained by fast solidification, before homogenization show a broad temperature interval of the martensitic transformation and reveal a small uniform grain size. The limited deforma-

tion of these alloys in the L2₁ parent phase show the pseudoelastic stress-strain behavior. The volume of deformation up to the fracture is in the range of 20-25%. TEM studies of the fractured specimen reveal a small amount of the induced martensitic phase. The mechanical behavior of the specimens, homogenized at 900°C for 50 hours, varies in comparison to those specimens, which were not homogenized. The long annealing at high temperature causes not only the compensation of the chemical composition but also the increase of the grain size, which may affect the mechanical behavior of the alloys.

12:15

Oral

Ni₂MnGa - polymer composites using polyester and polyurethane matrices

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Composites consisting of magnetic shape memory (MSM) particles embedded either in polyester [1] or polyurethane matrices were prepared. Single-crystalline MSM particles were obtained by mortar grinding of melt-extracted and subsequently annealed Ni_{50.9}Mn_{27.1}Ga_{22.0} (at.%) fibres. The crystal structure of the martensite is tetragonal (5M) with $c < a = b$. Magnetic characterizations of the deformed composites show indirect evidence for stress induced twin boundary motion within the MSM particles, as the compressed composite is easier to magnetize in the direction of compression compared with the directions perpendicular to it. The magnitude of this effect depends on the stiffness of the matrix material used and is therefore more pronounced for polyester than for polyurethane. The texture of all embedded MSM particles in the polyester composite is investigated before and after compression by means of synchrotron radiation [2]. In the initial state, the MSM particles in the composite have a random texture, i.e., there is no preferred orientation of the c axis. After 30% compression (height reduction), the MSM particles show a (004)-fibre texture in the direction of compression. This is unambiguous evidence for stress-induced twin boundary motion within the MSM particles. Finally, it is discussed whether it is more advantageous to use a polyurethane matrix in order to observe magnetic field-induced twin boundary motion.

[1] N. Scheerbaum, D. Hinz, O. Gutfleisch, K.-H. Müller, L. Schultz: *Acta Mater.* 55 (2007) 2707-2713.

[2] N. Scheerbaum, D. Hinz, O. Gutfleisch, W. Skrotzki, L. Schultz: *J. Appl. Phys.* 101 (2007) 09C501.

Lunch break

Wednesday afternoon, 19 September, 12:30

Parallel Session

Thin Films

Wednesday afternoon, 19 September, 14:00

14:00

Oral

Epitaxial Ni-Mn-Ga and Fe-Pd films for magnetic shape memory applications: Growth, martensitic transformation and magnetic properties

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Magnetic shape memory alloys like Ni-Mn-Ga and Fe-Pd can change their shape up to several percent by the reorientation of martensitic variants in a moderate magnetic field. However to date a significant strain is only obtained in single crystals bulk materials. It is expected that epitaxial films are most promising for observation of MSM effect in thin films.

In this presentation we show that Ni-Mn-Ga and Fe-Pd films can be grown epitaxially on MgO(100) substrates. Both material systems are compared with respect to their suitability for microactuator and sensor suitability.

Temperature depended X-ray diffraction and pole figure measurements are used to examine structure and its transformation and microstructural transitions (variant reorientation). Additionally temperature and orientation depended magnetic measurements are used to identify the anisotropic magnetic behavior of these films.

These experiments are used to analyze in detail the effect of constraints imposed by the substrate. This can result in significantly different behavior compared to bulk single crystal, e. g., the reduced symmetry of a thin film reduces the number of variants observed. Additionally the stress in the deposited film modifies the martensitic transformation temperatures. Due to the low blocking stress of few MPa's, no twin boundary motion of a thin Ni-Mn-Ga film on a thick, rigid substrate is expected. Contrary to this, magnetization and field dependent pole figure measurements indicate that - though constrained by the substrate - a reorientation of variants by a magnetic field can be possible in orthorhombic Ni-Mn-Ga films. A model explaining this unexpected behavior is proposed.

14:15

Oral

Integration Technologies for Single-Crystalline NiMnGa Films

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The implementation of the giant magneto-strain effect of ferromagnetic shape memory alloys (FSMAs) in microactuators requires novel single crystalline thin films with bulk-like intrinsic properties and microsystems-compatible integration technologies. In this paper, Ni-Mn-Ga films epitaxially grown on heated substrates by DC magnetron sputtering are examined. The films are martensitic and ferromagnetic at room temperature and thus fulfill basic requirements for applications. For a detailed analysis of the martensitic transformation temperature-dependent X-ray diffraction measurements are performed, which are discussed together with orientation-dependent magnetization and electrical resistance measurements.

Technologies for micromachining and integration of free-standing epitaxial NiMnGa films are developed to investigate magnetic-field and stress-dependent reorientation effects of the martensitic variants. In order to obtain free-standing films, the options of peeling the film off the substrate and of using a Cr buffer layer and chemical release have been pursued. The process of micromachining requires an etchant-resistant substrate. For this purpose, the thin film is bonded to an alumina substrate covered by an adhesive coating. The adhesive coating is selected to provide sufficient bonding strength during processing and to use it as a sacrificial layer later on. A transfer bonding process is developed allowing the integration of micromachined NiMnGa components to a target substrate. For electrical interconnection, the technologies of laser welding and gap welding are investigated.

14:30

Oral

Magnetostructural Phase Transformation and Shape Memory Effect of Fe-added Ni₂MnGa Films

Makoto Ohtsuka¹, Junpei Sekino², Keiichi Koyama³, Toshiyuki Takagi⁴, Kimio Itagaki⁵

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Ni₂MnGa alloy is a multifunctional material with ferromagnetism and shape memory effect (SME). The application of the alloy films as an actuator of micro-machine has been proposed. The SME induced by a magnetic field (MF) will enable the quick response and remote control. In the present study, the magnetostructural phase transformation and SME of Fe-added Ni₂MnGa films were investigated. The Fe-added Ni₂MnGa films were deposited on poly-vinyl alcohol substrates by a dual magnetron sputtering apparatus using the Fe target (sputtering power: DC 10, 20 W) and the Ni₅₁Mn₂₆Ga₂₃ target (sputtering power: RF 200 W). The composition of the films was Ni_{52.2}Mn_{24.5}Ga_{20.4}Fe_{2.9} and Ni_{50.4}Mn_{23.5}Ga_{20.0}Fe_{6.1}, respectively. After separating from the substrate, they were heat-treated at 1073 K for 3.6 ks and constraint-aged (CA) at various conditions to make the two-way SME. The Curie temperature was higher than room temperature and its martensitic transformation (MT) temperatures for the films. The X-ray diffraction (XRD) pattern for the CA films was indexed as body centered tetragonal (bct) and 7 layers modulated structure (14M) in the martensitic phase. The reversible two-way SME by the thermal change was confirmed through the MT and its reversion. The gradient of strain-temperature curve, the effective recovering strain and the width of thermal hysteresis were dependent on the CA conditions. The strain-temperature curves shifted to a high temperature region. The MF induced structural phase transformation was evaluated by an XRD apparatus in high MF up to 5 T. It was confirmed the martensitic phase was stabilized by the MF. Furthermore, the SME by the MF was observed around MT temperature on cooling for the films. It was considered that the MF induced SME was appeared by the inducing the martensitic transformation with MF.

14:45

Oral

Transformation behavior of Ni-Mn-Ga/Si(100) thin film composites with different film thicknesses

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The Ni-Mn-Ga magnetic shape memory alloys (MSMAs) show large magnetoplasticity and strain-induced change of magnetization. Thin film composites of MSMAs representing Ni-Mn-Ga thin films sputter-deposited on different substrates are prospective materials for (micro)nanosystem applications.

In this work, we have prepared two series of Ni-Mn-Ga/Si(100) thin film composites and studied the influence of the heat treatment and film thickness on the structure, martensitic transformation, ferromagnetic ordering and intrinsic stress state in the films. Each series containing 7 samples with thickness of 0.1; 0.3; 0.5; 0.7; 1; 2 and 3 μm was prepared by r.f. magnetron sputtering using $\text{Ni}_{49.5}\text{Mn}_{28}\text{Ga}_{22.5}$ and $\text{Ni}_{52}\text{Mn}_{24}\text{Ga}_{24}$ targets. These targets facilitate the formation of 10M and 14M ferromagnetic martensites at room temperature, respectively. A Si(100) wafer having amorphous 500 nm thick SiN_x buffer layer was used as a substrate.

According to X-ray diffraction, a disordered state can be inferred in the as-deposited films while annealed films (in vacuum at 1073K for 1h) show (110)-type fiber texture. Temperature dependencies of the electrical resistivity, $\rho(T)$, of films were measured precisely in the interval 250-450 K. On warming, as-deposited submicron films show a monotonous reversible decrease of resistivity which changes to the monotonous increase for the thicker films. The annealed films demonstrate the anomalies on the $\rho(T)$ curves associated with the martensitic transformation (MT) and Curie temperature (T_C) typical for bulk Heusler alloys. The values of MT and T_C temperatures are considerably dependent on film thickness in the submicron range. The stress in the films and stress anomalies produced by MT were determined with substrate curvature measurement method. The results of the resistivity and substrate curvature experiments are linked to explain the thickness dependence of the transformation temperatures.

15:00

Oral

Structural, magnetic and phase transformation properties of Fe-Pd thin films processed and characterized in situ with micro-hotplates

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Fe-Pd thin films were fabricated by sequential sputtering of elemental and alloy targets on micro-hotplate (MHP) arrays, in order to investigate

their properties as ferromagnetic shape memory alloys (FSMA). The advantages of Fe-Pd-based FSMA in comparison to Ni-Mn-Ga are a higher Curie temperature, saturation magnetization, transformation temperature as well as potentially higher magnetocrystalline anisotropy. The micro-hotplates – microstructured Si_3N_4 and SiO_2 membranes with Pt heaters and measurement electrodes – were used as material processing and characterization platforms: Films on such MHPs can be heated and cooled with rates up to several 1000K/s which enables rapid and precise annealing and quenching of metastable phases. Furthermore, during the heat treatment, the resistance of the thin film on the MHPs can be monitored, so that phase transformations can be identified in situ. The films were characterized by energy disperse x-ray spectroscopy, x-ray diffraction, resistance versus temperature measurements and temperature dependent vibrating sample magnetometry measurements. First results of MHP-processed Fe-Pd thin films show a reversible phase transformation with low hysteresis in a composition range from $\text{Fe}_{70}\text{Pd}_{30}$ to $\text{Fe}_{72}\text{Pd}_{28}$ in a temperature interval from 50°C to 90°C.

15:15

Oral

Several ways to adjust structural and physical properties of Ni-Mn-Ga thin films obtained by r.f. sputtering

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The aim of this communication is to present Ni-Mn-Ga thin films elaborate by r.f. sputtering with low power (less than 120W) and from Ni_2MnGa alloy target. Chemical composition, crystallographic structure identified from DRX, surface morphology from AFM, internal stress, nanoindentation modulus and strain induced by magnetic field of these films are systematically investigated as a function of the sputtering parameters (Argon pressure and power), the sputtering temperature (from 298 to 1073K) and the annealing treatment (from 473 to 873K). The films deposited at room temperature have nearly $\text{Ni}_{55}\text{Mn}_{23}\text{Ga}_{22}$ constant chemical composition, and, DRX analyses show 2 phases (one of them has a martensitic structure) that turn into only one phase when annealing temperature is increased. These results are confirmed by nanoindentation tests performed with few nm penetration depths. The set of these measurements give histograms with 2 indentation moduli M (respectively 135 and 210GPa) for films deposit at room temperature. These moduli decrease progressively with the annealing temperatures to reach at 873K a unique value of 97GPa also obtained in bulk ferromagnetic shape memory alloy (FSMA). Moreover, the internal stress in films deposited on Si beam cantilever is identified as a function of various sputtering conditions. It reveals three different areas delimited by 473K and 673K sputtering temperatures with a maximum stress value of 1,7GPa reached at 673K. Finally, the strain induced by magnetic field is measured via the deflection of the coated Si cantilever beam and a maximum strain value of -340ppm is reached for 1e6A/m magnetic field applied on films annealed 6h at 873 K. In conclusion, these results obtained from different techniques are coherent and consistent with the ones available in the literature. They show that the annealing treatments bring on an important transformation beginning at about 673K to finally give the FSMA properties to films from about 873K.

Coffee break

Wednesday afternoon, 19 September, 15:30

Joint Poster Session (Part II)

Poster Award Ceremony

Wednesday afternoon, 19 September, 15:50

Thursday, 20 September

Parallel Session

Devices and Systems I

Thursday morning, 20 September, 9:00

9:00

Invited

Design and Application of Shape Memory Actuators

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The use of shape memory alloys in actuators offers the opportunity to develop robust, simple and lightweight elements for application in a multitude of different industries. Over the years, the intermetallic compound Nickel-Titanium (NiTi or NiTiNol) has gained acceptance as a standard alloy in even though NiTiNol is not the perfect alloy for all kinds of applications and industries. The major disadvantages of the material open a wide field for improvements in both, the mechanical design with the existent alloys and the development of new, improved alloy systems:

- the lack of higher transformation temperatures in the available alloys in order to open the field of automotive applications ($M_f > 85^\circ\text{C}$),
- the non-linearity in the electrical resistivity in order to improve the controllability of the actuator,
- the wide hysteresis, which has a significant effect on both, the dynamics of the actuator and the controllability.

Hence, there is a constant strive in the field towards an improvement of the related properties. However, significant steps forward are already possible if the usage of existent materials is optimized for the projected application and if the system is designed in a most efficient way.

This paper provides an overview about existent designs, applications and alloys for actuators, as well as new shape memory actuator systems with improved performance.

9:30

Oral

Micropositioning using shape memory alloy actuators

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Actuators based on Shape Memory Alloys SMA benefit from a high power to weight ratio and have found applications in many areas. They have traditionally been used as “on-off” electromechanical actuators due to the non-linear and hysteretic nature of the martensite-austenite transformation. To accurately command the actuator to an intermediate position within its range of movement it is necessary to use feedback control. The ideal solution is to model the hysteresis mathematically so it can be compensated in the control loop. However, useful models

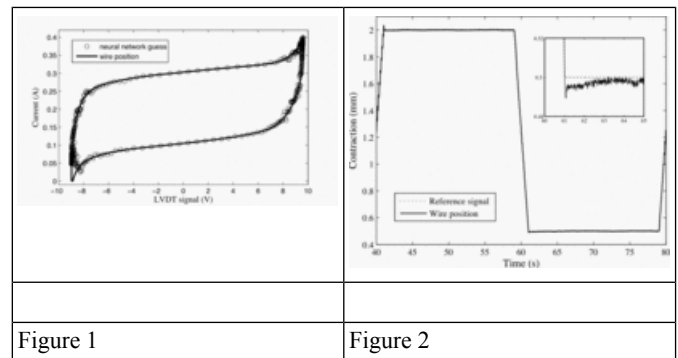
of phase transitions are difficult to obtain. Our approach to the problem is to model empirically the behavior of the wire. For this purpose we have trained a neural network.

The experimental set-up uses a commercial Nitinol wire, 75 mm long and 0.15 mm thick, suspended from a fixed support with its lower end fastened to the core of a LVDT (linear variable differential transformer) position sensor. An additional weight is hanged to the wire to make it recover its original length. The wire is joule-heated by a controlled current. The resolution attainable by the position sensor is half a micron, approximately.

The neural network is trained by applying currents to the wire with different amplitudes and frequencies. After the learning process, the neural network performs reasonably well as shown in Figure 1.

When used in a controller, the neural network nicely compensates the non-linear and hysteretic behavior of the wire, and any simple lineal controller works adequately. We have used a proportional-integral with anti-windup control, obtaining excellent results. Figure 2 shows the performance of the controller for set-points at 0.5 and 2 mm. The inset shows the magnitude of the error.

In conclusion, we demonstrate that shape memory alloys can be used as actuators with micrometer accuracy, provided that a suitable control strategy is implemented. A neural network is very effective for hysteresis compensation.



9:45

Oral

Marvellous method for improving properties of SMA coil springs

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This work is related with a SMA coil spring which shows a perfect recovery and a greater recovery force. When tension spring of common metals is fabricated, 'initial tension' can be provided by minus pitch winding. The initial tension is the force for tension springs to contract further in closed state. If a SMA spring has the initial tension, perfect recovery is expected with a greater recovery force. However, the initial tension cannot be given to SMA coils in the conventional way, since heat treatment for shape setting eliminates the tension generated by the minus pitch winding. We have developed a new method of supplying the initial tension to SMA springs by changing coil orientation after the shape setting treatment. In this paper, some examples are introduced that a perfect recovery with a greater force, though permanent deformation is involved in the deformation, can be attained by the initial tension.

10:00

Oral

Transfer Bonding Technologies for Batch Fabrication of SMA Microactuators

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The introduction of shape memory alloys (SMAs) into novel microsystems products is hampered by various constraints imposed by process technologies of SMA film or foil fabrication, thermo-mechanical treatment and, in particular, monolithic integration. Often, the costly piece by piece processing and pick-and-place assembly remain as the only solution. Many technology combinations promising novel functionality in various applications cannot be realized today. In order to overcome these problems, the technologies of transfer bonding on a full wafer scale and selective transfer bonding are investigated.

SMA materials to be transferred comprise either sputtered thin films or free-standing foils produced, e.g., by cold rolling. In either case, the thermo-mechanical treatment is performed in free-standing form to avoid any kind of temperature incompatibilities, such as interdiffusion or thermal stress. The SMA material is then fixed on a substrate to perform the micromachining. The resulting array of SMA microdevices is then bonded to a transfer substrate and released from the temporary substrate. In the case of wafer transfer bonding, the complete composite of transfer substrate and micromachined SMA layer is transferred and bonded to the final target wafer. Alternatively, individual SMA microdevices are transferred by selectively removing the bonding layer. Both technologies are compatible to target wafers of temperature-sensitive polymers, since critical steps are performed separately. The temporary and transfer substrate may be identical.

In the present study, the transfer technologies are tested for cold-rolled NiTi foils of 20 µm thickness, temporary substrates of alumina, transfer substrates of glass and target substrates of a polymer. Different adhesive coatings are used as bonding layers, which provide sufficient bonding strength during processing, but act as a sacrificial layer later on. The batch fabrication process is demonstrated for the example of microvalves.

10:15

Oral

Novel SMA composite for microapplication

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A crucial topic for advancing of microsystem technology is the development of an integrated microactuation mechanism compatible with microfabrication and able to provide a large displacement and a large actuation energy density. Shape memory alloys allow the development of microactuators with a resulting work density unequaled by other actuator types. However, the implementation of these materials into actual devices is quite complex and mostly limited to deposition of thin film shape memory materials onto fabricated MEMS structures. Potential drawbacks to this technology are the necessity of expensive

sputtering equipment, the accurate control of deposition parameters and the stability of the shape-memory effect in thin-film NiTi over the millions of cycles required for these devices. The current alternative, one-by-one assembly of discrete SMA elements (wire, spring or beam), is a time consuming and expensive process, not in accordance with the philosophy of batch fabrication, generally used for the production of the microstructures to be actuated.

Shape Memory Alloy composites could offer a cheap alternative, based on commercially available SMA wires embedded into a polymeric matrix. Integration of SMA fibres in large surfaces is already being investigated, e.g. by the aerospace industry for wing control. However, exploitation of these methods for wafer-scale integration is still in its infancy.

This paper is aimed at introducing a novel technique for producing SMA composites at microsystem level that overcomes the above mentioned limitations.

A fixture jig is used to position the fibres with the correct pitch. They are eventually annealed in order to set the memorized shape and obtain the same stress level amid the wires. The fibres treated as such are then embedded into the polymeric matrix, which had previously been deposited onto the target substrate. Curing of the polymer and patterning of the structures provide the last step to obtaining microactuators.

Coffee break

Thursday morning, 20 September, 10:30

Parallel Session

Devices and Systems II

Thursday morning, 20 September, 11:00

11:00

Oral

Manufacturing Technology for Fabrication a Discrete Actuator using Magnetic Shape Memory Alloy

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For the fabrication of a discrete micro actuator using Magnetic Shape Memory (MSM) alloys, various thin film fabrication technologies had to be investigated. Such an actuator needs an excitation coil system which is able to generate a suffice magnetic field of about 0.4T in the MSM material. This is the field strength necessary for affecting a crystalline reorientation of the MSM alloy which causes to the elongation of the material. Another important area for such a system is to coming up with highly permeable flux guide. In both cases, recent developments of the Institute for Microtechnology (imt) were taken advantage of.

The generation of a sufficiently great magnetic field depends on the thin-film coil design, in particular the number of turns and the cross section of the conductor. The latter determines the coil's current carrying capability. Contrary to previous work, a double layer spiral coil with an aspect ratio of 1.5 to one of the conductor was chosen. It resulted in a conductor with a width of 23.5 µm and a height of 35 µm. There are four turns per layer, resulting in a total number of 8 turns. To increase the filling factor, the insulation between the coil layers was fabricated of Si₃N₄ layer rather than SU-8TM used previously. The deposition was accomplished by Plasma Enhanced Chemical Vapour Deposition PECVD in a process minimizing film tensions.

The second development was in the area of improving the magnetic permeability of the used electroplated soft magnetic alloy. A parameter study was conducted, varying the current density in relation to the coated area, as well as the deposition pulse ratio. As a result, relative permeabilities μ_r of up to 100 and 80 could be achieved for flux guides with a thickness of 40 μm and poles with a height of 70 μm , respectively. This was the value which was required to reach the simulated flux density in the MSM material.

11:15

Oral

NiMnGa nanostructures produced by electron beam lithography and argon ion etching

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NiMnGa nanostructures open up an unforeseen variety of opportunities for the development of novel nanomachines, which may be controlled by local heating and a magnetic field. Nanoindentation experiments on conventional shape memory alloys have already demonstrated the scalability of the shape memory effect to nanometer-sized depths. The purpose of this study is to explore the technologies of electron beam lithography and Ar ion-etching for fabrication of NiMnGa structures with critical dimensions in the sub-micrometer range.

One micron thick films of Ni_2MnGa are deposited on a PVA substrate by RF magnetron sputtering at 200 W. The sputtering target of nominal composition $\text{Ni}_{52}\text{Mn}_{24}\text{Ga}_{24}$ is fabricated by hot pressing. During sputtering the substrate temperature is kept at 50 °C. An Argon gas flow is maintained at 230 mm^3s^{-1} . After release from the substrate the films are heat-treated at 800 °C for 36 ks.

Electron beam lithography is used to generate a primary pattern in a PMMA resist layer, which has been deposited on the Ni_2MnGa thin film. Its thickness is adjusted in the range of 1-3 μm by varying the spin-coating parameters. During the subsequent ion-etching process the resist is used as a protective layer for the functional structures. Therefore, the minimum resist thickness is given by the ablation rates of resist and thin film. The anisotropic behavior of the ion-etching process enables aspect ratios larger than 1, and thus, lateral structures with sub-micrometer dimensions. The process steps of lithography, pattern transfer and electrical contacting are performed on an auxiliary substrate with an adhesive bonding layer in-between, which is removed finally to obtain freely movable structures.

11:30

Oral

Confining Concrete Cylinders using Martensite Shape Memory Alloy wires

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This study proposed a new method to confine concrete cylinders or reinforced concrete columns using martensite shape-memory-alloy wires. The prestrained martensite SMA wire wrapped a concrete cylinder and, then, was heated by a heating jacket. In the process, the

confining stress around the cylinder was developed in the SMA wire due to shape memory effect, which can increase the strength and ductility of the cylinder under axial compressive load. In this study, 1mm diameter of martensite SMA wire was used for confinement. The transform temperature of M_f and A_f of the SMA were 37°C and 84°C, respectively. The cylinders had the length of 300 mm and the diameter of 150 mm. The space between wrapping wires was 2 mm. The unconfined concrete cylinders were tested and their results were compared to those of the confined cylinders with SMA wires. The SMA wire increased the compressive strength and the ductility of the cylinders due to confining effect. After the compressive load, the cylinder was bulged and the SMA wire was strained. At the state, the wrapped cylinder was heated by the heating jacket. Then, a portion of the bulge of the cylinder was recovered and the cylinder showed good strength and ductility in the second compressive test. The study showed the potential of the proposed method to retrofit reinforced concrete columns using SMA wires to protect themselves from an earthquake. **KEY WORDS:** shape memory alloy, martensite, shape memory effect, concrete, retrofit

11:45

Oral

A Novel Locking Coupling Designed For Orthopaedic Spinal Implant

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Introduction: Current orthopaedic implant locking involves tightening a nut against the rod and screw head to form a coupling. With body motion, fretting (wear) at coupling interface can occur, particulate debris is generated and the fixation loosens. The debris is also likely associated with numbers of post-operative complications. To overcome these problems a new locking coupling based on the special properties of nitinol is invented. Use a spinal implant as the first example of its application. The new spinal locking coupling will tightly close and lock the rod when its temperature is raised to 50°C (memory effect). If fretting occurs, the head will further tighten itself around the rod (super-elastic effect).

Methodology: Two versions of coupling have been developed. These new couplings are tested against 4 commonly used spinal implant locking mechanisms using Material Testing System (MTS) testing machine. The coupling is embedded to the setup with its screw perpendicular to the compression axis. The testing protocols include dynamic axial compression, static axial compression (load to failure or reach to the maximum point of 800N), and axial rotation in 20 and 40 degree.

Results: All the couplings survived dynamic axial compression between 330 and 370N. Under axial compression, conventional couplings failed between 570-740N, while the new couplings reached 800N without loosening. Similarly in axial rotation, all conventional devices failed between 1.8 to 5.3Nm, while the new couplings reached 6.5Nm without failure. A 'saw-tooth' pattern to the mechanical testing readout could be seen with the new coupling, and is indicative of the re-tightening effect once fretting occurs.

Discussion and conclusion: The ability of an implant coupling to self-tighten once fretting occurs is a new concept. This is made possible by use of the superelastic properties of NiTi alloys, so that it is able to exert a continuous and constant force on the rod even after tightening.

12:00

Oral

FE modelling of shape memory alloys considering thermo-mechanical couplings and finite deformations

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In this work we suggest a new thermomechanically coupled material model for shape memory alloys. In contrast to many earlier concepts of this kind the present approach includes arbitrarily large deformations. The work is motivated by the requirement to carry out finite element simulations of NiTi stents. Depending on the quality of the numerical results it is possible to circumvent, at least partially, expensive experimental investigations. Stent structures are usually designed to significantly reduce their diameter during the insertion into a catheter. Thereby large rotations combined with moderate and large strains occur. In this process an agreement of numerical and experimental results is often hard to achieve. One of the reasons for this discrepancy is the use of unrealistic material models which mostly rely on the assumption of small strains. In the present contribution we derive a new constitutive model which is no longer limited in this way. In the framework of large deformations this model is able to describe pseudoelasticity as well as the shape memory effect. Additionally a phase transformation criterion is introduced which includes the tension-compression asymmetry, typical for shape memory alloys. This transformation criterion depends on the second and third invariant of the deviatoric stress tensor. A further topic of this work is the efficient implementation of the model into a finite element formulation. One of the key issues in this regard is to fulfil "inelastic" incompressibility in each time increment. Here we suggest a kind of exponential map where the exponential function is suitably computed by means of the spectral decomposition. A series expansion is completely avoided. Finite element simulations of stent structures show that the new concept is well appropriate to demanding finite element analyses as they occur in practically relevant problems.

Lunch break

Thursday afternoon, 20 September, 12:30

Parallel Session

Modeling and Simulation II

Thursday afternoon, 20 September, 14:00

14:00

Invited

A Unified Thermodynamic Model for Ferroic Materials

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The paper presents a unified modeling framework for ferroelectric, ferromagnetic and ferroelastic materials with a particular focus on their hysteretic and nonlinear behavior. At the micro-scale, the physical mechanisms, which produce hysteresis and constitutive nonlinearities in these materials, differ quite considerably. At the meso-scale, however, these materials exhibit very similar features, which can be characterized by multi-well free energy functions typical for phase trans-

formations. Based on statistical thermodynamics and the theory of thermally activated processes, a unified thermodynamic model for the temperature and rate-dependent hysteretic behavior at the macro-scale is derived and illustrated through comparison with experimental data for piezoceramic, magnetostrictive and shape memory alloy (SMA) materials.

The talk concludes with an application of a phase field formulation of the above model. Representative for a number of typical applications such as micro-actuators or stent components, a coupled multi-field finite element analysis of a SMA cantilever beam using COMSOL is presented.

14:30

Oral

A Magnetization- and Strain-Dependent Free Energy Model for FEM Simulation of Magnetic Shape Memory Alloys

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Ferromagnetic shape memory alloys (FSMAs) allow the development of novel kinds of actuators, which can be controlled by a combination of applied stress, temperature, and magnetic field. For simulation and optimization of such actuators having an arbitrary shape, it is important to develop a validated model and an appropriate tool for field simulation such as the finite element method (FEM). The main goal of the present study is to develop a free energy model, which captures the main effects of variant reorientation on the strain and magnetization, and to implement the model in a FEM routine, which takes into account both the actuator geometry and the complex thermo-magneto-mechanical coupling of the material. A free energy function is constructed, which couples strain and magnetic moments in the FSMA material. The associated Gibbs function is used to calculate time-dependent transition probabilities between martensite variants in a magnetic field under applied stress. By keeping track of the variant fractions, the evolution of strain and magnetization is determined. The simulation model is applied to a beam actuator to verify magnetic field-dependent strain and magnetization characteristics. The influence of a coupling term between strain and magnetic anisotropy on these characteristics is discussed.

14:45

Oral

First ab initio determination of the phase transformations in Ni₂MnGa

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Ni₂MnGa is a typical example of a Heusler alloy that undergoes a martensitic transformation.

The high-temperature austenite has a cubic L2₁ structure, whereas below 200 K the structure is orthorhombically distorted. The transformation is completely reversible despite lattice deformations of more than 6% and large strains connected to this change. Ni₂MnGa is attractive

for application in actuators and sensors because magnetism can control its phases.

Before one can systematically use the composition to improve the transition temperature, the phases of the stoichiometric compound need to be understood in detail. Several (modulated) structures have been experimentally observed in the martensite, but their appearance and importance for the shape memory effect are not well known. We, therefore, systematically studied the various phases (austenite, premartensite, and both modulated and unmodulated martensite) and their transitions using ab initio DFT (in the GGA approximation and with the PAW method). We calculated phonon spectra in the quasiharmonic approximation to derive the finite temperature properties. A surprising result is that the soft phonon mode in the cubic structure does not disappear at elevated temperatures. We subsequently used a soft-mode analysis to determine the martensitic and premartensite modulations. We checked that the stable modulated structures do not show soft modes and reproduced the observed transition temperatures quite well. We found that the transition from premartensite to martensite includes both a decreasing c/a ratio and a change in the modulation. Finally, the unmodulated martensite competes with the modulated one at very low temperatures.

Keywords: shape memory, free energy, martensitic phase transition, phonon softening, ab initio

15:00

Oral

Temperature dependence of magnetically induced deformation of Ni–Mn–Ga martensite

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In the present work all known factors contributing to the temperature dependence of the magnetic field induced strain (MFIS) in Ni–Mn–Ga martensite are considered in the framework of statistical model elaborated in [1,2]. These factors are the temperature dependencies of: (i) crystal lattice parameters [3], (ii) Young modulus [4], (iii) saturation magnetization and (iv) thermal fluctuations of the microstress. Both individual and cooperative effects of these factors on MFIS are studied. The effect of the factors (i) – (iv) on the achievable MFIS value and on the characteristic values of magnetic fields, which "switch on" and saturate MFIS, is estimated. It is shown, that all factors affect both the achievable MFIS value and characteristic fields under the real experimental conditions, none of them can be neglected during the quantitative theoretical analysis of the experimental strain–field dependencies obtained for the different temperature values. In addition, the influence of specimen's shape on the characteristic fields is illustrated for different temperature values. For the available experimental dependencies (i) – (iii) and the reasonable set of model parameters the switching magnetic field proved to be equal to 160 kA/m when the temperature was 15 K below the martensite start temperature and to 320 kA/m when the temperature was 45 K below the martensite start temperature. Obtained results agree with the experimental data reported in [5].

References: [1] V.A. L'vov, O. Rudenko, N. Glavatska. Phys. Rev., B 71 (2005) 024421-6. [2] V.A. L'vov, N. Glavatska, I. Glavatskiy, Y. Ge, O. Heczko, O. Söderberg, S.-P. Hannula. Int. J. Appl. Electromagn. Mech., 23 (2006) 75-79. [3] N. Glavatska, I. Glavatskiy, G. Mogilyny, S. Danilkin, D. Hohlwein, O. Söderberg, V. Lindroos and A. Beskrovnij. J. Phys. IV, 112 (2003) 963-967. [4] V. A. Chernenko,

J. Pons, C. Seguí, E. Cesari. Acta Mater., 50 (2002) 53-60. [5] O. Heczko, L. Straka. J. Appl. Phys., 94 (2003) 7139.

15:15

Oral

Theoretical description of the phase transitions in Ni–Mn–Ga shape memory alloys under an external stress along [110] axis

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Cubic Heusler Ni–Mn–Ga shape memory alloys are of special attention, because of their structural and magnetic properties. In this kind of Heusler alloys martensitic phase transitions happen in the ferromagnetic state.

To present the phase diagrams of the cubic ferromagnet we take an expression of free energy [1] that contains only order parameter components causing structural and magnetic transitions with the account of external stress along [110] axis. The dimensionless expression for energy in the case $T_M < T_C$ when a structural transition occurs in a ferromagnetic state is

$$F = T(e_1^2 + e_2^2)/2 + \text{sgn}(b)e_3(e_3^2 - 3e_2^2)/3 + (e_3^2 + e_2^2)^2/4 + B(2^{1/2}e^2 \sin^2 \theta \cos 2\varphi/2 + 6^{1/2}e^2 \sin^2 \theta \cos 2\varphi/2 + 1/6) + K(\sin^2 2\theta + \sin^4 \theta \sin^2 2\varphi)/4 + \sigma(6^{1/2}/6e_3 + 1/2\xi \sin^2 \theta \sin 2\varphi)$$

Here T is the dimensionless temperature, ξ , B is the dimensionless magnetostriction constants, K is the dimensionless magnetic anisotropy constant, σ is the dimensionless stress, e_2 and e_3 are the combinations of the strain tensor, θ and φ are the polar and azimuthal angles of magnetization. In this work we consider the case of $B > 0$ and $K > 0$.

By minimization of free energy (1) with e_2 , e_3 , θ , φ we can obtain the following states.

1. The tetragonal phase T_1 with magnetization $\parallel$ [001] axes.
2. The orthorhombic phase R_1 with magnetization $\parallel$ [001] axes.
3. The tetragonal phase T_2 with magnetization $\parallel$ [110] axes.
4. The tetragonal phase T_3 with magnetization $\parallel$ [-110] axes.
5. The orthorhombic phase R_2 with magnetization $\in$ (001) plane.

The phase diagram with $b < 0$ allows us to describe the experimental sequence of the phase transitions which is observed in [2].

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[1] V.D.Buchelnikov et al., JETP, 92, (2001) 1019.

[2] V.V.Kokorin et al., Fiz. Tverd. Tela (Leningrad) 33, 1250-1252.

Coffee break

Thursday afternoon, 20 September, 15:30

Parallel Session

Modeling and Simulation III

Thursday afternoon, 20 September, 15:50

15:50

Oral

Phase transitions in Ni_2MnX ($\text{X}=\text{In, Sn, Sb}$) Heusler alloys with inversion of exchange interaction

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In ferromagnetic (FM) Heusler alloys the structural transition from high temperature cubic phase to low temperature tetragonal one takes place. The experiments show that in Ni_2MnX ($\text{X}=\text{In, Sn, Sb}$) alloys the next sequence of phase transitions occurs: paramagnetic (PM) cubic phase $\rightarrow$ FM cubic one $\rightarrow$ PM tetragonal one $\rightarrow$ FM tetragonal one [1]. This magnetic behavior can be explained by the existence in these alloys the inversion of exchange interaction [2]. In this work with the help of the Ginzburg-Landau theory the phase diagrams of Heusler alloys with inversion of exchange interaction are theoretically investigated. We used the expression for the Ginzburg-Landau functional from works [2,3]

$$F = \alpha m^2/2 + \beta m^2 \cos\varphi/2 - \gamma m^4 \cos\varphi/4 + \delta_1 m^4 \cos^2\varphi/4 + \delta_2 m^4/4 - \omega_1 m^2 (e_2^2 + e_3^2)/2 -$$

$$\omega_2 m^2 (e_2^2 + e_3^2) \cos\varphi/2 + a(e_2^2 + e_3^2)/2 + b e_3(e_3^2 - 3e_2^2)/3 + c(e_2^2 + e_3^2)^2/4 \quad (1)$$

where m is the normalized magnetization; φ is the angle between the magnetizations of two ferromagnetic sublattices; $e_{2,3}$ are the linear combinations of the strain tensor; $\alpha, \beta, \delta_1, \delta_2, \gamma$ are the exchange interaction parameters; ω_1, ω_2 are the magnetoelastic constants; a, b, c are the linear combinations of the 2nd-, 3rd-, and 4th-order elastic moduli. After minimization energy with respect to the order parameters $m, \varphi, e_{2,3}$ we constructed the phase diagrams on the $(\beta-a)$ plane. The analysis shows that in general case twelve phases exist: three paramagnetic, three ferromagnetic and six antiferromagnetic phases. At certain values of functional parameters (1) we obtained the same sequence of phase transitions as in experimental work [1].

This work was supported by grants RF and CRDF Y2-P-05-19, RFBR 05-08-50341, 06-02-16266, 07-02-96029-r-ural, 06-02-39030-NNSE, 05-02-19935-YaF_a RFBR and JSPS, RF President MK-5658.2006.2 and Human Capital Foundation.

1. T. Krenke, M. Acet, E.F. Wasserman et al., Phys. Rev. B 72 (2005) 014412.

2. C. Kittel, Phys. Rev. 120 (1960) 335.

3. M.A. Fradkin, Phys. Rev. B 50 (1994) 16326.

16:05

Oral

Effect of Magnetostatic Coupling on the Magnetic Shape Memory in Ni-Mn-Ga

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Present publication gives a detailed theoretical concept and also presents the relevant experimental results concerning the effect of the magnetostatic coupling between the twin layers on the magnetic-field-controlled strain response during the mechanical cycling in magnetic field in Ni-Mn-Ga. We also discuss the physical mechanism of this

effect using our latest model developments. Simultaneously we give the most important information about the main structural, magnetic and mechanical properties related to a family of nonstoichiometric Ni-Mn-Ga alloys with different crystal structures. We specially emphasize the fact that the main magnetic and magneto-mechanical properties of MSMA_s are strictly dependent on the details of twin and also fine internal magnetic domain microstructure within the twin bands. These magnetic domains was not expected and usually not taken into account in many early models of MSMA_s. Here we show that their presence may essentially modify the magnetization behavior and field dependence of magnetic driving forces at the twin boundaries in these materials.

16:20

Oral

Magnetic field dependent effects on the structural stability of MSM Heusler alloys

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In magnetic shape memory (MSM) Heusler alloys as Ni_2MnGa strains of up to 10% can be induced by external magnetic fields, making the material a technologically relevant candidate

for magneto-mechanical actuators. Magnetic field induced strains are either produced

by the field-induced motion of martensitic twin boundaries (as in Ni_2MnGa) by shifting the martensitic transformation temperature in a magnetic field (as in Ni_2MnIn).

We present calculations of magnetic field-dependent properties of MSM Heusler alloys by means of density functional theory calculations. The effects of an external magnetic field on structural properties are simulated by fixing the magnetic moments within the framework of the fixed spin moment (FSM) method. We investigate the phase stability of MSM Heusler materials by investigating phonon dispersions at different magnetizations and relate this to nesting features of the respective Fermi surfaces.

16:35

Oral

Origin of the tetragonal distortion in FePd shape memory alloys

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Magnetic shape memory alloys (MSMA) have attracted considerable attention as materials for actuator and sensor applications, due to large magnetically induced strains of up to 10%. A promising MSMA is disordered $\text{Fe}_{70}\text{Pd}_{30}$ with an induced strain of about 6% and a relatively high blocking stress of 10 MPa. We have calculated the electronic structure of disordered $\text{Fe}_{100-x}\text{Pd}_x$ alloys ($15 < x < 50$) in the framework of density functional theory using the full potential local orbital (FPLO) code. Disorder was treated in the coherent potential approximation (CPA). Our calculations show a good agreement with available experimental data on magnetic properties, lattice parameters, and specific heat. The origin of the tetragonal distortion in these completely dis-

ordered alloys is found to be a Jahn-Teller like effect, which allows the system to reduce its band energy in a narrow composition range.

On the basis of our results, we discuss the prospects for an optimization of the alloys' properties by adding third elements, including effects on the magneto-crystalline anisotropy energy.

16:50

Oral

Constitutive modeling of the magnetic field induced strain in MSMA

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Because of their actuating, sensing and power generating capabilities, the magnetic shape memory alloys (MSMA) have drawn an increasing interest in the past few years. Such materials are capable of attaining large actuation strains with actuation bandwidth which is greater than that of conventional shape memory alloys (SMA). In magnetoelastic magnetostrictive materials deformation can be triggered by magnetic field and the corresponding magnetic field induced strains (MFIS) are nonlinear, stress dependent and exhibit a hysteresis. Linear theory which have been most frequently used in practice and have been incorporated in a number of finite element codes, provides constitutive relations which characterize linear elastic, magnetic and magnetoelastic interactions, but neglects inherent hysteresis and nonlinear behaviour of MFIS. On the other hand, three-dimensional theories for magnetostrictive materials which have the capability of accurate modeling domain structure in strain and magnetization together with the corresponding nonlinear response of the homogenized system, are not developed to an extent comparable to similar theories for shape memory materials. In this work we develop a theoretical concept with higher-order energy relations which tends to incorporate some of the above mentioned phenomena. First we construct an appropriate Helmholtz energy relation in terms of temperature and magnetization. Then by invoking a double Legendre transform we introduce the Gibbs energy relation as a function of stress, strain, magnetic field, magnetization, and temperature, and by minimization seek the condition for local minimum in strains and magnetization. Derivation is conceptualized on truncated polynomial energy relations and results obtained from applying the described thermodynamic formalism are scrutinized for available experimental data and compared with some other existing models based on the theoretical concepts developed by Jiles-Atherton and Stoner-Wohlfarth.

Posters

Monday, 17 September

Joint Poster Session (Part I)

Monday & Wednesday

Monday afternoon, 17 September, 15:50

15:50

Poster

E1

Microstructure, martensitic transformation and shape memory effect in Ni_3Ta – novel high temperature SMA

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The Ni_3Ta intermetallic compound is quite known as a strengthening component for high temperature structural eutectic or directionally solidified materials (superalloys, composites etc.) [1]. The alloy of Ni_3Ta stoichiometry was arc-melted from electrolytic Ni and Ta of the highest purity in a pre-gettered argon atmosphere, re-melted 6 times and zone-melted for purification. Zone-melted specimens were homogenised by heat treatment in vacuum at 1673 K for 4 hours. Thin foils for TEM were prepared by Ar^+ ion milling. The bright field image presented in Fig.1 shows variants of martensite present in the specimen. The variants have different widths and can be found throughout the complete sample. By comparing simulated and experimental diffraction patterns, the monoclinic structure ($a=0.45319$ nm, $b=0.51253$ nm, $c=0.8632$ nm, $\beta=100.79^\circ$) as proposed by Zhao et al. [2] has been confirmed. Furthermore, an (001) twinning plane was determined from the diffraction pattern (Fig.2) taken along the [210] zone axis orientation.

It was found that Ni_3Ta undergoes a martensitic transformation (MT) from high (tetragonal) to low (monoclinic) phase with a 50 K temperature hysteresis at elevated temperatures: $M_s=530$ K, $M_f=500$ K, $A_s=550$ K, $A_f=580$ K (Fig.3). Thermal cycling over the temperature interval of the MT does not affect the MT temperatures except for a slight shift of the reverse transformation temperatures during the first cycle. The volume change during forward MT deduced from the austenite and martensite lattice parameters is -0.45%. The shape recovery after 10% compression at room temperature is almost complete although it takes place in a wide temperature interval – about 300 K. This alloy might open new perspectives for the application of high temperature shape memory alloys (HTSMA).

References:

[1] Mollard F. et al., 1974, Zeitschrift Für Metallkunde 65, 461-466

[2] Zhao J-T. et al., 1991, Acta Cryst. CA7, 479-483

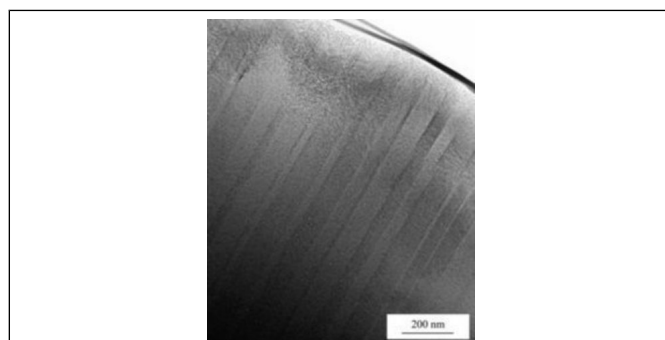


Fig.1

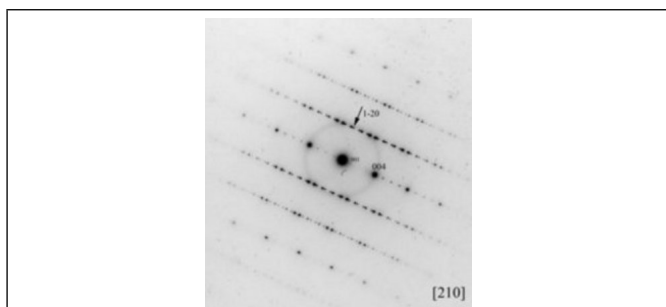


Fig.2

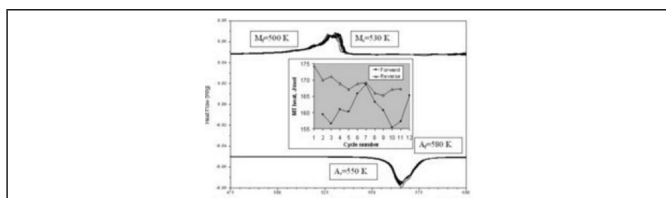


Fig.3

15:50

Poster

E2

Evolution of phase transformations after multiple steps of marforming in Ti-rich NiTi SMA

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The phase transformations associated with Shape Memory Effect (SME) can be one step, B19' (martensite) ↔ B2 (austenite), or two / multiple steps which include the intermediate R phase, depending on the thermal and thermomechanical history of the alloy. The transformation temperatures are generally observed above room temperature in Ti-rich Ni-Ti alloys, while those observed in Ni-rich alloys occur below room temperature.

The goal of the present work is to investigate the phase transformations evolution in Ti-Rich Ni-Ti SMA (Ni-51at%Ti) when subjected to two distinct thermal treatments (500°C for 30 minutes in air and 800°C for 300 minutes in vacuum) and subsequently multiple steps of marforming thermomechanical treatments intercalated with thermal treatments (500°C for 30 minutes in air) and subsequent four distinct final thermal treatments (400, 450, 500 or 600°C for 30 minutes in air). Further, the stability of phase transformations in the initial ten thermal cycles of these thermomechanically treated samples is also studied. Differential Scanning Calorimetry (DSC) and Electrical Resistivity (ER) and X-Ray Diffraction (XRD) were used to identify the transformation temperatures and the phases that are present after the thermomechanical treatments.

15:50

Poster

E3

Nanostructure and properties of the large size Ti_{49.4}Ni_{50.6} alloy samples, subjected to High Pressure Torsion Deformation with subsequent annealings

Aleksander V. Lukyanov¹, Dmitry V. Gunderov¹, Egor A. Prokofiev¹, V. G. Pushin²

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TiNi-based alloys are the functional materials with shape memory effect (SME). It was demonstrated that perspective way of improving properties of metals and alloys is formation of NC structure using methods of the severe plastic deformation (SPD). In earlier works amorphous and nanocrystalline (NC) structures in TiNi have been processed by high pressure torsion (HPT). Subsequent annealings lead to the formation of nanostructured state. However, these works considered only small HPT-processed samples 5-10 mm in diameter and approximately 0.1-0.15 mm in thickness, which hampered the investigation of mechanical properties and shape-memory effects and did not allow to use high-strength NC TiNi for practical application. A new HPT equipment was used in this work for the first time. It allows to produce large size HPT samples of Ti_{49.4}Ni_{50.6} alloy with the diameter of 20 mm and thickness of 1 mm. TEM studies of microstructure showed that the samples processed by HPT had amorphous-nanocrystalline state with the size of nanocrystals less than 20 nm. Fabrication of NC structure by subsequent annealings of such HPT samples is first investigated as well. After annealing at 400°C (1 hour) samples underwent a complete nanocrystallization and an average grain size constituted 30 nm. The subsequent annealing at 500°C leads to grain growth up to 50 nm. Microhardness measurements showed values increasing from initial 1800 MPa to 5600 MPa after HPT processing. Nanocrystalline state results in the record value of strength for this material - 2600 MPa. Thus, HPT combined with subsequent annealings at varying temperatures allowed to obtain integral nanostructured Ti_{49.4}Ni_{50.6} specimens with various grain sizes. Now, it is possible to carry out complex investigations of NC structure effect on mechanical and functional properties of TiNi alloys. Besides, it became possible to use such samples as a semi-product for various practice application.

15:50

Poster

E4

FEM Simulation Of A Nitinol Wire

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In recent years, one of the most promising new actuator technologies is based on the use of Shape Memory Alloys (SMA). The main challenge for the application of devices that use these materials is the hysteresis in the phase transition they suffer during actuation.

Finite element method (FEM) analysis is an important aid in the simulation of mechanical properties and thermal fields in actuators. Dynamic simulations give in many cases enough information without the necessity of building a prototype. We have used ANSYS and its FEM material module to simulate a Nitinol wire used in micropositioning actu-

ator. The accurate model parameters, not given by the supplier but required by the FEM analysis program, have been obtained by thermal and mechanical characterization of the material used.

Phase transition temperatures for wire specimens have been examined by differential scanning calorimetry (DSC) over a temperature range from -30°C to 180°C, with a heating rate of 5°C per minute. These results have been corroborated by electrical resistivity measurements in a climatic chamber (Angelantoni Industrie Challenge 250). Resistivity has been characterized in the range of 25°C to 130°C. Cyclic deformation of the Nitinol wire above the austenite finish temperature has been measured in a SHIMADZU AGS-5 mechanical testing machine.

When the SMA is resistively heated by an electric current applied to the wire the phase transition is induced and results in a contraction of the wire length. When the material cools off, a load applied to the wire stretches it back to the original left. The hysteresis of the system has been measured under different applied loads. These measured hysteresis loops can be compared to the loops obtained from the FEM simulations.

15:50	Poster	E5

Optimised Layout of Shape Memory Actuators by a Numerical Modelling Approach

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Shape-Memory-Alloy (SMA) actuators used in technical applications are still mainly parameterised by trial-and-error methods, because of a lack of computer-aided design tools for this active material. This paper describes a numerical modelling approach to design and optimise the geometry of SMA actuators and the load and heating conditions of the technical system. The Shape-Memory Effect used in most technical Systems is the extrinsic two way effect (2WE).

A numerical model was developed to simulate this effect. It was implemented in MATLAB/SIMULINK[®]. The focus of the model lays on the activation behaviour of the SMA actuator, which defines its rate of heating and cooling. These parameters describe the dynamical characteristics of the actuator and the complete SMA powered system. Various load conditions and actuator geometries and shapes, e.g. wire or spring actuator are simulated by a calculation of the energetic balance of the whole system. The numerical model can be used to simulate time variant heating currents in order to obtain an optimal system performance or match a defined time response of the actuator. It can also be used to calculate the activation behaviour of a SMA actuator with given heating and mechanical loading conditions.

The numerical model is validated by using a testing rig, which is capable of generating all the included parameters and boundary conditions of the numerical model that can be simulated for SMA-wire or SMA-spring actuators. Finally the archived knowledge is use to design the prototype of a SMA-powered Stepping Drive base on the moving principle of a Wave Drive Gear Set. In this concept the external engine is substituted by SMA-Wire-Actuators to combine the advantages of this Drive Set with the huge energy loading capabilities of Shape Memory Materials.

15:50	Poster	E6

New Spinal Implant Using Super-Elastic Nickel-Titanium Alloy For Progressive Deformity Correction: In-Vivo Demonstration In Goat Model

Kelvin Yeung¹, Keith Luk¹, William Lu¹, Jonathan Chung², Paul K. Chu², Chenglin Chu^{2,3}, Roy Chan¹, John K. Lam¹, Xiangmei Liu², Shuilin Wu², Kenneth Cheung¹

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Introduction: Current scoliosis surgery rarely achieves full correction due to visco-elastic properties of spinal tissues. Use of excessive force in attempting a full correction may result in bony fractures or neurological deficit due to spinal cord damage. Our new correction approach is to establish a correction force over the scoliotic spine that may not completely straighten the deformity at the time of operation, but will apply a constant and predictable correction force following surgery, allowing progressive and continuous correction of the deformity without inducing neurological problems or compromise of the implant-bone interface. This study aims to demonstrate in a goat model that the new implant using super-elastic nickel titanium alloy can be used as an internal fixator to safely overcome the viscoelastic properties of the spine and progressively change the spinal curvature.

Methodology: Lumbar spine segments of 5 goats were exposed posteriorly. A pre-contoured super-elastic rod, with a curvature of 60°, is first cooled to 20°C, straightened and then implanted. After implantation, the rods were warmed to body temperature and the rate of scoliosis development initially observed clinically, and then after wound closure by daily radiographs.

Results: Only a 15° curve was induced immediately after surgery. Scoliosis between 25° and 30° developed by 2 days, and progressed to between 45° to 50° by 1 week.

Discussion and conclusion: The results demonstrated that super-elastic rods can be used to progressively create scoliosis. The reverse should also be true, and their use should allow more complete correction of spinal deformities. Previous concerns with Ni release by NiTi alloys have been overcome by recent advances in nanotechnology surface treatment. A new generation of super-elastic spinal implants will help improve safety and efficacy of spinal deformity surgery.

15:50	Poster	E7

Shape memory behaviours of noble metal copper based beta-phase alloys

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A series of alloy systems exhibit a peculiar property called shape memory effect which involves the repeated recovery of macroscopic shape of material at different temperatures. The origin of this phenomenon lies in the fact that the material changes its internal crystalline structure with changing temperature. On cooling from high temperature, these alloys undergo a displacive transition following two ordering transitions. This transition is called martensitic transition and respons-

ible for the shape memory effect. In case these alloys are deformed in a temperature range in martensitic condition they change in shape and recover the original austenitic shape on heating over the reverse transition temperature after removing the strain. These materials regain the deformed shape on cooling to the martensitic state and cycle between deformed and undeformed shapes on cooling and heating. Shape memory alloys have attracted considerable interest as actuators and smart materials in engineering applications, to control the shape change and other properties. The choice of material as well as actuator and sensor to combine it with the host structure is very essential. Martensitic transformation and the associated mechanical shape reversibility in copper-based shape memory alloys is strongly influenced by cycling and ageing effects, due to the metastable character. These materials can be plastically deformed by variant transformations, in case of stress in the martensitic condition. The effects have been investigated on two shape memory CuZnAl and CuAlMn alloys. Ageing effects in CuZnAl alloys have been investigated by X-ray diffraction and optical microscopy. The alloys in the coil form were cycled between the closed and open coil shapes (deformed and undeformed conditions), and elongation parameters were measured following cycles under different loading conditions. Key Words: Shape memory effect, thermal actuators, and thermal cycles.

15:50 Poster E8

Effect of magnetic field on the TA_2 phonon branch softening in Ni_2MnGa

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The softening of the TA_2 phonon branch at $[\xi\xi0] 2\pi/a$, $\xi \cong 1/3$, as seen through inelastic neutron scattering experiments, is well known to be associated to the Parent (P) $\rightarrow$ Intermediate (I) phase, preceding the martensitic transformation (MT) on cooling in Ni_2MnGa alloys. There is still some controversy on the magnetic field dependence of both P $\rightarrow$ I and MT temperatures in these alloys, nevertheless the effect of magnetic field on the TA_2 softening has never been analysed. In this work, preliminary results of the P $\rightarrow$ I softening under a magnetic field are presented.

Neutron scattering experiments were carried out at the Institute Laue-Langevin, at IN3 triple axis spectrometer. A stoichiometric Ni_2MnGa single crystal ([100] axis perpendicular to the scattering plane) has been studied in the 220 - 260 K temperature range. The measurements without applied magnetic field reproduce the softening as reported in "*Phonon anomaly, central peak, and microstructures in Ni_2MnGa* ", A. Zheludev et al., Phys. Rev B 51 (1995) 11310; the minimum in the energy being at $T = 240$ K for $q \cong [0.35, 0.35, 0]$, thus close to $\xi = 1/3$ as expected. At lower temperatures, i.e. 230 and 220 K, the value at the minimum increases and the dip in the branch is less pronounced. Under the application of a magnetic field of 2 T along [100], the branch at 230 K approaches that of 240 K, both being closer to the 230 K branch without applied magnetic field. Therefore a slight decrease in the P $\rightarrow$ I temperature under the application of the field could be inferred, although this is a preliminary qualitative estimation. Additional experimental data, extending both the field and temperature ranges are

required in order to reach a quantitative estimation of the magnetic field effect.

15:50 Poster E9

Superelastic Porous NiTi with Adjustable Porosities Synthesized by Capsule-Free Hot Isostatic Pressing

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Porous NiTi with different porosities were fabricated by capsule-free hot isostatic pressing (CF-HIP). The effects of the cold compaction pressure and TiH₂ addition on the properties of porous NiTi alloys prepared by CF-HIP were systematically investigated. The porosity of the porous materials varies with the cold compaction pressure of the green compacts. TiH₂ addition significantly improves both the porosity and pore interconnection, but slightly impairs the mechanical properties of porous NiTi alloys. In comparison with porous NiTi fabricated by other methods, the stress-strain curves reveal that porous NiTi prepared by CF-HIP has higher compression strength and excellent superelasticity (SE). Furthermore, the phase transformation temperature shifts to a slightly lower value with TiH₂ addition, which favors the superelasticity of porous NiTi at human body temperature.

15:50 Poster E10

Design of an intelligent slades with shape memory materials

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The design of a device called "intelligent slades" is present. The device operate automatically with temperature changes open and closed the glasses of the slades with out employed electrical or electronic components typically employed in conventional automatic systems.

The designing of slades consist of a conventional mark doted with a mechanism which transmit the glasses weight to a set of plates made with a shape memory alloy (SMA).

The mechanism consists in two gears coupled to the mechanism of manual close-open and to a fixed shell which contain several shape memory plates.

For the designing of slades was developing an engineering model for determination of the SMA plate's displacement subject to a punctual variable load in cantilever arrangement.

15:50 Poster E11

Rapidly Solidified Ferromagnetic Shape Memory Alloys

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Ferromagnetic shape memory alloys have been manufactured by various techniques involving rapid solidification. Bulk alloys have been obtained by extracting the melted alloy in especially designed copper molds; glass coated wires have been obtained by drawing the melt from glass recipients followed by water cooling and ribbons have been fabricated by melt-spinning.

Microstructural observation show particular solidification aspects of fractured areas, while ferromagnetic behavior has been detected in glass coated wires obtained by rapid solidification in composition ranges where such a behavior has not been reported. The martensitic structure has been observed on Co-Ni-Ga rapid solidified bulk alloys and Fe-Pd ribbons.

The memory effect has been detected using a Vibran system that measures the shape recovery capacitively for the ribbons and by visual observation for other specimens. The conclusions of the observations are related to the relationship between the ferromagnetic behavior and the shape memory effect during the transition from nanocrystalline to crystalline state.

15:50 Poster E12

Giant Magnetostrain Based on Strong Single Ion Anisotropy of Rare Earth MaterialsMathias Doerr¹, Sebastian Raasch¹, Martin Rotter⁴, Matthias Frontzek¹, Dirk C. Meyer², Manuel Zschintzsch², Pavel Svoboda³, Michael Loewenhaupt¹

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The volume, shape and microstructure of solids can be influenced by magnetic fields [1]. Much effort is focused on magnetic shape memory (MSM) materials. Recently, the MSM effect has been discovered to occur also in the paramagnetic state, e.g. in RCu_2 compounds (R = rare earth) [2]. RSM materials distinguish themselves from conventional MSM materials by the new origin of the magneto-crystalline anisotropy: the strong rare-earth single ion anisotropy. Due to the pseudo-hexagonal symmetry of RCu_2 , three orientational variants exists, each of them rotated by about 60 deg with respect to the others. Switching these variants by an external field results in a change of the macroscopic shape. The strain is in the order of one percent (= Giant MagnetoStrain). The variant's fraction stays unchanged still after ramping down the field. The virgin state can be recovered by heating or by a perpendicularly directed field. This irreversibility shows the potential to construct field controlled actuators or switches.

We present temperature and field dependent measurements of magneto-strain and magnetization at the model substance $Tb_{0.5}Dy_{0.5}Cu_2$. The macroscopic characterization of the sample is complemented by a detailed microscopic analysis done by elastic neutron scattering. Although

the GMS effect of RCu_2 was worked out at single crystals, the principle of this magneto-mechanical coupling phenomenon is also useful for polycrystalline or microscaled applications. Futural preparation and characterization of thin or free-standing films are necessary to construct micro-mechanical actuators.

[1] M. Doerr et al.: Adv. Phys. **54** (2005) 1.[2] S. Raasch et al.: Phys. Rev. **B 73** (2005) 64402.

15:50 Poster E13

Residual stress in Ni-Mn-Ga thin films deposited on different substratesStephen Doyle¹, Volodymyr A. Chernenko², Stefano Besseghini³, Andrea Gambardella³, Manfred Kohl⁴, Peter Mullner⁵, Makoto Ohtsuka⁶

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Great attention has been recently paid to the development and study of MSM Ni-Mn-Ga/substrate thin film composites. The workability and reliability of small-scale devices utilizing such materials is highly dependent on the residual stress which such thin films exhibit due to the thermal expansion and lattice mismatches.

In the present work we report on residual stress measurements by X-ray diffraction in the standard $\theta/2\theta$ Bragg-Brentano geometry carried out at the ANKA synchrotron facility (FZK, Karlsruhe). In the method commonly known as " $\sin^2\psi$ ", the lattice spacing is assumed to be a linear function of $\sin^2\psi$, where ψ is the angle between the diffracting plane normal and specimen surface normal. A biaxial plane-stress model is then used to convert the measured strain into stress.

Four series of $Ni_{51.4}Mn_{28.3}Ga_{20.3}$ /substrate thin film composites, where the substrates are either Si(100), MgO(100), alumina or molybdenum foil, and three series of $Ni_{53.5}Mn_{23.8}Ga_{22.7}$ /substrate composites where the substrate is either Si(100), alumina or Mo foil, with different film thicknesses varying from 0.1 to 5 μm have been studied in the cubic phase by XRD stress measurements. An analysis of the results was made assuming the value of Young modulus and Poisson ratio to be equal to 50 GPa and 0.3, respectively, for all the films. The zero-stress lattice spacing was determined from the condition of $\sin^2\psi = 0$. The estimated level of residual stress is found to vary from 0.12 GPa for the 1 μm -thick film on MgO(100) to 0.53 GPa for the 0.1 μm -thick film on Si(100). In addition, a weak thickness dependence of stress is observed for films on MgO or Mo while it is much stronger and has the opposite sign for films on alumina or Si(100). The results are relevant to the evaluation of stress-induced magnetic anisotropy of films.

15:50 Poster E14

Ferromagnetic resonance in Ni—Mn-Ga thin films and thin-film tubesJanusz Dubowik¹, Iwona Goscińska², Yuri Kudryavtsev³

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Ferromagnetic resonance (FMR) has been proven to be useful tool to determine magnetic properties of thin films. The FMR data (the resonance fields and linewidth) for thin films enable evaluation of their effective magnetization, magnetic anisotropies, and their homogeneity and relaxation mechanisms. Usually the films are firmly fixed onto substrates and thus suffer a huge stress – tensile or compressive. We report on FMR experiments performed for the first time on thin (100 nm) Ni-Mn-Ga films clamped to the mica substrates and then fully released from them. The aim is to evaluate the role of magnetoelastic coupling in stressed Ni-Mn-Ga Heusler alloy films that undergo martensitic transformation. The as-deposited films are usually under stress and they form curled thin-film tubes after releasing from the substrates. We will discuss and compare the resonance conditions for metallic films and, in particular, for thin-film tubes and explain a peculiar FMR response in thin-film tubes as resulting both from a small microwave penetration depth and a specific angular dependence of the FMR linewidth in thin films. The temperature dependencies of the resonance field in the Ni-Mn-Ga films and thin-film tubes are measured in a wide temperature range of 78 – 400 K. The martensitic transformation is shown to occur in a range of 220 – 300 K. The experimental results show that the difference in the effective magnetization $4\pi(M_{\text{eff}}^{\text{tubes}} - M_{\text{eff}}^{\text{films}})$ is negligible in austenite phase and it increases to about 1 – 1.5 kG at temperatures well below the martensitic transformation. The data suggests that magnetoelastic coupling in the martensite phase of Ni-Mn-Ga thin films is typical of normal thin magnetic films with magnetostriction of about 50 ppm. The results will be compared with the FMR data obtained on Ni thin films and Ni thin-film tubes.

15:50 Poster E15

Powder metallurgy technology of NiTi shape memory alloyJan M. Dutkiewicz¹, Wojciech Maziarz¹, Tomasz Czeppe¹, Wojciech K. Nowacki², Stefan P. Gadał², Jerzy Luckner², Elżbieta A. Pieczyńska², Lidia Lityńska-Dobrzyńska¹

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Nickel-titanium alloys shaping and casting technology is difficult due to a high reactivity of titanium and brittleness in the as cast state. Therefore, powder metallurgy technology offering new technological possibilities and in addition controlled porosity was applied. Shape memory alloy was compacted from the prealloyed powder delivered by Memry SA showing $M_s = 10^\circ\text{C}$ and $A_s = -34^\circ\text{C}$ as results from DSC measurements. The hot compaction in the as delivered spherical particle's state was performed in a specially constructed vacuum press,

at temperature of 680°C and pressure of 400 MPa. The alloy powder was encapsulated in copper capsules prior to hot pressing to avoid oxidation or carbides formation. The alloy after vacuum high pressure and temperature of 680°C compaction (i.e. above the NiTi stability range) has shown similar transformation range as the powder. The porosity of samples compacted in the as delivered state was only 1%. TEM studies shown presence of small amount of B19' martensite and Ni_4Ti_3 particles at room temperature. The samples tested in compression up to $\epsilon = 0.06$ have shown partial superplastic effect and returned back to $\epsilon = 0.01$ after unloading. They have shown also high ultimate compression strength of 1600 MPa. Measurements of the samples temperature change during deformation allowed to detect temperature increase up to 2 degrees during loading and similar decrease during unloading.

15:50 Poster E16

Martensitic transformation and shape memory effect in NiTiCu strip covered by nanocrystalline platinum layerTomasz Goryczka¹, Jozef Lelatko¹, David Vokoun², Patrick Och³

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NiTi-based shape memory alloys (SMA) have been known from their practical application, especially, in medicine and biomedical engineering. However, risk of its influence on a human body is still reported. In order to prevent diffusion of nickel ions, various surface modification have been proposed. Coatings with nitrides, oxides or carbides seem to be an attractive way to create a barrier, which sufficiently reduces corrosion resistance. However, too thick and/or stiff layer can limit occurrence of the martensitic transformation. In consequence shape memory effect may be spoilt.

Proposed paper is focused on characterization of structure and thermal behaviour of the martensitic transformation in $\text{Ni}_{25}\text{Ti}_{50}\text{Cu}_{25}$ shape memory strip covered by thin nanocrystalline platinum layer. The strip was produced applying twin roll casting technique and covered by platinum layer using magnetron sputtering. Structure of the layer was studied by means of X-ray and electron diffraction. Transformation temperatures were determined from measurements carried out using differential scanning calorimetry (DSC). The layer reveals nanocrystalline structure with average grain diameter of 43 nm. Almost 25% of total grains are oriented along $\langle 111 \rangle$ direction. It was stated that the nanocrystalline platinum layer does not limit martensitic transformation in the covered strip, which reveals one step reversible martensitic transformation from the parent B2 phase to the orthorhombic martensite B19.

15:50 Poster E17

Transformation and twinning processes in CoNiAl alloy single crystalsSilvia Ignacova, Vaclav Novak, Petr Sittner

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Ferromagnetic shape memory alloy CoNiAl exhibiting paramagnetic/ferromagnetic transition besides a thermoelastic martensitic transformation is a promising material for high temperature and magnetic

shape memory applications. Due to the two-phase structure ($\beta+\gamma$) of the alloy, the Curie temperature and the martensitic transformation temperatures can be adjusted in the temperature range -150°C to 150°C by varying chemical composition of the β -phase or through annealing. In this work, $\text{Co}_{38}\text{Ni}_{33}\text{Al}_{29}$ shape memory alloy single crystals grown by Bridgman method were heat treated to obtain desired functional behavior. Stress induced transformation and twinning processes in this alloy were investigated on prism shaped samples deformed in compression in wide temperature range. Pseudoelastic stress-strain response at room temperature was characterized by a curious softening curve with narrow hysteresis (around 50 MPa) and 4% transformation strain for [001] load axis orientation. Twinning processes in L1_0 tetragonal martensite (test temperatures -5°C to -50°C) take place at very low stress $\sim 1\text{MPa}$, in contrast to available literature reports. Experimental results are analyzed and discussed in respect to twinning processes in studied alloy.

15:50 Poster E18

Effect of oxidation on transformation and deformation behavior in Ni-Ti alloy

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Since Ti reacts preferably with oxygen to form titanium oxide, Ni content increases between the matrix and the oxide, forming compounds and Ni-rich region. Because transformation temperature of Ni-Ti alloy depends strongly on Ni contents, the Ni-rich region affects transformation as well as deformation behavior. It is found out that the oxidation resultant including Ni-rich region lowers M_s and A_s temperatures in the alloys undergoing $B2 \rightarrow M$ transformation. In case of alloy of $B2 \rightarrow R \rightarrow M$ transformation, a significant decrease in $B2 \rightarrow R$ transformation temperature is observed near surface. Besides, no $R \rightarrow M$ transformation is observed when it is cooled to 123K, while M_s of the matrix is 250K. The Ni-rich region experiences more plastic deformation than the matrix during superelastic deformation, since slip occurs instead of formation of stress-induced martensite. It is summarized that increase in Ni content near surface by oxidation retards martensitic transformation and prevents SIM deformation by lowering enormously transformation temperature.

15:50 Poster E19

Cyclic Stability in Ultrafine Grained High Temperature Shape Memory Alloys

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NiTi alloys are the most widely used shape memory alloys (SMAs) due to their superior mechanical and functional properties, however their use is restricted below 100°C . In order to extend their utility in high temperature applications, higher martensitic transformation temperatures than 100°C , lower temperature hysteresis, and better cyclic reversibility are required. The transformation temperatures of NiTi alloys can be increased by the addition of Hf and Pd, however, these

additions lead to either the degradation of thermal cyclic response under stress due to the decrease in critical stress for slip or the reduction in fracture toughness due to the formation of detrimental precipitates/inclusions. In this study, severe plastic deformation via equal channel angular extrusion (ECAE) was used to increase the critical stress for slip by grain refinement down to submicron to nanometer range (100 to 300 nm) and by designing specific textures in NiTi and NiTi(Pd,Hf) alloys. A significant improvement in the thermal cyclic stability of the ECAE processed samples was shown which was attributed to the grain refinement. In addition, the severe simple shear deformation led to the dissolution and/or fragmentation of precipitates (mainly oxides or carbides) into very small sizes increasing the fracture strength of ternary high temperature SMAs. In this talk, we will summarize methods of engineering microstructure and texture using ECAE and resulting thermomechanical response including temperature hysteresis and enhanced cyclic reversibility in high temperature SMAs.

15:50 Poster E20

Generation and relaxation of reactive stress in active elements from shape memory alloys

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The main requirement to the active elements of communicative products as thermorelay is supply of operating curve constancy (force, deformation and operation temperatures at a given rigidity of counterbody). The most and sometimes even the determinant factor is the force, that develops in material with shape memory alloys (SMA) while heating by the given temperature.

The researches were carried out at the alloys of TiNi, which were subject to an annealing at 500°C , during 30 minutes after cold drawing, with temperatures of martensite transformation (MT):

Ti-50,2 at% Ni: $M_s = 16^{\circ}\text{C}$, $M_F = 12^{\circ}\text{C}$, $T_R' = 32^{\circ}\text{C}$, $A_s = 46^{\circ}\text{C}$, $A_F = 50^{\circ}\text{C}$;

Ti-50,0 at% Ni: $M_s = 62^{\circ}\text{C}$, $M_F = 40^{\circ}\text{C}$, $A_s = 80^{\circ}\text{C}$, $A_F = 86^{\circ}\text{C}$.

The effects of generation and relaxation depending on their growth rate and thus decrease of reactive force level at a given values of prestrain on the conditions of infinite rigidity ($K \rightarrow \infty$) and given rigidity of counterbody were studied in complete and incomplete intervals of temperatures MP. In the curve $P_r = f(\epsilon_{mp})$ two areas can be observed: linear and nonlinear. Relaxational capability of the materials was surveyed from the stress (P_r), registered at the linear area.

Thermomechanical hysteresis closes here, as a rule. However, by the multiple cycle repetition, heat « cooling of the operation temperature change. This fact is connected to the capability of the metals to relaxation, and also to the type of preliminary mechanical or thermal processing of alloy materials with SMA. Certain attention should be paid to the type martensite reaction. It is shown that alloy materials, experiencing the martensite transition $B2' \rightarrow B19'$, through the R-phase, provide high convertibility of the deformation in fairly narrow temperature range (till 10°C). Subjects, connected with operability of the active elements while bending and extension under the stress.

15:50 Poster E21

Effects of Fe and Co addition on the magnetoresistance in Ni-Mn-Ga Films

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Ni₂MnGa-type ferromagnetic shape memory alloy is one of the most promising candidates for smart systems. Thin film formation of Ni₂MnGa alloy is especially effective for the micro-systems application. Recently, we found that the heat-treated Ni-Mn-Ga-Fe sputtered films with Heusler type crystal structure exhibited both magnetic field induced shape memory effects and magnetoresistance (MR) effects [1]. The MR properties of the films are of interest in view of application to the smart systems. Addition of 3d transition element *M* such as Co or Fe to the Ni-Mn-Ga films has a potential for expansion of the operating range, because it enhances the Curie temperature *T_C*. Therefore, our purpose is to investigate the effects of Fe and Co addition on the MR in Ni-Mn-Ga films. Films with different Fe and Co composition *x* were prepared by co-sputtering method. The obtained films were heat-treated at 1073 K for 1 h and aged at 673 K for 1 h in a constrained condition. MR and magnetization σ were measured at fixed temperatures as a function of magnetic fields of up to 15 kOe by using four-point probe and vibrating sample methods, respectively. Martensitic transformation temperature *T_M* was determined by resistance-temperature measurements. With increasing *x*, σ enhanced for *M* = Fe, while it reduced for *M* = Co. These results are consistent with those in bulk alloys [2]. At 300 K, MR value at 15 kOe *MR*₁₅ was as low as 1%. The value of *MR*₁₅ was found to be decreased with increasing Fe content. In the case of Co, *MR*₁₅ increased with *x*. From these results, it was suggested that an origin of the MR in the present films is dominated by a s-d scattering model [3].

[1] K. Koike, M. Ohtsuka, Y. Honda, H. Katsuyama, M. Matsumoto, K. Itagaki, Y. Adachi and H. Morita, J. Magn. Magn. Mater. 310 (2007), e996.

[2] D. Kikuchi, T. Kanomata, Y. Yamaguchi, H. Nishihara, K. Koyama, H. Watanabe, J. Alloys Compd. 383 (2004), 184.

[3] M. Kataoka, Phys. Rev. B 63 (2001), 134435.

15:50 Poster E22

The martensite structure and the characteristics of thermoelastic martensitic transformation in Fe-Co based ferromagnetic alloys

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The Fe-Co-Ni-Ti alloy system belongs to the family ferromagnetic shape memory materials. Fe-Co-Ni-Ti alloys with different thermal hysteresis of martensite transformation have been studied by means

of optical microscopy and X-ray diffraction at room and low temperatures. The characteristic temperatures of martensitic transformation were determined by the low-field magnetic susceptibility. The thermal hysteresis, *M_s* temperature, martensite tetragonality and transformation volume change have been measured for specimens with different time of aging. Correlations among these factors are discussed. The influence of thermocycling over a temperature range of $\gamma \leftrightarrow \alpha$ transformation on features of the latter in Fe-Co-Ni-Ti alloys was studied.

Keywords: Fe-Co-Ni-Ti alloy, ferromagnetic shape memory alloys, thermal hysteresis, thermocycling

15:50 Poster E23

Magnetodeformations influence on spin-electron transport in nanofilms

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Heusler Ni_{2+x+y}Mn_{1-x}Ga_{1-y} ferromagnetic alloys with shape memory effect as on temperature, pressure, so on outer magnetic field are owing a number of phase transitions (PT) depending on stoichiometric constitution and alloying with other elements (Fe, Co, Al, etc.): ferromagnetic, premartensitic, martensitic and orientational PTs. With some constitutions of Heusler alloys PTs can be realized at room temperature. For Ni_{2.15}Mn_{0.81}Fe_{0.04}Ga temperatures of martensite-austenite transition and Curie are *T_M*=325 K and *T_K*=375 K and for Ni₅₃Mn₂₅Ga₂₂, Ni_{2.18}Mn_{0.82}Ga magnetic and structure PTs are coinciding.

Ferromagnetic films of Heusler alloys are showing PT at lower temperatures and magnetic fields in comparison with volumetric specimen. They also have substantial magnetodeformations (~1%) causing change of form surface and reallocation of a “down spin” electrons and “up” ones. Most strong influence of magnetodeformations on spin-electron transport is supposed to be in nanofilms of Heusler Ni₅₃Mn₂₅Ga₂₂ and Ni_{2.18}Mn_{0.82}Ga alloys with combined magnetostucture transition.

A quantum-mechanical calculation of quantum stages and zone structure of spoken above compositions was carried out with taking into account hybridization of s and d(f) electrons FPLMTO method. Computer modeling used molecular dynamics and half-electric quantum-mechanical methods. Shown that ~1% magnetodeformation in Ni₅₃Mn₂₅Ga₂₂ causes spin reorientation in ~10% free electrons.

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15:50 Poster E24

Magnetic and structural properties of non-stoichiometric Ni-Mn-Ga Ferromagnetic Shape Memory Alloys

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Ferromagnetic shape memory alloys are able to develop large magnetic field induced strains, up to 10% in non stoichiometric Ni-Mn-Ga alloys, making them very interesting for basic studies and applications. Structural and magnetic transition temperatures of those alloys present

a strong dependence on slight departures from the stoichiometry, as does the mobility of twin boundaries responsible for the large magnetic field induced strains.

In this work we study four non stoichiometric Ni-Mn-Ga polycrystalline alloys with compositions of 43-52 at. % nickel, excess manganese and deficient in gallium, and a single crystal of composition $\text{Ni}_{52}\text{Mn}_{26}\text{Ga}_{22}$. Those compounds are of technical interest due to the observed large room temperature magnetic field induced strains. We report the characterization of martensitic transition temperatures and determination of martensitic phase structure of these alloys in order to complete previous neutron diffraction results on the structures of the austenitic phases.

Calorimetric and magnetic measurements determined the martensitic transition and Curie temperatures of the alloys ($A_s = 331$ K and $T_{\text{Curie}} = 366$ K for 52 at. % nickel alloy). Nickel defective alloys present a martensitic transition region broader than excess nickel ones. This fact is directly related to the structures of the respective martensitic phases. Neutron powder diffraction analysis confirmed orthorhombic martensitic structures for nickel defective alloys (cell parameters for 49 at. % nickel are $a=4.217\text{\AA}$, $b=4.213\text{\AA}$ and $c=5.548\text{\AA}$), and tetragonal for excess nickel ones (cell parameters for 52 at. % nickel are $a=4.208\text{\AA}$ and $c=5.561\text{\AA}$). In the last case the crystallographic structure of the martensitic phase was also obtained on a single crystal with the same composition, trained to get a single variant, and it agrees with the determined in the powder sample.

15:50 Poster E25

Martensitic transformation in melt-spun NiMnGa ribbons

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Ferromagnetic shape memory alloys have been extensively studied since they were first time reported by Webster. He found that in a ternary intermetallic Ni_2MnGa alloy the parent phase, despite of the thermal and stress gradients, transforms to the martensite in a ferromagnetic state. The martensitic transformation may occurs as a one-step transition from the parent phase to one of martensites ($P \leftrightarrow 10M$, $P \leftrightarrow 14M$ or $P \leftrightarrow T$) or as a multi-step one with the longest sequence: $P \leftrightarrow 10M \leftrightarrow 14M \leftrightarrow T$.

Off-stoichiometric NiMnGa alloys, with various chemical compositions, were produced by melt-spinning technique. Transformation behavior was studied by means of differential scanning calorimetry (DSC). Ribbons and bulks reveal reversible martensitic transformation, which occurs in the range of temperatures: between 100°C and -150°C . Transmission electron microscope and X-ray diffractometer were used for structure examination. Dependently on chemical composition five-layered (10M) or seven-layered (14M) martensite was observed, whereas the parent phase shows the B2 structure. Due to high cooling rate realized in melt-spinning technique some amount of amorphous phase was found.

15:50 Poster E26

Alternative MSM alloy systems: Ni-Fe-Ga-Co and Ni-Co-Al

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The well-studied Ni-Mn-Ga system exhibits several drawbacks such as high brittleness and low working temperature (defined by martensitic transformation temperature T_m and Curie temperature T_C). The alloy systems Ni-Fe-Ga-Co and Ni-Co-Al are promising alternatives, as they are ductile and their working temperatures are similar or even higher compared to Ni-Mn-Ga. Here, we prepared both alloy systems by arc-melting. T_m and T_C were determined by DSC, SQUID magnetometer and VSM measurements and correlated with structural data.

For the quaternary Ni-Fe-Ga-Co, T_m and T_C can be tailored in a wide range by adjusting the composition and heat treatment. The effect of aging on the transition behaviour of a $\text{Ni}_{52}\text{Fe}_{17}\text{Ga}_{27}\text{Co}_4$ alloy was investigated. The magnetic transition in the annealed alloy exhibits a pronounced temperature hysteresis, indicating a coupled magnetocrystallographic transition. Aging at 400°C leads to an increase of both hysteresis of the martensitic and the magnetic transitions. However, the martensitic transformation itself and the magnetic transition hysteresis disappeared after aging at 500°C . The reason for this is the formation of γ precipitates, which suppress the occurrence of martensitic transformation.

For the ternary alloy Ni-Co-Al ten alloys with compositions $\text{Ni}_{32.5+X}\text{Co}_{39.5-X}\text{Al}_{28}$ and $\text{Ni}_{33.5+X}\text{Co}_{39.5-X}\text{Al}_{27}$ with $X=0/0.5/1/1.5/2$ were studied. Homogenisation was performed at 1300°C for 5 hours in a quartz ampoule filled with argon. Sample preparation for the subsequent measurements has a pronounced effect on the martensitic transformation. Simply crushing the samples in order to obtain small pieces for the measurements results in the absence of any martensitic transformation. A second annealing (again 1300°C 5h) yields $T_m = 0-120^\circ\text{C}$, depending on composition. Further, two effects were found to have an influence on T_m , the quenching procedure after annealing and low temperature annealing during measurements. Moreover $T_C = 80-100^\circ\text{C}$ is less dependent on composition.

15:50 Poster E27

Deformation of Ni-Mn-Ga martensite under the conditions of ultrasonic resonance

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A strong effect of the alternate mechanical stress on the process of magnetically/mechanically induced deformation (MID) of Ni-Mn-Ga martensite has recently been reported in the frequency range from 30 Hz to 10 kHz [1], being well below the longitudinal resonance frequency of the samples used ~ 100 kHz. To carry out the resonance ultrasonic experiments in the Ni-Mn-Ga martensite under magnetic field or/and axial mechanical stress is an issue of the day and the present communication is aimed at the stimulation of such experiments. To

this end, the process of deformation of rod-shaped twinned specimens affected by standing ultrasonic waves and slowly increasing field/stress has been studied in the framework of a statistical model of MID [2]. The additional deformation of the Ni–Mn–Ga specimen activated by the ultrasound in the presence of magnetic field or axial stress has been computed for several realistic values of wave amplitude and the actual values of Young elastic modulus, specimen length, and threshold field/stress (i.e. the field/stress, which "switches on" the process of MID). The decrease of the threshold field/stress under the influence of ultrasound is described quantitatively. It has been shown, in particular, that ultrasonic waves with a strain amplitude equal to $4 \cdot 10^{-5}$ cause a $\sim 30\%$ decrease of the threshold field/stress value; the additional deformation induced by the wave reaches its maximal value $\sim 2.5\%$ when the field/stress reaches the initial (i.e. obtained in the absence of ultrasound) threshold value. The slow evolution of MID in the stationary magnetic due to switching on the ultrasound has also been modeled.

References: [1]. B.W. Peterson, J. Feuchtwanger, J.M. Chambers, D. Bono, S.R. Hall, S.M. Allen, R.C. O'Handley, J. Appl. Phys., 95 (2004) 6963-6964. [2]. V.A. L'vov, O. Rudenko, N. Glavatska, Phys. Rev., B 71 (2005) 024421-6.

15:50 Poster E28

Microstructure changes in two phase $\beta+\gamma$ Co-Ni-Al ferromagnetic shape memory alloys in relation to Al/Co ratio

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The microstructure of annealed at 1200°C and water quenched $\text{Co}_{35+x}\text{-Ni}_{40-x}\text{-Al}_{25}$ ($x = 0, 2.5, 5.0$) ferromagnetic shape memory alloys has been investigated by optical (OM) and analytical transmission electron microscopy (TEM). The deformed microstructure consisted of elongated grains of martensite and γ phases as observed using polarized OM and Nomarski contrast. The annealing twins were found frequently in the γ phase. The volume fraction of γ phase has been determined using quantitative image analysis method. TEM observations revealed frequent stacking faults within martensite and its teragonality as well as dissociated dislocations within γ phase. The crystallographic relationship between martensite and γ grains has been determined basing on electron diffraction patterns. The elemental mapping technique and chemical point analyses allowed to determine the composition of particulate phases. The measured Al/Co ratio in the β phase of quenched alloys has been shown to be related to M_s temperature.

15:50 Poster E29

Characterization and properties of a modified NiTi shape memory alloy by N^+ ion implantation.

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NiTi alloys with a near equiatomic composition exhibit excellent shape memory effect, superelasticity, corrosion resistance and biocompatibility, tolerance to severe environmental conditions, and thus is one of the most important engineering materials. Their particular and superior properties are caused by a reversible thermoelastic martensitic transformation. NiTi shape memory alloys has been modified by N^+ ion implantation. In general, implantation process may be one solution to enhance the mechanical properties of surface, corrosion, wear and fatigue resistance of such instruments. The ion implantation method has precision capabilities for modification of thin surface layers on metals and alloys including their chemical composition, crystal structure, physical and mechanical properties.

The presented work is aimed at the investigation of structural changes in the modified near-surface layer of NiTi alloy, caused by ion implantation as well as at their influence on the mechanical and shape memory properties of this material. The investigations were carried out on an equiatomic commercial NiTi shape memory alloy. In the process of ion implantation of nitrogen at dose 10^{18}cm^{-2} and 55keV energy, the well-defined double-layer structure with different microstructure as well different chemical and phase composition was formed.

Differential scanning calorimetry was used to characterize the transformation sequence and transformation temperatures for the initial and surface-modified materials. The modifications of the phase composition before and after irradiation are studied at the room and martensitic transformation temperatures by X-ray diffraction methods. Experimental results of an structural-phase condition and functional mechanical properties of NiTi alloys are compared and discussed in this paper.

15:50 Poster E30

Effect of the Heat Treatment on the Pseudoelastic Properties of TiNi Wire for Medical Applications

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The unique properties of shape memory alloys (their ability to remember their shape, superelasticity, biological compatibility with human organism tissues) allow applying them successfully in medicine for a

long time as a perspective material for making implants and different medical devices.

In this given work the results of the pseudoelastic properties investigation of TiNi wire using for medical application are presented. The performed study is aimed on the effect of time and aging temperatures on the transformation temperatures, the width of the transformation hysteresis, and also, on the level of the yield stress and the meaning of the permanent strain by bending deformation of the investigated specimens at the body temperature (i.e. in austenite state). The heat treatment of the wire was realized at the temperature range 250–550°C with a number of different heat stages lasting between 15 and 120 minutes. The investigation of the effect of deformation wire cycling on its phase yield stress and the width of hysteresis loop stress-strain was carried out. Also the effect of ultrasonic vibrations on pseudoelastic and deformation properties of the material was studied. The results of the performed researches allowed optimizing the operating practices of the medical products manufacture from TiNi alloys.

15:50 Poster E31

Magnetic field induced strain and training process in NiMnGa single crystal

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Magnetic field applied in the direction of the hard magnetization axis causes rotation of the lattice (twinning) of tetragonal NiMnGa martensite single crystal in such a way that easy axis of magnetization becomes aligned with the field and the crystal changes its dimension resulting in magnetic field induced strain (MFIS) reaching theoretically ~6%. Twinning interfaces move only if the magnetocrystalline anisotropy energy is larger than the twinning stress [1]. The reverse reorientation is achieved by mechanical action of elastic spring when the magnetic field is switched off. This is well known as the physical principle of MSM actuator. It is known to much lesser extent that, in order to optimize the actuation functionality, the NiMnGa crystal has to be first “trained”. It is not clear why this is needed and what the training actually does to the material.

In this work, the effect of training on magnetic actuation was investigated. Training of the NiMnGa martensite single crystal was performed by successive compressive deformations on two different faces of cuboid sample. When magnetic field was applied along the loading directions of the training, MFIS of ~5% was observed. No MFIS was observed by application of the magnetic field in the third perpendicular “untrained” direction, though it should be so, theoretically. The influence of the training effect and the number of compressive cycles needed to observe the MFIS is studied. Observed results are confronted with the measured characteristics of stress-strain curves.

[1] J. Enkovaara, A. Ayuela, A. T. Zayak, P. Entel, L. Nordstrom, M. Dube, J. Jalkanen, J. Impola, R. M. Nieminen, Mat. Sci. Eng. A 378 (2004) 52-60.

15:50 Poster E32

Heating rate dependence of shape recovery stress in a Ti-49.7Ni alloy

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Effect of heating rate of shape recovery stress of a solution treated Ti-49.7Ni alloy was investigated by means of differential scanning calorimetry(DSC) and tensile tests. Shape recovery stress increased from 80 MPa to 100 MPa with increasing pre-strain from 1 % to 3 %, and then it went down to 80 MPa with increasing pre-strain up to 6 %, which was ascribed to the plastic deformation occurred during loading. Shape recovery stress generated by heating above Af(reversre transformation finish temperature) decreased by cooling and it disappeared at about 313 K, which is very close to Ms(martensitic transformation start temperature). Shape recovery stress measured after 3% pre-strain increased from 100 MPa to 140 MPa with decreasing heating rate from 171 K/min to 1.5 K/min, which was ascribed an adiabatic transformation condition developed by high heating rate. Stress plateau was observed on stress-temperature curves obtained at heating rates higher than 100 K/min. Moreover, shape recovery stress increased from 140 MPa to 308 MPa with increasing heating temperature from 363 K to 453 K which is very higher than Af measured from DSC(348 K). The increase in shape recovery stress above Af was attributed to the increase in Af by stress applied on the specimen.

15:50 Poster E34

The effect of γ -phase particles on microstructure and properties of Co-Ni-Ga alloys

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The paper concerns the determination of the role of the γ -phase particles on the microstructure and properties of Co-Ni-Ga alloys. The investigations cover the alloys of wide range of chemical composition. They show different microstructures: single phase martensite BCT(Body Centered Tetragonal) lattice with $c/a > 1$ (a), two phase B2 + γ (b) or BCT + γ (b). The particle analysis was performed to determine the amount of the g-phase in the studied alloys. The compression test on prism samples proved that the increase of the g-phase volume fraction significantly improves the strength and plasticity of the alloys. Thus the presence of the γ -phase precipitates cause the decrease of the brittleness observed in the single phase martensitic alloys. Additionally, the influences of the orientation of the grain boundary precipitates on the mechanical properties were studied. TEM observations were performed for specimens before and after deformation to reveal the mechanism of the γ -phase deformation. Studies of the para – to ferromagnetic and martensitic transformation were carried using different methods .

15:50 Poster E35

Reversible and irreversible martensitic transformations in Fe-Pd and Fe-Pd-Co alloys

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Ferromagnetic Fe-Pd shape memory alloys (SMA) undergo a martensitic phase transformation during cooling from a parent FCC phase to a tetragonal FCT martensite. This transformation is thermoelastic and reversible and has associated the shape memory effect. On further cooling an irreversible FCT to BCT transformation occurs that destroys the memory effect. Nevertheless, the transformation from reversible to irreversible phase is not complete since a volume fraction of the alloy is retained in the reversible phase even if the sample has been cool down well below the irreversible limit.

In this work, the changes in the magnetic susceptibility and the evolution of the neutron powder diffraction patterns have been analyzed in order to study the phase changes occurring during cooling in two different Fe-Pd and Fe-Pd-Co alloys. The addition of Co reduces the temperature of the reversible and irreversible phase transformation but also reduces the amount of transformed irreversible martensite after cooling to 10 K.

15:50 Poster E36

Magnetic Shape Memory Alloys: Route to commercial applications

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The commercial use of magnetic shape memory alloys in actuating devices demands high mobility of the twin boundaries in combination with high operational temperatures. For applications in automotive engines, industry requires temperatures of at least 140°C. Thus this project aims to expand the operational temperature range whilst enhancing the crystal's lifetime based on focused alloy design and sample preparation as well as crystallographic and mechanical characterizations. To cope with the insufficiency of the Bridgman technique for single crystal growth, the SLARE method, recently developed at HMI by A. Mecklenburg, was used for reliable and repeatable preparation of homogeneous single crystalline samples of known composition and low porosity.

Neutron diffraction experiments at BENSCH (HMI, Berlin) of $\text{Ni}_{49.65}\text{Mn}_{29.28}\text{Ga}_{21.07}$ revealed a modulated martensitic structure stable up to 65°C (Curie-point at approx. 105°C). Based on the above mentioned composition substituting Ni by Co ($\text{Ni}_{46.65}\text{Co}_{6.14}\text{Mn}_{30.9}\text{Ga}_{19.05}$) yields a martensite-austenite transition temperature of above 120°C

(Curie-point at approx. 157°C). Here the crystallographic and mechanical characterization of both alloys will be presented and discussed.

15:50 Poster E37

Theory of multidomain states in magnetic shape memory alloys

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Usual real samples of ferromagnetic shape memory alloys contain complex systems of crystallographic and magnetic domains [1,2]. We apply a phenomenological theory [3] to describe the evolution of multidomain states driven by external magnetic field and external stresses in magnetic shape-memory materials. Magnetization processes under external stress and magnetic fields can be analysed based on phase diagrams from phase-theory approximation that is applicable for volume systems. Generalizations allow (i) to introduce 180-degree magnetic domain structures within the twin variants and (ii) rotation of magnetic moments within magnetic domains in case of relatively weak magnetic anisotropies. Hysteretic processes can be described by an effective relaxation equation, which accounts for a thermally activated twin boundary motion. Geometrical domain models with 90-degree magnetization stripe structure allow to derive characteristic lengths of thermodynamically stable coupled multidomain structures applicable to twinned platelets or thin films with two homogeneously magnetized variants. The application of the theory for (textured) polycrystalline materials through an effective (mean-field) coupling in an assembly of variously oriented single-crystals is demonstrated. [1] O. Söderberg et al. *Smart Mater. & Struct.* 14 (2005) S223. [2] V. A. Chernenko et al. *J. Phys.: Condens. Matter* 17 (2005) 5215, *Acta Mat.* 53 (2006) 5461. [3] A. N. Bogdanov, A. DeSimone, S. Müller, U. K. Röbber, *J. Magn. Magn. Mater.* 261 (2003) 204.

15:50 Poster E38

Receiving of Bimetallic Joints of TiNi-steel, TiNi-TiNi Using High-Speed Strain

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From a technological point of view Nitinol is very difficult to join with dissimilar materials, especially stainless steel. At welding of Nitinol brittle intermetallic compounds (for example Ti_2Ni_3) are formed which considerably reduce joint strength.

In respect to technology of materials explosive welding is promising method for joining TiNi when influence of high temperatures is not enough and intermetallic phase at the interface practically does not present.

The use of high-speed strain allowed TiNi-steel joint, TiNi-TiNi to receive providing high thermoelastic and reflexive characteristics of the load-bearing elements operating on shape memory effect. At the expense of heat treatment selection it was possible to recover ther-

moelastic properties of TiNi alloys subjected to high-speed strain by explosive welding.

The microstructure studies of welded joints show high quality of the received bimetal; the weld joint has a wave character that is optimum from a technological point of view, taking into consideration subsequent mechanical and other kinds of treatments of the received half-finished products.

15:50 Poster E39

A Free Energy model for Magneto-Mechanically Coupled NiMnGa Single Crystals

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The paper presents a 1-D rate-dependent magneto-mechanical model for Ni-Mn-Ga single crystals. Motivated by previous models developed by the authors for conventional shape memory behavior and ferroelectricity, a constitutive Helmholtz free-energy landscape with strain and magnetization as order parameters has been constructed for a representative meso-scale lattice element. This landscape includes three paraboloidal energy wells representing two easy-axis martensite variants and one hard-axis variant relevant for the one-dimensional case.

The kinetics of the switching/phase transformation processes under combined mechanical stress and magnetic field loading follow from a system of evolution laws for the magnetic and martensitic phase fractions. They are based on the theory of thermally activated processes with transition probabilities derived from the energy barriers of the stress and field dependent Gibbs free energy landscape.

The phase fractions subsequently determine the macroscopic strain and magnetization of a sample of Ni-Mn-Ga by a standard averaging procedure. While the current version neglects local inhomogeneities and assumes “perfect” single crystal behavior, it reproduces several of the constitutive effects observed in single crystal Ni-Mn-Ga samples. Future work includes an extension to a full thermo-magneto-mechanical model accounting for the occurrence of austenite and inhomogeneity effects.

15:50 Poster E40

Diffusion Mechanisms near Tilt Grain Boundaries in Ni3Al Intermetallide

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Diffusion mechanisms near tilt grain boundaries of orientations [100] and [111] were studied by the method of molecular dynamics in the dependence on a disorientation angle. The interactions between different pairs of atoms in the alloy were presented by the model potentials constructed on the basis of tight-binding potentials. The calculated crystal block limited by rigid and periodical boundary conditions amounted to atoms. In the experiments, the crystal was impulsively heated during 100-300 ps at different temperatures. As the result, three basic mechanisms of grain boundary diffusion were distinguished: atomic migration along the nucleus of grain boundary dislocation,

cyclic mechanism of atomic migration near the nuclei, the formation of the chains of atomic moving from one dislocation nucleus to the other nucleus. The mechanisms began to act successively with the growth of temperature. It depended on disorientation angles of the boundaries. Then, a grain boundary slipping and intergrain slipping with dislocations emission, the formation of fault defects were added to the above mentioned mechanisms at the presence of uniaxial deformation. At relatively low temperatures, diffusion took place only over sublattices of Ni atoms without disturbing the superstructure order. The disturbance of the superstructure order took place at more elevated temperatures.

15:50 Poster E41

Determination of the recovery stress under constraint in Fe29Mn7Si5Cr SMA.

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Ferrous shape memory alloys do not have complete shape recovery. Several attempts to improve the shape recovery still fail to achieve 100% recovery. For some applications, however the recovery stress under constraint is more important. A procedure was designed to measure the stresses under constraint in a Fe30MnSiCr SMA.

The net recovery stress can be defined as the maximum in the difference between the stresses of a deformed and of an undeformed sample. The calculation of $\sigma = E \cdot \alpha \cdot \Delta T$ can replace the stresses obtained of the undeformed sample.

The effect of partial free recovery before recovery under constraint is also investigated. It was remarkable that after the annealing at 200°C, the net stress did not decrease although ~50% of the shape recovery has happened. This gives a good opportunity for the use as pipe joint. It means that the diameter of the joint can be larger than the pipes and that the free recovery will not result in a lowering of the recovery stresses under constraint.

15:50 Poster E42

Structure of Ni₄₉Mn₂₉Ga₂₁ shape memory alloy processed by Bridgman method

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Ferromagnetic shape memory alloys (FSMA) are capable of producing, magnetically induced strain of several percent at frequencies of the order of 1 kHz. The best performance exhibit alloys based on the Ni-Mn-Ga system. Off-stoichiometric single crystals of Ni₂MnGa phase can produce up to 10% strain in the field below 1T.

In this study the Ni₄₉Mn₂₉Ga₂₁ alloy was prepared by arc melting of the pure elements followed by the modified Bridgman growth method. Structure and phase transition temperatures were studied. The X-ray

diffraction patterns as well as magnetic measurements and SEM observations revealed a presence of martensite at room temperature. The magnetization vs. temperature curve showed the martensite-to-austenite transformation starting at 290K and the Curie temperature at 390K. The effect of thermal treatment on the structure was investigated.

Symposium F

Welcome

The binary ion exchange technique permits the doping of several materials in a controlled, out-of-equilibrium way, and finds its interest in different fields like materials science, photonics, sensoristics, chemistry, nanostructured materials. In particular it has a wide application in the synthesis of a variety of systems, for surface treatments of glasses, fabrication of optical dielectric and glass waveguides (planar, channel, buried), production of catalytic systems (metal-doped zeolites), sorption of heavy metals or radionuclides from waste waters, doping materials with ion metals or rare-earths for optoelectronic applications. The method is also used to understand fundamental physics problems (like the mixed-alkali effect) and for industrial applications (e.g., strengthening of glasses).

In order to achieve a full control of the process, the dynamics of the diffusion of the exchanging ions need also to be modeled in details. This requires the accurate assessment of the dependence of the diffusion parameters on macroscopical external parameters and/or microscopical parameters (matrix structure, local structure around diffusing species, ...). Furthermore, studies based on advanced investigation techniques (synchrotron radiation, neutrons) have recently shown a great potential in addressing some fundamental problems in the field.

With this symposium, we aim to put together communities of experimentalists (mainly materials scientists and chemists, but also physicists and engineers) and theoreticians of the ion exchange process in order to share competences and come to a better description and predictability of the process in the various application areas. Recent advances and new issues in the field (waste water treatment, production of nonlinear optical devices, results from advanced investigation techniques) will also be presented to the community. It should be underlined that since the ion exchange process concerns with several different fields, as described above, very rarely people from these different research areas have the opportunity to share their knowledge in all its aspects.

List of topics

- Ion exchange in glass
- Ion exchange in crystals
- Ion exchanged and nanostructured systems
- Ion diffusion modelling in ion exchange
- Field-assisted ion exchange
- Advanced characterization techniques
- Ion exchange and photonic materials and devices
- Ion exchange as surface treatment for industrial application
- Ion exchange in waste water treatments
- Ion exchange and metal doped zeolites
- Heavy metal and radionuclide immobilization

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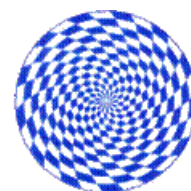
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Proceedings

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Programme

Monday, 17 September

Opening & Plenary Session

Czochralski Award Ceremony & Acta Gold Medal Ceremony
Monday morning, 17 September, 10:00

Lunch break

Monday afternoon, 17 September, 12:30

Session I

Monday afternoon, 17 September, 14:00

14:00	Invited
A trip in the history and evolution of ion-exchange process	
<u>Paolo Mazzoldi</u>	
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The first application of ion-exchange in the field of the cultural heritage may be attributed to Persians in X Century and, through the Islamic expansion in Europe, arrived to Italy in the XVI Century. A paper of "Mastro Giorgio" indicated the ion-exchange procedure.	
In this talk, the use of the ion-exchange process, followed by thermal treatments or irradiation, in different matrices, as polymers, ferroelectrics and dielectrics, will be discussed, in correlation with the application in the field of nanotechnologies, nanophotonics, optical and sensor devices.	

14:30

Oral

Silver and copper ions and nanoparticles in the glaze of Renaissance majolica

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Lustre pottery decorations of Renaissance majolica are mainly characterised by Cu and Ag nanoparticles dispersed in the glassy network of the glaze. Following this finding, a study has been carried out on several gold and red original lustre samples, with the objective to better understand the lustre formation mechanism and manufacturing. Preliminary measurements were carried out by XRF, SEM, TEM, Vis-NIR; then EXAFS measurements were carried out at the BM8 beamline GILDA of ESRF. While XRF and SEM-EDS measurements indicated that gold and red lustre decorations are characterised by the presence of Ag and Cu, surface plasmon resonances in Vis absorption spectra clearly indicated that mainly responsible of the colour for gold and red lustre are Ag and Cu metal nanoparticles respectively. These results have been confirmed by EXAFS measurements. However, in most cases, Cu and Ag have been also found in oxidised forms (Cu^+ and Cu^{2+} , with a large prevalence of Cu^+). The simultaneous presence of metallic and ionised Cu and Ag is coherent with a lustre formation mechanism where the first step consists of an ion-exchange between Cu and Ag ions from the oxides and salts used according to the original lustre recipe, and the alkali ions in the glaze (on average, total Na^+ and K^+ : 5-15% wt). The fractions of reduced Ag and Cu are mainly responsible of the colour. These fractions can be low and variable case by case, depending on used recipes, reductive conditions and temperature of the kiln. It has been found that even the metallic Ag/Cu ratio cannot be simply and univocally correlated with the colour. Further information has been recently obtained through the transnational access services offered by the Eu-ARTECH project within the 6th F.P. of the EU (<http://www.eu-artech.org>). In particular, through the MOLAB facility, XRF, micro-Raman, AFM, and UV-Vis measurements have been carried out on original plates of the Renaissance at the V&A Museum.

14:45

Invited

Ion Exchange for Glass Strengthening

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This paper presents an overview of glass strengthening by exchange of alkali ions in a molten salt, below the glass transition temperature (chemical tempering). The main applications are in the manufacturing of high strength glazing for high speed trains and aircraft cockpits, from where examples are given. The main parameters of the process which control the glass reinforcement are reviewed. The obtained residual stress state is characterized, thanks to differential refractometry, which is briefly described, along with different mechanical tests for the strengthening measurement. Beside reinforcement, other character-

istics of chemically tempered glass are presented. Finally, the concept of 'engineered stress profile' glass is presented and its interest for the applications is discussed.

15:15

Oral

The effect of Na-K ion exchange on the formation of Co nanoparticles in sodium silicate glasses doped with CoO

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The aim of the present report is the study of states of Co in Co-containing sodium-silicate glasses after $\text{Na}^+ - \text{K}^+$ ion exchange. The base binary $20\text{Na}_2\text{O} - 80\text{SiO}_2$ (in mol.%) glasses containing 0.5 wt.% CoO were studied. Polished plates approximately 0.4 mm thickness were prepared. The plates were immersed in a molten salt KNO_3 for periods of time (T_p) varying from 5 min to 7 h at 450 K. Some samples were annealed after ion exchange at 600 °C for 24 h in the H_2 atmosphere. The asymmetric EPR line with crossing-base $g = 4.4 \pm 0.1$ and $\text{DH}_{pp} \sim 100$ mT was observed at 4.2 K for the samples before an ion exchange and was attributed to Co^{2+} ions in distorted octahedral environment. The intensity of EPR line decreases with increasing T_p . New EPR line with $g = 2.31 \pm 0.02$ is observed along with the $g = 4.4$ line at 4.2 K. Its width ($\text{DH}_{pp} \sim 10 - 25$ mT) depends on T_p . The $g = 2.31$ line is not observed above 77 K. According to literature data we attribute the $g = 2.31$ signal to Co^{1+} ions. The small width of this line is associated with exchange interactions between ions Co^{1+} which form clusters. We assume the existence of $\text{Co}^{2+} @ \text{Co}^{1+}$ transition during the $\text{Na}^+ @ \text{K}^+$ ion exchange process. Starting from $T_p = 1$ h and $T_{ht} > 5$ h the single line with $g \sim 2.22$ and $\text{DH}_{pp} \sim 30 - 40$ mT was observed at room temperature. The dependence of the width and intensity of this line on measurement temperature indicates its superparamagnetic origin. An additional shoulder at 265 mT which appears at 4.2 K for the samples with $T_p > 1$ h and $T_{ht} \sim 15$ h indicates the formation of ferromagnetic particles of metallic Co. Optical absorption spectra and TEM of the samples confirm the formation of nano-particles of metallic Co in ion-exchange layers of heat-treated sodium-silicate glasses doped with CoO.

Coffee break

Monday afternoon, 17 September, 15:30

Joint Poster Session (Part I)

Monday & Wednesday

Monday afternoon, 17 September, 15:50

Tuesday, 18 September

Session II

Tuesday morning, 18 September, 9:00

9:00 Invited

Site of transition-metal ions in ion-exchanged metal-doped glasses

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Ion exchange is largely used to dope surface layer of glass with metal ions so inducing a modification of the optical properties of the doped layer, useful to fabricate low-loss optical waveguides. Post-exchange treatments, such as annealing in selected atmospheres and irradiation with light ions can induce the dopant aggregation in metallic clusters; laser irradiation can be further used to reduce the cluster size. These facts spread the possible application of ion-exchanged glasses as non-linear optical materials for optoelectronics.

I will present recent and new experimental results on glasses doped with metal atoms (Cu, Ag, Tl) by ion exchange from molten salt baths. X-ray absorption spectroscopy (XAS) allowed the investigation of the site of the metal ions dispersed into the matrix and of the metal aggregates induced by the post-exchange treatments, such as thermal annealing, laser or ion irradiation. Moreover, in presence of metal ions of different oxidation states diffusing into the matrix, XAS in grazing incidence geometry allowed to measure the depth profile of each species, giving the basic information to model the ion diffusion process. The experimental results indicate that the site of the dopant ion is different from that of that one of the replaced alkali ion (Na or K) and strongly depends on the glass used; moreover, the dopant-matrix correlation (i.e. the metal-oxygen distance) depends critically on the dopant concentration, suggesting that structural changes occur into the glass, caused by the ion-exchange process; about the aggregation of dispersed ions into metallic clusters, suitable post-exchange treatments can be exploited to tailor the cluster size, even towards the formation of small (~1 nm) aggregates.

9:30 Invited

Structure and properties of Li^+/H^+ or Li^+/D^+ ion-exchanged aluminosilicate glassesTetsuji Yano¹, Taketoshi Kagose¹, Nahoko Uno¹, Shuichi Shibata¹, Hideki Maekawa²

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Ion exchange of glass has been utilized to give various functionalities to the melt-quenched glass, where the size effects between the exchanged ions often play important roles like chemical strengthening of glass. However, the smallest monovalent cations of H^+ and D^+ have not been well investigated as a counter cation of the ion-exchange pair. They are bare of the electron shell (empty 1s orbital) and would show different interaction phenomena with anions like oxygens in the glass network from alkali ions. In this study, the ion-exchange treatments of H^+ or D^+ with the smallest alkali ion of Li^+ were conducted on lithium aluminosilicate glasses. Dipping and field-assisted methods were used as the ion-exchange technique. Non-bridging oxygen-free (NBO-free) glasses of $\text{Li}_2\text{O}-\text{Al}_2\text{O}_3-\text{SiO}_2$ system were prepared using melt-quenching method. The platelet glass samples were subjected to the ion exchange at 290-300 °C using conc- H_2SO_4 or D_2SO_4 as the sources of H^+ or D^+ , respectively. Dipping and field-assisted methods

gave the different ion-exchange ratios, and the latter enabled the fully ion exchange of glass samples with H^+ or D^+ without crack. The fully ion-exchanged samples showed low thermal expansivity below 400 °C and shrinkage at higher temperature due to the release of water from the glass network. The structural analysis using Infrared and Raman spectroscopies and nuclear magnetic resonance (NMR) spectroscopy were carried out on the samples. Some of the incorporated H or D were found to form H_2O or D_2O molecules in the glasses accompanying with the structural evolutions; the rearrangement of the network of TO_4 tetrahedra and the increase of the oxygen coordination number of Al^{3+} ions. In the presentation, the analysis results of $^1\text{H}/^{29}\text{Si}$ and $^1\text{H}/^{27}\text{Al}$ CP-MAS-NMR results and the measured properties of the ion-exchanged glass samples are shown to consider the ion-exchange phenomena in glass.

10:00 Oral

Characterization of silicate glasses doped with transition metals by field-assisted ion exchangeEli Cattaruzza¹, Giancarlo Battaglin¹, Francesco Gonella¹, Francesco Dacapo², Chiara Maurizio², Cinzia Sada³, Ali Shahid³, Giovanni Mattei³, Alberto Quaranta⁴

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In the presented experiment, gold and cobalt containing glasses were prepared by field-assisted ion exchange. A film of the metal was first deposited onto soda-lime glass substrates by the rf-sputtering technique, and then it was heat-treated under intense electric field. Structural and compositional characterization was performed by several techniques, namely, secondary ion mass and Rutherford backscattering spectrometries, optical absorption and extended X-ray absorption fine structure spectroscopies, and transmission electron microscopy.

10:15 Oral

Ion-Beam Induced Nano-Sized Ag-Metal Clusters in GlassHeinz-Eberhard Mahnke^{1,2}, Ivo Zizak^{1,3}, Vasil Koteski^{1,4}

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We have studied the formation of Ag-metal clusters in soda lime glass induced by heavy ion irradiation with x-ray absorption spectroscopy (XAS). The shape of the formed clusters was studied both with transmission electron microscopy (TEM) and with small angle x-ray scattering (SAXS). Glass platelets, 0.1 µm thick, were ion exchanged with silver according to standard procedures [1] and were irradiated at low temperature with 600-MeV Au ions from the ion beam laboratory ISL with fluences around 10^{12} ions/cm² resulting in not yet overlapping, more than 50-µm long ion tracks. XAS experiments on the Ag K-edge, performed at HASYLAB, revealed the transformation of Ag from the oxidic type local structure after the ion-exchange process into the metallic form following the swift heavy ion irradiation and annealing

[2]. Information on the shape of the formed clusters was obtained by transmission electron microscopy (TEM) on thin slices cut out of the samples parallel to the ion impact. While annealing under a reducing atmosphere of argon with a few % H₂ alone already leads to the formation of metal clusters (typical annealing conditions 30 minutes at 340°C), not very uniform in size and randomly distributed over the Ag-containing glass volume, ion beam irradiation yielded spherical clusters, larger in size and more homogeneous in their size distribution. The most remarkable feature is that these clusters are arranged in chains parallel to the direction of the ion beam. For a more detailed and more quantitative study of the arrangement, the shape, and the formation process SAXS experiments have been started at BESSY. First SAXS images confirm the TEM pictures in revealing column-like structures in q-space as disc-like images with dimensions translated into a diameter of the Ag-metal droplets of about 7 nm.

[1] G. Bataglin et al., Nucl. Instr. and Meth. B 200 (2003) 185

[2] H.-E. Mahnke et al., Nucl. Instr. and Meth. B 245 (2006) 222

Coffee break

Tuesday morning, 18 September, 10:30

Session II

Tuesday morning, 18 September, 11:00

11:00

Invited

Ag/Na ion exchange in soda-lime glasses and the formation of small Ag nanoparticles

Manfred Dubiel¹, Jörg Haug¹, Holger Kruth¹, H. Hofmeister², Klaus-Dieter Schicke²

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Nanosized metal particles embedded in glass have attracted much interest as material with potential applications because of their specific linear and non-linear optical properties. Especially, strong variations of the properties are expected for particle sizes much less than 10 nm. Therefore, we investigated the formation of very small Ag nanoparticles of 1 to 2 nm size in commercial soda-lime glass, silver-doped by Ag/Na ion exchange in a mixed nitrate melt such that Ag⁺ were incorporated into the glass replacing Na⁺ of the matrix. It could be shown by electron paramagnetic resonance and X-ray absorption spectroscopy that at the Fe K-edge that polyvalent Fe ions served as thermosensitive reductive during the ion exchange below the glass transformation temperature. This process could be described by a charge transfer between the iron ions and nuclei of Ag particles. The transmission electron microscopy, the optical spectroscopy as well as the X-ray absorption spectroscopy at the Ag K-edge were used to identify the formation of metal particles and to analyse their structure. For sizes from 0.5 to 2 nm the experimental data indicate the presence of Ag containing structures (like oxidic compounds) that obviously serve as predecessor for the formation of silver nanoparticles. The extensive analysis of the kinetics reveal that all previous models fail, in particular for nucleation and particle growth. It is assumed, that a new model must be established considering metastable intermediate phases in order to explain all experimental results.

11:30

Invited

Studies on Passive and Active Silver-Sodium Ion-Exchanged Glass Waveguides and Devices

Seppo Honkanen¹, Sanna Yliniemi¹, Jacques Albert²

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In this presentation, we review our recent work on exploring and developing new techniques for planar lightwave circuits utilizing silver-sodium ion exchange. In the experiments, two special kinds of glass substrates have been used: aluminoborosilicate glass (also called BGG31) specially developed for passive ion-exchanged waveguides and phosphate glass (commercial name IOG-1) designed for waveguide laser applications. Phosphate glass can be doped with large amounts of rare earth ions without significant lifetime reduction. This enables fabrication of short-cavity waveguide lasers, a desirable feature for example in high-repetition rate modelocked lasers operating at 1550 nm wavelength region.

First, birefringence properties of buried molten salt ion-exchanged waveguides in BGG31 glass are presented in detail. It will be shown that waveguides with zero birefringence can be obtained by properly adjusting the thermal annealing time. The effect of waveguide mask opening width on waveguide birefringence is described. Low birefringence, 10⁻⁵ or below, is demonstrated for both the even and odd mode with wide range of mask opening widths. Results of birefringence properties of an optical add-drop multiplexers/demultiplexer (OADM) fabricated into BGG31 glass are presented.

Photosensitivity properties of both undoped and Er-Yb-codoped IOG-1 glass are discussed. A new approach to fabricate waveguide Bragg gratings through UV exposure in IOG-1 glass will be presented. High quality waveguide Bragg gratings with reflectivity of the order of 80% are demonstrated in undoped IOG-1 glass. A single mode waveguide laser utilizing this kind of a narrowband UV-written waveguide Bragg grating as one of the cavity mirrors is demonstrated in hybrid IOG-1 glass. Waveguide Bragg gratings with lower reflectivity (R~15%) in Er-Yb-codoped IOG-1 glass are also described. Results of the material study into the effect of UV light exposure on phosphate glass will also be presented.

12:00

Oral

Study of deeply buried waveguides: a way towards 3D integration

Jérôme Grelin, Elise Ghibaudo, Jean-Emmanuel Broquin

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Ion-exchange on glass is now a mature integrated optics technology, indeed, many devices such as wavelength multiplexers, splitters, optical amplifiers, lasers or sensors have been already realized. The challenge is now to integrate all these functions on a single chip. Two different paths can be used to achieve this goal: the first one consists in a reduction of the waveguides' dimensions by an increase of the refractive index change, whereas the second one, which is addressed in this paper, is based on the realization of multilayered devices. Because ion-exchange on glass allows manufacturing either surface or buried waveguides, this technology is well adapted for 3D integration. However,

it is mandatory to prevent any parasitic light transfer between top and bottom waveguides. This condition can be fulfilled if they are sufficiently separated. In this article, we present the development of ion-exchanged waveguides deeply buried into a glass substrate and their application to 3D integrated devices. They have been realized by means of a two steps Ag^+/Na^+ ion-exchange on a dedicated silicate glass. First, a thermal ion-exchange has been carried out at 330°C during 2 min in a $0.8\text{NaNO}_3\text{-}0.2\text{AgNO}_3$ molten salt in order to create the core of the waveguide; then this core has been buried into the glass substrate by applying an electric field of 450 V/m during 1h30min in a NaNO_3 solution at 260°C . The obtained waveguide has been measured to be $22\text{ }\mu\text{m}$ under the glass surface. It is singlemode at $\lambda=1.55\text{ }\mu\text{m}$. In order to prove the good isolation between this waveguide and the surface, a top layer has been added to the device by the realization of surface channel waveguides through a thermal ion-exchange performed in a $0.8\text{NaNO}_3\text{-}0.2\text{AgNO}_3$ molten salt at 330°C during 2 min. The near-field observation of the device output has shown no coupling between the top and bottom layers demonstrating therefore the feasibility of 3D integrated optical devices by means of ion-exchange on glass.

12:15 Oral

Ion-exchanged optical waveguides fabricated in novel Er^{3+} and $\text{Er}^{3+}/\text{Yb}^{3+}$ -doped silicate glasses: The relationships between glass composition, basicity and waveguide properties

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Planar optical waveguide fabricated in erbium doped glasses became attractive due to their light emission at $1.5\text{ }\mu\text{m}$, thereby shown like candidates for optical amplifiers or waveguide lasers. In this study we focused on the influence of chemical compositions of specially formulated Er^{3+} and $\text{Er}^{3+}/\text{Yb}^{3+}$ -doped silicate glasses on the ion-exchange process and optical properties of the mm fabricated waveguides.

Optical waveguides were fabricated by a routine $\text{K}^+ \leftarrow \text{Na}^+$ ion exchange in a set of the glasses that had various contents of the RE ions and different optical basicity. Properties of the fabricated planar waveguides were studied by means of Dark Mode Spectroscopy at 637 nm while the chemical composition of the waveguides was determined using Scanning Electron Microscopy (SEM-EDAX). The intensities of the $1.5\text{ }\mu\text{m}$ emissions before and after the ion exchange were compared as well as the RE content after the ion exchange that was measured by the ion beam methods. The results are discussed from a point of view of the content of the active ions, glass permeability (i.e. glass covalence and glass basicity) for the exchanged ions and optical properties of fabricated waveguides. Diffusion that proceeds during the ion exchange will be characterized by diffusion coefficients and concentration depth profiles of the involved ions important for the resultant properties. It was found that presence of the RE could substantially lower the permeability of the glass matrix for the migrating ions.

Acknowledgement

The project has been supported by the GACR grant 106/05/0706 and National Research Centrum LC 06041.

Lunch break

Tuesday afternoon, 18 September, 12:30

Session III

Tuesday afternoon, 18 September, 14:00

14:00 Invited

Integrated optics devices based on glass ion-exchanged technology

Florent Gardillou, Cédric Cassagnettes, Jean-Christophe Maisonne, Denis Barbier

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During the last two decades, ion-exchange techniques on glass substrates have largely demonstrated their capacity to form channel waveguides with very low propagation losses, nearly perfect coupling efficiency with single mode optical fibers and small bending radii for compact designs. Based on that technique and on the development of custom made silicate and phosphate glasses dedicated to the Ag^+/Na^+ ion-exchange, Teem Photonics commercializes for 10 years passive and active integrated optics components mainly for telecom and sensors markets.

The achieved performances for some commercialized components like $1\times N$ and $2\times N$ splitters, fiber spacing concentrator (FSC) interface components, pump multiplexers, erbium-doped waveguide amplifiers (EDWA), distributed feed back (DFB) lasers and amplified spontaneous emission (ASE) sources will be presented. Perspectives of Teem's technology platform will also be developed.

14:30 Oral

Hybrid Neodymium-doped passively Q-switched waveguide laser

Rafael Salas Montiel, Lionel Bastard, Jean-Emmanuel Broquin

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In the mid 80's, the doping of optical fibers' core with rare earth atoms has been a major breakthrough in the field of optical telecommunications since it allowed the realization of in line optical amplifiers. However, erbium doped fiber amplifiers are a few meters long and a huge effort has been made in order to realize compact and efficient active devices based on rare-earth-doped waveguides. For this purpose the use of phosphate glasses instead of silicate ones has been investigated because they allow a better solubility of the inserted rare earths. In this paper we present the realization of a hybrid Neodymium-doped passively Q-switched waveguide laser made by an ion-exchange on a Schott IOG-1 Phosphate laser glass combined with the deposition of a BDN saturable absorber diluted in a cellulose acetate polymer cladding. In a first step, we present the CW operation of the laser with an undoped cladding. We show that for a $3.5\text{ }\mu\text{m}$ wide, 1.5 cm long waveguide realized by a silver-sodium ion-exchange, a 5 mW output has been achieved by creating a Fabry-Perot cavity with dielectric multilayers mirrors sticked to the chip facets. Then, the characterizations performed on the BDN doped layers are presented. It is shown that a proper selection of the hybrid guiding concentration and saturable absorber concentration entail an excess absorption ranging from 1 to 10 dB/cm at zero flux. Finally, we will present the last results we will obtain on the Q-switched behavior of the laser.

14:45

Invited

The potential of ion-exchange in sol-gel derived photonic materials and structures

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Sol-gel is a very convenient technique for the fabrication of high quality thin glass films, optical planar waveguides and photonic bandgap structures [1-3]. Ion exchange, on the other hand, is a well established technique to fabricate high quality waveguides in glass and, in fact, optical amplifiers made by this technique are commercially available. The possible integration of the sol-gel and ion exchange techniques for the fabrication of passive and active (rare-earth doped) channel waveguides in integrated optic devices has recently gained interest.

This presentation will start with a general overview of the fabrication of passive and active films and planar waveguides by sol gel, followed by a brief description of waveguide fabrication by ion exchange (in particular sodium-silver and sodium-potassium ion exchange) and the possible integration between the two processing technologies.

In the particular case of sol-gel waveguides doped with Er^{3+} ions, the possible use of sodium-silver exchange to define channel waveguides and simultaneously enhance the Er^{3+} fluorescence emission in the presence of Ag nanoparticles will also be considered, together with the possible use of ion exchange in the pore-doping of partially densified photonic sol-gel structures with rare-earth and silver ions in solution. Some considerations will also be made concerning the use of advanced characterization techniques like ellipsometry and m-line or RBS spectroscopies in the analysis of the ion-exchange concentration profiles.

[1] R. M. Almeida, J. Non-Crystalline Solids 259 (1999) 176.

[2] Rui M. Almeida, Paulo J. Morais and Ana C. Marques, Phil. Mag. B82 (2002)

707.

[3] Rui M. Almeida, Ana C. Marques and Sabine Portal, Optical Materials 27 (2005)

1718.

15:15

Oral

Silver Sensitized Er-doped Ion Exchanged Sol-gel Waveguides

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Rare earth ions such as erbium or neodymium have small absorption cross section, which limit their excitation efficiency. Sensitizers like silicon nanocluster can be used to increase the luminescence efficiency of rare earths ions and hence reduce the pump power needed to achieve inversion of population in a waveguide amplifiers. In fact it was demonstrated that optically generated electron-hole pairs in Si nano-

crystals can recombine and transfer energy to Er. Recently also silver has been proposed as erbium sensitizer as many absorption bands in the visible and near ultraviolet have been observed in glasses doped with Silver.

In this paper Ag and Er doped glass films have been synthesized with a combined sol-gel and ion-exchange route. The introduction of silver as erbium sensitizer in the film was obtained by ion-exchanging Er doped $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-Na}_2\text{O}$ sol-gel films. The films were subsequently annealed under controlled atmosphere to induce the migration and aggregation of the metal ions. Films showed different Er^{3+} photoluminescence behaviors depending on silver concentration and aggregation state. The interaction between erbium ions and Ag centers have been investigated and enhancement of the excitation cross section due to silver sensitizing effect have been demonstrated. The developed synthesis allowed also the realization of erbium doped channel waveguides by selective Na-Ag ion exchange process.

Coffee break

Tuesday afternoon, 18 September, 15:30

Session III

Tuesday afternoon, 18 September, 15:50

15:50

Invited

Chemical Composition Effects On An Ion Exchange Process In Glass

Martin Mika¹, Jarmila Spirkova¹, Pavlina Tresnakova¹, Linda Salavcova¹, Frantisek Ondracek², Frantisek Lahodny¹, Stanislava Janakova¹, Blanka Svecova¹, Hana Malichova¹

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An ion exchange technology represents the fast and effective process of the fabrication of low-loss optical waveguides in glass substrates at reasonable costs. Employing this technique, we successfully prepared Ag^+ , K^+ , Cu^+ , Cu^{2+} , and Li^+ planar and channel optical waveguides. Required ions diffused into substrates from a bath of molten nitrates, chlorides, and iodides replacing Na^+ ions present in glass substrates. For each waveguide type we developed a chemically durable sodium-aluminosilicate glass of optimized chemical composition that stabilized the cations and enabled efficient and well controlled ion exchange resulting in low-loss waveguides. For waveguide amplifiers we also formulated a series of sodium-zincaluminosilicate glasses doped mainly with Er^{3+} and Yb^{3+} ions. The optical properties of the prepared waveguides were characterized using the coupling prism or the dark mode spectroscopy in combination with the inverse WKB method. We determined the number of guided modes, depth of a waveguiding region, refractive index depth profiles, and refractive index increment. Employing the scanning electron microscopy (SEM-EDAX), ion beam methods (PIXE and RBS), and neutron depth profiling (NDP), we analyzed waveguide's concentration profiles. To optimize glass compositions and the ion exchange process, we developed composition-property models based on the first order expansion $y = \sum a_i x_i$ where y represents the measured glass property, x_i represents the molar fraction of the i -th glass component, and a_i is the i -th glass component effect representing a partial molar quantity. Using these models we quantified effects of glass components. We found that Zn^{2+} and Mg^{2+} facilitated the ion

exchange. On the other hand Ca^{2+} , Er^{3+} , and Yb^{3+} significantly decreased the ion exchange process rate. We interpreted the calculated components effects employing the optical basicity and ligand field theories.

Our project 106/05/0706 was funded by the Grant Agency of the Czech Republic.

16:20 Invited

Structure refinement of neutron and X-ray diffraction data on glassy systems for the generation of comprehensive three dimensional models

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Maximising the structural information that can be extracted from a diffraction experiment on a disordered system such as a glass is a long standing challenge. In this seminar I will demonstrate techniques that use a combination of a classical simulation method and experimental data to allow us to move beyond the traditional and limited information of the spherically averaged total pair distribution function. These new refinement methods allow us to build 3-dimensional models of the structure of complex glasses that are consistent with the available experimental constraints. Such models can then be interpreted to the level of partial pair distribution functions, spatial density functions and bond angle distribution functions. The results highlight the importance of extending our understanding of glass structure beyond the traditionally quoted statistical mean values. Examples of silicate glasses, SiO_2 and $\text{NaFeSi}_2\text{O}_6$ [1] will be used to highlight the possibilities for neutron diffraction, X-ray diffraction and the combination of the two.

[1] "Determination of Fe^{3+} sites in a $\text{NaFeSi}_2\text{O}_6$ glass by neutron diffraction with isotopic substitution coupled with numerical simulation", C.Weigel, L.Cormier, L.Galoisy, G.Calas, D.T.Bowron and B.Beuneu, *Appl. Phys. Lett.* **89**, 141911 (2006)

16:50 Oral

Effect of silicate glass composition on properties of ion-exchanged optical waveguides

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Planar ion-exchanged (IE) optical waveguides are important parts of photonics devices utilized for harnessing and distribution of optical radiation. Silicate glasses are very suitable material for the fabrication of the sought waveguides due to their good compatibility with commonly used optical fibres as well as due to flexibility of their composition. Knowledge of the relations between composition of the glass matrix and properties of the resulting IE waveguides would be a great help in tailoring the waveguides properties according to the desired applications.

Our research is focused on study and description of the relations between content of bivalent glass modifiers (as Ca^{2+} , Mg^{2+} and Zn^{2+} and their mixtures) on refractive index (n) depth profiles shapes, refractive index increments, depths of the in-diffused optical layers of the waveguides fabricated by the of silver -sodium or potassium-sodium ion exchanges. The relations will be illustrated on specially designed

dysprosium containing glasses that will find their potential usability in 1300 nm operating photonics devices. The results revealed very clearly expressed correlations between the content of the highly ionic Ca^{2+} that made the depths as well as n increments smaller, while the more covalent Mg^{2+} and Zn^{2+} increased both the depths of the optical layers and the refractive index increments.

Models of the processes involved will be also presented.

Acknowledgement

This research has been supported by the GACR grant 106/05/0706

Wednesday, 19 September

Plenary Session

Wednesday morning, 19 September, 9:30

Coffee break

Wednesday morning, 19 September, 10:30

Session IV

Wednesday morning, 19 September, 11:00

11:00 Invited

Modeling the structure and dynamics of glasses using *ab initio* and classical computer simulations

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Computer simulations are nowadays an ubiquitous tool in all branches of science. Their utility is particularly evident when investigating the behavior of disordered systems, as the determination of the structural and dynamical properties of non-crystalline materials is a extremely difficult task because any scientific insight must be achieved without the mathematical amenities useful in the crystalline solid state. Therefore a true understanding of the microscopic origin of the properties of glasses requires the combined use of multiple experimental techniques together with modern modeling capabilities. Here I will review the use of *ab initio* and classical Molecular Dynamics simulations as a tool to enhance the output and our understanding of experimental data. In particular the case of neutron scattering will be considered, as the characteristics of this technique and the typical time and length scales covered by modern neutron instruments match the properties that can be easily computed by means of standard MD simulations. Examples of recent research on the structure and dynamics of ionic and molecular glasses will serve to show the potential of modeling techniques, as well as the present limits and future possibilities.

11:30 Invited

Modelling the ion exchange process in glass: phenomenological approaches and perspectives

Alberto Quaranta¹, Elti Cattaruzza², Francesco Gonella²

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A great deal of work has been devoted in the last decades to the study of the modeling of ion exchange in glass, in order to tailor the physical properties of the modified surface, e.g. refractive index or stress profiles. The first models were based on simple ionic diffusion equations, while in the last years it became clear that the glass local structure changes during the ion exchange even at temperatures lower than the glass transition one. In fact, structural analyses underlined the local rearrangement of the ion neighbors as a function of the dopant concentration. This makes the analysis of the ion exchange process quite complex, with a corresponding difficulty in predicting and describing the physical properties of the treated surfaces. The aim of this work is to briefly review the main approaches simulating the ion exchange process in silicate glasses, and to suggest a further phenomenological approach, already used for the study of mixed-alkali glasses, which relates the transport parameters to the micro-structural properties of the glass network, in a simple but effective way. The ion exchange mechanism for different ionic species will be analyzed, taking also into account the different charge states of the migrating ions. The phenomenological coefficients will be connected to the microstructural information obtained for similar systems by different analysis techniques. Moreover, correlation effects between the ionic fluxes will be evidenced when different ionic species participate in the ion exchange process.

12:00 Oral

The influence of Cr-dopant on the properties of α -FeOOH particles precipitated in highly alkaline media

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The effects of the Cr-dopant on the phenomenology of the precipitation of acicular α -FeOOH particles, the formation of solid solutions, particle size and shape were investigated using X-ray powder diffraction (XRD), Mössbauer and Fourier transform infrared (FT-IR) spectroscopies and field emission scanning electron microscopy (FE-SEM). Acicular and monodisperse α -FeOOH particles were precipitated at a very high pH by heating the suspension obtained by adding a tetramethylammonium hydroxide solution to the aqueous solution of FeCl_3 . The influence of the Cr-dopant was investigated by adding varying amounts of Cr^{3+} ions to the initial FeCl_3 solution, where $r = 100 \cdot [\text{Cr}] / ([\text{Cr}] + [\text{Fe}])$ is the added amount of chromium. An XRD analysis of the obtained powders (with r values from 0 to 23.08) showed only the presence of the diffraction lines characteristic of α -FeOOH. Mössbauer spectroscopy showed a decrease in hyperfine magnetic field values of α -FeOOH sextets with an increase in Cr-addition, which indicates Cr incorporation into the α -FeOOH structure. OH bending bands in the FT-IR spectra showed only a slight change in position with an increased r , but a considerable increase in the lattice band wave number indicated a decreased thickness of α -FeOOH particles. This conclusion was confirmed by FE-SEM observations.

12:15 Oral

Precipitation of akaganéite and hematite particles in acidic pH media

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Iron oxides (a common name for iron oxyhydroxides and oxides) are important materials which have found application in traditional and

advanced technologies. These materials can be utilized as pigments, catalysts, sensors, carriers for waste metal cations, etc. Iron oxides also play an important role in natural aquatic systems, sediments and soils. Various methods of iron oxide synthesis using «wet» chemistry were reported, such as a slow or forced hydrolysis of Fe(III) -salt solutions, crystallization of ferrihydrite gel, sol-gel, microemulsion and crystallization induced by ultrasound, microwave or γ -irradiation. As a rule, these syntheses are conducted at low concentrations of Fe(III) -salts. In that case it is relatively easy to obtain monodisperse particles; these studies, however, are generally important from an academic standpoint. In the present work we have focused on precipitation from concentrated Fe^{3+} salt solutions partially neutralized with concentrated NaOH . The precipitation systems were autoclaved at 160 °C. The precipitation process was monitored by analysis of the precipitate by XRD, ^{57}Fe Mössbauer and FT-IR spectroscopies, and FE SEM. The kinetics of the reference system showed a formation of β -FeOOH (akaganéite) which was after a prolonged time transformed into β - Fe_2O_3 (hematite). The addition of Na_2HPO_4 (concentrations in the final volume 0.010, 0.025, or 0.050 M) at the beginning of the precipitation caused significant changes in the phase composition of the precipitates. An additional appearance of α -FeOOH (goethite) was observed as a direct result of adding Na_2HPO_4 to the precipitation systems. The specific adsorption of phosphates at acidic pH values, and the aggregation effect play an important role in the formation of particles in respect of their size and morphology. The formation of the end-product α - Fe_2O_3 was controlled by the dissolution/reprecipitation mechanism.

Lunch break

Wednesday afternoon, 19 September, 12:30

Session IV

Wednesday afternoon, 19 September, 14:00

14:00 Invited

Heavy metal uptake and ion exchange properties of functionalized mesoporous silica materials

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Silica-based mesoporous organic-inorganic hybrids become increasingly attractive because they display a regular structure at the nanoscale, large specific surface areas and pore sizes between 2 and 15 nm, and readily accessible organo-functional groups.¹ They are of particular interest for analytical and electroanalytical purposes, by exploiting their advanced properties in aqueous suspensions or at an electrode/solution interfaces.^{2,3}

The aim of this lecture is to highlight some recent achievements performed with using such new materials in analytical chemistry and electroanalysis, by focusing primarily on mesoporous hybrids containing organo-functional groups bearing a net positive or negative charge. I will first point out the definite advantage of mesostructured organically-modified silicas (ensuring good accessibility to the binding sites and inducing fast transport of the analyte because of unrestricted diffusion) over their non-ordered homologues,^{4,5} and their interest for electrochemical sensing.⁶ Then, discussion will be made on the effect of generating a net charge on the mesopore walls (e.g., protonation of amine groups) and the consequence it would induce on the reactivity of these nanoreactors (via the example of mercury(II) binding).^{7,8} Finally, the interest of surfactant templated sulfonic acid- and/or quatern-

ary ammonium-functionalized silica microspheres as new efficient ion exchangers and electrode modifiers will be presented.^{9,10}

1. Hoffmann *et al.*, *Angew. Chem. Int. Ed.***2006**, 45, 3216
2. Walcarius, *C.R. Chimie***2005**, 8, 693
3. Walcarius *et al.*, *J. Mater. Chem.***2005**, 15, 3663
4. Walcarius *et al.*, *Chem. Mater.***2003**, 15, 2161
5. Walcarius & Delacôte, *Chem. Mater.***2003**, 15, 4181
6. Walcarius *et al.*, *Electroanalysis***2003**, 15, 414
7. Walcarius *et al.*, *Anal. Chim. Acta***2004**, 508, 87
8. Walcarius & Delacôte, *Anal. Chim. Acta***2005**, 547, 3
9. Ganesan & Walcarius, *Langmuir***2004**, 20, 3632
10. Walcarius & Ganesan, *Langmuir***2006**, 22, 469

14:30

Oral

The phase development of the amorphous precursors of the ZrO₂-SnO₂ system during the thermal treatment

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The amorphous precursors of the ZrO₂-SnO₂ system on the ZrO₂-rich side of the concentration range were prepared by co-precipitation from aqueous solutions of the corresponding salts. The Zr/Sn molar ratio in the obtained products was determined by the results of energy-dispersive X-ray spectrometry (EDS). The thermal behaviour of the amorphous precursors was monitored using differential thermal analysis (DTA), X-ray powder diffraction (XRD), Raman spectroscopy and field emission scanning electron microscopy (FE-SEM). Crystallization temperature of the amorphous precursors rose with an increase in the SnO₂ content, from 405 °C (0 mol% SnO₂) to 500 °C (40 mol% SnO₂). The results of phase analysis show that the incorporation of Sn⁴⁺ ions has very small influence on the stabilization of high-temperature polymorphs of ZrO₂. A phase structurally closely related to monoclinic (*m*-) ZrO₂ is dominant in all crystallization products. Metastable products obtained upon crystallization of amorphous precursors also contain a phase structurally closely related to tetragonal (*t*-) ZrO₂, which disappears with further thermal treatment (up to 1100 °C). The first sign of a phase structurally closely related to cassiterite (*t*-SnO₂) appeared in the crystallization products with 30 mol% of SnO₂. A rise in calcination temperature leads to a decrease in the solid solubility of Sn⁴⁺ ions. The results of precise lattice parameter measurements show that the incorporation of Sn⁴⁺ ions causes an asymmetric local distortion of the *m*-ZrO₂-type crystal lattice.

14:45

Oral

Co-precipitation of As (III/V) and Se (IV/VI) with calcite

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The study of the adsorption of contaminants in soils is a very active area of research in the fields of geochemistry and soil physical-chemistry. Some important constituents of soils, like clays, iron oxides, carbonates or sulfate minerals are of particular interest because their

capacity to adsorb elements that are potentially risky for the environment, in the form of cations or oxyanions.

Carbonates are present in many near-surface environments. Specifically, calcite is a very abundant mineral and so its fundamental importance in many fields, related to immobilization of both inorganic and organic species. For this reason, ion incorporation in the structure of carbonate minerals is a subject of major concern, as well as the nanoscale mechanisms by which the ions are exchanged or substituted. In this context, co-precipitation is the most important mechanism by which an ion can get trapped into the bulk of a mineral, retarding its transport and subsequent availability to the environment.

In this work we present a systematic study of synthetic calcite samples co-precipitated with As(III), As(V), Se(IV) and Se(VI), to understand the sites and mechanisms of incorporation of their oxyanions. Combining spectroscopic (Neutron and X-ray Diffraction, EXAFS) and modeling (DFT) techniques, we probe the bulk minerals and gain insight into the substitution processes that take. Simulations help us to understand and predict which sites within the structure of the mineral would be more favorable for the substitution to occur.

The precise knowledge of the concentration of substituted As or Se in the bulk of the minerals is very important to predict the fate of these ions upon its release to the environment.

15:00

Oral

A model of the erbium ion exchange process in lithium niobate crystals

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Since the last decade, erbium ion has been the subject of an increasing interest as one of the most promising dopant for photonic materials, with application in integrated optoelectronics. Among different hosts, lithium niobate (LiNbO₃) has drawn a particular attention, thanks to its good intrinsic non linear optical properties. Several methods are currently under investigation for the erbium doping of the LiNbO₃ crystals, with the aim to control the erbium distribution, concentration and its site location. Local doping by thermal diffusion and by MeV ion implantation has been extensively studied [1,2] as well as the bulk doping in single domain and in periodically poled structures [3]. Recently we have demonstrated that also ion exchange is suitable for the erbium local doping of LiNbO₃ crystals [4] and we present the results of a systematic investigation of this process as a function of the main experimental parameters. In particular, we suggest a model based on the Nernst-Planck equations in order to explain the incorporation of trivalent ions by the ion exchange process in lithium niobate crystals. Due to the material anisotropy and the different valence state of the exchanged species, a correction to the ion flux expression is considered to include the strain effects. The model is then used to predict the erbium ion exchange in both X- and Z-cut slices. By comparing the predicted profiles to the dopant in-depth profiles measured by secondary ion mass spectrometry, we report on the model validity.

References

- [1] C.Buchal and S. Mohr, *Jour. Mater.Res.* 6(1), p.134, 1991
- [2] F.Caccavale, F.Segato, I.Mansour, J.M.Almeida, *Jour. Mater.Res.* 13(6) p.1672, 1998

[3] C.Sada, N.Argiolas and M.Bazzan Appl. Phys. Lett. 79 (14) p. 2163, 2001

[4] C.Sada, E.Borsella, F.Caccavale, F.Gonella, F.Segato, Yu.N. Korkishko, V.A. Fedorov, T.V.Morozova, G.Battaglin, and R.Polloni, Appl.Phys.Lett. 72 (26), p.3431, (1998)

15:15 Oral

Theoretical analysis and Characterization of the Refractive Index Profiles of Copper Ion-exchanged Glass Planar Waveguides

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Glass planar waveguides were fabricated by copper ion-exchange technique. Refractive index profiles caused by Cu^+ and Cu^{2+} were determined by Inverse-WKB method and the calculation from the profile of the ion concentrations. The calculation results shows that the changes of refractive index near the region of surface in the glasses were caused by Cu^+ and Cu^{2+} together, and the changes in the deep region of the glasses were caused by Cu^+ . Cu^{2+} only contributed to the index changes in the region near the surface. The index profile calculated from the ion concentrations also appeared to be a quasi-gradient index profile, and when the ion-exchange process was carried out for more than 5 minutes, the samples measured by prism-coupling technique showed no modes. It could be caused by the decrease of the surface index of the waveguides. Both the out-diffusion of Cu^+ and the stress release of Cu^{2+} caused by anneal contributed to the decrease of the surface index.

Coffee break

Wednesday afternoon, 19 September, 15:30

Joint Poster Session (Part II)

Poster Award Ceremony

Wednesday afternoon, 19 September, 15:50

Posters

Monday, 17 September

Joint Poster Session (Part I)

Monday & Wednesday

Monday afternoon, 17 September, 15:50

15:50 Poster F1

Nanocomposites on base of $\text{CdS}_x\text{Se}_{1-x}$ nanoparticles and polymer matrix, formed by combination of SILAR and ion-exchange methods

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Cadmium sulphide and selenide nanoparticles are one of the perspective materials in nanoscience. These materials could be used as biomarkers in medicine, production of high speed devices, catalysis. It was developed different methods of cadmium sulphide and selenide nanoparticles obtaining: such as, successful ion layered adsorption and reaction (SILAR), ultrasonic irradiation, matrix crystallization and etc. In given work we consider investigation of possibility of $\text{CdS}_x\text{Se}_{1-x}$ nanoparticles growth by combination of SILAR and ion-exchange methods. Firstly it was formed cadmium sulphide nanoparticles in polymer matrix with different sizes: 2, 3, 5, 7, 10 growth cycles. Then obtaining samples immersed electrolyte solution containing ions of selenium Se^{2-} . Band gap measurement shows that band gap of all samples is decreased. In high concentration and long persistence time band gap of particles approached to value of bulk crystals CdSe $E_g = 1,74$ eV. It was investigated chemical compound of received structures with the help EDAX. Results of analysis show that by selecting concentration and ion-exchange time, chemical compound of $\text{CdS}_x\text{Se}_{1-x}$ nanoparticles could be varied over a wide range.

This work carried out by STCU financial support, grant № 3486.

15:50 Poster F2

Effect of composition on ion exchange property of glasses

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Two glass samples BS1 $[(\text{Na}_2\text{O})_{0.22}(\text{B}_2\text{O}_3)_{0.05}(\text{SiO}_2)_{0.69}(\text{Al}_2\text{O}_3)_{0.011}(\text{K}_2\text{O})_{0.029}]$ and BS2 $[(\text{Na}_2\text{O})_{0.025}(\text{B}_2\text{O}_3)_{0.245}(\text{SiO}_2)_{0.69}(\text{Al}_2\text{O}_3)_{0.011}(\text{K}_2\text{O})_{0.029}]$ were prepared by the conventional melt-quench method. The ion exchange properties of these samples were studied using Cu^{2+} and Zn^{2+} ions. Based on these studies, it is seen that BS1 has higher ion exchange capacity than BS2. This is due to more number of nonbridging oxygen atoms present in the BS1 glass as revealed by the ^{29}Si and ^{11}B magic angle spinning nuclear magnetic resonance (MAS NMR) studies. Moreover, it is found that BS1 has more affinity for Cu^{2+} than Zn^{2+} . This is due to the formation of Cu^{2+} clusters as confirmed by ESR study.

15:50 Poster F3

Solid state field-assisted ion exchange for light waveguide application

Francesco Gonella¹, Elti Cattaruzza¹, Ali Shahid², Alberto Quaranta³, Cinzia Sada²

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A field-assisted ion exchange configuration has been recently developed for doping silicate glasses with different transition elements, namely, cobalt, gold, copper and silver. Metallic films deposited onto glass slides by the rf-sputtering technique were used as the metal ions source. In the presented experiments, optical propagation properties were investigated for various systems.

15:50 Poster F4

Study ion of Ag⁺/Na⁺ ion-exchange diffusion on germanate glasses : Realization of single-mode waveguides at the wavelength of 1.55 μm .

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Ion-exchange technology on glass has been successfully used for more than twenty years to realize dependable and low cost integrated optics devices on silicate or phosphate glasses for the realization of passive and active devices in the telecommunication wavelengths operations range (from $\lambda = 0.8$ to $1.7 \mu\text{m}$). However, the recent developments of integrated optics instruments for astronomical interferometers or biological sensors has lead to an increase of the devices operation range towards the mid-infrared. For these reasons, we present in this paper the development and the realization of single-mode waveguides by means of a binary ion-exchange on glass operating between $\lambda = 2$ and $4 \mu\text{m}$. In this study, the choice of germanate glass BGA-G115 from Kigre Inc. has been made because of both its similarity with silicate glass, its content of Na⁺ ions and its excellent transparency until $\lambda = 5 \mu\text{m}$. A complete study of the silver ion diffusion on this new glass matrix has been performed allowing the determination of silver and sodium ion-diffusion coefficients, which have been extracted by realizing slab waveguides with different ion-exchange times, temperatures or silver concentrations. Using these datas, simulations have shown that an ion-exchange of 90 min in a 0.03AgNO₃-0.97NaNO₃ molten salt at a temperature of 330°C can lead to the realization of single mode channel waveguides at either $\lambda = 1.55 \mu\text{m}$ or $\lambda = 3.39 \mu\text{m}$ depending on the diffusion window width. To demonstrate channel waveguides on BGA-G115, a specific technological process based on the deposition of a polysilicon masking layer has been implemented. Single-mode channel waveguides, with a $2.5 \mu\text{m}$ diffusion window width, have thus been realized and characterized at the wavelength of $1.55 \mu\text{m}$. Modal size has been measured to be $10 \mu\text{m} \pm 1 \mu\text{m} \times 7 \mu\text{m} \pm 1 \mu\text{m}$ for propagation losses of $1.2 \text{ dB/cm} \pm 0.5 \text{ dB/cm}$ for a $2 \text{ cm} \pm 0.1 \text{ cm}$ long device.

15:50 Poster F5

Solid state silver ion exchange in Er:glass sol-gel films: an EXAFS study of the Er environmentChiara Maurizio¹, Francesco Dacapito¹, Elti Cattaruzza², Francesco Gonella², Alessandro Martucci⁴, Ali Shahid³, Enrico Trave³

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The presence of silver in Er-containing dielectric matrices is known to enhance the photoluminescence properties of the rare earth element. In this experiment, Er:SiO₂:Na₂O sol-gel glass films were doped with silver by a field-assisted ion exchange procedure. EXAFS analysis of the obtained systems are used to detect the possible modification of erbium environment upon the Ag ion exchange. The results are then related to the optical response of the samples.

15:50 Poster F6

In-situ XRPD studies of anion exchange in layered double hydroxidesRune E. Johnsen¹, Poul Norby²

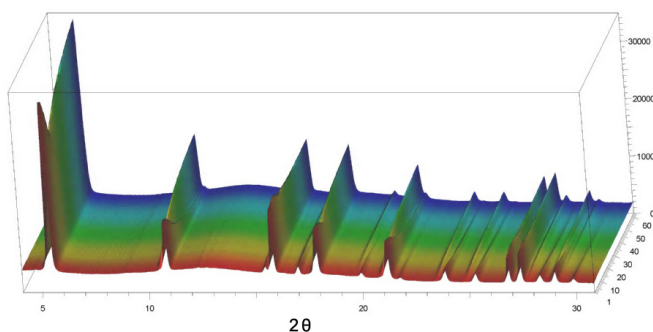
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In-situ X-ray powder diffraction is a powerful tool in studies of dynamic structural transformations and microtextural changes in crystalline materials. By means of a high intensity synchrotron source and a special designed in-situ cell even fast chemical reactions and transformations involving crystalline materials can be monitored. In this work we have followed structural changes of anionic clays in-situ during ion exchange.

Layered double hydroxides (LDH) comprise a group of materials with the general composition $[\text{M(II)}_{1-x}\text{M(III)}_x(\text{OH})_2](\text{A}^{n-})_{x/n} \cdot n\text{H}_2\text{O}$ where M(II) and M(III) are di- and trivalent metal ions and A^{n-} is the counter anion. LDHs have among other things been used as catalysts, catalyst supports, absorbents, and anion exchangers. The LDH structure can be described as a stacking of alternating positively charged metal hydroxide layers (brucite-type) and layers of negatively charged counter anions and water. The anion may be e.g. CO_3^{2-} , NO_3^- , OH^- , Cl^- , SO_4^{2-} or a combination of these.

Structural and microtextural changes during anion exchange in cobalt-aluminum layered double hydroxides were studied using in-situ X-ray powder diffraction (e.g. acid-enhanced ion exchange of carbonate LDH and subsequent back-exchange was studied). The ion exchange process is a one or two phase transformation depending of the composition of the initial LDH and the ion-exchange environment. Rietveld refinements of selected layered double hydroxides reveal structural changes as the ion exchange progresses.



15:50 Poster F7

Multitools approach for improved understanding of the structures and dynamics in LDH system: comparison of molecular dynamic simulations with X-ray diffraction refinements and micro calorimetric titrations.

Christine Taviot-Gueho, Yongjun Feng, Julien Pisson, Nicole Morel-Desrosiers, Fabrice Leroux, Patrice Malfreyt, Vanessa Prevot

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Layered Double Hydroxides (LDH) described by the general formula $[\text{M}^{2+}_{1-x}\text{M}^{3+}_x(\text{OH})_2]\text{A}^{m-}_{x/m} \cdot n\text{H}_2\text{O}$ (M^{2+} and M^{3+} are divalent and trivalent

cations forming the hydroxide layers and A^{m-} the interlayer anions surrounded by water molecules) have been widely studied in recent years as a layered host structure particularly suitable for the preparation of hybrid materials.

In order to achieve the objectives in materials synthesis, a good knowledge of the synthesis fundamental mechanisms is required. Although the exchange properties of LDH have been widely discussed into the literature, only a few papers have dealt with the exchange reaction mechanism so far. Recent technological and experimental advances in energy dispersive X-ray diffraction (EDXRD) using synchrotron radiation and recent developments in computational molecular modeling techniques provide new possibilities for the study of solid-state kinetics.

By means of EDXRD, we have shown the formation of second-stage intermediates during the exchange reaction of chloride anions in $Zn_2Al(OH)_6Cl \cdot nH_2O$ with a series of dicarboxylate anions. This phenomenon has only been observed for inorganic-organic anion exchange reactions so far while inorganic-inorganic exchange reactions occur in one-step process. The formation of such heterostructures offers new perspectives for LDH intercalation chemistry, particularly in direction of multifunctional materials.

Microcalorimetric measurements were performed indicating endothermic exchange enthalpies in the case of inorganic-organic anion exchange reactions and suggesting that staging is needed to overcome the energy barrier.

An other interesting aspect of the present work is the use of MD simulations combined with powder X-ray diffraction data to get better insights into the structure of $Zn_2Al(OH)_6Cl \cdot nH_2O$, $Zn_2Al(OH)_6(\text{tartrate})_{1/2} \cdot nH_2O$ and $Zn_2Al(OH)_6(\text{succinate})_{1/2} \cdot nH_2O$ with major emphasis on the interlayer structure and dynamical behavior of interlayer species.

15:50	Poster	F8

Advanced Wastewater Treatment for Heavy Metals Immobilisation Using Modified Fly Ash

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Wastewater treatment, using waste materials as adsorbents, represents a path for developing sustainable processes and for reducing the environmental burden. The use of fly ash in wastewater treatment, for heavy metal immobilisation, represents a topic much addressed to in the past years solving both, the problems of advanced treatment and the intelligent use of fly ash.

Ion exchange or chemical trapping of the ions, on the fly ash surface, are the expected processes for heavy metal removal on this substrate.

The fly ash surface properties depend on many factors, including the coal composition, the burning process and the furnace characteristics. Developing an up-scalable process requires reproducible surface properties, less (or no) related to the above mentioned influences.

The study presents the results obtained in Cd^{2+} and Ni^{2+} removal, using fly ash from CHP Brasov (Romania) with modified surface. The fly ash morphology is characterised using AFM while XRD provides composition and crystallinity data.

Metal ions solutions ($c = 10^{-4} - 10^{-2} \text{ M}$) are mixed in a reaction flask with fly ash (powder); samples of fly ash washed in ultra pure water or with modified surfaces after long contact with alkaline (NaOH), acid (HCl) and complexing agents (Complexone III, pyrocatechol violet) were used in series of experiments. Using the optimized adsorption parameters (contact time, wastewater volume: adsorbent mass ratio), the adsorption isotherms are obtained and the adsorption mechanisms are discussed.

Highly efficient chemical adsorption, even at very low heavy metals concentrations (50 ppm), is registered for fly ash with negative (HO^-) surface charge.

15:50	Poster	F9

Sorption removal of arsenic by cerium-exchanged zeolite

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Water pollution caused by arsenic, which is harmful to man and living organisms, is one of a major problems faced by many countries. Due to this scenario, development of new methods and sorbents for arsenic removal from water has been the subject of intense research lately. Zeolites have been used to sorb many metal cations but show no affinity for anions due to their permanent negative charges on their crystalline aluminosilicate surface. In this work modification of zeolite P was performed by exchanged of its sodium with cerium, in which the latter has strong affinity towards arsenate ($As(V)$) anion. The resulting cerium-exchanged zeolite P, (CeZP) was subsequently used to sorb $As(V)$ from aqueous solution. Parameters such as pH, initial $As(V)$ concentration, temperature, kinetics and effect of foreign ions were investigated.

Maximum sorption of $As(V)$ by CeZP occurred at pH range 3 to 6 at room temperature. In addition, the sorption capacity increased with increasing initial $As(V)$ concentrations. The sorption follows Langmuir model with maximum sorption capacity of 8.72 mg g^{-1} at 25°C and increased to 23.42 mg g^{-1} at 90°C , indicating an endothermic process. This is confirmed by the positive values of sorption enthalpy, $+22.25 \text{ kJ mol}^{-1}$. The sorption process was found to be spontaneous with free energy value of $-10.51 \text{ kJ mol}^{-1}$. A positive entropy change for sorption of $As(V)$ ion on CeZP could be due to fixation of the ions on the Ce(IV) that was randomly distributed on the zeolite P. Kinetics study shows that the $As(V)$ sorption follows pseudo second order kinetic with rate constant of $1.14 \times 10^{-3} \text{ g mg}^{-1} \text{ min}^{-1}$. The arsenic sorption by CeZP was not affected by the present carbonate, chloride, sulphate, bromide and nitrate but was reduced significantly in the presence of phosphate. This study shows that the as prepared CeZP was found effective for the removal of arsenic ion from wastewater sample of wood treatment industry.

15:50	Poster	F10

Removal of heavy metals from aqueous solutions by sorption on hydroxyapatite

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Heavy metal contamination of waters and soils can seriously affect living organisms. The bioavailability of metal ions can be reduced immobilizing them in minerals with low solubility. In this process some cations of the mineral are substituted by the metallic cations present in the solution. The removal of Cd, Cu, Zn and Pb from aqueous solutions by sorption onto synthetic hydroxyapatite (HA) surfaces has been investigated in batch conditions at 25 ± 2 °C. The metals were applied both as single or multi-metal (Cd+Cu+Zn+Pb) systems with initial metal concentrations ranging from 0 to 8 mmol/L. The removal capacity of HA in single-metal systems was 0.058-1.681 mmol of Cd/g, 0.016-0.764 mmol of Cu and 0.015-0.725 mmol of Zn/g of HA. In the multi-metal systems competitive metal sorption reduced the removal capacity by 63-83% for Cd, 13-76% for Cu and 10-63% for Zn, compared to the single-metal systems. The sorption of heavy metals by HA follows the Langmuir model. Heavy metal immobilization occurs through a two-step mechanism: first rapid surface complexation followed by partial dissolution of HA and ion exchange with Ca resulting in the formation of a heavy metal-containing HA.

The incorporation site of the metals in HA has been investigated by Extended X-ray Absorption Spectroscopy in the case of the four cited metals. Different situations appear for the various metals with detection of a metal-oxygen first coordination shell in all cases and a visible second coordination shell only for Pb and Cd. The metals appear to have a low coordination with O at distances much shorter than those typical of Ca. As a general finding, we observe different sites around the metal than that expected for a simple substitution of Ca and the possible scenarios will be described in detail for each case.

15:50	Poster	F11

Ionic Conduction Behaviour Study for small Proton Exchange Membrane Fuel Cells

Laurentiu Patularu, Daniela Stoenescu, Dumitru Mirica, Ioan Stefanescu, Vasile Stanciu, Mihai Culcer, Roxana Ionete

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Proton exchange membrane fuel cells (PEMFC) are promising power sources since they offer a highly efficient and environmentally friendly solution for energy conversion. In this paper there are presented the results obtained in different working conditions for Small Scale PEMFCs (25 cm² working electrode). The tests that were performed are: polarization curves, impedance measurement using EIS Spectroscopy, ionic behavior in different temperature conditions and variable pressures.

15:50	Poster	F12

Design and Characterization of Ion Exchange Membranes for PEM Fuel Cells

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The Proton Exchange Membrane Fuel Cells (PEMFC) is considered as the leading candidate to replace the internal combustion engine in the 21st century, as well as being a key technology both for small sta-

tionary power generation, transportation and mobile systems. The most commonly known and studied PEMs are based on nonaromatic perfluorinated hydrocarbons such as Nafion, Aciplex, etc. This paper presents the results obtained using a commercial block copolymers of the styrene-butadiene in structural design of a Proton Exchange Membrane for Fuel Cells. The membranes were structural tested by FTIR spectroscopy and X-Ray, and was measured their Ionic Exchange Capacity (IEC), Protonic Conductivity and Thermal Stability.

15:50	Poster	F13

New process for the treatment of radioactive waste using polyantimonic acid and its derivatives

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Extraction by ion exchange is one of the most known methods of radioactive waste treatment. The main problem of this method is that after the extraction process, the exchanger must be backwashed to convert the radioactive waste into the form suitable for immobilization. This leads to the formation of great volumes of secondary aqueous solutions. The exclusion of this stage from the process will make it safer and environmentally friendly. The polyantimonic acid and its derivatives are well known ion-exchangers. Besides that, some of mixed oxides containing antimony adopt pyrochlore structure that makes it possible to use them for immobilization. New process for the treatment of radioactive waste consisting of extraction and immobilization in the same crystalline matrix based on mixed antimony oxide are presented in this report.

Symposium G

Welcome

The EMRS Fall Meeting Symposium G (Genetic Algorithms in Materials Science and Engineering, acronym: GAMS2007) will provide a forum for establishing the current state-of-the-art in genetic algorithms and their applications in materials science.

Genetic or Evolutionary Algorithms, which mimic, in a simplified way, the evolutionary mechanisms (mutation, selection and crossover) that occur in living organisms, have been developing rapidly especially since the mid-1990s. Their applications cover virtually all domains of science, technology, medicine, and indeed everyday life, and have been facilitated by the availability of computer programs developed by specialists.

Problems that have been tackled using these techniques encompass a broad spectrum of applications including:

- modelling of materials, simulation of nanocrystals and atomic/molecular clusters,
- optimization of metallurgical processes, including iron making,
- search for new materials with desired properties,
- crystal/protein structure prediction,
- crystal structure solution,
- texture analysis, powder pattern indexing and indexing crystal faces,
- control of crystal growth and multilayer growth processes,
- catalyst design and drug design,
- solving computational problems in spectroscopy,
- RNA and protein folding,
- analysis of physical and structural properties of crystal surfaces and thin layer systems.

Such programs can be readily adapted to include specific user-defined tasks. The symposium is intended to collect together presentations concerning applications of these techniques in materials sciences, with the aim of promoting this methodology within the field and thus encouraging more widespread applications of these techniques to solve more difficult and challenging scientific and technological problems in the future. The symposium will give particular emphasis to the exchange of information on advances in methodology and in promoting the opportunities for applying this methodology among materials scientists.

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Proceedings

The proceedings of Symposium G "Genetic Algorithms in Materials Science and Engineering" (GAMS2007) will be published in: **Computational Materials Science (Elsevier)**

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Programme

Monday, 17 September

Opening & Plenary Session

Czochralski Award Ceremony & Acta Gold Medal Ceremony
Monday morning, 17 September, 10:00

Lunch break

Monday afternoon, 17 September, 12:30

Symposium opening

Opening
Monday afternoon, 17 September, 13:45

Fundamentals 1

Evolutionary approach: Tool and applications
Monday afternoon, 17 September, 14:00

14:00	Invited

Evolutionary method as a space searching tool - its basic features, advantages and drawbacks

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There is a common agreement that the evolutionary method (EM) is a space searching method inspired by the evolution theory and genetics. Usually, engineers use the EM as an optimization tool to perform e.g.

design or control tasks, rather than a tool for simulating the evolution. Therefore, it is useful to characterize the evolutionary method in terms of numerical optimization with much smaller stress on the biological metaphor.

EM consists in searching the space of candidate solutions by sampling it on random, according to a certain probability distribution, referred to as „sampling distribution” (SD). When EM is treated as an optimization tool, a crucial issue is to characterize the dynamics of the SD and, the areas where the candidate solutions will be most frequently sampled. It is believed that such „most interesting points” should be the points where the objective function takes its maximum. In the presentation, we provide counterarguments to that hypothesis.

The role of genetic operators are lively discussed. We argue that the mutation is indispensable, whereas the crossover can slow down the dynamics of the EM. We also discuss the issue of the population diversity, which is believed to be the factor that has to be maintained. We evidence that the diversity level is a result of the selection process rather than a separate factor that has to be specially controlled.

When solving a real world problem, one has to provide a reasonable solution in an acceptable time. We argue that it is more advisable to combine the evolutionary based search (which is robust to local maxima, but very imprecise) with local optimization methods (which in turn are rather precise and fast in finding local maxima). Possible methods for such hybridization are briefly discussed.

In case of practical problems, it is needed to introduce constraints to solutions infeasible solutions. EM allow for a variety of constraint handling techniques, which will be briefly mentioned and commented.

14:30 Invited

Unification of all evolutionary paradigms

Pierre Collet

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Evolutionary algorithms are quite ancient in the history of computing, traced back in 1953 in David Fogel's excellent Fossil Record, with works from Australia, the United States and Europe.

Of course, most people working on this strange field did not know each other, and many different paradigms started to emerge, the most popular being the Genetic Algorithms (US East Coast), the Evolution Strategies (Germany), Evolutionary Programming (US West Coast), and more recently Genetic Programming.

However, it happens so that all these different trends are so similar that it is possible to merge them into a more global and general Evolutionary Algorithm, that will be presented during this talk.

15:00 Invited

Dynamic modeling of material properties by intelligent systems

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This paper proposes intelligent system for dynamic modeling of material properties. Usability of the system is demonstrated by practical examples. The intelligent system is not limited only to the modeling of material properties, but it is a general approach for studying of different systems.

The proposed intelligent system consists of two parts: intelligent modeler and intelligent optimizer. The intelligent modeler is intended for the modeling of the system for which the behavior is not yet known. The intelligent optimizer is used for the optimization of the parameters for the system already described. Each of the two parts has its own input and output. The input to the modeler are the experimental data on the system studied, while the output is the model developed during modeling. The input to the optimizer is the model containing system parameters to be optimized. In most cases the modeler output is in the same time the optimizer input. The output of the optimizer is the optimized set of the system parameters.

The intelligent modeler gathers knowledge about the system studied using on-line sensing equipment. Since new knowledge is continuously coming into the modeler, the data are in fact dynamic environment. Consequently, the system as well as its optimized parameter values slowly change. The optimized parameters change the state of the environment (and vice versa) and influence general behavior of the system studied.

In the proposed concept, evolutionary algorithms are used for both intelligent modeler and intelligent optimizer. The genetic programming and the genetic approximation tools represent the core of the intelligent modeler. The core of the intelligent optimizer is genetic algorithm tool.

The paper concludes with the practical examples where the proposed intelligent system serves as a modeling tool for studying several different systems, i.e., modeling of bending capabilities of material, modeling of impact toughness, modeling of yield stress.

Coffee break

Monday afternoon, 17 September, 15:30

Joint Poster Session (Part I)

Monday & Wednesday

Monday afternoon, 17 September, 15:50

Tuesday, 18 September

Joint Session (9:00-10:30)

Nanoscale materials: modelling and applications

Tuesday morning, 18 September, 9:00

9:00 Invited

Semiconductor spintronics

Tomasz Dietl

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Prospects in applications of semiconductor spintronics for classical and quantum information hardware will be presented. Materials issues will be emphasized focussing on ferromagnetic semiconductors [1] and also on hybrid ferromagnetic metal/semiconductor systems, taking the Stern-Gerlach spin-filter as an example [2]. Various functionalities of ferromagnetic semiconductors will be listed, including electron and hole spin injection, tunnelling magnetoresistance [3], spin-reorientation

transition [4] as well as current-induced induced domain-wall displacement [5] and resistance [6].

Stimulated by demonstrated and foreseen functionalities, a considerable and successful effort has been devoted to develop semiconductors and oxides containing transition metals, in which ferromagnetic features persist up to above room temperature. Recent progress in understanding of these multi-component materials [7,8] will be described emphasising an interesting unexpected difficulties in characterisation of these systems by standard experimental probes and theoretical tools.

The work supported in part by projects NANOSPIN of EC and SPIN-TRA of European Science Foundation as well as by Humboldt Foundation.

1. T. Dietl and H. Ohno, *Materials Today* 9 (11) (2006) 18, and references therein.
2. J. Wróbel, T. Dietl, A. Łusakowski, G. Grabecki, K. Fronc, R. Hey, K.H. Ploog, and H. Shtrikman, *Phys. Rev. Lett.* 93 (2004) 246601.
3. P. Sankowski, P. Kacman, J. M. Majewski, T. Dietl, *Phys. Rev. B* 75 (2007) 045306.
4. K.-Y. Wang, M. Sawicki, K.W. Edmonds, R.P. Campion, S. Maat, C.T. Foxon, B.L. Gallagher, and T. Dietl, *Phys. Rev. Lett.* 95 (2005) 217204.
5. M. Yamanouchi, D. Chiba, F. Matsukura, T. Dietl, and H. Ohno, *Phys. Rev. Lett.* 96 (2006) 096601.
6. D. Chiba, M. Yamanouchi, F. Matsukura, T. Dietl, and H. Ohno, *Phys. Rev. Lett.* 96 (2006) 096602.
7. T. Dietl, *Nature Mat.* 5 (2006) 673.
8. S. Kuroda, N. Nishizawa, K. Takita, M. Mitome, Y. Bando, K. Osuch, and T. Dietl, *Nature Mat.* 6 (June, 2007).

9:30

Invited

Computer modelling in materials and nano-science

Richard A. Catlow

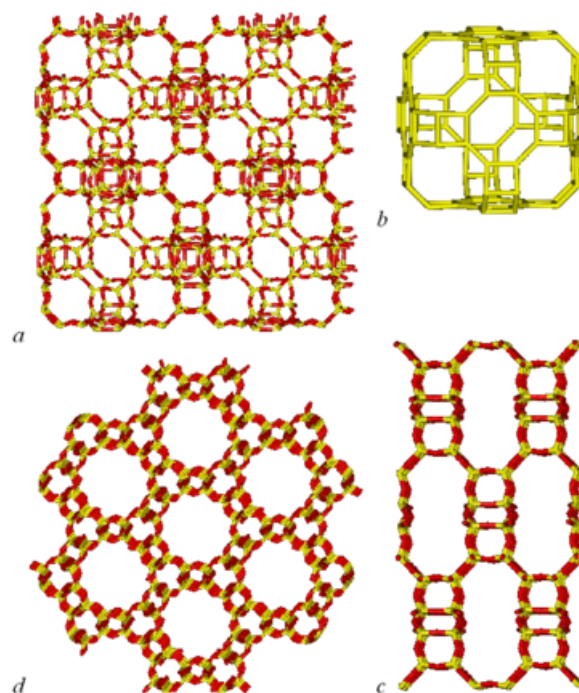
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Computer simulations techniques are an integral component of current materials science. This lecture will aim to present an overview of the current capabilities of atomistic modelling techniques in Materials science. It will focus on applications, although a brief introduction to techniques will be provided. The survey of applications will focus on:

- (1) Predicting the structures of complex materials.
- (2) Modelling surface structures and properties
- (3) Predicting nanoparticles structures and properties
- (4) Modelling reactivity

Topical examples will be presented in each case



Framework structures of some of the uninodal hypothetical zeolites derived from tiling theory: (a) structure 1_11, derived from one of the simple tilings, has a very low framework density of 11.4 T/1000 μ . The cages are shown more schematically in (b), illustrating the openness of the structure; (c) large-pore structure 1_71 has unidirectional 12-ring channels; (d) tetragonal structure 1_14 has elongated 12-ring channels, which run in orthogonal directions, intersecting to form a 3-D pore system.

10:00

Invited

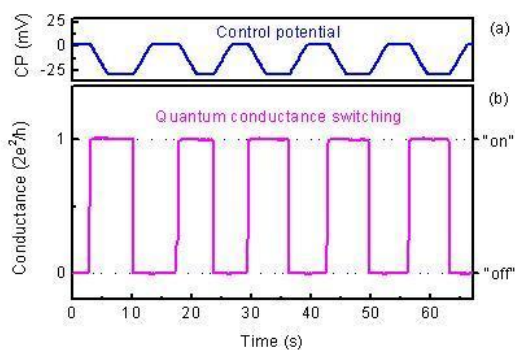
The Single-Atom Transistor

Thomas Schimmel^{1,2}, Fangqing Xie², Christian Obermair²

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An atomic-scale quantum conductance switch is demonstrated which allows to open and close an electrical circuit by the controlled and reproducible reconfiguration of individual silver atoms within an atomic-scale junction [1,2]. The only movable parts of the switch are the contacting atoms. The device is fabricated by electrochemical deposition of silver atoms and is entirely controlled by an external voltage applied to an independent third gate electrode. Controlled switching was performed between a quantized, electrically conducting “on-state” exhibiting a conductance of $G_0 = 2e^2/h$ ($\approx 1/12.9\text{k}\Omega$) or preselectable multiples of this value and an insulating “off-state”. The device, which reproducibly operates at room temperature, represents an atomic transistor or relay, opening intriguing perspectives for the emerging fields of quantum electronics and logics on the atomic scale.



- [1] F.-Q. Xie, L. Nittler, Ch. Obermair and Th. Schimmel,
Phys. Rev. Lett. **93**(12) 128303, (2004)
- [2] F.-Q. Xie, Ch. Obermair and Th. Schimmel,
Solid State Commun. **132**, 437 (2004)

Coffee break

Tuesday morning, 18 September, 10:30

Materials 1

Genetic algorithms for design of catalysts and protein studies
 Tuesday morning, 18 September, 11:00

11:00

Invited

Design of experiments and data analysis in the search for new compositions of heterogeneous catalysts

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In the search for new compositions of materials of maximal catalytic performance, various methods have been developed when using high-throughput experimentation for preparing and testing of catalysts. The methods are discussed along with the analysis of data obtained in experimentation.

For finding potential catalytic materials, a subset of representatives conveying the required information about all possibilities was addressed in the past by statistical design of experiments (DoE). DoE methods assume that interesting catalytic materials are uniformly distributed in the compositional space. If interesting materials form only one or several small clusters, methods of function optimization should be used. Most successful in the search of optimal catalytic materials have been evolutionary, i.e. a biologically inspired, stochastic optimisation methods. Their most well-known representatives are genetic algorithms (GA), in which the incorporation of random influences attempts to mimic the evolution of a genotype. Basically, that method comprises:

- random crossover;
- random mutation,

- selecting the locations for cross-over and mutation (parent locations) according to a probability distribution.

Subsequent data analysis may be done by artificial neural networks (ANN), which are distributed computing systems attempting to implement a part of the functionality of biological neural networks. From the point of view of data analysis, their most valuable feature is the ability to approximate arbitrarily complex dependences, such as the dependence of catalytic performance and composition of the material and its various physical and physicochemical properties. In this way, new insights into the science of heterogeneous catalysis may be obtained.

Applications of GA and ANN are illustrated for the development of catalytic materials for two reactions.

11:45

Invited

OptiCat: a general open source optimization platform for library design: application to catalyst design

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Catalysis is involved in more than 80% of commercial chemical processes. A wide variety of products are made through catalytic steps: chemicals, oil products, fertilizers, plastics, drugs and pharmaceuticals. Catalysts represent one of the most important mean to produce modern chemicals today: with the Gross Domestic Product in Europe being € 10,5 trillion, the “catalysis industry” accounts for 14% of the GDP in Europe.

Chemistry is a mature science. Many key catalytic processes, which have been setup hundred years ago, remain almost unchanged. Nevertheless, in the context of evolving environmental regulations and enhanced worldwide competitiveness, improved “drop-in” catalysts as well as “blockbusters” for dream reactions are seek.

In pioneer’s times, combinatorial studies were already used for the discovery and optimization of “breakthrough” catalysts. Even if the level of understanding in Catalysis reaches outstanding levels, the today’s research is still experimental and mainly based on “trial and error” approaches.

High Throughput experimentation has led to a new deal in research methodology. Screening technology enables to carry out hundreds experiments at a time. The direct consequence is the combinatorial explosion. The questions which arise are: Which are the most relevant experiments to carry out ? Which is the most efficient screening strategy to apply?

Innovative informatic solutions and library design methodologies will be discussed. New open-source optimization software (OptiCat) using cutting-edge technologies such as Web Services and Data Workflow treatments will be presented. The integration of best-of-breed technology in a consistent informatics infrastructure will be illustrated.

12:15

Oral

Protein-ligand docking using a multi-objective genetic algorithm

Danilo Di Stefano¹, Gianluca Tasco², Rita Casadio²

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Ligand docking is the computational prediction of the bound conformation of a small molecule in a complex starting from its atomic coordinates. A docking procedure is composed of two contributions: a scoring function determining the relative score of different configurations of the ligand and a search procedure exploring the space of the possible ligand orientations and conformations. Scoring functions are often based on calculation of binding energies between ligand and receptor; the search procedures have to find the ligand pose that this corresponds to the absolute minimum of the binding energy landscape. Binding energies are sums of different contributions. Search procedures generally look for the minimum of the global scoring function neglecting the possibility to optimize the search towards each single member or a group of them (for example bonded and non-bonded energies). This work describes the application of a Multi-Objective Genetic Algorithm (MOGA) as a search procedure for a protein-ligand docking problem. The MOGA implementation in modeFRONTIER™ is used to optimize the docking towards each of the different contributions of the docking program Autodock v. 3.05 (<http://autodock.scripps.edu>) scoring function. MOGA results give a set of solutions, each one representing a compromise between the different objectives (the so called Pareto frontier). This could be useful, for example, to get a docked complex that exalts one of the contributions to the total binding energy. The results consist of a set of docked ligand conformations within the 2-objectives Pareto frontier (minimization of bonded energy and minimization of non-bonded energy) having a root mean squared deviation from the crystal structure smaller than 1.5 Å. From the inspection of the Pareto frontier results that the non-bonded interactions effectively have a greater influence than bonded interactions in guiding the search.

Lunch break

Tuesday afternoon, 18 September, 12:30

Materials 2

Genetic algorithms for materials design and structure prediction
Tuesday afternoon, 18 September, 13:45

13:45

Invited

Tailor-made material design: An evolutionary approach using multi-objective genetic algorithms

Nirupam Chakraborti, Jayakanth Ravichandran, Sreevathsan Ramanathan, Baidurya Bhattacharya

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The main objective of this work is to design structural materials by interatomic potential based methods and to explore materials of high strength to weight ratio with a thermodynamically stable structure. The idea is to develop a material that is simultaneously, stiff, light and at the same time thermodynamically stable. Since these requirements are conflicting to each other, it leads to a Pareto-optimal problem, which in this study was computed through the rather well known NSGA II Algorithm. Proper quantification was made for the three objectives, stiffness, lightness and energy of the system. Attempts were made to maximize the first two while the third was minimized. The three objectives were optimized for simple cubic, body-centered cubic, face-centered cubic crystal lattices and also for the amorphous atomic clusters. For the crystalline materials simulations were done keeping the lattice parameters as variables, which were allowed to evolve through a genetic procedure. In case of the atomic clusters the individu-

al atomic coordinates were allowed to vary without any special regards for a specific lattice structure, as it has been done in the crystalline cases. In all the simulations the material behavior was modeled using the Lennard-Jones potential function. The Pareto frontier obtained in this study would lead to a series of hypothetical materials, each with its own lattice parameters which, in terms of the three objectives considered in this study would be non-dominating to each other, thus representing the best possible compromises between the objective functions. The solutions obtained after achieving the Pareto-optimality are individually examined and their properties are compared and contrasted with the existing materials, as and when possible. A typical Pareto front is shown in the attached figure.

14:30

Invited

High-accuracy protein structure prediction by global optimization

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We propose a new high-accuracy protein structure modeling method based on straightforward and rigorous application of a global optimization to the three consecutive layers of modeling: multiple alignment, chain building, and side-chain modeling. We applied the conformational space annealing method to a consistency based score function for multiple alignment. For chain building and side-chain modeling, we optimized the MODELLER energy function and a SCWRL-like energy function using a rotamer library constructed specifically for a given target sequence. The method was applied to CASP7 targets in a blind fashion. Assessment of the prediction, conducted after the native experimental structures are released, demonstrates that significant improvements over existing methods are achieved in backbones as well as in side-chains for TBM (template-based modeling) and high-accuracy TBM targets. For most high-accuracy TBM targets (17/26), the predicted model was more accurate than the model one can construct from the best template in a posteriori fashion. It appears that the proposed method can extract relevant information out of multiple templates. Advantages as well as limitations of the method will be discussed.

15:00

Invited

A parallel evolutionary algorithm for molecule structure prediction

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Solving the structure prediction problem for complex proteins is difficult and computationally expensive. In this paper, we propose a bicriterion parallel hybrid genetic algorithm (GA) in order to efficiently deal with the problem using the computational grid. The use of a near-optimal metaheuristic, such as a GA, allows a significant reduction in the number of explored potential structures.

However, the complexity of the problem remains prohibitive as far as large proteins are concerned, making the use of parallel computing on the computational grid essential for its efficient resolution. A conjugated gradient-based Hill Climbing local search is combined with the

GA in order to intensify the search in the neighborhood of its provided configurations. In this paper we consider two molecular complexes: the tryptophan-cage protein (Brookhaven Protein Data Bank ID 1L2Y) and alpha-cyclodextrin. The experimentation results obtained on a computational grid show the effectiveness of the approach.

Coffee break

Tuesday afternoon, 18 September, 15:30

Method and Materials I

Genetic algorithms for design of materials

Tuesday afternoon, 18 September, 15:50

15:50

Oral

Determination of the compositions of NiMnGa magnetic shape memory alloys using hybrid evolutionary algorithms

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Magnetic shape memory (MSM) alloys are a new class of actuator materials with high actuation frequency, energy density and strain. MSM effect occurs in alloys, which exhibit a martensitic transformation and are ferromagnetic. It involves, under effect of magnetic field, a high strain achieved via reorientation of twinned martensite plates. The major problem is that even a slight change in the alloy's composition causes drastic changes in the martensitic transformation temperature (MTT) and MSM effect is only possible in the martensitic region.

Therefore it is crucial to be able to predict the MTT of any NiMnGa alloy. Artificial Neural Networks (ANN) with their learning and generalization ability may act as a suitable tool to predict the MTTs of NiMnGa alloys. ANN are generally used when the problem cannot be explicitly described by an algorithm, a set of equations, or a set of rules. A genetic algorithm (GA) is a search technique used in computing to find true or approximate solutions to optimization and search problems.

In a previous study, in order to predict the MTT, the performance of a multi-layer perceptron has been studied. Training and validation stages of the approach are performed by using data sets from many separate analysis results and our chemical analysis results were used for testing.

In this paper, as an inverse design approach, we concentrated on finding the composition of any NiMnGa alloy by using the MTT as the input in our ANN model. To build an advanced solution, we used GA and ANN in a hybrid manner to obtain the composition values.

In order to compare the performance of the candidate solutions obtained from alternative methods, the required fitness function for the MTT was determined by the ANN developed in the previous study. Solution quality (accuracy) and computation time were used as the crosscheck parameters for the comparison of results obtained from either method.

16:05

Oral

The design of electroceramic compounds using a neural network and multi-objective evolutionary algorithm

Steven Manos, Daniel J. Scott, Peter V. Coveney

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In our research work to be presented, ceramic materials are designed with a multi-objective evolutionary algorithm which have optimal permittivity, an important property in the design of dielectric ceramics for microwave and telecommunications applications. Given the difficulty of large-scale manufacture and characterisation of these materials, we have attempted to guide a search for novel materials through the application of data-mining algorithms to a generic materials database which has been populated with data gleaned from the literature and contains composition and property information of over 1000 compounds. The composition-function relationship was then encapsulated using an artificial neural network.

Various constraints and objectives were incorporated to help search for designs that are optimal with respect to the permittivity, are stoichiometrically correct and are manufacturable. The stoichiometry is determined through charge calculations while a 'reliability index' calculation is used to ensure that permittivity predictions are likely to be accurate. In this way, we attempt to discover new compounds that are similar to the existing compounds stored in the database, along with new compounds that are very different. Finally, ceramic compounds that consist of 4 to 6 elements are given preference.

The use of a multi-objective design approach is particularly powerful since, rather than discovering a single 'perfect' solution, a range of solutions is found, where each solution satisfies the objectives and constraints to varying degrees. The final decision of 'optimality' is then left to a human decision maker, since this often involves other factors not originally encapsulated within the objectives or constraints. The algorithm used, along with the genotype (ceramic compound representation), constraints and objectives will be discussed, along with optimal compounds discovered by the genetic algorithm.

16:20

Oral

Optimization of strain field distribution for generation of compound curve surfaces using line heating technique

Nisith R. Mandal, Pankaj Biswas, Om P. Sha

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In shipbuilding, one of the most tedious and human skill dependent operations is plate forming for producing compound curve plates. Line heating technique can be gainfully utilized to develop necessary thermal strains in the plate to generate the desired shapes. Thereby the process of plate forming becomes amenable to mechanization once the line heating pattern and heating parameters become available.

For generating a curved surface one needs to know the extent of strain that is to be applied and its direction. The deformation occurs perpendicular to the line of heating. Clearly there is no single way to form a given curved plate by line heating. The same curved plate can be formed from various starting templates using various amounts of strains. So here the objective is to find initial plate templates (also called *blanks*) for which the minimum strain has to be applied so that the desired shape is generated.

The strain field distributions thus have a significant effect on plate forming using line heating process. Depending on this, the line heating parameters (heat input, torch speed, heating pattern, etc) are decided. The blank size and the strain energy required to develop the desired compound curved surface fully depends on strain field distribution. The objective is to obtain minimum strain configurations so that least amount of raw material is used and at the same time the work required to produce the desired surface is also minimized.

In the present work a GA based optimized strain field distribution approach has been applied such that the strain energy as well as the material required for blank generation is minimised.

The strain distributions as obtained using GA for a pillow shaped surface are shown in Fig.1.

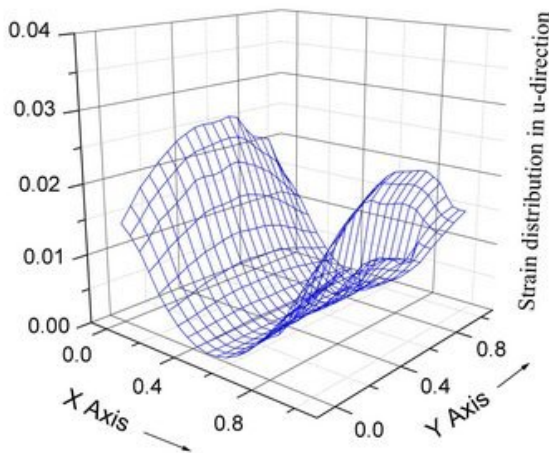


Fig.1 Strain distributions along u-direction

16:35

Oral

Searching for low energy configurations of disordered materials using genetic algorithms

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We show how a genetic algorithm together with energy minimisation can be used to find low energy configurations on the erratic and rugged potential energy surface of disordered periodic systems. Examples include simple test cases (e.g. one dimensional spin glass) disordered surfaces (e.g. the polar (100) UO_2 surface), alloys (e.g. Au-Cu) and mineral solid solution (e.g. CaCO_3 - MgCO_3). We pay particular attention to challenges in maintaining the diversity in the population due to the constraint associated with the fixed composition, and discuss the tuning of the parameters of the genetic algorithm. The genetic algorithm presented allows the study of disordered material in cases where a few configurations are thermally accessible and opens the way to efficient material design involving systems with a disordered structure

16:50

Oral

Genetic algorithm based multi-objective optimization of an ironmaking rotary kiln

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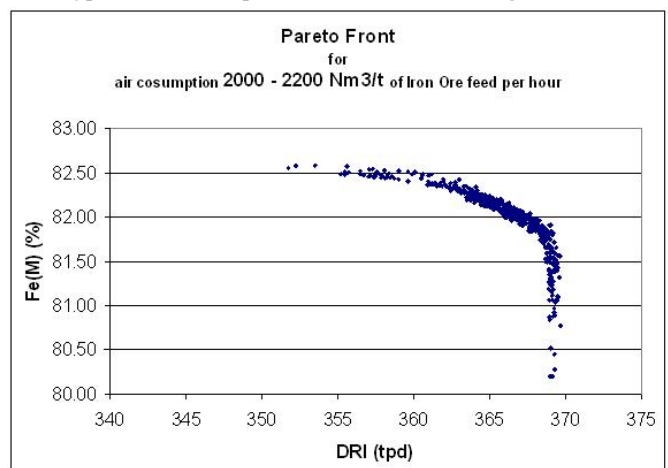
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In regions rich in iron ore and high ash non-coking coal quite often the preferred technology of steelmaking is the Rotary Kiln - Electric Arc Furnace route, as the minimum capacity level is of the order of hundred thousand tones and required investment is substantially less. The difference can be in the cost of production due the huge power requirement in electric steelmaking. Direct Reduced Iron (DRI) is produced in a rotary kiln and this route is commonly known as the DRI-EAF route.

In a rotary kiln the achievement of desired product quality depends upon the controlled addition of coal and air for developing the right reducing atmosphere for producing DRI of 90% metallization. DRI being a result of reduction of iron ore the production quantity and metallic iron content are conflicting i.e increasing the value of one will in principle reduce the value of the other. As both these objectives are simultaneously equally important they must be simultaneously optimized using multi-objective optimization techniques.

Multi-objective optimization facilitates the handling of data using constraints and one of the best ways to handle data with constraints is using genetic algorithms as the search space in this study is not very well defined. Genetic algorithms provide for a robust method of handling the data. The relationship between the individual inputs, a set of several variables like iron ore, coal and air input rate, ore and coal quality with the two simultaneous outputs, DRI production quantity and FeM content of DRI have been developed using artificial neural networks in this study while the search for simultaneous optimum results has been done using genetic algorithms to generate the input values of operating parameters and the corresponding output values.

Some typical results are presented in the attached Figure.



Wednesday, 19 September

Plenary Session

Wednesday morning, 19 September, 9:30

Coffee break

Wednesday morning, 19 September, 10:30

Structure*Evolutionary approach for crystal structure solution*

Wednesday morning, 19 September, 11:00

11:00

Invited

Optimization and applications of genetic algorithms for structure determination from powder diffraction dataKenneth Harris*Cardiff University, School of Chemistry, Park Place, Cardiff CF103AT, United Kingdom**e-mail: HarrisKDM@cardiff.ac.uk*

Many solids can be prepared only as microcrystalline powders and are therefore not suitable for structural characterization by conventional single-crystal X-ray diffraction techniques. For such materials, it is necessary to tackle structure determination using powder X-ray diffraction data [1]. Although traditional techniques for structure solution from powder X-ray diffraction data have been applied successfully in many cases, organic molecular materials represent a particularly challenging case. For these reasons, we are focusing on the development, implementation and optimization of new strategies for structure solution from powder X-ray diffraction data, with particular emphasis on molecular crystal structures. Our current work in this field is based on the direct-space strategy [2], in which a hypersurface defined by the powder profile R-factor is searched using a Genetic Algorithm [3,4]. In addition to applying this technique to tackle structure determination for a range of materials of academic and industrial interest, we are carrying out systematic explorations to find the optimum ways of implementing the Genetic Algorithm technique within this field [5,6]. The lecture will review fundamentals of the implementation of Genetic Algorithms in structure solution from powder diffraction data, and recent results from the application of this technique to determine crystal structures of molecular materials will be presented.

[1] K.D.M. Harris, E.Y. Cheung, *Chem. Soc. Rev.*, **2004**, 33, 526.[2] K.D.M. Harris, M. Tremayne, P. Lightfoot, P.G. Bruce, *J. Am. Chem. Soc.*, **1994**, 116, 3543.[3] B.M. Kariuki, H. Serrano-González, R.L. Johnston, K.D.M. Harris, *Chem. Phys. Lett.*, **1997**, 280, 189.[4] K.D.M. Harris, R.L. Johnston, B.M. Kariuki, *Acta Crystallogr.*, **1998**, A54, 632.[5] K.D.M. Harris, S. Habershon, E.Y. Cheung, R.L. Johnston, *Z. Kristallogr.*, **2004**, 219, 838.[6] Z. Zhou, V. Siegler, E.Y. Cheung, S. Habershon, K.D.M. Harris, R.L. Johnston, *ChemPhysChem*, **2007**, 8, 650.

11:45

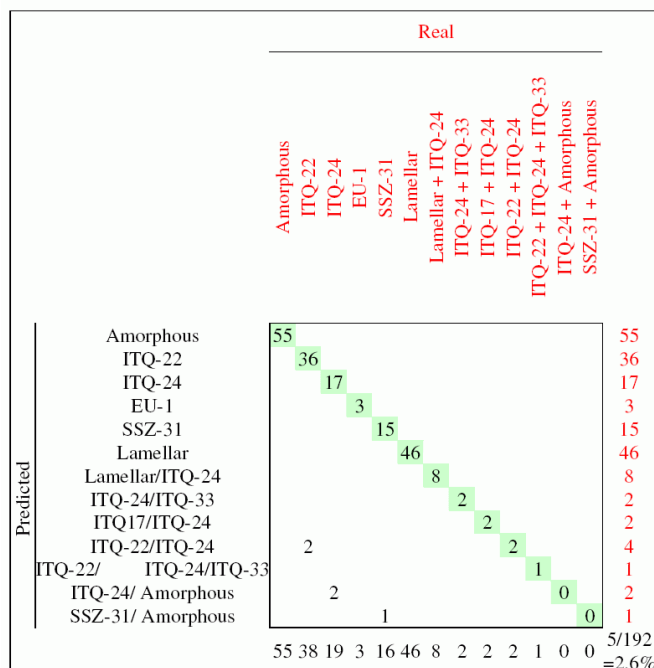
Invited

A robust methodology for high throughput identification of mixture of crystallographic phases from powder diffraction dataLaurent A. Baumes¹, Manuel Moliner¹, Remi Gaudin², Nicolas Nicoloyannis², Avelino Corma¹**1.** *Instituto de tecnologia quimica CSIC-UPV (ITQ), av de los naranjos, Valencia 46022, Spain* **2.** *ERIC University of Lyon II Lumiere, 5 Av Pierre Mendes France, Lyon 69676, France**e-mail: baumesl@itq.upv.es*

The knowledge of the structure at atomistic/molecular level of a material is required for any advanced understanding of its performance. Fortunately, characterization techniques are developing rapidly, with a major effort for parallelization and high-throughput (HT). On the other hand, the corresponding high volume of generated data requires an automatic data analysis system not to slow down the whole process. Most characterization responses take the form of "series" where the measurements follow an ordered variable; eg temperature (TGA, TPD), time (GC), wave length (photoluminescence, IR, UV), voltage (Voltammetry), angles (XRD)... Computing techniques for the treatment of such naturally ordered data, represents an actual challenge in data mining research.

Here we focus on zeolite since they are versatile materials for an increasing number of applications. The automatic determination of crystalline phases through X-ray diffraction data from powder represents a study of great interest for both chemistry and computer science. The algorithms initially created for multi-dimensional data are not effective when series are considered. We propose an approach called Adaptable Time Warping. ATW is a learning process which uses a genetic algorithm. According to a given classification problem, ATW approach adapts itself for better capturing data specificity. ATW effectiveness is demonstrated through numerous tests: 20 benchmarks and 3 real datasets of zeolite investigations [1, 2, 3]. The originality and power of the technique are clearly demonstrated when the complete phase diagram corresponding to the research space of the zeolite ITQ33 is automatically revealed (see Table1). ATW appears as innovative and a very promising methodology considering the numerous potential applications in chemistry.

Table 1. Confusion matrix with the real phases in the experimental design versus predicted classes obtained with ATW



1. M. Moliner et al., *Microp.Mesop. Mater.***2005**, 78, 73-81.
2. A. Corma et al., *Nature*. **2002**, 418, 514-517
3. A. Corma et al., *Nature*. **2006**, 443, 842-845

12:15

Oral

Computer simulation and quantum chemistry analysis as instrument for highly ordered supramolecular materials formation

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Computer simulation and quantum chemistry analysis were applied for a prediction of molecular architecture of biopolymer-inorganic systems for preparation of new nanoscale hybrid supramolecular materials. Polyaminoacid (PAA) fragments attached to cyclotriphosphazene (CTP) through spacer groups form organic/inorganic hybrids. Further modification of these compounds by photosensitive, biologically active species results in systems possessing multifunctional properties.

PAA/CTP fragments structure optimization done by methods of non-empirical, semiempirical quantum chemistry and methods of molecular mechanics. Quantum-topological analysis of electronic density revealed secondary non-valent interactions between C and H atoms in neighboring phenoxy-groups connected to phosphazene cycle. These interactions reflect mutual influence of spacer groups, providing their spatial orientation and stabilizing molecular structure.

We simulated the architecture of poly(g-methyl L-glutamate) (PMG) and poly(g-benzyl L-glutamate) (PBG) (total degree of polymerization $n=60$) derivatives of cyclotriphosphazene and confirmed the structure experimentally. PAA/CTP hybrids include six α -helices in trans-position to CTP core. α -helices in turn organized into bundles. Further

self-assembling leads to formation of aggregates comprising 5 molecules for PBG/CTP and 6 molecules for PMG/CTP, where helices are organized into two-dimensional hexagonal packing structure that was proved by SWAXS. Formation of this highly ordered supramolecular systems becomes possible owing to hydrogen bonding and non-covalent interactions, that is illustrated by IR and CD data.

Quantum chemical prediction and structural properties study permits to elaborate an approach for creation of multi-functional materials with defined properties.

The authors are grateful for financial support to Russian Ministry of Education and Science (Projects 5051 and 5118).

Lunch break

Wednesday afternoon, 19 September, 12:30

Nanomaterials and nanostructures 1

Optimisation of nanoalloys and semiconductor nanostructures I

Wednesday afternoon, 19 September, 14:00

14:00

Invited

Design and application of genetic algorithms for structural optimization of nanoalloys

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The desire to fabricate materials with well defined, controllable properties and structures, on the nanometre scale, coupled with the flexibility afforded by intermetallic materials, has generated interest in bimetallic alloy clusters or "nanoalloys". One of the major reasons for interest in nanoalloy particles is the fact that their chemical and physical properties may be tuned by varying the composition and atomic ordering, as well as the size of the clusters. Their surface structures, compositions and segregation properties are of interest as they are important in determining chemical reactivity, and especially catalytic activity. Nanoalloy clusters are also of interest as they may display structures and properties which are distinct from those of the pure elemental clusters. In this talk, I will start by introducing the field of nanoalloys and reasons for studying them. I will also summarise the important factors that determine the mixing or segregation properties of nanoalloy clusters. I will describe the model (the Gupta many-body potential) and the methodologies (genetic algorithms, Basin Hopping Monte Carlo and systematic optimisation studies of geometric shell clusters) that we have used to study the structures and segregation properties of a variety of nanoalloys - paying particular attention to the design of the genetic algorithm. The results of recent DFT calculations will also be presented.

14:45

Invited

Global optimization of 1-D and 2-D semiconductor nanostructures

Cristian V. Ciobanu

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In the cluster structure community, global optimization methods are common tools for arriving at the atomic structure of molecular and atomic clusters. The large number of local minima of the potential energy surface (PES) of these clusters, and the fact that these local minima proliferate exponentially with the number of atoms in the cluster simply demands the use of fast stochastic methods to find the optimum atomic configuration. Therefore, most of the development work has come from (and mostly stayed within) the cluster structure community. Partly due to wide availability and landmark successes of scanning tunneling microscopy (STM) and other high resolution microscopy techniques, finding the structure of periodically reconstructed semiconductor surfaces was not posed as a problem of stochastic optimization until recently, when we have shown that high-index semiconductor surfaces can possess a rather large number of local minima with such low surface energies that the identification of the global minimum becomes problematic. We have therefore set out to develop global optimization methods for systems other than clusters, focusing on periodic systems in one- and two- dimensions as such systems currently occupy a central place in the field of nanoscience. In this article, we review some of our recent work on global optimization methods (the parallel-tempering Monte Carlo method and the genetic algorithm) and show examples/results from two main problem categories: (1) the two-dimensional problem of determining the atomic configuration of clean semiconductor surfaces, and (2) finding the structure of freestanding nanowires. While focused on mainly on atomic structure, our account will show examples of how these development efforts contributed to elucidating several physical problems and we will attempt to make a case for widespread use of these methods for structural problems in one and two dimensions.

Coffee break

Wednesday afternoon, 19 September, 15:30

Joint Poster Session (Part II)

Poster Award Ceremony

Wednesday afternoon, 19 September, 15:50

Thursday, 20 September

Structure & Properties 1

Intelligent systems for studies of structures, microstructures and nanostructures

Thursday morning, 20 September, 9:00

9:00

Invited

In search of key structures (not global minima)

Scott M. Woodley, Richard A. Catlow

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Structures of inorganic compounds are sought from knowledge of the constituent atoms and how they interact. A global optimisation technique, based on the principles of evolution, has been refined for the prediction of meta-stable solutions, which contain desired structural features. In the case of crystal framework structures the microporous architecture is predefined through the use of exclusion zones – regions within the unit cell, from where the constituent atoms are repelled. In the case of small ZnS particles, to bias the search away from bubble-

like structures (which are typically found) towards bulk-like configurations, a characteristic structural fragment is fixed at the centre of the container. During the search the internal energy, which is based on the Born, or rigid ion, model of a solid, is used as the cost function. We present details of our implementation within the computational package GULP and show highlights from recent applications.

9:45

Invited

Calibration and evaluation of a combined fracture model for the microvoid formation that competes with shear in the polycrystalline microstructure by means of evolutionary algorithms

Jacek Jackiewicz

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Changes of stress or strain ratios during an operation can have an effect on cracking during metalworking processes. Therefore, there is a substantive interest of the aluminum and steel industries of the automotive and aviation manufacturing sectors in numerical simulations of fracture processes of typical structural materials. The motivation of the present contribution is to establish a detailed calibration procedure for a combined fracture model for the microvoid formation that competes with shear in the polycrystalline microstructure. The combined fracture model is hierarchically arranged according to the structure of the material. The established model is related to the common fracture criteria. Therefore, the various competitive physical modes of fracture can be taken into consideration (i.e. (a) shear fracture, (b) material interface decohesion, (c) reduction in material strength caused by the presence of the tensile hydrostatic stress that may lead to the brittle mode of fracture or to the ductile damage evolution), from which the only one with the greatest fracture likelihood determines the overall fracture limit. A set of experimental tests is taken as a reference to computer simulations, which have been carried out by means of evolutionary algorithms. The applied and developed evolutionary algorithms well match to computer simulations of this kind because the members of a population of individuals, which represent identifying material parameters of the combined fracture model, adapt to its environment in order to find the fracture mode with the greatest likelihood, after many generations. In other words, the members of the population of the identifying material parameters tend to disappear if they cannot adjust, but the others, which can adjust well enough, will live to reproduce, and in consequence they will give better adjustable parameters of the model.

10:15

Oral

Genetic algorithm based search of parameters for fabrication of uniform porous silicon nanostructure

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The promotion of silicon from being the key substrate material for microelectronic devices to a potential light emitter has emerged as a consequence of the possibility to reduce its dimensionality by different techniques. In this regard nanostructured porous silicon (PS) drew formidable attention because of its strong room temperature photoluminescence (PL) in the visible region and due to its very simple and

Si-IC technology compatible fabrication process. However, fabrication of PS with uniform pore and particle size distribution, which is essential for device application, is still an unresolved problem. We report a technique for non destructive estimation of porosity of the PS samples by measuring voltage across the electrodes of the electrochemical formation bath. A uniformity factor was defined in terms of formation current density (J), critical current density (J_{PS}) and measured porosity (P_m). The factors affecting P_m was linked to the voltage across the electrodes using Bruggeman's effective medium approximation (EMA) that included electrical conductivities of the electrolyte and the sample. An attempt was made to optimally design a set of parameters that can potentially produce PS with narrow band pore and particle sizes. The sensitive and nonlinear dependence of PS morphology on the formation and post-formation parameters and their wide range demanded a non-classical optimization technique. A multivariate, genetic algorithm (GA) based optimization of the uniformity factor was found suitable for determination of the range of the controllable variables and to develop a guide strategy for experiments. A series of experiments were performed on p-type Si substrates using the GA evolved parameters. Porosity was measured by conventional destructive and developed nondestructive method. The results provided important insight in designing uniform PS nanostructures.

Coffee break

Thursday morning, 20 September, 10:30

Nanomaterials and nanostructures 2

Optimisation of clusters

Thursday morning, 20 September, 11:00

11:00 Invited

Global structure optimization of atomic and molecular clusters: evolutionary strategies and results

Bernd Hartke

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Finding global minimum energy structures of clusters is an NP-hard problem, beyond current capabilities of deterministic algorithms. With stochastic heuristics (combining evolutionary algorithms with local search and additional structural selectors) it is possible to find minimum energy structures for clusters of relevant sizes efficiently.

Using these methods, we have arrived at unbiased minimum energy structures for alkali cation microhydration clusters. Simulations of their infrared spectra are in excellent agreement with experiment. Free and constrained molecular dynamics simulations supply additional stability information, leading to a new hypothesis on the reason for magic numbers in these systems.

Switching from the standard generational scheme of evolutionary algorithms to a flexible pool model made it possible for us to generate a massively parallel implementation without any serial bottlenecks. With this implementation, we are currently searching for the onset of ice cores in pure water clusters.

In collaboration with Stefan Bromley (Univ of Barcelona) we could identify similarities and differences between water and silica clusters, and map out structural changes upon switching from isotropic to tetrahedral building blocks.

Additionally, we are working towards including external pressure into our global structure optimization, modeling clathrate hydrate cages of

methan and carbon dioxide, in the presence of impurities. This line of research supports experimental studies of marine carbon dioxide sequestration scenarios, as part of the excellence cluster "Future Ocean" at the University of Kiel.

11:45 Invited

Basin hopping and the global optimisation of clusters

Mark A. Miller

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Basin hopping is a general approach to the global optimisation of high-dimensional continuous and differentiable functions. It falls into the category of methods in which the function to be optimised is transformed to make searching easier without affecting the solution. In basin hopping, the transformation maps the function onto a series of plateaus where the barriers between local minima have been removed [1]. In this talk I will review the early applications of basin hopping to the global optimisation of the potential energy of rare-gas clusters, illustrating the underlying reasons for its success [2]. I will also describe more recent work on clusters of model dipolar systems, which have revealed exotic ground state structures such as knots, links and coils [3].

[1] D. J. Wales and J. P. K. Doye; *J. Phys. Chem. A* **101** 5111 (1997)

[2] J. P. K. Doye, D. J. Wales and M. A. Miller; *J. Chem. Phys.* **109** 8143 (1998)

[3] M. A. Miller and D. J. Wales; *J. Phys. Chem. B* **109** 23109 (2005)

12:15 Oral

Genetic algorithm - Monte Carlo hybrid method for geometry optimization of atomic clusters

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An evolutionary type global optimization method for the geometries of atomic clusters has been developed and used for finding stable structures of carbon clusters as a test application. Carbon clusters have been chosen because of large barriers in the potential energy function which makes the optimization procedure harder. Simple optimization methods fail for the carbon clusters because of this property. Genetic algorithms (GAs) are known to be very powerful since they provide an efficient search of the configuration space. However, their power is still not enough when relatively larger carbon clusters are the subject. Combining GAs with a local optimization method reduces the search over all configuration space to a search over local minima and hence improves the efficiency of the GA. In this study, classical Monte Carlo (MC) method has been used as the local optimization between GA steps. MC optimization jumps over most of the local minima and this reduces the search space even further. Single parent GA has been preferred and a fitness based selection mechanism has been applied. Total potential energy of the cluster has been used as the fitness function. A geometrical mutation operation which rotates half of the cluster by an arbitrary angle has been applied. A shrinking operation has also been applied by multiplying all the atomic coordinates by a coefficient less than one, causing the MC local optimization to be an inflationary motion and this increases the efficiency of the method for cage like carbon clusters. Results obtained by this GA - MC hybrid method have been compared with available results in the literature and reliability

of the method has been justified for up to 38 atoms. This hybrid method has been parallelized by distributing individuals to the available computing nodes and a high efficiency has been observed for this parallel running version.

Lunch break

Thursday afternoon, 20 September, 12:30

Materials, nanomaterials and their analysis

Modelling of useful materials including the nanomaterials

Thursday afternoon, 20 September, 14:00

14:00

Invited

Search for new phosphors for displays and lightings using genetic algorithms

Kee-Sun Sohn, Jong Ho Kwak, Yu Sun Jung, Chandramouli Kulshreshtha

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New phosphors for use in white light emitting diodes, back light unit of liquid crystal displays and plasma display panels are necessary since currently available phosphors do not meet the requirement from the industry. Oxide phosphors are environmentally friendly and also suitable for the high-throughput synthesis based on the solution combinatorial chemistry. In this end, genetic algorithm-assisted combinatorial chemistry (GACC) was implemented in order to search for new phosphors in multi-cation oxide systems, including Li_2O , Na_2O , K_2O , CaO , MgO , BaO , SrO , B_2O_3 , P_2O_5 , TiO_2 , SiO_2 , Gd_2O_3 , Ce_2O_3 , Tb_4O_7 , and Eu_2O_3 . The GACC process was followed by a series of fine-tuning processes based on conventional high throughput screening employing quaternary and ternary libraries, in order to pinpoint promising compositions. The combination of genetic algorithm and combinatorial chemistry was proven to enhance the searching efficiency significantly when used for phosphor screening processes. Four independent GACC implementations adopting different initial generations and their ensuing fine-tuning processes yielded four new phosphor candidates, such as $\text{Eu}_{0.14}\text{Mg}_{0.18}\text{Ca}_{0.07}\text{Ba}_{0.12}\text{B}_{0.17}\text{Si}_{0.32}\text{O}_d$, $(\text{Ca}_{0.72}\text{Sr}_{0.24}\text{Ba}_{0.04})_2\text{P}_2\text{O}_7:\text{Eu}^{2+}$, $(\text{Na}_{0.92}\text{Li}_{0.08})(\text{Y}_{0.8}\text{Gd}_{0.2})\text{TiO}_4:\text{Eu}^{3+}$, $\text{Tb}_{0.01}\text{Gd}_{0.02}\text{Ce}_{0.04}\text{B}_{0.1}\text{Si}_{0.83}\text{O}_d$, and . The former three phosphors were proven to be crystallized into a definite crystalline structure but the latter one was proven to be a glassy phase. The luminescent property and color chromaticity of these phosphors were all acceptable from the practical point of view.

14:30

Invited

Hybrid optimization approach to modelling of semiconductor nanostructure and devices

Yiming Li

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Model and optimal parameters have been at the heart of CAD tools for semiconductor technology over the past decades. Equivalent circuit models continuously play an active role in bridging the nanoscale device fabrication technology and IC design. As the mainstream CMOS technology is scaled into the nanoscale regime, hundreds of parameters have to be optimized and extracted for the corresponding model

equations. Conventional manual adjustment is a bottleneck for advanced semiconductor materials and nanodevices.

In this work, we present an intelligent parameter extraction technique for nanoscale semiconductor device and nanostructures; in particular, for sub-100 nm CMOS transistors. Based on the multiobjective distributed genetic algorithm, advanced numerical methods, adaptive neural network scheme, parallel computing techniques, and physical-based empirical rules, a hybrid approach, shown in Fig. 1, has been developed and implemented. Suppressing those difficulties of the conventional methods, the approach automatically mimics engineer's rules to extract and optimize a set of parameters, in a global sense, for tens of transistors on a single wafer simultaneously. For the 90/65 nm CMOS technology, this window-based prototype demonstrates its capability to automatically extract the optimal parameters from several sets of measured I-V curves of more than 16 fabricated MOSFETs. Preliminary performance of the prototype is examined, as shown in Fig. 2. Applications of this intelligent extraction system in DC, AC, and RF parameters optimization for, such as CMOS, TFT, HBT, SOI, OLED, and power JEFT, have also been achieved. Besides, optimal electronic structure design of InAs/GaAs semiconductor quantum dot, ring, and molecules is further advanced by integrating a simulation-based technique in the prototype for energy state calculation. Semiconductor scientists and engineers would be very interested in this system.

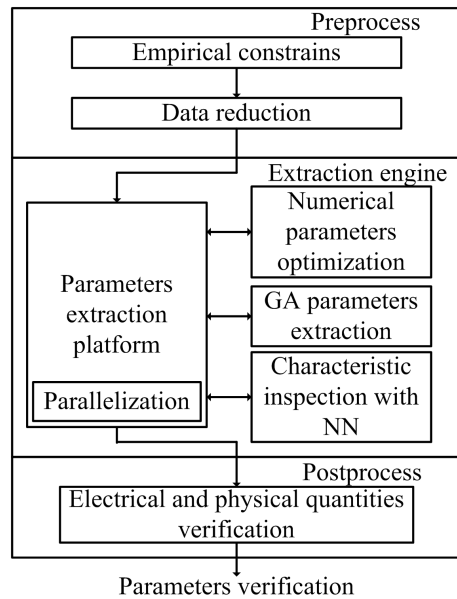


Fig. 1. An illustration of the hybrid optimization prototype.

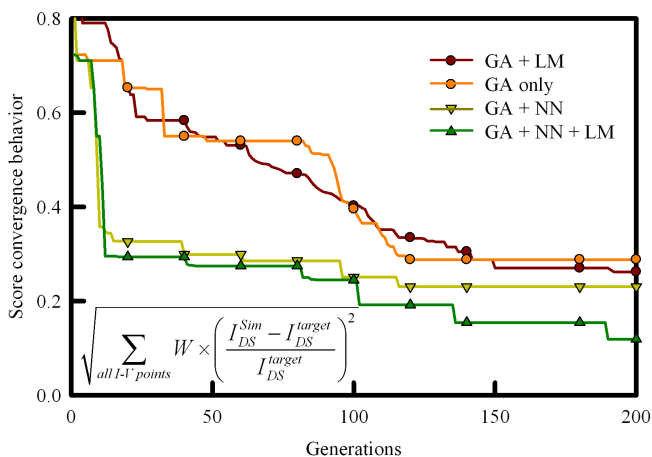


Fig. 2. The fitness score versus the number of generation among different approaches.

15:00 Invited

Genetic algorithms in texture analysis

Jacek Tarasiuk

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Texture of the polycrystal is one of the most important parameter characterizing the material. Many properties (mechanic, magnetic, electric) of the polycrystalline material depends not only on single crystal properties but also on distribution function of crystallographic orientations of grains in the sample, what is called texture. In some sense, the texture fulfill the weight factor roll for averaging properties of the material. Having texture function and tensor of single crystal properties it's easy to calculate tensor of any properties of the textured

material. The opposite case is extremely hard and in general exists no one procedure to determine texture fulfilling requested sample properties for a given single crystal properties. Till now, the new textures are projected using observations and trials and errors method. The paper will present general concept, based on Genetic Algorithms method, of completely automatic procedure realizing this goal. First the basic concepts and terms commonly used in texture analysis will be explained. Next two classical problems (pole figures and orientation distribution function recalculations) will be presented using classical and genetic algorithm approaches. The various possibilities of individuals representations will be discussed. Finally a simple case of prediction of a texture realizing requested property anisotropy will be presented.

Coffee break

Thursday afternoon, 20 September, 15:30

Method and Materials II

Genetic Algorithm for Materials Processing

Thursday afternoon, 20 September, 15:50

15:50

Invited

Application of Genetic Algorithm (GA) to estimate the rate parameters for reduction of iron ore-graphite composite pellets in a packed bed reactor.

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In the present article, Genetic Algorithm (GA) has been applied to estimate the rate parameters from experimental data during reduction of iron-ore/graphite composite pellet in a tailor made high temperature, high mass laboratory scale packed bed reactor. The reduction of iron-ore has been considered in three elementary steps, namely hematite to magnetite, magnetite to wustite and wustite to iron. Assuming all the above conversions follow the first order kinetics, the mass balance equations have been setup to define the rate of conversion of the various phases of oxide, as well as metallic iron. The unknown in the mass balance equations, namely three sets of frequency factors and values of activation energy, have been estimated by coupling the mass balance equations with the GA. The optimum values of the rate parameters have been obtained by minimizing the difference between the predicted and experimental degree of reduction. After optimization, a very close agreement between the experimental and predicted degree of reduction is obtained. The model has been utilized to calculate the evolution of various phases of oxide as well as metallic iron during reduction. Based on model predictions, it was found that hematite depletes fastest in the pellet, followed by magnetite, which passes through a maxima. Wustite initially build up and the gradually decreases while iron increases steadily. It is also seen that amount of metallic iron production increased with temperature. Temporal evolution of various oxide phases and metallic iron was also obtained through XRD and microstructural studies, which were found to be consistent with those predicted through kinetic model. The application of GA to estimate the rate parameters for iron ore reduction is unique and applied successfully for the first time.

Panel Discussion

Genetic Algorithms: Prospects for Improving Performance and Extending Applications

Thursday afternoon, 20 September, 16:20

Friday, 21 September**Genetic algorithms and alternatives: methods & applications**

Modelling of structure and properties

Friday morning, 21 September, 9:00

9:00

Invited

Alternative global search methods

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We will present an overview of global optimisation methods that differ from evolutionary approaches, and that can be also very efficient in broad ranges of applications. Improvements over the well-known simulated annealing algorithm will be discussed. Several finite-temperature simulation methods will be detailed in the light of the optimisation problem. Parallel tempering and flat-histogram methods, in particular, provide general and powerful schemes for sampling energy landscapes. Other approaches based on deformation of the energy surface will be presented, including the stochastic tunneling and basin-hopping approaches. Similarities and differences with genetic algorithms will be emphasized. Illustrations will be made for nanoscale and glassforming systems.

9:30

Invited

Cultural Differential Evolution: combined optimisation applied to structure solution from powder diffraction data

Maryjane Tremayne, Samantha Y. Chong

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Knowledge of the crystal structure of organic materials such as pharmaceuticals, pigments, agrochemicals, non-linear optics is essential if their solid-state properties are to be fully understood. However, when only polycrystalline samples of these materials are available, powder diffraction can be used as an alternative for structure determination. The most successful approach has been the development of direct space structure solution techniques in which a range of predicted models are compared with the experimental data using a global optimisation technique to locate the best crystal structure solution.

This presentation will focus on our recent work on the development and application of the Cultural Differential Evolution (CDE) technique, combining the traditional biological dictates of mating, mutation and natural selection in the Differential Evolution algorithm (DE) with an approach that models human social behaviour or cultural selection.

The DE method is a relatively new evolutionary algorithm that follows similar principles to conventional genetic algorithms, but differs in the

execution of these processes. Our implementation of the Cultural Differential Evolution algorithm uses ‘cultural’ behaviour – in this case, the distribution in values of structural parameters in each generation – to guide and enhance the DE process through the use of dynamic DE boundaries (i.e. constraints) re-defined at each generation. This ‘dual inheritance system’ uses social factors to guide and speed-up evolution but natural biological selection to drive the optimisation process. Our results show an average 40% improvement in terms of the efficiency of a structure solution calculation, in *all* test cases, over the DE method alone, demonstrating a simple ‘real-world’ implementation of a combined cultural and biological evolutionary approach that has potential applications in many other optimisation problems in materials science.

10:00

Oral

Modeling of recrystallization in cold rolled copper using inverse cellular automata and genetic algorithms

Sudipto Ghosh, Prashant Gabane, Anindya Bose, Nirupam Chakraborti

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An inverse modeling approach has been used in this work to model the process of recrystallization using genetically evolving cellular automata (CA). After formulating a CA model for primary static recrystallization, differential evolution (DE), a real coded variant of genetic algorithms, has been used to search for the values of nucleation rate and a constant factor in growth rate. These parameters are difficult to determine experimentally. This provided an acceptable matching between the simulated and experimentally observed values of average grain size and aspect ratio in the microstructure. Here, a realistic initial microstructure of cold rolled copper with statistically similar parameters as the experimental ones has been generated using a simple inverse CA coupled to DE. Dislocation density has been seeded in the CA cells of the initial microstructure as per the experimental observations on the variation of hardness in the mesoscopic scale. Each CA cell was assigned a nucleation probability based on the dislocation density value seeded in it. Recovery phenomenon has been incorporated by decreasing the dislocation density with time. The decrease in dislocation density was incorporated based on the experimental data variation in hardness with annealing time, at temperatures below the recrystallization temperatures. A good match could be found between the simulated and experimental parameters of the microstructure. Two simulated microstructures are shown in Figures 1 and 2.

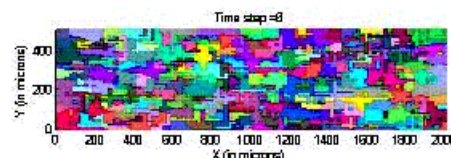


Fig. 1 At time step =1, Nucleation starts.

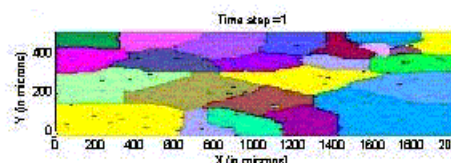


Fig. 2. At time step =6, Recrystallization partly over

Coffee break

Friday morning, 21 September, 10:30

Structure and properties 2*Genetic algorithms for understanding materials structure and properties*

Friday morning, 21 September, 11:00

11:00

Oral

On the definition of variable-space in direct-space structure solution from powder diffraction data using Genetic AlgorithmsZhongfu Zhou, Kenneth Harris*Cardiff University, School of Chemistry, Park Place, Cardiff CF103AT, United Kingdom**e-mail: zhouz5@cardiff.ac.uk*

Structure determination of organic molecular solids from powder X-ray diffraction data is nowadays carried out widely, in particular using the "direct-space strategy" for structure solution [1]. In this approach, the structure solution process involves exploring a powder-profile R-factor hypersurface $R_{wp}(\mathbf{\Gamma})$ to find the global minimum, where $\mathbf{\Gamma}$ represents the set of variables that define trial structures. In principle, any technique for global optimization may be used, and our own current work in this field is focused on the use of a Genetic Algorithm (GA) [2], implemented in the program EAGER [3]. Conventionally, the structural variables in the set $\mathbf{\Gamma}$ comprise, for each molecule in the asymmetric unit, the position $\{x, y, z\}$ and orientation $\{\theta, \phi, \psi\}$ of the whole molecule, and a set of n variable torsion angles $\{\tau_1, \tau_2, \dots, \tau_n\}$. Hitherto, all implementations of direct-space techniques for powder structure solution have defined molecular structures in terms of the variables in the set $\mathbf{\Gamma}$ given above, with each molecule in the asymmetric unit defined as a complete molecule. This approach is challenged here by an alternative definition of variable-space in which the molecule is broken into two or more smaller fragments by breakage of bonds, and which is demonstrated to be strikingly successful in solving the structures of conformationally complex molecules [4]. Here we assess the question of how the definition of variable space controls the evolutionary progress of GA calculations in this field.

[1] K.D.M. Harris, M. Tremayne, P. Lightfoot, P.G. Bruce, *J. Am. Chem. Soc.* **1994**, *116*, 3543

[2] K.D.M. Harris, S. Habershon, E.Y. Cheung, R.L. Johnston, *Z. Kristallogr.* **2004**, *219*, 838

[3] S. Habershon, Z. Zhou, G.W. Turner, B.M. Kariuki, E.Y. Cheung, A.J. Hanson, E. Tedesco, R.L. Johnston, K.D.M. Harris, *EAGER*, Cardiff University and University of Birmingham

[4] Z. Zhou, V. Siegler, K.D.M. Harris, E.Y. Cheung, S. Habershon, R.L. Johnston, *ChemPhysChem* **2007**, *8*, 650

11:15

Oral

Intelligent optical proximity correction using a model-based genetic algorithmYiming Li¹, Shao-Ming Yu², Yih-Lang Li²

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Optical lithography is one of the key technologies in semiconductor material and device fabrications. It is the process to transfer the layouts of desired pattern onto the wafers. However, the exposure on wafer has distortions due to the proximity effects. As the minimum feature sizes of explored samples continue to shrink, the mismatch between the pattern and the actual result on wafer is significant. Correction of mask patterns between the sample and post exposure result is thus necessary. Optical proximity correction (OPC) is the process of modifying the polygons to compensate for the non-ideal properties of the lithography process. Given the shapes desired on the wafer, the mask is modified to improve the reproduction of the critical geometry. In this work, we propose an intelligent OPC technique for process distortion compensation of layout mask. The proposed system consists of three parts: the pre-process, the OPC engine, and the post-process. During the pre-process, the partition analyzer will analysis all patterns and then divided them into many segments. Secondly, the OPC module is applied to correct the mask. The intelligent module searches the whole problem domain to find out the best combination of the mask shape by the genetic algorithm (GA). The corrected mask is verified by performing lithographic simulation to get the error norm between exposed result and desired layout. Finally, the mask verification is conducted in the post-process. To perform the mask correction in sub-wavelength era, the developed intelligent OPC technique integrates the GA and the model-based technique. We also propose a specific parameter extraction strategy for the OPC problem. Accuracy and computational efficiency of the methods are justified by a series testing and comparison between fundamental patterns and experiment data. We believe it is a constructive approach to advanced computer aided design for fabrication of semiconductor material, device, and integrated circuit.

11:30

Oral

Genetic algorithm based optimization for multi-physical properties of high strength steel through hybridization of neural network and desirability functionPrasun Das¹, Sandip Mukherjee¹, Subhas Ganguly², Bidyut K. Bhattacharyay³, Shubhabrata Datta²

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A genetic algorithm (GA) based optimization of the composite desirability of the tensile properties of thermomechanically processed steel plates has been done. The quality of a steel-rolled product depends on its characteristics like tensile strength; yield strength; elongation and so on so forth. The concept of desirability scale arises when there is a need to combine the magnitude of several characteristics to a dimensionless scale (say, d). This desirability function is so constructed that

any property value of a product is mapped between zero and one. Any value of d between zero and one gives an opportunity to improve the product quality depending on the closeness to these extreme two points. To provide a simultaneous balancing among physical properties of steel, viz. tensile strength, yield strength and elongation, a composite desirability scale (denoted by D) that represents average desirability is computed based on individual desirability values. The distribution of D is kept doubly bounded between zero and one to interpret the improvement aspect of product properties. The optimized solution of D provides a balance between multi-physical properties of steel under investigation. A neural network model has been developed to correlate between the tensile properties of steel and finally the desirability of the steel with its chemistry and several other rolling parameters in operation. Then, the model has been used for optimization of steel properties through the composite desirability by using GA. The GA technique, based on **population search technique**, has also been used in order to obtain the multiple feasible and implementable sets of solutions for the decision maker. The role of the variables on the work hardening behaviour of steel is also studied by varying the desirability function of yield strength and tensile strength. The results are validated from the existing knowledge of physical metallurgy in general and strengthening mechanisms in particular.

11:45

Oral

Genetic algorithm based search on the role of variables in the work hardening process of multiphase steels

Subhas Ganguly¹, Shubhabrata Datta¹, Nirupam Chakraborti²

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Multiobjective optimizations of strength and ductility of multiphase steels are made using genetic algorithm (GA) to find the role of the composition and process variables in the complicated work hardening process of such steel. Multi-phase steels offer attractive combinations of strength and ductility as a result of the different mechanical properties of the microstructural components and their interactions, and steadily increasing its usage in the automotive industries. Among the multi-phase steels the concepts of work hardening are different in case of microalloyed steels and that in transformation induced plasticity (TRIP) aided steel. The microalloyed steels show a very high work hardening respond only at low deformation, which gets continuously reduced with higher strain, which is almost constant over a wide range of deformation in TRIP-aided steel, due to transformation of retained austenite to martensite. The optimization of the two conflicting objectives viz. strength and ductility was attempted through rigorous experimental investigations by the researchers. Neural network based computational models, which can describe the complex correlations between the decision parameters for processing and materials chemistry of such steels, were developed using published data and were used for fitness functions. Two individual strength ductility maximization problems of multi-criterion nature were formulated for both types of steels. GA was performed to both these problems separately. The non-dominated strength-ductility Pareto optimal fronts were developed and compared. The role of the decision variables that ultimately affects the final properties in Pareto optimally balanced strength and ductility, as well as their role for work hardening behavior were investigated for both materials.

Symposium Closing

Friday afternoon, 21 September, 12:00

Lunch break

Friday afternoon, 21 September, 12:30

Coffee break

Friday afternoon, 21 September, 15:30

Posters

Monday, 17 September

Joint Poster Session (Part I)

Monday & Wednesday

Monday afternoon, 17 September, 15:50

15:50

Poster

G1

Fitting of X-ray reflectivity from periodic thin film structures: Improved convergence properties in genetic algorithm by rotation of coordinates

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Novel genetic algorithm (GA) is developed for precise X-ray reflectivity (XRR) curve fitting. The algorithm does the mating procedure in rotated coordinate frame to reduce effect of interparameter dependencies, called genetic linkage. The convergence properties of the novel GA using a few different rotation techniques are compared to real-valued conventional (CGA). Realistic XRR curve fitting problem is used as a test case where the curves are computed based on a periodic nanolaminate model. The importance of the used model arises from the wide tunability of the properties of these artificial materials. The desired nanolaminate properties such as conductivity depend heavily on structural properties of repetitive individual layers but cannot be determined from XRR measurements with currently existing fitting algorithms. The determination of unknown structural properties can be carried out most precisely by using Parratt's recursive formalism combined with Nevot-Croce interfacial roughness approximation in a curve calculation. In this formalism, one recursion cycle contains the calculation of reflectivity coefficient based on the previous coefficient and the three parameters, thickness, mass density and roughness. The calculation begins from the bottom substrate and continues until the surface is met. When applied to nanolaminates, the formalism includes tens of tightly linked parameters but interparameter dependencies can be reduced by the rotation of coordinates. In this work, the rotation of coordinates to reduce genetic linkage is found to improve significantly the convergence properties of the genetic algorithm. The present algorithm allows more efficient determination of parameters from complex periodic layer structures.

15:50 Poster G2

Optimisation of parameters of a dynamic penalty function for a test example of powder pattern indexing

Wojciech Paszkowicz

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In global-optimisation tasks, trapping the population at local minima (LM), and long stagnation periods at each of minima may lead to difficulties in achieving a satisfactory convergence of the algorithm. Hard or soft dynamic penalty functions (PFs) can be built into the definition of the cost function in any evolutionary global-search algorithm in order to limit the search area and so to improve the convergence. The concept, kinds and use of PFs have been reviewed in detail in [1]. The PFs are built in the general utility genetic-algorithm packages such as LHG [2] or BASIC [3], and their use in specific problems has been reported e.g. in [4,5]. In general, the choice of PF parameter values should be influenced by the extremum shape and extent in the given class of optimisation tasks. In [5], the use of varying-in-time soft penalty has permitted to improve the convergence for a powder-pattern indexing (PPI) task; the concept from [5] will be presented here in more detail and the influence of PF parameters on the convergence will be discussed. The applied definition of the PF discourages the algorithm from exploration of the earlier found LMs, moreover, a list of such minima is created. The PF varies smoothly in order to prevent against the loss of genetic information at the stage of birth of the next generation. Its use leads to reduction of the time of stagnation at the given LM, enabling thus to find numerous LMs and to select the global one from among them. It will be shown for the PPI example studied, how the choice of the PF parameter values influences the opportunity of finding the global optimum in a short cpu time.

[1] C.A.C. Coello, Comp. Meth. Appl. Mech. Eng. 191 (2002) 1245-1287.

[2] R. Östermark, Eur. J. Operat. Res. 152 (2004) 195-214.

[3] E.G. Shopova, N.G. Valdieva-Bancheva, Comput. Chem. Eng. 30 (2006) 1293-1309.

[4] K. Wang, A. Salhi, E.S. Fraga, Chem. Eng. Process. 43 (2004) 663-675.

[5] W. Paszkowicz, Mater. Sci. Forum 228 (1996) 19-24.

15:50 Poster G3

Influence of temperature, pressure, magnetic field on the solid's structure by means of internal stresses

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In closed thermodynamics system the influence of any external thermodynamics force causes change of all thermodynamics parameters of the system [1], which within the linear approaching can be described by the generalized system of equations:

$$dx_i = M_{ij} dX_j \quad (1)$$

where dX_j is the increment of thermodynamics forces (temperature, magnetic field, stress $dT, dH, d\sigma$), dx_i is the change of thermodynamics

parameters of system (entropy, magnetized, deformation $dS, dJ, d\epsilon$), and M_{ij} is the matrix of thermodynamics coefficients, including the elastic modules of solid, coefficients of thermal expansion, magnetic receptivity, pyro- and piezomagnetic coefficients ($S_{\lambda\mu}, \alpha_i, \gamma_{ik}, \rho_i, b_{i\mu}$). In one's turn external influences cause the return reaction of the system. As a result own «internal thermodynamics forces» were activated in a body: internal stresses, temperature, own magnetic field. The internal stress state was selected from thermodynamic parameters as the most sensible parameter to structure of solid. Internal stresses is the integral feature of solid given by chemical bond and related to the location of electronic clouds of neighboring atoms in a lattice. These stresses are changed under external mechanical forces, heating and influence of the magnetic fields according to equation:

$$d\sigma_\lambda = s_{\lambda\mu} (\alpha_\mu dT + b_{k\mu} dH_k + s_{\mu\nu} d\sigma_\nu) \quad (2)$$

The value of internal stresses are enough great and achieve a few GPa (according to results [1,2]). Structural phase transition will be realized, when some critical value of internal stresses, being parameter of the material, is achieved. There are different ways to attain the critical value of stresses: by change of any parameters H , T and P according to (2) and results [1]. The analysis of experimental data [1] allowed to make a conclusion about the deciding role of internal stresses in the nature of phase transitions.

1. P.I. Polyakov, S.S. Kucherenko. MMM, 278, (2004), p. 138-155.

2. Table of Physical quantities. Ed. N.K. Kikoin, Moscow, (1976), - 1008 p.

15:50 Poster G4

Analytical and finite element modelling of an ohmic contact test structure for low specific contact resistivity

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Ohmic contacts in semiconductor devices are typically quantified by their contact resistance [R_c (Ω)]. In researching such contacts, the more appropriate parameter for comparing two-layer contacts is the geometry independent interfacial resistance parameter termed specific contact resistivity [ρ_c (Ωcm^2)]. Various test structures have been employed to determine ρ_c , and Cross Kelvin Resistor (CKR) test structures are most suitable for estimating low ρ_c values. The value determined by CKRs includes parasitic resistances that have been difficult to account for when ρ_c is small. In this paper, an analytical technique for determining the parasitic resistance for particular CKR designs is presented. Finite element modeling (FEM) and experimental results for metal to silicide contacts will be used to validate the analytical expressions; and silicides have been incorporated in the designs as they are used in CMOS technology and contribute to the low ρ_c values.

This paper describes an analytical model for circular contacts based on Bessel function expressions. Using several contacts of different diameter (d) with $d/w < 0.4$ (w is the width of the CKR arms), the parasitic resistance can be accurately accounted for by extrapolation of experimental data to $d/w \rightarrow 0$. This model assumes that the concentric equipotential contours around the contacts extend to the voltage tap. CKR structures with the same geometry were modeled using FEM and the results for both the analytical and FEM models were in agreement for ρ_c values as low as $1 \times 10^{-9} \Omega\text{cm}^2$. FEM contour plots show that the equipotentials are circular around the contact and the value on

the tap closely approximates to that obtained using the Bessel function expressions.

Experimental results for ρ_c will be presented for aluminium to titanium silicide contacts. These demonstrate that the technique is reliable for determining low values of ρ_c ; and a value of $8 \times 10^{-10} \Omega \text{cm}^2$ was obtained for these contacts using the proposed method.

15:50 Poster G5

Effect of modifiers on the ion exchange properties of borosilicate glasses

Jayshree Ramkumar, Vv Sudarsan, Rk Mishra, Aweshkumar Tyagi, Tulsii Mukherjee

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Borosilicate glasses with barium oxide modifier BBS $[(\text{Na}_2\text{O})_{0.143}(\text{B}_2\text{O}_3)_{0.268}(\text{SiO}_2)_{0.473}(\text{BaO})_{0.116}]$ and without modifier SBS $[(\text{Na}_2\text{O})_{0.162}(\text{B}_2\text{O}_3)_{0.303}(\text{SiO}_2)_{0.535}]$ were used for the uptake of uranyl ion. The amount of metal ion taken up is expressed in terms of Kd which is a ratio of concentration of metal ion in solid to that left behind in solution. variation of Kd value with metal ion concentration for both SBS and BBS glass samples shows that the presence of BaO modifier (in BBS sample) improves the efficiency of metal ion uptake to a great extent. The variation of equilibration time at a fixed uranyl ion concentration showed that the Kd values increased to a maximum value after 24 h. Therefore, all equilibrations were carried out for 24 h. It is seen that for uranyl ion, the Kd value increases a maximum at a concentration of 0.01 M (2380 ppm) and then decreases. This could be due to the fact that with increase in the sorbate concentration in solution, there is an increase in the amount left behind in solution after equilibration and this factor affects the value of Kd. The difference in ion exchange behaviour between the two glass samples could be due to the difference in the number of non-bridging oxygen atoms present as revealed by ^{11}B and ^{29}Si MAS NMR studies. ^{29}Si MAS NMR patterns revealed the addition of BaO as modifier (BBS) results in the increase in the concentration of the Q^3 structural units containing more non-bridging oxygen atoms. ^{11}B MAS NMR patterns revealed that BBS contain only one sharp peak around -22 ppm, characteristic of the tetrahedrally coordinated boron (BO_4^-) structural units whereas for SBS, in addition to the sharp peak at -22 ppm, a broad peak characteristic of trigonally coordinated boron (BO_3) structural units is observed. The (BO_4^-) structure is charge neutralized by Na^+ ion. From ^{11}B and ^{29}Si MAS NMR studies, it is established that increase in the Si-O-Na^+ and B-O-Na^+ results in the higher exchange capacity of BBS.

15:50 Poster G6

Genetic algorithm applications in calculation of the classical ground state configurations and management of the ab-initio LMTO package

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The 2D-dimensional ground-state configurations of the classical point-charge particles have been calculated using a new genetic-algorithm-based approach. The systems consist of a number of particles interacting through the Coulomb potential which are trapped by the parabolic confining potential. The structures obtained have confirmed the recent

Monte-Carlo findings, including the metastable states. The genetic algorithm approach follows the Holland scenario and finds the global minimum of the potential energy which corresponds to the optimal geometrical structure. The chromosomes are defined as the set of the Cartesian coordinates of all the particles. The problem is solved by using the following parameters: the size of population $S=200 \div 500$, and the number of generations $N_g=100000000$, and the probability of crossover $p_c=0.3 \div 0.7$ and probability of mutation $p_m=0.02 \div 0.15$. The numerical calculations are based on the flow-point coding.

We have worked out the program which finds the total energy of an electronic system by using the LMTO subroutines *lm.run* with the atomic radii found by the genetic algorithm. In this way the computer resources needed to run the TB LMTO code have been reduced in computations of the total energy requiring the interactive user-dependent mode. The LMTO programs are performed in the background and the disc space is reduced to that needed to store the corresponding *S* output files *LM* irrespective of the number of iterations. Results obtained have been illustrated for the ordered alloy Ni_2MnGa [1].

References

- [1] G. Kamieniarz et al. *Comp. Meth. Sci. Technol.* **13(1)**, 13, (2007).

Symposium H

Welcome

Motivation:

This E-MRS symposium is aimed to give an overview of the current status of optical and x-ray metrology for materials characterization and quality assurance of thin films, layer-structured materials, and one-dimensional nanomaterials, with a particular emphasis on state-of-the-art metrology. Two basic approaches will be emphasized. First of all, we will emphasize the trends and advances in the techniques of optical and X-ray metrology for thin film materials, nanowires and nanotubes; secondly, we will address the application to thin-layered, wide band gap nitrides and functional oxide films (high-k dielectrics, ferroelectrics, ZnO and novel p-type transparent conductive oxides), as well as nanotubes and nanowires based on such materials.

As manufacturing processes become more complicated, it is imperative to employ in-situ metrology; this is particularly true in the microelectronics and micro-systems industry, such as compound semiconductor electronics, photonics, MEMS and sensors. The need for improvement of optical and x-ray metrologies for microelectronics, optoelectronics and micro-systems is driven by the increasing diversity of materials used in the industry, the complex fabrication processes and the poorly understood structure-properties relationships. One main objectives of the symposium is to promote and encourage the interaction between academic and industrial research (instrument manufacture, IC and optoelectronics industry and materials suppliers) to address scientific and technological challenges associated with the improvement of standard analytical methods and characterization of newer techniques.

Among the various X-ray-based methods, standard X-ray diffractometry, X-ray reflectivity and diffuse scattering are acquiring an increasing relevance for characterization of materials and devices in academic and industrial laboratories, since these techniques are rapid, high resolution, non-destructive and non-contacting. In addition, the availability of high brilliance X-ray Synchrotron sources and the recent development of new X-ray scattering techniques offers new opportunities for non-destructive characterization of microstructures and the unique opportunity of characterization of micrometer-sized (or smaller) objects.

The current trends in optical metrology mainly concern spectroscopic ellipsometry (SE), polarisation and modulation spectroscopy, anisotropic reflectance and Scanning Near-Field Optical Microscopy (SNOM).

Both optical and X-ray related techniques have gained considerable interest in the last decade and are currently involved in the characterization of thin film materials and nanomaterials. In this symposium, these methods will be discussed with particular attention paid to their application, as well as their limitations and complementarities.

Topics:

- Trends and advances in optical and X-ray metrology for thin film materials (nitrides and functional oxides) qualification, for microelectronics, optoelectronics, and microsystems, including the application concerning the larger integration on Si.
- Trends and advances in optical and X-ray metrology for nanotubes and nanowires (nitrides and functional oxides) qualification, for microelectronics, optoelectronics, and microsystems
- Optical and X-ray characterization of novel nanomaterials and heterostructures including novel n-type and p-type transparent conductive

oxides, wide band gap nitrides, high-k materials and ferroelectrics, organic materials for OLED applications, GaN and diluted nitrides.

- Advances in numerical method-based procedures for X-ray reflectivity and diffraction data interpretation.
- Metrology physics - light/matter interaction, including interferometry, near field and transient based optical probing.
- Advances in the development of generalized ellipsometry and real-time ellipsometry, in modulation and anisotropic reflectance spectroscopy and in Scanning Near-Field Optical Microscopy (SNOM).
- Advances in X-ray diffractometry, X-ray diffuse scattering and reflectometry applied to thin-layered materials for microelectronics, optoelectronics and microsystems.
- Recent development, using X-ray Synchrotron sources, of new X-ray scattering techniques for non-destructive characterization of thin-layered materials and of micrometer-sized (or smaller) objects.
- Spatially resolved techniques for the investigation of small objects and micro-devices.

- Real time in situ diagnosis & control during epitaxial growth (stress & strain measurements, alloy composition & growth rate determination...) and/or complex stacked structure elaboration (e.g., metal/interface/dielectric/ interface/substrate).

- Characterization of III-N based optoelectronic devices (VCSEL, LED, QCL, OPO) and nitride-based transistors (HBT, HEMT).

- Characterization of microelectronic devices by scatterometry and polarimetric methods-determination of critical dimensions.

- Examination of critical dimensions in lithography using real time polarization optics.

Symposium H Workshop: “Novel p-type transparent conductive oxides”, organized in conjunction with the EU project NATCOFP6-511925

List of invited speakers/talks, additional invited talk may be selected from the outstanding submitted papers (to be confirmed):

V.Holy (Masaryk Univ., Czech Republic)

Dave Aspnes (NC State, USA)

A.Gibaud (Univ. du Maine, France)

Davor Balzar (NIST, USA)

John Budai (ORNL, USA)

U. Pietsh (Siegen University, Germany)

D. Lübbert (Forschungszentrum Karlsruhe, Germany)

J. Garnaes (Danish Fundamental Metrology, Denmark)

Tentative list of scientific committee members (to be confirmed)

B. Drevillon (Ecole Polytechnique, France), S. Logothetidis (Aristotle University of Thessaloniki, Greece), J. Hosea (Univ. Surrey, UK), J. Humlicek (Univ. Brno, Czech Republic), T. Lohner (Academy of Science, Hungary), N.Tomozeiu (Oce,Holland) T. Baumbach (FZK, Germany), M.Losurdo (IMIP-CNR, Italy), D.Chateigner (CRISMAT-ENSICAEN, France) M.Gartner (Romanian Academy, Romania), B. Servet (Thales, France), J. Misiewicz (Univ. Wroclaw, Poland), S.Bosch (Universitat de Barcelona, Spain), E. Zolotoyabko (Technion, Israel), Marie-Antoinette Poisson (Alcatel-Thales III-V lab), J. Bak-Misiuk (Polish Academy of Science)

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Programme

Monday, 17 September

Session I : Data analysis for spectroscopic ellipsometry

Monday afternoon, 17 September, 14:00

14:00	Invited

Survey of methods to characterize thin absorbing films with Spectroscopic Ellipsometry

James N. Hilfiker¹, Neha Singh¹, Diana Convey², Steven M. Smith², Harland G. Tompkins³

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Spectroscopic Ellipsometry (SE) is routinely used to measure thickness and optical constants of dielectric and semiconductor films. However, it is difficult to insure unique results for thin absorbing films, such as metals. SE methods to enable simultaneous determination of metal layer thickness and optical constants include multiple sample analysis, interference enhancement, optical constant parameterization, and

simultaneous analysis of SE and intensity-based optical measurements. Methods are tested on a series of Chromium thin films prepared with multiple thicknesses and multiple substrate types to allow direct comparison between methods.

14:35	Oral

Nanocrystals characterization in porous silicon using model dielectric function

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We used Adachi's model dielectric function (MDF) to characterize porous silicon thin films of systematically changing nanocrystal size. Since both the surface and interface roughnesses have to be taken into account, and the E_0 , E_1 , and E_2 critical point (CP) features are all described by a combination of several lineshapes (two-dimensional CP, excitonic, damped harmonic oscillator) with numerous parameters in each layer, the number of fitted parameters were reduced by parameter coupling and selecting sensitive parameters for the fit. The porous silicon layers were prepared by electrochemical etching of p-type single-crystal Si (c-Si) of varying dopant concentration resulting in systematically changing morphology and nanocrystal (wall) sizes in the range of 2-25 nm. The broadening parameters of the interband transitions

in the measured photon energy range correlate with the long-range order in the crystal, and, as a result, with the nanocrystal size. The advantage of this method over the robust and simple effective medium approximation (EMA) presented recently (Petrik et al., Appl. Surf. Sci.

253 (2006) 200) using a composition of voids and c-Si with a nanocrystalline Si reference is that the combined EMA+MDF multilayer method of this work provides a more detailed description of the material and layer structure. Error bars and parameter correlations have to be investigated thoroughly, because of the increased number of fitted parameters.

14:55	Oral

Optical characterization of HfO₂ by spectroscopic ellipsometry: dispersion models and direct data inversion

Jordi Sancho Parramon¹, Mircea Modreanu², Salvador Bosch³, Michel Stchakovsky⁴

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Hafnium oxide (HfO₂) has attracted much interest as high-k material of choice for gate oxide replacement in future CMOS technologies and for its use in optical coating technology. The determination of optical properties, like refractive index and band-gap is focus of intense research, since the optical constants of HfO₂ depend on the physical microstructure and the deposition methods and conditions. In particular, HfO₂ ultrathin films use to show significantly different properties than the bulk material.

In the present work the optical characterization of very thin HfO_2 films deposited by plasma ion assisted deposition and annealed at different temperatures is carried out. The characterization is performed using ellipsometric measurements in the spectral range from 1.5 to 8 eV and employing two different approaches: i) fitting of the data using a dispersion model to describe the optical constants of HfO_2 , and ii) the direct inversion of the ellipsometric data. In particular, two dispersion models are tested: Tauc-Lorentz and Cody-Lorentz. These models, intended to describe the optical interband transitions of amorphous materials, can be extended to include of sub-gap absorption in terms of an exponential Urbach tail. The validity of the models is analyzed according to the fitting quality and the confidence limits and correlation of the parameters defining the dispersion model. On the other hand, the direct data inversion is shown to be useful to provide additional information like the presence of sub-gap absorption and point out features associated to the crystallinity of the material.

15:15 Oral

Use of Gaussian Voigt oscillators to characterize microelectronics materials by Spectroscopic Ellipsometry.

Mickael Gilliot, Jean-Philippe Piel

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Complete understanding of ellipsometric data needs the use of a model of either the sample structure and the materials to be characterized within the layers, including the delicate step of choosing a good dispersion function to model these materials. Particular problems appear when modelling materials with large absorption bands where classical Lorentzian oscillators cannot fit experimental data. This is for example the case of materials which present transitions with large inhomogeneous broadening in the visible and infrared spectral range. Oscillators in the collection of nominally identical atoms may then have slightly different resonance frequency because, for example, of local surroundings local crystal structures, because of defects, dislocations or lattice impurities. We report on the use of a dispersion function to model materials with such non Lorentzian bands. This dispersion function is the correlation of a Lorentzian oscillator by a Gaussian distribution, also known as the Voigt function. For the same area Gaussian have lower peaks, larger FWHM but have a faster extinction and so a smaller background. The use of Voigt profiles allows to introduce alternate variations between Lorentzian (strongly homogeneous limit) and Gaussian (strongly inhomogeneous limit). We propose the computation of this function using the complex scaled error function also known as the Faddeeva function. It is shown that the use of such oscillators greatly helps in the fitting procedure by reducing the number of fitting parameters. This is illustrated by different examples concerning microelectronics materials in thin layers such as H-doped Silicon nitride (SiN), Polybenzoxazole (PBO) polymer dielectrics (PBO) or Fluorinated silicate glass FSG which belongs to the low k dielectrics family. We also propose an alternate formulation of this oscillator when the broadening is not symmetric with frequency.

Coffee break

Monday afternoon, 17 September, 15:30

Joint Poster Session (Part I)

Monday & Wednesday

Monday afternoon, 17 September, 15:50

Tuesday, 18 September

Session II : Advances in XRD characterization of small objects I

Tuesday morning, 18 September, 9:00

9:00 Invited

Structural characterization of semiconductor quantum dots by three-dimensional x-ray diffraction mapping in reciprocal space

Vaclav Holy¹, Cristian Mokuta², Kiran Mundboth², Julian Stangl³, Till H. Metzger², Günther Bauer³

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High-resolution x-ray diffraction is frequently used for the determination of positions, shapes, elastic strains, and indirectly also local chemical composition in semiconductor quantum dots. In these studies the reciprocal space mapping is usually confined to a single scattering plane in reciprocal space (perpendicular or parallel to the sample surface), so that a full characterization of the quantum dots is possible only if a certain dot symmetry is assumed. We present a new diffraction method yielding a full three-dimensional distribution of diffracted intensity in reciprocal space. The method was used for the investigation of a random array of SiGe quantum dots deposited by liquid-phase epitaxy on a Si (001) surface. Three-dimensional distributions of diffracted intensity were measured around various reciprocal lattice points and the measured data were compared with numerical simulations based on continuum elasticity and semikinematical scattering theory. From the comparison we could reconstruct a full three-dimensional image of the shape of an average quantum dot, as well as its local chemical composition, without any a-priori assumption about the dot symmetry.

9:35 Invited

Shape and stacking analysis of semiconductor nanowires using grazing-incidence diffraction and coherent scattering on single nanowires

Vincent Favre-Nicolin^{1,2}, Joël Eymery¹, Pascal Gentile¹, Robert Koester¹, Erik Bakkers³, Rienk Algra³, Thomas Mårtensson⁴, Lars Samuelson⁴, Ian Robinson⁵, Ross Harder^{5,6}

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Vertical semiconductor nanowires (NW) are used as building blocks for the development of new electronic and photonic devices. To achieve a suitable efficiency it is necessary to tune the structural properties of the wires, both individually and for the assembly of NW.

We have conducted a study of an assembly of InAs/InP NW: for these NW grown by Chemical Beam Epitaxy it is possible to separate the diffraction of the wires from that of the substrate and surface overgrowth using grazing incidence diffraction. By measuring several diffraction rods in the direction perpendicular to the surface of the sample it is possible to extract the different contributions to the diffraction, and analyse the wurtzite/zincblende stacking along the wires.[1]

However a complete analysis of NW structure cannot always be obtained by scattering from an assembly of wires, as the distribution of shape and size (diameter & length) will allow access only to the average properties of the wires. It is therefore necessary to conduct the analysis on single nanowires, which can be done using X-rays with the development of micro-focused (1-10 microns) coherent beam.

We will present the results of coherent diffraction on single NW:

- for GaP/GaAs NW the stacking faults induce phase shifts (of the scattered X-ray wave) along the wire for each fault. The resulting scattering can be analysed as the Fourier transform of the stacking faults and can be used to model the stacking along the wire

- for Si nanowires we obtained coherent diffraction images for NW with a diameter smaller than 100 nm. The recorded 3D diffraction exhibits a 6-fold symmetry which can be used to reconstruct the shape of the original wire.

We will discuss how coherent diffraction could be used for the reconstruction of the shape, strain and composition of single NW, particularly for vertical and radial heterostructures.

[1] J. Eymery et al., Nanoletters (submitted).

10:10

Oral

Neutron, X-ray and laser diffraction in inverse opal films

Alexander Sinitskii¹, Vera Abramova¹, Tatyana V. Laptinskaya¹, Kirill Napolskii¹, Sergey O. Klimonsky¹, Sergei Grigoriev², Natalia Grigorieva³

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Films with 3D-ordered macroporous structures attract a growing attention because of their wide applications in separations, catalysis, and optical information processing. A number of methods have been developed for their preparation, among which templating of opal films provides a simple and efficient path. Recently, thus prepared porous films, also referred to as inverse opals, were extensively studied as photonic crystals, which are expected to revolutionize modern optoelectronics resulting in fabrication of high-performance light emitting diodes and lasers, optical waveguides with sharp bends and all-optical microchips. Typical tools for study of inverse opals include scanning electron microscopy and optical spectroscopy. However, even though inverse opals possess highly-ordered microstructure, they were scarcely investigated by diffraction methods.

We report a complex diffraction study of inverse opals based on different materials including metals (Ni and Co) and oxides (TiO₂, WO₃ and Mn₂O₃). For the sample preparation we fabricated colloidal crystals by self-assembly of polystyrene microspheres onto vertical substrates. Inverse opals were synthesized by infiltration of the voids of opal films with a desirable material (we employed sol-gel technique for synthesis of oxide materials, while electrodeposition was used in case of metals) and removal of polymer microspheres by either annealing or dissolution in toluene.

We discuss the results of diffraction experiments on inverse opals using different types of radiation, including neutrons, X-rays and visible photons. A very special attention was paid to angle-dependent laser diffraction experiments, which provide information on layer stacking in close-packed structure of inverse opal, which is inaccessible by conventional microscopy methods. In case of magnetic films (Ni and Co) we performed special neutron experiments, in which we monitored the intensity of diffraction spots as a function of applied magnetic field.

Coffee break

Tuesday morning, 18 September, 10:35

Session III : Optical characterization of small objects

Tuesday morning, 18 September, 11:00

11:00

Invited

Fast hybrid scalar and Fourier modal method for scatterometry with least square measurements validation

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The range of validity and the accuracy of scalar analysis with renormalization for periodic diffractive phase elements is evaluated by a comparison with the Fourier modal method. It is found that the error of renormalized scalar diffraction theory is small for normal incident light with wavelengths in the range from 350 nm to 1000 nm and for height pitch ratios less than 1. The scalar method is implemented in a hybrid scalar-vector diffraction method that reduces the solving time for one-dimensional gratings by a factor up to 100. The scatterometry parameters, such as height, fill factor and wall angles, are obtained using a generalized least square method. The input to this least squares method is 1) best estimates and the associated covariance matrix of all measured quantities, including influence quantities, and 2) a measurement model describing all known physical relations between the measured quantities and the scatterometry parameters. The output includes best estimates and the associated covariance matrix of the scatterometry parameters.

11:35

Oral

Nanoscale resolution sub-surface fluorescence imaging with aperture Scanning Near-Field Optical Microscopy

F. Bonaccorso^{1,4}, A. Mazzaglia², L. Monsù Scolaro³, A. Valerio⁵, A. Rencurosi⁶, L. Lay⁵, U. Giorgianni⁴, N. Micali¹, V. Villari¹, O. M. Maragò¹, Pietro G. Gucciardi¹

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In this paper, we demonstrate the capability of aperture scanning near-field optical microscopy (SNOM) to attain topography-free sub-surface

fluorescence imaging with sub-diffraction resolution on supramolecular aggregates of amphiphilic β -cyclodextrins (CDs) and porphyrins. The system is prepared by mixing CD covalently tagged with a dansyl fluorescent group (SC6CDDns) and CD targeted by receptor specific group (SC6CDGal). Static and Dynamic Light Scattering show that the formed nanoaggregate is constituted by both SC6CDDns and SC6CDGal species. The mixed nanoaggregates (MixCDDansGal) can encapsulate cationic porphyrins (H_2T_4) in solution as shown by bathochromic shift of H_2T_4 Soret band in UV-Visible spectra. Solutions of MixCDDansGal/ H_2T_4 at 10:1 molar ratio have been cast onto glass substrates, evaporated overnight and analysed by SNOM. We have imaged localized porphyrin aggregates, fluorescing at $\lambda_{\text{fluo}} \sim 660$ nm, below the surface of mixed porphyrin-cyclodextrin micrometric vesicles, with 150 nm spatial resolution. Similarly, nano-bubbles have been localized below the surface of thin films of amphiphilic β -cyclodextrin, marked with dansyl fluorescent molecules ($\lambda_{\text{fluo}} > 500$ nm). Both commercial and homemade optical fiber probes have been used. SNOM subsurface imaging opens up fascinating possibilities for chemical and structural analysis at the nanometer scale of nanocomposite materials, bio-recognition of carriers including drugs and specific receptor protein or single fluorescent molecules embedded under transparent membranes.

11:55

Oral

FTIR Characterization of Photonic Bands in 2D Silicon Photonic Crystals

Zdenek Kral¹, Josep Ferre-Borrull¹, Trifon Trifonov², Lluís F. Marsal¹, Angel Rodriguez², Josep Pallares¹, Ramon Alcubilla²

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Photonic band gap materials, also known as photonic crystals, are very promising optical materials because of their particular properties to interact with light. They consist of materials with a periodical variation of their refractive index in one, two or three spatial dimensions and can be seen as a generalization of the well-known multilayer coatings. The most relevant result of this periodic variation is that the propagating photons must obey special dispersion relations, very different from those found in common materials. Since these dispersion relations are analogous to the bands in electronic materials, they are known as photonic bands. Photonic crystals are manufactured by several methods, depending on their dimensionality and on the used base materials. In this work we will focus on two-dimensional photonic crystals based on macroporous silicon made by electrochemical etching of silicon wafers previously patterned with a standard photolithography method.

In order to characterize these materials, it is important to be able to measure their photonic bands. We have developed a characterization procedure for these photonic crystals based on angle-resolved spectroscopic reflectometry and on the comparison of the measurement with the corresponding model simulation. The specific lattice constants of the studied photonic crystals range from 4 to 11 microns, which results in photonic bands in the spectral range of the Mid-IR. We have adapted the existing photonic band measurement techniques to this spectral range using a FTIR spectrometer equipped with a reflection attachment. Furthermore, we have implemented numerically the model for the interaction of the incident light with the photonic structures and we will show the results of the comparison between measurement and simulation. Finally, we will demonstrate that the same technique can be ex-

tended to polarimetric measurements to improve the recognition of the photonic bands.

Lunch break

Tuesday afternoon, 18 September, 12:15

Session IV : Advances in XRD characterization of small objects II-Spatially resolved techniques

Tuesday afternoon, 18 September, 14:00

14:00

Invited

Polychromatic X-ray micro- and nanodiffraction for spatially-resolved structural studies

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The availability of intense, focused synchrotron x-ray microbeams has enabled new techniques for materials investigations with sub-micron spatial resolution. The scanning microbeam setup we have developed at the UNI-XOR beamline at the Advanced Photon Source (APS) is versatile in that it provides 3D scans (including depth resolution), and can alternate between polychromatic- and monochromatic-beam modes. Focusing in both modes uses elliptical Kirkpatrick-Baez (K-B) reflecting mirrors. Beam diameters of ~ 400 nm FWHM are now routine, and 90 nm focus has been demonstrated. In white-beam mode, a CCD detector records a complete Laue diffraction pattern, which is analyzed with an automated indexing program. These diffraction patterns provide real-space maps of the local lattice structure, crystal orientation, grain morphology, and strain tensor. In monochromatic mode, individual diffraction peaks are monitored while scanning the x-ray energy to provide absolute lattice parameter measurements.

This presentation will illustrate the application of polychromatic scanning x-ray microscopy with examples from 1D, 2D and 3D materials systems. Using 1D ZnO nanorods, we have mapped the structure, orientation and lattice perfection along individual nanostructures as small as 200 nm diameter. Using deposited 2D thin films, x-rays studies have revealed the mechanisms for local crystallographic tilting in two different systems: epitaxial cerium oxide films grown on textured Ni substrates for superconducting applications, and pendeo-epitaxial GaN films grown on Si substrates. 3D materials investigations have included studies of thermal grain growth in polycrystalline aluminum and phase separation in multiferroic (Eu,Y)MnO₃ manganite single-crystals.

Support by DOE Office of Science, Division of Materials Sciences under contract with ORNL, managed by UT-Battelle, LLC; XOR/UNI beamline supported by ORNL, UIUC-MRL, NIST, UOP and DOE-BES; APS supported by DOE.

14:35

Invited

X-ray microdiffraction imaging of epitaxially laterally overgrown Gallium Nitride structures

Daniel Luebbert^{1,2}, tilo baumbach¹, Vaclav Holy^{3,4}, P. Mikulik⁴, L. Helfen^{1,5}, Muthaffar Elyyan¹, P. Pernot^{1,5}, S. Keller⁶, S. P. Den-Baars⁶, J. S. Speck⁶

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We present recent advances in X-ray microdiffraction imaging as a tool for studying the crystal lattice properties of epitaxially laterally overgrown (ELO) semiconductor structures. The method of Rocking Curve Imaging (RCI) permits us to quantitatively investigate the local crystal quality and the nature of crystal defects, while simultaneously imaging their spatial distribution with micrometer resolution. This technique of full-field imaging (as opposed to scanning methods) proceeds by recording sequences of X-ray diffraction topographs. The small angular source sizes available at synchrotron sources make it possible to simultaneously achieve high spatial and angular resolution. As a result, the RCI technique offers high sensitivity to many kinds of distortions of the crystal lattice. It can be used to reveal, among others, lattice plane curvature, strain fields, grain boundaries, crystallite tilts, dislocations and other defects as well as the regularity of the local crystal lattice.

The talk will focus on recent applications to Gallium Nitride (GaN) ELO structures. Here, the RCI method was used to distinguish between the ELO wing and window regions, to reveal wing tilt and the formation of subgrains in the ELO wings, and to monitor changes in the local crystal quality. Information on ELO wing tilt can be obtained from sample volumes as small as $(2\text{ }\mu\text{m})^3$. Wing tilt is found to fluctuate considerably on a local scale (between adjacent ELO periods), in addition to more long-range variations of lattice orientation (macroscopic curvature). Advanced data analysis is used to visualize wing tilt in three dimensions as well as to compare lattice perfection in the different sample regions in a statistically representative way. Finally, combinations with the theory of dislocations and numerical simulations allow us to assess dislocation densities on a local basis.

15:10

Oral

Production of metallic X-ray capillaries with nanometer-rough reflecting surfaces

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The X-ray guiding monicapillaries belong to a few only types of the X-ray concentrators. Their quality depends essentially on the controlled shape of the walls, wall material and the quality of the internal reflecting walls. The simple analytical ray tracing of the photon trajectories in X-ray capillaries is described. It establishes the parameters of capillaries and conditions to guide the X-ray photons towards the semi-focus. The conditions for the roughness of the mirroring surfaces are determined to allow efficient reflection of X-rays – it means the range of rms should be below 5 nm. The capillaries with the walls made of selected metals are preferred, due to some advantages: better control of the capillary shape and the straightness of the main axis, greater critical reflection angles. The metallic capillaries with gold and rhodium internal surfaces are manufactured according to the galvanoplastic method elaborated at KUL. The internal shape of capillaries was controlled with the laser scan micrometer. The measurements gave the estimation of the long-distance shape distortions, termed a roughness correlation length. The metallic capillaries produced in our laboratory had the long-distance distortions of the order of 60-200 nm. The transmission of the laser light along the main axis of the capillary and registration of the escaped photons on the CCD screen enabled the estimation of the symmetry of transverse cross sections of capillaries. The symmetry was circular. The field emission scanning electron microscope (FESEM) and atomic force microscope (AFM) studies gave the images of the internal surface of the capillary. The AFM measurements determined the surface roughness (rms) and the best gold internal surfaces had the rms in 2 nm range, as measured by AFM on 20 mm x 20 mm areas. The roughness is controlled by changing the composition of the electroplating bath, electropolishing conditions and the measurements of the surface strains.

Coffee break

Tuesday afternoon, 18 September, 15:30

Session V : Modulation Spectroscopy

Tuesday afternoon, 18 September, 15:50

15:50

Invited

Advances in contactless forms of modulation spectroscopy for probing the optical properties of nano-scale objects

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Contactless modulated reflectivity spectroscopy, in forms of photorelectance (PR) and contactless electroreflectance (CER), is presented as a useful tool for the investigation of semiconductor structures including nanometer-sized quasi 0D objects (like quantum dots or quantum dashes) obtained by self-assembly. There are discussed their advantages with respect to a certain kind of structures and including the problems of spurious background signals, below-band-gap oscillations and modulation mechanisms. Finally, recent results regarding the modulation spectroscopy on several types of novel 0D-like structures will be presented and it will concern the following: (i) modulation spectroscopy followed 2D-3D growth transition: wetting layer thickness determination and detection of the quantum dots formation in Stranski-Krastanow growth mode in different strain regimes for InGaAs/GaAs QDs; (ii) intermixing processes in self-assembled InAs/GaAs QD structures; (iii) excited states spectrum for new material/design quantum-dot-like structures (e.g. columnar InGaAs/GaAs QDs or InAs quantum dashes on InP substrate with different kind of quaternary barriers); (iv)

electronic properties of semiconductor laser structures: different material quantum-dot-based lasers for fiber telecommunication range: InAs/GaAs dots for 1.3-1.55 μm ; InAs/InP dashes for 1.55 μm and longer wavelengths; quantum dot (or dash) – quantum well tunnel injection systems based on GaAs or InP substrates; (v) modulation response intensity versus the nano-object size in different quantization regimes: for different size (volume) InAs/GaAs QDs or InAs/InP QDashes; (vi) modulation spectroscopy in high spatial resolution mode (microphotorefectance on plane QD structures or single micrometer-sized pillar microresonators).

16:25

Invited

Photorefectance in the mid-infrared : a study of the bandgaps and spin-orbit splittings of ternary and pentenary InAsSb-based alloys

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Recent interest in mid-infrared (MIR, 2-5 μm) light emitters and detectors is driven by the wide range of potential applications in this spectral region. However, losses by non-radiative Auger recombination paths result in low quantum efficiencies so that many operating devices have to be cooled. Auger rates are sensitive to the fundamental band gap energy, E_0 and the spin-orbit splitting energy, D_0 . However, the band-structures of many narrow gap materials have not been very well characterised which makes engineering their properties very difficult. Part of the reason for this is that modulation spectroscopies such as photo-modulated reflectance (PR), the key non-destructive tool for optical characterisation of semiconductor band-structures, becomes increasingly difficult as one attempts to look further out into the IR. Here, we report on MIR PR of E_0 and E_0+D_0 features in high-quality, InAs-rich InAsSb and GaInAsSb samples as functions of both temperature and antimony contents $\leq 22.5\%$, up to wavelengths as long as 4.75 μm . We find that the temperature dependence of E_0 is generally consistent with that accepted in the literature. We are able to measure E_0 up to 300K and E_0+D_0 over a wide temperature range, confirming that D_0 is temperature-independent. However, with regard to the behaviour as a function of Sb content, while our PR measurements confirm the literature value of the E_0 bowing parameter, we find a dramatically different, and oppositely signed, D_0 bowing as compared to the currently accepted value. Due to the importance of D_0 in the prediction and interpretation of the performance of InAsSb-based devices, our new result is expected to have an important impact.

17:00

Oral

Surface photovoltage spectroscopy of quantum well and quantum dot systems

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Surface photovoltage spectroscopy (SPS) has been used for many years to study the properties of electronic states near the surface in bulk semiconductors. With the advent of crystal growth techniques, allowing the production of high quality semiconductor heterostructures, SPS spectroscopy found additional applications. It was used to study electronic states in heterostructures, quantum wells (QW's), quantum dots (QD's) and also electronic device structures.

We present room temperature SPS results obtained on MBE grown semiconductor structures with QW's and QD's. Obtained results were compared with other spectroscopic methods like photoluminescence (PL) and photorelectance (PR).

The SPS measurements were performed at contactless mode at normal incidence using a probe light chopped at 160 Hz. The absence of electric contacts makes a contactless mode more convenient to perform and non-destructive comparing to standard surface photovoltage measurements.

We have investigated GaAsSb/GaAs and GaAs/InGaAs/AlGaAs QW structures. In both cases SPS measurements gave us additional information about QW's electronic structure comparing to PL and PR. In case of GaAsSb/GaAs QW's the SPS spectra show additional features corresponding to optical transitions between light hole state in valence band and electronic state (1lh-1e) and conduction band edge (1lh-cb) in conduction band. In GaAs/InGaAs/AlGaAs QW case, SPS spectra give us some information about band offset between semiconductors forming QW.

Except of QW structures we have investigated InGaAs/GaAs single layer QD structures and InAs/GaAs vertically stacked QD structures. Obtained spectra usually show additional features, connected with higher energy QD optical transitions, comparing to PL and PR. Surface photovoltage spectroscopy with photoluminescence and photorelectance are turning out to be supplementary techniques for investigating semiconductor heterostructures (QW's, QD's) and device (VCSEL, laser) structures.

Wednesday, 19 September

Plenary Session

Wednesday morning, 19 September, 9:30

Coffee break

Wednesday morning, 19 September, 10:30

Session VI : Grazing incidence X-ray scattering techniques

Wednesday morning, 19 September, 11:00

11:00

Invited

Grazing-incidence X-ray diffraction from free-standing InAs/GaAs nanorods

Ullrich Pietsch¹, Andreas Biermanns¹, Felix Ntui Ayuk¹, Jörg Grenzer², Jens Bauer³, Volker Gottschalch³

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Vertically aligned group IV and A(III)BV nanorods (NR) typically grow by VLS mechanism onto $\langle 111 \rangle_B$ oriented semiconductor sub-

strates nearly independent from lattice mismatch between NR and substrate. In case of InAs NR onto GaAs this mismatch exceeds 7%. The mechanism of strain accommodation is not clear. Strain calculation in terms of a Valence-Force Field model suggest a strain energy which is 10 times smaller for epitaxy on [111] oriented support compared to epitaxy onto [001] material. However, the knowledge of strain accommodation is essential for the general understanding of the VLS growth process.

InAs NR were grown on GaAs [111B] by VLS technique using gold droplets as seed. Prior to x-ray measurements the NR were inspected by SEM showing pyramidal shape. First we measured reciprocal space maps in the vicinity of the (111) out-of plane Bragg peak of GaAs. It shows separate Bragg reflections of NR and substrate and appearance of out-of plane strain. Grazing-incidence in-plane X-ray diffraction experiments have been performed at ESRF beamline ID10b. Running in-plane longitudinal scans ($Q - 2Q$ scans) at different a_i close to the critical angle of total external reflection, a_c , we measured the strain evolution as function of depth close to the interface between NR and substrate. Besides small peaks caused by scattering from Au seed we find strong zinc-blende type Bragg peaks for InAs NR and the GaAs substrate. Both indicate compressive and tensile strain components at NR and substrate, respectively. These features show the same depth dependence as the substrate reflection whereas the NR diffraction shows bulk behaviour. Our findings will be used to determine the nature of strain accommodation at the interface. In between the InAs NR and GaAs substrate peaks we find an additional peak of small intensity which can be interpreted by a thin layer made of solid solution of InAs and GaAs which has been grown prior to the NRs.

11:35

Invited

The analysis of mesostructured thin films by grazing incidence x-ray scattering techniques

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It is nowadays possible to make and control the organization of mesostructured materials at the nanoscale both laterally and perpendicularly to the substrate either by nanolithography or by self assembly. At the nanoscale, x-ray scattering techniques in grazing incidence are ideally suited to characterize in a non intrusive way the structure of such materials. Along the normal to the surface, X-ray reflectivity is a unique tool to investigate their electron density profile. GISAXS (Grazing Incidence Small Angle X-ray Scattering) is on the contrary used in a complementary manner to ascertain the morphology of nanostructured materials parallel to the plane of the substrate. In this work, a brief description of these techniques will be given and we will illustrate their use on various kind of materials ranging from gratings to mesoporous materials.

12:10

Oral

The international VAMAS project "X-ray reflectivity measurements for evaluation of thin films and multilayers" for X-Ray metrology

Laura Depero¹, Dileep K. Agnihotri, Victor E. Asadchikov², Elza Bontempi¹, Keith Bowen³, Chang-Hwan Chang⁴, Paolo Colombi¹, Mark Farnworth⁵, Toshiyuki Fujimoto⁶, Alain P. Gibaud⁷, Matej Jergel⁸, Michael Krumrey⁹, Tamzin Lafford³, Alessio Lamperti¹⁰, Richard J. Matyi¹¹, Mojimir Meduna¹², Silvia Milita¹³, Kenji Sakurai¹⁴, Leonid Shabelnikov¹⁵, Alex Ulyanenko¹⁶, Arie Van Der Lee¹⁷, Claudia Wiemer¹⁸

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X-ray reflectivity (XRR) is a unique technique for thin film analysis providing information on thickness, density, and roughness. Indeed, XRR is becoming more and more popular in the characterization of both crystalline and amorphous layered samples.

This technique starts now to be applied in several industrial fields, involving thin film deposition and/or control, but the lacking of good manual practice and standard reference samples makes it hard to norm for metrology.

The VAMAS project "X-ray reflectivity measurements for evaluation of thin films and multilayers" has the goal to assess the accuracy and precision of thickness, density and roughness as determined by XRR measurements and simulations. Preliminary results are encouraging and reference samples are going to be evaluated by the laboratories participating to the project. The goal of this work is to show and discuss the results of the round-robin process involving for the first time 20 laboratory around the world and to show that XRR is a mature technique for nanometrology.

Lunch break

Wednesday afternoon, 19 September, 12:30

Session VII : Optical Polarization Metrology

Wednesday afternoon, 19 September, 14:00

14:00

Invited

Study of the light depolarization induced by sharp dielectric and metallic tips and of its effects on the selection rules of polarized Tip-Enhanced Raman Scattering on crystals

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In Apertureless Scanning Near-Field Optical Microscopy, a sharp tip is used to locally enhance the optical tip-sample interaction, permitting to map the electron and/or phonon properties of the samples on the 10-nm scale. In this paper, we present a quantitative study of the depolarization properties of sharp metallic and dielectric tips. Depolarization effects are particularly important for Tip-Enhanced Raman Spectroscopy (TERS) of solid crystals, in which the Raman emission can be strongly polarized according to symmetry-related selection rules, and therefore dependent on the actual polarization state of the incident light. Our results show that metallic tips can depolarize linearly polarized light from a few % up to 25 – 30 %, depending on whether the incident polarization state is parallel or orthogonal to the tip axis. Effects of tip-induced depolarization are studied by investigating the Raman emission of Si (100) at 520 cm⁻¹. Because of polarization selection rules, the emission from this band is totally depolarized if the sample is illuminated from the far-field with light polarized along the [100] direction. The proximity of the tip, instead, induces a strong component of Raman signal polarized along the incident polarization, which is interpreted as an effect of the tip-induced near-field depolarization. Comparisons between the 1-phonon band at 520 cm⁻¹ and the 2-phonons bands at 300 and 980 cm⁻¹, characterized by different polarization selection rules, allow drawing conclusions on the local tip-sample optical interaction, and on the actual field enhancement provided by the tip.

14:35

Oral

Normal-incidence generalized ellipsometry: learning more about the optics of graphite

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Graphite is one of the most important materials in our industrialized economy, but many of its fundamental properties are poorly understood. In particular, the optical properties are not even known in the visible part of the spectrum, due primarily to the unavailability of large single crystals of graphite. The best samples are highly oriented pyrolytic graphite (HOPG), which consist of microcrystals of graphite roughly oriented with the c-axis oriented perpendicular to the surface. In this paper, we present a technique to determine the complex optical functions of graphite using a combination of standard ellipsometry and normal-incidence generalized ellipsometry. Two ellipsometry measurements are made on oriented HOPG: 1) standard ellipsometry measurements are performed on cleaved HOPG with the optic axis perpendicular to the sample surface and 2) two-modulator generalized ellipsometry microscope (2-MGEM) measurements are made on HOPG cut and polished such that the optic axis is parallel to the sample surface. The measurements are performed at 9 different wavelengths from 405

to 750 nm and determine the optical functions of graphite for light polarized perpendicular and parallel to the optic axis (ordinary and extraordinary). The measurements show that the optical functions for the two different polarizations are quite different: the ordinary optical functions show evidence of band-to-band transitions throughout the visible and a Drude-like behavior in the red part of the spectrum. In contrast, the extraordinary dielectric functions do not show any Drude-like behavior, but do show band-to-band transitions throughout the observed spectrum. The 2-MGEM measurements also generate images of optical polarization parameters such as diattenuation, retardation, and the direction of the principal axis. Research sponsored by Division of Materials Sciences and Engineering and the DOE Office of Nuclear Energy, Science and Technology's Advanced Gas Reactor program.

14:55

Oral

In-Situ and Real-Time Protein Adsorption Study by Spectroscopic Ellipsometry

Sylvie Lousinian, Stergios Logothetidis

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Spectroscopic ellipsometry (SE) is a non-destructive technique, with high sensitivity, that can be applied both in air and in liquid ambient and perform very fast measurements. These advantages together with the use of advanced modeling procedures regarding SE data can provide valuable information for the study of biological samples. The aim of this work is to explore the protein adsorption phenomena and mechanisms of two basic blood plasma proteins on amorphous hydrogenated carbon (a-C:H) thin films, grown with rf reactive magnetron sputtering on c-Si substrates. The proteins Human Serum Albumin (HSA) and Fibrinogen (Fib), which play an important role in preventing and causing thrombi formation, respectively, were studied. Their adsorption was monitored in real-time (msec time) in Vis to UV spectral region for two pH values (at physiological conditions and at the protein's isoelectric point) and on a-C:H thin films with different deposition conditions, in order to better comprehend protein adsorption and contribute to the improvement of potential biomaterials properties through controlling their growth conditions. Complementary AFM and Fourier Transform IR-SE measurements were made to provide additional information about surface nanotopography and bonding configuration of the a-C:H films, to describe in detail the bio/non-bio interactions. The a-C:H films deposition conditions as well as the pH of the protein solution affect protein adsorption rate. This is due to the surface nanostructural characteristics and bonding structure (in the case of different growth conditions of the a-C:H thin films) and due to the change of the protein molecules charge and the change of the lateral electrostatic interactions between them, for different pH of the protein solution.

15:15

Oral

Ellipsometric and ion beam analytical studies on sputtered and annealed niobium oxide thin films

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For preparation of niobium oxide films radio frequency sputtering was used with Nb₂O₅ crystalline powder. The sputtering gas was an argon-oxygen mixture (Ar/O₂=4/1). During the sputtering the substrate was water-cooled, and chamber pressure was kept at 0.3 Pa. The deposited film thickness was adjusted to about 50-350 nm by selecting an appropriate sputtering time. The precise knowledge of the optical properties and the thickness are important in the design of optical film systems. The thickness and the complex dielectric functions of the as-prepared and annealed niobium oxide films were determined by spectroscopic ellipsometry using the Cauchy dispersion model and the Tauc-Lorentz oscillator model. The insertion of a roughness layer between the ambient and the niobium oxide film in the optical model resulted in better agreement between the experimental and the generated spectroellipsometric data. The composition of the layers was studied using Rutherford Backscattering Spectrometry with 2 MeV ⁴He⁺ ion beam. The Nb/O ratio was found in the close vicinity of the stoichiometric one. The films contained small amount of argon. Upon annealing (500 °C, 1 hour), the extinction coefficient decreased by about 20% in the ultraviolet spectral range. The optical properties of the sputtered films were compared to those obtained by plasma enhanced chemical vapor deposition [1].

[1] J.-P. Masse, H. Szymanowski, O. Zabieda, A. Amassian, J. E. Klemberg-Sapieha, L. Martinu, *Thin Solid Films* 515 (2006) 1674.

Coffee break

Wednesday afternoon, 19 September, 15:30

Joint Poster Session (Part II)

Poster Award Ceremony

Wednesday afternoon, 19 September, 15:50

Thursday, 20 September

Session VIII : X-ray scattering techniques-Stress and Strain measurements

Thursday morning, 20 September, 9:00

9:00 Invited

X-ray Characterization of Crystalline Defects and Strains in Thin Films

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Sensing materials' properties at nanoscale presents new challenges for diffraction techniques. As much as novel synchrotron and neutron sources made measurements at small scales feasible, analyzing the results requires improved models in order to accurately extract properties of interest. One of the most serious impediments to improving the methodology relates to sometimes orthogonal approaches to data analysis. A comparative analysis of some widely used approaches will be given with the emphasis on the use of full-powder-pattern fitting techniques, such as Rietveld refinement, to obtain information about coherently diffracting domain size, strain, defect densities, and residual stress. A topic of considerable interest is tungsten thin films used in the fabrication of photon number-resolving transition-edge sensor (TES) and arrays of TES detectors for astronomical and quantum in-

formation applications. Tungsten can form in one of two crystal structures: alpha (bcc), with a superconducting transition temperature (T_c) of 15 mK, and beta (A15), with a T_c between 1 and 4 K. Films with intermediate T_c s are composed of both alpha and beta phases. Variation of deposition conditions, and also the choice of the underlayer/coating for equal deposition conditions, affect the T_c s of tungsten films. We have used both laboratory and synchrotron (APS high-energy 6-ID-MU beamline at 52 keV in transmission geometry) X-ray diffraction to determine the structure of tungsten thin films and film stress at both room temperature and 8 K.

9:35

Invited

Influence of crystallographic orientation on local strains in silicon: a combined High-Resolution X-Ray Diffraction and Finite Element Modelling investigation

Michel Eberlein¹, Stephanie Escoubas¹, Olivier Thomas¹, Pascal Rohr², Romain Coppard²

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With the general trend of reduced dimensions in microelectronic devices, local mechanical stresses are increasingly considered a very important issue. The local strain field is worth investigating in particular in Shallow Trench Isolation (STI) which is a key step in the production of non-volatile-memories and transistors. We investigate by High Resolution X-ray Diffraction (HRXRD) the periodic strain field induced in silicon by the STI process. HRXRD is shown to be very sensitive ($\sim 10^{-4}$) to local strains and has the distinct advantage of being non destructive. Investigated samples are periodic arrays of SiO₂-filled trenches in single crystal (001) Si. The trenches are etched either along [100] or along [110]. The HRXRD experiments have been performed on a 4 circles goniometer with a laboratory source. We have recorded Si 004 and asymmetric Si 224 or Si 404 reciprocal space maps in order to extract axial strains in the 3 directions. The trenches array induces a periodic strain field in silicon, which gives rise to distinct satellites in reciprocal space, shifted from the substrate peak position. The intensity of these satellites is related to the strain field. We also performed elastic calculations with a Finite Element Modelling (FEM) code. Finally structure factor calculations are performed using the displacement field determined from mechanical modelling. The simulated diffraction maps are compared with experimental ones and allow for a complete validation of the stress field. We focus here on the effect of silicon elastic anisotropy on the strain field in 580nm and 200nm period samples. The influence of line crystallographic orientation ([100] or [110]) on the strain field is evaluated by FEM. These results are compared with experimental ones and discussed.

10:10

Oral

Strain measurements with depth resolution by energy-variable X-ray diffraction

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Energy-variable X-ray diffraction technique is established as a novel method for depth-resolved measurements of lattice d -spacings and residual strains in layered polycrystalline materials. Achieving high depth

resolution in X-ray diffraction measurements is still a problem because of rather large penetration lengths. In single-crystalline structures, an advantage is taken from the coherent interaction between X-rays and crystal lattice. In polycrystalline materials, the phase correlations exist within individual grains only and the coherence is lost on larger length scales. As a result, the obtained diffraction profiles are free of interference features and cannot be used for analysis of structural parameters with depth resolution.

In our method, the depth sensitivity is achieved by the controlled changing the X-ray energy and, hence, the X-ray penetration into the sample. Theoretical analysis of the problem accomplished for bulk materials [1] and thin films [2] showed that X-ray quanta scattered at different depths will be registered with different probabilities in the detector's system. Based on this fact, a characteristic depth is analytically derived, which provides the maximum of the detected X-ray intensity. An analysis of this expression allowed us to conclude that a sub-micrometer range for the in-depth steps is easily achievable.

The developed technique is applied to characterize the metal-metal, ceramic-metal, and ceramic-ceramic multilayers produced by different methods. We show a capability to characterize the strain changes at interfaces buried a 30-50 microns beneath the sample surface, to detect strain variations on a 100 nm scale and to collect experimental data in steps of 10 nm, when working not far away from absorption edges.

[1] E. Zolotoyabko, B. Pokroy, J. P. Quintana. *J. Synchrotron Rad.* 11, 309-313 (2004).

[2] E. Zolotoyabko, B. Pokroy, T. Cohen-Hyams, J. P. Quintana. *Nucl. Instr. & Meth. Phys. Res. B* 246, 244-248 (2006).

Coffee break

Thursday morning, 20 September, 10:30

Session IX : Characterization of functional oxides

Thursday morning, 20 September, 11:00

11:00

Invited

Investigation of strain and strain relaxation in functional epitaxial oxides using X-ray scattering techniques

Florine Conchon¹, Alexandre Boulle¹, René Guinebretière¹, Stéphane Pignard², Cécile Girardot², Eric Dooryhée³, Jean Louis Hodeau³, Laure Libralesso⁴, Tien Lin Lee⁴

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Functional oxide based on the perovskite structure are the subject of intense research in the field of micro- and nanoelectronics. However, in contrast with semiconductor films, oxide layers are much more difficult to grow with a high crystalline quality level which results in very broad and complicated reciprocal space features. Therefore it is often extremely challenging to obtain reliable information concerning the nanostructure of oxide layers using X-ray diffraction (XRD).

In this communication we will address some important issues that can be encountered in the study of RNiO₃ perovskites (R = rare-earth). These compounds are extremely difficult to stabilize because of the

less stable 3+ oxidation state of Ni. A possible way to stabilise RNiO₃ phases is to use epitaxial strain. It can hence be expected that strain will profoundly affect the structure and microstructure of these epitaxial films.

Using the particular example of SmNiO₃ grown on different substrates (SrTiO₃, LaAlO₃), we will show in this communication that using different complementary X-ray scattering techniques allows us to access a wealth of information concerning the defect structure of the films:

- the contribution of chemical and mechanical effects to the lattice parameter can be rigorously separated using laboratory based X-ray reciprocal space mapping and synchrotron grazing incidence diffraction.
- the effect of relaxation-induced misfit dislocations are investigated using high resolution synchrotron XRD (HRXRD). The presence of misfit dislocations induces asymmetrical longitudinal profiles which are quantitatively analyzed using a semi-kinematical scattering model based on B-spline functions.
- highly localized strain fields due to a particular type of planar faults (Ruddlesden-Popper faults) are studied by HRXRD. These defects give rise to extended diffuse scattering in reciprocal space which can be analyzed using a recently developed phenomenological scattering model.

11:35

Oral

Thickness determination of monolayered or multilayered SrZrO₃ thin films using XRD reflectometry

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High-*k* SrZrO₃ perovskite thin films, suitable for microelectronics applications were deposited under various conditions, on plane Si(100) substrates by direct liquid injection metal-organic chemical vapor deposition (MOCVD) method. Depending on the deposition temperature, films were either crystallized either amorphous. Amorphous films were annealed in order to obtain the expected orthorhombic perovskite structure.

X-ray reflectometry method is a versatile and non-destructive technique for a fast and accurate thickness determination of thin layered heterostructures. In that case, the model-independent Fourier-inversion method applied to a reflectivity curve allows a determination of the individual film thicknesses.

Reflectometry measurements provide the thicknesses of individual layer as well as multi-layers of a stack formed in the films. To estimate the order of the multi-layers, SIMS measurements were also conducted on all the studied thin films. Thicknesses were successfully confirmed by this destructive method and chemical environment of the films was determined as well as the composition of the layers. This information on as-deposited and post-annealed films was also compared with infrared transmission measurements (FTIR) giving clues on desorption of volatile species during deposition or annealing. Thus, crystallographic and chemical structures were precised on studied samples.

11:55

Oral

Photoelectrochemical studies of semiconducting photoanodes for hydrogen production via water dissociation

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It is well known that the photoelectrochemical (PEC) hydrolysis of water via solar energy can directly produce both gaseous hydrogen and oxygen. The PEC process is, however, very inefficient when using semiconducting n-type photoanode materials that are chemically stable in the highly basic aqueous environment required for PEC cell operation. Solving this problem will require the identification of materials that are chemically stable in basic solutions, that have a sufficiently small band gap to convert a reasonable part of the solar spectrum, and that have band edges located at potentials which allow the evolution of both H₂ and O₂. Here we describe the photoelectrochemical properties of n-type potassium tantalate (KTaO₃) and zinc oxide (ZnO) single-crystal PEC cell photoanodes. Although the band gaps of these two materials are not ideal solar collectors (the band edges are 3.64 and 3.34 eV respectively), they do not spontaneously disassociate in high pH aqueous solutions, and they are well-known indirect and direct gap semiconductors. Variations of these pure materials are obtained by doping KTaO₃ with alkaline-earth elements (Ca²⁺ and Ba²⁺) or other metallic impurities and by doping ZnO with Ga, Li, In, Gd, Er, or Mg. Both materials can form diodes when one face is in contact with a high-pH electrolyte (such as 8.5M NaOH) and can act as a photochemical cell, converting the photons above their respective band gaps into electricity and/or oxygen and hydrogen. The PEC characteristics include current-voltage (I-V) curves under illumination and spectral quantum efficiency (QE) measurements. Our results show that the maximum QE of KTaO₃ PEC cells occurs at 260 nm – with some photo-response for all wavelengths below 340 nm. ZnO PEC cells disassociate slowly in the electrolyte solution, but show significantly higher QE's for wavelengths shorter than ~365 nm. Research sponsored by the Division of Materials Sciences and Engineering, U.S.D.O.E.

12:15

Oral

Ceramic materials Ba_(1-x)Sr_xTiO₃ for electronics – synthesis and characterization

Ciceron A. Berbecaru¹, Horia V. Alexandru¹, Constantin Porosnicu¹, Alin Velea¹, Andrei Ioachim², Liviu Nedelcu², Mariana Toacsen²

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Oxide based ceramics Ba_(1-x)Sr_xTiO₃ -BST are important for applications in electronics. Samples prepared by solid-state reaction from BaO, SrO and TiO₂ were sintered 1200-1260 °C for 2 hours. The following compositions were tested x = 0.25, 0.40, 0.35, 0.50, 0.60, 0.75, 0.90 and small amounts of 1 wt. % MgO and 1 wt. % MnO₂ were added to improve the grains crystallisation. Archimedes's method for ceramic density measurements gives for x content the following values r(x) = 5.50–1.00 x, (g/cm³). XRD, EDAX, SEM methods were used for sample characterization. Perovskite structure, [1,2] was dominant

and other minor phases were detected. Grains of ~5 mm size, well faceted, could be “seen” by SEM analysis for x = 0.75 samples. Pores and their dimensions grow for Sr content and sintering temperature increase. Dielectric permittivity and loss were investigated (1 kHz) and -150 to +150 °C temperature range at a pace of ~2 °C/min. Sharp transitions were noticed with the sintering temperature increase with some well defined peaks of permittivity of higher values (2000-4000). A shift of the Curie points to higher values was noticed with Sr content according to the eqn: T_c = 130 - 323·x, [3]. At room temperatures the permittivity measured at 1GHz show increasing values (1300 to 2500) with sintering temperature and decreased values of losses (2 % to less than 0.5 %). The x = 0.35 composition being in the ferroelectric phase could not be measured in microwave at room temperature. Our results points to important application of these BST compositions for microwave devices.

References: [1] Jae-Ho Jeon, J. Europ. Ceram. Soc., 24 (2004) p.1045–1048, [2] H.V.Alexandru et al, Appl. Surf. Sci., 253 (2006) p.354-357, [3] A.Ioachim et al Mater. Sci. EnG. C, 26 (2006) p.1156-1161.

Lunch break

Thursday afternoon, 20 September, 12:35

Session X : NATCO Workshop I

Thursday afternoon, 20 September, 14:00

14:10

Oral

Dopant Ionic Radius and Electronic Structure Effects on the Transparency of Doped Cu₂O Transparent Conducting Oxide

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Cu₂O derived materials are currently leading candidates for p-type transparent conducting oxides (TCOs). In the "Novel Advanced Transparent Conducting Oxides" (NATCO) project, we are using first principles density functional theory (DFT) calculations in collaboration with experiment to develop and characterise candidate p-type TCO materials. Currently leading p-type TCOs based on Cu₂O are SrCu₂O₂, CuAlO₂ and CuGaO₂, which are alloys of Cu₂O and a second oxide material. Simple substitutional doping of Cu₂O with nickel has recently been studied. In this presentation, we use DFT to study substitutional doping of Cu₂O, with particular emphasis on the effect of dopant ionic radius and electronic structure. We firstly show that in Cu₂O, the origin of the small band gap arises from the presence of Cu-Cu interactions. In terms of doping, ions with larger ionic radius than Cu⁺ (Sn²⁺, La³⁺, Ce⁴⁺) strongly distort the atomic structure around the dopant site, disrupting the Cu-Cu interactions, while doping with ions that show a smaller ionic radius (Mg²⁺, Al³⁺, Cr⁴⁺) has no effect on the local atomic structure around the dopant site. For Sn and La, the distortions to the Cu-Cu interactions open up the band gap, while for Al and Cr, the lack of structural distortion leads to a small change in band gap. This demonstrates explicitly the important, yet unexplored, role of Cu-Cu interactions in Cu₂O in determining the transparency of doped Cu₂O. However, the dopant electronic states also play a crucial role, as exemplified by Ce and In, both of which have larger ionic radii than Cu⁺ and show strong distortions around the dopant site; however, the presence of the dopant electronic states that interact with the Cu₂O valence or conduction band edge, reduces the band gap and transpar-

ency. From these extensive calculations a set of rules for choosing potential dopants are presented.

14:30 Oral

Investigation on optical properties and conduction mechanism of p-type SrCu_2O_2

Mircea Modreanu¹, Michael J. Nolan¹, Simon D. Elliott¹, Olivier Durand², Evie L. Papadopoulou⁶, Guy Garry², Bernard Servet², Guido Huyberegts³, Jean-Luc Deschanvres⁵, Elias Aperathitis⁴

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Transparent conductive oxides (TCOs) are materials displaying a remarkable combination of high electrical conductivity and optical transparency. Most of the currently known TCOs exhibit only *n*-type electrical conductivity and due to the lack of controlled and reproducible *p*-type conduction in these oxides there is a limited number of oxide-based *p-n* junctions. In this paper, we report on first principles *ab initio* modeling and experimental studies of optical and microstructural properties of both bulk and thin films of SrCu_2O_2 . Using periodic density functional theory (DFT, PBE exchange-correlation functional) plane wave calculations of the atomic and band structure of SrCu_2O_2 , we have investigated the formation of Cu vacancies as the origin of *p*-type character in SrCu_2O_2 . The DFT calculated lattice parameters were found to be in good agreement with the experimental ones obtained from XRD. Fourier Transform Infrared (FTIR) and Raman spectroscopies are used in the investigation of the vibrational properties of both bulk and thin film SrCu_2O_2 deposited by two deposition techniques, namely Pulsed Laser Deposition and MOCVD. We report here the fundamental infrared and Raman active modes for the tetragonal phase of SrCu_2O_2 . The spectroscopic ellipsometry and UV VIS NIR reflectance results show that the tetragonal phase of SrCu_2O_2 has an optical bandgap of 3.34 eV.

14:50 Oral

The effect of PLD deposition parameters on the properties of p- SrCu_2O_2 /n-Si diodes

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There have been many attempts towards the fabrication of *p*-type transparent oxides since the discovery of the first *p*-type transparent CuAlO_2 by Kawazoe et al. in 1997. The research had been focused on investigating the properties of these *p*-type oxides for realizing novel electronic and light emitting or light detecting devices. These devices either utilize both *n*-type and *p*-type transparent oxides for forming the *p/n* junction thus creating transparent devices, or make use of one type (*n*- or *p*-) of these transparent oxides on conventional semiconductors like Si and GaAs, leading to devices with unique properties.

In this investigation we report on the properties of *p*- SrCu_2O_2 (SCO) films on *n*-Si wafers and the resulting *p/n* junction. The SCO films were fabricated by pulsed laser deposition (PLD) from a SCO target on UV-grade fused silica and Si substrates and their properties (structural, electrical and optical) were investigated as a function of PLD parameters like substrate temperature (300–600°C) and oxygen pressure (10^{-2} – 10^{-5} mbar). All SCO films were shown to be *p*-type semiconductors, with a carrier density in the order of 10^{14} – 10^{16} cm⁻³ and resistivity in the order of 300 Ωcm. Their transparency reaches 80%. Post-deposition annealing in O_2 environment reduced resistivity from 293 Ωcm to 60 Ωcm. The properties of the *p*-SCO/*n*-Si diodes were investigated using I-V, C-V and DLTS measurements and the use of SCO/Si heterostructure for electronic as well as optoelectronic applications will be addressed.

15:10 Oral

Effect of Chlorine doping on electrical and optical properties of ZnO thin films

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ZnO is a semiconductor oxide material with low resistivity, high transmittance up to UV spectral range and with a good chemical stability under strong reducing environments. It is thus a promising Transparent Conductive Oxide (TCO) and a possible alternative to tin oxide and indium tin oxide to be used as transparent electrode for photovoltaic solar cell for example [1]. We present a study of chlorine doping of ZnO. ZnO:Cl layers have been grown by MOCVD technique, in a

vertical geometry reactor. The optimal growth temperature for the quality of the layers was found $T=425^{\circ}\text{C}$. This relatively low growth temperature is compatible with further processing of TCO based devices. The growth was performed on c-sapphire and fused silica. The theta-2theta X-ray diffraction scans present the ZnO wurtzite symmetry structure without any additional phase. Transport properties were studied for samples with different content of chlorine. Hall Effect measurements show the increase of electron carrier concentration and decreases of electron mobility while increasing the amount of chlorine incorporated in ZnO. Carrier concentration as high as 6.510^{20}cm^{-3} has been achieved with resistivity of $\rho=1.4\times 10^{-3}\text{ Wcm}$. Activation energy related with chlorine centre has been determined from temperature dependent Hall measurements. Optical transmittance is greater than 82%. Low temperature cathodoluminescence and photoluminescence show strong UV excitonic emission for all ZnO:Cl, reflecting the conservation of the optical properties of the layers. First principles calculations using density functional theory are presented for the electronic structure of models of Cl-doped ZnO. These results demonstrate that the use of Chlorine is an interesting route to achieve high level of n-type doping by MOCVD with keeping high VIS-IR transparency of the ZnO layers.

[1]A.Guillen-Santiago, M.Olivera, A.Maldonado, et al. Phys.Stat.Sol.(a) **201**, (2004), 952

Coffee break

Thursday afternoon, 20 September, 15:30

Session XI : NATCO Workshop II

Thursday afternoon, 20 September, 15:50

15:50 Oral

Study of the growth conditions of SrCu_2O_2 thin films deposited by injection MOCVD

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Divalent ion in a copper oxide matrix $\text{M}^{2+}\text{Cu}_2\text{O}_2$ are intensively studied for the application field of p-type transparent conductive oxide in order to achieve transparent electronic devices. In this paper, we have studied the SrCu_2O_2 (SCO) growth conditions by means of an injection-MOCVD apparatus. Few works have been realized by MOCVD despite its high facility to prepare different ratio of cation. The precursors for this study were strontium and copper tetramethylheptadionate and the deposition temperature has been ranging from 480°C to 570°C . For these temperatures, FTIR and XRD analyses have shown that the films were composed of a mixture of copper oxide (CuO) and strontium carbonate. This later compound is due to the low deposition temperature. So to achieve the SCO compounds, two kinds of annealing have been studied: the *ex-situ* Rapid Thermal Process (RTP) and the *ex situ* conventional annealing. The ability to obtain the pure SCO phase with the different annealing has been analyzed related to the film composition. For the RTP, the atmosphere was Ar with or without O_2 . Without O_2 , the annealing is too reductive and metallic copper appears on the

surface of the annealed films. Whereas an O_2 partial pressure is enough to burn all the carbonate and make the crystallization of $\text{Sr}_3\text{Cu}_5\text{O}_{8+z}$ phase as observed by FTIR and XRD measurements. Then, after a final pure Ar RTP, the SCO phase is obtained. The structural and electrical properties of the annealed films are discussed related to the deposition conditions, the film compositions and the annealing conditions.

16:10 Oral

Defects and the Origin of p-Type Conductivity in Cu_2O : a First Principles Investigation

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The search is on for novel p-type transparent conducting oxide (TCO) materials, with Cu_2O derived materials currently leading candidates. As part of the "Novel Advanced Transparent Conducting Oxides" (NATCO) project, we are using first principles density functional theory (DFT) to study the properties of Cu_2O derived materials, with the aim of using the resulting band gap and computed effective hole masses to screen potential candidate p-type TCO materials. Before investigating TCO candidates, it is necessary to understand the properties of Cu_2O , the origin of the electronic structure and the p-type character of Cu_2O , which provide important insights for investigating more complex Cu_2O derived materials. In this talk, we present DFT calculations of stoichiometric Cu_2O and Cu_2O with Cu and O vacancy defects. From these calculations, we find the following (i) the small band gap in Cu_2O arises from the Cu-Cu interactions in Cu_2O , (ii) Cu vacancies have a significantly smaller formation energy than O vacancies, (iii) at finite temperature and pressure, Cu vacancies will form spontaneously, while O vacancies have a much larger formation energy and will not dope Cu_2O n-type or compensate holes (iv) neutral Cu vacancies are the origin of the p-type character of Cu_2O , leading to the formation of hole states 0.2 eV above the valence band. The implications of these results for candidate TCO materials are discussed.

16:30 Oral

Combination of the Optical Waveguide Lightmode Spectroscopy Method with Electrochemical Measurements

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Optical waveguides are normally sensitive to the various inhomogeneities inside the waveguide and also to the surrounding media and surface contaminations. Various sensor systems were developed on this principle. One of the most sensitive and effective systems is the optical waveguide lightmode spectroscopy (OWLS). The necessary OWLS sensor chips were developed on the basis of Silicon-Titanium-Oxide ($\text{Si}_{1-x}\text{Ti}_x\text{O}_2$), containing a grating for incoupling of laser light into the waveguide layer. The incoupling angle dependence of the photocurrent is measured. This basic waveguide layer can be covered with further solid layer in order to sensitise for selected specific applications. The sensor chips can be sensitised also for label free microbiological investigations. OWLS method allows not only qualitative, but also quantitative detection of biological species.

The electrochemical methods, on the other hand, are widely used both in inorganic and organic chemistry, and also advantages in microbiology were demonstrated.

In this work first results are presented in combination of these two methods for simultaneous measurement of refractive index changes of the waveguiding system and electrical current changes due to the presence of the cells/molecules/ions to be investigated. An Indium-Tin-Oxide (ITO) nanolayer is deposited and activated on the top of the sensor chip. ITO is known as n-type conductor. This electrically conductive oxide layer serves as working electrode in the specially developed electrochemical cuvette.

The basic results are demonstrated using H_2O_2 and toluidine blue solutions, using KCl and TRIS solutions as buffer and transport media. Measurements show the changes of the incoupling angles and that of the effective refractive indices (both electrical and magnetic modes) with the changes in buffer solutions and with the injection of other solutions and species to be studied and also on the applied potential in cyclic and chrono voltammetric measurements.

16:50 Oral

Optical properties of ZnN thin films fabricated by rf-sputtering from ZnN target

Maria G. Androulidaki¹, Sotiris Dounis^{1,3}, Popi Voulgaropoulou^{1,3}, Vicky Kambilafka^{1,5}, Michal Ružinský², Vladimír Šály^{1,2}, Patrik Prokein², Zacharias Viskadourakis⁴, Elias Aperathitis¹

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ZnN is a relatively new material, and even though its physical properties are not well studied and there is a controversy concerning its optical band gap, it has been used for the fabrication of p-type ZnO:N films through oxidation at temperatures $\geq 400^\circ\text{C}$ and recently as electrode in Li-ion batteries. Oxygen contamination has been considered donor for ZnN leading to higher carrier concentration and larger band gap energy due to Moss-Burstein effect. Intentional oxygen doping can create oxynitride phases (ZnON) but annealing in oxygen environment can convert n-ZnN into p-ZnO:N.

In this investigation, ZnN thin films were deposited by magnetron sputtering from ZnN target in plasma consisting of mixture of Ar- N_2 gases on silica and Si substrates. The ZnN films were n-type semiconductors having around 10^{20}cm^{-3} carrier concentration, resistivity between 10^{-2} - $10^{-3}\Omega\text{cm}$ and low transmittance. The amount of nitrogen in plasma affected more the optical properties than the electrical properties of the films. Annealing, in N_2 environment, at temperatures below 400°C improved the electrical properties but above 400°C the carrier concentration was reduced (10^{15}cm^{-3}) and the resistivity increased ($10^2\Omega\text{cm}$). The transmittance of the films was $\sim 85\%$ in the near UV-VIS region regardless the annealing temperature. X-Ray Diffraction was used for monitoring the phases in the structure of the films. Low temperature photoluminescence (PL) was employed to study the bound exciton states in the range 3.326 to 3.365eV. Films deposited in pure Ar or N_2 and after high temperature annealing, ex-

hibited a sharp emission peak at 3.360eV and another one at 3.306eV. These PL peaks were shifted to higher energies for films deposited in a mixture of Ar- N_2 plasma. Green PL, associated with defect states in the band gap, appeared at around 2.5eV. PL assignments were achieved by monitoring the temperature evolution of the peaks.

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17:10 Oral

Derivation of the complex refractive index in the infrared region of the spectrum from the analysis of optical spectra

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We have developed an algorithm to derive the IR-dispersion of the complex refractive index, $n = \eta - i k$, from the analysis of Reflectance and Transmittance optical spectra. Within the framework of the Drude model, this derivation allows the calculation of the plasma wavelength λ_p as well as the relaxation time τ . The mathematical approach to the problem is made by the use of the Newton-Raphson method [1]. The theoretical expressions for the Reflectance and the Transmittance, as well as their partial derivatives with respect to η and k which are necessary for the application of the method are introduced analytically improving speed and accuracy. The method has been applied to the analysis of optical spectra of Indium-Tin-oxide (ITO) as well as Indium-Tin-oxynitride (ITON) films produced by sputtering at different rf-power levels, these levels ranging from 150 to 550 W.

Within the wavelength range between 1.5 and 2.5 μm we notice the following. The detected values of τ range from 0.1×10^{-14} to 1×10^{-14} sec while those of λ_p from 2.0 to 5.0 μm , approximately. Thermal annealing at 600°C results in a decreased λ_p as well as an increased τ in the cases of ITO films, while for ITON films, the same trend is observed as regards λ_p , the opposite however as regards τ . This latter remark is consistent with an expected post annealing deterioration of the structure produced from the release of nitrogen. In the post annealing cases $\lambda_p \approx 2.0 \mu\text{m}$ for either ITO or ITON films.

[1] W. H. Press, B. P. Flannery, S. A. Teukolsky and W. T. Vetterling, *Numerical recipes in Fortran* (Cambridge University Press, Cambridge, U.K., 1986).

Friday, 21 September

Session XII : Characterisation of functional Materials

Friday morning, 21 September, 9:00

9:00

Oral

An old technique (Umweganregungen) for X-ray characterization of an epitaxial new material (Chromium doped GaN)

Martina Von Der Ahe, Yong Suk Cho, Nicoleta Kaluza, Hilde Hardtdegen, Michel Marso, Detlev Gruetzmacher

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Up to now the structural characterization of non-perfect epitaxial thin films always needs a series of different X-ray reflection measurements to determine strain and relaxation and also crystal quality. This is a very time consuming procedure for layer evaluation. In 1937 the appearance of *Umweganregungen* was discovered by Renninger – a method in which X-ray multiple diffraction for forbidden or quasi forbidden reflections is studied mostly for single crystals. Their intensity shows a strong dependence not only on the size of a crystal but also to an increasing degree on structural quality. In a Renninger scan a crystal is rotated about the normal to a set of diffracting planes while the diffraction from these planes is measured. The higher the intensity of the *Umweganregungen* is the higher the quality of the crystals. In this paper we will present the use of this old technique in a new context which will allow structural assessment of thin GaN layers doped with Chromium by just one measurement.

This method we applied to the MOVPE growth optimization of GaN:Cr layers to assess the best growth temperature and the influence of the dopant Chromium on GaN growth. The Bragg angle of the exactly (0001) reflection of GaN was used. To confirm the results additionally the conventional X-ray evaluation methods were carried out.

With both approaches we come to the same conclusion: an intermediate growth temperature and the incorporation of Cr are advantageous for the crystal quality. This report will give a comparative overview on both structural investigation methods, their advantages and limitations. It will be shown that Renninger scans are promising for future layer evaluation using only one measurement.

9:20

Oral

Undoped and Al-doped ZnO films with tuned properties by pulsed laser deposition

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Zinc oxide (ZnO) has been the focus of intense investigation due to the unique properties it possesses. It is a transparent, wide band gap

semiconductor ($E_g \sim 3.4\text{eV}$) with high exciton binding energy ($\sim 60\text{meV}$). Its conductivity, if properly doped, approaches the high value of the widely used n-type indium-tin-oxide (ITO), and if it could also be reproducibly of p-type, ZnO would be the ideal material for many emerging transparent optoelectronic applications.

However, if ZnO is to be used as an active layer in optoelectronic devices, it must have electrical and optical properties within a wide range of values. Thus, it would be of paramount technological importance if ZnO films can be fabricated with tuned properties by adjusting the deposition parameters.

In this investigation the properties of undoped and Al-doped ZnO films fabricated by pulsed laser deposition (PLD) on UV-grade fused silica and Si substrates are presented. The ZnO and ZnO:Al(2%) target were ablated by a 248 excimer laser and the films were deposited in a wide range of PLD parameters, including substrate temperature (up to 500°C), oxygen pressure (from 10^{-2} to 10^{-5} mbar) and at different target-substrate distances. Polycrystalline films having the wurtzite structure were fabricated, with carrier concentration between 10^{15} and 10^{21}cm^{-3} , resistivity from 100 to $3 \times 10^{-4}\ \Omega\text{cm}$ and E_g spanning from 3.2 to 3.7eV . Sharp emission excitonic peak at 353.6nm was observed for highly conductive ($3.3 \times 10^{-4}\ \Omega\text{cm}$) ZnO:Al films.

The properties of the ZnO films were investigated using atomic force microscopy (AFM), Hall-effect measurements and various spectroscopic techniques such as UV-NIR optical measurements, X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS) as well as low temperature photoluminescence (PL) for identifying excitonic emission peaks and band gap defects. The use of the ZnO films, fabricated in this investigation, in optoelectronic devices like LEDs, TFTs, etc., will also be addressed.

9:40

Oral

Electrical Conduction and Dielectric Relaxation of a-SiO_x (0<x<2) Thin Films Deposited by Reactive r.f. Magnetron Sputtering

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Silicon oxide (a-SiO) is one of the most used silicon-based materials in optoelectronic and microelectronic technologies. Previously, we have reported that the a-SiO_x (0<x<2) physical properties related to the material structure show a kink point in their evolution when x varies. It was found that highly oxygenated a-SiO_x thin films are inhomogeneous (silicon-rich nanoclusters embedded in oxygen-rich material). In this paper, we focus on electrical conductivity and carriers' transport mechanisms in a-SiO_x. Dc current-voltage characteristics are analysed by using the Pollak and Riess model of the percolation theory. A special attention is paid to the trapped charge in so called "dead ends" of the carriers' path. The current-time (for constant voltage pulses measurements) characteristics' analysis reveals the chargeability behaviour of the material.

The ac measurements have also been employed to investigate the electrical character of the SiO_x thin films. Both the modulus and the argument of the complex impedance have been analysed on SiO_x (with x>1), when the frequency was varied between 10^2 and 10^6Hz . The variation of the impedance modulus with the frequency showed a capacitive behaviour: in $\log \hat{Z} = f(\log \omega)$ plots, the slope of the linear variation was found around -1 . Similar results have been obtained studying the argument variation [$\arg \hat{Z}(-75^\circ; -90^\circ)$].

The intensive parameters as dielectric constant and electrical conductivity have been calculated. The results of this study revealed that a-SiO_x sputtered thin films are characterised by dielectric relaxation and electrical conductivity (with a transport mechanism described by variable range hopping).

10:00

Oral

Possibilities of thin film multilayer structures study by X-ray and ion beam complex of nondestructive methods

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The investigation of nanosize thin film multilayer structures is needed in knowledge of the element concentration, the crystal structure and the thickness for every layer of the structures. Moreover, it is very important to have the information about properties of interlayer spaces. Again, the knowledge is usually obtained in result of intercomplementary investigations. Because of this, the control methods must be characterized by the nondestructive influence on the structures. The complex of specific X-ray and ion beam testing methods can be effectively used for this problem solution.

The Rutherford backscattering of He⁺ and H⁺ ion fluxes by a multilayer target is the perfect method which allows to determinate the depth multielement concentration profile for it with thickness resolution near 10-20 nm. In case of the insufficient sensitivity of RBS method the measurements can be completed by the nuclear reaction spectrometry, the recoil nuclears method and the registration of X-ray fluorescence yield. The sensitivity maximum of X-ray fluorescence spectrometry can be achieved in condition of X-ray exciting beam total reflection on the surface target studied (in the framework of TXRF method). The using of planar X-ray waveguide-resonator (PXWR) for the exciting beam formation allows to improve TXRF method characteristics additionally. The method diagnoses the target element composition for very thin surface layer with thickness near 5 nm. The depth element diagnostics is possible in condition of the angular variation for X-ray exciting beam.

The crystal structure for every layer of the multilayer target can be studied by the initial X-ray beam grazing diffraction. The grazing diffraction efficiency can be enhanced by PXWR application. The complex method investigation is suitable for any structure state of layer, but it's efficiency maximum will achieve at the epitaxial structure study. All possibilities of the complex method are illustrated experimentally.

Coffee break

Friday morning, 21 September, 10:30

Round table

Future trends in Optical and X-ray metrologies
Friday morning, 21 September, 11:00

Lunch break

Friday afternoon, 21 September, 12:30

Posters

Monday, 17 September

Joint Poster Session (Part I)

Monday & Wednesday

Monday afternoon, 17 September, 15:50

15:50

Poster

H/PII.01

Effect of multi-layered bottom electrodes on the orientation of strontium-doped lead zirconate titanate thin films

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This paper discusses the results from Bragg-Brentano X-ray diffraction (XRD) and glancing angle X-ray diffraction (GA-XRD) analysis of piezoelectric strontium-doped lead zirconate titanate (PSZT) thin films deposited on multi-layer coatings on silicon. The films were deposited by RF magnetron sputtering, and a metal coated substrate was desired to exploit the pronounced piezoelectric effect that is theoretically expected normal to the substrate. This highlighted the influence that the bottom electrode architecture had on the final crystalline structure and surface roughness of the deposited thin films. A number of bottom electrode architectures were used, with the uppermost metal layer on which PSZT was deposited being gold or platinum. The choice and the thickness of the intermediate adhesion layers influenced the intensity, and in some cases, the presence of the desired perovskite peaks.

Gold was chosen as its lattice co-efficients were closely matched to that of PSZT; and considering the gold-silicon eutectic point of 363 °C, deposition on gold coated substrates was limited to 300 °C. Interdiffusion between gold, the titanium adhesion layer, and silicon was observed. A silicon dioxide layer was introduced to overcome the interdiffusion, and appeared to have an effect on the final crystalline state, which was determined using XRD.

For platinum bottom electrodes (which are preferred choice in literature), a number of multi-layer combinations were experimented with; these included different combinations and thicknesses of platinum, titanium, titanium dioxide, and silicon dioxide. Depositions were carried out at temperatures ranging from 500 °C to 750 °C, with post deposition annealing performed in certain cases. This paper shows the different reactions which occur, and highlights cases in which changing the bottom electrode architecture distinguished the dominant perovskite orientation.

15:50	Poster	H/PI.01

The optimization of the registration condition for the X-ray reflectivity of real multilayer thin films

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The X-ray reflectivity method is the useful tool for the determination of parameters describing thin- and multilayer film as: thickness (τ - the range from 10 to 2000 Å), density (ρ) and roughness (σ). The Cu, Au, Ag real multilayer films with different thickness, have been obtained by vacuum evaporation technique on silicon substrate, was analyzed under different registration condition.

Moreover, the Grazing Incident X-ray Diffraction Geometry (GIXD) method was applied for different incident angles for structure determination of the real multilayer thin films.

The measurements were performed by using the Philips X'PERT diffractometer. The calculations of the characteristic parameters of reflectivity data were carried out by the WINGIXA and SUPREX software. The standard X'PERT software was applied for the determination the characteristic parameters of Grazing Incident X-ray Diffraction Geometry (GIXD) method.

15:50	Poster	H/PII.02

TiON as multifunctional nanostructured film

Christos Trapalis¹, Mariuca Gartner², Petre Osiceanu², Tionica Stoica³, Toma Stoica³, Theophilou Giannakopoulou¹, Nikos Todorova¹, Anastasios Lagoyannis⁴

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Based on the excellent physico-chemical properties (high melting point, extreme hardness, high chemical stability, high electronic conductivity, interesting optical properties) nanocrystalline titanium oxynitride (TiON) films have wide applications for cutting tools, heat mirror, wear resistant coatings in bone surgery and not in the last row as photocatalyst in visible light applications (having the optical bandgap, Eg, in VIS range). Undoped TiO₂ sol-gel films were obtained by five alternative coatings TiO₂ - PEG₂₀₀₀-10% (PEG₂₀₀₀ means polyethylene glycol with molar weight 2000) on quartz substrate, thermally treated at 500°C for 2h in O₂ while the N doped TiO₂ films were obtained after an additional treatment at 500°C for 2h in NH₃ atmosphere. To choose the optimal temperature of treatment the N doped films were supplementary treated at various temperatures up to 1100 °C. Spectroscopic ellipsometry (SE) was used to follow the changes in the optical properties of the films. Different simulation models were handled based on the information received from X-Ray Diffraction and different spectroscopic analysis such as: Rutherford Backscattering, X-ray Photoelectron, Fourier Transform Infrared and UV-VIS.

The RBS measurements exhibited that the biggest N concentration and the smallest O concentration were found in the film treated in NH₃ at 1100 °C where the osbornite (TiN_{0.9}) is formed (based on XRD analysis).

is). In agreement with these data the UV-VIS shows a smaller transmittance when the samples are calcined up to 1100 °C. The value of absorption index (from SE analysis) increases from 0.09 at 600 °C to 1.58 at 1100 °C, at $\lambda=633$ nm. Related to refractive index, there is a competition between the densification processes (which leads to the increasing of refractive index) and enhancement of dopant concentration (which has the contrary tendency) reflected in its behavior.

15:50	Poster	PI.02

Raman spectra intensity as a function of the penetration depth of the visible light in the in-situ grown YBCO films

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We determined the penetration depth of the visible light in the YBCO films as a function of the visible light frequency using our ellipsometric spectra determined previously. We correlate this results with the Raman profiling spectra in the depth of YBCO films. We establish the optimal thickness of the YBCO films to obtain the maximum laser hitting effect in the depth of the YBCO films. We optimised the Raman signal as a function of the visible light frequency and establish that the YBCO resonance for the most important Raman peaks are at the blue light.

15:50	Poster	H/PI.03

High temperatures X-ray diffraction study of Tantalum – Oxygen phases

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ABSTRACT The structures and lattice parameters of the Tantalum and Ta₂O₅ phases were determined by high temperature X-ray diffraction under vacuum and low pressure carbon monoxide between 293 and 2278 K. We show that the metallic phase structure is stabilized by inclusion of oxygen into the metal octahedral hole. We determined the crystalline characteristics at high temperatures for Ta and Ta₂O₅ Phases.

15:50	Poster	H/PII.03

Growth and characterization of doped Ga₂O₃ and SnO₂ nanofibers using GaN and Sn powder mixtures

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One of the most exciting areas in materials science is the study of nanomaterials, due to their novel physical properties stemming from the reduced dimensions. Since monoclinic gallium oxide (b-Ga₂O₃) is chemically and thermally stable with a band gap of 4.8 eV, the b-Ga₂O₃ with low-dimensional nanostructure will likely provide an attractive promise for its practical application as well as for fabricating nanodevices with novel properties. Particularly, Ga₂O₃ has gained much attention as a new phosphor material for emissive display applications. In addition, Tin oxide (SnO₂), an n-type semiconductor with a wide

band gap ($E_g=3.6$ eV at 300 K), is regarded as one of the most promising materials for gas sensors, heat mirrors, photovoltaic solar energy conversion devices, and transparent electrodes. Accordingly, various structural and morphological forms of SnO_2 1D nanostructures have been fabricated over the past several years. Herein, we have investigated the production of gallium oxide (Ga_2O_3) and tin oxide (SnO_2) one-dimensional (1D) nanostructures by heating a mixture of GaN and Sn powders at 900 °C. TEM analysis revealed that the product corresponded to a mixture of monoclinic Ga_2O_3 nanofibers and tetragonal rutile SnO_2 nanofibers. EDX spectra suggested that Ga_2O_3 and SnO_2 nanofibers were doped with Sn and Ga elements, respectively. The PL measurement with a Gaussian fitting exhibited visible light emission bands centered at 445, 572, and 665 nm.

15:50	Poster	H/PI.04

Fourier analysis of optical profilometry and BRDF in paints art investigations

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The scope of this work is concerned with paints (i.e manual painting) and manual/maschine polishing of metallic surfaces. The optical profilometry (OP) by the measurements of scattered light scattered gives many information about surface topography, optical indices, and thickness of films in cases of their occurring as a resultant those factors. Combined PO investigations with for example mechanical profilometry allow to separate light scattering produced by surface topography from another another scatterings as volume or material scatterings. - To be continued

15:50	Poster	H/PII.04

Surface-enhanced Raman scattering of gold nanowires : role of dipolar and multipolar localized surface plasmons

Marc Lamy de la Chapelle, L. Billot, M. Lopes, A.-S. Grimault, A. Vial, D. Barchiesi, J.-L. Bijeon, P.-M. Adam, P. Royer

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We have studied the Surface-Enhanced Raman Scattering (SERS) of shape controlled metallic nanowires which can act as nano-antenna, designed through electron beam lithography and lift off techniques. We have notably studied the influence of localized surface plasmon resonance (LSPR) on the efficiency of SERS [1,2].

The deposited molecular probe used is the trans-1,2-Bis(4-pyridyl)ethylen (BPE) with two different excitation wavelengths of 632.8 nm and 676 nm. The nanowire lengths vary from 50 nm up to 1 μm (the width and the height of the nanowires is fixed). The observation of the dependence of the Raman enhancement versus the nanowire length is clearly demonstrated and remarkably the enhancement is observed to be maximum for a specific length. Moreover, we have compared our SERS results to the particle LSPR and we have determined that the best enhancement is obtained for a LSPR position close to the Raman wavelength. For such arrays, we also observe the existence of multipolar surface plasmon modes (up to 7th order) and we show clearly that multipolar LSP modes exhibits a stronger efficiency than the first dipolar order in SERS process [2].

Our results support the fact that the optimization of SERS efficiency, relative to the LSPR, strongly depends on the shape of the metallic nanoparticles. The enhancement strongly depends on the nanowire parameters which is a crucial point to determine the optical efficiencies of such structure and to optimise the SERS processes.

[1] J. Grand, M. Lamy de la Chapelle, J.-L. Bijeon, P.-M. Adam, A. Vial, and P. Royer, *Phys. Rev. B* **72**, 033407 (2005)

[2] L. Billot, M. Lamy de la Chapelle, A.-S. Grimault, A. Vial, D. Barchiesi, J.-L. Bijeon, P.-M. Adam and P. Royer, *Chem. Phys. Lett.* **422**, 303 (2006)

15:50	Poster	H/PII.05

Comparative ellipsometric and ion beam analytical studies on ion beam crystallized silicon implanted with Zn and Pb ions

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The near-surface structural changes of silicon implanted with Zn and Pb ions and subsequently crystallized using high energy silicon ion beam or annealed thermally have been studied by spectroscopic ellipsometry and Rutherford Backscattering Spectrometry in combination with channeling. Zn and Pb ions were implanted at 100 keV into silicon, in order to form amorphous layers. Ion Beam Induced Epitaxial Crystallization of the implanted amorphous silicon was induced at 400°C using 3 MeV Si^+ ion beam. Rutherford Backscattering Spectrometry and channeling techniques were applied to measure the thickness of the as-implanted amorphous as well as ion beam recrystallized layers and to obtain the depth distribution of Zn and Pb. Spectroscopic ellipsometry measurements were performed to determine the thickness of the surface oxide layer as well as the thickness of the amorphous and the recrystallized silicon layers. The evaluation of the ellipsometry data was carried out using different optical models consisting of a surface layer, one or more underlying layers and single crystalline silicon substrate. The dielectric function of single crystalline silicon, silicon dioxide, ion implantation amorphized silicon and the implanted-annealed silicon were taken from Refs [1] and [2]. In the case of Pb implantation the thermal annealing (525°C, 60 min) was not enough for the complete recrystallization of the implanted silicon layer. The highest annealing dose (of the 3 MeV Si^+ ion beam) was sufficient for almost complete recrystallization of the ion implantation amorphized silicon layers for both implanted species.

References

[1] Handbook of Optical Constants of Solids, edited by E. D. Palik (Academic Press, 1985)

[2] M. Fried, T. Lohner, W.A.M. Aarnink, L.J. Hanekamp, A. van Silfhout, *Journal of Applied Physics* **71** (1992) 5260

15:50 Poster H/PI.05

SnO₂ films deposited by spray pyrolysis: Intensity of XRD as a parameter for structural characterizationGhenadii Korotcenkov¹, Vladimir Brinzari¹, Johannes Schwank², Valeriu Blaja¹**1.** Technical University of Moldova (TUM), Bld. Stefan cel Mare, 168, Chisinau 2004, Moldova **2.** University of Michigan (UM), 3085 H.Dow Building, 2300 Hayward, Ann Arbor 48109-2125, United States

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The study of the influence of additives, used for gas sensing and magnetic properties optimization, on structural properties of SnO₂ films, deposited by spray pyrolysis method, was the main goal of research, discussed in this report. Films had thickness from 40 to 400 nm. Cu, Fe, Co, or Ni (0-16 at. %) were used as doping elements. For structural analysis of tested films we used XRD, SEM, and AFM techniques. The change of such parameters as film morphology, grain size, texture, and intensity of X-ray diffraction peaks were controlled. Annealing at 850-1000 °C was used for estimation thermal stability of film structure. It was established that tin oxide doping by Fe, Co, Ni, and Cu promoted the change of film morphology. However the decrease of grain size estimated on the base of XRD data was not big. On the base of obtained results it was concluded that the presence of big contents of fine dispersion phase, which does not provide reflexes in XRD patterns, make us to be careful with any quantitative evaluations, made on the base of XRD data, because in this case we obtain information mainly about grains given contribution in XRD patterns, first of all about crystallites incipient at initial growth stage and grown through the whole of film thickness. It was shown that at certain conditions, connected with keeping measurement conditions invariable, the intensity of X-ray diffraction peaks can be considered as a certain parameter, characterizing the crystallinity of studied material. The smaller intensity of X-ray diffraction peaks at the same film's thickness is, the worse is the crystallinity, i.e. the bigger is the contents of fine dispersion amorphous-like phase in studied film. It was found that the contents of fine dispersion amorphous-like phase in doped films is increased in the line SnO₂:Ni, SnO₂:Fe, SnO₂:Co, SnO₂:Cu. It was shown that thermal unstability of SnO₂ film structure in temperature range 600-800 °C is connected with the presence of this phase.

15:50 Poster H/PII.06

Skew asymmetric arrangement of X-ray diffraction for structural diagnostics of multi-layer semiconductor materialsZbigniew Świątek¹, Igor M. Fodchuk², Elżbieta Bielańska¹, Marek S. Michalec³**1.** Polish Academy of Sciences, Institute of Metallurgy and Materials Sciences (IMIM PAN), Reymonta 25, Kraków 30-059, Poland **2.** Chernivtsi National University (ChNU), 2 Kotsubinsky Str., Chernivtsi 58012, Ukraine **3.** Jagiellonian University, Faculty of Chemistry, Ingardena 3, Kraków 30-060, Poland

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Progress in fabrication of the optoelectronic devices based on epitaxial thin-films and semiconductor multilayer structures, requires an improvements of techniques used for the material characterization.

The method in a skew asymmetric arrangement of X-ray diffraction in Bragg geometry for a structural diagnostic of defected and multi-

layers semiconductor materials is presented. New possibilities of this method for a layer-by-layer visualization of structural changes in sub-surface crystal layers are demonstrated for semiconductor materials after various types of surface treatments such as a chemical etching, laser irradiation and ion implantation.

The lattice strain and structural changes in materials produced by existing and introduced defects were characterized by a combination of X-ray topography and diffraction techniques using single- and double-crystal configurations in the symmetric, asymmetric and skew-asymmetric geometries. Finally, to visualise of microstructural changes and to compare with their theoretical models, X-ray structural investigation was accompanied by transmission electron microscopy, scanning electron microscopy, atomic force microscopy and secondary ion mass spectroscopy measurements.

To build a full possible picture of microstructure of such materials, the simulation and interpretation of diffraction reflection curves of crystalline structures with a heterogeneous near-surface layer have been performed on the basis of a kinematics diffraction theory, analytical solutions of the Takagi-Topan equations in semi-kinematics approximation and generalized dynamic theory of X-ray scattering. Moreover, additional simulations of processes of defects generation in model materials caused by an ion implantation and dopant diffusion were carried out.

The presented method in this skew-asymmetrical mode is both a recommendable and useful tool for studying epitaxial thin films as well as multi-layer systems.

15:50 Poster H/PI.06

Spectroscopic cathodoluminescence study of nanostructured SnO₂ films deposited by spray pyrolysisGhenadii Korotcenkov¹, Mihail Ivanov¹, Iurie Blinov¹, Mihail Nazarov²**1.** Technical University of Moldova (TUM), Bld. Stefan cel Mare, 168, Chisinau 2004, Moldova **2.** Gwangju Institute of Science and Technology (GIST), 1 Oryong-dong, Buk-ku, Gwangju 500-712, Korea, South

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Carried out research has shown that the shape and cathodoluminescence intensity of nanostructured SnO₂ films is the reflection of structural state of analyzed object, and therefore the measurement of CL is effective method for characterization of thin metal oxide films, which have low photoluminescence intensity at room temperatures. It was found that cathodoluminescence of undoped and (Cu-, Fe-, or Co)-doped SnO₂ films, deposited by spray pyrolysis, is characterized by the presence of several broad emission bands in the range of the wavelength 370-650 nm, which intensity depends on deposition conditions and the following thermal treatment. The most intensive band on CL spectra of as-deposited films is the one at 500 nm. At that the growth of pyrolysis temperature is accompanied by a considerable increase of the CL intensity. It was established that SnO₂ doping by Cu, Fe, or Co leads to the decrease of CL and PL intensity. On the contrary, the annealing promotes the increase of the CL intensity, which is connected with improvement of SnO₂ films' crystallinity. After annealing at T~850 °C, an intensive band peaked at 370 nm appears in spectra for films with big size of crystallites. At that the doping does not change general character of CL spectra change, observed during annealing process. It was suggested the mechanism, explaining changes taking place in CL and PL spectra. It was made an assumption that observed transformation of CL spectra is connected with the change of both grain size and concentration of structural defects in SnO₂ surface layer.

It was concluded that the radiative recombination takes place through shallow donor levels, associated with oxygen-vacancies, and trapped centers, forming deep levels, located at 0.8-0.9, 1.35-1.45 and ~ 1.6 eV from the top of valence band.

15:50	Poster	H/PI.07

Influence of Nd dopants to change of lattice parameters in YVO₄ single crystals.

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YVO₄ single crystals (yttrium orthovanadate) are interesting materials for laser and optical application. The pure YVO₄ single crystal (crystallized in D_{4h}¹⁹ - I₄/amd space group) can be used for making polarizers, beam displacers and optical isolators due to its large birefringence ($n_e - n_o = 0.223$ at 1.06 mm). Compared with the YAG laser crystals, the Nd-doped YVO₄ crystal has a low pumping threshold, large emission section (2.7 times at 1.06 mm), large absorption coefficient (4 times), wide absorptive area at 809 nm and strongly polarized laser output and low susceptibility to gamma and electron irradiations. It is an excellent material for making the diode-pumped solid lasers.

The influence of neodymium doping of YVO₄ single crystals has been studied. These crystals have been grown by means of Czochralski method. One of them has been pure YVO₄, others have been doped with the Nd (YVO₄:1% at. Nd, YVO₄:2% at. Nd and YVO₄:5% at. Tm+0.4%Ca).

The precise changes of the lattice parameters were determined by the X-ray diffraction method – the Bond method ($\delta d/d = 10^{-6}$). The lattice parameters was measured on symmetric reflections 800 and 008 CuK α_1 , $\lambda = 0.15405929 \pm 5 \times 10^{-7}$. We have observed increase of the lattice parameters with the increasing concentration of Nd atoms and slighty changes of the unit cell volume. These changes are connected with site location of the atom dopants in YVO₄ crystal structure.

15:50	Poster	H/PII.08

Effect of high pressure annealing on defect structure of GaMnAs

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A rapid development of spintronics is observed recently. This exciting new field of research combines magnetism and electronics or optoelectronics. The best materials are so called diluted magnetic semiconductors, i.e. mixed crystals based on classical semiconductors, with a controlled fraction of nonmagnetic atoms being substituted by the magnetic ones, e.g. in GaMnAs.

The 001 oriented GaMnAs layers were grown by the MBE method. The samples were subjected to processing at temperatures T , up to 650 K for 1h under 10^5 Pa and up to 920 K under high hydrostatic pressure ($HP = 1.1$ GPa) in argon atmosphere.

The defect structure of samples was determined by high resolution X-ray diffraction methods using conventional and synchrotron radiation sources (HASYLAB-DESY). Synchrotron radiation in the grazing incident diffraction geometry was applied to examine the near-surface region of samples. Conventional X-ray investigations were carried out using MRD-PHILIPS diffractometer in the double and triple axis configurations. Lattice parameters for GaMnAs, before and after the treatment, were measured. Reciprocal space maps for (004) symmetrical reflections were registered. Roughness in the direction perpendicular to the surface and Mn depth profiles were determined using Atomic Force Microscopy and Secondary Ion Mass Spectroscopy, respectively.

The temperature-pressure treatment makes it possible to modify the GaMnAs structure. The high pressure treatment effects in changed diffusivity of Mn interstitials in GaMnAs. Enhanced pressure applied during annealing of the MBE grown layers results in increased strain. Changed strain, from the compressive to tensile one, can be related to a creation of zincblende Mn(Ga)As nanoclusters. A creation of nanoclusters under HP - T conditions is more effective than that caused by annealing under 10^5 Pa.

Acknowledgements:

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15:50	Poster	H/PI.08

Sub-pixel detection of a grid's node positions for optical diagnostics

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Determination of the position of a square grid's nodes is a frequently encountered image processing task in optical metrology and diagnostics. For example, many optical shape metrology tools use a grid projected onto the object; further, grids are used in instrumentation and processes for alignment purposes. The accuracy of the grid detection greatly influences the accuracy of the whole measurement system and the speed of the grid detection determines the throughput of the measurement or process.

In the present paper, we describe a complete grid detection algorithm. Our research is motivated by application in quantitative Makyoh topography [1]; this method uses the projected image of a grid to determine the height topography of closely planar mirror-like surfaces. First, the software generates the correlation function using a special (cross-like) weight function, then, it finds the local maxima of the correlation function with one-pixel accuracy. Then, the neighbourhood relations are determined for the proper ordering of the data into a matrix. Finally, the algorithm refines the positions of the detected grid points with sub-pixel accuracy.

We have developed several versions of the algorithm for different tasks. We study and compare the accuracy and speed of the algorithms as a function of grid geometry, grid distortion, grid defocus, image noise and contrast using simulated images. Experimental application

in quantitative Makyoh topography using semiconductor wafers are demonstrated as well.

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15:50	Poster	H/PII.09

X-ray study of Ge nanoparticle formation in Ge:SiO₂/SiO₂ multilayers

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We present the study of the multilayer germanosilicate (Ge:SiO₂) films which have been grown at room temperature, by magnetron sputtering with Ar as a working gas. Each Ge:SiO₂ (molar ratio: 60:40) layer is separated by a pure SiO₂ layer with equal thickness (7.2 nm). The samples were then annealed for 1h at different temperatures Ta (up to 900 °C) which produces Ge nanoparticles. It is shown that the morphology of such a heterogeneous material can be well interpreted by combining the information obtained from the three techniques: grazing incidence wide/small angle x-ray scattering (GIWAXS/GISAXS) and x-ray reflectivity (XR). The analysis of GIWAXS data revealed that the formation of Ge nanoparticles starts at Ta = 600°C and that they have maximum fraction at Ta = 750 °C. XR superlattice reflections revealed that the period of the multilayer stack decreases for annealed samples: for Ta = 600-750 °C by ~1.6 nm (as a result of Ge crystallisation), and for higher Ta by additional 1 nm (crystallisation + out diffusion of Ge atoms from the sample). 2D GISAXS patterns evidence a high degree of vertical alignment of the nanoparticles along whole multilayer stack for Ta = 600-800 °C. The average size of nanoparticles and their in-layer correlation are obtained using Guinier approximation and the position of diffuse scattering maxima, respectively.

15:50	Poster	H/PI.09

Reconstruction of lattice structure of ion-implanted near-surface regions of HgCdTe epitaxial layers

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It has been presented the results of AFM, SEM, X-ray and SIMS studies of near-surface regions of HgCdTe graded-gap epitaxial layers obtained by high-temperature annealing of them in the vapour of the main components. We used grown on CdTe substrates the ISOVPE layers of HgCdTe whose surface was implanted by arsenic ions. The AFM investigation has showed that the morphology of surfaces of HgCdTe structures obtained at the same conditions is significantly influenced by the crystal orientation of the initial substrates of CdTe. By means of SIMS and SEM analyses it has been observed a substantial

increase of the molar content of HgCdTe solid solution on the surface of ion-implanted epitaxial layers after high-temperature annealing. For X-ray investigation we used the technique of two-crystal spectrometry at skew-asymmetrical setup of the sample under study. On the basis of the observed structural changes taking place at different angles of azimuth scanning it has been made comparison of the calculated profiles of deformations and the experimental SIMS distribution of the main components of HgCdTe solid solution in the near-surface regions of the structure. Large gradients of composition HgCdTe solid solution have been shown to be formed in the near-surface regions of the epitaxial layers due to small changes in the thermodynamically equilibrium conditions of the process of high-temperature annealing. These gradients are the cause of formation of graded-band-gap structure in which strains of lattice crystal are present. The influence of these factors on the diffusion of an electrically active impurity is analyzed.

15:50	Poster	H/PII.10

Methods of stress investigations in dielectric layer of MIS structures

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In this work, results of stresses measurements in dielectric layer under the gate area of metal-insulator-semiconductor (MIS) structures obtained by means of several methods are presented. The objects of our investigations were Al/SiO₂/Si (traditional), Al/La₂O₃/Si and Poly-Si/SiO₂/Si systems. In these studies, the methods such as interferometry, profilometry and spectroscopic ellipsometry have been applied.

Interference measurements allowed determination of curvature radius of oxidized silicon substrate (by Fizeau interferometer) and analysis of the gate area and the gate vicinity (by MII-4 interferometer). Contact method (by alpha step 200 profilometer of Tencor Instruments Company) gave the directly profile of gate surface and its surrounding. Ellipsometric method (by variable angle spectroscopic ellipsometer) allowed determination of thickness and refractive index of the dielectric layer. In this paper, we mainly focused on the investigation of changes in the shape of the gate surface which occurs sometimes after annealing of the gates at temperature of 450°C. To analyses of obtained measurements results, model based on Stoney formula and its later modifications were used.

15:50	Poster	H/PI.10

Anisotropy of sapphire tube-shaped single crystal

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Dependence of mechanical properties of sapphire single crystal on crystallographic orientation was investigated with the tube-shaped samples. Three different parameters were measured: longitudinal sound waves speed, surface roughness and X-ray rocking curve width. The data show a strong correlation between the measured parameters. Evaluation of relative anisotropy coefficient for major orientation planes in sapphire possesses to characterize them as: C-plane – the most isotropic and the A-plane – the most anisotropic. C-plates (C-axis is normal to the plate face) have the highest isotropy and non-birefringence widely used in protective windows working under static

or dynamic load. In addition high isotropy of C-plates helps to achieve the highest requirements to the window form (flatness, waveform distortion). Rather close results can be achieved on M-plates.

The maximal surface roughness and minimal sound speed characterize R-plane. This coincides with the minimal value of Young modulus in [01-12] direction. R-plane can be recommended for use under through thickness directed thermal gradient working conditions. The anisotropy of A-plates ([1-210] axis is normal to face) leads to aggravation of polished surface flatness and strength.

The experimental method proposed within this study has been proven to be effective in evaluation of angle dependence of different parameters of single crystals. Influence of both material non-homogeneity and machining conditions variation is minimal.

15:50	Poster	H/PII.11

Laser diffraction and optical spectroscopy study of colloidal crystal films

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It is well-known that single crystal electron diffraction is a powerful tool for visualization and analysis of crystalline structures of atomic and molecular crystals. However, despite the analogy of colloidal crystals (also referred to as opals, when made of self-assembled silica or latex microspheres) and ordinary crystals, similar laser diffraction experiments on opals were reported only in a few works.

In the present work we report a laser diffraction and optical spectroscopy study of colloidal crystal films based on polystyrene or silica microspheres. The films were grown by vertical deposition method employing self-assembly of colloids onto vertical substrate. Optical spectra at different angles of incidence were recorded both in transmission and reflection modes. In diffraction experiment we varied the geometry and the wavelength of radiation using a tunable laser.

Diffraction phenomena in colloidal crystals are shown to be very complex. When measuring optical transmission spectrum of opal film in normal direction, a number of peaks, corresponding to diffraction of light on different families of crystallographic planes in a 3D structure of close-packed microspheres, can be observed. At the same time, each individual hexagonal close-packed layer can act as a 2D diffraction grating resulting in sixfold diffraction pattern when illuminating colloidal crystal film with a light of proper wavelength. A very special attention has been paid to angle-dependent laser diffraction experiments, in which we monitored the intensity of diffraction spots as a function of laser beam angle of incidence. Such study provides information on layer stacking in close-packed structure of colloidal crystal film, which is unattainable by conventional microscopy methods.

15:50	Poster	H/PI.11

Influence of Illumination and Decay of Electrical Resistance of ITO Nanoscale Layers

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Indium tin oxide (InSnO_2 , ITO) is known as a transparent oxide with n-type electrical conductivity. However, the as grown ITO layers have

high resistivity and the transparency is also very limited. In this work ITO layers were deposited by evaporation and then underwent a post-growth annealing. Annealing leads to low electrical resistivity and to an enhanced transparency. Annealed samples show n-type conductivity.

Since sensor chips used in optical waveguide lightmode spectroscopy (OWLS) can be coated with various solid layers in order to achieve either simply improved or more selective sensor sensitivity, ITO coating layers are envisaged for measurements where the layer conductivity plays an important role. One of such cases is when the optical sensitivity can be combined with specific advantages of the electrochemical processes.

ITO layers of typically 10 nm thickness were deposited onto OWLS sensor chips and on SiTiO covered glass substrates. The basic measurement after the annealing was the conductivity determination. On sensor chips also the angle dependent photocurrent spectra were evaluated. This allows the selection of the chips.

The electrical conductivity was estimated first by two point direct resistance measurements, then by van der Pauw configuration and by collinear four point probe method. W needles, Sn containing mechanical contacts, In, Ga and In+Ga wetting contacts, and some alloyed contacts (In, Au, Sn) were used. All types of contacts proved to be applicable. Also the repetitivity of the technological processes was controlled partly by electrical measurements.

The storage time dependent stability and light sensitivity were studied. It is demonstrated that the resistance decreases due to the illumination, but in a very small extent. The decrease depends on the wavelength and the process is very slow (tens of minutes, up to hours). The recovery is also slow and also some drift in resistance is observed under continuous repetitive measurements.

15:50	Poster	H/PI.12

Surface Scattering Optical Loss Measurements in Thin Oxide Planar Waveguide Layers

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In the case of optical waveguides, there are some typical problems, like incoupling of the laser light and optical losses in the waveguide. There are great differences also in these meanings between the fibbers and planar waveguides. In the case of optical waveguide lightmode spectroscopy very thin, nm scale transparent oxide layers are used as planar waveguides. In our case the incoupling of the light is realised using a grating on the surface of the layer. On the other hand, the optical losses can be significant in thin layers, which can remarkably decrease the sensibility and remarkably increase the noise in the measurements performed on these sensors. This effect is emphasised in this case, since such waveguide sensors are primarily sensitive to the surface contaminations.

The thickness of the sensitive oxide layers is about 200nm and/or 10-20nm. The overall transparency measurements performed perpendicular to the surface are not sufficiently sensitive to characterise losses in the layer under investigation.

In this work efforts made to characterize or visualise losses of the incoupled light propagating in the plane of the waveguide layer. An experimental setup was built for such measurements. The light is incoupled via the grating and the light scattered by various centres and scattered out from the layer is studied along the layer. First results are presented in this paper.

The first observations showed already that not the losses caused by density, refractive index or similar inhomogeneities play dominating role, in agreement also with some literature data. Scattering or refractive effect of the surface and interface roughness plays decisive role in the case of such thin planar waveguides. This is important not only in the optical losses, but also in increase of noise level of the measurements. The scattered light, the position of the scattering centres, etc. are observed by a CCD camera and the corresponding software allows also some numerical evaluation.

15:50	Poster	H/PII.12

An RBS Study of Thin PLD and MOCVD Strontium Copper Oxide Layers

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Strontium copper oxide (SCO) layers have been extensively studied as p-type transparent (VIS) conductive oxide material. Also theoretical studies suggested p-type conductivity of the SrCu_2O_2 composition. This oxide is promising also in replacing of oxides containing an In component.

SCO thin layers, with thicknesses 50 to 1500 nm were deposited either by pulsed laser deposition method, or by MOCVD. According to the needs of various measurements, both glass and silicon substrates were used. The as grown layers showed high electrical resistance. Due to an annealing process the resistivity was significantly decreased and the layers showed p-type conductivity. Optical transparency was measured on samples grown on glass substrates and it was found about or above 80 % was found, including also some thickness dependence.

These layers are intended to be used in various devices, the structure and composition of the oxide have great importance. The study of the surfacial and, especially, interfacial regions is difficult, partly because of the small layer thicknesses. RBS measurements were applied for the determination of the chemical composition profile of the layers. These measurements included samples grown with the mentioned technologies and substrate types. In this study one pair of halves of a PLD sample was analysed such a way that one half was left in as grown state and the second one was annealed. This unique comparison revealed some specific differences between these halves. Furthermore, a vertical inhomogeneity was observed demonstrating an effect of the interface.

Measurements revealed also differences between samples grown by the two different deposition techniques, though the substrates were also different. Special modelling evaluation software was also developed with the aim to facilitate choice of the experimental conditions for the real measurements. This way conditions and requirements for the live experiment can be formulated more precisely and more quickly.

15:50	Poster	H/PII.13

Advances in X-ray Characterization.

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The continued scaling of silicon-based device is rapidly reaching the physical limit of Moore's law and the semiconductor industry will soon replace the traditional CMOS (Complementary Metal-Oxide Semiconductor) technology with new materials and processes. Films of few nanometers thicknesses, quantum dots, quantum wires are the basis of the modern electronic industry and X-ray diffraction techniques will play a new and very important role as a powerful characterization tool for determining the detailed structural information of ultra-thin film such as: the evolution of strain relaxation, defect formation, interfacial properties between film/substrate, the effects of the reduced dimensionality and their correlation to the physical properties (transport and magnetic). Materials of technological interests are: SiGe, exotic oxides, high and low K dielectrics, new materials for interconnects and formemory storage/spintronics, organic thin films.

In this talk, few representative systems are chosen from the broad category of materials (semiconductors, metals, oxides and organic thin films) to illustrate some of the unique features of the different X-ray scattering techniques to modern material problems in the electronic industry

15:50	Poster	H/PI.13

Luminescence properties of $\text{Ca}_3\text{MgSi}_2\text{O}_8:\text{Eu}^{2+}$ thin film phosphors by a pulsed laser deposition

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It is generally agreed that the phosphorescence of Eu^{2+} in most of hosts is believed to be caused by the 4f-5d transitions, and the peak positions in the mission spectra depend strongly on the nature of the Eu^{2+} surroundings in host lattices. Alkaline earth silicates are useful luminescent hosts with stability, and therefore, a great deal of work has been done on rare earth ion activation of these hosts. $\text{Ca}_3\text{MgSi}_2\text{O}_8$ and Eu^{2+} ion have been attracted as a host material and an activator for phosphor, respectively. In $\text{Ca}_3\text{MgSi}_2\text{O}_8$ crystal structure, there are two kinds of coordination number for Ca^{2+} , and so the corresponding r is 0.112 nm ($n=8$) and 0.118 nm ($n=9$), respectively. And r is 0.065 nm for Mg^{2+} with the coordination number 6. $\text{Ca}_3\text{MgSi}_2\text{O}_8$ thin films have been grown on various substrates using a pulsed laser deposition technique. The substrate was Al_2O_3 (0001). Thin films were grown under the various oxygen pressures and various substrate temperatures. The crystalline structure, surface morphology characteristics, chemical states, and photoluminescence properties of the Eu-doped $\text{Ca}_3\text{MgSi}_2\text{O}_8$ films were analyzed using X-ray diffraction (XRD), scanning electron microscope (SEM), and atomic force microscopy (AFM), respectively. The films grown at various deposition conditions show the different crystallinity, surface morphology, and luminescent properties. The

room temperature photoluminescence spectra were dominated by the emission peak at 477 nm radiated from the transition of 4f-5d within Eu^{2+} ions. We found that the luminescence spectra of the films are highly dependent on the crystallinity and surface roughness of the films. And the surface roughness and luminescence intensity of the films showed similar behavior grown on various deposition condition.

15:50 Poster H/PII.14

Lifetime improvement of organic light-emitting diodes using silicon oxy-nitride as anode modifier

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Silicon oxy-nitride (SiO_xN_y) films prepared by radio-frequency magnetron sputtering were investigated as an anode modifier in organic light-emitting diodes (OLEDs). SiO_xN_y films were deposited between ITO and the hole transport layer of an OLED with a configuration of indium tin oxide (ITO)/ SiO_xN_y /α-naphthylphenylphenyl diamine (NPB)/8-hydroxyquinoline aluminum (AlQ)/Mg:Ag. By varying the argon and oxygen flow ratio during deposition of SiO_xN_y films, devices with improved electroluminescent performance and operation lifetime were obtained. The atomic composition of the SiO_xN_y films were analyzed by X-ray Photoelectron Microscopy. The best device with the optimal SiO_xN_y film showed a half brightness lifetime 5 times better than the control device.

15:50 Poster H/PI.14

Optical properties of nanocrystalline titanium oxide

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The optical properties of titanium oxide were investigated in relationship with the presence of chemically-optically active centers and their evolution in argon, oxygen and hydrogen ambient. Samples of nanocrystalline TiO_2 powder were annealed at 1100°C for 30 min., under argon atmosphere. The as-obtained polycrystalline material was further subjected to two types of treatments: *i*) at 800°C for 30 and 120 min., respectively, under oxygen, and *ii*) at 750°C for 30 and 120 min, respectively, in forming gas with various concentrations in hydrogen, from 10% up to 70%.

The optical emission spectra were measured by cathodoluminescence in the scanning electron microscope (SEM-CL) and analysed in correlation with the structure and morphology of the surface, which were investigated by X-ray diffraction and SEM.

The nanocrystalline TiO_2 powder showed a broad emission spectrum in the visible region with the main emission peak at 450-500 nm and several small peaks situated between 400 and 620 nm. As the effect of the treatment under argon, besides the visible emission at 480-550 nm, a well resolved emission band appears in the near infrared region whose peak is present at 720 nm.

We assign the optical emission bands evidenced in the visible region to oxygen vacancy centers. The peak intensity varies as a function of duration and ambient of the treatment. The emission band at 450 nm

was found to be sensitive to treatment in oxygen, while the emission band at 480 nm was highly sensitive to both duration of treatment in hydrogen and the hydrogen content.

The optical emission band situated in the near infrared region is associated with the presence of titanium ions. The treatments in hydrogen and oxygen determines the shift of the peak to 820 nm. The structure of polycrystalline TiO_2 and the growth phenomena of protrusions, terraces and polygonal structures on the polycrystal surfaces as a result of treatments under oxygen and hydrogen are discussed.

15:50 Poster H/PII.15

Investigation of lanthanum and hafnium-based dielectric films by X-ray reflectivity, spectroscopic ellipsometry and X-ray photoelectron spectroscopy

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The use of insulating materials bearing higher dielectric constant than that of silicon oxide in CMOS devices is compulsory for all technology nodes requiring gate dielectric with EOT smaller than 1 nm. Two of potential candidates are LaAlO_3 and HfAlO . Investigation of film and high-k/Si interlayer composition by several non-destructive optical methods is a possible way to control the integrity of the performed structure during the fabrication process. We discuss here the morphology and composition of LaAlO_3 and HfAlO thin films accessed by X-ray reflectometry (XRR), spectroscopic ellipsometry (SE), and X-ray photoelectron spectroscopy (XPS). LaAlO_3 and HfAlO gate dielectrics were sputtered from ceramic target and deposited on HF last clean or thermally nitrided Si. Experimental XRR profile and its associated Fast Fourier Transform were used to extract absolute thicknesses, densities and interfacial roughnesses of thin films. Absolute thicknesses given by XRR were used in SE measurements to determine optical constants and composition of the films. XPS complementary analysis was performed to analyze the chemical bondings in thin films, in particular at the high-k/substrate interface.

A three-layer model was found both in XRR and SE analysis to be the best optical model for as-deposited and annealed films. It was composed of an interfacial layer, covered by a layer of pure LaAlO_3 or HfAlO , and a surface-roughness layer. XRR analysis showed that the dielectric/Si interface of the $\text{HfAlO}/\text{SiON}/\text{Si}$ structure had a significantly smaller roughness than those of HfAlO/Si . SE studies revealed that in the case of LaAlO_3 and HfAlO deposited on Si, the interfacial layer is a mixture of high-k material and amorphous-Si inclusions. The SiON-like interface did not change significantly after thermal annealing in O_2 . XPS also pointed out the superior diffusion barrier properties brought by SiON/Si substrates before LaAlO_3 , and to a greater extent, HfAlO deposition.

15:50 Poster H/PI.15

Hydroxyapatite films obtained by sol-gel and sputtering methods

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Hydroxyapatite (HA) biocoatings were obtained using two different deposition methods (sol-gel and sputtering). The depositions were performed on bare silicon or covered with a TiO₂ buffer layer. Comparison of physico-chemical properties between different samples prepared by sputtering and sol-method, as well as between films deposited with and without TiO₂ buffer layer is a main subject of this paper. The microstructural, chemical and optical characterizations of the films were performed by IR, XPS, UV-VIS Absorption and Ellipsometric Spectroscopies. The studied HA/Si films are highly transparent and colorless, owing to low refractive index of hydroxyapatite (1.556 at wavelength 590 nm, as estimated from ellipsometric data). Interference color, which depends on refractive index and thickness, was slightly observed increasing film thickness from 160 to 2500 nm. Ca/P atomic ratio of 1.7 (specific for HA) was evaluated from XPS spectra. Silicon signal derived from substrate or any other signals from impurities except carbon from surface were not observed. Similar FTIR spectra recorded on sputtering and sol-gel films were obtained. FTIR measurements showed two peaks from 550-600 cm⁻¹ and 1000-1200 cm⁻¹ which are attributed to the major absorption modes associated with the presence of phosphate (S. W. Russell et al., J. Am. Ceram. Soc. 79 (1996), 837). Films annealed at 400-550°C showed a band at 1600 cm⁻¹ and one more faint at 861 cm⁻¹ related to the CO₃²⁻ ions. The OH⁻ group was detected by XPS analysis after O1s line deconvolution. It was found that OH⁻ group presents a relative concentration of about 15% from the total amount of oxygen. Although the investigated films show similar properties with previous published results, future improvements are necessary to reduce the carbon contamination due to organic residuals and to increase the stoichiometry of the films.

15:50 Poster H/PI.16

Advances in micro-crystalline inclusion multi-elementary analysis (12<Z<92) by coupled absolute PIXE and RBS

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Multi-elementary major to trace element analysis of light matrices by PIXE requires the use of a 'funny filter' (a filter with a hole drilled at its center placed in front of the detector window) to attenuate the X-Ray emission of the major elements. Previous work by Gama et al. (2001) showed that, assuming the main setup parameters were well constrained (charge, detector physics ...), the application of computer-aided PIXE as a multi-elementary absolute analytical method required precise knowledge of the detection geometry parameters (detection

solid angle, thickness of the filters and diameter of the hole in the filters). We have developed a simplified procedure that allows multi-elementary PIXE to be applied in an absolute fashion. It relies on the definition of an energy-dependant standardization function $G(E) = T(E) \cdot W$ (T : filter transmission; W : detection solid angle, E : characteristic X-ray energy of element Z).

Let us consider the geometrical detection parameters of a PIXE setup and a solid chemical standard the PIXE spectrum of which yields the peaks of N detectable certified elements ($N \gg p$). The computer-assisted PIXE spectrum treatment yields N values of $G(E)$, from which the p detection parameters can be adjusted by a least-square fit procedure. The method is illustrated using a natural basalt standard provided by the SARM laboratory (Nancy, France). 17 certified elements from Al to Ba are detected on its PIXE spectrum using an Al-Be 'funny filter'. The 4 geometrical detection parameters are adjusted to within ± 2 to 6 % on the standard spectrum using the Levenberg-Marquardt algorithm.

We show that, by combining RBS and PIXE measurements, quantitative PIXE can be applied to intracrystalline inclusions placed within the range of protons, as RBS spectrometry provides the geometrical information needed for quantitative elemental analysis of occluded phases.

15:50 Poster H/PII.16

New results in calculating critical thickness and the degree of relaxation for epitaxial grown Silicon-Germanium layers on Silicon

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Due to the higher lattice constant of SiGe compared to Silicon, an epitaxial SiGe layer deposited on Si will be stressed compressively. When a certain thickness value has been reached, called *critical thickness*, the layer will start to relax. This fact is well known since the early beginning of SiGe epitaxial growth. There were many investigations to calculate the critical thickness of combined semiconductor structures. Unfortunately, the results differ by more than one order of magnitude for Ge concentrations which are of interest for the semiconductor industry. Since the benefit of strain engineering by SiGe has come to the focus of scientific and industrial research, critical thickness, especially for SiGe, became an important topic again.

We have achieved new results describing the behaviour of LPCVD epitaxial grown SiGe layers on 200mm Si substrates. The SiGe layers deposited have various Germanium concentrations up to 45% as measured by AES and XRD. The measured values for critical thickness, i.e. where the onset of relaxation could be identified, show a good correspondence between the calculated values by Cohan-Solal. We also calculated the degree of relaxation by XRD measurements. We derived an equation which describes this value in dependence of the germanium concentration and layer thickness pretty accurately. Furthermore, when the SiGe layer starts to relax, its surface roughness as been measured by AFM increases drastically and typical crosshatch

patterns can be observed on the surface. This is in particular true for small relaxation values.

The project was funded in line with the technology funding for regional development (EFRE) 2000-2006 of the European Union and by funds of the Freestate of Saxony.

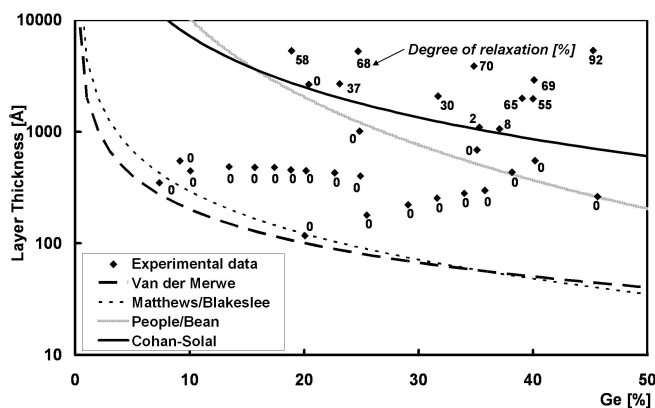


Fig. 1. Degree of relaxation of $\text{Si}_{(1-x)}\text{Ge}_x$ layers in dependence of layer thicknesses and Ge concentration. Comparison between experimental data and different theoretical descriptions

15:50

Poster

H/PII.17

Influence of the substrate orientation on the composition of solid solutions grown by LPE and MOCVD

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It has been shown that the segregation coefficients in III-V ternary systems depend on substrate orientation. This effect was observed, in particular, in the system GaInP grown on GaAs substrates by LPE. All articles studying this effect were based on results obtained growing on substrates with different orientations. In these studies the liquid phase composition was changed in each experiment. Analyses of their growth methodology show that important parameters such as the initial supercooling and liquidus temperature can differ from one experiment to another. These uncontrolled growth parameters introduce, naturally, some uncertainty on the results. Moreover the evaporation of phosphorus from the liquid phase can change its composition and this could be a reason of another experimental error.

All these errors can lead to different conclusions and even some authors do not observe any effect of the substrate orientation at all. In this work, to eliminate these uncertainties, we have grown simultaneously GaInP on GaAs substrates with different orientations but from the same liquid phase. In this way we are sure that the growth conditions are the same for different substrates even if the liquidus temperature and supercooling are not exactly known. The samples obtained were studied by HRXRD, photoluminescence and SIMS. It was found that the Ga concentration in the epitaxial layers diminishes in the order (100) > (111)A > (111)B. Additionally, analogue experiments were done by MOCVD using as substrates GaAs (100), (111)A and (111)B growing the GaInP simultaneously during the same growth experiment. The results were very similar to those obtained by LPE.

15:50

Poster

H/PI.18

Highly oriented doped and undoped tin oxide thin films grown on multicrystalline silicon substrate.

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Abstract

Polycrystalline antimony doped and undoped tin oxide thin films are grown by chemical vapor deposition on multicrystalline silicon and on glass substrates. The latter substrate was considered for comparative study on growth and characterization. The doping was achieved with various content of Sb in the starting material. X ray diffraction patterns let us to find preferential growth which is not the same according to the substrate. On the glass substrate the grain grows in the (200) crystallographic direction while it grows in the (110) orientation when deposited on polycrystalline silicon substrate at the same time and in the same reactor. The resultant films possess a columnar microstructure perpendicular to the substrate and exhibits grains with nanometer size. The sheet resistance measured on doped layers are significantly improved compared to that obtained on undoped ones, in the same conditions i.e. substrate temperature $T_s=440^\circ\text{C}$, duration of the deposition 10 min and flowing oxygen about 2L/min. Contrary to results obtained on tin oxide layer deposited on monocrystalline silicon in previous work the microstructure seems to be dependant on the kind of the substrate.

15:50

Poster

H/PII.18

Single crystals of gallium nitride doped with gadolinium-synthesis and properties.

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During recent years rapid development of investigations over diluted magnetic semiconductors (DMSs) is observed. Gallium nitride is well known semiconductor, dopant of gadolinium in GaN lattice could result in ferromagnetic properties in this material. Mixture of gallium and gadolinium metals was used as a substrate in presented in this work research. Experiments were carried out in horizontal reactor with resistance heating at temperatures from 1150 to 1250°C in an ammonia atmosphere. Measurements involving SEM, Raman studies and EXAFS were used to investigate the single crystals obtained.

15:50

Poster

H/PII.19

Makyoh-topography study of the swirl defect in Si wafers

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Swirl defects in heavily *p*-doped CZ Si single crystals have been known for a long time [1, 2]. Such defects are spiral features that are generated by the agglomeration of point defects while the crystals cools after pulling. Swirl defects are usually studied by microscopy after preferential etching and various X-ray methods. Swirl defects are detrimental to device quality and yield.

Makyoh topography (MT) is an optical method for the qualitative observation of the surface topography of mirror-like surfaces. The studied surface is illuminated by a collimated light beam, and an image is projected onto a distant screen, where an image is formed due to the focusing/defocusing action of the morphological features of the surface [3]. MT has been used in semiconductor technology from the 90's, mainly for the assessment of the polishing quality of wafers.

Although swirl defects have also been observed by MT [4], a systematic study is still lacking. The aim of the present paper is just to perform such a systematic investigation. We compare two series of *p*-type wafers of varying dopant concentration from different vendors. We show the unambiguous correlation with the high dopant concentration, and point out the differences between the wafers of the two vendors. Other defects are also observed and the possible origins are discussed.

The results are interpreted using a geometrical optical model of MT imaging of sine surfaces which predicts the observed contrast as a function of surface feature amplitude and period [5]. The results are compared to surface stylus measurements, and the possibility and limitations of the extraction of quantitative data are discussed.

1. P. M. Petroff, A. J. R. De Kock, J. Cryst. Growth 30 (1975) 117.
2. T. W. Fan, J. J. Qian, J. Wu, L. Y. Lin, J. Yuan, J. Cryst. Growth 213 (2000) 276.
3. F. Riesz, Proc. SPIE 5458 (2004) 86.
4. D. Korytár, M. Hrivnák, Jpn. J. Appl. Phys. 32 (1993) 693.
5. F. Riesz, Mater. Sci. Semicond. Processing, 9 (2006) 220.

15:50	Poster	H/PI.19

Transparent thin-film transistors with zinc oxide active layer fabricated using metal zinc

Woo-Seok Cheong, Chi-Sun Hwang, Min-Ki Ryu, Jae-heon Shin, Sang-Hee Ko Park, Doo-Hee Cho, Chun-Won Byun, Shinhyuk Yang, Sung Min Yoon, Jeong-Ik Lee, Hye Yong Chu, Kyoung Ik Cho

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In this study, transparent thin-film transistors (TTFTs) with active channel of zinc oxide (ZnO) were fabricated, where as well as bottom-gated structures, top-gated structures were used. The electrode was ITO, and gate insulator was Al₂O₃ by atomic layer deposition. ZnO films were made by two methods; one is the oxidation of sputtered metal Zn film, where Zinc could be oxidized by various methods such as furnace anneal, plasma treatment, and UV-lamp heating process, while the other is radio frequency (RF) reactive sputtering method using metal zinc target. Reactively sputtered ZnO films were formed under the condition of different working pressures, gas ratios (oxygen/oxygen+argon) and rf powers. The band-gap energies of reactively sputtered ZnO films were changed by both deposition condition and post-anneal condition, which affected device properties. ZnO films from both methods had good optical transmittances at 550nm (around 80% or higher). The correlation between ZnO films and the device properties was investigated by X-ray diffraction (XRD), transmission electron microscopy (TEM) and atomic force microscopy (AFM). Current-voltage (I-V) properties measured through the

gate showed that the channel was n-type, and I_{on}/I_{off} ratio was more than 10^6 .

15:50	Poster	H/PI.20

Determination of dislocation density and crystallite size distribution in deformed lath martensite by X-ray peak profile analysis

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Microstructural characteristics of extra low carbon lath martensite after equal channel angular pressing of four passes and cold rolling to 85% reduction were studied by means of X-ray peak profile analysis and transmission electron microscopy. The modified Williamson-Hall and Warren-Averbach methodologies were implemented to determine crystallite size distribution, dislocation density and character of prevailing dislocations of the deformed structures. It was found out that lath boundaries disappear at large strain which successively turn to a highly and uniformly dislocated structure. Cold-rolled steel showed lamellar dislocation cell blocks in which strong preferred orientation could be deduced from anisotropy of integral width according to the classic Williamson-Hall approach. However, equiaxed dislocation cells of random orientation were found in the equal channel angular pressed steel. It is demonstrated that further straining would be necessary to realize a bulk nanostructured steel with high fraction of high angle boundaries.

15:50	Poster	H/PII.20

Phase transformation during the annealing of thin films in Sr-Cu-O system analyzed with in situ studies by X-ray diffraction and Raman spectroscopy

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Divalent ion in a copper oxide matrix $M^{2+}Cu_2O_2$ are intensively studied for the application field of p-type transparent conductive oxide in order to achieve transparent electronic devices. In this paper, we have studied the phase transformation during the annealing of thin films in Sr-Cu-O system analyzed with *in situ* studies by X-ray diffraction and Raman spectroscopy. The Sr-Cu-O were deposited on silicon wafer by means of an injection-MOCVD apparatus. FTIR and XRD analyses have shown that the films were composed of a mixture of copper oxide (CuO) and strontium carbonate. This later compound is due to the low deposition temperature. So, to achieve the SrCu₂O₂ compound, it is necessary to anneal the samples. The ability to obtain the pure SCO phase with the different annealing has been analyzed related to the film composition with *in situ* experiments by X-ray diffraction up to

700°C under vacuum or different gaseous atmosphere and by Raman spectroscopy up to 600°C.

15:50

Poster

H/PI.21

Structure of Si:Mn annealed under enhanced stress conditions

Jadwiga Bak-Misiuk¹, Przemysław Romanowski¹, Elzbieta Dynowska¹, Artem Shalimov¹, Andrzej Misiuk², Sławomir Kret¹, Piotr Dłużewski¹, Adam Barcz^{1,2}, Jarosław Domagała¹, Wolfgang Caliebe³, Jerzy Dąbrowski¹

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Silicon implanted with Mn (Si:Mn) is magnetically ordered after appropriate annealing [1]. This work concerns the Si:Mn structure as well as its dependence on temperature during implantation, T_s , and on subsequent annealing parameters.

Si:Mn was prepared by implantation of Czochralski silicon with 160 keV Mn^+ ions at $T_s = 340$ K and 610 K to a dose $1 \times 10^{16} \text{ cm}^{-2}$. Si:Mn was processed at temperature, T_a , up to 1270 K for up to 10 h under ambient (10^5 Pa) and enhanced Ar hydrostatic pressure (HP, up to 1.2 GPa). Concentration profile of Mn was measured by SIMS; the defect structure was determined by high resolution X-Ray diffraction and diffraction in the grazing incidence geometry (synchrotron).

The near-surface area of Si:Mn implanted at $T_s = 340$ K is amorphous. In the case of implantation at $T_s = 610$ K, this area is better ordered, with well-defined layered structure, while the buried layer is polycrystalline. Subsequent annealing of Si:Mn results in re-crystallization of disturbed layer and formation of the Mn_4Si_7 phase (Fig. 1). These effects depend, among others, on T_s , T_a and HP. After processing at 1270 K, the samples implanted at $T_s = 340$ K indicated the presence of dislocation loops while vacancies and / or interstitials were formed in Si:Mn prepared at 610 K. The treatments under 10^5 Pa and under HP result, respectively, in the dominating presence of interstitials and vacancies.

Our results help to understand the origin of magnetic ordering in Si:Mn.

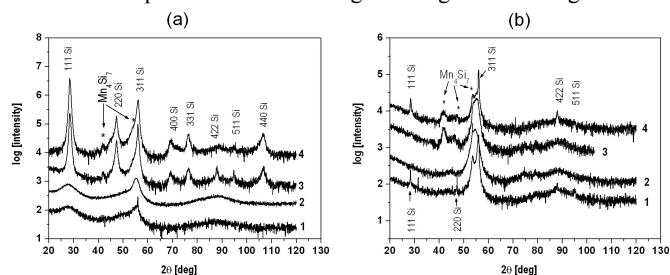


Fig. 1. Powder diffraction patterns in grazing incidence geometry for Si:Mn implanted at $T_s = 340$ K (a) and 610 K (b): 1 - as-implanted; 2 - annealed for 1 h at 610 K under 10^5 Pa; 3, 4 - annealed for 1 h at 870 K under 10^5 Pa and 1.1 GPa, respectively.

References:

[1] M. Bolduc, C. Awo-Affouda, A. Stollenwerk, M. B. Huang, G. G. Ramos, G. Agnello, V. P. LaBella, Phys. Rev. B, **71** (2005) 033302.

Symposium I

Welcome

New opportunities and challenges in material research using synchrotron and free electron laser sources is a topical subject of debate more than ever, since the first decade of this century is marked by an exponential increase of the number of new ultra-bright light facilities that are already operational or are in advanced commissioning or construction stages. The scope of this symposium is to feature the cross-disciplinary efforts and achievements in material science and technology at the Synchrotron and Free Electron Laser (FEL) laboratories, highlighting the latest progress and the emerging new fields.

The Organisers invite participants from academic, research and industrial institutions to present their results, interact and share their knowledge and discuss future research directions and development of appropriate research tools. Representatives from industry, graduate students and post-docs who wish to learn more about the new research opportunities at various Synchrotron and FEL facilities are especially welcome.

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Programme

Monday, 17 September

Opening & Plenary Session

Czochralski Award Ceremony & Acta Gold Medal Ceremony
Monday morning, 17 September, 10:00

Lunch break

Monday afternoon, 17 September, 12:30

Opening Remarks

Monday afternoon, 17 September, 14:00

Session 1

Micro and nanostructured materials: Fabrication and characterization

Monday afternoon, 17 September, 14:05

14:05

Invited

Pushing the limits of nanolithography with coherent extreme ultraviolet light from synchrotron sources

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Nanometer-scale patterns are increasingly required in many scientific and technological applications such as the continuing miniaturization of electronic circuits, sub-wavelength optics, x-ray optics, templated

self-assembly and patterned magnetic media. Extreme Ultraviolet interference lithography (EUV-IL) has emerged in recent years to become a viable technique for producing periodic nano-structures. Several EUV-IL tools based on spatially coherent undulator radiation have been set up at synchrotron radiation facilities. The highest resolution structures obtained so far using EUV-IL have a pitch of 25 nm, which rivals e-beam lithography that has been the main tool for original pattern creation at this resolution level for decades. Both diffraction and reflection optics have been used in setups to create interference fringes. Achromatic interference schemes based on diffraction gratings are particularly advantageous as they make efficient use of the wideband beam available from undulator sources. This scheme also enables considerable flexibility in the types of achievable patterns, including two-dimensional, curved, or variable-period patterns. Being a parallel patterning technique EUV-IL provides higher throughput than e-beam lithography. Other advantages over e-beam include the practical absence of the proximity effect and the potential to avoid field-stitching problems. In this talk I will outline the main features of the technique and give examples from its applications.

14:40

Oral

Quantum dot superlattices in amorphous systems

Maja Buljan¹, Uroš V. Desnica¹, Goran Dražić², Mile Ivanda¹, Nikola Radić¹, Pavo Dubček¹, Kresimir Salamon³, Sigrid Bernstorff⁴

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Influence of deposition temperature on self organized growth of Ge quantum dots (QDs) during magnetron deposition of (Ge+SiO₂)/SiO₂ multilayers is investigated. We demonstrate formation of three-dimensional QD superlattices in completely amorphous matrix for narrow range of deposition temperatures. Self-ordered growth and QD superlattice formation is explained by surface morphology of growing layer combined with diffusion mediated nucleation. In addition we show that self-ordering leads to formation of size uniform QDs and induces collective behaviour, observed as coherent acoustic vibrations of QDs.

15:00

Invited

Capturing the Moment- High Performance Magnetic Materials Assembled from Gas Phase Nanoparticles

Chris Binns¹, Steven H. Baker¹, Sharon Louch², Sarnjeet S. Dhesi³

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It is well known that gas-phase magnetic transition metal nanoparticles have enhanced magnetic moments and novel internal spin arrangements. It is also known that enhanced moments are preserved in isolated particles if they are supported on a surface or embedded in a matrix. Several experiments using XMCD have revealed that a significant proportion of the increased total moment arises from orbital magnetism. As the flux from cluster sources has increased to the point where thin films of clusters can be deposited in short times, it has become a goal of cluster-assembly to produce usable magnetic materials whose magnetisation exceeds that of conventional alloys. This inevitably involves embedding the nanoparticles at high volume fractions within a

matrix of a different material but the behaviour of dense interacting assemblies of nanoparticles is less well understood than the isolated particles. The effect of dipolar and exchange interactions between the clusters radically modifies the behaviour of the film. In addition the embedding matrix can change the atomic structure of the clusters and modify their behaviour. The talk will show how the magnetic properties of thin films of clusters embedded in various matrices develops as the volume fraction and hence the inter-particle interaction increases from the isolated cluster limit to pure cluster films. The possibility of producing materials whose flux density exceeds the Slater-Pauling limit will be discussed from various approaches.

15:35

Oral

XEOL study of ZnO and rare-earth doped ZnO nanostructures

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Extensive studies have recently appeared on the optical properties of nanostructured broad band semiconductors, due to their close dependence of morphology and optical responses. Among others, ZnO is promising for future optoelectronic devices due to its wide band-gap (3.37 eV at room temperature) and peculiar optical properties, which is also suitable as a host material for optically active impurities doping, such as rare-earth ions.

A variety of morphologies of pure and RE-doped ZnO nanostructures (RE = Eu, Tb) have been grown by metal thermal evaporation in reactive atmosphere. The structures and chemistry of the sample depend on the temperature and concentration gradient during deposition and the added doping species.

We studied light-emitting properties from different ZnO nanostructures, both in energy and time domain, using the synchrotron techniques X-ray excited optical luminescence (XEOL) and time-resolved X-ray excited optical luminescence (TR-XEOL). Besides structural aspects, we investigate the time behavior of luminescence from nanostructured ZnO(RE). In general, defect state luminescence decays much slower than the fast band-gap emission. Detailed analysis of the different decay components, in particular of the excitonic decay, give insight into the optical structural property correlation of nanostructured materials. The role of rare earth dopants is likely to be of structure nucleating nature, still the opening of new lanthanide-related optical channels is subject of ongoing investigations.

Coffee break

Monday afternoon, 17 September, 15:50

Joint Poster Session (Part I)

Monday & Wednesday

Monday afternoon, 17 September, 15:55

Session 1 (continuation)*Micro and nanostructured materials: Fabrication and characterization*

Monday afternoon, 17 September, 17:20

17:20

Invited

Advances in synchrotron based imaging with hard X-raysJosé Baruchel, Jean Susini*European Synchrotron Radiation Facility (ESRF), Grenoble 38043, France**e-mail: baruchel@esrf.fr*

A substantial fraction of synchrotron radiation-based research is being carried out today with an "imaging-type" approach, where the relevant information such as density, chemical composition, chemical states, structure, and crystallographic perfection are mapped in two, or, increasingly, in three dimensions. Techniques are clearly heading towards fulfilling the nanoscale challenge, this implying **higher spatial resolution X-ray imaging**. Many different scientific communities, such as materials science, soft condensed matter, biology, and cultural heritage, require this. High spatial resolution, beyond the detector resolution, is being achieved by nanofocused beams or by lensless coherent diffraction imaging, with a generalized use of phase contrast. A second obvious trend is the **improvement of temporal resolution**, made possible by the specific development of X-ray detectors and computing upgrades. The availability of very efficient lenses in the hard X-ray range (2-100 keV) led to a dramatic progress of the scanning version of X-ray imaging (**microbeam based imaging**). This is used for structural (combining diffraction and imaging) and chemically-selective X-ray imaging (high spatial resolution fluorescence maps, or chemical state using energy dispersive micro-spectroscopy). Recent developments exploit the **coherence** of the synchrotron X-ray beams for sophisticated phase contrast imaging or coherent diffraction imaging. These techniques rely on improvements of detectors and algorithms, in particular for the reconstruction of "holotomographic" images, and for the iterative determination of the phase of the scattering amplitude in coherent diffraction imaging. In addition the increasing demand to access SR-based imaging techniques requires implementation of automation, and high throughput. These new opportunities will be discussed and illustrated by examples of applications to a wide variety of materials, which reveal features not observable otherwise.

17:55

Oral

Martensitic transformation of individual grains within multiphase TRIP steels studied with high energy synchrotron X-ray diffractionNiels H. Van Dijk¹, Enrique Jiménez-Melero¹, Lie Zhao², Jilt Sietsma², Sven E. Offerman², Sybrand Van der Zwaag³

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Martensitic transformations are acquiring an increasing technological relevance in a broad scope of high-performance materials. However, there is a lack of in-situ experimental information on the martensitic

transformation behaviour of individual grains, which is essential to optimize the mechanical properties of the material. In this sense, in-situ three-dimensional synchrotron X-ray diffraction has become a powerful tool to monitor individual grains within the bulk of polycrystalline materials. In this study, we have focused on TRIP steels, which are attracting a great interest due to their high strength and good formability. The Transformation Induced Plasticity (TRIP) effect, which is considered to yield a significant contribution to the large elongation of these steels, stems from the transformation of the metastable (face-centred cubic) austenite phase into the (body-centred tetragonal) martensite phase. The martensitic transformation of the metastable austenite can be induced mechanically by applied stress or thermally by cooling. In recent experiments we have monitored in-situ the martensitic transformation of a significant number of individual grains during cooling, using an intense microbeam of high-energy X-rays (80 keV). For each of the grains, the martensitic transformation temperature has been correlated to local microstructural parameters like the interstitial carbon concentration and the grain size. For the smallest grains we demonstrated that the grain stability of the metastable phase depends strongly on the grain size, indicating a critical size below which the martensitic transformation is completely suppressed. Future developments at synchrotron and free electron laser sources will strongly improve the spatial and time resolution and thereby lead to new opportunities to probe martensitic transformations of single grains in situ.

[1] N.H. van Dijk et al., *Acta Mater.* 53 (2005) 5439.[2] E. Jiménez-Melero et al., *Scripta Mater.* 56 (2007) 421.

18:15

Invited

"Stress in Nanoparticles"Ian Robinson*London Centre for Nanotechnology (LCN), Gower St, London WC1E6BT, United Kingdom**e-mail: i.robinson@ucl.ac.uk*

Quantitative three-dimensional maps of the deformation of a crystal from its equilibrium lattice spacing have been obtained by inversion of the coherent X-ray diffraction pattern. This requires solving the crystallographic 'phase problem' which is achieved by oversampling and a support-constrained HIO algorithm. The lead crystal we investigated [1] was grown on a SiO₂ substrate and shows internal strain arising from its contact with the substrate. Closer examination of the strain distribution, after an important correction for X-ray refraction has been included, shows an expansion of the surface layers of the faceted hemispherical nanocrystal. Over most of the surface of the crystal there is a clear outward displacement, which decays exponentially into the bulk. The displacement is suppressed on the (111) facet itself and stronger on the surrounding regions, indicating an orientational variation in surface stress.

[1] M. A. Pfeifer, G. J. Williams, I. A. Vartanyants, R. Harder and I. K. Robinson, "Three-dimensional Mapping of a Deformation Field inside a Nanocrystal", *Nature* 442 63-66 (2006)

18:50

Oral

Quantitative Assessment of Inhomogeneous Creep Cavity Distribution

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The damage evolution of in-situ creep experiment of a brass alloy Cu-40Zn-2Pb has been studied using high resolution synchrotron X-ray microtomography. This technique (XMT) offers new possibilities for a non-destructive determination of creep damage evolution and it provides direct images of the bulk of the materials. Different creep damage extents between the surface and in the bulk of samples was previously reported, however no clear evolution of damage of those regions was deduced. In the present study, an in-depth and systematic investigation of the spatial inhomogeneities of creep cavities using XMT has been carried out. The well-known analysis of nearest neighbor distances (NND) and the clustering tendency of cavities were performed for the bulk and the surface of the sample. Furthermore, comparisons of individual cavities evolution in those regions of the sample are given.

Tuesday, 18 September

Session 2

Biomaterials and Soft matter

Tuesday morning, 18 September, 9:00

9:00

Invited

Synchrotron infrared micro-spectroscopy in Biomaterials Research

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The application of micro-spectroscopy, and hence imaging, to biological and pathological problems in particular relies on informations obtained with great details. During the recent years, the application of infrared synchrotron radiation to microscopy has flourished. Because of the high brightness of the synchrotron source, enhanced lateral resolution, faster acquisition time and superior spectral quality could be obtained. Thus, synchrotron infrared micro-spectroscopy is a demanded analytical technique and several beamlines are operational, planned or being designed around the world. Spectroscopic imaging provides diagnostic informations in a visual form, a prospect appealing to physicians and biologists. Imaging methods can provide potentially far more information to non-specialists than their non-imaging counterparts. However, the interpretation and diagnostic potential of IR imaging strongly depends on the quality of the spectra acquired. A crucial parameter, the signal-to-noise ratio, has strong impact on the image quality. For biological or biomedical investigations, a comprehensive understanding of all informations buried in a complex sample is rather elusive based on known functional group analysis. Computer-based multivariate methods are available for valid and statistically

relevant classification of infrared patterns. In this presentation, we shall illustrate the imaging capabilities of synchrotron infrared micro-spectroscopy for individual cells and human tissues. Chemical images (also called univariate images), as well as multivariate images, have been obtained for several examples, demonstrating the high potential of this synchrotron-based microanalytical technique. We anticipate further interesting developments of synchrotron-based infrared micro-spectroscopy if combined with other synchrotron-based techniques like x-ray microscopy performed on the same samples.

9:35

Oral

X-ray and IR micro-analysis studies of bone mineral and related biological samples

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Commonly considered as inert, the mineral of hard tissues (bone, teeth) appears in fact as a very reactive and sophisticated material. The composition, the crystal size, the crystal orientation and the surface properties all contribute to adapting the tissue to its biological functions.

The various existing types of biological mineralization are based on apatitic calcium compounds and, in the case of vertebrates, on apatitic calcium phosphate compounds (apatite biominerals).

For bone, providing mechanical resistance and acting as a primitive ion reservoir are two crucial functions. They are obtained through a tissue organisation involving apatite nanocrystals with a very high specific surface area and a well-developed hydrated layer responsible for ion exchanges with body fluids. Maturation, ion exchange and absorption are involved in the dynamic behaviour of apatitic biominerals.

Consequently, bone mineral presents a complex composition. In addition to calcium and phosphate, it contains numerous other ions (hydrogenophosphate, carbonate, magnesium, sodium), metals and trace elements. All these compositions vary strongly in bone through and with time, but also depending on the diet, turn over rate of the mineral, medical treatments...

We will report the studies carried out on processes of maturation of bone mineral, but also to present some pathologies which perturb the natural bone turnover and maturation process, related proposed medical treatment, and biomaterials (analogues to bone mineral and implants). Complementary information obtained using a combination of synchrotron imaging techniques (X-ray microscopy -absorption, fluorescence-, micro-XANES, infrared microscopy, micro-diffraction) will be presented.

9:55

Invited

Self-assembled matter probed by synchrotron-based techniques

Michael Zharnikov

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Frontier areas of modern technology rely on the possibility to tailor surface properties such as wetting, adhesion, lubrication, corrosion,

and biocompatibility on both microscopic and macroscopic scales. To a large extent, these options are provided by self-assembled monolayers (SAMs), which are 2D polycrystalline films of semi-rigid molecules that are chemically anchored to a suitable substrate. In addition, such layers represent a general platform for the arrangement of future molecular electronics devices, framework for Chemical Lithography, and a model system for macromolecular and biological assemblies. Synchrotron-based techniques represent a powerful tool box for the characterization of SAMs. A basic information on the chemical identity, integrity, molecular conformation and orientation in these films can be obtained by a combination of X-ray photoelectron and X-ray absorption spectroscopy (XPS and XAS, respectively). More specific questions, such as, e.g., bonding configuration of the anchor group or dynamics of the charge transfer along the molecular backbone require special experimental tools such as high-resolution XPS and resonant Auger electron spectroscopy, respectively. Using several model systems, we will illustrate application of the above techniques for the characterization of SAMs. In addition, we will show that SAM can be used as prototypes of highly organized biological systems and provide important implications for cryogenic approaches in advanced electron and x-ray microscopy and spectroscopy of biological macromolecules and cells.

Coffee break

Tuesday morning, 18 September, 10:30

Session 2 (continuation)

Biomaterials and Soft matter

Tuesday morning, 18 September, 11:00

11:00

Invited

NEXAFS Microscopy and Soft X-Ray Resonant Scattering

Harald W. Ade

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NEXAFS microscopy and soft x-ray resonant scattering are soft x-ray techniques that offer high intrinsic contrast to image materials in real space or characterize structure in reciprocal space, respectively. Furthermore, the high contrast is strongly photon energy dependent and correlated to the chemical moieties present in the sample, thus allowing compositionally sensitive characterization. The carbon, nitrogen and oxygen NEXAFS is particularly sensitive to differences in composition in organic matter, and NEXAFS microscopy with presently ~30 nm spatial resolution and resonant scattering with a q-range of ~2 nm⁻¹ are thus excellent tools to characterize organic soft condensed matter and organic/inorganic hybrid materials. I will present the present state of the art of these characterization tools with a focus on the most recent developments in resonant scattering and resonant reflectivity. These results show that resonant soft x-ray scattering will be a powerful alternative tool to neutron- and conventional x-ray scattering for the characterization of structured soft condensed matter nanomaterials. The advantages of these soft x-ray resonant methods are primarily their high, tunable contrast compared to conventional x-ray scattering and the lack of special sample preparation requirements, i.e. isotope substitution (deuteration), which is typically required to enhance contrast for neutrons.

11:35

Invited

Charge transfer in conjugated materials and at interfaces thereof in dynamic and static descriptions

Wojciech Osikowicz, Michel P. De Jong, Slawomir Braun, Mats Fahlman, William R. Salaneck

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X-ray absorption and photoelectron spectroscopy has been used to investigate charge transfer processes in conjugated materials and across their organic-organic as well as hybrid interfaces. The dynamics of a charge transfer is probed using resonant photoemission spectroscopy in combination with core-hole-clock principle. The results show orbital-specific femtosecond charge transfer across specific interfaces as well as in between conjugated units. On the other hand photoelectron spectroscopy applied to the energy level alignment studies at various hybrid and organic-organic interfaces provides information on the charge exchange at equilibrium. For many interfaces that are of industrial importance the charge transfer is restricted to much slower tunneling processes and is limited to transfer of complete charges.

12:10

Oral

Effect of glass composition on crystallization of anorthite phase in CaO-Al₂O₃-SiO₂ glass-Al₂O₃ filler composite

Minji Ko, Soo Hyun Lyoo

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A mixture system of MO-SiO₂ (M: Ca, Sr, Ba) glass and Al₂O₃ crystalline powder has been investigated as a substrate material for low temperature co-fired ceramics (LTCC). This type of glass-ceramic mixture is known to be crystallized during the sintering process. We found that glass composition was strongly related with degree of crystallized anorthite phase. And it was observed that the degree of crystallization was affected by sintering condition as well as glass composition. Crystallization behavior was monitored by means of powder X-ray diffraction (XRD). The microstructure of sintered body was examined by Scanning Electron Microscopy (SEM).

Lunch break

Tuesday afternoon, 18 September, 12:30

Session 3

Disordered materials and extreme conditions

Tuesday afternoon, 18 September, 13:45

13:45

Invited

Pressure induced phase transitions: the local point of view

Anne-Marie Flank¹, Jean-Paul Itié¹, Pierre Lagarde¹, Alain Polian²

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Most of materials exhibit pressure induced phase transformations which can induce strong modifications of their properties (electronic, magnetic...). These properties are often related to the interaction between the neighboring atoms and therefore the knowledge of the local order in the new phases is required to understand what happens to the material. When long range order does not exist (amorphous material, glasses or liquid) or is only an average of the local order local investigations, like X-ray Absorption Spectroscopy (XAS) are needed to follow the modifications of the short range order. Moreover, in case of dilute species in a matrix, the local probes become unique tools to determine the effect of the phase transformation on the impurity.

After a short introduction on high pressure technology, the limitations of measuring XAS specially at low energy when using a pressure set-up will be presented. Thanks to the combination of new high pressure cells with perforated diamonds and preindented gasket and of the highly focused XAS LUCIA beamline (SLS-PSI, Switzerland), low energy edges (2-8 keV) are now accessible to high pressure experiments above 20 GPa. Some examples illustrating these possibilities will be given.

- Phase transformation on the dilute magnetic semiconductor $\text{Zn}_{1-x}\text{Mn}_x\text{O}$ [1] for $x=0.25$ and $x=0.05$. For both compositions, Mn atoms substitute to Zn in the low pressure phase wurtzite structure. Under pressure the phase transition from wurtzite to rocksalt structure occurs above 7 GPa for the two compositions studied. The transition is shown to be reversible for $x=0.05$ and irreversible for $x=0.25$.

- LiV_2O_4 is a mixed-valence compound with equal ratio of V^{3+} and V^{4+} . The observed pressure induced phase transition can be attributed to a charge ordering as it was observed in the isostructural compound, AlV_2O_4 [2].

[1] Pellicer-Porres J. et al, *Appl.Phys.Lett.*, 2006, 89, 231904

[2] Pinsard-Gaudart L. et al, to be published in PRB

14:20

Oral

Infrared measurements at high-pressures at the SIS-SI@Elettra beamline

Andrea Perucchi, Leonetta Baldassarre, Emanuele Arcangeletti, Daniele Di Castro, Paolo Postorino, Stefano Lupi

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Strongly correlated electron systems are of great interest because of their complex phenomenology. Difficulties in achieving a proper understanding of these phenomena lie in the subtle interplay of electron-electron (U) interaction, electron-phonon ($e-ph$) interaction, and the electron bandwidth (W). Experimentally, W can be varied either by chemical doping or by applying hydrostatic pressure. The latter procedure is preferable since it permits to *continuously* tune W without introducing impurities and disorder.

High pressures can be achieved through the use of a diamond anvil cell (DAC). However, the small dimensions of the samples to be placed in a DAC make infrared measurements at high pressure a very challenging technique. The high brightness of an infrared synchrotron radiation source (IRSR) is of great help for performing measurements in a DAC with an adequate signal to noise ratio and over a broad spectral range. We present here both reflectivity and transmission measurements performed at the IRSR beamline SISSI@Elettra. A special focus will be dedicated to the study of pressure-induced metal to insulator transitions in different vanadium oxide compounds.

14:40

Invited

X-ray powder diffraction and absorption experiments under pulsed magnetic fields up to 30T

Fabienne Duc¹, Carsten Detlefs², Paul Frings¹, Johan Vanacken³, Karine Chesnel¹, Olivier Mathon², Emilio Lorenzo⁴, Marc Nardonne¹, Julien Billette¹, Aziz Zitouni¹, Wim Bras², Geert Rikken¹

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Intensive research on advanced materials has led to the discovery of new compounds with spectacular properties driven by quantum correlation effects. Studies of these materials under high magnetic fields are of fundamental importance since magnetic field finely tunes the delicate balance between different correlation effects. Because of the strong dependence of these interactions on the atomic arrangement, the determination of the correct crystal structure at these extreme conditions is a prerequisite for the validation of any model of the ordered state and of the underlying interactions. In this context, numerous initiatives have been undertaken recently to develop X-ray techniques (diffraction, absorption spectroscopy) in high magnetic fields.

Here, we report X-ray powder diffraction and X-ray absorption spectroscopy (XAS) experiments carried out at the ESRF (beamlines BM26B, ID20 and ID24) in 20 ms long pulsed magnetic fields. For these experiments, a mobile pulsed magnetic field installation developed at the LNCMP, Toulouse, was used to generate peak fields of 30T with a rise time of about 5 ms. A liquid He flow cryostat allowed us to vary the sample temperature accurately between 4.2 and 300K.

Powder diffraction patterns of several samples were recorded in a broad temperature range using 21 keV monochromatic X-rays and an online image plate detector exposed for 5 ms around the maximum of the field pulse. More particularly, we observed the effect of large magnetic fields on the Jahn-Teller distortion of TbVO_4 .

XAS spectra could be measured and finite XMCD signals, directly proportional to the magnetic moment on the Gd absorber atom, were measured in thin Gd foils. The detector used was a CCD FReLoN (Fast Readout Low Noise) camera which allows to record spectra with high enough frame rates and gives the possibility to track the evolution of the signal as a function of field strength during the pulse.

15:15

Invited

Inelastic Scattering at the Nanoscale

Claudio Masciovecchio

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The physics of systems without translational invariance such as liquids, dense fluids and glasses, has been fascinating scientist for many years. Indeed, the understanding of the liquid-to-glass transition mechanism, thermal anomalies at low temperatures, divergence of the transport properties and relaxation phenomena, are challenges that deserve strong experimental and theoretical efforts.

The recent construction of an inelastic scattering instrument working with UV photons has opened up the possibility to study collective dynamics in an energy-momentum transfer region not accessible before,

allowing engendering a large amount of new information about the complex behaviour of glass-forming materials.

I will also present the idea to extend the standard transient grating spectroscopy towards higher energies in a way such that nanoscale dynamics may be investigated. This would be extremely interesting to better understand the physics of disordered systems and could also allow sensitive probing of interfaces and extremely thin films, as well as heat transport and correlations in nanostructured materials.

Coffee break

Tuesday afternoon, 18 September, 15:50

Session 4

Materials reactivity

Tuesday afternoon, 18 September, 16:25

16:25

Invited

Oxidation, structure and chemical activity of catalysts monitored by in-situ surface diffraction

Andreas Stierle

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Chemical reactions at solid / gas and solid / liquid interfaces play a key role in industrial applications and environmental processes, such as high temperature corrosion protection, heterogeneous catalysis, energy conversion (fuel cells), gas sensors or dissolution of minerals. In my talk I will present, how chemical reactions at interfaces and nanoparticles can be followed in real time under operational or processing conditions using x-ray diffraction as an *in-situ* tool. The final goal of our experiments is to obtain atomic scale information on the structure formation at chemically active interfaces and nanoparticles under industrially and environmentally relevant conditions, such as reactive gas mixtures at atmospheric pressures, humid environments and elevated temperatures. I will discuss in detail the *in-situ* x-ray observation of Pd and Rh nanoparticle shape changes (size range 5-10 nm) as a function of the applied oxygen pressure and its reversal by CO exposure.

17:00

Invited

High Pressure XPS : A tool for the investigation of heterogeneous catalytic processes

Axel Knop-Gericke

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High Pressure XPS (X-ray photoelectron spectroscopy) in the millibar range became an in situ tool for investigations of heterogeneous catalytic processes within the last years. The success of this method is based on the possibility to correlate the catalytic activity of a catalyst with the electronic structure of the working catalyst surface. The application of this technique at a synchrotron radiation facility provides the possibility to measure depth profiles by changing the photon energy and therefore the kinetic energy of the photoelectrons. This kind of measurement allows the identification of subsurface species present below the active surface of a working catalyst. Subsurface species

might influence the electronic structure of the active surface and influence the catalytic properties of the catalyst.

Subsurface oxygen was found in polycrystalline Cu foil during the partial oxidation of methanol to formaldehyde. The abundance of this species is correlated to the yield of formaldehyde. Investigations of the same heterogeneous catalytic reaction over Ru(0001) shows that RuO₂ is active in the total oxidation of methanol to CO₂ and H₂O, whereas a RuO_x species catalyses the partial oxidation to formaldehyde.

The selective hydrogenation of pentyne to pentene over polycrystalline Pd foil is characterised by the formation of a subsurface carbon species, which blocks the diffusion of dissolved hydrogen to the surface. Dissolved hydrogen is known to be not selective. In this case only the gas phase hydrogen, which is selective, is available for the active surface. Under reaction conditions favouring the total hydrogenation of pentyne to pentane the carbon layer forms to a much lower extent. These examples show the potential of this in situ technique.

An overview about recent developments in the field of high pressure XPS will be given. The availability of the method for the community will be discussed.

17:35

Invited

Is there any possibility to use carbon nanotube as sensors?

Andrea Goldoni, Silvano Lizzit, Luca Petaccia, Rosanna Larciprete

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There are two main characteristics that suggest carbon nanotubes (CNTs) as promising candidates for extremely sensitive gas sensors. First, as one-dimensional systems, their "quantum nature" makes the CNT intrinsic properties highly sensitive to very tiny external perturbations. Second, the amount of active surface area is huge. Many experimental evidences demonstrate the high sensitivity of CNTs to several molecular species in gas phase, both organic and inorganic. Although most of the experimental reports must be taken cum grano salis and a number of technical problems must be deeply investigated and solved, there is the effective possibility to a sensor technology based on CNTs within the next five years. Coatings and/or functionalization are the goals.

18:10

Oral

Electron--electron correlation in graphite

Alexander Gruneis¹, Claudio Attaccalite², Ludger Wirtz², Thomas Pichler¹, Angel Rubio³

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The full three-dimensional dispersion of the pi-bands, Fermi velocities and effective masses are measured with angle-resolved photoemission spectroscopy and compared to first-principles calculations. The band structure by density-functional theory strongly underestimates the slope of the bands and the trigonal warping effect. Including electron-electron calculation on the level of the GW approximation, however, yields remarkable agreement in the vicinity of the Fermi level. This demonstrates the breakdown of the independent electron picture in semi-metallic graphite and points towards a pronounced role of electron correlation for the interpretation of transport experiments and double-resonant Raman scattering for a wide range of carbon based

materials.

Wednesday, 19 September

Plenary Session

Wednesday morning, 19 September, 9:30

Coffee break

Wednesday morning, 19 September, 10:30

Session 5

Electronic structure and magnetism

Wednesday morning, 19 September, 11:00

11:00

Oral

Phonon and vibrational spectra of real crystals obtained using the synchrotron radiation

Eugen M. Sheregii¹, J Cebulski¹, Jacek Polit¹, B V. Robouch², A Marcelli², M Piccinini², A Kisiel³, Andrzej Mycielski⁴

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Data of measurements of optical reflection obtained in wide range of wave numbers ($30 - 8000 \text{ cm}^{-1}$) in far and middle infrared regions and at temperatures ($20 - 290 \text{ K}$) with using of synchrotron radiation have been summed up in given paper for the CdTe, CdHgTe, ZnHgTe, ZnCdTe and ZnCdHgTe crystals produced by different methods. An unique ownership of the synchrotron radiation have allowed to resolve subtle structure of the phonon and vibrational bands in researched crystals. The main results can be summarized as following: 1) in case of CdTe crystals different lines corresponding to the local mode were observed; additional lines associated with red shift with respect to the main phonon mode of the heavily hydrogenated CdTe were interpreted as a resonant or Additional Phonon Modes (APM) caused by the Cd-Te oscillations in tetrahedra containing one or two additional H atoms[1]; 2) in case of ZnCdTe crystals eight phonon modes, canonical for ternary alloys (canonical phonon mode – CPM) generated by defect free tetrahedra, were observed and successfully interpreted [2]; 3) in case of CdHgTe crystals phonon spectra can not be interpreted without taking into account intrinsic defects for n- and p-type of materials: tetrahedra containing the Hg-vacancies generate APM[3]; 4) similar situation is observed in ZnHgTe-crystals; 5) in case of quaternary ZnCdHgTe generally, we could observe 30 CPMs but really it is possible to resolve 10 - 15 ones to which it is necessary to add APMs caused by defect-bearing tetrahedra [4]. A statistical model of deformed tetrahedra was developed for quantitative analysis of observed spectra. [1] J.Polit, E.M.Sheregii, et al. *J. Appl. Phys.*, **100**, 013521, 2006; [2] J.Polit E.M.Sheregii et. al. *Journal of Alloys and Compounds*, **371**, 172, 2004; [3] J.Polit, E.M.Sheregii et.al. *Eur. Phys. J. Appl. Phys.*, **27**, 321, 2004; [4] E.M. Sheregii, J.Polit et.al. *Infrared Physics & Technology*, **49**, 13, 2006;

11:20

Invited

Low-energy excitations in oxides from temperature-, angle- and polarization-dependent resonant soft-x-ray emission spectroscopy

Federica Bondino¹, Marco Zangrando¹, Tefvik O. Montes², Michele Zacchigna¹, Elena Magnano¹, Fulvio Parmigiani^{1,3}

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Resonant x-ray emission spectroscopy (RXES) is a powerful technique for the investigation of the elementary electronic excitations in solids. In RXES, an incident x-ray photon excites a core electron to the absorption threshold, and the x-ray emission resulting from the decay of the excited state is energetically analyzed. In recent years, this spectroscopy has progressed remarkably, becoming an effective tool, with elemental specificity, for the study of low-energy neutral electronic transitions, charge-transfer excitations, interband transitions, and energy-band dispersion.

I will present some applications of this technique in the study of oxides.

In the first part I will show how a Zhang-Rice-singlet excitation can be unambiguously identified in the O 1s RXE spectra of CuGeO₃ measured as a function of temperature, angle and polarization [1].

In the second part I will show some examples of Mn 2p RXE studies of manganites, showing how this spectroscopy can be used not only for the identification of the low-energy transitions [2], but also for the study of the charge-transfer dynamics [3] of these compounds.

[1] F. Bondino, M. Zangrando, M. Zacchigna, G. Dhalenne, A. Revcoleschi, and F. Parmigiani “Crystal-field and Zhang-Rice-singlet excitations in CuGe_{1-x}Si_xO₃ (x=0, 0.05, and 0.1) from temperature-, angle-, and polarization-dependent resonant soft x-ray emission”, *Phys. Rev. B* **75**, 153311 (2007)

[2] F. Bondino, M. Platè, M. Zangrando, M. Zacchigna, D. Cocco, A. Comin, I. Alessandri, L. Malavasi, and F. Parmigiani, “Doping-dependence of the electronic structure of La_{1-x}Na_xMnO₃ by resonant x-ray emission and absorption spectroscopy”, *J. Phys. Chem. B* **108**, 4018 (2004).

[3] T. O. Montes, F. Bondino, E. Magnano, M. Zangrando, K. Kuepper, M. Neumann, and F. Parmigiani, “Excitation Dynamics in La_{0.875}Sr_{0.125}MnO₃ Measured by Resonant Auger Electron and Resonant X-ray Emission Spectroscopies”, *Phys. Rev. B* **74**, 205409 (2006).

11:55

Oral

Surface structure and magnetism in a perpendicular exchange bias [NiO/CoO]/[PtCo]/Pt(111) system

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We report on a combined structural and magnetic study of the [NiO(3ML)/CoO(3ML)]x3/[PtCo]/Pt(111) perpendicular exchange bias system prepared by electron beam epitaxy. The coherent epitaxy was followed and thoroughly characterized *in situ* by Surface X-ray Diffraction. The sample shows a maximum in the coercivity associated

to TN and an exchange bias shift characterizing a strong exchange coupling. *Ex situ* PMOKE characterization also reveals, below TN, a spin reorientation within the ferromagnetic layer, induced by the strong anisotropy of the antiferromagnetic oxide. The depth dependence of the out-of-plane magnetization across the interface between the antiferromagnetic and the ferromagnetic layer has been investigated taking advantage of the chemical and spatial sensitivity of Soft X-ray Magnetic Reflectivity. A net magnetic moment is present in the oxide layers, both in the Co and Ni atoms. Upon TN, these moments are not confined to the ferromagnetic layer but extend in the oxide layer up to four atomic layers above the interface. These results show that interfacial effects observed in parallel exchange bias systems have their counterpart in perpendicular ones and that valuable insights into the surface structure and magnetism in these system emerge from combination of *in situ*, *ex situ*, and in-depth sensitive structural and magnetic characterizations.

12:15 Oral

Structure, Composition, Morphology, and Electrical Properties of Amorphous SiO_x (0<x<2) Thin Films

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Lunch break

Wednesday afternoon, 19 September, 12:35

Session 5 (continuation)

Electronic structure and magnetism

Wednesday afternoon, 19 September, 14:00

14:00 Invited

Photoelectron emission microscopy of layered magnetic materials containing antiferromagnets

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Imaging of magnetic domains has contributed essentially to our present level of understanding of magnetic materials. Among these, structures consisting of several ultrathin layers of ferromagnetic, antiferromagnetic, and nonmagnetic materials exhibit many new and interesting phenomena. One major issue is to understand the magnetic interaction between ferromagnetic and antiferromagnetic layers.

Photoelectron emission microscopy (PEEM) with synchrotron radiation is a technique capable of delivering layer-resolved magnetic information on a microscopic scale. Based on the element selectivity of X-ray magnetic circular dichroism (XMCD) and linear dichroism (XMLD), it allows the layer-resolved visualization of magnetic domain patterns at surfaces and in buried layers.

I will present investigations of layered magnetic structures containing single-crystalline antiferromagnetic FeMn layers. Layer-resolved magnetic domain images clarify how the magnetic coupling between the ferromagnetic and antiferromagnetic layers depends sensitively on the atomic-scale morphology of the interface [1]. Because of a three-dimensional non-collinear antiferromagnetic spin structure of FeMn, the XMLD signal of the antiferromagnetic layer vanishes. However,

it was possible to identify the tiny XMLD signal of the induced magnetic moments of the FeMn layer at the interface [2]. Interestingly, the XMLD signal of a ferromagnetic Co layer shows a pronounced anisotropy, which is triggered by the contact to the antiferromagnetic FeMn layer.

Results were obtained in collaboration with F. Offi, L. I. Chelaru, M. Kotsugi, K. Fukumoto, J. Wang, J. Kirschner and J. Kunes.

[1] W. Kuch et al., Nature Materials 5 (2006) 128.

[2] W. Kuch et al., Phys. Rev. B, in print (2007).

14:35 Oral

Antiferromagnetism vs. Paramagnetism: Revisiting the magnetostructure of MnAs on GaAs

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Ferromagnetic MnAs is a promising candidate for electrical spin injection into GaAs and Si based semiconductors, since it exhibits a large carrier spin polarisation, small coercive field and relatively high saturation magnetisation. Bulk MnAs is ferromagnetic at RT (α -phase) and shows near 40°C a second order transition to the paramagnetic β -phase. On contrary, epitaxial MnAs films on GaAs substrate show at RT the coexistence of both phases due to the large and anisotropic lattice mismatch. Elastic domains of both phases form a periodic stripes pattern in a self organised way [1]. The nature of the nonmagnetic phase, which coexists in thin films is of great interest[2]. Until recently β -MnAs has generally been considered to be paramagnetic but the effects just mentioned and first principles calculations[5] suggest antiferromagnetism.

Stimulated by this controversy, we have recently reexamined our XMCD-PEEM [6],[7] results and have complemented them with corresponding linear dichroism (XMLD) studies. We present the results of this comparison and will demonstrate that β -MnAs is antiferromagnetic. These important results end a more than 50 year old scientific controversy and allow to give a new revisited description of the magnetic behaviour in striped MnAs thin films.

XMCDPEEM image (left), showing the magnetic domain structure in the ferromagnetic phase and XMLDPEEM image (right), showing the coexisting antiferromagnetic phase, of a 300 nm thick MnAs film on GaAs (100). Diameter of field of view 5 μ m.

[1] T. Plake et al, Appl. Phys. Lett. **80**, 2523 (2002).

[2] L. Däweritz, Rep. Prog. Phys. **69**, 2581 (2006)

[3] K.-S. Ryu et al, Phs. Rev. B **71**, 155308 (2005)

[4] J. Mira et al, Phys. Rev. Lett. **90**, 097203 (2003)

[5] M. K. Niranjan, B.R. Sahu and L. Kleinman, Phys. Rev. **B 70**, 180406 (2004)

[6] E. Bauer et al. J. Vacuum Sci. Technol. B **20**, 2539 (2002)

[7] L. Daeweritz et al, J. Vacuum Sci. Technol. B **23**, 1759 (2005)

14:55

Invited

Domain wall spin structures and their interaction with spin-polarized currents

Mathias Kläui¹, Dirk Backes^{1,2}, Lutz Heyne¹, Daniel Bedau¹, Andrea Locatelli³, Onur Mentès³, Laura Heyderman², Frithjof Nolting², Aranxta Fraile-Rodriguez², Ulrich Rüdiger¹

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Magnetic head-to-head domain walls have recently become the focus of intense research and rather than using conventional field-induced reversal, a promising approach for switching magnetic nanostructures is current-induced domain wall motion (CIDM), where due to a spin torque effect, electrons transfer angular momentum and thereby push a domain wall [1-4]. Since this interaction is strongly dependent on the wall spin structure, we have employed primarily X-ray magnetic circular dichroism photoemission electron microscopy (XMCD-PEEM) to image such domain walls in NiFe wire nanostructures and correlate the above mentioned effects with the imaged spin structure [1-4]. By high resolution imaging of the wall spin structures we have been able to directly probe the energy barrier separating the wall types [5,6].

In addition to wall movement, changes in the wall spin structure have been predicted [2], and we have recently observed such wall type transformations using PEEM [3] and found that the velocity depends strongly on the wall type and the transformations occurring [3].

In addition to moving domain walls in wires, vortex cores in disc elements have been studied, since their movement allows for a direct measurement of the non-adiabaticity of the transport. Due to the high magnetization gradients, larger non-adiabatic contributions to the transport are expected compared to the transport across wide domain walls with low gradients [7].

1. M. Kläui et al., PRL 94, 106601 (2005); A. Yamaguchi et al., PRL 92, 77205 (2004).

2. A. Thiaville et al., EPL 69, 990 (2005).

3. M. Kläui et al., APL 88, 232507 (2006); F. Junginger et al., APL 90, 132506 (2007).

4. M. Laufenberg et al., PRL 97, 46602 (2006).

5. M. Kläui et al., APL 85, 5637 (2004); M. Laufenberg et al., APL 88, 52507 (2006).

6. M. Laufenberg et al., APL 88, 212510 (2006); M. Kläui et al., APL 86, 32504 (2005).

7. J. Xiao et al., PRB 73, 54428 (2006).

Coffee break

Wednesday afternoon, 19 September, 15:30

Joint Poster Session (Part II)

Poster Award Ceremony

Wednesday afternoon, 19 September, 15:50

Session 5 (continuation)

Electronic structure and magnetism

Wednesday afternoon, 19 September, 17:20

17:20

Invited

Studies of Magnetization Dynamics using synchrotron based methods

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In this talk I will review some of the recent advances in studying magnetization dynamics with high spatial resolution using methods based on XMCD. Using X-PEEM it is possible to gain information on the dynamic response of magnetic micro- and nanostructures with a spatial resolution of about 50 nm. Some experiments using pulsed magnetic field excitation will be reviewed.

Using a scanning transmission X-ray microscope it is possible to employ lock-in techniques which drastically increase the signal to noise ratio while maintaining the high spatial resolution.

Data will be presented that elucidate the complex magnetic mode structure of simple ferromagnetic objects in a frequency range from some hundred MHz up to a few GHz.

17:55

Oral

On the spin reorientation of Co/Au based magnetic nano-dot arrays.

Andreas Persson¹, Lidia Gridneva¹, Miguel Angel Nino^{2,5}, Julio Camarero², Juanjo de Miguel², Rodolfo Miranda², Christian Hofer³, Christian Teichert³, Thomas Bobek⁴, Andrea Locatelli⁵, Dimitri Arvanitis¹

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We discuss here, an X-ray Absorption Spectroscopy (XAS) study of (Co/Au) and (Au/Co/Au) ferromagnetic nano-dot arrays grown in-situ on self-organized SiGe substrates, using grazing angle shadow deposition as presented earlier for Co. /1/ We characterized the samples by X-ray Photoelectron Emission Microscopy (XPEEM), X-ray Magnetic Circular Dichroism (XMCD) at the Co L-edges in the XAS mode and XMCD-PEEM. Experiments were performed both at ELETTRA and MAX-lab. These dot arrays exhibit a considerable degree of lateral order, are ferromagnetic and single domain at room temperature. Macroscopic magnetic domains composed of several dots are observed. At low Co coverage, for Co/Au dots, dots with an out of plane moment were evidenced by XMCD-PEEM, at room temperature. Indeed, using angular dependent XMCD studies, versus x-ray incidence angle, for dots containing 2.5 ML Co layers on a 5 ML Au buffer grown on top of the SiGe nano-structured template, a strong stable macroscopic out-of-plane remanence, can be directly observed by XMCD at 100K. Such a sample can also be remanently magnetized in the sample plane. At room temperature no stable out of plane macroscopic remanence can be observed, only smaller out of plane domains can be evidenced by means of XMCD-PEEM. With a thicker Co coverage of 6 ML for Co/Au dots, it is found that the magnetization of the dots lies within the surface plane. A study of the Co orbital moment as a function of

both Co and Au cap thickness in these (Co/Au) dot arrays reveals a systematic increase of orbital moment, correlated with the appearance of a macroscopic out-of-plane magnetization. This work is supported by the EC as part of the NAMASOS project STRP NMP2/CT/2003/505 854.

I. A. M. Mulders, A. Fraile-Rodríguez, D. Arvanitis, C. Hofer, C. Teichert, M. Á. Niño, J. Camarero, J. J. de Miguel, R. Miranda, K. Lyutovich, E. Kasper, S. Heun, A. Locatelli, Phys. Rev. B vol.71, 214 422, 2005.

18:15 Invited

Domain wall dynamics studied by time-resolved XMCD-PEEM

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The propagation of magnetic domain walls, driven by pulsed magnetic fields or spin-polarized currents, plays an important role in present and future magnetic storage devices. Many of these devices, like spin-valves or magnetic random access memories (MRAM), consist of multilayered magnetic systems. We have used time-resolved photoemission electron microscopy (PEEM) combined with x-ray magnetic circular dichroism (XMCD) to study domain wall propagation in multilayered continuous films and nanostructures. We have used the element selectivity of XMCD to obtain separate, microscopic information on the domain wall dynamics in FeNi/X/Co trilayer systems (X = Cu, Al₂O₃). Domain wall propagation speeds obtained with magnetic field pulses are much larger than with pulses of spin-polarized currents. Domain walls in the Co layer strongly influence the domain wall dynamics in the FeNi layer, through magnetostatic interactions induced by domain wall stray fields. We have also used time-resolved XMCD-PEEM to study the influence of the Co layer on the reduction of the critical currents for current-induced domain wall propagation in this kind of trilayers.

This work was performed together with S. Pizzini, V. Uhler, M. Bonfim, J. Camarero, W. Kuch, K. Fukumoto, V. Cros and F. Petroff

Thursday, 20 September

Session 6

Time-resolved and Ultra-fast x-ray Science
Thursday morning, 20 September, 8:50

8:50 Invited

Electron Dynamics in magnetic Nanostructures

Wolfgang Eberhardt

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Over the last decade the processes following excitation of a magnetic recording medium by an optical fs laser pulse have attracted a lot of interest not only from a fundamental physics point of view but also under the aspect of understanding the fundamental limitations of the magneto-optical recording process. Here I will report our studies on Ni

films carried out by fs laser spectroscopy and pump probe experiments at the BESSY soft X-ray fs slicing source, providing new insight into the ultrafast electronic and magnetic response stimulated by optical excitation by a fs laser pulse.

Magnetic recording media are comprised of individual nanometer size particles. As these dimensions are reduced in order to increase the storage density, the individual electronic properties, derived from quantum confinement gain in importance. At the extreme limit of mass selected clusters containing less than about 100 atoms not only the ground state properties, but also the dynamic optical response depend uniquely and strongly on the exact size of the particle/cluster.

As an example, we have explored the relaxation processes of electronically excited states in mass selected Au clusters. The phenomena observed depend strongly on the size of the cluster ('each atom counts') and range from electron-electron to electron-phonon scattering and from vibrational wavepacket oscillations to a quasi 'melting' of the cluster.

Presently this type of spectroscopy is limited by the photon energies available from laboratory laser sources or the low intensity of the slicing source. However the new soft X-ray free electron laser light sources such as FLASH at DESY and the planned BESSY-FEL offer a unique potential to expand these studies in the future.

9:25 Invited

Molecular Dynamics using Ultrashort X-Ray Pulses

Christian Bressler

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We will present our recent results on the light induced structural dynamics of molecules in solution using an optical probe/X-ray probe method based on X-ray absorption spectroscopy. This approach was applied to the case of Iron (II)-based complexes, which undergo a spin change from singlet to quintet. We have determined the structure of the high spin excited state, which has a lifetime of 0.6 ns. The structural determination was obtained using exploiting both the XANES and the EXAFS features. In a recent development, 100 femtosecond hard X-ray pulses have been extracted from the SLS synchrotron. We will present our preliminary results where we follow the ultrafast XANES changes, the evolution of the system from the initially excited singlet charge transfer state into the lowest excited quintet state, on a time scale of 1 ps.

10:00 Invited

Ultrafast structural dynamics in solids studied with accelerator-based femtosecond X-ray sources

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no abstract content

Coffee break

Thursday morning, 20 September, 10:35

Session 6

Time-resolved and Ultra-fast x-ray Science
Thursday morning, 20 September, 11:00

11:00 Invited

Single pulse diffractive imaging of a biological samples

Filipe Maia, Janos Hajdu

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Single pulse diffractive imaging allows, in principle, to visualize arbitrary samples to atomic resolution by recording its diffraction, which can then be inverted to give an image of the object, without any extra information (Neutze, Wouts et al. 2000). In practice the technique is limited by the intensity and wavelength of existing light sources, as well as radiation damage caused during the pulse. By combining several images taken in different orientations it will be possible to reconstruct the 3D structure of reproducible objects, as long as it is possible to determine the relative orientation of the different images. By removing the need for crystals this technique will revolutionize atomic scale imaging. The talk will outline current results obtained at FLASH, including the imaging of fixated and vaporised cells, and outline what will be the future developments, as well as what problems still left unresolved.

11:35 Invited

6 Million Pixels for Advanced Diffraction Studies: the PILATUS 6M

Miroslav Kobas, Christian Broennimann

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A novel x-ray detector has been developed at the Paul Scherrer Institut for protein crystallography at the Swiss Light Source (SLS). The PILATUS 6M detector is a two-dimensional hybrid pixel array detector, which operates in single-photon counting mode [1]. It consists of 2527 x 2463 pixels with a pixel size of 0.172 mm. The basic building block consists of a module with 487 x 195 pixels. The 6M detector is composed of 60 modules arranged in 12 banks each containing 5 modules. The total active area amounts to 424 x 435 mm².

This detector features several advantages compared to current state-of-the-art CCD and imaging plate detectors. The main features include: no readout noise, superior signal-to-noise ratio, a point spread function of one pixel, readout time of 4 ms, framing time of 80 ms, a dynamic range of 1:1'048'576, high detective quantum efficiency (100%-50% from 8-16 keV) and the possibility to suppress fluorescence by an energy threshold. This energy threshold can be set individually for each pixel with a precision of 50 eV. The short readout and fast framing time allow to take diffraction data in fine-phi-slicing mode with continuous rotation of the sample without opening and closing the shutter for each frame.

The high dynamic range of the PILATUS detector makes it possible to acquire simultaneously weak and strong diffraction phenomena of high quality. The superior signal-to-noise ratio enables the detection of very weak diffraction signals like diffuse scattering. The impressive speed of up to 12 Hz for the complete 6M detector and up to 400 Hz for a single module allows to carry out time-resolved experiments.

Results from various x-ray experiments are presented, including diffraction data from protein and aperiodic crystals, as well as results from diffuse scattering and x-ray absorption experiments. All data have been collected at the X06SA and X05LA beamline of the SLS.

[1] Broennimann, Ch. et al., J. Synchrotron Rad., 2006, 13, 120-130.

Lunch break

Thursday afternoon, 20 September, 12:30

Special Session on Research Frontiers with UV and x-ray FEL

invited presentations

Thursday afternoon, 20 September, 14:00

14:00 Invited

The European X-ray Free-Electron Laser Project in Hamburg

Massimo Altarelli

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A number of projects worldwide are pursuing the realization of a source of extremely brilliant (peak brilliance $\sim 10^{33}$ photons/s/mm²/mrad²/0.1%BW), ultra-short (~ 100 fs) pulses of spatially coherent x-rays with wavelengths down to 0.1 nm, and to exploit them for revolutionary scientific experiments in a variety of disciplines spanning physics, chemistry, materials science and biology. In the US and in Japan, Free-Electron Lasers (FEL) are being developed based on room-temperature linear accelerators (Linacs). In Europe, the superconducting linear accelerator technology developed at DESY within the TESLA collaboration, and successfully applied to produce laser-like radiation in the 100 to 13 nm range at the FLASH facility at DESY, is adopted. The most important advantage of the superconducting technology is the possibility to produce up to 30 000 pulses per second, instead of 60 or 120 foreseen in the Japanese and American projects, respectively. In the European facility, it is foreseen that electron bunches, accelerated to 17.5 GeV in a ~ 1.7 km long Linac, pass through long (up to 200 m) undulators, where they generate bursts of coherent x-rays via the process known as SASE (Self-Amplified Spontaneous Emission). Commissioning with first beam of the facility is expected to take place in 2013. An initial contingent of 3 photon beamlines with 6 experimental stations (later to be upgraded to 5 photon beamlines and 10 experimental stations) is foreseen, where experiments exploiting the high intensity, the coherence and the time structure of the new source are going to be performed. A brief illustration of some of the potential experiments is presented.

14:30 Invited

4GLS - the UK's Fourth Generation Light Source Project

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4GLS is a suite of accelerator-based light sources planned to provide state-of-the-art radiation in the THz-soft X-ray regime. Superconducting

energy recovery linac (ERL) technology will be utilised in combination with free electron lasers (IR to XUV), undulators and bending magnets. The undulators will generate spontaneous high flux, high brightness synchrotron radiation (SR), of variable polarisation from 3 - 800 eV, optimised in the lower harmonics up to about 200 eV. Viable radiation at energies up to several keV may be provided from multipole wiggler magnet radiation. The ERL technology of 4GLS will allow shorter bunches and higher peak photon fluxes than is possible from storage ring sources. VUV and XUV FELs will be used to generate fs-regime pulses that are broadly tuneable and more than a million times more intense than the equivalent SR. 4GLS will probe ultra-fast dynamics in a wide range of fields. Pump-probe experiments will allow the study of chemical reactions and short-lived intermediates on the timescale of bond formation, even for very dilute species. Circularly polarised light sources in a variety of frequency regimes will be used to manipulate and monitor carrier charge and spin transport in device structures. The high intensity of the FEL radiation will allow high resolution in imaging and the opportunity to probe nonlinear regimes. The lower intensity, high repetition rate ERL SR provides ideal sources for ultra-high energy resolution spectroscopy, especially in the solid state. Around £22 M funding has been obtained for the first stages of the project (construction of a prototype source, science demonstrations and design study work, currently nearing completion at STFC Daresbury Laboratory). It is anticipated that the full 4GLS facility will be available to users in 2014.

15:00 Invited

Frontiers in Science with FELs: The FERMI@ELETTRA project

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The FERMI@Elettra free electron laser will be realized in two principal phases. FEL-1 is designed to operate in the time domain and with two complementary operating modes: a) high stability and b) high intensity. FEL-2 is designed to operate in the frequency domain (high energy resolution) and relatively long photon pulses (~ 1 ps). These design choices will yield a FEL source quite unique among the other FELs under construction or proposed for construction in Europe, USA and Japan. The initial FERMI science program is structured to allow experiments with different, and increasingly more demanding photon parameters. This plan will allow performing high quality experiments from the very beginning of the FEL1 and FEL2 commissioning. For these reasons we plan the following order of experiments. • Single Shot Experiments (High Peak Brilliance Experiments) • Pump-Probe Experiments (Experiments in both the time and frequency domains) • High Energy Resolution Experiments (Experiments in the Frequency and Time Domain, non-linear spectroscopy experiments).

Coffee break

Thursday afternoon, 20 September, 15:30

Special Session (continuation)

Thursday afternoon, 20 September, 16:00

16:00 Invited

The PSI - Free Electron Laser Project

Albin F. Wrulich

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The PSI-XFEL covers the wavelength range from 0.1 to 10 nm and incorporates the development of an electron gun with small emittance and an acceleration with high gradient in a diode configuration immediately after the electron emission from the cathode. The fast initial acceleration, combined with a pulse of moderate initial current and correspondingly larger length, will alleviate space charge effects. A higher compression of the beam is required, which is effectively reached by an appropriate shaping of the longitudinal phase space with a newly developed 2-frequency cavity.

An overview of the project is given with a status of the R&D work and the achievements.

16:30 Invited

Free Electron Lasers at MAX IV

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The planned MAX IV facility is intended to be the successor of the existing MAX-lab. Three synchrotron radiation storage rings at 0.7, 1.5 and 3.0 GeV will provide spontaneous radiation of very high quality covering a broad spectral range. A 3 GeV linear accelerator will be used as a common injector for all three rings. This linear accelerator will also serve as the electron source for spontaneously emitted short-pulse X-rays in the first phase and for Free Electron Lasers (FEL) in a second phase. The FEL facility is constructed to take advantage of the present and projected fast technical development in this field, aiming towards the construction of a 2nd generation FEL facility. The main line is to benefit from seeding techniques and to use Self Amplified Spontaneous Emission (SASE) amplifiers.

17:00 Invited

The BESSY Soft X-Ray FEL in Berlin Adlershof

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BESSY is operating Europe's largest third generation synchrotron radiation user facility for the VUV and Soft X-Ray range. For this presentation special emphasis will be placed upon introducing the BESSY FEL project. BESSY is planning to build a Soft X-Ray FEL facility covering the photon energy range from 24 eV to 1 keV at the site next to the existing BESSY II storage ring. This second generation FEL facility will offer laser like photon beams with fully coherent, high power (mJ) pulses of ≤20 fs duration, enabling a whole set of novel experiments dedicated to understand dynamical processes in matter or for the investigations of very dilute systems. Complementary to the SASE FEL facilities such as FLASH or LCLS, the pulses will be generated in a HGHG scheme (High Gain Harmonic Generation) which offers controlled, reproducible pulses as determined by the ex-

ternal seed UV (430-230 nm) laser pulse. Additionally this scheme allows for an inherent synchronization for pump-probe investigations. The HGHG-FEL covers the traditional BESSY II photon energy range, which is especially suited for electronic structure investigations of atoms, molecules, clusters, and solids. With the anticipated temporal resolution of ≤ 20 fs charge transfer processes and time resolved ‘femtochemistry’ studies as well as magnetization dynamics in magnetic materials establish some of the major areas of scientific interest in this new facility. Furthermore, in exploiting the coherence of the photon pulses in microscopy applications it is possible to acquire an image with about 10nm resolution using a single laser pulse. Thus, for example, stroboscopic time resolved images of dynamical processes in living cells can be recorded.

Round Table

Thursday afternoon, 20 September, 17:30

Friday, 21 September

IRUVX meeting

IRUVX meeting

Friday morning, 21 September, 11:00

Posters

Monday, 17 September

Joint Poster Session (Part I)

Monday & Wednesday

Monday afternoon, 17 September, 15:55

15:55	Poster	11

Influence of high temperature annealing on the local atomic structure around Mn atoms and magnetic properties of (Ga,Mn)As layers

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In recent years a rapid development of the field of research called spintronics is observed. The goal of the studies is to create conceptually new devices which will utilize spin of electron. To achieve this goal it is necessary to fabricate effective injectors of polarized spins and to learn how to manipulate and detect spin polarization. On the other hand the integration of magnetic materials with conventional semiconductor technology should be also possible to achieve. All of these imply the fabrication of suitable materials with appropriate properties at room temperature and manufacturing the reliable spin based device.

As suitable candidates for these studies GaMnAs structures were chosen being a composite multiphase material, after high temperature post-growth annealing, with small ferromagnetic nanoparticles of MnAs immersed in the semiconductor host lattice. Changes of the local structure around Mn atoms in (Ga,Mn)As layers after high temperature annealing were determined by an X-Ray absorption spectroscopy (namely EXAFS) and high resolution X-Ray diffraction (HRXRD) measurements, and were compared with their magnetic properties. The X-Ray absorption is sensitive enough to detect crystalline structure changes around Mn atoms in consequence of annealing at the temperatures of 500 and 600 °C. The qualitative and the quantitative analysis of EXAFS spectra gives unambiguously evidence of transition of the local structure around Mn atoms from cubic to hexagonal (i.e. new phase formation, namely hexagonal inclusions of MnAs). The sample with MnAs hexagonal inclusions exhibits ferromagnetic properties up to room temperature with almost all Mn atoms being in ferromagnetic phase.

ACKNOWLEDGMENTS

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15:55	Poster	12

Magnetic frustration in “Spin-Ice” nanostructures networks.

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The recent progress made in lithography technologies allows the elaboration of well-controlled artificial structures exhibiting exciting behaviors such as quantum magnetism, order - disorder statistics... This is also the case for magnetic nanostructures for which the elaboration artificial networks of ferromagnetic single-domain structures allows to explore local magnetism, and in particular magnetic interactions between artificial nanostructures.

In a geometrically ordered network[1], these interactions generate magnetic frustrations similar to those observed in Spin-Ice systems[2]. The size and shape of the nanostructures are chosen so that only the single-domain configuration is stable (a few tens of nanometers and strong shape anisotropy) in order to avoid any influence of micromagnetism. The system can then be modeled by a network of spins (magnetic single-domain) arranged in the form of networks treated within the Ising model approximation[3]. Using lithography techniques, we can then create but also control the problem of magnetic frustration.

We present the results of a first study of frustrated magnetic systems composed of hexagonal (Kagomé) and square ordered networks using the combination of high-resolution X-PEEM microscopy and Magnetic Force microscopy. The goal of this work is to highlight the importance and the influence of short-range interactions and of the geometry on

the magnetic frustration phenomena and relaxation.

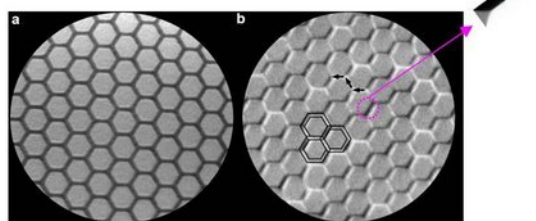


Figure : 'Spin-Ice' Kagomé Network : periode 700nm. Nanostructure individual size : 100x400nm. a) LEEM microscopy image b) Magnetic domains images obtained using X-PEEM microscopy highlighting the magnetic frustration.

[1] Moessner, R. Can. J. Phys. **79**, 1283-1294 (2001).

[2] R. F. Wang, C. Nisoli, R. S. Freitas, J. Li, W. McConville, B. J. Cooley, M. S. Lund, N. Samarth, C. Leighton, V. H. Crespi et P. Schiffer Nature **439**, 303 (2006)

[3] M. Tanaka, E. Saitoh, H. Miyajima, T. Yamaoka et Y. Iye. Phys Rev B **73**, 052411 (2006).

15:55 Poster I3

fea modelling and flow behavior studies of aluminum-copper alloys using vision system

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Aluminum-Copper binary alloys (2%, 4% & 6% by weight) were deformed (compression) between flat platens to predict the metal flow. Friction and aspect ratio ($H_0/D_0 = 1.0$ and 1.5) are studied as process parameters. Surface lubrication (Vacuum grease as lubricant) minimized barreling for compared to dry compression. Non uniform deformation was observed in terms of variation in Vickers micro-hardness. The same has been correlated with microstructural features. Adoption of Vision system to analyze the flow behavior of materials during upsetting has been investigated by using PC based video recording system and CCD camera was used to analyze the grid deformation. Finite element software ANSYS has been applied to model, simulate, and analyze the upset forming process. Friction coefficient, measured from Ring test was used for FEM modeling.

15:55 Poster I4

Modeling of 2D GISAXS of nano-crystalline silicon thin films

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Grazing-Incidence Small-Angle X-ray Scattering (GISAXS) spectra of nano-crystalline silicon thin films were simulated using the freely available FORTRAN program IsGISAXS. This program calculates the particles form factor in the distorted-wave Born approximation, which describes correctly the reflection-refraction effects at the substrate surface. In model calculations, the samples were assumed as a

mixture of random oriented nano-crystals and voids 2-30 nm in size, embedded in an amorphous matrix. Several particle shapes and size distributions were tested using, as initial values in the calculation, the data estimated by HRTEM. The calculated spectra were compared with GISAXS measurements done at the Austrian SAXS line at the synchrotron Elettra (Trieste). By varying the initial values in, the calculated, spectra were fitted to the experimental values until a reasonable match was obtained. The in that way obtained individual particle sizes, size distribution and volume contribution of each element used in the model calculation were compared with the results from Raman and optical spectroscopy. GISAXS was performed at different grazing incidence angles which allows for depth resolution and enables a better comparison with the other applied methods. The obtained results and usefulness of the described procedure in the analysis of thin films will be discussed.

15:55 Poster I5

Spin structures in micro-patterned highly spin-polarized materials

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Highly spin-polarized materials have become the focus of intense research due to their possible applications in spintronic devices and their exciting magnetotransport properties [1]. 100% spin-polarized materials where the density of states exhibits a gap in one spin channel, are commonly called half-metallic ferromagnets and one of the most prominent examples is magnetite (Fe_3O_4) [2].

Spin-dependent transport properties are in general strongly dependent on the spin polarization but in the usual geometry (multilayer pillar elements), interfaces can play a crucial role for magnetoresistance and spin transfer torque effects and often the polarization at interfaces is strongly reduced compared to the bulk value.

In order to make use of the high bulk spin polarization for spin transfer torque effects, single layer elements can be used, where the complications of interfaces are avoided. In such single layer elements, the interplay between currents and domain-walls leads to domain-wall magnetoresistance [3] and strong currents have been predicted to displace domain walls due to the spin transfer torque effect [3,4].

Before such devices can be envisaged, the spin structures of the domain walls has to be determined. We use high resolution X-ray magnetic circular dichroism photoemission electron microscopy to image the spin structure in highly spin - polarized magnetite (Fe_3O_4) patterned elements. Due to the cubic anisotropy, the domain wall spin structures strongly differ from those usually observed in permalloy [5]. In addition to head-to-head domain walls, also 90° domain walls are observed and the exact spin structure depends on the orientation of the magnetization with respect to the magnetocrystalline anisotropy axes.

[1] K. Dörr, J. Phys. D **39**, R125 (2006).

[2] M. Fonin et al., J. Phys. Cond. Mat. (in press 2007)

[3] C. H. Marrows, Adv. Phys. **54**, 585 (2005)

[4] M. Kläui et al., PRL **94**, 106601 (2005)

[5] M. Laufenberg et al., APL **88**, 52507 (2006)

15:55 Poster I6

Identification of Mg based phases in ilmenites by X-ray absorption spectroscopyMarcin Klepka¹, Krystyna Lawniczak-Jablonska¹, Iraida N. Demchenko¹, Roman Minikayev¹, Maciej Jablonski³, Velichka Kontozova²

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Ilmenites are important minerals used as a source for TiO₂ (white pigment) production. Ilmenites originated from Norway, India, China and Australia are frequently being used in industrial technological processes. This raw material is mainly used in titanium dioxide production by sulphate method. This method requires the knowledge about elemental and phase composition for adjusting the efficiency and the safety of kinetics of the reaction. The main phases which involved Ti and Fe are known but content and chemical states of other elements is also important because of their impact on quality of the final product. In the presented paper Mg based phases were studied in minerals listed above. To characterize these minerals we have applied X-ray absorption spectroscopy (XAS), X-ray diffraction (XRD) and single particle electron probe microanalysis (SPEPMA). From the performed SPEPMA and XRD measurements it results that Mg phases constituted quite substantial part of investigated minerals. The Mg chemical states were evaluated by applying the principal components analysis of X-ray absorption K-spectra of Mg (XANDA code was used). The spectra were measured at BEESY beamline UE52PGM. The Mg was found in three chemical compounds MgTiO₃, MgSiO₃ and Mg(OH)₂. The content of the particular phases depends on the place where minerals were collected. These findings were the starting point in Rietveld analysis of diffraction data and they were compared with the results from SPEPMA cluster analysis.

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RII3-CT-2004-506008 (IA-SFS).

15:55 Poster I7

High Temperature in-situ X-ray Investigation of Liquid Phase Sintering of Zinc OxideAndreas Klimera, Mohammad Lutful Arefin, Friedrich Raether

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ZnO-based ceramics doped with M₂O₃ show highly non-ohmic current-voltage behaviour that is important for surge arresting in electrical circuitry. The addition of Bi₂O₃ to ZnO lowers melting temperature and promotes liquid-phase sintering. In contrast, the presence of Sb₂O₃ influences grain size distribution and the formation of new intermediate compounds. Grain boundary phases with spinel and pyrochlore structures affect the electrical behaviour of ZnO-based varistors.

High temperature in-situ X-ray diffraction by synchrotron source is used to monitor progressive formation of new phases during sintering. Partial evaporation of (Bi,Sb)₂O₃ leads to gradients away from sample surface in liquid phase distribution, so conventional HT-XRD is not reasonable because of its small penetration depth in the surface region (10-20 µm).

Therefore, synchrotron X-rays were used at the HASYLAB, Hamburg to analyze the phase formation in samples of 1 mm thickness. The samples were heated in a resistive furnace which was specially developed for those experiments and measured in transmission mode. Different compositions in the ZnO-Bi₂O₃-Sb₂O₃-system were investigated.

15:55 Poster 18

X-ray Absorption Fine Structure study of nickel grains embedded in the carbonaceous filmsMirosław B. Kozłowski^{1,2}, Robert Nietubyc³, Elżbieta Czerwosz^{1,4}, Ryszard Didusko^{1,2,5}, Piotr Dłużewski², Joanna Rymarczyk¹, Edmund Welter⁶

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We have measured Ni K-edge extended x-ray absorption fine structure (EXAFS) spectra for 300 nm thick carbonaceous films containing Ni clusters of the size in the range 2-20 nm. The films with various nickel concentrations have been deposited onto a Mylar® foil substrate by PVD method. EXAFS measurements have been performed in fluorescence and transmission mode at the A1 beamline at DORIS III in Hamburg.

The EXAFS spectra were interpreted by fitting calculated scattering paths contributions to the experimental contributions extracted from the measured fine structure by Fourier Transform filtering. The analysis was supported by ab initio model calculations (FEFF) performed for distorted *fcc* structures and structures optimized for Ni₁₃ clusters.

It has been found that for none of the studied samples the range of crystalline order exceeded 5 neighboring shells (6 Å). Spectra measured for the samples containing the most diluted nickel showed an enhanced white line and other spectral features which are not observed for metallic bulk nickel. For samples having Ni concentration of 2.7 wt% the crystalline order has been found to be limited to the nearest neighbors.

15:55 Poster 19

Gd atoms on Si (111) surface – AFM and photoemission studyBronisław A. Orlowski¹, Bogdan J. Kowalski¹, Elżbieta Guzewicz¹, Elżbieta Lusakowska¹, Victor Osinniy¹, Iwona A. Kowalik¹, Mieczysław A. Pietrzyk¹, Elżbieta Nossarzewska-Orłowska², Andrzej Bukowski², Robert L. Johnson³

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The vacuum deposition of Gd atoms on Si(111) surface leads to the appearance of some unexpected effects. The Gd atom as a next after

Eu with configuration $\text{Eu } 4f^7 6s^2$ it has configuration $\text{Gd } 4f^7 5d^1 6s^2$ of electrons. Lightly bound $\text{Gd } 5d^1$ electron contributes as a donor and Gd atom appears in most chemical compounds as Gd^{3+} ion. The atom is very reactive and in reaction with Si gives a sparking effects. Deposition of Gd atoms by evaporation in UHV on Si(111) leads to creation of craters on Si(111) surface in interface region. Negative magnetoresistance giant effect was observed in low temperatures below 80 K for $\text{Gd}_x \text{Si}_{1-x}$ silicide as an effect of RKKY interaction between isolated Gd ions and free carriers.

The paper presents study of Gd atoms deposited on Si(111) surface by means of Atomic Force Microscopy (AFM) and Fano-type resonant photoemission. The layers of thickness 2, 20, 150 and 3000 Å were deposited in UHV conditions. The remarkably deep craters were observed on silicon surface under deposited 150 Å of Gd layer. The application of synchrotron radiation in the region of energy $h\nu$ corresponding to the Gd 4d-4f transition (130 – 170 eV) gave the possibility to measure Fano-type resonant photoemission spectra. The spectra of Si valence band with contribution of Gd 4f and 6s electrons were studied for layer of Gd with 2 Å thick deposited on Si(111) clean surface. The photoemission study gave the curve of the Fano resonance shape with resonance for $h\nu=151.8\text{ eV}$ and antiresonance for $h\nu=146.8\text{ eV}$. The Gd 4f localized electrons gave the contribution to the valence band density of states located 9.8 eV below the valence band edge.

15:55 Poster I10

Phase structure investigations of the bulk glassy FeCoZr-WB alloys

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Changes of nanostructure with the cooling conditions were studied on ribbon samples of various thicknesses produced by melt-spinning and rod and tube samples of various diameters prepared by suction-casting technique for the $\text{Fe}_{61}\text{Co}_{10+x}\text{Zr}_{15}\text{W}_{4-x}\text{B}_{20}$ (where $x=0, 2, 3$ at.%) alloys.

Mössbauer spectra analysis for the amorphous rod, tube and ribbon samples allowed the determination of hyperfine parameters for the amorphous phase and showed its ferromagnetic ordering in as-cast state. The hyperfine field B_{hf} distributions calculated from the Mössbauer spectra, also indicated non-equivalent surroundings for the Fe atoms. Furthermore, dissimilar B_{hf} distributions were observed for samples produced by various rapid solidification methods. The glass transition temperature T_g , crystallization temperature T_x and melting temperature T_m were compared for all alloy compositions. Furthermore the activation energies for crystallization have been evaluated using the Kissinger's equation for particular alloy composition from the DSC plots.

High-energy X-ray diffraction (XRD) measurements were performed using monochromatic synchrotron radiation of 112 keV ($\lambda=0.110696$ Å) at undulator beamline PETRA 2 of synchrotron facility DESY/HASYLAB (Hamburg, Germany). The analysis of diffraction patterns revealed fully glassy structure for ribbon samples of all compositions investigated. In case of the bulk samples, some traces of crystallinity were detected for the rod samples of all compositions, due to lower cooling rates applied for processing bulk glassy samples. The low fraction of crystalline phase present in the rod samples was im-

possible to detect by conventional XRD equipment, therefore the synchrotron radiation is due to its advantages more preferred technique of studying the glass forming ability of the alloys. Similar studies of the thin walled tubes samples revealed their fully amorphous nature.

Work supported by the Polish Ministry of Science and Higher Education (grant: 3T08A 046 27)

15:55 Poster I11

Mn 3d electrons in the valence band of $\text{Mn}/\text{Ge}_{0.9}\text{Mn}_{0.1}\text{Te}$ - a resonant photoemission study

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We present the results of the electronic band structure study of $\text{Ge}_{0.9}\text{Mn}_{0.1}\text{Te}$ epitaxial layer surface, clean and enriched in Mn by *in situ* deposition. GeMnTe is a ferromagnet with a relatively high Curie temperature (up to 140 K) strongly dependent on Mn concentration.

The $\text{Ge}_{0.9}\text{Mn}_{0.1}\text{Te}$ layers were grown on BaF_2 (111) substrates by an MBE method with use of GeTe, Te_2 and Mn solid sources, at the substrate temperature of 450°C. The sample surface was protected against interaction with ambient atmosphere (during transfer to the photoelectron spectrometer) by a layer of amorphous tellurium. Prior the photoemission experiments, the tellurium layer was removed by sputtering and annealing. The Mn overlayers were deposited *in situ* at room temperature.

The valence band and shallow core levels of $\text{Mn}/\text{Ge}_{0.9}\text{Mn}_{0.1}\text{Te}$ were investigated by means of resonant photoemission spectroscopy. This experimental technique is a tool enabling determination of the emission of electrons from the transition metal 3d orbitals. Therefore, the sets of photoemission spectra were measured for the photon energy range covering the energy of the Mn 3p@3d transition. They were acquired for the clean surface of $\text{Ge}_{0.9}\text{Mn}_{0.1}\text{Te}$ sample, after evaporation of 0.5 ML Mn and after annealing. The annealing was carried out in order to induce the diffusion of Mn atoms into the $\text{Ge}_{0.9}\text{Mn}_{0.1}\text{Te}$ crystal. The Mn 3d states contribution occurred in the upper part of the valence band, with a maximum at the binding energy of about 4 eV. The shape of the spectra corresponds to the emission from Mn ions occupying the lattice sites with octahedral coordination. Deposition of Mn and annealing the system lead to an increase of Mn 3d feature intensity. This proves that diffusing Mn ions occupy the same sites in the lattice as those introduced during the layer growth, increasing the Mn contents in the subsurface layer.

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15:55 Poster I12

Development of a photoelectric method to measure the MOS system flat-band voltage

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Results of the application of the LPT (Light Pulse Technique) method to accurately measure the flat-band voltage V_{FB} value are presented. This method, known for many years, has never been thoroughly tested and brought to practical applicability. We develop this method as an alternative to the commonly applied V_{FB} determination from the C(V) characteristics of MOS structures. The accuracy of V_{FB} determination, using the C(V) characteristics is not better than ± 100 mV and the LPT method is expected to assure the ± 10 mV accuracy.

The LPT method consists in illuminating the MOS structure gate with the pulsed UV light beam. The electrical signal u from the structure depends on the semiconductor surface potential Φ_S and changes sign when $\Phi_S = 0$. Hence, the V_{FB} value is determined by taking the u vs gate voltage V_G characteristic and finding the value of V_G which corresponds with $u = 0$.

A model allowing correct prediction of the $u = f(V_G)$ characteristic has been developed and a comparison of V_{FB} measurement results by the LPT and C(V) methods has been made.

The LPT method gives excellent reproducibility of the measurement results and is very sensitive (both quantities are better than ± 10 mV). The V_{FB} values obtained by both methods are usually close to each other. However, in some cases, differences between V_{FB} values obtained by both methods were considerable (sometimes exceeding 100 mV). We ascribe these differences to the limited accuracy of the C(V) method, but we can not exclude yet the other factors which may influence the measurements results. This problem is currently being studied.

The next step will be the development of the SLPT (Scanned LPT) method, which should allow determination of the V_{FB} local values distribution over the gate area of MOS structures. In this case the structure will be scanned by a pulsing UV light beam of small (in comparison with gate dimensions) diameter. Initial results of this kind of measurements are promising.

15:55	Poster	I13

Comparison of the interfaces ZnO/Si and ZnO:Al/Si by soft X-ray photoemission spectroscopy

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Transparent conductive oxide (TCO) films have been extensively used in optoelectronic devices because of their high visible transmittance and low dc resistivity. Recent investigations point out that ZnO, which combines semiconducting behavior with optical transparency in the visible range, will be one of the most competitive semiconductors in the near future.

Pure ZnO and Al-doped films about 100 nm thick were prepared onto Cz-Si (1000) type p (carrier concentration 10^{20} cm⁻³) substrates by spray pyrolysis. The deposition parameters were chosen to ensure a good optical transparency. The structure and chemical analysis confirmed the formation of Al-doped and undoped ZnO and the absence of other phases or compounds. More details about preparation and characterization of undoped and Al-doped ZnO films have been published elsewhere.

PES spectra were recorded using synchrotron radiation at VUV PHOTOEMISSION Beamline of the ELETTRA synchrotron (Trieste). The interfaces were studied using VB, Al 2p and Si 2p (bulk binding energies 72.5 and 99.2 eV) spectra. Ar⁺ ion beam sputtering was used to reduce the pyrolysis layer thickness progressively, in order to reveal

the ZnO:Al/Si or the ZnO/Si interface, where an intermediate SiO_x oxide layer has been detected. In order to maintain the oxygen stoichiometry, ion beam sputtering was followed by light oxidation (20 Langmuirs 10⁻⁶ Torr for 20 seconds). In spite of the weak signal coming from Al 2p state (nominal doping was 3%), the high photon flux at low energies in ELETTRA facility, has allowed to distinguish contributions due to two different chemical environments, octahedral and tetrahedral. Their respective intensities change with sputtering deep, which suggests Al segregation at the junction with SiO_x.

15:55	Poster	I14

A new powder diffraction beamline at Pohang Light Source and its recent results

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We introduce a new beamline, fully dedicated to X-ray powder diffraction measurements, at Pohang Light Source. The design is for a powder crystallography, i.e., very high angular resolution and various sample environments. The technical characteristics of the beamline and some performance indicators are listed, such as the incoming photon flux and the angular/energy resolutions obtainable under typical experimental conditions. We present some recent results using the high-resolution data of this beamline, not detected by previous powder diffraction experiments.

15:55	Poster	I15

Study of Si doped AlGaIn by synchrotron radiation x-ray microprobe techniques

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Blue and UV light emitting diodes (LEDs) and lasers based on AlN and GaN semiconductor compounds represent one of the most important breakthroughs in electronics and optoelectronics of recent years. By varying the composition of ternary Al_xGa_(1-x)N alloys and by the achievement of controlled doping, the optical and electrical properties can be tuned over a wide spectral range.

So far, n-type conductivity is commonly obtained by the incorporation of Si, which is supposed to substitute for Ga or Al in the GaN or AlN lattice, respectively. The dependence of the AlGaIn material properties on Si doping has already been reported, but some observed trends are not well understood yet, e.g. the effect of Si on the defect formation, alloy disorder, and stress relaxation in AlGaIn is still under discussion. The observed strong correlation between electrical and optical properties is generally explained by a random distribution of impurities and defects that may produce local strain and potential fluctuations. However, little direct information on the spatial compositional distribution and local structure of Si in AlGaIn lattice is available.

In this study, we report the application of synchrotron radiation X-ray microprobe to the study of Si impurities in plasma-induced molecular bema epitaxy (PIMBE) grown $\text{Al}_{0.32}\text{Ga}_{0.68}\text{N}$. The synchrotron-related data were collected at the LUCIA beamline of Soleil installed at the Swiss Light Source (Villigen, Switzerland). Elemental maps obtained by micro-x-ray fluorescence spectrometry show inhomogeneous distributions of Si, Al, and Ga on the micron scale. X-ray absorption near-edge structure spectra taken at the Si and Al K edges provided information about their local chemical environment and revealed the change of the spectral features as depending on the position compared to the sample surface and on the concentration of Si.

15:55 Poster I16

A XAS study of surface oxidised Fe particles

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A study of surface oxidised iron particles with X-ray absorption spectroscopy (XAS) at the Fe:K, L_2 , L_3 edges and the O:K edge is presented. Powder samples were prepared by ball milling and subsequently thermally treated in vacuum or air. The XAS study of the samples and the reference Fe oxides in the XANES range of the Fe:K edge has been carried out at the synchrotron laboratory HASYLAB, Hamburg. Simultaneous measurements in the transmission geometry and total electron yield (TEY) have been carried out. The transmission spectra which correspond to probing of the bulk do not reveal the presence of iron oxides within 1% error margin, except for the sample annealed in air at 300 °C. Its spectrum consists of the contributions of 95% metallic Fe, 4% magnetite and 1% hematite. The TEY spectrum of the sample, which corresponds to a few hundreds of nanometers probing depth, contains contributions of 45% metallic Fe, 42% magnetite and 13% hematite. Taking into account different probing depths of both methods the average thickness of the oxide layer could be determined. The XANES spectra of the samples studied and the reference oxides at the Fe: L_2 , L_3 edges, O:K edge and the EXAFS spectra at the O:K edge have been measured at the synchrotron laboratory ELETTRA, Trieste. The TEY detection mode was used, which in the case of the Fe:L edges and the O:K edge has the probing depth of a few nanometers. The O:K edge XANES spectra and their derivatives as well as the EXAFS spectra have been fitted with a linear combination of the spectra of the reference oxides. This provided the information on the content of individual oxide species in the surface layers, which is particularly valuable for the samples thermally untreated and annealed in vacuum, where the oxide layer is of nanometric thickness.

15:55 Poster I17

Stability of Jahn-Teller distortion in LaMnO_3 under pressure: direct study by X-ray absorption

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The physics underlying the remarkable properties of the manganite LaMnO_3 and its doped alloys is rich and complex. The actual path followed by a given system towards ferromagnetism and/or metallization, through chemical substitution, thermal treatment or pressure involves modifications of a delicate balance between delocalization and carriers trapping. Even in the parent stoichiometric compound LaMnO_3 the actual relationship between the conduction state and local distortion - related to the Jahn-Teller character of the Mn^{3+} ion - is not fully understood. The present study aims to describe the local process accompanying the pressure-induced abrupt changes in the resistivity in the Jahn-Teller distorted LaMnO_3 .

We observed directly the modifications of the average local distortions around the manganese atoms induced by application of high pressures, in the range 0-15 GPa, from the modifications of X-ray absorption spectra in the near edge range. The MnO_6 octahedra keep unchanged by application of hydroscopic pressures up to 8 GPa, whereas above this value a continuous long Mn-O distance reduction is observed. However, the Jahn-Teller bond splitting persists over the whole pressure range. A total quenching of this splitting is expected to take place only for pressures larger than 30 GPa, i.e. at pressure where the metal to insulator transition also takes place. An hysteresis in the XANES features is observed down to ambient pressure, suggesting the coexistence of MnO_6 distorted and undistorted units.

Ramos et al., Phys. Rev. B 75 (2007) 052103.

15:55 Poster I18

EXAFS investigation of the local atomic structure of di-alanino acid derivative of protoporphyrin IX

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EXAFS can be successfully applied to resolve local atomic structures of very low symmetry. We used this technique to get a better insight into the local environment of iron atoms in di-alanino acid derivative of protoporphyrin IX. Right now, these compounds undergo intensive clinical tests as potential photosensitizing candidates for photodiagnosis and photodynamic therapy (PDT). In PDT, the controlled photosensitization of reactive oxygen species (ROS), including singlet oxygen ($^1\Delta_g$), is believed to be responsible for triggering a cascade of subsequent biochemical processes resulting in eradication of tumor cells. In particular, di-amino acid derivatives of protoporphyrin IX attract a lot of attention since they reveal selective retention by malignant tissues and are also efficient ROS generators. Our preliminary electron spin

resonance (ESR) study revealed the presence of high-spin, trivalent iron atoms (FeIII, S=5/2) in di-alanino acid derivative of protoporphyrin IX nominally iron-free compound. EXAFS measurements were performed at station BM26A at ESRF. The analysis of EXAFS spectra of Fe-K absorption edge indicated iron presence inside the protoporphyrin IX ring and revealed the bromine atom as one of the first neighbors. The comparison of the shift of Fe-K absorption edge for di-alanino acid derivative of protoporphyrin IX and the material of reference, Fe₂O₃, confirm the presence of the trivalent iron centers in this novel photosensitizer.

Most probably the location of iron and bromine depends on technological process of preparation the samples. Consequence of precise determination of place of bonding the heavy atoms will be finding the most efficient and no expensive purification procedure to obtain iron and bromine free photosensitizer and allowance to concentrate on most effective technology production process.

Acknowledgments :

This work was partially supported by grant N202 053 32/1197

15:55	Poster	I19

Electronic structure of Mn atoms in (Ga,Mn)As layers modified by high temperature annealing

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We attempt to address here an actual question facing material scientists today, namely novel materials for devices explicitly utilizing the electron spin for microelectronic applications. Among many, GaMnAs is commonly considered as a promising material for such applications. Most of the studies on this compound were devoted to the uniform ternary alloy which is a diluted ferromagnetic semiconductor; however it is fairly easy to obtain GaMnAs samples containing precipitations of ferromagnetic MnAs. These precipitations were usually considered as a major drawback. However, since MnAs is a metallic ferromagnet with Curie temperature of about 318 K, it is possible to prepare the GaAs:MnAs system in such a way that small ferromagnetic nanoparticles are immersed in the semiconductor host lattice. Such a composite material could be considered as a good semiconductor filled with nanomagnets providing a built-in magnetic field at room temperature. These magnetic MnAs nano-clusters can be produced from a single-phase GaMnAs material by the post-growth annealing at temperatures higher than 500 C. We focus here on the atomic and electronic structure of the Mn atoms relating to the cluster formation. The changes in the electronic structure of Mn, Ga and As atoms in (Ga,Mn)As layers after high temperature annealing were determined by x-ray absorption near edge spectroscopy (XANES). The experimental spectra were compared with the predictions of the ab initio full multiple scattering theory (FEFF 8.4 code). The nominal concentration of the Mn atoms in the investigated samples was 6%, therefore we do not observe changes in the electronic structure of Ga and As. However, we found considerable changes in the case of Mn atoms.

This work was partially supported by national grant of Ministry of Science and High Education N202-052-32/1189 as well as by DESY,

MAX-lab and the European Community under Contract RII3-CT-2004-506008 (IA-SFS).

15:55	Poster	I20

The VUV luminescent spectroscopy of Pr-doped scintillators based on single crystalline films of garnets and perovskites

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The Liquid Phase Epitaxy (LPE) of wide band gap oxides activated by rare-earth ions with fast and efficient broad band UV emission opens new possibilities for development of thin-film scintillators and screens for visualization of invisible images from biological objects. The Ce³⁺ and Pr³⁺ ions were considered for such application, and the latter is the less studied one. In 2005, at LOM Lviv National University as well as at SUPERLUMI station in HASYLAB, DESY, Hamburg we started a new program of growth and investigation of Pr³⁺ luminescence using the synchrotron radiation excitation in scintillators based on single crystal (SC) and single crystalline film (SCF) of garnet and perovskite compounds.

Pr-doped LuAG, YAG, LuAP and YAP SCF were grown by LPE from melt-solution (MS) based on Pb-B₂O flux on the YAG (garnets) and YAP (perovskites) substrates. The Pr₂O₃ concentration in MS was varied in a range of 1.1-10.5 mol % for determination of the maximum of light yield (LY) under excitation by α -particles of Pu²³⁹ sources. Some samples were additionally co-doped with Ce³⁺ for study of the Pr³⁺→Ce³⁺ energy transfer. The comparison with LuAG:Pr and YAP:Pr bulk SC analogues was performed as well.

The following main features of Pr³⁺ ion luminescence in SCF and SC of garnets and perovskites it is considered: **(i)** excitation energy transfer between d-f states and ratio of the d-f and f-f emission intensities; **(ii)** dependence of the d-f and f-f luminescence intensity on the activator concentration; **(iii)** influence of Lu_{Al} and Y_{Al} AD on the luminescence and energy transfer in LuAG and YAG SC; **(iv)** excitation energy transfer between Pr³⁺ and Ce³⁺ centres.

It has been also considered the possibilities of application of the LuAG:Pr and YAG:Pr SCF as thin film UV scintillators for α - and β -particles monitoring as well as LuAP:Pr and YAP:Pr SCF as luminescent light sources in a range of 250-260 nm for visualization of invisible images.

Symposium J

Symposium Scope

Symposium Scope:

Nowadays the need of advanced materials for high-end applications increases considerably, while the grain size of their constituents decreases down to the nanometric scale. Therefore, fundamental research and design of novel materials with tailored properties requires the use of sophisticated microscopy and spectroscopy characterization tools.

The aim of this symposium is to introduce a wide range of participants to the entire potential of materials characterization techniques available today. It is of crucial importance to present the broad range of capability and quantity of possible data that microscopic characterization methods offer to the scientists and engineers. A good example may be the transmission electron microscopy, which is often used for simple imaging of the material microstructure, while it can be applied for sophisticated analysis of various defects at the atomic scale, qualitative and quantitative elemental mapping and reconstruction of 3-D structures. Therefore, tools like electron or surface probe microscopy equipped with various satellite tools will be in the scope of this session. Particular interest will be focused on high-end spectroscopic tools allowing chemical characterization of the materials down to the atomic bonding. Contributions in advances and innovations in instrumentation are particularly solicited.

All scientists and engineers interested in contributing to the development of knowledge in microscopic characterization tools are cordially invited to participate in this symposium.

The symposium will consist of four sessions, listed below, each of them devoted to a different characterization method:

1. Application of Analytical Electron Microscopy
2. Surface Probe Microscopy & Spectroscopy at Micro & Nanoscale
3. Non-Destructive Characterization Methods
4. Nanoindentation and Nanotribology of Advanced Materials

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Proceedings

The Proceedings of the Symposium J will be published in "Micro" (International Research and Review Journal for Microscopy) Elsevier.

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Programme

Monday, 17 September

Opening & Plenary Session

Czocharlski Award Ceremony & Acta Gold Medal Ceremony
Monday morning, 17 September, 10:00

Lunch break

Monday afternoon, 17 September, 12:30

Parallel Session

Nanotribology

Monday afternoon, 17 September, 14:00

14:15

Invited

Mechanisms of atomic friction studied by friction force microscopy

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Force microscopy is a versatile instrument to investigate physical phenomena on surfaces. The first emphasis is on the study of friction on the nanometer-scale, also called nanotribology. It will be shown that atomic-scale stick-slip is relatively well understood, where the dependence on velocity and normal force is experimentally investigated and interpreted in terms of an extended Tomlinson model. It is essential to include thermal actuation to understand the observed phenomena. The transition from atomic-scale stick slip to continuous sliding will be described [1]. The detailed analysis yields the energy corrugation and the lateral contact stiffness as a function of load. It is found that continuum mechanics is not anymore valid at these dimensions. Recently, it has been shown that atomic friction can be controlled with rather high accuracy by electrostatic actuation [2]. It will be shown that this type of actuation leads to variations of the energy barriers, which makes it possible to enter the regime of superlubricity. Recent experiments on hetero-structures of KBr on NaCl have shown that the energy barriers can also vary in space, which leads to transition from regions with atomic stick slip to regions with smooth sliding [3].

[1] A. Socoliuc, R. Bennewitz, E. Gnecco and E. Meyer, Transition from Stick-Slip to Continuous Sliding in Atomic Friction: Entering a New Regime of Ultra-low Friction, *Phys. Rev. Lett.* **92**, 134301-1 (2004).

[2] A. Socoliuc, S. Maier, O. Pfeiffer, E. Gnecco, A. Baratoff, R. Bennewitz and E. Meyer, Atomic-Scale Control of Friction by Actuation of Nanometer-Sized Contacts" *Science*, **313**, 207, July 14 (2006).

[3] S. Maier et al., to be submitted (2007).

14:45

Invited

Nanoparticle manipulation using Scanning Probe Techniques: A new approach to Nanotribology

Udo D. Schwarz¹, Claudia Ritter¹, Dirk Dietzel^{2,3}, Andre Schirmeisen², Harald Fuchs^{2,3}

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In recent years, considerable efforts have been directed towards the clarification of the atomic origins of friction, largely spurred by the miniaturization of moving components in technological devices. By analyzing the friction between an atomic force microscopy (AFM) cantilever tip and the sample surface, friction force microscopy (FFM) has proven to be a powerful tool for nanotribology. Unfortunately, FFM has some limitations inherent to the experimental configuration. Apart from the limited variety of material combinations, the fixed tip radius makes it difficult to analyze effects as a function of the contact area. However, the contact area dependence is one of the most fundamental yet unsolved current questions in nanotribology.

In order to achieve significant progress by overcoming the limitations of FFM, the friction between two objects in relative motion with a well-defined contact area should be measured. One possible approach is to measure the lateral force between nanoscale islands supported by flat substrates while being pushed by an AFM tip. Thus, experiments have been conducted where the frictional resistance of antimony nanoparticles featuring contact areas from 10000 nm² to 310,000 nm² is measured.

In this configuration, if the interface is atomically clean, a state of virtually frictionless sliding is anticipated, often referred to as 'structural lubricity'. Here, the lattice mismatch at the interface causes a decrease of the potential barrier between stable states with increasing contact size that ultimately leads to vanishing friction. However, experiments performed under UHV and ambient conditions showed two distinct frictional states: While some particles assume a state of frictionless sliding, other particles show finite friction increasing linearly with contact area, thus resembling Amonton's law at the nanoscale. This unexpected duality of friction states might be related to contamination of the interface altering the frictional properties.

Coffee break

Monday afternoon, 17 September, 15:30

Joint Poster Session (Part I)

Monday & Wednesday

Monday afternoon, 17 September, 15:50

Tuesday, 18 September

Parallel Session

Spectroscopy

Tuesday morning, 18 September, 9:00

9:00

Invited

Optical fields at micro and nanostructures

Othmar Marti, Ralf Ameling, André Siegel, Manuel R. Gonçalves

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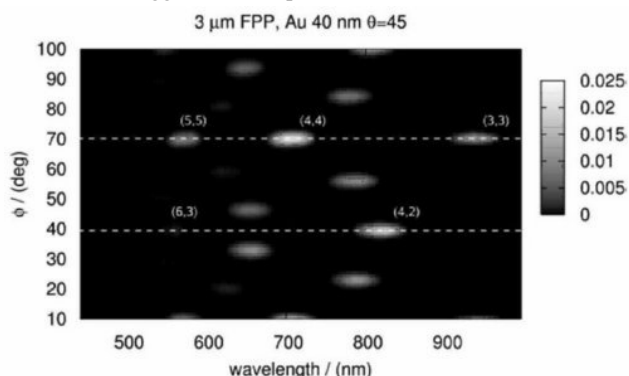
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The interaction of light with metallic microparticles and nanoparticles is strongly modified by resonance effects such as plasmon-polaritons. Size, shape and material of the structures affect the spectral angular scattering distribution. To date only model calculations of light scattering by simple shapes are available. To test analytical and finite element models both near field and far field measurements have been carried out [1,2].

The nanoparticles and microparticles were created with colloidal crystals. Metal evaporation created arrays of triangular particles. Other structures were created by transforming the adsorbed colloidal spheres, by embedding them or by transforming the arrays of metal particles.

The samples were illuminated either in total internal reflection geometry or with light perpendicularly incident on the plane of the particles. Near field measurements were carried out in a WITec alphaSNOM. Scattering data was acquired both with a confocal microscope and with a goniometer setup.

SNOM measurements and confocal microscopy data are compared to model calculations. It was observed that the shape of the particles and their material composition have a strong influence of the intensity distribution in Bragg diffraction patterns.



Intensity of different Bragg diffraction at gold nanoparticles measured by a goniometer. The array of nanoparticles has lattice constant $D=3\mu\text{m}$. (Ref. 1)

References:

1. A. Siegel, M. R. Gonçalves, R. Ameling, and O. Marti, "Investigation of the light scattering form factor of metallic nanostructures using Bragg diffraction", *Journal of Optics A* accepted for publication (2007).
2. M. R. Gonçalves, A. Siegel, and O. Marti, "Influence of the light scattering form factor on the Bragg diffraction patterns of arrays of metallic nanoparticles", *Journal of Microscopy* accepted for publication (2007).

This work was supported in part by the German Science Foundation (SFB 569).

9:30 Oral

A new tool for nano-scale x-ray absorption spectroscopy and element-specific SNOM microscopy

Silvia Larcheri¹, Francesco Rocca¹, Daniel Pailharey³, Franck Jandard³, Roberto Graziola², Giuseppe Dalba², Aleksej Kuzmin⁴, Robert Kalendarev⁴, Juris Purans⁴

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Investigations of complex nanostructured materials used in modern technologies require special experimental techniques able to provide information on the structure and electronic properties of materials with a spatial resolution down to the nanometer scale. The "X-TIP" STRP project supported by the European Commission under the 6th Framework Programme strove to address these needs through the combination of x-ray absorption spectroscopy (XAS) using synchrotron radiation microbeams with scanning near-field optical microscopy (SNOM) detection of the x-ray excited optical luminescence (XEOL) signal.

The new instrumentation developed within this project offers the possibility to carry out a selective structural analysis of the sample surface with the subwavelength spatial resolution determined by the SNOM probe aperture. In addition, the apex of the optical fibre plays the role

of a topographic probe, and chemical and topographic mappings can be simultaneously recorded.

Our working XAS-SNOM prototype is based on: a quartz tuning-fork head mounted on a high stability nano-positioning system; a coated optical fibre tip, operating as a probe in shear-force mode; a detection system (spectrometer with a CCD detector for fast spectral analysis or an analogue detector for imaging) coupled with the microscope head control system; a dedicated software/hardware set-up for synchronization of the XEOL signal detection with the synchrotron beamline acquisition system.

In this talk, we will present the state of the art and the new experimental results obtained with the XAS-SNOM prototype operating at the European Synchrotron Radiation Facility (ESRF) in Grenoble, France. We will illustrate the possibility to obtain an element-specific contrast and to perform nano-XAS experiments by detecting the Zn K and W L3 absorption edges in luminescent ZnO and ZnWO₄ nanostructured thin films.

9:50 Oral

Optical characterization of quantum dash based tunnel injection laser structures for 1.55 μm emission

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The tunnel injection structure idea is to place an auxiliary quantum well (QWell) in the vicinity of quantum dot (QDot) area, separated with a thin barrier only. QWell serves as a reservoir of carriers, which next tunnel laterally into the QDot states. In such a system several fundamental laser parameters can be improved: carrier collection, threshold current and modulation rate.

There is presented an optical characterization of tunnel injection structures designed for 1.55 μm laser emission. The structures are based on InP substrate with InAs quantum dashes (QDashes – elongated QDots) as an active area and InGaAs QWell as a carrier collector. There have been applied different optical spectroscopy techniques for full characterization of tunnel injection structures properties. Photoluminescence (PL) has been used to follow QDash and QWell ground state emission, photorefectance (PR – highly sensitive absorption-like modulation technique) has given an information about energies of optical transitions in the whole structure and photoluminescence excitation (PLE) has allowed to probe directly the energy transfer from QWell to QDash ground state.

PL temperature dependence has revealed two recombination channels: through quantum well and via quantum dash ground states [1]. The strong relative QDash emission has been observed up to approximately 100 K. At higher temperatures the QDash emission decreases significantly in favor of QWell radiative recombination. Low temperature PLE experiment combined with PR measurements has proved the presence of the QWell-QDash ground state energy transfer [2]. The PLE temperature dependence has indicated the carrier transfer presence up to temperatures above 100 K.

[1] P. Podemski, R. Kudrawiec, J. Misiewicz, A. Somers, J. P. Reithmaier, A. Forchel, *Appl. Phys. Lett.* 89, 061902 (2006).

[2] G. Sek, P. Poloczek, P. Podemski, R. Kudrawiec, J. Misiewicz, A. Somers, S. Hein, S. Hofling, A. Forchel, *Appl. Phys. Lett.* 90, 081915 (2007).

10:10

Oral

Effect of the hydrostatic pressure on quantum dot - quantum well tunnel injection structures

Przemysław Poloczek¹, Grzegorz Sęk¹, Krzysztof Ryczko¹, Jan Misiewicz¹, Elizavieta Semenova², Aristide Lemaître², Abderrahim Ramdane²

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Tunnel injection (TI) structure is a novel construction approach to build quantum dot based lasers keeping their main advantages like low threshold current, high temperature stability, wide spectral range, but overcoming simultaneously typically occurring limitations, like low carrier collection efficiency or low modulation rates due to hot carrier problems.

We present the results of optical spectroscopy measurements (photoluminescence (PL) and photoreflectance (PR)) for investigating the properties of a tunnel injection structure based on InAs/GaAs quantum dots (QDs) and InGaAs/GaAs quantum well (QW) separated by a 3 nm thick GaAs tunneling barrier. A set of samples including QD and QW references has been studied.

First, we take advantage of modulation technique (eg. high sensitivities and absorption character of PR spectra) to probe the electronic structure details (transitions energies, energy levels and band gap discontinuities). Further, we investigate the photoluminescence properties versus temperature to probe the thermal sensitivity of the emission intensity and tuning of the TI device properties. As it shows a very strong influence of the thermal quenching on the PL efficiency. The hydrostatic pressure has been used (up to 15 kbar) to modify the band structure (spectral fine tuning of the device). As expected, it has shown a detuning of the QW and QD ground state energies (the pressure coefficients of both parts the TI structure agree with those for the reference samples). The latter, in general, should cause a decrease in the tunneling probability, however, a very strong increase in the QD PL intensity has been observed with the increase of pressure. Its explanation has been based on a compromise between the phonon assisted tunneling process (pressure induced energy shift with respect to the LO phonon energy) and changes in the probability for carrier escape from the dots out of the active region through the well states.

Coffee break

Tuesday morning, 18 September, 10:30

Parallel Session

Spectroscopy

Tuesday morning, 18 September, 11:00

11:00

Invited

3D Characterization of Defect Gradients in the Near Surface Microstructures and Bulk Alloys Via Polychromatic Microdiffraction

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Unprecedented three-dimensional (3D) measurements of unpaired dislocation-density distributions are now possible due to an emerging class of instrumentation: the 3D x-ray crystal microscopes. These instruments use ultra-intense synchrotron x-ray sources and advanced x-ray optics to probe polycrystalline materials with submicron x-ray beams. By employing polychromatic x-ray beams and a virtual pinhole camera method, called differential aperture microscopy, 3D distributions of the local crystalline phase, orientation (texture) and elastic and plastic strain tensor distributions can be measured with submicron resolution in all directions. The elastic strain tensor elements can typically be determined with uncertainties less than 100 ppm. Orientations can be quantified to ~0.01 degrees and the local unpaired dislocation density can be simultaneously measured. Efforts are underway to improve the spatial resolution to less than 50 nm and to extend the technique to allow for measurements deep within materials. Because the 3D x-ray crystal microscope is a penetrating nondestructive tool, it is ideal for studies of mesoscale evolution of defects in materials. The unprecedented new information now coming from this new class of instrumentation will revolutionize our understanding of the materials physics underpinning materials properties.

11:30

Oral

Photon-in photon-out soft X-ray spectroscopy for the study of electronic properties in nanostructured materials: advantages and problems.

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Materials with characteristic structure sizes of a few nanometers are a forefront area of modern materials science due to the possibility to tailor many properties by changing size, shape and dimensionality. Often, such nanomaterials have to be embedded in a matrix in order to be suitable for technological applications, for example in order to achieve stability under ambient conditions or in order to interface to other components of an integrated device. The understanding of how the electronic properties change with size and how they are influenced by their surrounding are important tasks from both industrial and academic points of view.

A large number of publications is devoted to the examination of properties of nanostructured materials by different experimental and theoretical methods. Synchrotron radiation based soft X-ray absorption and emission spectroscopies are excellent non-destructive tools to study the electronic structure of solids. The bulk sensitivity in conjunction with the atom selectivity of the methods allows to study the electronic structure of sophisticated materials such as embedded clusters or nanotubes in bundles. The local and chemical sensitivity allows to

investigate atoms with different chemical environment and trace changes in the electronic structure. The photon-in-photon-out mode is applicable to all materials including isolators. These technical aspects will be demonstrated with results on two specific material systems.

(i) For Si nanoclusters embedded into a silicon dioxide matrix it was found that the density of the states in the valence and conduction bands are size dependent and the band gap size is strongly influenced by the dioxide matrix.

(ii) The electronic properties of Mo-I-S nanotubes and nanowires depend on their stoichiometry and can be explained by changes in the S-Mo bond due to the strong hybridization between S p and Mo d states in the vicinity of the Fermi energy.

11:50

Oral

Contactless electroreflectance spectroscopy in UV region

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Ultraviolet light sources based on III-nitride semiconductors are attracting much attention because of their promising applications in medicine, electronic devices and military systems. Because of wide band gap, AlGaIn/GaN structures are a very promising system to create high power electronic devices operating at high temperatures. However, the realization of high performance devices requires many material investigations by using various experimental techniques including electromodulation (EM) spectroscopy [i.e. photoreflectance (PR) and/or contactless electroreflectance (CER)]. This technique, due to its absorption and differential character is an excellent tool to study the energies of optical transitions (including the excited state ones) in quantum wells (QWs) and quantum dots as well as energies of bulk-like QW barriers or intermediate layers. Because of its high sensitivity to the Franz Keldysh effect, this technique can be used to determine the value of built-in electric fields in semiconductor structures. This issue is especially important for III-nitrides since they are polar structures with high internal piezoelectric fields. However, the application of PR spectroscopy in the UV region can be limited due to the wavelength of modulation beam (deep UV laser sources are still less available and very expensive). This problem can be avoided in CER spectroscopy when a wirelike electrode is used in the capacitor system. In this work we will present our recent progress in the application of CER spectroscopy to study GaN-based structures including AlGaIn/GaN transistor and AlGaIn/GaN QW structures. We will discuss electromodulation mechanism in the CER technique as well as the efficiency of electromodulation in CER technique depending on the capacitor construction.

12:10

Oral

Nanoscale Characterisation of Exciton and Carrier Diffusion in Wide Band Gap Semiconductors using Low Voltage Cathodoluminescence Microscopy and Spectroscopy

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A modern Schottky field emission scanning electron microscope is capable of injecting electron hole pairs into sub-10nm volumes when operating at low voltage (< 1 kV). Under these conditions the electron

probe can generally be considered to be purely diffusive and consequently carrier behaviour can be investigated using scanning cathodoluminescence microscopy and spectroscopy at high spatial resolution. In this paper, the utility of low voltage scanning cathodoluminescence will be demonstrated by presenting temperature resolved studies of carrier and exciton diffusion in technologically important materials that exhibit short recombination lengths, such as Gallium Nitride and Zinc Oxide. Factors that determine the free exciton lifetime in these materials and the impact of the surface on these cathodoluminescence measurements will also be discussed.

Lunch break

Tuesday afternoon, 18 September, 12:30

Parallel Session

Spectroscopy

Tuesday afternoon, 18 September, 14:00

14:00

Invited

X-ray photoelectron spectroscopy for surface and thin film chemical analysis.

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The chemical composition of the surface of a material mostly differs significantly from the bulk or average composition due to adsorption, oxidation and dissolution reactions. This typically occurs in situations such as catalysis, tribology and corrosion. On the other hand the functionalization of metal and metal oxide surfaces with adsorbed molecules is nowadays an attractive technique to form well-adherent molecular layers at the interface. This method might allow the production of tailor-made materials with controlled surface properties.

A technique considered essential for the understanding both the film growth and properties of nanosized films is x-ray photoelectron spectroscopy. It allows not only the identification of all elements present on the surfaces but also their speciation via the chemical shift of the elements belonging to the various functional groups. Quantitative analysis allows determination of the thickness and the composition of the overlayer and of the substrate. The rapid development of new spectrometers together with the possibility of applying new algorithms for data processing and quantification results in non-destructive in-depth profiles of thin films. Imaging – XPS can provide high lateral resolution chemical information. Some recent examples as e.g. self-assembled monolayers on gold, the oxidation of iron and nano-crystalline NiP alloys and the characterization of nano-biofilms will be presented.

14:30

Oral

Luminescence and EPR analysis in investigations of cubic boron nitride

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Due to their exceptional properties cubic boron nitride (cBN) is considered as real advanced and multifunctional material for applications

in microelectronics, optoelectronics, cold emission devices, detectors and of high power microwave devices all working in hard conditions. Luminescence as spectroscopy techniques of characterization of cBN has been used by us for many years. EPR analysis of cBN was rarely used. It is clear now in which way these methods can deliver knowledge about defective structure of cBN and which difficulties can be obtained. As compared to diamond and GaN cubic boron nitride has shown itself as very difficult to be explored in luminescence as well as in EPR analysis. The main reason of these problems is that the material is very pure to show luminescence and EPR output as result of doping or other external influence.

As diamond has showed a lot of luminescence centers after doping, irradiation and implantation which are most of "inter-center" nature, as GaN has got a lot of donor-acceptor pairs and excitons optically active defects... cBN, probably, behaves in different way.

After irradiation and implantation with different ions cBN has shown just 5 optically-active defect, RC-1, RC-2, RC-3 and RC-4 considered as simple Frenkel pairs composed of nitrogen vacancy and some unknown defects. The last fifth optically-active defect C was registered after ion implantation and was considered as large complex based on nitrogen vacancies. Just beryllium has performed itself in luminescence spectra as very reliably identified. A nature of electronic transitions, which is a reason of luminescence centers in cBN, is sorted between the "inter-centers" ones and donor-acceptor recombination. Just one luminescence center related to exciton in cBN was considered.

The same picture is there in EPR analysis. Still 3-4 paramagnetic active defects are obtained in cBN. No irradiation, implantation or doping of cBN has delivered any signals in EPR spectra of cBN.

14:50

Oral

Nanoscale analysis of defects in semiconductors by means of capacitance- and charge-transient spectroscopy

Štefan Lányi¹, Vojtech Nádaždy¹, Miloslav Hruškovíc², Ján Hribík²

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We shall report on the possibilities of analysis of electrically active deep defects in semiconductors by means of modified Deep Level Transient Spectroscopy, the Isothermal Capacitance-Transient Spectroscopy and the Isothermal Charge-Transient Spectroscopy, applied on the sub-micrometre level. While the first of them utilises the relaxation of the depletion layer, caused by emission of trapped charges, and requires sufficient conductivity, in the second the transient current is directly integrated. It can be applied also to low-conductivity materials like dielectrics.

The capacitance microscopes achieved a resolution sufficient to resolve the charge of a single electron. However, the standard interpretation of ICTS response is statistical, therefore many defects should be within the reach of the probe, to be applicable. Our charge-transient spectrometer achieved the resolution of a few hundred electrons but we hope to increase it approximately by one order of magnitude. With optimal shape of the probe a spatial resolution of tens of nanometres can be achieved in materials with relatively high defect concentration. At low concentrations, e.g. in device quality silicon, a resolution on the hundred-nm level can be achieved.

We shall present some results obtained on pentacene films and on thin-film transistors fabricated in crystalline islands in amorphous silicon films.

15:10

Oral

Comprehensive theoretical and experimental evaluation of the impact of the internal polarization fields on the optical emission energy of Si-doped GaN/AlGaIn MQWs and superlattices

Morteza Esmaili¹, Mahmood Sabooni², Hamid Haratizadeh^{1,3}, Bo Monemar³, Per Olof Holtz³

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We report results from detailed optical spectroscopy from MOCVD grown GaN/AlGaIn multiple QWs and superlattices, as opposed to most previous studies where MBE was employed. The wurtzite group III nitrides are tetrahedrally coordinated direct band gap semiconductors having a hexagonal Bravais lattice with four atoms per unit cell. As a consequence of the noncentrosymmetry of the wurtzite structure and the large ionicity factor of the covalent metal-Nitrogen bond, a large spontaneous polarization oriented along the hexagonal c-axis is predicted. In addition, GaN is highly piezoelectric [1]. These macroscopic polarizations play an important role in nitride based QW by producing large built-in electric fields along c-axis.

In this paper we will present theoretical and experimental results demonstrating how polarization induced electric fields and bound interface charges in GaN/AlGaIn MQWs affect the emission peak energy, PL line shape as well as the emission line width. We have calculated the approximate value of spontaneous and piezoelectric and depletion field in these structures. Our results demonstrate that both of polarization fields and depletion fields need to be considered in the interpretation of the experimental data. The theoretically estimated fields in this work are consistent with the experimental data. The transition energy of the heavy hole and electron ground state, E_{e-hh} , in GaN/AlGaIn MQWs were measured using triangular well profile approximation [2]. The theoretically calculated transition energy have been compared to experimental energy position and it is found to be in good agreement with the data.

[1] H. Morkoc; *Nitride Semiconductors and Devices*; ed. By R. Hull, R. M. Osgood, Jr. H. Sakaki and A. Zunger; Springer-Verlage, (1999).

[2] R. Cingolani, A. Botcharev, H. Tang, H. Markoc, G. Traetta, G. Coli, M. Lomascolo, A. Di Carlo, F. Della Sala, and P. Lugli; *Phys. Rev. B* **61**, 2711 (2000).

Coffee break

Tuesday afternoon, 18 September, 15:30

Parallel Session

SEM

Tuesday afternoon, 18 September, 15:50

15:50

Invited

Application of orientation mapping in TEM and SEM for the study of inhomogeneous microstructural evolution during annealing of aluminum alloy with bimodal particle distribution

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In recent years, orientation mapping (OM), i.e. determination of topography of crystallite orientations, has become a well established technique of examining and quantifying microstructures of any mono- and polycrystalline material. The OM technique, performed in the TEM and SEM, covers a spatial resolution range from several to hundreds of nanometers, respectively.

Examination of recrystallization process in aluminum alloy 6013 have been selected to show the application of the OM in microstructure analysis. The alloy contains both large ($>1\ \mu\text{m}$) and small ($<<1\ \mu\text{m}$) second-phase particles. During deformation, the laminar structure and zones of localized strain around large particles (both with characteristic local textures) are formed. *In situ* investigations in TEM as well as calorimetric measurements, combined with OM in TEM and SEM, demonstrate, that recrystallization can be considered as a number of partly overlapping processes, proceeding in two stages. In the initial stage, there occurs particle stimulated nucleation and migration of high angle grain boundaries (HAGB) limited to the areas of deformation zones (DZ) around particles. A certain enlargement of newly-created grains was also observed outside the DZs. Here, however, it appears due to the annihilation of low-angle grain boundaries (LAGB) between the chains of subgrains lying in layers parallel to the sheet plane. As a consequence, new grains often have a plate-like habit, with the shorter axis parallel to the sheet plane normal direction (ND). In the second stage, HAGBs were observed to migrate in the direction of high orientation gradient. This migration, mostly in the ND, was limited to "free areas" of the deformed matrix between bands of the new grains formed in the zones of localized strain in the initial stage of recrystallization.

16:20

Oral

Estimation of the Low Voltage BF/DF STEM Image in an UHR FE-SEM

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Recently, Field Emission Scanning Electron Microscopy (FE-SEM) has been strongly needed in the fields of nano-technology to observe super-fine structure on the sample with ultra high resolution (UHR). In addition, low voltage (30kV) Scanning Transmission Electron Microscopy (STEM) in FE-SEM has been used for inspection and analysis of the nano-scale sample with high contrast image. In order to realize the UHR-STEM observation in FE-SEM, it is essential to have high performance objective lens, high vibration resistance, and contamination free evacuation system. Hitachi S-5500 is an in-lens type UHR FE-SEM with these conditions and provided the new STEM function which can obtain the bright field (BF) and the dark field (DF) images

simultaneously. Performance of the objective lens in FE-SEM is a very important factor to obtain the clear STEM images. We have estimated the spatial resolution of the S-5500 optical system by calculation the optical transfer function (OTF). As a result, it is shown that there is an information value at 0.3nm order in the OTF. We have also obtained the carbon nanotube (CNT) BF-STEM image and it is recognized 0.34nm carbon lattice fringes. On the other hand, DF-STEM can obtain the Z-contrast image. DF-STEM detection system in S-5500 can control the detection angle of the scattered electrons in the sample using motorized detector which can be moved up and down along the optical axis. Therefore, the optimum DF signal corresponded to composition or thickness of the sample, can be obtained by detecting signal scattered wide angle and adjusting detection angle continuously. We have obtained the DF-STEM images of the CNT included iron (Fe) as growth catalyst. When the detection angle is set to 200~400 mrad range, iron part of the image is clearly shown with optimized contrast. In this case, image contrast corresponds to composition (material) information.

16:40

Oral

The use of charging technique for lithium niobate and lithium tantalate crystal investigation by SEM.

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Lithium niobate (LN) and lithium tantalate (LT) ferroelectric crystals are widely used in the field of nonlinear and integrated optics, in acoustics and in other fields due to their outstanding properties. We report scanning electron microscopy (SEM) application for non-invasive domain structure of the crystals visualization and investigation. The SEM was used to study periodically poled (PPLN) and opposite domain structures (ODLN). In addition to the information about the domain shape and sizes, SEM can give information about potential relief distribution on the crystal surfaces, different defects and impurities due to various behavior of surface charging upon electron beam irradiation. Using the surface charging technique, we studied different type of domain walls.

In the investigated samples, periodic domain inversion was initiated by the aftergrowth thermoelectrical treatment[1] and by crystal growth conditions [2]. The congruent LN crystals and crystals with different dopants were investigated and compared.

Analysis of the structures has shown that "tail-to-tail" domain walls are distorted more than others and displayed special images at negative surface charging. The results were supported by x-ray examination of different domain walls in LN and LT. The charge domain boundary images in different crystals were comparatively investigated. The experimental results have demonstrated possibilities of the charging technique for periodical domain structure diagnostic and investigation.

1. N.A.Sorokin, V.V.Antipov, A.A.Blistanov, *Ferroelectrics* **167**, 267 (1995)

2. Red'kin Boris, *Ferroelectrics*, **158**,145 (1994).

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17:00

Oral

Quantitative Dopant Profiling in the SEM including surface states.

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Two-dimensional dopant mapping using secondary electrons (SEs) in a FEG-SEM is a potentially useful technique for studying dopant distributions in semiconducting materials at high spatial resolution. A novel implementation of an attachment to the through-the-lens detector has enabled quantification of dopant profiles to be developed more readily from SE imaging, allowing a non-destructive and rapid in-situ characterisation method of semiconductor devices. However, the reason that it has not yet found widespread application is the lack of an accurate and robust quantification procedure. Although it is known that secondary electron dopant contrast observed across a p-n junction is a function of the built-in potential across the junction, surface band-bending and external local fields (patch fields) above the specimen, the lack of understanding on the respective contributions from all these factors has hampered accurate quantification. To analyse the problem, we have used detailed computer modelling to investigate the effects of surface states and doping concentrations as well as doping feature geometries, on the surface band-bending and external patch fields. We have tested the validity of our calculations against experimentally measured secondary electron energy spectra. The methodology described in this work will help to enable accurate quantification in the dopant mapping of semiconductors in the SEM.

17:20

Oral

Structural Characterization of Carbon Nanofibers using Scanning Electron Microscopy

Makoto Suzuki¹, Takeshi Ogashiwa¹, Shuichi Takeuchi¹, Mitsugu Sato¹, Quoc Ngo², Toshishige Yamada^{2,3}, Alan M. Cassell³, Jun Li³, Cary Y. Yang²

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Carbon nanofiber (CNF), grown by plasma-enhanced chemical vapor deposition, is a nanoscale 1D structure with a diameter of 10-300 nm and has been studied for a wide range of applications including electron emitter array, electronic devices, and interconnects for next-generation integrated circuits. In such applications, the interface structure between the CNF and adjacent materials including the underlying substrate, as well as internal structure of the CNF itself, is essential to ensure its high-performance electrical and thermal properties. Scanning electron microscopy (SEM) is one of the powerful tools to investigate such structures. Especially for the carbon-based nanostructures including CNFs, the surface information and internal structure are obtained by using a secondary electron detector and a transmission electron detector, respectively, if the samples are supported on the electron transparent film. To study the interfacial or internal structure of samples on bulk substrate with SEM, however, generally requires sample thinning, which is destructive. We present two new SEM imaging techniques

developed for CNF devices fabricated on a thick Si substrate without any sample modification. One is an imaging method for the interface between the CNF and substrate, providing the information whether the CNF is in contact with the substrate using top-down SEM imaging. The other technique enables us to obtain a bright-field scanning transmission electron microscopy (STEM) contrast, providing the internal graphitic layer structure of the CNF without destructive sample thinning. We show that the thermal and electrical transport characteristics of the CNF devices are effectively evaluated with these techniques. Detailed analysis using Monte Carlo simulation shows that the secondary electrons from the substrate and efficient detection of these electrons play a key role in both of these imaging techniques.

Wednesday, 19 September

Plenary Session

Wednesday morning, 19 September, 9:30

Coffee break

Wednesday morning, 19 September, 10:30

Parallel Session

Special Session Thin Films

Wednesday morning, 19 September, 11:00

11:00

Invited

Characterisation of phases in nanostructured, multilayered titanium alloys by analytical and high resolution electron microscopy

Aleksandra Czyrska-Filemonowicz¹, Philippe A. Buffat²

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The microstructure, chemical and phase compositions of the hard layer formed on the surface treated Ti-6Al-4V alloy were investigated by LM, XRD, SEM as well as analytical/high resolution TEM, STEM, EDS, electron diffraction and FIB.

The advanced electron microscopy techniques used for unambiguous identification of phases present in the surface multilayer are critically discussed.

Phase identification based on electron diffraction revealed the presence of several binary phases in the system Ti-Ni-P, sometimes partial substitution of Ti by Al. However one of the main phases remained not indexed satisfactorily. EDS microanalysis on FIB samples, free from the composition bias due to forward and backscattered electrons toward the thick part of the sample, shows that its composition is close to TiNiP with an accuracy of a few atomic percents. This "new phase" is absent from the ICCD (PDF-4) and ICSD databases

These investigations show that improved mechanical and tribological properties of surface treated alloy are related to the presence of a multilayered microstructure containing several phases from the Ni-Ti-P-Al system; some of them are nanocrystalline.

11:30

Oral

Piezoelectric response characterization using atomic force microscopy with standard contact-mode imaging

Sharath Sriram¹, Madhu Bhaskaran¹, Ken T. Short², Glenn I. Matthews³, Anthony S. Holland¹

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Standard atomic force microscopes (AFM) require extensive modifications to operate as piezoresponse force microscopes. This paper discusses a technique for observing and quantifying piezoelectric response of thin films, using standard AFMs. The technique has been developed and verified using strontium-doped lead zirconate titanate (PSZT) thin films, which are known for their high piezoelectric response. Quantification of the electro-mechanical voltage co-efficient d_{33} (in pm/V) is directly made based on the applied peak-to-peak voltage and the corresponding peak-to-peak displacement in the obtained scan image.

Under the proposed technique the AFM is configured in contact mode, where the silicon nitride tip is set to follow the film displacement at a single point. A known sinusoidal voltage is applied across the film and the displacement determined as a function of time, rather than the typical AFM measurement of displacement versus tip position. The resulting raster image contains several bands, which are directly related to the AFM scan frequency and the applied sinusoidal voltage and its frequency.

Different combinations of the AFM scan frequency and the applied sinusoid frequency have been used to characterize the PSZT thin films. The films analyzed were deposited under different conditions and were of varying thickness, on gold and platinum coated silicon substrates. Large displacements have been observed for the PSZT thin films, which correspond to d_{33} values above 550 pm/V. These results have also been verified by nano-indentation based piezoresponse tests. The effect of positioning the tip away from the top electrode of samples has been studied, with results in agreement with expected decrease in the electric field strength with distance from the top electrodes.

11:50

Oral

In – situ mechanical characterization of the TiN/NbN, NbN/CrN and TiN/CrN multilayer thin films

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TiN/NbN, NbN/CrN and CrN/TiN multilayer coatings with 20 periods and a thickness of a single layer of around 25 nm were synthesized by

magnetron sputtering. Hardness and Young's modulus of the multilayers films were measured by nanoindentation and compared to reference samples of TiN, NbN and CrN. Some multilayer systems exhibited an increase of hardness, compared to the reference samples. To understand the underlying deformation mechanisms a new nanoindentation instrument based on the Hysitron Picoindenter has been developed for use inside a high resolution scanning electron microscope. The technique allows for observation of pile-up, sinking-in and crack propagation as well as other phenomena during the indentation loading cycle. The in-situ indentations revealed that the pile-up/sink-in behavior of the multilayer is different to the one observed for pure coatings.

To understand the governing sub-surface deformation mechanisms, transmission electron microscopy (TEM) samples from the indented areas were prepared in cross-section via focused ion beam machining. The related TEM micrographs revealed that the thickness of individual layer decreased significantly within the residual imprint. Possible deformation mechanisms related to these observations will be discussed.

12:10

Oral

Nanomechanical properties of Fe-W alloy composite coatings

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In this project, we studied the processing, microstructure and nanomechanical properties of iron-tungsten composite coatings prepared using electro-deposition on copper substrate. We studied the effect of deposition temperature, pH values of the electrolyte bath and the applied current density on the deposits. The microstructure of the deposited Fe-W composites have been studied using electron microscopy and X-ray diffraction techniques. Both amorphous and amorphous/nanocrystalline composite coatings have been made and depth-sensing nanoindentation technique has been used to evaluate the properties of the coatings. The effect of strain rate on nanomechanical properties will be discussed, in conjunction with the nanomechanical properties measured using nanoindentation technique from the iron-based bulk metallic glass. In addition, we will discuss the size effects observed in the nanoindentation tests, including indentation size effect, sample size effect.

12:30

Oral

Surface properties and ozone interaction of indium oxide prepared by metalorganic vapour phase epitaxy

Marcel Himmerlich, Chunyu Wang, Thomas Kups, Volker Cimalla, Juergen A. Schaefer, Oliver Ambacher, Stefan Krischok

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Polycrystalline In_2O_3 thin films have great potential for low cost and low energy consumption environmental sensors, e.g. for the detection of ozone. We have examined the properties of indium oxide thin films (In_2O_3) prepared by metal organic chemical vapour deposition using X-ray and ultraviolet photoelectron spectroscopy (XPS, UPS), electron energy loss spectroscopy (EELS), atomic force microscopy (AFM) and high resolution transmission microscopy (HRTEM). The growth of In_2O_3 was performed on $\text{Al}_2\text{O}_3(0001)$ in a horizontal MOCVD reactor using trimethylindium and H_2O vapour as precursors and N_2 as

carrier gas. The influence of the growth temperature and rate on structure, morphology, stoichiometry as well as the surface electronic properties has been investigated. A dependence of the core level binding energy and the valence band density of states on the growth temperature is found, induced by changes in the surface band bending linked to differences in the resistivity of the films. For samples grown at low temperature (200°C), strong emission from a state below the valence band maximum, which is to our knowledge unidentified up to date, is observed. Additionally, differences in the XPS, UPS and EELS spectra of bixbyite In_2O_3 and samples with rhombohedral crystal structure are presented and discussed. In order to identify the photoreduction and oxidation mechanisms related to the ozone detection capability of In_2O_3 , we have investigated the interaction of indium oxide thin films with O_3 and UV radiation. From the changes of the valence band electron spectra it is concluded that the O_3 interaction is induced by a reversible saturation of defect states at the In_2O_3 surface resulting in adsorption of an oxygen species with two distinct states at 6.1 eV and 11.3 eV in the valence band and an increase of the sample work function.

12:50

Oral

Microstructural studies of $\text{La}_{0.7}\text{Ca}_{0.3}\text{MnO}_3$ (LCOM) films on different (011) substrates deposited by pulsed laser deposition

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Cross-sectional TEM was carried out to understand the effects of microstructure on the physical properties of manganite films on different (011) substrates. $\text{La}_{0.7}\text{Ca}_{0.3}\text{MnO}_3$ (LCMO) films have been grown on (011) surfaces of various (pseudo-) cubic substrates, SrTiO_3 (STO), LaAlO_3 (LAO) and $\text{La}_{0.3}\text{Sr}_{0.7}\text{Al}_{0.5}\text{Ta}_{0.35}\text{O}_3$ (LSAT) by pulsed laser deposition. (011) LSAT substrate and LCMO film have the smallest lattice mismatch, but, (011) LAO substrate induces compressive strain while (011) STO induces tensile strain to the LCMO films. Two different epitaxial relationships have been observed in three different substrates. On LSAT substrate, $[001]_o$ of LCMO is parallel to the $[011]_{\text{LSAT}}$ while the LCMO films show a $[100]_o // [011]_{\text{LSAT}}$ relationship on STO and LAO substrates. LCMO films on STO and LSAT show the single orientation variant with twins as the dominant defects. LCMO film on LAO substrate shows at least 3 variants and with twins. High resolution TEM reveals that LCMO on LSAT possesses a perfect interface and has no misfit dislocations in the area of examination. LCMO films on (110) LAO and (110) STO, have interfaces with atomic sharpness while misfit dislocations can be identified. Films grown on STO and LAO have an easy magnetization direction when the applied magnetic field is in plane along the long-axis of LCMO. In the case of LSAT, there is no in-plane anisotropy. The different in-plane crystal direction and strain distributions in the film may account for the different magnetisation behaviours of the LCMO films on different (011) substrates.

Lunch break

Wednesday afternoon, 19 September, 13:10

Parallel Session

Special Session Thin Films

Wednesday afternoon, 19 September, 14:00

14:00

Oral

Structural ordering of thin amorphous- nano-crystalline films studied by HRTEM, GISAXS and Raman spectroscopy

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Thin nano-crystalline silicon films were deposited on glass, using the PECVD method and 13,6 MHz (RF) gas discharge in silane gas diluted by hydrogen. By varying the dilution, the crystal to amorphous fraction was changed from a few percent up to 70%. The samples were analysed by Raman, HRTEM and GISAXS spectroscopy. The ratio of the areas under corresponding TO phonon peaks in Raman was used to estimate the ratio between the crystal and the amorphous fraction. The crystalline TO peak positions varied between 500 and 521 cm^{-1} which corresponds to average crystal sizes between 2 and 20 nm, considering only phonon confinement due to the nano-size of the crystals. However, the TO line was asymmetric and the broad line-width suggested the existence of also smaller and larger crystals. The size and size distribution of nano-crystals estimated from HRTEM and compared with Raman, show an excellent match in the average individual sizes. However, the distribution was slightly different than those obtained by Raman and suggested a larger contribution of smaller crystals. GISAXS of the deposited samples indicated particles with Gyro radii between 2 and 6 nm. Larger particles are present in samples with a higher degree of crystal fraction and larger crystals. However, the samples are porous to a certain degree and the direct correlation between particles and crystals is not easy to establish. GISAXS obtained by varying the grazing incidence angle allowed to estimate the in-depth distribution of the particles. For growth conditions that favor smaller nano-crystals, the particles were spherically symmetric and their individual sizes were uniform from the surface towards the depth of the sample. For growth conditions that resulted in larger crystals the particles were elongated parallel to the surface and smaller in the sample "bulk". These results were used in the discussion of the difference in the distributions of individual crystal sizes obtained by HRTEM and Raman.

14:20

Oral

Nanoindentation of heterogeneous n-carbon films containing Ni nanocrystals

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Composite films of nano-size nickel grains embedded in a nanocarbon (n-carbon) or amorphous carbon matrix were synthesized by a two source PVD evaporation process of C_{60} fullerenes and Ni acetate.

The structure of the nanocomposite films were characterised by SPM methods, transmission electron microscopy (TEM), selected area electron diffraction (SAD), X-ray diffraction, chemical analysis and atomic force microscopy. Correlations with deposition parameters and typical structure changes are found. The Ni metallic component leads to bi-layered structures of the film and changes its mechanical properties comparing with the carbonaceous matrix properties. The tribological properties were studied using a HYSITRON Triboscope (electrostatic transducer) attached to a Scanning Force Microscope and controller Nanoscope IV. Single and multi-cycling indentation are used to study the depth dependent hardness and indentation modulus. Pop-ins reflects the layered nano-structure.

14:40

Oral

Growth, Optical and Nanostructural properties of ZnO thin films deposited on high-barrier inorganic/polymeric and hybrid/polymeric flexible substrates

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The combination of Transparent Conductive Oxides, characterized by desired optical and electrical properties, with high barrier (hybrid and inorganic) films deposited onto flexible polymeric substrates, is of considerable importance, in order to improve the efficiency, lifetime and stability of Flexible Electronic Devices. In this work, ZnO thin films were deposited onto flexible high-barrier inorganic/polymeric and hybrid/polymeric substrates by Pulsed DC Magnetron Sputtering, at room temperature and by applying different target power voltages. In-situ and real-time Vis-farUV Spectroscopic Ellipsometry (SE) in the 1.5-6.5 eV spectral region has been employed in order to investigate the growth mechanisms of ZnO onto inorganic (SiO_x , AlO_x) and hybrid barrier films from the early stages of growth. The detailed study of the time evolution of the ZnO films' thickness and optical properties (energy gap, absorption peaks, conductivity, etc.), in combination to the investigation of their bonding structure by in-situ and real-time FTIR SE ($900\text{-}3500\text{ cm}^{-1}$), has provided important insights on the layer and interface quality, which have a major impact on the final device performance. Finally, X-Ray Diffraction measurements revealed information on the nanostructural properties and on the effect of the applied power on the ZnO films' crystallinity.

15:00

Oral

Surfactant-based synthesis and characterization of nanostructured V_2O_5 thin films

Alessandro Cremonesi², Gianluca Calestani², Gianni Antonioli¹, Danilo Bersani¹, Pier Paolo Lottici¹

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A surfactant-assisted sol-gel route was chosen in order to synthesize low temperature nanostructured V_2O_5 thin films of orthorhombic structure. The effects of the surfactants on the final structure of vanadium oxide films have been evaluated by XRD, SEM and Raman and XAS spectroscopy.

No traces have been found of hydrated phases. On the contrary, Raman and EXAFS show a high degree of equatorial and axial order, comparable or, for Brij[®]76 and Tween-60, enhanced with respect to commercial crystalline V_2O_5 powder, even in films annealed at 300 °C.

The removal of surfactants from the host metal oxide network as a function of the thermal annealing has been monitored by Raman and EXAFS. Brij[®]76, Triton X-100 and Tween-60 are extracted around 300 °C, while CTAB persists in the matrix at temperatures up to 400 °C. This was confirmed by XRD measurements, too.

The morphologies are mostly driven by the strong anisotropic requirements of the oxide network, forcing the stacking of V_2O_4 chains in lamellar structures. In all cases the spin-coating/evaporation process leads to the deposition of more or less ordered lamellar particles on the substrate. Two different behaviours, related to the persistence of surfactant aggregates during the thermal treatment, may be evidenced:

- In the case of CTAB, the surfactant is still present when the extended crystallization of the oxide network starts around 300 °C; the sintering process becomes effective only after surfactant extraction (around 400 °C): at this stage the nanoparticles are mostly isolated and randomly oriented, so that V_2O_5 lamellas and nanorods elongate at random out of plane distribution with increasing temperature.
- If the surfactant is removed when the crystallization starts (as for other surfactants employed), the in-plane growth of the crystallites is much less perturbed, leading to textured coverage of the substrate by nanobelts and nanorods.

15:20

Oral

Single phase formation mechanism of $\text{Se}_x\text{Te}_{100-x}$ binary alloy confirmed by XRD and TEM method

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High resolution transmission electron microscopy (HR-TEM) and x-ray diffraction (XRD) measurements are done for the $\text{Se}_x\text{Te}_{100-x}$ ($x=16, 29, 38$) thin films crystallized with rapid thermal annealing at 250 °C for 5 min. Composition of each sample is analyzed by Rutherford backscattering spectroscopy and the crystal structures of $\text{Se}_{16}\text{Te}_{84}$, $\text{Se}_{29}\text{Te}_{71}$ and $\text{Se}_{38}\text{Te}_{62}$ thin films are identified as a hexagonal structure of Te basis. Diffraction patterns for each composition are identical, meaning that the structure of the $\text{Se}_x\text{Te}_{100-x}$ films does not change with different compositions. Also, there are no peaks addressed to a hexagonal Se structure. But the 2 theta value which is corresponded to the Te {0 1 1} peak increases with the increase of Se content. This means that the interplanar spacing between adjacent Te {0 1 1} planes becomes to be shorter with the Se content. The calculated spacing between Te {0 1 1} planes for $\text{Se}_{16}\text{Te}_{84}$ is 3.189 Å, that is 3.162 and 3.134 Å for $\text{Se}_{29}\text{Te}_{71}$ and $\text{Se}_{38}\text{Te}_{62}$, respectively. From the HR-TEM images of $\text{Se}_{16}\text{Te}_{84}$ and $\text{Se}_{38}\text{Te}_{62}$, it is found that the interplanar spacing between (0 1 -1) planes of $\text{Se}_{16}\text{Te}_{84}$ is longer than that of $\text{Se}_{38}\text{Te}_{62}$. This is well agreed with the calculated result from XRD. According to the models for the crystal structures consist of Se and Te basis based on the HR-TEM images and the simulated lattice images, it is indicated that the Se atom substitutes the Te atomic site in the Te hexagonal structure, leads to the variation of plane spacing. Because that the atomic size of Se is smaller than Te, it is plausible that the interplanar

spacing between (0 1 -1) planes of $\text{Se}_{16}\text{Te}_{84}$ that has less content of Se atoms to substitute the Te atomic sites is longer than that of $\text{Se}_{38}\text{Te}_{62}$ in the same hexagonal structure.

Coffee break

Wednesday afternoon, 19 September, 15:30

Joint Poster Session (Part II)

Poster Award Ceremony

Wednesday afternoon, 19 September, 15:50

Thursday, 20 September

Parallel Session

Surface Probe Microscopy

Thursday morning, 20 September, 9:00

9:00 Invited

Surface force spectroscopy of polymeric and biological materials under extreme conditions: touching spider legs and fish skin

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We utilized surface force spectroscopy (SFS) for direct point-load measurements of the nanomechanical response of the cuticle and pads on the legs and wind-sensing hairs of live wandering. These measurements allowed for calculations of the spring constant of the hair itself, the torsional constants, the elastic modulus of cuticle and pads, and viscoelastic deformation and adhesion of pads (the damping coefficient). In other related study, superficial sensor cupulae of several fish were compared in terms of size, distribution, morphological differences, and mechanical responses (the elastic modulus and the retardation time) in comparison with the micropatterned, dome-shaped hydrogel structures. The applicability of the AFM and SFS for unambiguous and quasi-quantitative measurements of surface nanomechanical properties is discussed and illustrated for a range of model elastic surface layers including composite polymers,

[1][2] grafted block-copolymers[3], homo-[4] and binary brushes[5] in dry state, under liquids, and at elevated temperatures.[1] T. Choi, J.-H. Jang, C. K. Ullal, M. C. Lemieux, V. V. Tsukruk, E. L. Thomas, The elastic properties and plastic behavior of two-dimensional polymer structures fabricated with laser interference lithography, *Adv. Funct. Mater.* 2006, 16, 1324

[2] V. V. Tsukruk, Z. Huang, Micro-thermomechanical Properties of Heterogeneous Polymer Films, *Polymer*, 2000, 41, 5541

[3] Y.-H. Lin, J. Teng, H. Shulha, V. V. Tsukruk, In-situ Observation of Switchable Nanoscale Topography for Y-shaped Binary Brushes in Fluids, *NanoLett.* 2005, 5, 491

[4] M. Lemieux, S. Minko, D. Usov, M. Stamm, V. V. Tsukruk Direct Measurement of Thermo-Elastic Properties Of Glassy And Rubbery Polymer Brushes Grown By Grafting From Approach, *Langmuir*, 2003, 19, 6126

[5] A. Kovalev, H. Shulha, M. Lemieux, N. Myshkin, V. V. Tsukruk Nanomechanical probing of layered nanoscale polymer films with atomic force microscopy, *J. Mater. Res.* 2004, 19, 71677777

9:30

Oral

DEEM: a novel electron spectromicroscope

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The idea and realisation of the novel electron spectromicroscope DEEM equipped with 5keV electron gun and recently obtained experimental results are reported for the first time. The original DEEM concept of the combination of two parallel imaging columns with a lens system of spherical beam deflectors allows for the simultaneous observation of the two different electron optical planes - selective area diffraction plane (angular distribution) and imaging plane - at two independent imaging units. The dispersive properties of the deflector are used for the selection of the energy range of the emitted electrons. This feature is especially useful if the energy of the illumination radiation is high enough to cause emission of the core level electrons from the sample. As a consequence, energetically and laterally resolved measurements and imaging are possible.

The novel idea of the sample illumination with a high energy (5keV) electron primary beam allows for electron microspectroscopy and electron spectromicroscopy measurements under laboratory condition. A plurality of contrast mechanisms and techniques is used for imaging, including chemical contrast (nanometric imaging- ESCA), AES, energy and laterally selective diffraction, work function contrast, selected area electron spectroscopy etc. An example of selected area electron spectroscopy and energy selective imaging with secondary electrons for a Pd/Si test sample are presented for the first time.

Keywords: imaging nanoscale ESCA, electron spectroscopy, spectromicroscopy, XPEEM, AES

9:50

Oral

Estimating the Surface Diffusion Barrier Difference and the Indirect Gap of ScN(001) by Nano-probing: Scanning Tunneling Microscopy/Spectroscopy

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Scandium nitride thin film is grown on MgO(001) using radio frequency molecular beam epitaxy. Both X-ray diffraction and reflection high energy electron diffraction show that the grown films have a rocksalt structure and are 001-oriented. For growth under N-rich conditions, at which the N-effective flux is more than the Sc flux, the film is found to be stoichiometric resulting in surface morphology of flat-topped mounds and four-sided spiral pyramids. However, under Sc-rich growth conditions the resultant films are under stoichiometric with surface morphology of rounded spiral mounds. This difference of the surface morphology is attributed to the change of the surface diffusion barrier of both growth modes. Since the Sc (N) atom has 3 (5) electrons in the outer shell, 3d1 4s2 (2s2 2p3), ScN is expected to exhibit a

semiconducting behavior. Moreover, due to stabilizing in a rocksalt structure, ScN is predicted to have an optical gap at the X point and an indirect gap between the Γ point (in the valence band) and the X point (in conduction band). Optical absorption measurements confirm the transition at the X point, showing a direct gap of 2.15 eV. However, the measurement of the indirect gap using the same technique is very difficult due to two reasons. First, the absorption coefficient of the indirect gap is significantly less than that of the direct one. Second, the vapor pressure of Sc is low which requires high temperature to evaporate, of 1400 oC, leading to high level of impurity incorporation in the film. This impurity incorporation causes other transitions due to impurity states. In this work, it is presented that how nano-probing, using scanning tunneling microscopy and spectroscopy, is employed to investigate the surface diffusion barriers and the existence of the indirect gap of ScN(001), showing good agreements with the experimental observations and recent theoretical results.

10:10

Oral

Vanadium nano-needles growth on silicon 7x7 surface investigated by scanning tunneling microscopy

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Vanadium oxides, alloys and silicides exhibit a broad variety of electronic, magnetic and structural properties, including high melting point, high hardness and corrosion resistance which made it compounds important for the material engineering field. In order to understand the properties of nanostructured vanadium and vanadium oxides, surface science research has been devoted to investigate the growth of vanadium deposited on different substrates, such as, SiO₂, TiO₂, Cu and Cu₃Au-O. Studies concerning self-organized vanadium oxide nanostructures and vanadium surface reconstructions show the potential applications of vanadium on nanostructured materials as model catalysts and nanodimensional standards. In this work, we investigate the size distribution and shape transition of self-assembled vanadium silicide nano-needles and nanoclusters growth on Si(111) 7x7 surface by scanning tunneling microscopy (STM). The nanostructures were formed by submonolayer vanadium deposition at room temperature up to 1100K (called as reactive deposition epitaxy). As a function of temperature, both, flat (2D) hexagonal shaped clusters and needle-clusters have been obtained. Substrate temperature and also deposition rate can be adjusted in order to tuning the nanostructures features. The nano-needles grow following a severe surface energy minimization, as a step-by-step growth at single half unit 7x7 cell as a detailed image analysis suggest. The nano-needles growth direction follow a corner-hole alignment which grows at certain 60° with respect each other at the substrate isotropic directions. The nanostructures have typical aspect ratio range of 4-8 and height range of 1-2nm. Based on high resolution STM images an on-top structure model for the nano-needles exposing atomically flat surfaces was proposed.

Acknowledgements

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Coffee break

Thursday morning, 20 September, 10:30

Parallel Session

Surface Probe Microscopy

Thursday morning, 20 September, 11:00

11:00

Invited

Scanning Force Microscopy – Applications in Magnetism and Surface Science

Hans J. Hug^{1,2}, Iris Schmid¹, Peter Kappenberger¹, Sara Romer¹, Tim Ashworth², Magdalena Parlinska-Wojtan¹, Quang Thai¹, Sasa Vranjkovic¹, Raphaëlle Dianoux¹, Shigeki Kawai¹

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Since its invention in 1986 the AFM has evolved into an instrument that is able to map various physical or chemical properties on a local scale with up to atomic resolution. The measured tip-sample interaction is due to long and short range forces. Among these are electrostatic, magnetic, van der Waals forces and short-range forces due to incipient chemical bonds between the tip apex and the sample atoms, respectively.

In a first part, I will first concentrate on the mapping of magnetic structures with high sensitivity and spatial resolution. Using ultra-high aspect ratio silicon tips coated with ultrathin ferromagnetic layers, magnetic stray fields can be routinely imaged with 10nm spatial resolution, and sub-monolayer sensitivity. The latter turned out to be particularly useful to map the spatial distribution of uncompensated spins at the interface between ferromagnetic and antiferromagnetic thin films. Based on this information an improved understanding of the exchange bias effect was obtained and materials with higher exchange anisotropy energies could be designed and fabricated.

In a second part, atomic resolution imaging will be addressed. Since the first demonstration in 1995 atomic resolution images were obtained on a large variety of surfaces. Site-specific measurements of the tip-sample interaction forces performed at cryogenic temperatures have provided relevant information on the nature of the incipient chemical bond between the tip apex and selected surfaces sites and on the imaging mechanisms. However, the routine use of AFM in surface science has still remained much more challenging than in the case of STM. This is presumably due to the presently used operation modes. Approaches to overcome the present limitations in AFM will be discussed.

11:30

Oral

Local Dynamic Mechanical Analysis

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At the nanometer scale, thin film coating represents unique enhancement in terms of mechanical properties compared to conventional

coating. Even though deposition process and characterization of such films have been widely reported in the literature, quantitative mechanical measurement remains difficult. Measurements made on thin films bonded on thick substrate often show the substrate properties.

Among the discussed characterization techniques, Nanoindentation provides material properties measurements and can directly be compared to well-established Dynamic Mechanical Analysis (DMA). Traditional nanoindentation refers to quasi static indentation testing in the submicrometer range. In such measurements, a diamond tip is pressed into the sample, load and displacement of the probe are recorded and analysis models based on load displacement data can be then interpreted to obtain mechanical properties of the sample.

In contrast to quasi-static measurements, Nanoindentation can be used in ac mode by modulating the force and/or the displacement; this mode enables quantitative studies of dynamic mechanical properties like viscoelasticity and can be compared to classical Force Modulation Microscopy (FMM). Existing dynamic nanoindentation setups are based on force modulation while FMM is operated in both force and sample modulation.

We have realized a dynamical nanoindentation setup, Local-Dynamic Mechanical Analysis (L-DMA), based on both sample and tip modulation, in order to allow a direct comparison between those techniques and consistent quantitative mechanical measurement.

Bulk materials as well as thin films of ceramics and polymers have been used for testing and validating the setup. The instrument has been modelled in sample modulation mode and experimental results obtained for different materials where compared with simulation data.

11:50 Invited

Quantitative transmission electron microscopy for the analysis of interfaces and quantum nano-structures

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Quantitative TEM characterisation techniques have been successfully developed in the recent years. We are now able to probe physical properties such as structure, electronic and magnetic structure quantitatively with sub-Ångström to nanometer resolution. The challenge in quantitative TEM methods consists in the correlation of spectroscopic, image or diffraction intensities with the physical properties of a given sample. Several challenges must be met such as highly reproducible acquisition conditions and a quantitative understanding of the electron sample interaction. In this paper, we will present examples in which TEM techniques in quantitative electron energy loss spectroscopy (EELS) and quantitative image interpretation were developed and refined.

In tribology, hard coatings are subject to friction against metals and to understand wear, the initial states of the tribological processes are studied. The FIB is used to localise these areas of starting wear and to prepare a TEM lamella from this area. By applying energy filtered imaging (EFTEM) and high resolution TEM, we obtain atomic to nanometer scale information. This chemical information can be correlated with tribological models of wear on surfaces.

In the field of semiconductor physics, the understanding of the energy level structure of individual QDs remains non-trivial and essential to engineer the optical properties of a QD. The quantitative characterisation of the QD structure involves the localisation of the quantum dots, optimisation of the inherently low signal to noise ratios and the char-

acterisation of the 3D structure. The correlation between QD structure and luminescence spectra through energy level calculations yielded excellent agreement.

Lunch break

Thursday afternoon, 20 September, 12:30

Parallel Session

EFTEM

Thursday afternoon, 20 September, 14:00

14:00

Invited

Study of Tribochemical Processes by Energy-Filtering TEM

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Energy-Filtered TEM is now a technique widely used in the field of tribology, in lubricated wear as well as in dry friction. Two preparation methods have been developed in the past 5 years, first is the observation of thin wear particles extracted from the tribofilm collected on a carbon film, the second is the direct observation of the tribofilm itself by the FIB technique. Also two kind of microscopes can be used, either the in-column integrated filter working in the image-spectrum mode or the FEG-HRTEM microscope equipped with the EELS detector, more dedicated to line scan studies.

Here we present applications of these techniques to investigate different tribological systems. First example is the formation of tribofilm by the tribochemical reaction with antiwear additives (containing phosphorous, sulfur and boron). A second example is the tribology of diamond-like coatings. A third example is the use of nanolubricants to replace phosphorous and sulfur in future engine oils. Fullerenes, nanotubes and onions have been used and the results of the tribochemical reaction has also been investigated by EF-TEM.

14:30

Oral

Analytical TEM study of the formation of perovskite-type oxides by the EDTA/citrate complexing method

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Several complex oxides of cubic perovskite-type are excellent mixed ionic-electronic conductors (MIEC). Interest in these materials increased in the last years as they allow the supply or removal of oxygen to or from reaction mixtures with unrivalled selectivity and high oxygen fluxes. Today, the synthesis of highly-doped double perovskites is widely done via EDTA/citrate complexing methods (or derivatives of them) as they allow to obtain phase pure products and to control precisely their stoichiometry. However, details of the formation of the perovskite crystal are little known. The synthesis process for cubic perovskites of (Ba,Sr)(Zn,Fe)O_{3-d}-type and (Ba,Sr)(Co,Fe)O_{3-d}-type have been studied in very detail by analytical transmission electron microscopy in a field-emission instrument. The methods applied are diffraction contrast (bright-field and dark-field), high-resolution transmission electron microscopy (HRTEM), elastically-filtered selec-

ted area electron diffraction (SAED), energy-filtered transmission electron microscopy (EFTEM), scanning transmission electron microscopy (STEM) in bright-field (BF) and high-angle-annular dark-field (HAADF) contrast, energy-dispersive X-ray spectroscopy (EDXS), and electron energy-loss spectroscopy (EELS) with special emphasize on energy-loss near-edge structures (ELNES). It turns out that the perovskite-type oxides are formed already at moderate temperatures at around 700 °C via nanoscale solid state reactions between finely-dispersed crystalline intermediates identified as a spinel and a carbonate. The analytical TEM study gives a clear picture of reaction pathways and mechanisms as it includes orientation relationships between intermediates and products.

Coffee break

Thursday afternoon, 20 September, 15:30

Parallel Session

TEM Holography

Thursday afternoon, 20 September, 15:50

15:50

Invited

Off-axis electron holography: beyond atomic structure determination

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Recent progress in instrumentation, namely the invention of the corrector for the spherical aberration of the objective lens (Cs-corrector), allows now the almost dislocalization-free imaging of the atomic structure in a transmission electron microscope (TEM). Since the atomic structure determines the macroscopic properties, the answering of the key question "which atoms are where" is an important step for derivation of structure-properties relationships.

But even a Cs-corrected TEM is a compromise because aberrations are deliberately introduced to produce phase contrast imaging. Furthermore, the recording of conventional image intensity causes the loss of the electron wave's phase resulting in a considerable lack of long-ranging object information like e.g. inner electric potentials and magnetic fields.

In a TEM, off-axis electron holography solves the phase loss problem of imaging by interfering the object-modulated electron wave with an unmodulated reference wave. From the recorded interference pattern, amplitude and phase of the electron wave is numerically reconstructed using the laws of Fourier optics.

By this, a new window into the nanoworld is opened allowing direct measurements of tiny phase differences caused by electric potentials and magnetic fields. For example, the structured incorporation of dopants into the crystalline Si-substrate produce potential differences in semiconductor devices. Since the phase shift of the electron wave is directly proportional to the potential difference, magnitude and position can be measured by holography, giving important insights into the relationships between the microscopic electric structure and the macroscopic performance of the device.

The close cooperation with the group of Prof. Hannes Lichte from Triebenberg Lab (TU Dresden) is gratefully acknowledged.

16:20

Oral

Quantitative analysis of two-dimensional dopant profile in Mg doped GaN

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Recently, owing to their wide direct band gap, GaN materials have used increasingly in the field of commercial industries, such as back lighting of liquid crystal display device, white light, traffic light and car lighting. To improve the efficiency of GaN devices, it is necessary to achieve high P-type doping levels of $10^{19} \sim 10^{20} \text{ cm}^{-3}$. However, in Mg doped p-type GaN, native defects, residual impurity and complexes passivate or compensate Mg acceptors during growth [1-5]. The ability to control the Mg concentration and the other impurities during the p-GaN growth will lead to improved GaN device quality and functionality [5]. Therefore, it is important to know Mg doping distributions and concentrations in p-GaN.

In this study, electron holography, scanning capacitance microscopy (SCM), and cathodoluminescence (CL) were used to obtain two-dimensional doping profile of Mg doped p-type GaN. The samples were grown by Metal Organic Chemical Vapour Deposition (MOCVD) and consist of alternating 250nm layers of un-doped GaN and Mg doped GaN. We were prepared for electron holography measurement using FIB (focused ion milling) with low kV milling for which samples was removed ion damage layers. Successfully, we obtained quantitative doping profile with Mg doped p-type GaN. We compared the doing profiles of electron holography, SCM, and CL to secondary-ion mass spectrometry (SIMS) data and discussed their results.

Reference

- [1] H. Amano, M. Kito, K. Hiramatsu, and I. Akasaki, *Jpn. J. Appl. Phys.* **28**, L2112 (1989).
- [2] U. Kaufmann, M. Kunzer, M. Maier, H. Obloh, A. Ramakrishnan, b. Santic, and P. Schlotter, *Appl. Phys. Lett.* **76**, 1326 (1998).
- [3] F. Shahedipour, and B. W. Wssels, *Appl. Phys. Lett.* **76**, 3011 (2000).
- [4] O. Gelhausen, H. N. Klein, M. R. Phillips, and E. M. Goldys, *Appl. Phys. Lett.* **81**, 3747 (2002).
- [5] O. Gelhausen, M. R. Phillips, and E. M. Goldys, *J. Phys. D: Appl. Phys.* **36**, 2976 (2003).

16:40

Oral

High sensitivity and high resolution reconstruction of sample structure, chemical composition and crystalline phase by combining of transmission, fluorescence and diffraction synchrotron microtomography

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When illuminating a sample with x-rays, a number of interactions occur revealing different physical properties of the sample. Using appropriate detectors suitably arranged around the sample, we propose a novel synchrotron measurement tomographic scheme to map structural, chemical and nanocrystalline details of the inner part of a sample.

Microtomography is a well-established technique to reconstruct the sample internal structure. With the advent of third generation synchrotron sources, high-resolution tomography could be reached that reveals details within a micron. With fluorescence tomography, 3D quantitative chemical imaging is also possible by measuring fluorescent x-rays in a first generation scanner geometry.

We propose here to record a diffraction pattern simultaneously to the fluorescence and transmission signal. It consists in translating the sample in front of a high intensity monochromatic pencil beam. At each point along the linear scan, we record the incoming and outgoing intensities for transmission tomography, a fluorescence spectrum for chemical imaging, and a diffraction pattern for crystalline phase imaging. Then, we rotate the sample and we repeat the process until one turn is achieved. After integration of the images to obtain linear diffraction patterns, region of interest corresponding to each Bragg peaks are selected to generate sinograms. Using a classical algebraic reconstruction technique, we compute an image of the inner distribution of each crystalline phase.

Measurements have been performed at the ESRF beamline ID22. Apart from the validation of the method with phantoms, three examples covering three engineering topics have been tested. High contrast images have been obtained when classical tomography did not work. The method gives a highly suitable combination of chemical and crystalline data and its sensitivity has been proved to be much higher than in classical microdiffraction analysis.

17:00 Oral

Small angle neutron scattering study of interface nanostructure in sintered Nd-Fe-B magnets processed under high magnetic fields

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Sintered Nd-Fe-B magnets are the most promising materials for a driving motor of the hybrid electric vehicle (HV). In this application, a large value of coercivity H_c is required, because the operating temperature of magnets reaches beyond 450 K. As a symptomatic treatment, Nd has been replaced partly by Dy to enhance H_c . But the problem is an estimated short supply of Dy in the HV mass-production stage due to its low natural abundance. We have recently shown^[1] that H_c of Nd-Fe-B magnets can be enhanced by applying a high magnetic field during the annealing. In order to investigate the reason for the H_c increase, we started a small angle neutron scattering (SANS) study. SANS technique has an advantage over microscopic methods. That is,

we can non-destructively obtain a quantitative information averaged over a bulk sample. In the present study, two kinds of Nd-Fe-B samples with and without Cu and Al additives were prepared. The 99% enriched ¹¹B isotope was used to avoid high neutron absorption of ¹⁰B in natural boron. SANS experiments were performed with SANS-J and PNO apparatus equipped at JAEA in which the incident wave length of neutrons were 0.65 and 0.2 nm, respectively. In the samples without Al and Cu additives, the intensity of scattered neutrons was proportional to the q^{-4} where q is the scattering vector. When the sample contains Al and Cu additives, however, we observed a marked deviation from the simple relation above for $q > 0.3 \text{ nm}^{-1}$. We have reproduced these experiments by a calculation, assuming a structure such that a core of Nd₂Fe₁₄B phase is surrounded by a Cu-rich shell with a thickness of 4-8 nm. We already reported^[1] that the DSC measurements suggest an existence of Nd-Cu and Al-Cu eutectic phases at the grain boundary. The DSC and SANS results thus suggest that an existence of the Cu-containing liquid phase is strongly correlated with the H_c enhancement by the high field annealing.

[1] H. Kato et al., J. Magn. Magn. Mater. 310, 2596 (2007).

Friday, 21 September

Parallel Session

TEM

Friday morning, 21 September, 9:00

9:00 Invited

HRTEM and TEM studies of amorphous structures in ZrNiTiCu base alloys obtained by rapid solidification or ball milling

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Amorphous structure in melt spun or ball milled alloys was studied in ZrNiTiCu system with Mn, Nb and Ag additions. TEM studies of melt spun ribbons from alloys far from eutectic composition has shown usually presence of nanocrystals while that near eutectic only amorphous structure. The introduction of even small amount of 3 at. % of Al into high copper eutectic ZrTiNiCuMn alloys resulted in formation of additional coarse dendrite like primary crystallites. They were eliminated by decreasing the Al content down to 1,5 at%, i.e. from Ti₁₅Zr₁₅Ni₂₅Cu₃₉Mn₃Al₃ to Ti_{15,5}Zr₁₄Ni₂₇Cu₃₉Mn₃Al_{1,5} (in at. %). The glass transition range $\Delta T_x \sim 26^\circ\text{C}$, $T_x \sim 482^\circ\text{C}$ and the reduced glass transition $T_{gr} \sim 0,59$ was determined in melt spun ribbons. The *in-situ* heating of thin foils within the crystallization range was applied to introduce controlled amount of nano-crystallites.

TEM studies of ball milled powders showed presence of nanocrystallites of size 2-5 nm and in consolidated compacts Cu₅₀Ag₁₀Zr₃₀Ti₁₀ amorphous matrix with nanocrystals of size close to 15 nm. The electron diffraction pattern showed amorphous hallow and a weak rings due to crystalline phases like Zr₂(Cu,Al), Zr₂Cu, Ti₂Ni and α -Ti, β -(Ti,Zr). The diffused rings makes difficult identification of a crystal structure. The crystallites were imaged using HREM. Interplanar distances were measured in pictures obtained by IFFT (Inverse Fast Fourier Transform) of atomic planes to obtain better contrast. Following

the HREM analysis of fringe spacing and angles CuZr_2 phase was identified. Similar analysis was performed in the consolidated $\text{Zr}_{36}\text{Cu}_{29.5}\text{Ni}_{19}\text{Ti}_{10}\text{Nb}_{5.5}$ alloy from ball milled powders. Analysis of the IFFT from high resolution micrographs allowed to identify $\text{Cu}_{10}\text{Zr}_7$ phase. Point analysis and elemental mapping performed using nondispersive X-ray energy spectroscopy showed uniform elements distribution indicating that chemical segregation to nanocrystals is within a measurement error.

9:30

Oral

TEM microstructural analysis of a single crystal CMSX-4 superalloy creep deformed at high temperature

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To withstand extreme conditions of high temperature and stress, modern aircraft turbine engines require the use of single crystal (SC) nickel-base superalloy turbine blades. A microstructure of SC superalloys consists of a cuboidal coherent precipitates of γ' phase surrounded by γ phase channels. The high temperature creep strength of SC alloys results both from the coherency and the nanoscale dimensions of the γ phase. The aim of the present work was to investigate the microstructural changes during creep of second generation SC CMSX-4 superalloy. Chemical composition of the alloy investigated is as follows: Ni - 9.5Co - 6.4Cr - 6.4Ta - 6.4W - 5.6Al - 2.9Re - 1Ti - 0.6 Mo - 0.1Hf (wt %). The creep tests were carried out at 750°C and 675 MPa as well as 950°C and 185 MPa. The temperature and stress values were selected to cover the range typical for operating conditions of turbine blades. The tests were terminated at the beginning of the secondary creep stage. TEM microstructural analysis were performed on thin foils prepared from {001} longitudinal sections of creep deformed specimens.

In the CMSX-4 deformed at 750°C and 675 MPa, stacking faults incorporated within extended dislocation ribbons passing through γ matrix and γ' particles were observed. Both extrinsic and intrinsic character stacking faults were identified. Inside the γ channels the formation of dislocation networks was detected.

Microstructure of the specimen creep tested at 950°C and 185 MPa exhibited development of directional coarsening of γ' particles from a cuboidal to a plate-like morphology, so-called rafting. TEM analysis showed the dislocation networks on γ - γ' interfaces and rarely cutting of rafted γ' particles by single dislocations.

The results of TEM investigations indicated the two different creep mechanisms of CMSX-4 superalloy. It is of importance in interpreting the creep behavior of the single crystal aircraft turbine blades, and thus determining their life-time.

9:50

Oral

Enhanced thermal stability of gold and silver nanorods through thin surface layers

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The thermal stability of noble metal particles is of interest both for fundamental reasons and because gold and silver nanoparticles show a unique optical response (plasmons) making them useful for a range of optical applications.

Using in-situ TEM, we find that a carbon shell governs the morphological transitions of gold and silver nanorods upon heating.

Performing the in-situ heating experiment with silver nanorods leads to a surprising observation: the silver nanorods are stable against heating to temperatures close to the bulk melting point, show no or little shape transformation, but eventually a strongly anisotropic sublimation. A thin cage-like structure with the original particle dimension is left behind.

To study the role of the material, we also investigated samples of gold nanorods. Covered rods slowly change their shape only close to the bulk melting temperature, i.e. at considerably higher temperatures than pristine gold nanorods. However, gold nanorods never show the strongly anisotropic sublimation behavior as observed with silver nanorods.

The fast and anisotropic sublimation behavior of silver particles may be explained by the different vapor pressures of the two materials. The observed difference between gold and silver nanorods is a strong hint how slightly different surface stability and surface chemistry affects the nanocrystal stability of otherwise similar materials to an unexpected degree. The thermal stabilization of gold rods by a thin surface coating may help in high power applications (e.g. enhancement of nonlinear optical effects) by pushing the thermal destruction threshold considerably.

10:10

Oral

TEM observations of capillarity-driven migration of a thin wedge in the Au/Ge system.

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This work reports on the morphological evolution of an anisotropic, solid, thin wedge on the deformable substrate. Mazed bicrystal films of Au have been grown epitaxially on {001} and {111} Ge substrates by physical vapor deposition. The epitaxial relationships and morphological features of these films were characterized by transmission electron microscopy and diffraction in plan view and cross section. In the course of our study on the kinetics of grain coarsening of Au mazed bicrystal thin films, we observed interesting phenomena at the triple line where a free-standing film encounters a wedge shaped substrate. The morphological effects dictated by the capillary forces at the Au/Ge interface result in a diffusion induced grain boundary migration in the film as well as a retraction of the wedge shaped substrate. The wedge shaped film/substrate geometry is of particular interest as a simple model system for the study of the stability of nonequilibrium configurations. Combination of in situ TEM annealing experiments with FIB specimen preparation resulted in 3-dimensional characterization of the observed morphological changes and enabled us to elucidate the mechanism of the transformation. A direct parallel with the behavior of spreading/retracting of a liquid droplet on a deformable substrate was observed. In the absence of kinetic limitations, capillarity as a driving force acts on two levels: the thin wedge migrates over the large distances in order to decrease overall energy of the system; in addition, rearrangements at the wedge tip result in the establishment of local equilibrium at the triple line. The local equilibrium configuration at the triple junction is strongly influenced by the anisotropic nature of

the solids involved. This work is supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Materials Sciences Division of the U.S. Department of Energy under Contract No. DE-ACO3-76SFOO098

Coffee break

Friday morning, 21 September, 10:30

Parallel Session

TEM + Closing

Friday morning, 21 September, 11:00

11:00

Oral

Electronic Structure of Pd Nanoparticles on Carbon Nanotubes

Alexandre Felten¹, Jacques Ghijsen¹, Jean-Jacques Pireaux¹, Wolfgang Drupe², Robert L. Johnson³, Duoduo Liang⁴, Gustaaf Van Tendeloo⁴, Michel Hecq⁵, Carla Bittencourt⁵

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The performance of modern electronic devices based on carbon nanotubes (CNTs) is influenced by a potential barrier at the metal–CNT contact that governs electron injection. The study of metal–CNT interactions is essential to achieve low-resistance ohmic contacts with nanotubes and thus their integration in new nano-devices.

Palladium appears to be the most promising contact metal; ballistic transmission of electrons has already been reported. However, there is no obvious reason why Pd should give a smaller barrier than e.g. Ti.

In order to understand the Pd–CNT interaction, MWCNTs with different amounts of Pd evaporated onto their surface were studied by high-resolution transmission electron microscopy (HRTEM) and Photoemission Spectroscopy. XPS and UPS measurements were performed at BW2 and FLIPPER II beamlines respectively, at Hasylab.

HRTEM images show the evolution from dispersed clusters to a quasi-continuous coating for increasing amounts of Pd evaporated onto the pristine CNT surface. Oxygen plasma treatment improved the cluster dispersion and reduced the size distribution by grafting oxygen groups on the CNT surface, which mediate the overlayer morphology. The preserved structural characteristic of the graphene layer under the Pd cluster and the absence of features near the Pd 3d doublet and C 1s on the XPS spectra are strong indications of the absence of a mixed Pd–C phase.

The observed shift in the Pd 3d core level towards higher binding energy can be associated with a poor final-state screening or charging possibly resulting from the reduced size of the clusters and poor charge transfer between the CNTs and Pd clusters. The different electrical contact behaviour of Pd and Ti will be discussed.

This work is financially supported by the Belgian Program on Interuniversity Attraction Pole (PAI 6/1), by Nano²Hybrids (EC-STREP-033311), and by DESY and the European Commission under contract RII3-CT 2004-506008 (IASFS). JG is research associate of NRSR (Belgium).

11:20

Oral

Evaluation of crystal structure and polytypism of SiC in microwave sintered diamond–SiC composite by TEM and HR-TEM

Paulina Unifantowicz^{1,2}, Sébastien Vaucher, Malgorzata Lewandowska², Krzysztof J. Kurzydłowski²

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TEM technique is used for an investigation of silicon carbide crystals produced during reactive microwave sintering of diamond monocrystals and silicon powder. The presence of double SiC layer on the diamond (100) face has been revealed which consists of SiC nanocrystals at the diamond interface and microcrystalline SiC at the top. Despite the presence of disordered interlayer, crystallographic orientation of the latter was found to match the orientation of the diamond substrate. Electron diffraction images show several polytypes of alpha-SiC. The results of the TEM and HR-TEM analysis will be presented and discussed in detail, and their implication to the SiC growth mechanism will be discussed.

Lunch break

Friday afternoon, 21 September, 12:30

Parallel Session

Friday afternoon, 21 September, 14:00

Coffee break

Friday afternoon, 21 September, 15:30

Parallel Session

Friday afternoon, 21 September, 15:50

Posters

Monday, 17 September

Joint Poster Session (Part I)

Monday & Wednesday

Monday afternoon, 17 September, 15:50

15:50 Poster J1

TiO₂-noble metal particles based porous nanocomposites for pollutants sensitive detection via surface-enhanced Raman spectroscopy

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Surface-enhanced Raman spectroscopy (SERS) became in the last decades one of the most promising techniques for a large variety of applications due to the significant enhancement (10^3 - 10^{12} order of magnitudes) of the Raman signal of the molecules adsorbed on noble metal nanostructured surfaces. The enormous Raman signal enhancement of a species adsorbed on a metallic surface is basically explained by considering the contribution of two enhancement mechanisms: an electromagnetic (EM) mechanism associated with large local fields caused by electromagnetic resonances occurring near metal surface, and a charge transfer (CT) mechanism involving a scattering process associated with chemical interactions between molecules and the metal surface. Several research groups have exploited the benefit of the Raman enhancement and used even metal colloids (gold and silver), gold-metal island films or other substrate types based on silver-polydimethylsiloxane nanocomposites or ultrathin TiO₂ deposited on silver island films for the detection of pollutant molecules [1-3]. By taking into account the affinity of the sulphates, chlorates, amino and hydroxyl based pollutants to bind to the noble metals surface we propose in the present work to show the potential of some recently prepared nanocomposites based on titania aerogel and silver or gold colloidal particles to detect pollutant species adsorbed from an aqueous solution on the noble metal surfaces. Prior evidencing the detection efficiency of the composites for different pollutant molecules the existence of their porous-like structure was proved with the help of scanning electron microscopy (SEM) images.

References

1. H. Schmidt, N. B. Ha, J. Pfannkuche, H. Amann, H. D. Kronfeldt, G. Kowalewska, *Marine Pollution Bulletin*, 49, 229–234 (2004).
2. M. A. De Jesús, K. S. Giesfeldt, M. J. Sepaniak, *J. Raman Spectrosc.*, 35, 895-904 (2004).
3. L. Bao, S. M. Mahurin, S. Dai, *Anal. Chem.*, 76, 4531-4536 (2004).

15:50 Poster J2

Photoemission study of the electronic structure of tris-(8-hydroxyquinoline) aluminum / clean Al and tris-(8-hydroxyquinoline) aluminum / self oxidation Al interfaces in organic light-emitting devices.

Kwanghee Cho¹, Sang Wan Cho¹, Yeonjin Yi², C. N. Whang¹, Kwangho Jeong¹

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The electronic structures of tris tris-(8-hydroxyquinoline) aluminum (Alq3)/ clean Al and Alq3 /self oxidation Al interfaces were studied

using in situ ultraviolet and x-ray photoelectron spectroscopy (UPS and XPS). The UPS and XPS spectra allowed us to evaluate the complete energy level diagrams and to analyze the chemical interactions at the interfaces. For structure of Alq3 / self oxidation Al, the onset of the highest occupied molecular orbital level (HOMO) of Alq3 was shifted 0.31 eV toward high binding energy comparing to structure of Alq3 / clean Al. Work function of Al is reduced from 4.2 to 3.58 by self oxidation of Al and this work function reduction is reason of reducing height of the electron injection barrier.

15:50 Poster J3

Growth and characterization of branched carbon nanostructures arrays in nano-patterned surfaces from porous silicon substrates

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A method to fabricate carbon branched nanostructures arrays is presented. We employ the electron-beam-induced deposition method using a transmission electron microscope in poor vacuum conditions where hydrocarbons are present in the chamber. The hydrocarbons are attracted to the substrates by the local electric fields. The process can be monitored in real time at the nanoscale level. Saw-tooth nano-patterns were made with a focused ion beam in porous silicon substrates with high porosity in order to create sites with high local electric fields. We found that the adequate ion dose to create well defined saw-tooth nano-patterns was between 2 and 8 nC/mm². Micro Raman studies for the branched nanostructures showed two sharp peaks near 1500 cm⁻¹ and 1350 cm⁻¹ which are consistent with the G and D peaks of amorphous carbon. Electron energy loss spectroscopy in the high loss region confirms this assignment. Raman and energy-loss near-edge structure were compared in terms of sp²/sp³ calculations. Selected area electron diffraction, high resolution images and energy dispersive X-ray analysis are also presented.

15:50 Poster J4

New applications for interference microscopy

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Interference optical microscopy (IOM) is a 3D nanometrology tool with sub-nanometer vertical resolution. It is widely used in both academic and industrial research and development. We have developed our own IOM in order to address several scientific and industrial applications that would not have been possible with most standard IOM setups.

For example, we have written our own optical path difference (OPD) reconstruction algorithms [1] and compared their performance with standard reconstruction methods in white-light interference microscopy.

We also integrate the IOM to other instruments such as an Atomic Force microscope (AFM) and a nanoindenter [2], resulting in a very powerful tool for MEMS characterization.

The full access to the individual correlograms enabled us to implement a "thickness mode", i.e. a way to image multiple interfaces of semi-transparent layers [3]. Along the same line, a special mode have been

implemented which allows to quickly image with nm resolution two surfaces which have a height difference up to 200µm.

Finally, we have used a high resolution CCD with a 2.5X Tolanski objective, which provides large field of view images (1.5mm x 1mm) with optical lateral resolution (1µm) and nanometer vertical resolution.

References:

- [1] M. Jobin, A. Du and R. Foschia, *NSTI Nanotech*, **2**, 695 (2005).
- [2] M. Jobin, Ph. Passeraub and R. Foschia, *Proc.SPIE*, **6188**, 61880T (2006)
- [3] M. Jobin and R. Foschia, *Microsc. Microanal.*, **12** (Supp2), 1774CD (2006)

15:50	Poster	J5

Functionalization of MWCNTs Using Atomic Nitrogen: Effects on Electronic Structure

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The plethora of potential applications associated with carbon nanotubes (CNTs) has triggered intense research on their synthesis and characterization. Their electronic properties range from semiconducting to metallic, depending on their chiral angle and diameter. However, tailoring their electronic properties by controlling their chiral angle and/or diameter is non-trivial, moreover, bundling during synthesis appears as a technological difficulty for obtaining their dispersion in matrixes. Instead, one uses post-growth treatments such as grafting molecules or metal clusters to the tube sidewall. Hence, understanding the effect of post-treatments is essential in terms of assessing process controllability, CNT stability and eventual integration. The question to be addressed in this work is: How does the grafting of nitrogen atoms at the tube surface influence on CNT electronic structure and dispersion characteristics?

In this study, multi-wall CNTs (MWCNTs) were exposed to atomic nitrogen formed in a microwave Ar + N₂ plasma. Their electronic structure was thereafter investigated using X-ray and UV photoelectron spectroscopy (at beamlines BW2 and Flipper II at Hasylab), showing that plasma treatment induces the formation of nitrogen chemical groups at the CNT surface altering the density of electronic states. The modification of the relative intensities of photoemission near 3.5 eV (assigned to 2p-π states), and around 5.5 eV (top of 2p-σ states) suggests that the groups introduced by nitrogen-plasma promote the 2p-σ states.

Dispersion characteristics found for plasma-functionalized-MWCNTs will be discussed in terms of reduction in the π-π interaction between tubes due to the promotion of 2p-σ states.

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15:50	Poster	J6

Characterization of Al-Ni / a-Si interface by high resolution Rutherford backscattering spectrometry

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Interfaces of Al-Ni alloy and a-Si films in a thin film transistor (TFT) for liquid crystal display (LCD) were analyzed by high-resolution Rutherford backscattering spectrometry (HRBS). In conventional TFT-LCDs, a barrier metal, e.g. Cr or Mo, is used between Al-Ni alloy and a-Si films to prevent interdiffusion of Al and Si atoms. It was proposed that a combination of the Al-Ni films and the nitridation of a-Si is a promising technique for future TFT-LCDs that enables a direct contact of Al alloy and a-Si without any additional barrier metal. In order to realize a good contact between Al alloy and a-Si films, however, the interface characteristics such as elemental profiles have to be optimized. We carried out HRBS measurement using a magnetic spectrometer, which has a high depth resolution and nondestructive features, to determine the elemental depth profiles for the samples of Al-Ni (50 nm) / a-Si stacks, where the Al-Ni films were deposited by sputtering. The results revealed that the Ni contents and the oxygen contamination at the interface have a considerable influence on the contact characteristics. It is concluded that optimization of the elemental profiles at the extremely thin interface region is necessary for the future TFT-LCDs and HRBS is a powerful tool for such analyses.

15:50	Poster	J7

A study of surface oxidised Fe particles with Mössbauer spectroscopy

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A Moessbauer spectroscopy study of isotopically enriched surface oxidised iron particles is presented. Powder samples enriched with 20% ⁵⁷Fe were prepared by ball milling and subsequently thermally treated in vacuum or air. The Moessbauer spectra have been measured in transmission mode at the temperatures between 4.2 K and room temperature. Conversion electron Moessbauer spectra at room temperature were also obtained. The CEMS spectrum of the sample annealed in air at 300 °C consists of the contributions of iron, magnetite, hematite and maghemite in the relative amounts of 55%, 34%, 8% and 3%, respectively. This shows that the dominant oxide forming at the surface on heating to 300 °C at ambient atmosphere is magnetite. The spectra of the sample measured in transmission show the bulk amount of 10% magnetite, 2% hematite and 88% of metallic bcc iron. A much smaller

amount of oxides than that observed in the CEMS measurement is related to the difference of probing depths, which is of 300 nm in CEMS, whereas it is of a few micrometers in transmission an probes the bulk of the particles. This also proves that oxides are located mainly at the surfaces of the particles. The low temperature spectra of the thermally untreated and the vacuum annealed at 300 °C samples measured in transmission show the presence of oxides, mostly magnetite, in bulk amount below 1%, which corresponds to the oxide surface layer of nanometric thickness. Such a small amount of oxides could not be detected in the corresponding iron powders of natural isotopic abundance of ^{57}Fe , i.e. 2%. The Moessbauer signal from the oxide layer decreases with increasing temperature and disappears well below room temperature. The effect corresponds to a decrease of the probability for the recoilless absorption with increasing temperature and is much stronger than that in the bulk iron oxides. The observed features are analysed and a possible relation to the magnetotransport properties of the materials is discussed.

15:50	Poster	J8

$\text{SnO}_2/\text{SiO}_x$ core-shell nanoneedles

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Tin oxide (SnO_2), an important and inexpensive semiconductor with a wide band gap ($E_g=3.62$ eV, at room temperature), is well known for its potential applications in gas sensors, transparent conducting electrodes, flat display devices and solar cells. On the other hand, the formation of SiO_x sheaths, which exhibit excellent insulating characteristics on the exterior of nanowires is essential not only to avoid interference between building blocks of a complex nanoscale circuit, but also to protect nanowires from contamination. Furthermore, the SiO_x coating layer is optically transparent for the visible absorption/emission of semiconductor nanowires, resulting in limited destruction of their intrinsic optical properties. While semiconductor crystals can be obtained in the nanometer length scale in various geometries, the one-dimensional (1D) variants warrant special attention because the anisotropy in quantum confinement potentials can be used to produce unusual optical, magnetic, and electronic properties. One of the most interesting and urgent challenges is the fabrication of 1D material with a novel morphology. Preparation of the nanoneedle is important not only for scientific interests but also for future industrial applications. In the present work, we report on the fabrication of SnO_2 needle-shaped nanostructures sheathed by a sputtering technique, their structural characterization, and also their photoluminescence (PL) spectrum.

15:50	Poster	J9

Tunneling charge transfer in manganise oxides.

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Tunneling measurements of manganite ceramic samples exhibiting a CMR effect in a wide range of voltages are performed. The analysis

of the background conductance spectra was carried out and an assertion in support of the presence of nanoscale metallic regions embedded into the insulating matrix was presented. We argue that this phenomena can be caused by the phase separation in the near-barrier region. The data obtained are discussed in terms of the Glasman-Matveev model for localized states in the barrier and these localizations can stimulate the resonance mechanism for tunneling transfer of charge in systems. Effective spectral functions of the electron-boson interaction have been extracted from a fine structure in the conductance curves. It is supposed that structure between 100 and 200 meV may be attributed to the orbital ordering in manganite. Experiments with high-voltage treatments exhibit a tendency of the suppression of the electron-phonon interaction with increasing disorder at the manganite surfaces, as it follows from the theory of weak localization effects.

15:50	Poster	J10

Carrier recombination under one-photon and two-photon excitation in GaN epilayers

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III-nitride semiconductors are successfully applied for blue and UV optoelectronic devices such as light emitting and laser diodes, as well as high-voltage and high-power electronic devices. Although these devices are commercially available, there are a lot of unsolved problems related to the high density of nonradiative traps in GaN epilayers. The conventional heteroepitaxial growth of GaN films results in high dislocation densities ($10^8 - 10^{10} \text{ cm}^{-2}$) which affect both optical and electrical device properties. One of the ways to reduce threading dislocations in the film is growth of the homoepitaxial layers of large thickness by hydride vapor phase epitaxy (HVPE).

In recent decade, significant progress has been achieved in growth of GaN layers, which has an impact on dominating luminescence decay processes. For the surface excited high quality GaN layers, due to efficient diffusion created carriers rapidly escapes from the excited region, thus luminescence lifetime cannot be treated as materials quality factor anymore. Recently, we proposed application of two-photon excitation conditions for luminescence characterization of high quality GaN.

Here we present time-resolved luminescence properties of $\sim 100\text{-}\mu\text{m}$ thick GaN epilayers grown by HVPE over three different substrates excited under one-photon (1P) and two-photon (2P) conditions. The excited sample area is much bigger than carrier diffusion depth and we can neglect carrier diffusion processes. Experimental data was fitted with consideration to theoretical spatial-temporal distribution of the plasma density.

We show that the investigation of luminescence transients obtained for 1P and 2P excitations allows for the estimation of carrier lifetime, related to the density of nonradiative traps, and of the carrier bipolar diffusion coefficient. The impact of the equilibrium carrier density on the luminescence dynamics of highly excited GaN is discussed.

15:50 Poster J11

The metals chemical states in hydrated vanadium oxides

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Vanadium pentoxide based xerogels, $H_2V_{12}O_{31-y} \cdot nH_2O$, have been extensively studied over past years because of their particular properties. These materials can be used as reversible cathodes in lithium batteries, electrochromic layers for display devices, host materials for the intercalation of guest species, humidity sensors, starting material for switching elements, and so on. These xerogels have a layered structure where V_2O_5 chains are bonded by interlayer water, and can intercalate guest ions and molecules.

Partial substitution of vanadium ions by a metal of higher valence (Mo, Cr) in hydrated vanadium oxides leads to a decrease of the electronic part of conductivity. The X-ray photoelectron spectroscopy (XPS) spectra of hydrated vanadium oxides

$(VO)Mo_xV_{12-x} \cdot nH_2O$ ($x = 0, 1, 2, 3$) has been studied to provide the vanadium ions reduction ratio $RR = V^{4+}/(V^{4+}+V^{5+})$ dependence on the molybdenum ions concentration and the thermal treatment. The analysis of vanadium and molybdenum XPS spectra of the thermally untreated samples demonstrate that either V $2p_{3/2}$ and Mo $3d_{5/2}$ lines shape is actually independent on the molybdenum concentration within $0 \leq x \leq 3$ interval. It was shown, that $RR \approx 0.18$ is same that for the all tested samples, while molybdenum is in the main Mo^{6+} oxidation state. It indicates that charge transfer $V^{4+}+Mo^{6+} \leftrightarrow V^{5+}+Mo^{5+}$ between V and Mo ions do not take place. The temperature desorption mass spectroscopy analysis shows that for $(VO)V_{12} \cdot nH_2O$ xerogel intensive desorption of oxygen from xerogels begins at $T_k \approx 460K$. The significant, of about 0.13, increase of vanadium ions RR for the samples backed below and above T_k may be related with an appearance of additional O^{2-} vacancies due preferential oxygen evaporation above T_k .

It was concluded that the appearance of V^{4+} ions in V_2O_5 -based xerogels is mainly related with the stoichiometry of the samples, while higher valence admixture ions acts only as a diluting component.

15:50 Poster J12

The nature of carrier confinement in quantum dashes of various cross-section shapes

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As it has previously been demonstrated the novel quasi 0D structures called quantum dashes, which are strongly elongated dots or finite size quantum wires in shape, present a potential in a number of optoelectronic applications, in the near infrared especially, due their intrinsic properties like naturally high surface density or wide emission wavelength tunability [1-2]. However, the electronic structure of the dashes needs deeper understanding yet, as many of the optical properties are still unclear, e.g. the interpretation of the optical transitions or the importance of the largest size confinement.

We have calculated the electronic states and optical transitions exploiting the model of the two dimensional quantum confinement (wire-like approach). We have used the adiabatic approximation within the effective-mass theory to calculate the states of a quantum dash with different cross-section, like rectangular or triangular. The model takes into account the realistic dash crystallographic orientation, its shape, material composition and strain distribution. The model has been applied for the dashes in two different material systems: AlGaAs-GaAs and InGaAlAs-InP. We have calculated the confined states energies of the dashes versus their lateral size following the existing structural data and compared them to the experimentally determined optical transition energies.

Further, we have calculated the electronic structure for a pair of quantum dashes in order to probe the quantum-mechanical coupling effects. We have calculated the energies of confined states as a function of base dash width and the distance between quantum dashes.

1. R. Schwerberger, D. Gold, J. P. Reithmaier, A. Forchel, IEEE Photonics Technology Letters 14, 735 (2002).
2. A. Bilencia, R. Alizon, V. Mikhelashvili, G. Eisenstein, R. Schwerberger, D. Gold, J. P. Reithmaier, A. Forchel, Electronics Letters 39, 170 (2003).

15:50 Poster J13

New solid solution: $(Li_{1-x}Na_x)CoO_{2\pm z}$ ($0.0 < x < 0.8$, $z \pm 0.3$) system

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In this study, we prepared the $(Li_{1-x}Na_x)CoO_{2\pm z}$ ($0.0 < x < 0.8$, $z \pm 0.3$) system by solid-state reaction. This process has become reproducible. The synthesis of polycrystalline powders in the $(Li_{1-x}Na_x)CoO_{2\pm z}$ ($0.0 < x < 0.8$, $z \pm 0.3$) system with $x = 0.0$ (LC), 0.2 (LC2), 0.4 (LC4), 0.6 (LC6) and 0.8 (LC8) was designed to obtain 2 g of sample, using the following reagents Li_2CO_3 , Na_2CO_3 , and Co_3O_4 . Subsequent thermal treatment, appropriate to each compound, was sufficient to yield the pure phase.

By Differential Thermal Analysis we attribute the changes for the dehydration, and decomposition of the carbonates. In Thermogravimetric Analysis the changes of weight decrease the temperature and we point those changes with varying the structure. This last observation was carried out by X-ray diffraction powder (XRD) with temperature to corroborate that the changes of weight correspond to the changes of the unit cell. It is observed a rhombohedral structure PDF 50-0553 by XRD analysis at ambient temperature in LC and LC2 composition. In the others LC4, LC6 and LC8 compositions we observed the Co_3O_4 reagent. The particle size and morphology of the polycrystals were examined by Scanning Electron Microscopy and EDX.

15:50 Poster J14

Substructure of the metal nanomaterials after the intensive external influenceAndrzej Misiuk¹, Rostyslav V. Shalayev², Boris M. Efros², Vladimir A. Ivchenko³

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Nowadays, the problem of producing nanomaterials with 10-100 nm grain size is among the main actively developing scientific problems. It is already known that such materials possess unique physical and mechanical characteristics. In this respect, the reviewed paper aimed at the intensive external influence on sub-structure of nanometals is urgent.

The investigation objects were polycrystalline iridium, tungsten, nickel and copper metals of 99.95-99.99 % purity (with the initial grain size » 20-50 nm). The field ion microscopy which is practically the unique method providing the direct experimental observation of separate atoms constituting of sample, was taken as the basis one.

The object of investigation by field ion microscopy is various metals. In our experiments we took iridium, nickel, tungsten and copper pre-treated with intensive external influence (ion implantation and severe plastic deformation).

Some interesting results have been obtained in this work. For example, it has been revealed that in nano-iridium influenced by severe plastic deformation a nanostructure is formed (the grain size of 20-30 nm), but in the bodies of grains there are practically no defects of structure, however, after implantation a sub-nano structure, (sub-grain size of 3-5 nm) is formed, and in the bodies of sub-grains there are defects.

The sub-nano structure in the surface and near-surface volumes of iridium was for the first time found as the resulting from the implantation of argon ions at distances, which are the order of magnitude larger than the projective path of argon ions from the irradiated surface.

The obtained results are enough new, they are important for doing investigations in the fields of nanotechnology and nanostructure materials science.

15:50 Poster J15

Sm³⁺ AND Eu³⁺ in Clay Interlayer: Neutron Diffraction with Isotopic SubstitutionOleg Sobolev¹, Laurent Charlet¹, Gabriel J. Cuello², Gehin Antoin¹, Jocelyne Brendle³

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Montmorillonite is swelling clay consisting of negatively charged aluminosilicate layers held together by interlayer cations. It was selected as the most suitable buffer material for the repository for high level radioactive waste. The main function of the clay buffer material is to

retard the migration of the radio nuclides in the case of corrosion of the waste package material. The neutron diffraction experiment was carried out with using diffractometer D4 at the Institut Laue-Langevin (Grenoble, France). The aim of this experiment was to investigate by means of neutron diffraction with isotopic substitution the structural parameters of the hydration, hydrolysis and sorption of Sm and Eu, in the interlayer space of the montmorillonite and their dependence on chemical and physical factors, which are important for radioactive waste management. The experimental results are obtained for the hydrated samples of Sm-montmorillonite, prepared at low pH and pH>7 in order to find out whether Sm is present as aqueous Sm(OH)⁰₃, Sm³⁺, or intermediate hydrolyzed species at pH>7, and how it is connected to the clay surface. The measurements with hydrated samples of Eu-montmorillonite, prepared at low pH were carried out for the estimation of the feasibility of our future neutron diffraction experiments, having the aim to study the possible changes of Eu coordination in clay due to the M³⁺ → Mn²⁺, Fe²⁺ → Fe³⁺ reaction. It was found that Sm³⁺ is bind to the clay surface, most probably in the syloxane cavities. In the case of pH>7 the slightly shorter Sm – O distance is indicated in comparison to the low PH samples. This might be interpreted as an indication of the fact that at least some of Sm is present as hydrolysed species. For the Eu-montmorillonite samples we obtained curves very similar to those for Sm. This result provides the proof of the feasibility of our future experiments on Eu – Fe-doped montmorillonite systems.

15:50 Poster J16

Microscopic Description Of Layer Crystals Intercalated By 3d-metalsNatalia K. Tovstuyuk¹, Yosyp M. Stakhira¹, Bogdan A. Lukiyanets²

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Foreign atoms or molecules inserted into the layer crystal conduct themselves to a certain extent as independent system preserving almost the same features as before their insertion. Such atoms form monolayer planes in van der Waals gaps. It allows us to consider them as flat nanostructures. So intercalation as a way of nanoplanes formation is caused by i) specific structure of the crystal layer structure, ii) atoms of the most different features particularly 3d-atoms can be inserted.

Presence of the magnetic active clusters in the van der Waals gap supports the Coulomb blockade of the electric current. Besides it creates the conditions of new unique magnetic properties, which serve the base for the new technologies of the medium materials. However crystal lattice should not be considered as completely insensible to such nanostructure. It will have a certain influence on average magnetic moment of the 3d-impurity.

Microscopic description of electron subsystem of layer crystal intercalated by 3d-impurities is based on the Anderson-like Hamiltonian [1]. Within the frame of electron mixing we considered not only structural changes caused by the appearance of intercalante but Coulomb interaction of electrons with opposite spines on the same 3d-atom. Transcendent equation for average number of occupation by electron with spin up and down in site *i* is obtained. It depends on microscopic energy parameters of crystal lattice and it is determined through integral equation. As the result effects of the magnetic momentum changes depending on the system parameters are analyzed.

1. Lukiyanets B.A., Tovstuyuk N.K. Electron density states of intercalated layer crystals // Mol.Phys. Rep. V.23, p.162-166 (1999).

15:50 Poster J17

Specific aspects of total internal reflection spectroscopic ellipsometryOndřej Vlačín, Kamil Postava, Richard Dvorský, Jaromír Pištora*Technical University of Ostrava (VSB), 17. listopadu 15, Ostrava 70800, Czech Republic**e-mail: ondrej@vlasin.cz*

Modern science and advanced technology require measurement of anisotropies and inhomogeneity of various thin films and nanostructured materials with ever higher demands for precision. Classical spectroscopic ellipsometry is limited by relatively narrow gamut of incident angles. Wider range of tangential propagation constant in a material enables more accurate determination of inhomogeneity and anisotropy. Methods of the total internal reflection ellipsometry (TIRE) and the surface plasmon resonance (SPR) overcome this drawback by introducing a prism between sample and incident beam and taking advantage of specific properties of noble metals. These methods are used to analyze various nanostructured solid samples and liquids with dispersed nanoparticles. Phase modulation ellipsometer UVESEL (Horiba - Jobin Yvon) with spectral range of 0.6 to 6.5 eV has been applied. The main advantage of these methods is fast nondestructive probing with high sensitivity.

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15:50 Poster J18

Dielectric permittivity mappings obtained by GHz region Microwave Microscopy using non-contact probe and their noise reduction of Fourier transform refinementsHirofumi Kakemoto¹, Takakiyo Harigai¹, Jianyong Li¹, Song-Min Nam², Satoshi Wada³, Takaaki Tsurumi¹

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Recently, dielectric devices, such as multi - layer ceramic capacitor (MLCC) prepared by BaTiO₃ (BT) based material (X7R property) have been miniaturized and used at high frequency. The high frequency dielectric measurement technique for microscopic region is useful for the designing and preparation of these dielectric devices. In experimental, the microwave reflection (*r*) intensity measurement using scanning non - contact state probe was carried out for measuring the dielectric distribution in the ferroelectric material and device. The dielectric measurement using non-resonant and contact probe was setup with an oscillator inserted a Gunn diode, directional coupler, detector (diode), coaxial tube and probe (6μmφ). The measurement frequency was changed from 9GHz to 11GHz using an oscillator. The *r* intensity was measured at room temperature as a function of distance between sample and probe. The sample was selected for BT single crystal and MLCC (cross - section). Dielectric mapping image obtained by *r* intensity minimum value, however image was not clear because of the noise insertion slightly to the image. Fourier transform (FT) analysis was hence carried out for reduce the noise insertion to the image as a function of space frequency. A two dimensional discrete FT was expressed as $F(m_1, m_2) = (1/MN) \sum \sum f(k_1, k_2) \exp[-j2\pi(m_1 k_1 / M + m_2 k_2 / N)]$, where m_1, m_2 are discrete integers for *x* and *y* axes, and *M* and *N* are also discrete integers, and k_1, k_2 are space frequencies. From FT image, the noise was selected from raw data, and then inverse Fourier transformation (IFT) was carried out with filtering of the noise. As the result, noise reduction was carried out, and clear dielectric image was reconstructed by FT refinement.

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15:50 Poster J19

Statistics of misorientation across grain boundaries in nanostructured Fe-Ni-Mn steel prepared by cold rolling and annealing of lath martensiteSyamak Hossein Nedjad¹, Hassan Shirazi², Mahmoud Nili Ahmadabadi²

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Nanostructured Fe-Ni-Mn steel was prepared by heavy cold rolling and isothermal aging of lath martensite. Kikuchi patterns obtained by convergent-beam electron diffraction analysis was used to study the statistics of misorientation across grain boundaries. It was found out that high fraction of grain boundaries are high angle. However, low angle boundaries were also indicated frequently. The evolution of the misorientation statistics was studied during isothermal aging to outline the mechanism of ultrafine grain formation. It is found out that the statistics of misorientation is unlikely to substantially change with aging time. Following that, it is assumed random nucleation of new grains is unlikely to occur during aging and, subsequently, ultrafine grains along with misorientation statistics are inherited from deformed structure.

15:50 Poster J20

Surface fractal dimensions of sintered porous niobiumLeonid I. Skatkov*PCB "Argo", 4/23 Shaul ha-Melekh Street, Beer-Sheva 84797, Israel**e-mail: sf_lskatkov@bezeqint.net*

the fractal geometry of different natural and artificial objects was a subjects of active investigations during the last years. the present paper reports an experimental study of the surface fractal dimension of porous solid niobium obtained by vacuum sintering niobium powder. here we present observation of modified Porod law and also give stipulations of the obtained surface which are based on the employment of the independent method of Hg porosimetry. results of small-angle X-ray scattering agree very closely with those of Hg porosimetry. the surface fractal dimension are stated to be of the order of 2.8 which is a stipulation of a highly developed porous structure. our results provide experimental support to the theory developed earlier.

15:50 Poster J21

The identification and quantification of ε martensite in a FeMnSiCrNi shape memory alloy by means of EBSDKim Verbeken, Nele Van Caenegem*University of Ghent, Civil Engineering, metallurgy and materials science, Technolopypark 903, Zwijnaarde, Gent 9052, Belgium**e-mail: Nele.VanCaenegem@UGent.be*

The determination of the volume fraction of ϵ martensite is of crucial importance to study the shape memory behaviour of an iron based shape memory alloy (SMA). Several techniques have been used in literature trying to obtain a reliable measurement of this volume fraction. The results of these attempts were, however, not satisfying. The ϵ martensite appears as thin lines in the optical microscopy. In TEM, the sequence of very thin lines forms an ϵ band of only 0.4 μm thickness.

In the present work, the amount of ϵ martensite in a FeMnSiCrNi shape memory alloy was measured by means of Electron BackScatter Diffraction (EBSD). Despite the specific nature of the austenite to ϵ martensite transformation, which affected the image quality parameter, it was proven, through the high confidence index, that a correct indexing of the diffraction pattern of ϵ martensite is possible with this technique. The evolution of the ϵ martensite volume fraction could readily be followed with a reasonable accuracy, through the entire processing: before deformation, after deformation and after subsequent annealing. These results showed that EBSD has a high potential to quantify the ϵ martensite fraction in an iron based SMA.

15:50	Poster	J22

Melting of copper nanoparticles revealed by absorption spectroscopy

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We report size-dependent melting of spherical copper nanoparticles embedded into silica matrix. Based on the temperature dependence of the surface plasmon resonance energy and its width we observe two distinct melting regimes. For particles smaller than 20 nm the absorption spectrum changes monotonically with the temperature, and this allows us to assume the gradual solid-liquid phase transition (melting) of the nanoparticles or existence of superheated solid nanoparticles. In contrast, for nanoparticles larger than 20 nm, we observe a jump-like increase of the bandwidth and non-monotonic dependence of surface plasmon energy at the temperatures below the bulk melting point. This indicates that the melting of large nanoparticles is a first-order phase transition similar to the melting of bulk copper.

15:50	Poster	J23

AFM/MFM studies of low temperature nitrided layers on austenitic steel

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Low temperature nitriding enables the corrosion and wear resistant layers to be formed on austenitic steel due to presence of so-called S-phase. It was found that depending on the process parameters and nitrogen contents this phase exhibits magnetic or non-magnetic properties. The previous investigations shows that the layers with different morphology can be produced depending on the nitriding parameters as well as on the plasma parameters used for activation before gas nitriding. Especially when nitrogen was used as sputtering gas, the additional subsurface layer was produced, which has non-magnetic properties.

The main aim of the paper was to investigate nitrided layers structure with the special attention paid to application of magnetic force microscopy, which enable that specific morphology of these layers can be revealed. The investigation were performed on the layers obtained in various conditions of the treatment. Additionally to AFM/MFM studies, X-ray diffraction (XRD and GXR) together with CEMS, XPS and EBSD techniques was used to evaluate the phase composition of the layers. Elements content and distribution were determined by means of EMPA and SIMS methods. The microstructure was investigated using scanning and transmission electrons microscopy. The results confirmed complex microstructure of the layers formed during low temperature nitriding of austenitic steel, and nanocrystalline structure of subsurface non-magnetic zone. This particular characteristic of the latter is discussed in terms of increased oxygen content in subsurface area.

15:50	Poster	J24

Cleavage surfaces atomic and electronic structure of the layered In_4Se_3 crystals as natural 2D-materials

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Layered indium-selenides attract attention of researchers as natural 2D-materials [1, 2]. Among the layered chalcogenides crystals In_4Se_3 seems to be less studied, although this crystals possesses not only interlayer anisotropy properties, but also a superstructure [1] and peculiarities of the interfaces formation [2], depending on the cleavage conditions [1,2].

This work is a further step to study properties of cleavage surfaces of In_4Se_3 layered crystals by means of electron microscopy and spectroscopy: LEED, AFM, SEM, STM/STS, XPS. In this study we focus on the cleavage surfaces atomic (LEED, STM, AFM), and electronic (STS, XPS) structure characterization and peculiarities of the interfaces formation on these surfaces of pure In_4Se_3 and copper intercalated $\text{In}_4\text{Se}_3(\text{Cu})$ crystals.

Crystal bulk and cleavages surface structure (just after cleavage) studied by XRD and LEED shows fine on orthorhombic symmetry of the crystal. The cleavages do not appear to exhibit any lateral reconstruction but are not stable in UHV along time. The quasi 2D of these layered crystals is the reason of their peculiarities with respect to interface layer formation and surface nanophases educing. The formation of In-In metallic binding and metallic phase (STS) on the free cleavage surfaces of In_4Se_3 and Cu-In-Se bindings for $\text{In}_4\text{Se}_3(\text{Cu})$ intercalated crystals have been found by XPS. SEM and STM also observe phases educing. STM/STS, AFM study shows complicated surfaces nano-, micro- and electronic structure on the cleavage surfaces of In_4Se_3 crystals that have been exposed in UHV.

[1] O. Balitskii, V. Savchyn, B. Jaeckel, W. Jaegermann, Phys. E. Low-dim. syst. & nanostr. **22**, (2004) 921.

[2] P. Galiy, A. Musyanovych, Ya. Fiyala, Phys. E. Low-dim. syst. & nanostr. **35**, (2006) 88.

15:50 Poster J25

Investigation of the Virus of the Tobacco Mosaic on the Si(111) Surface by atomic-force microscopyVolodymyr L. Karbivskyy¹, Anatoly P. Shpak¹, Taras A. Korniyuk¹, Natalya A. Kurgan²

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Viruses belong to the organisms that have features of objects of the animate and inanimate nature. To have the ability to self-collecting, adhesion to various surfaces and, showing unique optical properties, viruses became interesting object of researches not only in the field of biology, but also in physics and chemistry. Recently, due to the small size of viruses (10-100 nanometers), the special attention is focused on their application as components of nano-electronic and nano-optical devices. However, at the same time the important problem is the research of atomic-molecular structure of viruses, its influences on physical properties of viruses.

The purpose of this work was the research of the structure of virus of tobacco mosaic using ultrahigh vacuum atomic-force microscopy. During the experiment the technique of preparation of virus preparation for research in high vacuum conditions ($\sim 10^{-8}$ Pa) has been developed. Pictures have been received with the use of ultrahigh vacuum atomic-force microscopy JSPM-4610 of firm "Jeol" with resolution 1 Å in a contactless mode of shooting. It is established, that in conditions of high vacuum there is a formation of the ordered structures of virus of tobacco mosaic on surface Si (111). Interaction of virus particles with the surface Si (111) is accompanied by change virion conformation (FIG 1).

The received results have shown an opportunity of application of technique ultrahigh vacuum atomic-force microscopy for research of biological objects and have opened a potential opportunity for carrying out the researches of interaction of wild and mutant types of viruses with the surfaces of various materials and influences of such interactions on properties of the given materials.

15:50 Poster J26

Comparison of current-voltage characteristics between AFM modification and ICP etching in fabrication method of a single-channel superconducting flux flow transistorSeokcheol Ko¹, Seong-Jong Kim², Byoung-Sung Han³

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In order to develop superconducting flux flow transistors (SFFT), we propose a model to describe the current-voltage characteristics of fabricated devices by considering the Biot-Savart's law. A single-channel SFTTs were successfully fabricated using an atomic force microscope (AFM) modification method and inductively coupled plasma (ICP) etching method in order to compare to the current-voltage characteristics. The measured and calculated values including flow voltage,

transresistance and current gain are discussed in relation to the vortices flow in a single-channel SFFT. The predictions agree well with measured results.

15:50 Poster J27

In-situ investigation of ultrathin polypyrrole film formation using AFMMargus Marandi¹, Jüri Tamm¹, Väino Sammelselg^{1,2}

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The properties of polypyrrole (PPy) films grown by electrodeposition on solid anode are dependent on surface characteristics of the electrode, on dopant and solvent used, and on experimental conditions such as potential and current applied. In order to understand the film formation mechanisms in more detail it is necessary to investigate in-situ the first stage of its growth, which is the main task of this work.

The electrodeposition of PPy films was carried out from aqueous solutions on Au/mica electrodes. The Au electrodes were prepared in clean vacuum by thermal evaporation on freshly cleaved mica substrates. The morphology of the substrates and film surface was studied with a multimode SPM using both contact and intermittent contact modes, and a modified liquid cell.

Atomically flat gold substrates are ideal for imaging with AFM the first stage of PPy film growth. For the method the use of flat substrates is essential to be able to distinguish the topographical features of the film and substrate.

Electrodeposition of the films was carried through in galvanostatic mode. As the film growth mechanism depends sufficiently on current density different deposition series with varied time and/or current values were done. The film topography was studied after each deposition cycle. Using relatively high current density values for the synthesis we observed the formation of a thin polymer layer over the whole electrode area already after the first cycle. Earlier investigators doing both ex-situ and in-situ AFM measurements observed similar results. As our earlier ex-situ AFM experiments showed that the film growth carried out at low synthesis current densities starts mainly on the active centers of the electrode surface, and only after applying more charge the polymer-film starts to cover the whole electrode surface, we are concentrating to the use of low current density values now. In the presentation our last results of in-situ PPy film growth studies will be presented.

15:50 Poster J28

Tip lenght influence on the shear force imaging in liquidsValentinas Snitka¹, Gediminas Seniunas¹, Kestutis Nemciauskas¹, Vida Mizariene¹, Ingrida Bruzaite²

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Shear force near-field microscopy in liquid environment loses considerable sensitivity and resolution as a result of liquid viscous damping. The Q-control technique allows to increase significantly a quality factor (Q -factor) in water, without changing its intrinsic resonance frequency

and spring constant. However, Q-control technique is quite expensive and complicated to incorporate into commercial instrumentation. In this work, we investigate the possibility to use a tuning-fork with a long (4mm) tungsten tip to operate in liquid environments. We have investigated the damping of the tip oscillation as a function of its shape and as a function of its depth under the liquid surface. The degradation of the quality factor from 1300 in air down to 400 in contact with liquid was observed with a tip with diameter of 130 microns and the length 0.4 mm and down to 980 with the tip length 1.4 mm at the conventional 32.768 kHz tuning fork first overtone resonant frequency of ~ 190 kHz. The shear force between a glass, a biological cells sample and approaching near-field probe using tuning fork detection is studied in detail. The tuning-fork and a tip oscillation amplitude-frequency dependence in air and a tip in contact with surface were investigated using Finite Element Modelling and good agreement between experiment and theory have been shown. Shear force imaging of silicon calibration gratings in liquid with a 1.2 mm long tip and a tuning-fork Q -factor of 460 have shown a high imaging sensitivity (30 nm) and stability. The tip diameter and the length variations allow to control the stiffness of the tip. To test whether the interaction forces are low enough to image soft biological material, a sample containing fixed MH22 hepatoma cells was prepared. The shear force image is comparable to an image obtained using tapping mode AFM in liquid with a silicon nitride cantilever with a spring constant of 0.10 N/m and a resonance frequency of 28 kHz at a similar scanning speed.

15:50 Poster J29

Application of Atomic Force Microscopy (AFM) in imaging of sol-gel derived mesoscopic objects and thin structured layers

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Since its discovery in 1986 Scanning Tunneling Microscopy (STM) and all related techniques developed in next years known as Surface Probe Microscopes (SPM) have been applied in many scientific and industrial domains. The spectrum of applications of SPM techniques is not only limited to the simple surface imaging (including imaging with molecular or atomic resolution) but may be also extended for determining the mechanical, electrical, magnetic, frictional and many other parameters of conductors, semiconductors, insulators and biological samples.

This study presents few examples of application of Atomic Force Microscopy (AFM) in investigation of sol-gel materials.

Sol-gel is versatile technique commonly used in materials' engineering allowing the fabrication of many metal oxides, composites, organic-inorganic hybrids and other materials in a form of powders, thin films, nanoparticles, fibers and porous materials.

Here, the application of AFM in imaging of chosen types of sol-gel derived materials is presented. As a first example silica nanotubes prepared with the use of various templates is shown. Another part of this study concerns the imaging of porous titania thin films prepared with surfactants or polymer beads which may serve as catalyst or photonic crystals. The last part is devoted to the imaging of organic-inorganic hybrids based on silica films containing organics.

15:50 Poster J30

The Study of Electrical Characteristics of Heterojunction Based on ZnO Nanowires using Ultra-High-Vacuum Conducting Atomic Force Microscopy

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Numerous studies have demonstrated novel nanodevices and applications based on ZnO nanostructures, such as nanolaser, nanogenerator, and acoustic resonator. In the field of photodetection, although a few studies have demonstrated novel photodiode based on nanostructures recently, little work has been done in electrical characteristics of heterojunction based on nanostructures.

In the present study, the electrical performances of the nano-photodiode based on a heterojunction of n -ZnO NWs with p -Si substrate with spatial resolution at the nanometer scale have been characterized using an ultra-high-vacuum conducting atomic force microscopy. Compared with the expected values of 1.0 to 2.0 reported in ZnO-based p - n junction in the previous studies, the abnormally high diode ideality factor (>2) was obtained. A theoretical model was proposed to analyze the anomalously high ideality factor of the prepared n -ZnO/ p -Si junction diode.

15:50 Poster J31

A New Tool for in-situ Electrical Measurements on Metal Nanowires within a Scanning Electron Microscope

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Nanoscale objects like metal nanoparticles or nanowires with sizes down to a few nanometers are of great interest due to their size dependent electrical properties. However, the electrical addressing of such small objects is still a great challenge. Methods available so far based on lithographically fabricated electrodes and they are accompanied with huge technical efforts. Other methods like STM or conductive AFM have high local restrictions.

Our integrated manipulating/probing system within a SEM presented here allows us to image nanoscale objects as well as to manipulate and characterise them electrically *in-situ* by using up to four metallic tips. With this nanomanipulator system we can precisely address objects with nanometre precision. Additionally, any arbitrary area of interest on the sample can be addressed which gains enormous flexibility.

Using this versatile nanomanipulating system we performed electrical transport measurements on gold nanowires. These wires were prepared on prestructured substrates formed by MBE techniques, where layers of GaAs and AlAs form a periodic heterostructure. Lamellar structures of 2.5 nm and 20 nm width were obtained by selective wet chemical etching. These lamellae were then partially covered with gold by selected angle evaporation to form metal nanowires. In this work we will show the electrical characteristics of individual nanowires recorded *in-situ* while imaging the structures in a SEM.

15:50 Poster J32

SEM and TEM investigations of misorientation in ultra-fine grained aluminum alloysSonia Boczekal¹, Maria Richert², Marzena Lech-Grega¹

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The samples of AlMg5 and AlCu4Zr0.5 alloys deformed to large strains about $\epsilon = 14$ (33 cycles) by cyclic extrusion compression (CEC) method were investigated by electron backscatter diffraction (EBSD) technique on SEM and additionally the misorientation was measured by author's software KILIN cooperates with TEM. The EBSD results concerned larger areas of sample in comparison to TEM investigations. In this range of deformations materials achieved structure consisting with the ultra-fine grains with large misorientation. Measurement the misorientation by SEM and TEM technique allowed to compare results and recognized possibility to determine the geometric and crystallographic features obtained microstructure. The micro-texture was recognized in the investigated areas of samples showing the preferred orientation.

15:50 Poster J33

SEM and EDX studies of bioactive hydroxyapatite coatings on titanium implantsGabriela Ciobanu¹, Gabriela Carja¹, Octavian Ciobanu², Ion Sandu³, Andrei Sandu⁴

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Titanium is a successful biocompatible material that is used for biomedical applications, especially for bone-anchoring systems, such as dental and orthopaedic implants. It has advantageous bulk mechanical properties such as a low modulus of elasticity and passive surface properties such as excellent corrosion resistance, low rates ion released and a high degree of biocompatibility which is largely attributed to an inert surface oxide film. Hydroxyapatite is widely used as a bioactive ceramics since it forms a chemical bonding to bone. Applications include coatings of orthopedic and dental implants and scaffolds for bone growth. This work presents an alternative coating method based on biomimetic techniques which are designed to form a crystalline hydroxyapatite layer very similar to the process corresponding to the formation of natural bone. The hydroxyapatite deposits are investigated by means of scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDX). In vitro hydroxyapatite formation studies show that the morphology and structure of hydroxyapatite layers alter in different condition of preparation.

15:50 Poster J34

Measurement of distorting magnetic field inside the scanning electron microscope with the use of microscope itselfMariusz Płuska^{1,2}, Andrzej Czerwinski¹, Jacek Ratajczak¹, Jerzy Kątki¹, Łukasz Oskwarek², Remigiusz Rak²

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The electron-microscope image distortion generated by electromagnetic interference (EMI) is an important problem for accurate imaging. Available commercial solutions to this problem utilize complex hardware for EMI detection and compensation.

However, the distortion reduction can be based on the measurement of magnetic field inside the microscope. Because the distortions are visible as deformations of registered images, digital identification of these deformations could be even applied for an automatic compensation.

A method is proposed for measurement of magnetic field inside the scanning electron microscope (SEM), using only its microscopic images. It is an easy in use and fast method that enables calculation of magnetic field with use of electron beam deflection revealed in images. Changing the scanning rate serves for separation of nonstationary distortions from stationary specimen shapes. The method is applicable for periodic and constant magnetic fields, nevertheless the field is uniform or nonuniform. In the proposed approach the field-generated electron-beam deflection is separated from image deformations generated by induction of additional currents in electronic circuits of SEM. This separation is based on measurements of image distortions at various distances between the final aperture of electron column and the specimen (i.e. working distances).

The results were validated with a magnetic-field meter placed inside the microscope chamber, although this additional measurement has many limitations avoided in the proposed method. Another validation is the identity of results obtained for different electron beam energies. The impact of the magnetic field on various parts of SEM was measured and the parts of microscope unit most sensitive to the field were determined. Comparison of the results obtained for the field generated inside and outside the microscope chamber allowed to determine the microscope shielding efficiency.

15:50 Poster J35

Intermetallic phase identification in the cast AlSi5Cu2Mg aluminium alloy in the T6 stateJolanta B. Romanowska, Małgorzata U. Wierzbńska, Grazyna E. Mrowka-Nowotnik

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The purpose of this work was to identify phase components in the aluminium – AlSi5Cu2Mg casting alloy after the standard heat treatment - T6. The alloy was subjected to differential scanning calorimetric analysis in order to study phase transformation temperatures and the processes sequence which take place during continuous heating to the melting temperature. The studied alloy was heated up to 800°C at the heating rate of 4°C/s using the SETARAM SETSYS Evolution – 1200 DSC calorimeter. Afterwards, the alloy was hold at the selected temperature in the range of solidus-liquidus region and then quenched to

the room temperature in water to freeze the microstructure. A microstructural analysis of the heat treated aluminium alloy was conducted by the means of light (LM) and scanning electron microscopy (SEM) techniques. Chemical composition of the precipitates was conducted by EDS using HITACHI-3400N scanning microscope with Thermo-Noran x-ray microanalysis system.

The results of the analysis have enabled the determination of the sequence of microstructural components formation, their morphology and evaluation of their pseudo-stoichiometry. Therefore the demonstration of intermetallic phases distribution at different stages of the process was possible, which might be used to formulate the model assumption for the microstructure and process of the alloy occurred in the different stages of phase components development, what might be used to formulate appropriate model assumptions for the microstructure and processes of shaping and for the estimation of chemical and structural composition

15:50 Poster J36

New hybrid nanostructures based on oxacillin – hydrotalcite like anionic clays loaded with nanoparticles of iron oxides: a TEM study of their textural properties

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Oxacillin sodium, a widely used beta-lactam antibiotic in the penicillin class, may cause side effects as cholestatic hepatitis, upset stomach and diarrhea [1, 2]. Hydrotalcite-like anionic clay is a biocompatible synthetic clay with pharmaceutical applications such as: excipient, drug stabilizer, component in adhesive for transdermal delivery and for the therapy of digestive disorders. We report here the synthesis of new hybrid structures based on hydrotalcite-like anionic clays incorporated with oxacillin. The porous clay matrix is modified by supporting nanoparticles of iron oxides on the clay surface. The coprecipitation method and the ion exchange method are used to obtain the drug-supported clay hybrid structure. XRD and FTIR analyses give information about the structural features of the obtained samples while TEM analyse is used to study their textural characteristics. These results can open new perspectives to reduce the toxic side effects of oxacillin, its aggregation process in aqueous solution and also to control the drug targeted delivery.

[1] A. H. Homaidhi, N. M. Abdel-Haq, M., El-Baba, B.I. Asmar South. Med. J. 95(2002) 650.

[2] L. Seoane, P. Martinez Landeira, L. Besada, J. M. Ruso, F. Sarmiento, G. Prieto, Coll. Polymer Sci. 280(7) (2002) 624.

15:50 Poster J37

Transmission Electron Microscopy Observations on Self-Assembled Si-Ge Nanorings

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Self-assembled single-crystal ultrasmall nanorings (NRs), as small as 10 nm, on Si and Si-Ge alloys have been fabricated by the mediation of Au nanodots. Si-Ge thin films were transformed into Si-Ge nanorings

(silicon nanorings doped with controlled amount of Ge) with the assistance of Au catalysts deposited directly onto the thin film with a simple process. In situ ultrahigh vacuum transmission electron microscopy (in situ UHV-TEM) at high temperature has employed to reveal the process of island formation, evaporation of Au-Si eutectic liquid droplets, and NR formation in real time.

To examine the ring-like nanostructures on the substrates, High-resolution transmission electron microscope (HRTEM) and the line profile of energy dispersive spectrometer (EDS) analysis were conducted. By controlling the size of Au nanodots, the adjustment of the diameter of NRs has been achieved. Moreover, the composition of NRs could be modulated by the deposition of various thin films, such as Si, Si_{0.8}Ge_{0.2}, and Si_{0.7}Ge_{0.3}. The process promises to be an effective nanofabrication technique to produce high density and uniform in size Si and Si-Ge NRs.

15:50 Poster J38

Mechanical and microwave absorbing properties of carbon-filled polyurethane composite

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Polyurethane (PU) matrix composites were prepared with various carbon fillers at different filler contents in order to investigate their structure, mechanical and microwave absorbing properties. As fillers, flat carbon microparticles, carbon microfibers and multi-walled carbon nanotubes (MWNT) were used. The microstructure of the composite was examined by scanning electron microscopy and transmission electron microscopy. Mechanical properties, namely universal hardness, plastic hardness, elastic modulus and creep were assessed by means of depth sensing indentation test. Mechanical properties of PU-composite filled with different fillers were investigated and composite always exhibited higher hardness, elastic modulus and creep resistance than un-filled PU. Influence of filler shape, content and dispersion was also investigated. Microwave characteristics were determined from reflexivity measurements on 3 cm waves.

15:50 Poster J39

Hydrogen induced changes in 316L stainless steel microstructure

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Transmission electron microscopy (TEM), light microscopy and X-ray diffraction investigations of the effect of cathodic hydrogen charging on microstructure of 316L stainless steel were conducted. The objective of this study was to explain in more detail the mechanism of hydrogen induced microstructure changes which affect the ductility loss of this material.

X-ray diffraction examination confirmed that cathodic H-charging of 316L steel, like in other grades of stainless steels induces partial phase transformation of austenite to several hydride phases. The solution annealed (initial state) and 50% cold worked specimens were examined by TEM before and after hydrogen charging. It was found that hydrogen charging introduces numerous dislocations and causes microtwins formation. Examination of the solution-annealed hydrogen charged specimens revealed that their microstructure exhibits similar features to that of the uncharged but 50 % cold-worked specimens. In both cases, a high dislocation density increase and microtwins formation took place. It means that cathodic hydrogen charging produces heavy plastic deformation of the matrix.

The similarity of the microstructure of the annealed and hydrogen charged material to the microstructure resulting from 50 % cold-working indicates that the amount of plastic deformation induced by hydrogen charging is of the same order

15:50 Poster J40

Characterization of the effects of hydrostatic extrusion on structure and surface properties of 303 austenitic stainless steel

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The stability of the passive film formed on austenitic 303 stainless steel in the as-received state and after severe plastic deformation by hydrostatic extrusion (HE) leading to nanostructurisation was investigated in an aggressive environment containing Cl⁻ ions.

TEM and stereological image analysis were used to examine structural changes introduced by HE. The effect of surface oxide film on the corrosion resistance of 303 stainless steel in a borate buffer + 0.1 M NaCl was investigated by potentiodynamic polarization. Surface analytical techniques such as light microscopy, SEM, AES and SAM were used to characterize the morphology, grain size, and chemistry of the surface, including local characterization of nonmetallic inclusions and their surface before and after HE. SAM analysis revealed discontinuities of the passive oxide film on MnS inclusions. TEM and STEM examinations confirm a drastic reduction of grain size, accompanied by a distinct reduction of the size of sulphide inclusions in the HE matrix. These changes in the 303 HE stainless steel are apparently responsible for its reduced pitting resistance compared to the as-received material.

This work was financially supported by the Polish Ministry of Education and Science through grant 3T08C 013 29. Surface characterizations were performed using a Microlab 350 located at the Physical Chemistry of Materials Center of the Institute of Physical Chemistry, PAS and of the Faculty of Materials Science and Engineering, WUT.

15:50 Poster J41

Electron microscopy studies of magnesium hydride

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Hydrogen driven vehicles with fuel cells offer the most environmentally friendly automotive technology. Among the known metals and alloys with potential use in hydrogen storage, magnesium is generally regarded as the most promising because of its high capacity (7.6 wt.%) and low cost. Mechanical (ball) milling has proved to be simple and very effective technique for synthesizing Mg-related materials exhibited sufficiently fast hydriding/dehydriding kinetics for technical applications.

Microstructure and morphology of Mg/MgH₂ powder (or compacts) are crucial material parameters for fast sorption kinetics. However, to our knowledge, there are only a few works in the literature about microstructure and morphology of MgH₂ powder after processing and cycling hydrogen loading. One of that reasons is the behavior of this hydride under the electron beam. MgH₂ starts to release hydrogen as soon as the electrons interact with thin sample and decomposed to Mg. The microstructural and structural investigations were performed using scanning electron microscope (FE SEM) equipped with duo-STEM detector and transmission electron microscope (TEM). This investigations were carried out on nanocrystalline MgH₂ without and with Cr₂O₃ nanopowder as a catalyst.

Morphological investigations of mechanical (ball) milled MgH₂ powders showed a MgH₂ particle size of 0.5-10 μm. The bright field STEM images of milled MgH₂ exhibited a homogeneous crystallite size distribution with average crystallite size smaller than 100 nm. The brittle nano-Cr₂O₃ particles are embedded by the MgH₂ matrix, forming a nanocomposite structure. The particle size of Cr₂O₃ was found to be 10-300 nm. TEM investigations showed the presence of small fraction of MgO phase not only in the surface of MgH₂ particles but also within the micrometric particles. These oxide regions are formed probably by aggregation of passivated MgH₂ particles during the ball milling.

15:50 Poster J42

On the structured imperfections of bulk GaSb using High Resolution Transmission Electron Microscopy

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GaSb is a promising III-V direct band gap semiconductor with sphalerite type FCC structure. Its band gap value made it an excellent candidate for the conversion of infrared radiation to electricity. Furthermore, it can be used as a substrate material due to its lattice parameter matching ternary and quaternary III-V compounds. From the device point of view, GaSb based structures have shown potentiality for numerous applications such as booster cells for improved efficiency of photovoltaic cells and high efficiency thermophotovoltaic (TPV) cells.

All the above clearly show the interest in any structural microscopic imperfections of the bulk GaSb, which affect its macroscopic usable properties.

The wafers of GaSb, that were studied, originated from ingots grown with the Liquid Encapsulated Chochralski method. In all cases, Energy Dispersive X-ray Spectroscopy and Particle Induced X-ray Emission measurements demonstrated an excess of Sb, suggesting the presence of Ga vacancy (V_{Ga}) and/or Sb antisite defects (Sb_{Ga}). The aim of the present work is the study of the micro structural imperfections using High Resolution Electron Microscopy (HRTEM) to determine the specific point defects which lead to the detected Sb excess.

The samples heating was chosen to trigger the gathering of the point defects, so that they could be determined. Based on this, small clusters of ordered material from 10 up to 25nm, formed during heating experiments in the electron microscope, were studied by means of Electron Diffraction and HRTEM. The ordered structure axes were found to have a general relation with respect to the cubic ones as follows: $a_0 = n2^{1/2}a_c$, $b_0 = 2^{1/2}a_c/2$ and $c_0 = [001]_c$. Moreover orientation variants in the two equivalent cubic [110] and $[-110]$ directions were observed. From HRTEM image analysis a structural model is suggested. This is based mainly on the ordering of Sb antisite defects (Sb_{Ga}) in two groups of sites and with different occupation values.

15:50 Poster J43

Structural characterization of plasma nitrided austenitic stainless steel

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Austenitic stainless steels are known for their excellent corrosion resistance, however their hardness and wear resistance are relatively low. These can be improved by proper surface treatment. It has been shown that the plasma nitriding of the stainless steel surface at temperatures below 475°C leads to improving its wear resistance, tribological and corrosion properties. These positive changes are due to a phase called expanded austenite or S-phase. The temperature of nitriding plays a decisive role in the formation of the nitrogen super-saturated austenite. If it exceeds 500°C the nitrided layer contains CrN precipitates which results in a decrease of the corrosion resistance. In recent years, many efforts have been made to obtain a metastable nitrogen super-saturated layer on the surface of different stainless steels by various nitriding process, but the formation, structure and properties of the S-phase are still not fully understood. In the present work, XRD and cross-sectional TEM were used to study the structure of the nitrided layer formed during nitriding the AISI 316L stainless steel at temperature of 450°C. It has been found that the applied treatment led to the formation of the 1-2 mm thick layer of the S-phase. There is no evidence of CrN precipitates. The XRD experiments showed that there occurred the austenite lattice expansion, the value of which depends of the crystallographic direction. The TEM studies confirmed that the layer consists of a single cubic phase. It contains a lot of defects such as dislocations, stacking faults, slip bands and twins. The interface between the layer and the substrate can be clearly visible, it does not create a barrier for the defects propagation. The HREM observations showed that the most prominent feature are nanotwins consisting of a few atomic planes. The outmost region of the nitride layer contained rounded areas of amorphous structure, due to high plastic deformation caused by the cathode sputtering process.

15:50 Poster J44

Analysis for Phase States of Nickel Silicides Using Plasmon Energy Mapping

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Recently, nickel silicides have been used as a material for contact of the source, drain and gates in advanced complementary metal-oxide-semiconductor (CMOS) devices. Since the phase state of nickel silicides ($NiSi_x$) influences their resistivity, it is necessary for the development of a number of modern devices to understand the two-dimensional (2D) phase state. We demonstrate 2D mapping of nickel silicides using an energy-filtered image-series technique in a low-loss region and show the results of our analysis for phase state in CMOS devices. We prepared a test pattern for CMOS devices including contacts and gate. TEM specimens for cross-sectional observation were first prepared using an FIB system (Hitachi, FB-2100). Energy-filtered images were acquired using TEM (Hitachi, HF-2000) coupled with an energy filter (Gatan, GIF-Tridium). The width of the energy-selecting slit was adjusted to 2 eV and the energy step between subsequent images was adjusted to 0.2 eV. The exposure time of each energy-filtered image was 5 s. The total energy range in which energy-filtered images were acquired was 20 eV, and 100 energy-filtered images were recorded. After the sample drift had been corrected between energy-filtered images, the EEL spectrum of each pixel was extracted and the maximum of the plasmon peak was determined using a least squares fit of Pseudo-Voigt function. The maximum energy was displayed as a 2D map. Using a contrast of a TEM image, we were unable to distinguish the different phase states of contacts and gate. However, the plasmon energy map showed the different phase states of contacts and gate, and we were able to identify that the gate corresponds to the $NiSi$ phase and the contacts correspond to the $NiSi_2$ phase. In conclusion, this technique is indispensable for detailed failure analysis of the latest semiconductor devices, because it can be used to easily obtain a 2D map of low-loss energy regions and analyze phase states of nickel silicides.

15:50 Poster J45

An investigation of Pt Island Formation on p-GaN (0001)

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We investigated Pt island formation on surfaces of p-GaN (0001) epitaxial layer treated with HCl, annealed at 550 C for 10 minutes and nitrogen-sputtered for 15 minutes. Auger electron spectroscopy (AES), synchrotron photoemission spectroscopy (PES) and low energy electron diffraction (LEED) were used to study surface cleanliness, electronic properties and surface structure of the p-GaN (0001) surfaces, respect-

ively. Using AES, Cl, C and N were found on the HCl-treated sample. After annealing at 550 °C in vacuum for 10 minutes, Cl and C disappeared but oxygen was found instead. Sputtering of nitrogen ion onto the sample surface removed oxygen peak. Nitrogen was believed to replace oxygen in the surface layers of GaN crystal and results on a shift of Ga3d peak in PES results. LEED results taken at various electron beam energies demonstrated (1x1) surfaces. The closer the Ga 3d peak to the valence band edge indicates the smaller surface barrier height. After cleaning, platinum was e-beam evaporated on the clean p-type GaN (0001). One sample was studied as deposited and the other annealed at 530 °C for 1 minute in air. Atomic Force Microscopy (AFM) results show that after annealing at 530 °C for 1 minute in air, the number of islands increases. Cross-sectional Transmission Electron Microscopy (TEM) results show island thicknesses of ~4 nanometers. Lengths of the islands were found to vary from ~5-50 nm. High resolution electron micrographs (HREMs) suggest that islands formed on the non-damaged surface region. This indicate that cleaning process of the p-GaN wafer would result in difference microstructure and, hence, electronic properties of the contact between metal and p-GaN.

15:50 Poster J46

Determination of densities of various dislocation types in GaN – comparison of TEM and improved hot acid etching method

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GaN is a wide band gap semiconductor with many optoelectronic applications. High density of threading dislocations is present in heteroepitaxially grown layers, reducing the device performance. The influence of dislocation on layer properties and especially on device electrical properties depends on the type of dislocation. It necessitates a method to determine densities of any specified dislocation type. Transmission electron microscopy (TEM) is an accurate but time-consuming technique. Chemical selective etching delineates dislocations by revealing etch pits, however a special approach have to be used to determine the densities for various dislocation types.

In this work GaN epitaxial layers grown by metal-organic vapour phase epitaxy on sapphire substrates, with total threading dislocation density of $2.4 \times 10^9 \text{ cm}^{-2} \pm 0.2 \times 10^9 \text{ cm}^{-2}$ (as determined by TEM), were subjected to wet chemical etching in hot phosphoric acid, with various etching times and temperatures.

A new statistical approach, i.e. the method based on size-distribution of revealed etch pits, was used for revealing densities of various types of dislocations. Etched samples were examined in scanning electron microscope (SEM) and afterwards the obtained images were digitally processed to determine the etch-pit size distribution. If appropriate etching conditions are applied, the distribution is multimodal. TEM results serve for an attribution of the peaks to the types of dislocations and as the values that should be achieved from the hot-acid etching method, which allows to determine optimal etching conditions. Hence, SEM (or Atomic Force Microscopy, AFM) technique is suitable for determining densities of various types of threading dislocations in GaN layers.

Thus, a simple and low time-consuming method, with an application of selective etching, SEM (or AFM) observations and size-distribution approach, is presented and calibrated by analyses based on TEM.

15:50 Poster J47

In situ micro-Raman analysis and X-ray diffraction of nickel silicide thin films on silicon

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This paper reports on the *in situ* analysis of nickel silicide (NiSi) thin films formed by thermal processing of nickel thin films deposited on silicon substrates. The *in situ* techniques employed for this study include micro-Raman spectroscopy (μ RS) and X-ray diffraction (XRD); in both cases the variations for temperatures up to 350 °C has been studied.

Nickel silicide thin films formed by vacuum annealing of nickel on silicon (at 350 °C for 30 minutes) were used as a reference for measurements. These silicide thin films have been studied using Auger electron spectroscopy depth profiles to verify the 1:1 nickel to silicon stoichiometry and glancing angle X-ray diffraction to determine the crystal orientation. Micro-Raman analysis results for these films are reported, indicating very low stress in the films, evident from a null shift of the major 214 cm^{-1} peak (compared to the spectra for NiSi powder).

In situ analysis was carried out on nickel thin films on silicon, while the samples are heated from room temperature to 350 °C. Data was gathered at regular temperature intervals (in steps of 25-50 °C) and other specific points of interest (such as 250 °C, where the reaction between nickel and silicon to form Ni_2Si is expected). The transformations from the metallic state, through the intermediate reaction states, until the desired metal-silicon reaction product is attained are discussed. The evolution of nickel silicide from the nickel film can be observed from both the μ RS and XRD *in situ* studies. Variations in the evolution of silicide from metal for different silicon substrates are discussed, and these include (100) n-type, (100) p-type, and (110) p-type silicon substrates.

15:50 Poster J48

Investigation of nickel silicide to silicon interface using transmission electron microscopy

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This paper discusses the results of cross-sectional transmission electron microscopy (TEM) based investigation of nickel silicide (NiSi) thin films grown on silicon. Nickel silicide is currently used as the CMOS technology standard for local interconnects and in electrical contacts. In addition to high resolution imaging, the analysis of these films also utilized other TEM-based techniques which consisted of energy filtered imaging, energy dispersive X-ray analysis (EDX), electron energy loss spectroscopy (EELS), selected-area electron diffraction (SAED), and hollow cone illumination (HCI).

The nickel silicide thin films were formed by vacuum annealing thin films of nickel (50 nm) deposited on (100) silicon. The cross-sectional samples indicated a final silicide (metal-silicon reaction product) thickness of about 115 nm. This investigation studied and reports on three aspects of the thermally formed thin films: the uniformity in composition of the film using EDX; the crystalline orientation of the thin films using SAED and HCI; the quality and nature of the interface using a combination of high resolution imaging, SAED, EELS, and EDX.

The analysis highlighted uniform composition in the thin films, which was also substantiated by spectroscopy techniques; desired and preferential crystalline orientation corresponding to stoichiometric NiSi, supported by glancing angle X-ray diffraction results; and an interface exhibiting the desired abrupt transition from silicide to silicon. Analysis was carried out to confirm the absence of oxygen at the interface, as the presence of oxygen affects the electrical conductivity of the films and increases the resistance posed by the silicide-silicon interface to electrical current.

15:50	Poster	J49

Characterisation of nickel silicide thin films by spectroscopy and microscopy techniques

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Nickel silicide (NiSi) is the material currently used to define local interconnects in the CMOS industry. It is also highly suitable for microsystems fabrication, exhibiting suitable mechanical properties and good resistance to bulk micromachining etchants. This paper discusses the formation and detailed materials characterisation of nickel silicide thin films. Nickel silicide thin films have been formed by thermally reacting electron beam evaporated thin films of nickel with silicon. Nickel, on formation of nickel silicide, consumes about 1.83 nm of silicon for every nm of metal, resulting in 2.54 nm of silicide. It was observed that the metal step height of 50 nm had reduced to 25 nm on (100) n-type samples after nickel silicide formation.

The nickel silicide thin films have been analysed using Auger Electron Spectroscopy (AES) depth profiles, Secondary Ion Mass Spectrometry (SIMS), and Rutherford Backscattering Spectroscopy (RBS). The AES depth profile shows a uniform NiSi film, with a composition of 49-50 % nickel and 51-50 % silicon. No oxygen contamination on the surface or at the silicide-silicon interface was observed. SIMS depth profile confirms the existence of a uniform film, with no traces of oxygen contamination. RBS results indicate a nickel silicide layer of 114 nm, with the simulated spectra in close agreement with obtained data.

Atomic Force Microscopy has been used to study the surface morphology of the nickel silicide thin films. The average grain size and average surface roughness of these films was found to be 25 nm and 0.67 nm respectively. The film surface has also been imaged using high resolution scanning electron micrographs, and studied using Kikuchi patterns obtained by electron back-scatter detection.

15:50	Poster	J50

Influence of oxygen partial pressure on the composition and orientation of strontium-doped lead zirconate titanate thin films

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The influence of oxygen partial pressure during the deposition of ferroelectric strontium-doped lead zirconate titanate thin films is reported. The thin films have been deposited by RF magnetron sputtering in an atmosphere of high purity argon and oxygen (in the ratio of 9:1); on platinum-coated silicon substrates (heated to 650 °C). The post deposition cooling rate has been identified to influence the extent of the desired perovskite orientation of the thin films; with slower cooling rates resulting in more pronounced perovskite orientation. The influence of oxygen partial pressure is studied to understand the manner in which the stoichiometry of the thin films is modified, and to understand the influence of stoichiometry on the perovskite orientation.

This paper reports on the results obtained from films deposited at oxygen partial pressures of 1, 2, 3, 4, and 5 mTorr. The thin films have been studied using a combination of X-ray photoelectron spectroscopy (XPS), glancing angle X-ray diffraction (GA-XRD), atomic force microscopy (AFM), and optical characterisation. XPS analysis highlights the marked influence of variations in oxygen pressure during sputtering, observed by variations in oxygen concentration in the thin films, and in some cases by the undesirable decrease in lead concentration in the thin films. GA-XRD is used to study the relative variations in perovskite peak intensities, and has been used to determine the deposition conditions to attain the optimal combination of stoichiometry and orientation. AFM scans show a trend of increasing surface roughness with increase in oxygen partial pressure, and also variations in grain structure and size. Films have also been optically characterized by refractive index measurements using a prism coupler. The films exhibit a high refractive index of approximately 2.3 (varying with oxygen partial pressure) at 633 nm.

15:50 Poster J51

Investigation of composition irregularities and inter-diffusion in strontium-doped lead zirconate titanate thin films on gold-coated silicon substrates

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This paper discusses the results of extensive transmission electron microscopy (TEM) based characterisation of strontium-doped lead zirconate titanate thin films. The thin films were deposited by RF magnetron sputtering at 300 °C on gold-coated silicon substrates, which used a 15 nm titanium adhesion layer between the 150 nm thick gold film and (100) silicon. The films analyzed include cross-sectional samples of films deposited for durations of 2 and 4 hours, which were about 600 nm and 1400 nm thick respectively. The TEM analysis was carried out using a combination of high resolution imaging, energy filtered imaging, energy dispersive X-ray analysis (EDX), selected-area electron diffraction (SAED), and hollow cone illumination (HCI).

At the interface of the films and gold, an amorphous zirconium-rich layer (about 4 nm thick) was observed, while the composition of the films appeared to be uniform elsewhere (verified by EDX). The films were found to be polycrystalline with a preferential columnar orientation perpendicular to the substrate; this was observed using a combination of SAED and HCI. The highly crystalline nature of the films was also evident from the accentuated roughness of the film surface, which was highlighted by the presence of faceted peaks. The films deposited for four hours exhibited a higher density of these peaks. These crystalline peaks on the film surfaces were also verified using atomic force microscopy.

Inter-diffusion between the bottom metal layers and silicon was observed; which could be due to the temperature of deposition being close to the eutectic point of gold and silicon (363 °C). The diffused regions in silicon were composed primarily of gold (analyzed by EDX), and appeared to stop at (111) silicon planes, highlighted by the triangular diffused regions observed in the 2-D TEM image. The influence of introducing 200 nm of silicon dioxide to isolate the metal layers from silicon is also discussed in this paper.

15:50 Poster J52

In situ investigation of thermally influenced phase transformations in $(\text{Pb}_{0.92}\text{Sr}_{0.08})(\text{Zr}_{0.65}\text{Ti}_{0.35})\text{O}_3$ thin films using micro-Raman spectroscopy and X-ray diffraction

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Thin films of ferroelectric strontium-doped lead zirconate titanate [PSZT, $(\text{Pb}_{0.92}\text{Sr}_{0.08})(\text{Zr}_{0.65}\text{Ti}_{0.35})\text{O}_3$] deposited by RF magnetron sputtering have been analyzed by *in situ* analysis techniques. These analyses are used to study the phase transformation from room temperature perovskite or near-perovskite to cubic orientation at higher temperatures; this is used to identify the Curie point of the thin films deposited under different sputtering conditions. The *in situ* techniques employed for this study include micro-Raman spectroscopy (μRS) and X-ray diffraction (XRD); and variations in temperatures up to 350 °C and 750 °C for the respective techniques has been studied.

The PSZT samples analyzed include samples deposited on gold and platinum coated silicon substrates (with varying adhesion layers); and samples deposited at either room temperature or higher temperatures (up to 750 °C) on these substrates. The analyses were carried out in chosen gas ambient or vacuum, based on the type of sample; with data collected at regular temperature intervals (steps of 25-50 °C). To determine the transformation temperature, and the presence of hysteresis, analysis was carried out during both temperature ramp-up and ramp-down cycles.

Significant variations in peaks for both μRS and XRD exist for samples of PSZT deposited on gold and on platinum. These peaks have been systematically analyzed and variations relative to other gathered spectra have been discussed, given the limited literature available on PSZT. The results highlight variations in the Curie point based on deposition parameters.

15:50 Poster J53

Analysis of amorphous-nanocrystalline multilayer structures by optical, photo-deflection and photocurrent spectroscopy

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Nano-crystalline silicon is a promising material for its application in thin film photovoltaic solar cells. For industrial purposes it is of interesting the deposition on large scale by plasma enhanced chemical vapour deposition (PECVD) on glass substrate, what sets upper limit on the substrate temperature. For deposition of amorphous layer, the working gas uses to be silane and plasma is produced by radio frequency excitation. For deposition of nano-crystalline layers, in order to induce formation of crystalline phase, the working gas is highly di-

luted by hydrogen, which implies strong plasma-surface interactions during growth.

In this work we examine multilayer structure consisting of 100 nanometres-thick layers of nano-crystalline silicon between two thin amorphous silicon layers. First, we measured the optical gap, Urbach slope and estimate the defect level of single layers of nanocrystalline silicon by standard transmittance and reflectance measurements, photo-deflection spectroscopy and photoconductivity. The same characterization has been done on the multilayer structures. The results indicate that the optical properties can not be simply explained in terms of the individual properties of the single layers. As first, the interference fringes disappear, proving the increase of interface roughness. Furthermore, additional absorption appears at the longer wavelength spectral range. The results are compared with structural measurements done by Raman spectroscopy, high resolution transmission electron microscopy (TEM) and grazing incidence small angle x-ray scattering (GISAXS). The observed behaviour can be explained as consequence of strong hydrogen ion bombardment which results in changes of surface structure (roughness) and structural changes in the inside of thin films. In that way it was confirmed that the nano-crystal are formed in the already grown film and not in the plasma or, at least, not all of the nano-crystals are formed before built in the film.

15:50	Poster	J54

Correlation between surface and electrical properties of ITO and ITON thin films

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Indium tin oxide (ITO) is widely used as an optical as well as an ohmic contact material for many III-V and III-N based optoelectronic devices due to its outstanding properties, like high conductivity and high transparency in a wide range of wavelength. The use of N₂ during deposition results in the incorporation of nitrogen into the material leading to indium tin oxynitride (ITON) films with improved optical and electrical performance. In this study we investigate the surface properties of polycrystalline ITO and ITON films using X-ray and ultraviolet photoelectron spectroscopy (XPS, UPS) together with electron energy loss spectroscopy (EELS) as well as Auger electron spectroscopy (AES) depth profiling measurements. Surface changes upon rapid thermal annealing (RTA) are correlated to the optical and electrical properties of the films. The deposition was performed by rf sputtering using an ITO target and Ar or N₂ as process gas. Deposition in N₂ plasma results in the formation of several types of incorporated nitrogen chemical states, which are mainly located in the first 40 nm of the surface: nitrogen bond to In and Sn, oxynitride bonds as well as unbound nitrogen are found. Comparing the core level spectra of ITO and ITON films, a direct correlation between carrier concentration and existence of an oxygen reduced phase is found. Additionally the UPS spectra of ITON films exhibit a shoulder below the valence band

maximum due to the incorporated nitrogen. Upon RTA in N₂ the surface properties of ITO films remain unchanged while the desorption of unbound nitrogen from the surface together with a strong segregation of Sn towards the surface at temperatures above 550°C is found for ITON. These processes result in a shift of the absorption edge of ITON towards lower wavelength together with a strong increase of the carrier concentration which influences the contact properties between ITON and p-GaN. The formation of ohmic ITON contacts on p-GaN is examined and discussed.

15:50	Poster	J55

Thermal Diffusivity in InN and GaN Thin Films

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GaN and InN have attracted immense importance due to their unique properties. These two kinds of materials have attracted much attention as materials for their optoelectronic applications and high power electron devices. On the other hand, the heat management in these III-nitride based semiconductor devices becomes a serious issue for the electronic industry. A proper treatment of this problem must include the effect of interfaces and thin film layers on heat flow. In this study, we have focused on the thermal properties in GaN and InN thin films on different substrates. The thermal diffusivities (α) of GaN and InN films were measured by the travelling wave technique, investigating the effect of thickness on InN films. This method is based on the well known principle of phase lag of travelling thermal wave. The thermal diffusivities (α) of GaN and InN films were measured by the traveling wave technique which is based on the well known principle of phase lag of travelling thermal wave. A thermal diffusivity of 0.83 cm²/s ($k=216$ W/mK) was obtained in GaN/Sapphire combination. The effective thermal diffusivity of InN/Sapphire, InN/GaN/Sapphire and InN/ITO/Glass were estimated to be 0.626, 0.666 and 0.467 cm²/s respectively. According to thermal diffusivity, the thermal conductivity (k) of different kinds of materials were obtained of about 122, 145 and 94 W/mK. This high α (or k) value suggests a lower degree of phonon scattering in our InN films with fewer structural defects than reported. These results showed a successful synthesis of InN and GaN thin films on various substrates, which is important for device applications.

15:50	Poster	J56

Characterization of calcium phosphate coatings doped with Mg, deposited by pulsed laser deposition technique using ArF excimer laser

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Calcium phosphate layers were deposited on Ti6Al4V substrates with TiN buffer layers by use of pulsed laser deposition method. With this technique three pressed pellets consisted of tricalcium phosphate (TCP

– $\text{Ca}_3(\text{PO}_4)_2$, hydroxyapatite (HA – $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$) and hydroxyapatite doped with magnesium ($(\text{Ca,Mg})_3(\text{PO}_4)_2$, 4% of Mg) were ablated using ArF excimer laser ($\lambda=193$ nm). The using of different targets enabled to determine the influence of target composition on the nature of deposited layers. The obtained deposits were characterized by means of Fourier Transform Infrared Spectroscopy (FTIR) and X-ray diffraction method (XRD). The obtained Fourier spectras revealed that spectral bands corresponding to presence of absorbed H_2O in obtained Mg-doped layer are weaker than in the other layers. The analysis from XRD showed that Mg-doped HA layer has crystalline structure and TCP and HA layers composition is characterized by amorphous nature.

15:50 Poster J57

Comparative study of superthin TiN_x layer by XPS and low energy SIMS

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Gradient-index (GRIN) materials have received wide attention in material sciences for considering the effect of materials graded inhomogeneity on the optical, piezoelectric and nonlinear effects as well as due to MEMS applications.

Gradient index composite structures based on TiN_x materials are interesting for photonic, optoelectronic and sensor techniques. The TiN_x layers were deposited on Si-wafers (100) by laser ablation of a Ti target with different nitrogen gas pressure up to 3×10^{-2} mbar. The layer thickness of 22 nm and composition was controlled by parameters of the excimer laser pulses (wavelength at 193 nm) and nitrogen gas pressure.

The sample was annealed using Rapid Thermal Process (RTP) at 600°C in Ar flow by 10 min. A profile of TiN refractive index and a nitrogen concentration depends on process parameters. Surface and interfacial layer-substrate energies dominate both a bulk and a strain-energy term, which encourages the formation of metastable phase with a low inner-energy. The TiN_x layers show hydrogen and oxygen high sensitivity and change of Ti/TiN ratio at as prepared - and RTP processed - sample. XPS and SIMS was applied to compositional study of TiN_x layers. Samples were analysed by AFM and spectroscopic methods.

15:50 Poster J58

Microscopy and spectroscopy techniques in characterization of thick GaN and $\text{Ga}_{1-x}\text{Mn}_x\text{N}$ layers grown by Sublimation Sandwich Method

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The synthesis and characterization of GaN and $\text{Ga}_{1-x}\text{Mn}_x\text{N}$ layers are reported. The layers were prepared by the modified Sublimation Sandwich Method (SSM). The GaN layers having a current maximum size of 200 mm thickness (10 mm x 10 mm area), and $\text{Ga}_{1-x}\text{Mn}_x\text{N}$ layers of 60 mm thickness (10mm x 10mm area) were obtained. Characterizations of the materials were performed using following methods: atomic force microscopy (AFM), Raman spectroscopy, electron microprobe, secondary ion mass spectrometry (SIMS). Electron probe measurements found the $\text{Ga}_{1-x}\text{Mn}_x\text{N}$ layers containing 4%at Mn. AFM studies showed that the GaN RMS roughness was found to increase with increasing of thickness. Raman investigations supported the conclusion on the well ordered structure of the prepared layers. SIMS measurements were accomplished in order to check the manganese concentration, as well as distribution of gallium, nitrogen and impurities - such as hydrogen, carbon, oxygen and silicon.

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15:50 Poster J59

Structural evolution of germanium-bismuth-telluride phase change materials for PRAM application with modulated RF-sputtering power on bismuth target

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RF-power modulated Ge-Bi-Te ternary chalcogenide thin film was deposited by co-sputtering Ge-Te and Bi target on SiO_2/Si substrate in order to search the appropriate candidate for alternate phase change material. A differential scanning calorimetry (DSC) analysis, x-ray diffraction (XRD) analysis, auger electron spectroscopy (AES), and high resolution transmission electron microscopy (HR-TEM) studies are conducted in order to investigate the crystallization behavior of Ge-Bi-Te ternary alloy. The XRD results of each annealed samples showed that Ge-Bi-Te alloy crystallized into GeBi_2Te_4 , $\text{Ge}_1\text{Bi}_4\text{Te}_7$, $\text{Ge}_2\text{Bi}_2\text{Te}_5$ phase around 300 °C according to Ge content and expelled Ge single phase appeared over 400 °C. Not only GeBi_2Te_4 , $\text{Ge}_1\text{Bi}_4\text{Te}_7$, $\text{Ge}_2\text{Bi}_2\text{Te}_5$ phases but Ge phase was also confirmed with HR-TEM images and diffraction patterns of various zone axes. It is noted that

some of $\text{Ge}_2\text{Bi}_2\text{Te}_5$ grains show specific faceted planes like $\{0113\}$, $\{0112\}$, and $\{0001\}$. Through the successive analysis, we revealed the Ge-Bi-Te ternary alloy varies structure according to Ge contents and confirmed potential of Ge-Bi-Te alloy for the PRAM application.

15:50 Poster J60

Investigation of mechanical properties of thin nitrided layers produced on stainless steel

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Stainless steel is widely applied in industrial practice thanks to its good corrosion resistance. Nitriding enables a hard and wear resistant layer to be formed and if the process is conducted below 500°C the layers maintain good corrosion resistance thanks to formation of “expanded austenite” or “expanded martensite”. Good corrosion resistance and biocompatibility of the layers together with their high wear resistance makes this process particularly interesting for medical purposes, both for implant and medical equipment and tools. However, these applications demand particular mechanical properties, which should minimise the chance that during exploitation hard wear particles will be formed. Moreover, in specific applications (e.g. implants) the deformation of the layers under load could not produce the sharp and brittle asperities on the surface, which could increase the wear of counterpart. The mechanical properties of the nitrided layers depend mainly on the nitrogen content, which can be controlled by process parameters. The main purpose of this work was to investigate the mechanical properties of gas nitrided layers with various nitrogen contents produced on stainless steel. The nanoindentation and scratch tests were used in combination with GDOES and EMPA techniques. The investigations were particularly focus on determination of the relationship between the mechanical properties (young modulus and hardness) and nitrogen content, which can be used for modelling the layers behaviours under load condition. The several hardness tests were performed from upper surface (continuous stiffness measurement technique) as well as on the cross-sections of the layers. The results were analysed on the base of nitrogen profiles, to overcome two main technical problems: - soft matrix influence (5-times harder layer than matrix) and - gradient nitrogen distribution in the layer. The surface deformation and topography were investigated by atomic force microscopy.

15:50 Poster J61

Investigation of the effect of surface properties and stoichiometry on haemocompatibility of titanium nitride thin films

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Introduction: Growth interest in the study of Titanium nitride thin films as coatings for biomedical applications has been generated due to their good mechanical stability, chemical inertness and enhanced biocompatibility. The objectives of this study is to evaluate the thrombogenic potential and subsequent haemocompatibility of stoichiometric (TiN) and non- stoichiometric (TiNx) films and its correlation with chemical

composition, surface, optical and electronic properties. The thin films were developed by magnetron sputtering under different deposition conditions, and human platelets response towards them was estimated by Atomic Force Microscope (AFM) observations. The surface topographic characteristics and reflection images of TiN nanocoatings were obtained with high resolution, by the implementation of Scanning Near Field Optical Microscope. In addition, Spectroscopic Ellipsometry in the energy region of 1.5-6.5 eV was used for compositional and stoichiometry characterization of TiNx films.

Results: It was found that the nitrogen partial pressure and the applied bias voltage during deposition influence the surface properties and stoichiometry of TiNx films whereas haemocompatibility was found to be related to these parameters. The platelets AFM study during one and two hours' adhesion on TiNx films, revealed the variation in their morphological characteristics indicative of different degrees of platelet activation. Thrombogenicity of the examined films was correlated with surface nanotopography and their stoichiometry. Therefore, it can be deduced that the stoichiometry and surface characteristics of TiNx films influence their haemocompatibility paving the way for the improvement of their biological properties.

15:50 Poster J62

Water absorption by epitaxial LaNiO_{3-x} thin films

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LaNiO_3 is one of conductive oxides with a crystal structure suitable for integration in epitaxial heterostructures with perovskites of enormous technological potential such as colossal magnetoresistance materials, high-temperature superconductors and ferroelectrics. The surface segregation of elements and possible hydrooxidation of LaNiO_{3-x} films were studied by means of Tunable High-Energy X-ray photoelectron spectroscopy using synchrotron radiation. Epitaxial LaNiO_{3-x} films deposited onto (100)-plane oriented NdGdO_3 substrate was obtained by using a reactive DC magnetron sputtering technique and demonstrate the excellent in-plane orientation and the surface La/Ni ratio close to the bulk stoichiometric value. The films were examined at different angles to distinguish between the chemical state of lanthanum and nickel species at the surface and slightly deeper into the material. The evident difference were observed between spectra measured at normal ($\Theta = 0^\circ$) and grazing ($\Theta = 89.3^\circ$) take-off angles, indicating that the significant alteration of the film chemical composition take place within thin (scanning depth ~ 3 nm) surface layer. Both core and satellite peaks were used for the identification of the chemical state of elements and further quantitative analysis. It was shown, that the hydroxyl-containing phase, located near the film surface may be attributed to the lanthanum and nickel hydroxide species. For the all spectral lines in question hydroxide/oxide peaks intensity ratio strongly affected by the geometry of the experiment, thus directly indicating that the thickness of the hydroxide enriched layer is of about few nanometers. The more quantitative analyses were carried out assuming the exponential and step spatial distributions of hydroxide species concentration within the bulk of the film. The thickness of about 2 nm

for hydroxide enriched layer, was estimated from the oxide and hydroxide peak intensities ratio.

15:50 Poster J63

EBS and EDX characterisation of copper films

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Microstructure of copper films is investigated by electron back scatter diffraction technique (EBSD). The automatic analysis of these EBSD pattern yields for every scan point the crystallographic orientation and phase and a value indicating the quality of the diffraction pattern. From this data the microstructure of the scanned area can be reconstructed. The resulting crystal orientation maps give a vast amount of information on the sample, including kind and distribution of different phase, size, form and defect condition of grains, kind and position of grain boundaries, local crystal orientation and misorientation distribution (texture) and other more. Aim is to prove that EBSD is a useful characterisation tool to identify copper films. Because of its films low resistivity, high resistance to electromigration and high thermal conductivity, copper is replacing aluminum as metal for multilevel interconnections. These copper precursors are believed is good stuff for interconnection metallization. Energy dispersive X-ray analysis has been used to confirm and complement the results obtained by EBSD. The phase copper and copper oxide could be identified by discriminate crystallographically dissimilar phases.

15:50 Poster J64

Pulsed positron beam for atomic and nanoscale characterization of thin films

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Positron annihilation spectroscopy is an experimental method to probe the local electron density and atomic structure at the site chosen by the electrostatic interaction of the positron. Information of the structure can be obtained from the time and energy spectra of the positron annihilation radiation. The positron lifetime is a measure of the electron density, whereas the Doppler broadening of the annihilation radiation reflects the electron momentum distribution. With these methods it is thus possible to investigate local structures embedded in the bulk of the material, such as missing atoms, clustering of atoms, superlattices, quantum dots, as well as free volumes and void sizes in polymers and biological materials. These imperfections often determine the crucial properties of new materials, such as mechanical properties, electrical conductivity, diffusivity or light emission. Hence combining positron annihilation with electrical and optical measurements leads to quantitative studies of electrical compensation, light absorption and emission in new and functional materials.

For studying thin layers, slow monoenergetic positrons are needed. These are produced with slow-positron beams, in which positrons emitted e.g. in β^+ -decay are moderated and accelerated before being implanted into the specimen. Positron beams suitable for Doppler broadening studies are technically simple and therefore very common.

However, knowledge of the moment-of-birth of the positron is lost in the moderation process. In order to perform lifetime measurements, this problem must be overcome for example by pulsing the beam.

We have constructed a pulsed positron beam for lifetime measurements in thin layers. We present the details of the system and demonstrate its performance. The positron implantation energy can be varied between 3–30 keV corresponding to a mean implantation depth of 0–2 μm . For temperature-dependence studies, the sample temperature can be controlled in the range 20–800 K.

15:50 Poster J65

The effect of magnetic field in Cu and Al thin film production of DC magnetron sputtering

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Magnetrons Sputtering are used for thin film deposition and sputter etching [1]. In these devices an electric sheath and external magnetic field are configured to trap electrons [2, 3]. In front of the cathode surface the magnetic field creates a zone "magnetic trap" where the electron loss rate is reduced and the ionization probability enhanced [4]. The ensuing ions, having Larmor radii much larger than the magnetic trap size, are accelerated by the electric field towards the cathode where they sputter particles which deposit as a film over a suitable substrate [5]. In this paper the effect of magnetic field on production of Copper and Aluminum thin film have been studied. Cu and Al films were deposited on glass substrates without heating by DC magnetron sputtering in pure Argon gas. The coil current increased from 2A to 9A. X-Ray diffraction, Spectrophotometer, SEM and Atomic Force Microscopy were used to observe the structural characterization of the films. The results show that there is an optimum magnetic field for the maximum sputtering rate. Surface properties could be affected by this experiment dramatically. Details and a model which plasma sheath plays an important role will be discussed in full paper.

References:

- [1] J. A. Thornton and A. S. Penford, Thin Film Processes, J. L. Vossen and W. Kern, Eds. New York: Academic, 1978, pp.75.
- [2] B. Chompan, Glow Discharge Processes. New York: Wiley, Interscience, 1980, pp.177-184, 265.
- [3] T. E. Sheridan, M. J. Goeckner and J. Goree, J. Vac. Sci. Tech., vol. A8, pp. 30-37, 1990.
- [4] A. S. Penfold, Handbook of Film Process Technology. IOP (1995), Sec. A. 3.2, p. 1.
- [5] G. Seriani, V. Antoni, R. Cavazzana, G. Maggioni, E. Martines, N. Pomaro, V. Rigato, M. Spolaore and L. Tramontion, 27th EPS Conference on Contr. Budapest. ECA Vol. 24 B 17020, 2000.

15:50 Poster J66

Verification of Grain Boundaries in Annealed Thin ZrO₂ Films by Electrical AFM Technique

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Zr and Hf based high-k films have been identified as promising candidates to replace SiO₂ based dielectrics for the continued down scaling of semiconductor devices. In high-k films, amorphous and crystalline phases coexist depending on the used thermal budget. Hence, different and more complex current conduction mechanisms occur at the nanometer scale and new advanced methods for both, physical and electrical characterization are required. Tunneling atomic force microscopy (TUNA) is a very promising method which allows concurrent locally resolved characterization of the electrical properties and the surface topography of the thin dielectric films. In this work, electrical and surface properties of thin high-k ZrO₂ films were studied using TUNA. The electrical analysis is performed by measuring the tunneling current through the dielectric film in a range of 50 fA to 120 pA with extremely high current sensitivity (noise level of 40 fA). ZrO₂ films in a thickness range of 2 to 10 nm were deposited by atomic layer deposition (ALD) on silicon and annealed (RTA). Their film properties were studied by TUNA mapping. For example, as-deposited 4.8 nm thick amorphous films show smooth surfaces in the topography and randomly distributed leakage current spots in the TUNA current maps. After RTA at 450 and 600°C for 30 to 300 s, all films show a crystalline morphology which is proven by TEM analysis. While the AFM topography measurements do not register the changes in film morphology, TUNA mapping visualizes the film crystallization as ring shaped structures with significantly higher current densities, which are due to grain boundaries. The sensitivity of the TUNA technique will be discussed and a comparison to conventional AFM topography measurements will be performed. Summarizing, the presented results will successfully demonstrate that TUNA is an extremely sensitive mapping tool for the detection of small changes in film morphology of high-k layers at a nanometer scale.

15:50 Poster J67

Influence of the thin Si cap layer on SiGe quantum dots morphology

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We have investigated how a deposition of thin Si layer has influenced on a structure, and composition of SiGe QDs. The samples were grown

in ultra high vacuum chemical vapour deposition process (UHVCVD) controlled with an *in-situ* reflection high energy electron diffraction (RHEED). An initial layer of germanium quantum dots were grown on Si (001) substrates at 700°C. The resulted Si substrates with SiGe QDs layer were covered by Si caps with nominal thickness between 1.4 – 20.6 nm at temperatures 550°C and 700°C.

Cross sectional TEM samples were prepared by grinding, polishing, dimpling, and ion milling, as well as by tripod polishing method. The conventional (TEM) and high-resolution (HRTEM) investigations were performed with a use 2000EX and 2010F Jeol instruments, both operating at 200 keV. The local energy dispersive X-ray (EDX) analysis was carried out with a nanoprobe produced by the field emission gun (FEG) and PGT X-ray analyser. Surface of the samples was examined by an atomic force microscopy (AFM).

We found that Si capping process influences strongly on the shape of the SiGe QDs in both 550°C and 700°C deposition temperatures. At the initial stage three types of QDs were existed: pyramid, dome, and asymmetric. The dimension of QD base, taken from 80 images, gave average value 114 nm and was the same for the dome and pyramid shape. The average height was different and gave value 20 nm for dome and 11.5 nm for pyramid respectively. After deposition of 20nm Si cap a shape transition of the QDs has take a place. The both shapes lost facets, QDs were uniformed to flat lens-like shape with height to length ratio diminished by factor of two.

Germanium distribution in SiGe QDs was examined by EDX analysis and a measuring of the local lattice distortion. Both methods gave coherent result 50-60 at% Ge in the case of not covered QDs. For the Si capping layers the Ge content in QDs diminished to around 40-50 at% Ge. It was most pronounced for thicker cap layers.

15:50 Poster J68

Optical investigation of surface morphology of the WC tool-plate subjected to nitriding preprocessing

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The performance of tribological coatings depends greatly on the adhesion strength between coating and substrate and the substrate morphology strongly affects the mechanical properties of coatings. It has been recently shown that a comparatively low-energy nitrogen ion beam (1-2 kV) with a much higher beam current density can increase the hardness of some high-speed tools. When the nitrogen ion implantation is used as a preliminary process, it can lead to increase of the surface roughness. Before covering, the tools are cleaned by glow discharge in nitrogen atmosphere (nitriding preprocessing). After cleaning, a short-time titanium bombardment is used (titanizing preprocessing). The bias voltage in both cases is of the order of 1 kV and the final coverage is made after these preliminary processes.

In this work, we have investigated the influence of the implantation with low-energy N- and Ti-ions on the surface roughness of wolfram carbide (WC) high speed tool-plate. We have also studied the surface topography of TiN layers deposited on the WC high speed tool-plate as a function of time of nitriding and titanizing preprocessing. TiN coatings have been prepared by the ARC physical vapour deposition (PVD) technique in a commercial equipment PUSK 83. We have carried out complex optical, atomic force microscopy (AFM) and scanning

electron microscopy (SEM) investigations. The optical studies include X-Y optical profilometer measurements and bidirectional reflection distribution function (BRDF) measurements, being complementary to AFM and SEM methods. The mean initial value of s_{rms} (root-mean square) roughness of WC substrates is equal to 255 nm and after 20 min of nitrogen cleaning process s_{rms} rises to about 550 nm. For short deposition time of the subsequent TiN layer, its surface roughness reflects the substrate roughness, while with longer deposition time the TiN surface becomes more and more smooth.

Keywords: TiN, nitrogen implantation, surface roughness

15:50 Poster J69

SBF studies of Bioglass Nanostructured Thin Films obtained by pulsed laser deposition or magnetron sputtering: a comparative study

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Nanostructured thin films have been obtained by pulsed laser deposition (PLD) [1,2] and magnetron sputtering (MS) methods from bioglass (BG) targets. Three types of powders with different silica content (54,57 or 61 %) were selected for these experiments. Titanium alloys, silicon and quartz were used as substrates for micronic thin films deposition. PLD films resulted partially crystallized as an effect of substrate heating at 400 deg C during deposition. MS layers showed a prevalent amorphous structure, even after post annealing at 750 deg C in air. Quite unexpectedly, MS films displayed "droplets" structured at a nano-micro level, as evidenced by SEM analyses. PLD films displayed typical nanoparticulates present either on surface or embedded inside obtained nanostructures. The stoichiometry of the targets was conserved in case of PLD films but largely modified for MS depositions (namely an increase of P/Si ratio was observed). The behavior in SBF solutions of all obtained structures was tested in dedicated chemical growth experiments. The samples were kept in SBF for 1,3,7,14, or 21 days then extracted and investigated by FTIR, SEM and XRD in comparison with pre-immersion evidence. The observed chemical evolutions could be assigned to dissolution and precipitation processes at solid liquid interface due to an ionic exchange between BG and SBF. This mechanism is quite similar to that observed in the case of SBF in-growth onto crystalline and amorphous hydroxyapatite sputtered films [3]. In view of increasing adhesion to substrates, functional graded compositional structures have been also prepared by magnetron co-sputtering, using titanium and bioglass targets. The method efficiency was demonstrated by "pull off" studies of deposited layers. References: [1] E. Gyorgy, et al., to appear in: Appl. Surf. Sci. (doi:10.1016/j.apsusc.2007.02.146); [2] D. Tanaskovic, et al., accepted at E-MRS'07, Spring Meeting; [3] Liliana Verestiuc et al, J Cryst. Growth 264 (2004) 483

15:50 Poster J70

Elaboration and characterization of barium silicate thin layers

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Historically, dielectric material used for silicon technology in the field of micro electronic is SiO₂. This is mainly due to the quality of the Si/SiO₂ interface as well as the easiness to perform and control the thickness of such films. However, the limits encountered in the miniaturization of transistors come now from the gate oxide. Combination of the low dielectric properties of SiO₂ and the requirements of thickness induces non negligible leakage current. Thus, SiO₂ is no longer adapted to the next generations of transistors and new dielectric materials are tested, such as metallic oxides (HfO₂, ZrO₂, TiO₂, Pr₂O₃, ...) and also nitrides (Si₃N₄, AlN₃, ...) and oxy-nitrides (Si-O-N, ...).

In this field, our goal is to test the capability of mixed oxide layers such as silicate ones in order to combine the advantages of the natural Si/SiO₂ interface and high dielectric constant of metal oxides. The materials we point out are barium silicates, mainly because of the good dielectric properties of BaO ($\epsilon_r(\text{BaO}) = 34$). The first step of this work plays with high reactivity of barium towards metal oxides. It consists in studying the Ba/O/Si system in order to determine the optimal conditions for barium silicate elaborations. The elaboration technique consists in the incorporation of barium in silicon oxide film through a deposition step in UHV followed by an annealing and/or an oxygen exposure.

After barium deposition, first results were obtained *in-situ* by XPS and AR-XPS. They revealed the presence of barium carbonate. After annealing, barium carbonate is decomposed and lets place to the formation of barium silicate. Further analysis were carried *ex-situ* by TEM and SIMS and they confirmed the formation of the silicate. Finally, the presence of different morphologies (depending on the presence, or not, of initial SiO₂), revealed by AFM, will also be discussed.

15:50 Poster J71

Characterization of Co-Cu thin films obtained by vapor deposition

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CoCu thin films are grown by vapor deposition method. As a substrate we use three kinds of materials: large salt monocrystal, metallic, glass and mica slides. The grain size of thin films was measured from TEM images taken with Philips CM120ST microscope. The measured data was fitted assuming a lognormal distribution and result an average size less than 10 nm. The structural properties of films were carried out by means of SAED and HRTEM. SAED pattern confirm cubic structure of nanocrystals and HRTEM images are used for evaluate uniformity of thin films and investigate defects. Fast Fourier transform was used

to measure interplanar distance and for improvement of HRTEM image by applying blob type filter and recalculate image without noise. The films are grown using a different substrate. Finally we studies influence of substrate nature in structural properties of film. In other words, we investigate morphology, crystallographic information and defects from thin films deposited to three kind of substrate: glass, metallic and salt (or amorphous, conducting and nonconducting substrate).

15:50 Poster J72

Epitaxial Growth and Band Alignment of (GdxLa1-x)2O3 Films on n-GaAs (001)

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Herein we demonstrate the epitaxial stabilization of single-crystalline (Gd_xLa_{1-x})₂O₃ films on *n*-GaAs (001) with a controlled lattice match. (Gd_xLa_{1-x})₂O₃ films have an in-plane epitaxial relationship with a 2-fold rotation on GaAs (001). Spectroscopic characterization by photoemission and absorption confirms that the band gap of (Gd_xLa_{1-x})₂O₃ film is approximately ~5.8 eV. However, the conduction band offset is increased by the unpinned Fermi level of the *n*-GaAs in the (Gd_xLa_{1-x})₂O₃ film (*x* = 0.97). The correlation of the crystalline property and the interfacial band offset by the electrical properties, as probed by capacitance and leakage current measurements, is also discussed.

15:50 Poster J73

Morphological and optical near field characterization of tin dioxide thin films deposited by spray pyrolysis method

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The morphological features and grain structure of thin films play a key role in determining the main physical and chemical properties of films. In this paper, we demonstrate that a simple method based on spray pyrolysis technique provides a powerful tool not only for integrating tin dioxide films at the termination of standard optical fibers, but also for creating diverse film structures (from the point of view of grain habits and their associations) with significant differences in morphology and near field behavior. In particular, an accurate investigation of the fabricated samples has been carried out by using a complex scanning probe system capable of simultaneous collection mode NSOM (Near Field Scanning Optical Microscopy) and normal force AFM (Atomic Force Microscopy) imaging using the same probe. We show that the gaussian profile of the near field intensity (emerging from uncoated optical fiber or from a flat SnO₂ layer) appears strongly perturbed by the presence of the SnO₂ grains with mean spatial dimensions comparable with the light wavelength (λ = 1550 nm). Moreover, when the structures spacing is greater than about 1 μ m, the structures are able to confine light leading to an effective light localization and field enhancement effect. We finally, show how by acting on the deposition parameters and by using post processing stages, such as thermal anneal-

ing, it is possible to modify the film morphology in terms of grain size, spacing and distribution, leading in turn to consequent modifications of the optical near field. This methodology opens the way to the manipulation of light through metal oxide micro and nano sized structures; in fact, by finding the appropriate deposition parameters it would be possible to obtain the desired structures onto the optical fiber core, in order to concentrate the total emerging field in precise localized spots.

Equipment and products, services and publications exhibition

Programme

Exhibitions

Alphabetical order

Exhibitor

AJA International, Inc.

Michael Hale

AJA INTERNATIONAL Inc., 809 Country Way PO.246, Scituate 02060, United States

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www.ajaint.com

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Exhibitor

Alfa Aesar GmbH & Co KG

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Exhibitor

Caburn MDC Europe Ltd

Gerhard Althoff, Heiko Hagen

Caburn-MDC Europe Ltd, Uckfield TN221QG, United Kingdom

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www.caburn.co.uk

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Exhibitor

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Andrzej Wiśniewski¹, Grzegorz Przeliorz¹, Maria Tymieniecka², Paweł Czuma²

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Exhibitor

COST Cooperation in the field of Scientific and Technical Research

Piotr Świątek, Zina Terziki, Inge De Prins

European Science Foundation, COST Office (COST), 149 avenue Louise, Brussels 1050, Belgium

e-mail: office@cost.esf.org

www.cost.esf.org

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Exhibitor

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Exhibitor

Esteco srl Headquarters

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www.esteco.com

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Lot Oriel GmbH & Co. KG

Henryk Dłużewski, Agnieszka Kowalczyk, Jurgén Schlutter

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Exhibitor

NT-MDT - Molecular Devices and Tools for Nano Technology

Ignacy Mościcki, Grzegorz Kaszyński, Dimitry Kozodaev

NT-MDT Europe BV (NT-MDT), De Pinckart 54, Nuenen 5674CC, Netherlands

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Satellite events

The expertise in nanotechnology of CSEM focuses on characterization, optics, microscopy, and surface engineering. The combination of these technologies with other ones such as microfluidics, microsystems, photonic optics and micro-optics makes CSEM able to integrate successfully nanotechnology to novel products.

Biochips and biosensors, environmental systems, instrumentation, sensing and actuating, surface engineering are applications which are addressed by the technology with impact on markets such as tool manufacturing, food safety, healthcare and pharma, telecom/datacom.

Examples and the mechanism of introducing these technologies to industry will be discussed.

Coffee break

Monday afternoon, 17 September, 15:30

Joint Poster Session (Part1)

Monday & Wednesday

Monday afternoon, 17 September, 15:50

Session AGM 1

Monday afternoon, 17 September, 15:51

15:51

Invited

Electronically tunable nanomaterials

Horst Hahn

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The properties of materials are typically controlled in a static manner by the microstructure. This implies control of the grain size, defect concentration, structure and metastability. As long as the microstructure does not change during the use of the material, the properties of the material are fixed, or irreversible. In semiconducting materials, properties can be tuned by the application of an external field due to the space charge regions which extend far from the interfaces. In metallic systems, this effect cannot be observed unless the dimensions of the structures are in the nanometer regime. The reason for this different behaviour is the small spatial dimension of the space charge regions due to the effective screening of the induced charges by the conduction electrons. In nanoporous metals and thin films exposed to appropriate electrolytes, it has been demonstrated that substantial changes of physical properties can be induced by the application of a potential between the nanostructured metal and a counter electrode. Examples of the changes of surface stresses and the electrical resistivity of thin Gold films and nanoporous Gold will be presented. The experimental results can be interpreted by the modification of the electron density distribution at the interface of the metal and the electrolyte. Furthermore, results on the tunability of magnetic properties will be summarized. The extension of the concept to organic molecules whose properties can be switched by temperature or light will be discussed.

16:20

Invited

Blue laser diodes by low temperature plasma assisted MBE

Czesław Skierbiszewski

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We present recent progress in growth of nitride based semiconductor structures made by plasma assisted MBE (PAMBE) for electronic components like laser diodes (LDs), high electron mobility transistors (HEMTs) and resonant tunneling diodes (RTDs). This technology is ammonia free and nitrogen for growth is activated by RF plasma source from nitrogen molecules. We describe new approach for growth of nitrides by PAMBE at low temperatures. The crucial for this technique is to use thin, dynamically stable, metal (In or Ga) layer on (0001) GaN surface, which significantly reduces barriers for N adatom diffusion enabling high quality 2D step-flow growth mode at low temperatures. We demonstrate importance of low threading dislocation (TDs) density substrates for high quality epitaxy in PAMBE. The growth of nitrides on low TDs density bulk GaN substrates allows to achieve step-flow epitaxy with parallel atomic steps and eliminates problems related with formation of hillocks due to the dislocation mediated growth - e.g., a high mobility twodimensional electron gas at GaN/AlGaIn interface (exceeding $110\,000\text{ cm}^2/\text{Vs}$ at 4.2 K) is achieved.

The new perspective for PAMBE in optoelectronics has been opened recently by a demonstration of blue-violet LDs [1]. We show room temperature, continuous wave (cw) operation of InGaIn multiquantum wells LDs made by PAMBE at 411 nm wavelength. The threshold current density and voltage were 4.2 kA/cm^2 and 5.3 V respectively. High cw optical power output of 60 mW is achieved. The LDs were fabricated on low TDs density bulk GaN substrates at growth temperatures $600^\circ\text{C} - 700^\circ\text{C}$.

In this work, we discuss also properties of InGaIn/GaN quantum wells, p-type conductivity limits in GaN:Mg and InGaIn:Mg layers and point out potential of PAMBE for UV and green emitters.

Acknowledgements: This work was partially supported by Polish Ministry of Science and Higher Education Grant Nr 3T11B04729

[1] C. Skierbiszewski, et. al., Appl. Phys. Lett. **88**, 221108 (2006)

Tuesday, 18 September

Special Joint Session with Symp G

Nanoscale materials: modelling and applications

Tuesday morning, 18 September, 9:00

9:00

Invited

Semiconductor spintronics

Tomasz Dietl

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Prospects in applications of semiconductor spintronics for classical and quantum information hard-ware will be presented. Materials issues will be emphasized focussing on ferromagnetic semiconductors [1]

and also on hybrid ferromagnetic metal/semiconductor systems, taking the Stern-Gerlach spin-filter as an example [2]. Various functionalities of ferromagnetic semiconductors will be listed, including electron and hole spin injection, tunnelling magnetoresistance [3], spin-reorientation transition [4] as well as current-induced induced domain-wall displacement [5] and resistance [6].

Stimulated by demonstrated and foreseen functionalities, a considerable and successful effort has been devoted to develop semiconductors and oxides containing transition metals, in which ferromagnetic features persist up to above room temperature. Recent progress in understanding of these multi-component materials [7,8] will be described emphasising an interesting unexpected difficulties in characterisation of these systems by standard experimental probes and theoretical tools.

The work supported in part by projects NANOSPIN of EC and SPIN-TRA of European Science Foundation as well as by Humboldt Foundation.

1. T. Dietl and H. Ohno, *Materials Today* 9 (11) (2006) 18, and references therein.
2. J. Wróbel, T. Dietl, A. Łusakowski, G. Grabecki, K. Fronc, R. Hey, K.H. Ploog, and H. Shtrikman, *Phys. Rev. Lett.* 93 (2004) 246601.
3. P. Sankowski, P. Kacman, J. M. Majewski, T. Dietl, *Phys. Rev. B* 75 (2007) 045306.
4. K.-Y. Wang, M. Sawicki, K.W. Edmonds, R.P. Campion, S. Maat, C.T. Foxon, B.L. Gallagher, and T. Dietl, *Phys. Rev. Lett.* 95 (2005) 217204.
5. M. Yamanouchi, D. Chiba, F. Matsukura, T. Dietl, and H. Ohno, *Phys. Rev. Lett.* 96 (2006) 096601.
6. D. Chiba, M. Yamanouchi, F. Matsukura, T. Dietl, and H. Ohno, *Phys. Rev. Lett.* 96 (2006) 096602.
7. T. Dietl, *Nature Mat.* 5 (2006) 673.
8. S. Kuroda, N. Nishizawa, K. Takita, M. Mitome, Y. Bando, K. Osuch, and T. Dietl, *Nature Mat.* 6 (June, 2007).

9:30

Invited

Computer modelling in materials and nano-science

Richard A. Catlow

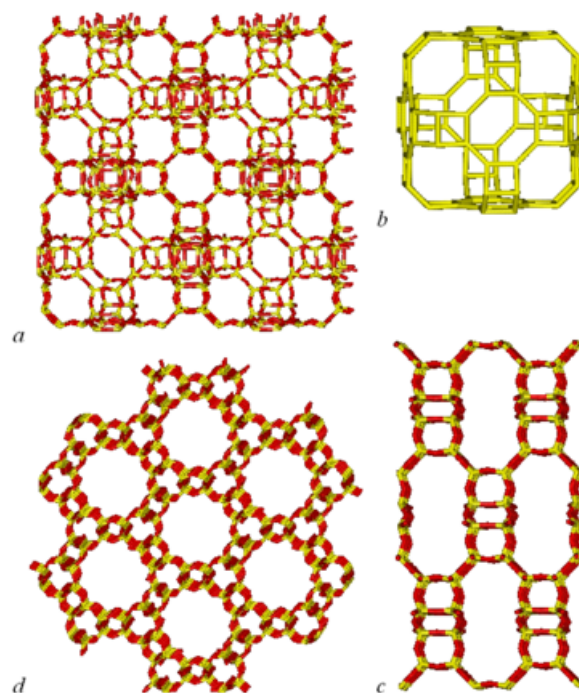
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Computer simulations techniques are an integral component of current materials science. This lecture will aim to present an overview of the current capabilities of atomistic modelling techniques in Materials science. It will focus on applications, although a brief introduction to techniques will be provided. The survey of applications will focus on:

- (1) Predicting the structures of complex materials.
- (2) Modelling surface structures and properties
- (3) Predicting nanoparticles structures and properties
- (4) Modelling reactivity

Topical examples will be presented in each case



Framework structures of some of the uninodal hypothetical zeolites derived from tiling theory: (a) structure 1_11, derived from one of the simple tilings, has a very low framework density of 11.4 T/1000q. The cages are shown more schematically in (b), illustrating the openness of the structure; (c) large-pore structure 1_71 has unidirectional 12-ring channels; (d) tetragonal structure 1_14 has elongated 12-ring channels, which run in orthogonal directions, intersecting to form a 3-D pore system.

10:00

Invited

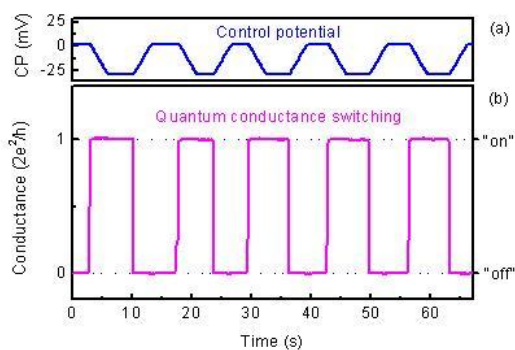
The Single-Atom Transistor

Thomas Schimmel^{1,2}, Fangqing Xie², Christian Obermair²

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An atomic-scale quantum conductance switch is demonstrated which allows to open and close an electrical circuit by the controlled and reproducible reconfiguration of individual silver atoms within an atomic-scale junction [1,2]. The only movable parts of the switch are the contacting atoms. The device is fabricated by electrochemical deposition of silver atoms and is entirely controlled by an external voltage applied to an independent third gate electrode. Controlled switching was performed between a quantized, electrically conducting “on-state” exhibiting a conductance of $G_0 = 2e^2/h (\approx 1/12.9 \text{ k}\Omega)$ or preselectable multiples of this value and an insulating “off-state”. The device, which reproducibly operates at room temperature, represents an atomic transistor or relay, opening intriguing perspectives for the emerging fields of quantum electronics and logics on the atomic scale.



- [1] F.-Q. Xie, L. Nittler, Ch. Obermair and Th. Schimmel, *Phys. Rev. Lett.* **93**(12) 128303, (2004)
- [2] F.-Q. Xie, Ch. Obermair and Th. Schimmel, *Solid State Commun.* **132**, 437 (2004)

Coffee break

Tuesday morning, 18 September, 10:30

Session AGM2

Tuesday morning, 18 September, 11:00

11:00 Invited

Magnetic Nanostructures

Andrzej Maziewski

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The formation of out-of-plane magnetization states in ultrathin films is an important problem for general knowledge and in terms of application for data storage. Discussion of the reorientation phase transitions (RPT) from out-of-plane magnetization into in-plane magnetization state is focused on metallic nanostructures. The RPT can be induced by different changes of: (i) magnetic layer thickness d ; (ii) buffer and (iii) overlayer structures [1]. Changes of such overlayer thicknesses on nanometer or even sub-nanometer level are responsible for the appearance of the RPT. Magnetization spatial distributions were studied by micromagnetic simulations and novel types of distributions were found [2,3]. Approaching the RPT magnetic domain structure in ultrathin film changes from the classical Kittel structure into sinusoidal-like one; domain structure size drastically decreases down to sub-micrometer range. By shrinking the lateral size of a nanostructure one can induce reorientation from an in-plane to an out-of-plane magnetization state. Geometrically constrained magnetization distributions will be discussed using as examples nanodisks and nanowires.

The work was supported by the project EC MC ToK NANOMAG-LAB Combined study of nanostructured magnetic materials.

1. M.Kisielewski, A.Maziewski, M.Tekielak, A.Wawro and L.T.Baczewski, *Phys. Rev.Lett.* **89**, 8, 87203 (2002).

2. M. Kisielewski, A. Maziewski, T. Polyakova, and V. Zablotskii, *Phys.Rev.B* **69** (2004) 184419.

3. Maziewski, V. Zablotskii, and M. Kisielewski, *Phys. Rev. B* **73** (2006) 134415.

11:30 Invited

Nanostructure Processing of Advanced Biomaterials

Jackie Y. Ying

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Nanostructured materials are of interest for a variety of applications. Through controlled synthesis in reverse microemulsions, my laboratory has achieved polymeric nanoparticles for the glucose-sensitive delivery of insulin. These stimuli-responsive materials allow for the appropriate insulin delivery to diabetic patients only when their blood sugar levels are high, without the need for external blood sugar monitoring. We have also developed apatite-polymer nanocomposite particles for the sustained, zero-order delivery of protein therapeutics. By adsorbing valuable bone morphogenetic proteins on carbonated apatite nanocrystals that are then encapsulated within biodegradable polymeric micro-particles, we are able to achieve controlled release of this growth factor for the bone healing process over an extended period of time. Functionalized nanoparticles and quantum dots have also been tailored for bioimaging, biolabeling and bioseparation applications.

In addition, nanostructure processing has been employed in artificial implant and tissue engineering applications. For example, nanocomposite processing has been applied to obtain orthopedic implants and bone scaffolds with superior mechanical strength and bioactivity. By combining microfabrication and nanotechnology, we have also created various microstructures in kidney-specific dimensions and shapes. These structures can be used as bioartificial renal assist microdevices, and may serve as three-dimensional templates for tissue engineering.

12:00 Invited

Synthesis of nanowires and molecular devices through amyloidogenic self-assembly of proteins and peptides

Wojciech Dzwolak

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Stacking of incorrectly-folded protein molecules has profound medical implications, as it has been associated with a number of fatal disorders such as Alzheimer disease. From the physicochemical perspective, however, the linear aggregation (fibrillation) reflects a common generic feature of proteins as polyamides. Therefore the aggregation can be induced in benign proteins, as well. I am showing how dynamics and structural variability of aggregating proteins may inspire novel strategies in engineering of molecular devices. Aggregation of insulin molecules under high hydrostatic pressure yields uniquely regular nanorings. On the other hand, high specificity of the docking interactions between incoming protein monomers and fibrils' termini (during the elongation process) may translate into the possibility of employing fibrils as selective protein receptors. As growth of protein fibrils may be triggered by seeding, and the conformational pattern of the template easily overrides environmental biases, it is now possible to obtain highly homogenous (in terms of morphology) samples of the fibrils. Several applications of aggregating insulin molecules, and sequenceless

polypeptide chains for nano-drug-delivery, and pH-controllable ‘molecular Velcro’, respectively, will be demonstrated. Such novel bionanosystems may be easily transformed into smart enzymes and/or antibodies layers for a renewable immobilization atop biosensors and biochips.

Lunch break

Tuesday afternoon, 18 September, 12:30

Session AGM3

Tuesday afternoon, 18 September, 14:00

14:00

Invited

Generation and analysis of complex organic structures

Harald Fuchs

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We have developed new approaches to self organize organic molecular systems into long range ordered ‘superstructures’ exhibiting novel physical properties as compared with the individual molecules. The methods make use of intermolecular interactions and substrate-film interactions which can be varied over a wide range. Mono- and multilayer preparation was done, for example, at the air/water interface by using the Langmuir-Blodgett technique using wetting instabilities, by self organizing by making use of intermolecular hydrogen bond formation of chiral systems, and Organic Molecular Beam Epitaxy (OMBE) under ultrahigh vacuum (UHV) conditions. The latter method allows us to generate in plane (A-B) structured films of molecular layers consisting from differing molecular systems. Some opto-electronic properties these systems will be discussed. Results from advanced scanning probes for the characterization of the organized systems by dynamic force spectroscopy, and a novel type of near field optical probe with nanometer resolution capability will be presented. Complementary to self organization techniques local probe techniques were applied to selectively modify the chemical nature of monolayers films and subsequent adsorption of other organic compounds, and metallic nanoclusters. In this way complex patterns could be locally generated for the investigation of electronic properties, for example, of nanowires.

References

W. C. Wang, D. Y. Zhong, J. Zhu, F. Kalischewski, R. F. Dou, K. Wedeking, Y. Wang, A. Heuer, H. Fuchs, G. Erker, and L. F. Chi; *Patterned Nucleation Control in Vacuum Deposition of Organic Molecules*; Phys. Rev. Lett. 98, 225504 (2007)

S. Lenhart, P. Sun, Y. Wang, H. Fuchs, C. A. Mirkin: *Massively parallel dip-pen nanolithography of heterogeneous supported phospholipid multilayer patterns*, Small 3, 71-75 (2007)

14:30

Invited

Nanomaterials for Biomedical Applications

Thomas Tsakalakos

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While general principles of nanotechnology are clear, systematic and comprehensive studies of the effects of processing and the resultant evolution of structure on the overall functional properties, performance and thermal stability, under complex loading conditions, have thus far not been performed in sufficient depth. This overview will emphasize the “on development” of useful implementations and applications of nanotechnology. One way to address this question is by using bio-inspiration – techniques, where we apply lessons learned from living systems, to design new materials with localized feedback mechanisms. This approach leads naturally to the development of multifunctional nanomaterials for use in applications through basic research of bio-inspired nanostructural materials. Nanoparticles are generating much enthusiasm in molecular biology and medicine due to their unique optical properties, facile surface chemistry, and appropriate size scale. Noble metals, especially Au, nanoparticles have great potential for cancer diagnosis and therapy due to their surface plasmon resonance enhanced light scattering and absorption. Magnetic nanoparticles show promise *in vivo* as site specific drug delivery agents. Iron oxide, magnetite (Fe₃O₄) specifically, has been the focus of most. With this behavior, it is hoped that the particles can literally be dragged with a magnet to wherever is needed. In this preliminary study, the effects of gold sodium thiomalate (Myochrysin) and magnetite (Fe₃O₄) nanoparticles on human intestinal primary epithelial cell (HIEC) growth, differentiation, and function is observed and measured. Differentiation is observed via optical microscopy imaging, proliferation is measured via a cell count, and function is measured via IL-8 cytokine analysis. It was found that Myochrysin had inhibitory effects on cell function and that magnetite caused abnormally large cell size in HT-29 cells.

15:00

Invited

Nanotechnology for Improving Implant Function

Thomas J. Webster

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Nanotechnology is being used to mimic structural components of our tissues in synthetic materials intended for various implant applications. Recent studies have highlighted that when compared to flat or micron rough surfaces, surfaces with nanofeatures promote optimal initial protein interactions necessary to mediate cell adhesion and subsequent tissue regrowth. This has been demonstrated for a wide range of implant chemistries (from ceramics to metals to polymers) and for a wide range of tissues (including bone, vascular, cartilage, bladder, and the central and peripheral nervous system). Importantly, these results have been seen at the *in vitro* and *in vivo* level. This talk will cover some of the more significant advancements in creating better bone and cartilage implants through nanotechnology efforts. It will also cover recent *in vitro* and *in vivo* studies which highlight better bone and cartilage regeneration. Advancements made in understanding possible toxic effects of nanomaterials for these tissues will also be discussed.

Coffee break

Tuesday afternoon, 18 September, 15:30

Session AGM4

Tuesday afternoon, 18 September, 15:50

15:50

Invited

Nano Au/oxide support systems in catalytic oxidationBarbara A. Grzybowska-Świerkosz, Małgorzata Ruszel*Polish Academy of Sciences, Institute of Catalysis and Surface Chemistry, Niezapominajek 8, Kraków 30-239, Poland**e-mail: ncgrzybo@cyf-kr.edu.pl*

Reports by Haruta et al. on high activity of Au nanoparticles deposited on oxide supports in low temperature CO oxidation, have stimulated in the last decade extensive studies on catalytic properties of gold dispersed on oxides. Beside CO oxidation –an important reaction in protection of environment, and purification of hydrogen for fuel cells from traces of CO- other reactions studied on supported Au catalysts, of great concern in protection of environment and chemical synthesis, include: total oxidation of hydrocarbons, reduction of NO_x, decomposition of N₂O, propene epoxidation, Water Gas Shift reactions.

The literature data on catalysis on nano-gold, will be surveyed, the special attention being paid to oxidation reactions. The methods of the catalyst preparation, ensuring nano dispersion of Au particles will be described.

The results of the authors' studies on activity of nano-Au on supports of different nature in oxidation of CO and hydrocarbons, will be discussed. The catalysts approached in the latter studies comprise nano-Au dispersed on: a) transition metal, TM oxides (including new, original supports – chromites of TM, and b) oxides of main group elements. It has been found that the nature of the support oxide and its properties (in particular the reducibility) determine the size of Au nanoparticles and the catalytic properties. The catalytic activity in low-temperature CO oxidation is higher when reducible oxides are used as the supports, particularly good performance being observed for easily reduced Au/TM chromites. Nano-Au dispersed on oxides of main group elements catalyse, on the other hand selective oxidation of propene to propanal, ethanal at temperatures higher than ~200°C.

The factors which affect catalytic performance of Au/oxide support catalysts will be considered, including:

- size of Au particles

- electronic state of Au

- role of the oxide support not only in dispersion of the Au particles but also in different steps of the reaction.

16:20

Invited

Nanoporous crystals of transition metal oxidesWuzong Zhou*University of St Andrews, School of Chemistry, St. Andrews KY16-9ST, United Kingdom**e-mail: wzhou@st-andrews.ac.uk*

Mesoporous silicas, SBA-15, KIT-6, FDU-12 and SBA-16, have been used as hard templates, allowing crystal growth of transition metal oxides, such as Cr₂O₃, Co₃O₄, WO₃, NiO, CeO₂, In₂O₃, Fe₂O₃, MnO₂, etc, inside the pores. After removing the silica templates, the final products are porous crystals of metal oxides with various morphologies depending on the pore systems in the templates. The materials have a strong application background. For example, they might be developed into self-supported nanoscale catalysts, advanced materials for fuel cells and Li-batteries. Synthesis, structural characterization and the most recent progress of these materials are reviewed. Three most popular synthesis methods, namely the surface functionalization method, the evaporation method and the solid-liquid method, are discussed. In addition to X-ray powder diffraction and nitrogen adsorp-

tion/desorption methods, electron microscopy, including transmission electron microscopic imaging, electron diffraction and energy dispersive X-ray spectroscopy techniques, is a powerful tool in studies of these new nanomaterials. Some examples of electron microscopic studies of crystal structures, defects, crystal orientations and crystal growth mechanisms with possible confinement effect of nanopores are demonstrated. Future developments of these materials are also discussed.

16:50

Invited

Nano-sized powder by high energy milling for high performing fine grain size electro ceramicsMarija Kosec, Tadej Rojac, Danjela Kuščer, Janez Holc, Barbara Malic*Jozef Stefan Institute, Jamova 39, Ljubljana, Slovenia**e-mail: tina.rucigaj@ijs.si*

The synthesis of nano-size powder of complex chemical composition by wet chemical methods often yields agglomerated particles and not enough controlled chemical homogeneity. The latter is due to different chemical reactivity of precursors in solution.

The high energy milling sometimes called mechanochemical synthesis successfully overcomes these problems.

The kinetics and mechanism of formation of perovskite powders by high energy milling are studied. Mechanochemical synthesis is modelled. For the synthesis of NaNbO₃ from Na₂CO₃ and Nb₂O₅ the critical cumulative energy is between 7-12 KJ/g (1). Mechanism of NaNbO₃ formation includes transformation of precursors to amorphous phase and subsequent formation of sodium-niobium carbonato complex and finally perovskite crystallization. The particle size of the NaNbO₃ is about 20 nm and independent of ball impact energy.

This might be quite general mechanism, as confirmed in the Pb(Mg_{1/3}Nb_{2/3})O₃ - PbTiO₃ (PMN-PT) system (2).

Using high energy milling the powder of lead free piezoelectric (K,Na,Li)(Nb,Ta)O₃ is prepared. After hot pressing the completely dense ceramics with very high piezoelectric coefficients is obtained.

Structural and functional properties of sintered ceramics of selected compositions of relaxor ferroelectrics PMN-PT, multiferroic Pb(Fe_{1/3}Nb_{2/3})O₃ (PFN) and PFN- Pb(Mg_{1/2}W_{1/2})O₃ (PFN-PMW) are also discussed. The nano-size powder combined with a suitable densification technique resulted in ceramics with large variation in grain size ranging from nano to micron dimensions. This enables a so-called size effect studies in ferroelectrics. The results demonstrate a high potential of the mechanochemical synthesis as a method yielding chemically homogeneous fine ceramic powders and consequently high performing ceramics with well controlled microstructure.

(1) T. Rojac, M. Kosec, B. Malic, J. Holc, J Eur. Ceram. Soc., 2006, **26**, 3711-3716.

(2) D. Kuscer, J. Holc, M. Kosec, J. Am. Ceram. Soc., 2007, **90**, 29-53.

17:20

Invited

Nanopores and NanowiresUlrich Goesele*Max Planck Institute of Microstructure Physics (MPI), Weinberg 2, Halle 06120, Germany**e-mail: goesele@mpi-halle.de*

Nanopores and especially ordered arrays of nanopores are of interest as building blocks for nanotechnology. Various types of material sys-

tems enabling the fabrication of ordered arrays of nanopores will be described including porous anodic alumina and arrays based on diblock copolymers. The resulting nanopores may be filled with various materials leading to nanowires or nanotubes depending on the fabrication procedure. Nanorods and nanowires, which can be used as basic elements of nanoelectronic devices, may also be generated in a number of other template-free ways which will be discussed. Potential applications will also be touched upon.

Wednesday, 19 September

Plenary Session

Wednesday morning, 19 September, 9:30

Coffee break

Wednesday morning, 19 September, 10:30

Session AGM5

Wednesday morning, 19 September, 11:00

11:00 Invited

Bulk nanostructured copper with strength of stainless steel and color of gold

Alain R. Yavari

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Copper was the first metals used by mankind and bronze (copper and tin-based) and brass (copper and zinc-based) have been in use since thousands of years ago. Like in the distant past, copper is widespread in modern civilization with applications as varied as in wires and cables, jewelry, radiators and pipings, roofing, tooth filling, screws and nails and computer boards. Modern science and engineering can fashion various materials in desired nanostructures with new properties and it is timely to design and explore nanostructured copper.

We have developed a method for creating scratch-resistant nanocrystalline layers on near eutectic alloys containing 90% copper by copper-mould casting. Some of our alloys have yield strengths in excess of 1.8 GPa making them mechanically more resistant than many stainless steels but with a range of colors. Depending on elemental additions, the colors can vary from copper-like to gold-like.

11:30 Invited

Recent Advances in Making Nanometals by Heavy Plastic Deformation

Malgorzata Lewandowska

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Nanometals defined as metals or alloys having grain size reduced to less than 100 nm have attracted a considerable research interest due to their advanced properties such as very high strength combined with good ductility, increased high cycle fatigue life, enhanced charging capacity and diffusion rate of hydrogen, improved resistance to corrosion and wear. They can be produced following the bottom-up methods,

e.g. consolidation of nanopowders, electrodeposition, nanocrystallization from amorphous phase. However, it seems that better technological perspectives are related to top-down approach which consists in the transformation of metals or alloys having a conventional grain size into bulk materials possessing submicron- or nanoscaled structure by using the methods of severe plastic deformation (SPD). To date, a significant development in SPD techniques has been achieved and such methods as ECAP, HPT, ARB and CEC have been successfully applied to grain refinement in various materials.

The major challenge for nanometals is their commercialization. To achieve this goal a significant effort must be undertaken. The main restriction which limits the industrial application of nanometals is due to a small volume of obtained products. Therefore, there is a strong interest in development of new processing techniques. Recent results have shown that more conventional techniques such as rolling in liquid nitrogen or hydrostatic extrusion are also efficient in grain refinement and allow to obtain substantially greater volumes of products. Nanometals are known as high strength materials. However, the detailed description of other mechanical and physico-chemical properties and their stability during exploitation is essential in order to introduce products made of nanometals into the market. Nanometals are unknown for industrial community. Some examples of potential application of nanometals will be shown.

12:00 Invited

Nanoscale Materials For Hydrogen Storage

Maximilian Fichtner, Christoph Frommen, Aline Leon, Wiebke Lohstroh, Horst W. Hahn

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The technical challenges in the field of hydrogen storage will be outlined and principles of hydrogen storage in general and of hydrogen storage in solids will be explained. The state-of-the art of selected physisorption and chemisorption materials will be presented and advantages and constraints will be mentioned of the different types of materials which are currently under investigation. Examples will be shown where the particular microstructure of hydride-based nanocomposites plays a major role in the transformation behaviour of the materials and a model description is presented which was developed based on detailed knowledge of the processes on the nanoscale.

Future trends in the development of hydride materials are outlined and it will be shown that nanotechnological approaches can open new fields in this area and play a major role in the development of viable hydrogen storage materials.

Lunch break

Wednesday afternoon, 19 September, 12:30

Session AGM6

Wednesday afternoon, 19 September, 14:00

14:00 Invited

Fundamentals of Thermal Stability of Granular NanosystemsLazar S. Shvindlerman^{1,2}, Günter Gottstein²

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The main goal of the presentation is to treat the fundamentals of thermal stability of grain microstructure in nanosystems. The consideration is based principally on the experimental, theoretical and computer simulation research which are carried out at the Institute of Physical Metallurgy and Metal Physics, RWTH Aachen University and the Institute of Solid State Physics, Russian Academy of Sciences (Chernogolovka). The contribution of different mobile structural elements and defects of nanocrystalline materials - foreign atoms, vacancies, grain boundary junctions (triple junctions and quadruple points), immobile and mobile particles - to grain growth dragging is quantitatively examined. The results of the first direct measurements of grain boundary excess free volume and the influence of this grain boundary parameter on the stability of nanocrystals are presented and discussed. It is shown that grain growth controlled by grain boundary junctions (triple junctions and quadruple points) kinetics can be used to stabilize a microstructure which will be stable during subsequent annealing at grain boundary kinetics. The derived hierarchy of drag efficiency constitutes an effective engineering tool to assess and compare the role of chemistry and crystals defects on the microstructural stability of nanocrystalline and ultrafine grained materials.

14:30 Invited

The Onset of Plasticity in Small Single- and PolycrystalsEugen Rabkin¹, David J. Srolovitz²

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We review recent experimental data that indicate an exceptional strength of small metallic objects (nanopillars, nanowires and nanoparticles), exceeding the strength of their bulk counterparts by orders of magnitude. To understand the origins of this high strength, we performed a series of molecular dynamics simulations of the uniaxial compression of single- and bicrystalline gold nanopillars. Simulations were performed for different nanopillars crystallographic orientations, nanopillar cross-sections, misorientation parameters of grain boundaries, and over a range of temperatures using several different interatomic potentials. For some nanopillar orientations, the initially perfect samples exhibit buckling or shear instabilities that can be interpreted in terms of the fcc-hcp phase transition at high pressure and the stress dependence of the shear modulus. For the others, the yield stress was observed to be either a linear or parabolic function of temperature, depending on the choice of interatomic potential, nanopillar cross-section and/or nanopillar size. For example, the temperature dependence of the yield stress of the <111> oriented nanopillars was linear for those with hexagonal and square cross-sections, but parabolic for those with circular cross-sections. We suggest a simple yield nucleation criterion in which the nucleation of the first Shockley partial at the surface of a nanopillar occurs at a critical strain at the surface, which includes contributions from thermal vibration, elastic loading and thermal ex-

pansion. We demonstrate that the yield condition correctly describes the temperature dependence of the yield stress observed in the full set of computer simulations.

15:00 Invited

Stability, instability, metastability and grain size in nanocrystalline ceramic oxide systemsGiora Kimmel, Jacob Zabicky

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The following nanocrystalline binary oxide systems were studied: Mg-Ti, Zn-Ti, Fe-Al, Fe-Ti, Ni-Ti, Mg-Ti, Zr-Al, Zr-Mg and Y-Ln (Ln = rare earth element). Some pure or doped unary oxides were also studied. The xerogels were heated at constant T (200 to 1600 °C) for 3 to 6 hours. There was a threshold T for oxide formation and in many cases the products were metastable nanocrystalline phases, depending on grain size and composition, including doping. The oxide phases of Ni-Ti, Mg-Ti, Zr-Al, and Ln-Y formed at T < 900 °C are different from those formed at high T. New ranges of solid solution and formation of higher temperature structures were found. A transition phase can be defined as a structure formed at relative low T, irreversibly transforming higher T into an equilibrium phase of the same elemental composition. Some low T transition phases have a structure similar to that of a high T equilibrium phase, e.g., (equilibrium phase given in parentheses) tetragonal ZrO₂ (monoclinic) and low-T qandilite-like solid solutions (qandilite + geikielite). Others are unique with no representation in the equilibrium phase diagram, e.g., gamma-like aluminas (corundum) and anatase (rutile), which are formed as nanocrystalline oxides due to small growth rate caused either by low-T of calcination or due to additives. To assess the importance of crystal size in the stabilization of transition phases, we conducted the following studies: (a) XRPD analysis of all unary, doped and binary compositions; (b) evolution of transition phases in HT XRPD of the Mg titanates; (c) at temperatures where mixtures of transition and equilibrium phases were found, phase evolution was studied with time; (d) the retention of pure tetragonal ZrO₂ on quenching Al-Zr oxides after calcinations at high T; (e) additional evidence from HRTEM, SEM and DTA experiments is also being collected. A model correlating the size effect with the unusual phases and structures is proposed.

Coffee break

Wednesday afternoon, 19 September, 15:30

Joint Poster Session (Part II)

Wednesday afternoon, 19 September, 15:50

Thursday, 20 September**Session AGM7**

Thursday morning, 20 September, 9:00

9:00 Invited

Nanofluid – A New Concept for Advanced Heat Transfer ApplicationsIndranil Manna

e-mail: imanna@metal.iitkgp.ernet.in

11:00

Invited

Nano-Oxides and Hydroxides With Low Dimension Structures for Application in Optoelectronic and Biomedical Engineering

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In this paper we present and review the effective route for synthesize the oxides and hydroxides of nano-particles, -colloid, -rod, -tube from Y, Eu, Tb, Gd and Ti, Fe. The hydroxides and oxides of the above elements have been synthesized by using a polymer as soft template or derived from sol gel process via complexation.

The nanoparticles of Y_2O_3 , Eu_2O_3 , Tb_4O_7 and Gd_2O_3 have been prepared by using diethylene glycol. Nanoparticles of TiO_2 derived from sol gel process via complexation. The nanocolloid of Fe_3O_4 obtained by co precipitation of Fe(II) and Fe(III) in starch or methylpolyethylenglycol.

The (Y,Tb) hydroxides nanorod, and tube have been prepared by using polyethylene glycol with the different molecular weights. The (Y,Tb) oxides nanotubes and rods can be obtained by calcination of the hydroxides at 800°C. TiO_2 nanorod and tubes have been obtained from complexation synthesis with tri(propylene glycol)diacrylate by using the sol gel process.

The methods of XRD, FESEM, TEM, EDS, and thermogravimetry have been employed to characterize these nanomaterials. The growth mechanism of the above nanostructures could be curiously affected by a solution-solid process, anisotropic nanocrystal structures and incorporated with complexation agent or polymer conformation. Especially the transformation from nanorod to tube in reaction process has been observed well in the case of $Y(OH)_3$ and TiO_2 .

The BET surface area data for $Y(OH)_3$ nanotube, 139,26 m²/g, for TiO_2 nanotube, 244,69 m²/g are much larger than that of nanoparticle or -rod, indicating that their hollow and tubular characteristics.

The magnetization and colloidal properties of (Fe,Gd) nanosuspensions were characterized to biocompatible materials for contrast enhancements in medical MRI.

The optical, spectroscopic, and emission of some nanophores: $Y(OH)_3$: Eu, Tb; Y_2O_3 : Eu, Tb; Eu_2O_3 ; Tb_4O_7 as particles, colloid, rod and tube have been investigated for display, waveguide devices and active materials in coding card.

11:30

Oral

Local magnetic and electronic properties of surface oxidised Fe nanoparticles

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A combined XRD, Moessbauer, STXM and NMR study of surface oxidised, ball milled Fe nano-powders exhibiting magnetoresistive properties is presented. The XRD patterns show the peaks of bcc-Fe phase with linewidths increasing with increasing milling time. This is attributed to a decrease of the effective size of crystallites and increased strain due to accumulation of defects. Thermal treatment causes a reduction of the line-widths to their initial values from before milling. A similar effect is observed for the line-widths in the Moessbauer spectra, whereas other parameters, i.e. hyperfine field, quadrupole splitting and isomer shift remain unchanged. Scanning Transmission X-ray Microscopy (STXM) measurements provided the oxygen maps of the particles and, hence, the information on the distribution of oxides on particles surface. The O_{1s} and Fe $L_{2,3}$ near-edge absorption spectra (XANES) of selected areas of particle surface show differences between oxides in samples prepared under different conditions. The ^{57}Fe spin echo NMR spectra consist of a dominant resonant line corresponding to bcc-Fe core and a much weaker resonance corresponding to the iron oxides in the surface layer of nanometric thickness. The resonant frequency of the main line of the oxide layer is slightly lower than that of the bulk magnetite. The effect can be attributed to the exchange coupling between the oxide layer and the bcc-Fe core of the particle and/or to the presence of strain in the oxide layer. A lack of the signals from oxides in the Moessbauer spectra can be attributed to a small thickness of the oxide layers and the related low probability for the recoilless absorption. The studies constitute a detailed investigation of the nature of the iron oxide surface layer and its interaction with the iron core in ball milled iron nanoparticles.

11:45

Oral

High pressure-low temperature synthesis of new nanostructured compounds

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Mild thermodynamic conditions (in terms of pressure and temperature parameters) specific to hydrothermal or solvothermal reactions are able to initiate through chemical bonding directly from solutions new

developments in inorganic and molecular chemistry. The enhanced reactivity at the solution/nuclei or inorganic/organic interfaces is one way able to improve chemical bonding between different species. Such chemical bonds can be used to produce in soft conditions nanostructured oxide compounds or hybrid inorganic/organic molecules. More over, the addition of external electric field is able to produce oriented grain growth deposition of thin films starting from these nanocompounds.

The paper presents the opportunities offered by the methods developed by our research group in obtaining different new materials. Examples presented are doped nanocrystalline zinc oxide – maleic acid copolymers with controlled nanostructure and electronic properties, hydroxyl apatite – polysaccharides and layered silicates-polymer compounds - calcium phosphate-polymer hybrids for tissue engineering and regenerative medicine.

Acknowledgment: Romanian Agency for Scientific Research for financial support in the frame of Research for Excellence contracts 46-ReteBdent, 69-SINAPS and 16-TECOREMED and COST D30 – High pressure Tuning of Materials.

12:00 Oral

Diamine biomolecules surface functionalization single-walled carbon nanotube as gene-delivery vector

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The development of new and stable gene carrier is of great importance to improve the transfection efficiency and achieve to certain fundamental needs in gene therapy such as therapeutic protein formation, immunogenicity enhancement and apoptosis induction. The stability and applicability of the system which contains single-walled carbon nanotube (SWCNT) to combine with certain sequence of plasmid DNA(EGFP-C1) were examine by computer simulation. Raman spectroscopy was used as supporting information to verify the process and phenomenon of complex formation. Two diamine molecules, 1,4-diaminobutane and polyoxyethylene bis-amine, were applied in order to load positive charges onto the surface of SWCNT under physiology condition and also enhance the binding ability of CNT to EGFP-C1. The formation of peptide bond was examined with FTIR to confirm the success crosslinking. The amount of surface functional groups was checked out with TGA, which found that they are at an extremely low concentration but show great influence on the physical property. Biocompatibility was evaluated with LDH, MTT and total DNA. The results show a significant increase in cell viability and number for SWCNT-1,4-diaminobutane complex. IL-6 release was measure to justify the inflammatory inducing and give the hint of translocation mechanism. The binding efficiency of functionalized SWCNT to EGFP-C1 was analyzed through the binding strength under electrophoresis. In-vitro transfection was also done to justify the transfection efficiency but showing no significant increase compare with other non-viral vectors which may due to the binding force of such complex.

Keywords: *gene carrier, single-walled carbon nanotube, 1,4-diaminobutane, polyoxyethylene bis -amine, biocompatibility*

12:15 Oral

Growth of gold tips onto hyperbranched CdTe nanostructures

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The creation of hybrid metal-semiconductor nanostructures is an important next step towards active devices from inorganic nanocrystals. There is also considerable interplay between the optical and electronic behavior of the components of such hybrid materials, potentially leading to enhanced or new properties. The gold tipped hyperbranched particles provide not only an significant progress in the level of control over inorganic hybrid nano-materials but may allow creating blends with organic molecules for novel electronic and optical materials.

We have prepared a novel type of hybrid metal-semiconductor Au-CdTe nanostructures with very complex shapes, enormously large surface to volume ratio and well defined evenly sized gold anchor points at the tips. It may give rise to a new class of multifunctional materials, in particular in combination with organic molecules, with possible application for example in energy conversion devices, bio-functionalized materials, and infrared emitting/detecting devices.

Lunch break

Thursday afternoon, 20 September, 12:30

Session AGM9

Thursday afternoon, 20 September, 14:00

14:00 Oral

High performance nano fuel cell based on 1D nano proton transport channels

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Numerous investigations have demonstrated novel nanodevices and applications based on nanomaterials. But such devices can not work without external power supply, thus nano power source is urgently demanded accompanying with the increasing study on nanodevices. Here we present a method to fabricate Nafion®-115 nanowires as 1D nano channels for proton transportation. Furthermore, a high performance nano fuel cell has been accomplished on the nanoscale based on the nano proton transportation channel.

An extrusion method, using anodic aluminium oxide (AAO) membranes as templates, has been successfully developed to fabricate Nafion nanowire arrays. A surface modification is carried out to improve the filling rate in the synthesis of Nafion nanowire arrays. By using sodium dodecyl sulfate as a surfactant in the surface decoration, the filling rate of the Nafion® nanowires in the corresponding template exceeds 95%, compared with the filling rate of 42% in an undecorated template, while on using cetyltrimethylammonium bromide as a surfactant, the filling rate is only about 20%. Systematical investigations show that the effect of surface decoration is induced by the combination of surface

tension and electrostatic force. This result is significant in organic nanowire fabrication.

The as-synthesized nanowires are about 85nm in diameter and several to over ten micrometres in length. Near-IR Raman and IR spectrum studies show that the polymer nanowires are composed of pure Nafion only. The Nyquist Z spectrum shows that the proton conductivity in an individual Nafion nanowire is much higher than that in the Nafion® membrane.

The best performance of the as-synthesized nano-fuel cell can be obtained: open circuit voltage 430mV, maximum current density and power density $4.33\mu\text{A}/\mu\text{m}^2$ and $0.44\mu\text{W}/\mu\text{m}^2$, respectively, which is enhanced by orders of magnitude compared with the traditional fuel cell. The nano-fuel cell provides a potential future for integrated self-powered nanodevices.

14:30 Oral

Nano-intermetallic/ceramic dispersed Al-based amorphous/nanocrystalline matrix composites synthesized by mechanical alloying

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Development of high specific strength-structural material is of significant interest to the transportation and aviation industry. The strength of crystalline and age hardenable aluminum alloys is limited a maximum of about 600 MPa. However, recent studies claim that the compressive strength of Al-alloys could reach over 1200 MPa in amorphous or nanocrystal dispersed amorphous condition. In the proposed talk, the genesis of complete/partial solid-state amorphization of Al-Cu-Ti and Al-Ti-Si alloys by high-energy planetary ball milling and mechanical properties of the same in compacts consolidated by high pressure and/or spark plasma sintering will be presented.

Dispersion of nano-intermetallic phases in amorphous matrix may be achieved either by continued milling or by subsequent controlled annealing. The identity and sequence of phase-evolution have been monitored by x-ray diffraction, high-resolution scanning and transmission electron microscopy and differential scanning calorimetry. The microstructural evolution during mechanical alloying follows a complex and interdependent sequence of grain refinement, mutual dissolution (alloying), nanocrystallization and/or amorphization. The genesis of solid-state amorphization or nanocrystallization has been investigated by positron annihilation spectroscopy, nuclear magnetic resonance and thermodynamic calculations based on modified-Miedema approach. Subsequent evaluation of mechanical property of the composites after compacting and sintering under special conditions of high pressure and/or spark plasma sintering suggests that fairly high hardness and compressive strength are possible to achieve under special conditions of milling and sintering.

Thus, it appears that the present approach may be useful in developing high specific strength Al-based amorphous and nano-intermetallic dispersed amorphous matrix composites by the inexpensive and versatile processing route of mechanical alloying.

14:45

Oral

Development and study of nanostructured titanium alloys with enhanced fatigue properties

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Titanium and titanium based alloys are among the most widespread structural materials applied in various spheres of engineering and medicine. However the problem of enhancement of their mechanical and fatigue properties still remains the issue of the day which we resolve by means of nanostructuring of metals and alloys through severe plastic deformation (SPD) techniques [1].

The objects of our research are commercially pure titanium and its alloys, Ti-6Al-4V and Ti-6Al-7Nb. The principles of nanostructure formation in the titanium materials by SPD processing are discussed in the present report. SPD processing of titanium billets included equal channel angular pressing (ECAP) in combination with conventional methods of thermomechanical treatment (TMT). Among them are various regimes of extrusion, rolling combined with annealing and strain-heat treatments in conditions of superplasticity.

For example, homogeneous ultrafine-grained (UFG) structure with a grain size less than 400 nm has been formed in the billets from Ti-6Al-4V alloy produced by combined SPD processing [2]. As a result of grain refinement, the strength of up to 1400 MPa and endurance limit on the basis of 10^7 cycles constituting no less than 700 MPa have been achieved.

The present report demonstrates the possibility of producing pilot articles from UFG titanium materials for applications in medicine and considers the advantages of the articles over the standard products.

[1] R.Z. Valiev, Nanostructuring of Metals by Severe Plastic Deformation for Advanced Properties, *Nature Materials*, Vol. 3, pp. 511-516 (2004).

[2] I.P. Semenova, G.I. Raab, L.R. Saitova, R.Z. Valiev, The effect of equal-channel angular pressing on the structure and mechanical behavior of Ti-6Al-4V alloy, *Mater. Sci. Eng., A* 387-389 (2004) p. 805-808.

Coffee break

Thursday afternoon, 20 September, 15:30

Session AGM10

Thursday afternoon, 20 September, 15:50

15:50

Oral

Combining microwave and pressure techniques for hydrothermal synthesis of ZnO and ZrO₂ nanopowders doped with a range of metal ions

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Combining high pressure and microwaves permits to decrease the reaction time one order of magnitude comparing to conventional hydrothermal reaction because of direct heating of the reacting fluids. On the other hand pressure permits to use high microwave power without sparking and boiling.

We carried out synthesis of nanopowders of zirconia doped with rare earth ions and ZnO doped with Al, Mn, Co, Ni, Ce. We studied also the effect of annealing on the nanopowders properties. The nanopowders were characterized as follows: density specific surface, grain size and grain size distribution, lattice constants and zeta potential, FTIR, luminescence and true chemical composition using ICP technique.

It was found that hydrothermal synthesis followed by annealing is a promising method for production of nanopowders doped with ions. Work supported by COST action D30 and Polish Research Network WITNANO

16:20

Oral

Automated X-Ray characterization of microstructure of ZrO₂ nanopowders

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Quantitative analysis of microstructure of nanocrystalline materials based on full-length XRD patterns is mathematically complex and time consuming task. On the other hand, number of laboratories producing nanopowders rapidly grows and there is a need of quick and cheap, routine high-quality characterization of nanomaterials.

In the present work an approach to an automated on-line characterization service will be shown. XRD powder patterns measured at home diffractometer or using synchrotron radiation can be uploaded to the internet expert system using its www website. The expert system performs spectral analysis of the noise, figures-out characteristic spots of

the pattern and attempts to analyze profiles of the best (usually: the strongest) reflections. Advanced Grain Size Distribution (GSD) profile analyses can be made for all phases present in the powder. Full data evaluation takes less than a minute and provides a report with publication-ready curves and numbers.

To be effective, the expert system contains some predefined knowledge about material being investigated. We present well known example of zirconia as a two-phase system of a simple tetragonal (almost cubic) and complex low-symmetry monoclinic nanocrystals. The expertise can be extended for other polycrystalline materials if needed.

16:35

Oral

Preparation and characterization of poly(1,4-butylene adipate-co-polycaprolactam/ polycaprolactone (PCL) multi-walled carbon nanotubes nanocomposites

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This study describes the preparation, characterization and properties of nanostructured materials based on poly(1,4-butylene adipate-co-polycaprolactam) (PBA-co-PCL)/polycaprolactone (PCL) and multi-walled carbon nanotubes (MWCNTs). Polymer/MWCNT nanocomposites were achieved by mixing polymers with various ratios of MWCNTs using solution process or melt process. The dispersion of MWCNTs into the polymer matrix was analyzed by transition electron microscope (TEM), and thermal stability of nanocomposite materials was studied by thermogravimetric analysis (TGA). Differential scanning calorimetry (DSC) was used to study crystallization and melting behaviour.

16:50

Oral

Synthesis of titania nanostructures and their application as supports for hydrogenation and oxidation reactions

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Nanoparticles of titania promise to have diverse applications such as environmental cleaning and purification, photovoltaics, antifogging, self-cleaning surfaces, pigment, and cosmetic sunscreen emulsion, etc. Nanotubular structures are of great importance because of their exceptional electronic and mechanical properties. Over the years the synthesis of nanotubes of oxides such as TiO₂ have attracted much attention and TiO₂ nanotubes are particularly interesting because of their large specific surface area, which may influence photocatalytic activity and potentially lead to application in environmental purification.

In our work TiO₂ derived nanotubes with relatively large specific surface areas (~270 m²/g) were synthesized using a simple and short methodology. In order to understand the morphology, crystal structure, surface properties and the basic nature the materials were characterized by various techniques such as SEM, TEM, BET analysis, XRD, TPD and TPR. It has been found that the proportion, particle size, and the crystallinity of tubes formed depend on the synthesis conditions employed and that each material exhibits unique properties. Composite materials with Au were synthesized by deposition precipitation (DP) method. Au supported on titania has been widely used in CO oxidation reactions, and numerous factors have been shown to enhance the activity of the Au/TiO₂ catalyst including Au particle size. In the current study, we have used a titanate obtained from KOH/TiO₂ as a support

for the Au catalyzed CO oxidation reaction. We also report on the effect of morphological and structural modification of titania on the activity and **stability** of Au supported catalysts for CO oxidation. The effect of the tubular structures on Au particle size is addressed and compared with that of P25.

Pd nanoparticles were also incorporated into the TiO₂ derived nanotubes and used as catalyst for the hydrogenation of phenol and ortho-chloronitrobenzene.

17:05 Invited

How we can stimulate intra-shell emissions of rare earth and transition metal ions in thin films and in nanoparticles

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Nanoparticles and quantum dots of wide band gap compounds activated with either transition metal (TM) or rare earth (RE) ions are perspective light emitting phosphors and fluorescence labels for applications in optoelectronics and biology and medicine. Unfortunately, intra-shell transitions of these ions are often inefficient, since are deactivated by parity and spin selection rules. In the consequence, intra-shell transitions of RE and TM ions are characterized by relatively long luminescence decay times and low efficiencies of radiative decay. This often limits possibilities of their applications as emission activators in modern light sources and as fluorescence labels.

In my talk I will discuss methods of stimulation of emission of intra-shell transitions of RE and TM ions. Concepts of co-doping, plasmonic mechanism of emission enhancement and idea of super radiance will be discussed based on the recent experimental results.

Posters

Monday, 17 September

Joint Poster Session (Part1)

Monday & Wednesday

Monday afternoon, 17 September, 15:50

15:50 Poster 1

Influence of magnetic structure on pinning in ferromagnet/superconductor bilayers

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The use of magnetic particles or ferromagnetic (FM) domains as pinning centers for vortices in the ferromagnetic/superconducting (FM/S) hybrid layered structures has been demonstrated in recent years. The goal is the creation of new devices in which this type of magnetic

pinning (MP) enhances the control of the critical current in the S layer. However, the influence of various materials parameters on the MP is not yet well understood.

In this work we investigate the effect of the shape of the FM domains on the MP in a series of FM/S bilayers which consist of niobium film as the S layer, and Co/Pt multilayer with perpendicular magnetic anisotropy as the FM layer. First, using the MFM technique we show that the variation of the thickness of platinum in the Co/Pt multilayer in the range $0.3 < t < 2$ nm leads to a change of the domain pattern, from less dense collection of domains of diverse sizes when $t=0.3$ nm, to a more dense collection of uniform smaller domains when t increases. We then use the array of small Hall sensors to study the flux penetration into the FM/S bilayers built of these Co/Pt multilayers with various t . We find that the enhancement of the critical current induced by the MP is present in all cases. However, the pattern which contains diverse sizes, but fewer domains induces smaller critical current enhancement than the pattern with dense small domains. In addition, large flux instabilities are observed during the flux penetration in the first case, while in the second case the flux penetration is smoother. The origins of these effects will be discussed.

15:50 Poster 2

Morphological and magnetic properties of cobalt nanostructures fabricated by focused ion beam

Jerzy Jaworowicz^{1,2}, Marek Kisielewski^{1,2}, Jacques Ferre¹, Nicolas Vernier¹, Jean-Pierre Jamet¹, Vitalii Zablotskii^{1,2,3}, Andrzej Maziewski², Iosif Sveklo², Jacques Gierak⁴, Eric Bourhis⁴

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Controlled and reproducible fabrication of nanostructured materials are one of the main challenges for next years. High resolution focused-ion-beam (FIB) patterning has been recently used to pattern magnetic nanoelements [1,2]. 30-keV Ga⁺ ions were focused on to ultrathin Pt/Co(3 nm)/Pt film as a 5 nm beam spot diameter to create arrays of square nanodots, nanostripes and irradiated narrow lines. Atomic Force Microscopy (AFM) evidence a film wetting effect [3] in FIB-irradiated regions and, for high doses the sputtering of the film surface. Considering the above film with in plane magnetization, high resolution Magneto-optical Kerr Microscopy combined with Magnetic Force Microscopy reveal that out-of-plane magnetization states can be generated by choosing properly patterning and dose in the FIB process. Particular magnetization configurations near edges of FIB affected areas are observed and discussed taking into consideration the edge effect recently predicted in [4].

[1] T.Aign, P.Meyer, S.Lemerle, et al. Phys. Rev. Lett. 81, 5656 (1998)

[2] A. Ruotolo, S. Wiebel, J.-P. Jamet, N. Vernier, D. Pullini, J. Gierak, J. Ferré,

Nanotechnology 17, 3308 (2006)

[3] X. Hu, D. G. Cahill, and R. S. Averback, Appl. Phys. Lett. 76 (2000) 3215.

[4] A. Maziewski, V. Zablotskii, and M. Kisielewski, Phys. Rev. B 73 (2006) 134415.

15:50 Poster 3

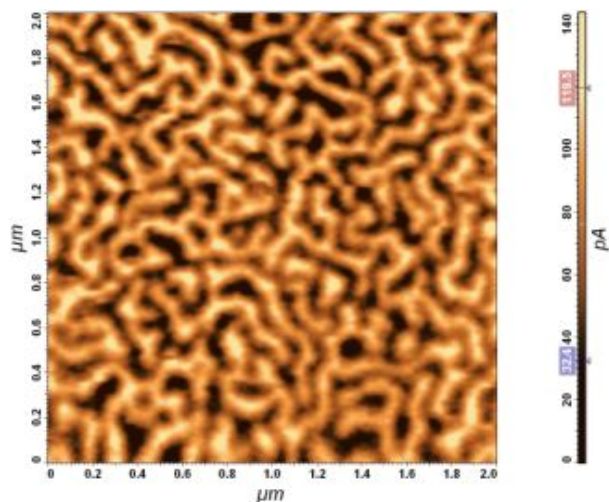
Magnetic properties of (FeAu)_n alloys

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Magnetic domain structures of FeAu superlattices are studied by experiment and modelling. The domain period, magnetic hysteresis loops and magnetic susceptibility were measured for a wide range of the film thickness, 3- 50 monolayers. Domain structures were observed by utilizing a magnetooptical installation based on the Kerr effect (MOKE) and magnetic force microscope (MFM). Typical MFM images of domain structures are shown in Fig.1. It is found that the zero-field domain period is inversely proportional to the saturation field. The thickness dependence of the domain period is well described by the classical domain models using the modified relationships for the domain wall energy density [1].



MFM domain image for the 30 monolayers FeAu.

[1] M. Kisielewski, A. Maziewski, T. Polyakova, and V. Zablotskii. Phys. Rev. B 69, 184419 (2004).

15:50 Poster 4

Acoustic Spectrum of Nanoparticles of a Heysler Shape Memory Ferromagnetic Alloy

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Nanofilms of Heysler $\text{Ni}_{2+x+y}\text{Mn}_{1-x}\text{Ga}_{1-y}$ shape memory alloy received by methods of laser and magnetron spray or molecular-beam epitaxy have differences in comparison with specimen of the same stoichiometric

constitution: they have lower phase transition temperatures and magnetic moments. Obvious features of ferromagnetic nanoparticles are big surface to volume ratio and limited amount of atoms. Difference of a surface power constants from their values inside the infinite crystal (that is due to non-compensated powers on the free surface) leads to a grid deformations, appearance of surface tension and localized surface waves, as far as to strong anharmonism. Sizes of an elementary crystallographic cell $a=0,5825$ nm for room temperature and $a=b=0,5920$ nm, $c=0,5566$ nm at for low temperatures for Ni_2MnGa .

Photonic spectrum of Heysler alloy nanoparticles in austenite phase was received from the frequency spectrum infinite crystal, taking into account the Einstein model of a solid body, atomic-sphere approximation and contribution of the particle surface. While N increases, nanoparticle photonic spectrum quickly comes close to an oscillation spectrum of an infinite crystal. Numeric estimate of the maximum critical diameter of the Ni_2MnGa one-domain nanoparticle makes D_c approximately equal to 50 nm. Particles with sizes bigger than $d=4$ nm are owning ferromagnetism. There is no shape memory effect in one-domain nanoparticles.

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Photonic effect in ferromagnetic nano-films of Heysler alloy

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The idea of a spintronic analogue of a field-effect transistors, spoken in [1] and based on the control of spin polarization of a charge carriers with use of a spin-orbit interaction with outer electric field is still not realized experimentally. The same is true for idea of a polarization transistor [2], the essence of which is in polarization magnetooptic bistability effect in magnetoordered films. Such effect, been found experimentally, would make it possible to realize binary logic in s- and p-polarization of a light beam (or even single photon) and to build quantum computer with colossal parallel information processing in perspective.

Nowadays there is a growth of interest to the only one known magnetoordered Heysler Ni_2MnGa alloy, in which a shape memory effect as on temperature, so on outer magnetic field was reliably discovered. A 90 nm and 30 nm films of this substance were synthesized, i.e. ones containing only several tens of crystallographic cells in a specified direction. Here of Kerr's and Faraday's effects in films of $\text{Ni}_{53}\text{Mn}_{25}\text{Ga}_{22}$, $\text{Ni}_{2.18}\text{Mn}_{0.82}\text{Ga}$ are considered with combined magnetostructured phase transition with use of a scalar relativistic version of the tight-binding linear muffin-tin-orbital atomic-sphere approximation within the local-spin-density approximation. An outer magnetic field is deforming the film and consequently causing reallocation of a "down" and "up" spin polarized electrons on the film surface, influencing on photons polarization change as at photons reflection from a metal film, so at their transition through it. It seems that electrons spin overturn can cause amplification effect (transistor) of transformed light pulses.

Author is grateful for BRFFI-RFFI F04R-080 grant for partial financial support

References

[1] Datte S., Das B. Appl.Phys.Lett.1991.U.56.P.665

[2] Kuzavko Yu.A., Shavrov V.G. Digests of XII Soviet seminar "Novel magnetic materials for microelectronics" Novgorod, USSR, 1990 v.2. p.97

15:50 Poster 6

Surface waves in nanofilms of Heysler ferromagnetic alloy

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Giant magnetodeformations reachable in ferromagnetic films of $\text{Ni}_{2+x}\text{Mn}_{1-x}\text{Ga}_{1-y}$ Heysler alloy with magnetocontrolled shape memory effect are stimulating their use as components of micromechanical devices (scanning ones, sensor graphic displays, switches, etc.), but until now such explorations are on the initial stage. Differentiated information was received about their synthesis, and only structural magnetic characteristics were measured. Mechanical and acoustical qualities of films are totally unexplored, despite the fact that values of reversible magnetodeformations, elasticity and plasticity are very important characteristics of their practical usage.

Here we consider a Ni_2MnGa (001) film on a (001) substrate of a GaAs, Si cubic crystall. In such structure a volumetric TA_2 with $k=[110]$ satisfies the boundary conditions and is not connected with LA and TA_1 . Two last waves are forming Relay wave due to a boundary conditions or a Stounly wave, which are weakly influenced by such phase transition. Axis are to be chosen normally to the film free surface. TA_2 transforms into a Lyav wave in such system. For structures of (001) Ni_2MnGa - (001) GaAs (Si) zero modes of Lyav waves were numerically explored, as most practical and easy-watching ones. It is shown that at their spread along the $[110]$ axis in (001) plane their substantial softening takes place, as far as high sensibility to the film coating thickness and to changes of $c'=(c_{11}-c_{12})/2$ elasticity modulus which is responsible for martensite and premartensite phase transitions. Dispersion and frequency dependences are presented for films of 30, 90, and 1000 nm for ferromagnetic, premartensite and martensite stages.

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15:50 Poster 7

Magnetite-based nanoparticles as contrast agents for MRI

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Results of a XAS study of new magnetic nanoparticle materials based on iron metal and iron oxides are presented. The materials have potential biomedical applications, e.g. as contrast agents in MRI. Several samples of carbon- silica- and zeolite coated nanoparticles have been obtained by ball milling, arc melting and co-precipitation techniques. The coatings prevent agglomeration of the particles, slow down their removal from the circulation in blood and limit absorption of the harmful transition elements. In order to determine the local structure and the Fe valence state in the materials, the X-ray absorption spectroscopy in the XANES (X-ray Absorption Near Edge Structure) range was used. The experiments were performed at the Fe:K edge at room temperature in Hasylab/DESY, Hamburg. Metallic Fe, hematite, maghemite and magnetite were used as standards. The edge energy and shape of the spectra of the materials prepared by coprecipitation and milling of magnetite are the closest to those of maghemite. This reveals the oxidation occurring during the preparation procedure. No noticeable difference is observed between the co-precipitated materials annealed in air and argon atmosphere. The results show that the standard characterization of nanomaterials using X-ray diffraction is not sufficient as it does not provide information on the real stoichiometry of iron oxide. The edge energy and shape of the spectra of the nanoparticles prepared by the arc melting of the magnetite filled graphite electrodes are similar to those of metallic iron, which reveals a reduction of magnetite to iron. This fact can be explained by the reducing effect of carbon during melting of magnetite. Preliminary MRI experiments performed on water suspensions of the materials studied indicate their high efficiency in decreasing the T_2 and T_2^* relaxation times.

15:50 Poster 8

Magnetic anisotropy of Co films annealed by laser pulses

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Change of magnetic anisotropy from in-plane to perpendicular state was observed in Co films after laser pulse annealing. High power laser pulses generated by the Chameleon Ultra Coherent laser were focused with a lens of 20 mm focal length on the epitaxial Au/Co/Au sandwich

prepared by magnetron sputtering. The laser pulses are of 700 nm wavelength, 200 fs width, the repetition rate is 80 MHz, and average power 2W. Magnetic properties of irradiated samples are characterized by magneto-optic Kerr microscopy, magnetic force microscopy (MFM) and magneto-optic vector magnetometry. Irradiation effect is studied as function of Co thickness, laser power and exposition time. Perpendicular magnetization states were obtained for Co films thicker than corresponding spin reorientation transition (SRT) thickness of non-irradiated one.

15:50	Poster	9

Molecular dynamics simulation study of titanium-decorated fullerene cluster

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The titanium decorated carbon nanotubes or fullerenes are considered to be a potential hydrogen-storage media [1-3]. We have studied (MD method) the small titanium-decorated cluster ($C_{60}[TiH_2]_6$) over a wide range of energy, from solid state to the vaporization of the nanosystem. The low energy, solid state structure of the cluster was obtained (pentagonal bipyramid). The energy evolutions of several time-correlation functions of physical observables of $C_{60}[TiH_2]_6$ molecule were calculated, analyzed and discussed.

References

1. T. Yildirim, Jorge Íñiguez and S. Ciraci Phys. Rev. B **72**, 153403 (2005)
2. T. Yildirim and S. Ciraci Phys. Rev. Lett. **94**, 175501 (2005)
3. E. Durgun S. Ciraci W. Zhou and T. Yildirim Phys. Rev. Lett. **97**, 226102 (2006)

15:50	Poster	10

The liquid crystal phase in small mesogene cluster (9CB)_n - MD study

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Using molecular dynamics (MD) technique, we have investigated the nano droplet composed of twenty mesogene molecules 4-n-alkyl-4'-cyanobiphenyl (9CB). The geometry of 9CB molecule was calculated by DFT method. The intermolecular interaction is taken to be the full site-site pairwise additive Lennard-Jones (LJ) potential. We calculated the radial and orientational distribution functions in the temperature range from 100 to 350 K, as well as the linear and angular velocity autocorrelation functions and their Fourier transforms. We have observed the liquid crystal ordering in nanoscale system studied, up to its vaporization temperature.

15:50	Poster	11

MD study of the plastic and liquid phase of endohedral potassium ion fullerene cluster ($K^+@C_{60}$)₇

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We have simulated (MD method) the nanosystem composed of only as little as seven endohedral fullerene $K^+@C_{60}$ molecules. The interaction is taken to be the full site-site pairwise additive Lennard-Jones (LJ) potential, which generates both translational and anisotropic rotational motions of each endohedral fullerene. Our atomically detailed MD simulations allow to analyze the dynamics of the motion of $K^+@C_{60}$ molecule inside the cluster. The radial distribution function, the mean square displacement, the translational velocity correlation functions and their Fourier transforms of endohedral fullerene have been calculated for several energies of the nanosystem studied.

15:50	Poster	12

Ab initio study of electronic properties of alkali doped fullerene dimer

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The electronic properties of fullerene-based nanostructures $A(C_{60})_2$ (A – alkali atom) are examined by first-principles calculations based on the density functional theory. The electron conductivity of the $(C_{60})_2$ dimer is low owing to the constraint of the junction of the molecules. When the fullerene dimer are doped by inserting an alkali atoms A , the unoccupied state around the junction is filled and the conductivity can be significantly improved.

15:50	Poster	13

Quantumchemical Simulation of Growth and Structural Stability of Silicon Nanotubes

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The results of quantumchemical simulation (B88-LYP DFT, NDDO-PM5) of growth and structural stability of single-walled silicon nanotubes (SiNT) are presented. Three type of SiNT - the square, pentagonal, and hexagonal have been studied. The heat of formation of SiNT as a function of the number of forming plates and the energy per atoms are analyzed. The growth of SiNT was modeled by the sequential attachment of forming plates, broking bonds being saturated with hydrogen atoms. More stable structure was demonstrated is hexagonal SiNT, the growth of square SiNT being unstable. We also analyzed the growth of mixed nanotubes. On initial stage of growth (when the number of plates in SiNT is less 5) the formation of mixed pentagonal SiNTs are more favorable. With growing of the number of SiNT plates ($n > 14$) more favorable is the syntheses of mixed hexagonal SiNTs. On initial stages of growth, the formation both pure

and mixed SiNTs are possible. This fact may be the reason why an experimental synthesis of the perfect SiNTs is difficult. The distribution of charge of SiNT atoms has been analyzed also.

15:50 Poster 14

Molecular dynamics simulation of the properties of homocysteine ultrathin layer covered carbon nanotube

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Excessive of homocysteine in the human body is recently considered as a factor increasing the risk of the cardiovascular system diseases. The nanosystem composed of finite number of homocysteine molecules covered single walled carbon nanotube have been studied by MD technique. The translational and rotational velocity correlation functions and their Fourier transforms have been calculated for several temperatures. The qualitative interpretation of translational and reorientational dynamics of homocysteine molecules in such specific environment is presented.

15:50 Poster 15

Dielectric relaxation of cholesterol domain near graphite wall – computer simulation

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Molecular dynamics (MD) studies are presented of the cluster composed of cholesterol molecules near graphite wall. We have investigated the dynamical observables of cholesterol as a function of temperature. We focus our attention on the temperature evolution of the calculated total dipole moment autocorrelation function of cholesterol molecules and dielectric loss of the cluster. The comparison with the dielectric relaxation of pure cholesterol cluster (without graphite wall) is also presented and discussed.

15:50 Poster 16

The influence of carbon nanotube on the dynamics of cholesterol molecules in the lodgment localized near transmembrane protein – MD study

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We have made the molecular dynamics (MD) simulations for the cluster of cholesterol molecules localized near the transmembrane protein at the physiological temperature $T = 309.75$ K. We have observed that the cholesterol molecules form a lodgment on the surface of protein. Next we have studied the influence of carbon nanotube on the several physical quantities of cholesterol molecules among those: radial distribution function, the mean square displacement, diffusion coefficient, linear and angular velocity autocorrelation function.

15:50 Poster 17

The effect of grain size distribution on mechanical properties of nanometals

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Predicting material properties from the knowledge of the internal microstructures is attracting some interest in the fields of Materials Design and Engineering. The most commonly used equation known as Hall-Petch Relationship (HPR) deals with the relationship between flow stress and *average* grain size. On the other hand, there are many evidences that other statistical quantities of grain size distribution in materials may have impacts on the mechanical properties. These could even be more pronounced in the case of grains of the nanometer size, where the HPR is not valid anymore and the Reverse-HPR is applicable.

In the present paper, the statistical model of the relationship between the flow stress and grain size distribution is proposed. The model considers different *deformation mechanisms* and may be applied to nanometals.

The results obtained with the model shows that the dispersion of grain size distribution plays an important role in the design of desirable mechanical properties.

15:50 Poster 18

Evaluation of the efficiency of semiconductor materials for quantum computing

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Solid-state implementations of quantum computing (QC) are particularly attractive because of the possibility of using existing computer technology to scale small numbers of qubits up to the 10^5 or so that would be needed for nontrivial computations. In this communication we examine the decoherence times for some of the existing proposals based on electron-spin qubits in the different Si-based heterostructures. We calculate the relaxation rate ($1/T_1$) as a function of uniaxial strain, temperature, magnetic field and alloy content. In general, we find that the relaxation rate is strongly decreased by uniaxial compressive strain, making this strain an important positive design feature. Si-rich structures are to be preferred because relaxation time increases with increasing Si content in the investigated alloys.

15:50 Poster 19

DLS and AFM in measuring of gold nanoparticles.

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This paper compares the capability of dynamic light scattering (DLS) analysis and atomic force microscopy (AFM) for characterizing size of various nanoparticles in the size range 15-40 nm. Polyelectrolyte-protected gold nanoparticles have been obtained by heating different polyethylenimine containing polyelectrolyte/HAuCl₄ aqueous solutions. Experimental data indicate that different initial molar ratios of polyelectrolyte to gold allow to control the size of well-stabilized gold nanoparticles. The average AFM size of gold nanoparticles is 18.4 +/- 2.6 nm and is slightly lower than that obtained using DLS (22.0 +/- 3.5 nm). Comparison of experimental data from DLS and AFM demonstrates good correlation in measurements of size and its distribution using both methods. Measurement of changes in nanoparticles' size during their functionalization is relatively easy using either of these methods.

15:50 Poster 20

Impact of Triple Junctions on Grain Growth in Nanocrystalline Materials

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Effect of finite mobility of grain-boundary triple junctions on grain growth is studied using numerical simulation. These junctions are shown to retard grain growth, especially at small grain sizes, and can transform the growth kinetics from parabolic into linear one. It is concluded that grain-boundary junctions can contribute to microstructure stability in single-phase nanocrystalline materials. Magnitude of triple-junction mobility in nanocrystalline Fe is estimated from the experimental data on grain growth kinetics.

15:50 Poster 21

Chemical Patterning and Chemical Contrast Imaging with the Tip of an Atomic Force MicroscopeStefan Walheim¹, Matthias Barczewski², Roland Gröger², Matthias Müller², Andreas Pfrang³, Birgit Riedel¹, Christian Obermair², Thomas Koch¹, Thomas Schimmel^{1,2}

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When monitoring surface processes and chemical surface reactions, it often is of great importance to be able to monitor chemical surface changes with lateral resolution down to the nanometer scale. We demonstrate that using an oscillating tip in the contact mode of the AFM, chemical contrast can be visualized. The technique of **Chemical Contrast Imaging (CCI)** is described, and examples for imaging surface reactions are given, including carbon layer deposition and carbon layer etching.



At higher forces in the range between 100 nN and 10 µN, the AFM tip can be used as a “chemical pen” to induce chemical processes locally at predefined positions. In this way, local etching can be induced, and tribo-chemical as well as tribo-electrochemical processes can be studied with lateral resolution on the nm scale. Site-selective AFM-tip induced molecular exchange reactions on the nm-scale are demonstrated for different systems, including polymer brushes, and the corresponding structuring mechanisms are discussed.

AFM topography (left) and chemical contrast due to different tip-sample interaction (right) on a droplet-etched silicon wafer surface. Scale: 12 µm x 12 µm.

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Formation of carbon fibers in high-voltage low-current electrical dischargesArkadiusz T. Sobczyk¹, Anatol Jaworek^{1,2}, Eryk Rajch, Maria Sozańska³

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The paper presents results of investigation of growth of carbon fibers in low-current electrical discharges. The method is based on hydrocarbon pyrolysis in electrically generated plasma in oxygen-free atmosphere. Various types of corona discharges of both polarities were tested with regard to their applications to carbon fiber synthesis.

The discharge was generated between a stainless steel needle, and a plate made of a nickel alloy, spaced at 15 mm. The experiments were carried out in argon atmosphere in normal temperature and pressure. Cyclohexane or n-hexane were used as the carbon feedstock gases. The voltage of the discharges was changed in the range from 2 kV to 30 kV to obtain a stable low-current arc discharge. The current of the arc discharge was varied from 1 to 6 mA. The changes in morphology of the carbon fiber surface was observed under a SEM Hitachi S3400N.

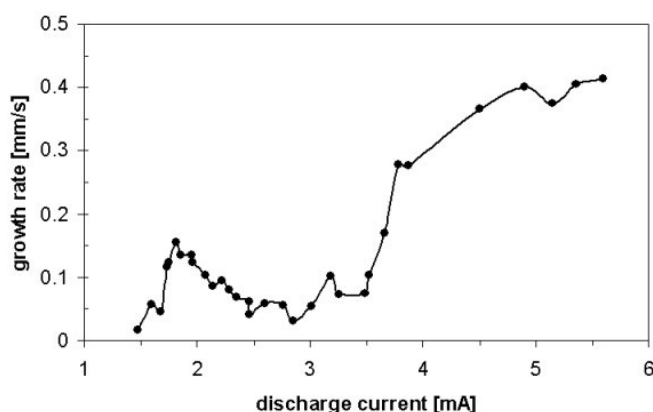


Fig. 1.

It was noticed during the experiments that carbon fibres were built at the tip of the discharge needle electrode as an effect of plasma pyrolysis. The length of synthesised carbon fibers varied in the range from 0.5 to 13 mm after a constant time of syntheses of 30 s, and their diameter was from about 20 to 100 nm. With the current increasing up

to about 2 mA, the growth rate increased (Fig.1). For larger currents the growth rate started to decrease, and next, for the current >3 mA, increased slowly again. For small currents (<2 mA), the structure of the fiber was homogeneous and the surface was smooth. For larger currents (>3 mA), the fiber consisted of two different co-axial structures: a central core and an external porous shell. An example of carbon fiber synthesised in the discharge is shown in Fig.2.

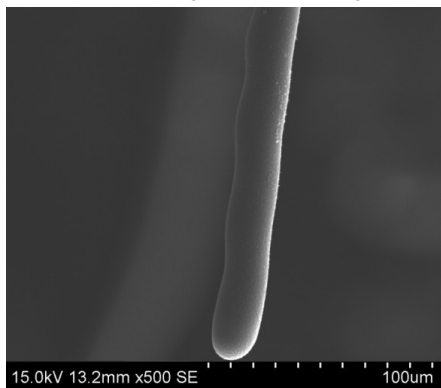


Fig. 2.

The synthesis of carbon fibers in argon at atmospheric pressure was more efficient than in nitrogen. The carbon fiber does not grow in carbon dioxide atmosphere. Synthesis in the low-current arc discharge is the fastest process from all types of discharges tested.

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Imprint properties of $\text{IrO}_2/\text{Sr}_2(\text{Ta}_{1-x}\text{Nb}_x)_2\text{O}_7/\text{SiO}_2/\text{Si}$ structure device formed by rf sputtering and oxygen radical treatment

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Recently, nonvolatile memory devices having a ferroelectric gate structure have considerable attention from the view points of nonvolatile, high-speed, high-density, and obeying the scaling rule. $\text{Sr}_2(\text{Ta}_{1-x}\text{Nb}_x)_2\text{O}_7$ (STN, $x=0.3$) is suitable for use as a ferroelectric material for ferroelectric memory field-effect transistors, because it has a low dielectric constant. However, the fabrication of STN on an amorphous insulator, such as SiO_2 , is difficult. In particular, in the case of STN, because its crystallization annealing temperature is 950°C , the metal element of STN and Si react mutually during crystallization annealing. We have already developed and reported [1] the STN formation technology on amorphous SiO_2 and MFIS structure device operation. These were realized using (1) a thin seed layer formed by rf sputtering and microwave-excited plasma treatment, and (2) by the formation of a ferroelectric film, whose oxygen vacancy is reduced by ferroelectric multilayer stack (FMLS) deposition. This plasma treatment can oxidize the surface of the film and supply ion bombardment energy to the surface of the film. In this work, we report imprint and data-programming properties of the MFIS structure device. Threshold voltage shift of the device is within 10mV and 30 mV after writing operation of 10^9 and 10^{10} cycles, respectively.

[1] I. Takahashi et. al., Japanese Journal of Applied Physics, Vol. 45, pp. 3207-3212, 2006

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Epitaxial Growth of InN Films by Molecular-beam Epitaxy Using Hydrazoic Acid (HN_3) as an Efficient Nitrogen Source

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Epitaxial InN films have been successfully grown on c-plane GaN template by gas-source molecular-beam epitaxy with hydrazoic acid (HN_3) as an efficient nitrogen source. Results in residual-gas analyzer show that the HN_3 is highly dissociated to produce nitrogen radicals and can be controlled in the amounts of active nitrogen species by tuning HN_3 pressure. A flat and high-purity InN epilayer has been realized at the temperature near 550°C , and a growth rate of 200 nm/hr is also achieved. Moreover, the epitaxial relationship of the InN(002) on the GaN(002) is reflected in the X-ray diffraction, and the full-width at half-maximum of the InN(002) peak as narrow as 0.05° is related to a high-quality crystallinity. An infrared photoluminescence (PL) emission peak at 0.705 eV and the integrated intensity increasing linearly with excitation power suggest that the observed PL can be attributed to a free-to-bound recombination. These findings reveal that HN_3 has a great potential to be an alternative nitrogen source in the growth of high-quality nitride semiconductors. Therefore, we believe that the HN_3 could be a high-performance nitrogen source, which can be applied to other growth techniques, such as MOVPE, HVPE, etc.

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Effect of growth temperature on the characteristics of ZnO films grown on Si(111) substrates by metal-organic chemical vapor deposition

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ZnO is a wide band gap semiconductor with $E_g=3.36$ eV at room temperature. Since the report on the stimulated emission from ZnO films was published in 1997, ZnO has attracted more and more attention. In order to fabricate the ZnO based optoelectronic devices, high quality ZnO films are necessary. In addition, for the large-scale production, the research on the metal-organic chemical vapor deposition (MOCVD) growth of high quality ZnO films is also very important.

Diethylzinc (DEZ) and O_2 or H_2O are normally used as the precursors for the MOCVD growth of ZnO films. However, in this the reactions are so fast that the gas line should be designed more carefully in order to prevent the pre-reaction. Therefore, we used the DEZ and CO_2 as precursors to grow ZnO, in which the reaction is mild.

The ZnO films were grown on Si(111) substrates by MOCVD with DEZ and CO₂ as the precursors. The effects of the growth temperature on the growth rate, surface morphology, structure and optical properties of the ZnO films were investigated. The temperature window was found between 450 °C and 700 °C. In this region, with the increase of growth temperature, the growth mode was changed from kinetics limited to mass transfer limited and finally terminated as desorption limited. In addition, the ZnO crystalline grains were increased, resulting in the improvement of structure and optical property. Moreover, the rougher surface at high growth temperature also increased the PL intensity. Using CO₂ as oxygen source, the optimal growth temperature was found to be near 600 °C.

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Surfactant assisted hydrothermal synthesis of Ba-ferrites

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Barium hexaferrite in a fine form is a major candidate for perpendicular magnetic recording medium and for the absorbers of the electromagnetic radiation. The demand for the synthesis of nanosized particles is large since they can be used in important technical applications. Nanoparticles of Ba-hexaferrite can be prepared using various methods, most commonly including high energy milling, co-precipitation, sol-gel method and hydrothermal syntheses. The hydrothermal synthesis of Ba-ferrite was performed in the presence of large organic molecules which stabilize the small crystallites during their formation and prevent the Ostwald ripening. The nanosized Ba-hexaferrite particles were prepared using surfactants assisted hydrothermal synthesis. The particle size and the phases formed after the hydrothermal syntheses were strong dependent on the selection of the proper surfactant and the conditions of the syntheses. A bi-modal particle size distribution of Ba-hexaferrite particles with an average size of 20-30 nm in a form of round crystallites and 100-200 nm sized platelets were obtained. The optimization of the particle size distribution will be performed by selecting the proper synthesis conditions.

15:50 Poster 27

Electrocatalytic activity of PtWO_x and WO_x catalysts on different carbon support

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The reduction of oxygen has always been a cause of significant power loss in fuel cells technology. Pt based catalysts still remain the optimum electrocatalysts for oxygen reduction. For the catalyst preparation, Vulcan XC72 is commonly used as carbon support. A main focus of this research is the synthesis of PtWO_x and WO_x on different carbon support as alternative catalyst for fuel cell technology.

PtWO_x and WO_x catalysts on Vulcan XC-72, carbon aerogel and MWNTs are prepared by impregnation-reduction method. SEM and EDAX analysis give the information about catalysts morphology and

composition. The electrocatalytic ORR activities were characterized using voltammetric technique. We have also attempted an initial comparison of the electrocatalytic activities of PtWO_x and WO_x with commercially-available Pt/C catalyst (E-tek) under same voltammetric measurement conditions. The results revealed important insight into the electroactivity of our catalyst and give the possibility of replacing precious metal anode catalysts with transition metal compounds for hydrogen oxidation.

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ZnS/Silica Nanocable as Biological Nanosensors Using Avidin-Biotin System: Nanodevice Fabrication and Application

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Compound semiconductor/isolator (ZnS/silica) core/shell nanocables have been used to fabricate field effect transistors (FETs) using focused ion beam (FIB), which involved two major steps. First, cutting the nanocables to bare ZnS core using FIB could be achieved by accelerating concentrated Gallium ions to a specific site, which etches off any selected area, leaving a very clean surface of ZnS core. Secondly, the Pt mixture was selectively deposited using FIB to contact the prepatterned electrodes and ZnS core. Electrical characteristics of nanocable-based FET show the typical behavior for n-type FET devices.

To explore NWs-based sensors for biological recognition, silica shell of nanocable was functionalized with biotin. Biotinylation reagent used here is amine-reactive to react with APTES-modified ZnS/silica nanocable. Biotin could be efficiently attached silica shell with little change in the electrical characteristics of nanocables. The binding of avidin-biotin with low dissociation constant would be utilized for specific recognition. Conductance-time measurement shows that the conductance of biotin-modified nanocable increases rapidly and considerably to a constant value upon addition of the avidin solution and that this conductance value is remained even after the addition of BSA solution

In summary, we have also demonstrated the nanocable FET devices for the sensitive, label-free, real-time, selective, and electrical detection of protein using avidin-biotin system. This work presents the potential of core-shell-nanowire-based FETs for biological sensing using FIB nanofabrication and biotinylation.

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Effects of Intermediate Laser Ablation of Mn on Deposition of ZnS Films on ZnO Nanorods Grown by Chemical Vapor Deposition

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We have studied of deposition of ZnS:Mn films on ZnO nanorods grown by low-pressure chemical vapor deposition. In this study we report on the effects of laser ablation of Mn that is performed after ZnO nanorods' growth and before deposition of ZnS:Mn films. We call this ablation as an intermediate laser ablation. The ZnO nanorods are grown on Si(111) substrate by chemical vapor deposition (CVD) combined with laser ablation of Mn. Metal Zn vapor and O₂ gas are

used as precursors to synthesize ZnO and N₂ is used as carrier gas. The Mn pellet is placed near a substrate in a deposition chamber and ablated by a pulsed Nd:YAG laser beam (10 shots/sec, 0.04 J/shot). Firstly, ZnO nanorods with 100 nm diameter and 1700 nm height are grown for 10 min growth-time that are vertically aligned to a substrate. The pressure is 26.6 Pa, the temperature is 550 °C and O₂ mass flow rate is 0.88 SCCM. Then only the laser ablation of Mn is performed for 5 min without O₂. Finally, ZnS:Mn films are deposited by changing the precursor from O₂ to H₂S for 10 min. The pressure is 67 Pa and 133 Pa, and mass flow rate of H₂S is 2.0 and 4.0 SCCM. It is found that the intermediate laser ablation of Mn has a drastic effect on growth of ZnS:Mn films. When the intermediate laser ablation is not performed, the ZnS:Mn film mainly grows along the axis-direction of ZnO nanorod. When it is performed, however, the growth for lateral direction becomes dominant, although the growth rate along the axis-direction is not so changed. For example, when the mass flow rate of H₂S is 2 SCCM and pressure is 67 Pa, the total length of nanorod is 3600 nm and diameter is 100 nm when the intermediate laser ablation is not performed. On the other hand, when the intermediate laser ablation is performed the total length of nanorod is 3000 nm and diameter is 540 nm. It is concluded that the intermediate laser ablation has an effect to enhance the growth rate of ZnS:Mn layer for the lateral direction of ZnO nanorods.

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Continuous Flow Supercritical Fluid MOCVD of Cu and Ag Thin Films: Reactor Design and CFD Modelling.

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Supercritical Fluids (SCFs) are unique solvents that possess thermodynamic and transport properties intermediate of those of the liquid- and gas-like states. These unusual solvents have previously been used to thermally batch deposit thin metal films within micro- and meso-scale templates [for example 1,2]. We have developed a new continuous flow reactor that allows deposition of metals and metal oxides from SCFs, which opens the possibility of rapid multi-layered conformal growth without the need to depressurize. However, the engineering of performing this task in flow is not straightforward. SCFs are highly dynamic, where changes in temperature or pressure lead to large thermal and diffusion gradients that strongly influence flow patterns. The use of SCFs and high-pressures also introduces obvious analytical challenges. We have deposited Cu and Ag thin films from SCF-soluble MOCVD reagents, with an aim to understand, predict and effectively utilize the SCF flow.

In order to design successful tubular SCF-MOCVD flow reactors, we have incorporated custom thermodynamic FORTRAN routines into a commercially available CFD package, CFD-ACD+; this allows us to calculate many SCF properties. We will present simulations and experimental validations of flow and metal deposits from MOCVD reagents within these reactors. Reactor development, in combination with ACE+ calculations, has led to several generations of warm- and hot-walled reactors. Hot-walled reactors allow for much higher temperatures to be achieved, i.e. >500°C inside SCF reactors, and hence semi-conductor materials may be rapidly deposited. However, as we will demonstrate,

their use further increases the need for specialized engineering solutions.

1. Watkins, J et al., Microelec. Eng., 2002, 64, p53
2. Holmes J et al., J.Phys:Condens. Matter, 2003, 15, p8303

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Electrospray nanocoating of microfibers

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Electrospraying is a method of liquid atomisation by subjecting a liquid at a capillary nozzle to an electrical stress. The liquid meniscus maintained at high electric potential elongates into jet and disrupts into fine droplets. The droplets obtained by this method are highly charged and can be of submicron size, which can be readily controlled via liquid flow rate and voltage applied to the nozzle. Since last decade, there is increasing interest in applying electrospray processes in nanotechnology. The deposition efficiency of a charged spray on an object is manifold higher than for un-charged one. The droplets can be directly deposited onto a substrate enabling surface coating or direct writing in submicron scale.

The paper presents experimental results of deposition of nanopowder on microfibers using electrospraying. The process is aimed at formation of fibrous filters of enhanced collection efficiency in submicron range by making tighter unwoven fabric covered with a catalytic material.

In the presented experiments a layer of MgO particles was formed on an polyamide fiber 30 mm in diameter by deposition the particles from a methanol suspension via electrospraying. Low particle concentration and ease evaporation of the solvent used enabled deposition of uniform tight layer on the fibers. The spraying process was carried out using a fine stainless steel capillary of 200 mm i.d., and 400 mm o.d. The obtained layer was grainy and composed of particles smaller than 200 nm.

The electrospray process is very efficient, cheap, flexible, and can take place at atmospheric pressure and ambient temperature.

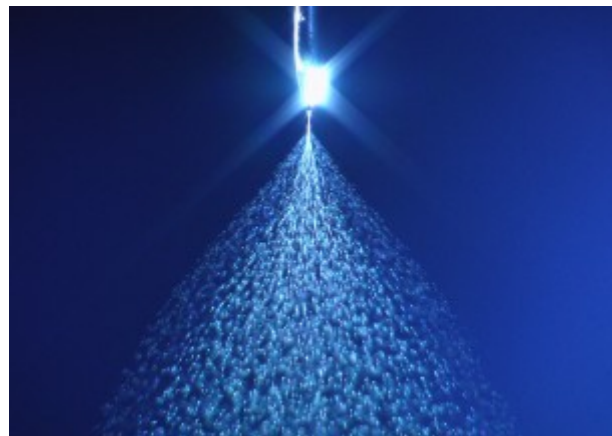


Fig. 1. The cone-jet mode of electrospraying. Exposure: 3 ms. Liquid: MgO suspension in methanol, voltage: 5.1 kV.

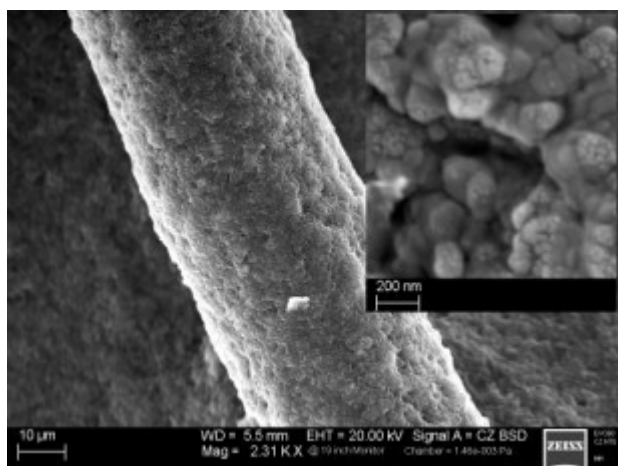


Fig. 2. SEM images of MgO particle layer deposited by electrospraying on a polyamide fiber 30 mm diameter. Inset: a fragment of the coated fiber showing grainy structure of the layer.

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Nanosized barium hexaferrite powders obtained by single microemulsion techniques

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The present work aims to prepare barium hexaferrite ($\text{BaFe}_{12}\text{O}_{19}$) particles by a single microemulsion technique and to study the possibility to control their sizes. One of the advantages of the single microemulsion technique is considerably lower cost compared to the other microemulsion methods. We use a water-in-oil microemulsion system, where cetyltrimethylammonium bromide (CTAB) acts as a cationic surfactant, n-butanol as a co-surfactant, n-hexanol as a continuous oil phase, and an aqueous phase contains FeCl_3 and $\text{Ba}(\text{NO}_3)_2$ (with the Ba/Fe ratio was 1/10). The co-precipitation was performed by a concentrated solution of NaOH at pH = 12. The second step of the synthesis includes calcination of the precursor at low temperature (900°C) in air ambient.

Since the technological synthesis conditions possess a significant influence on the chemical, structural and physical properties of the obtained materials, we investigated the relation between the experimental conditions of $\text{BaFe}_{12}\text{O}_{19}$ synthesis and their properties. We studied the influence of the stirring time, the water/surfactant ratio (W_0) and metallic ions (Ba^{2+} and Fe^{3+}) concentration on the particle size distribution and crystallinity of $\text{BaFe}_{12}\text{O}_{19}$. The correlation among the microstructure, the magneto-crystalline structure, and the magnetic properties of the barium hexaferrite powders thus prepared was examined as well. The microscopy investigations show a narrower grain size distribution and the average particle size is between 115 and 180 nm depending on the synthesis conditions. These sizes are well below the critical mono-domain diameter for barium hexaferrite (460 nm). We also measured the magnetization-versus-magnetic field at 4.2 K and room temperature. The coercive force is 4.3 kOe for the two temperatures. A saturation magnetization value (M_s) of 43 emu/g

was obtained from the magnetization curves in high magnetic fields up to 130 kOe. The magnetic properties may be attributed to the mono-domain structure.

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ZnO thin films for organic/inorganic heterojunctions

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Zinc oxide is a wide band gap II-VI semiconductor which is characterized by unique physical and chemical properties, which led to many applications of this material. Thin films of ZnO are used for the photo-generation of charge carriers in the solar cells. Appropriately doped ZnO may be transparent and conductive, and can therefore be used as a transparent electrode in the solar battery and microelectronics.

ZnO is also a prospective semiconductor candidate for making hybrid organic/inorganic devices. However, because of destruction of organic materials at high temperatures, these applications of ZnO require extremely low growth temperature. Our studies show that extra-low temperature ZnO growth is crucial to avoid formation of foreign phases and spinodal decomposition when transition metal atoms are introduced during the growth process.

The ZnO thin films were grown by Atomic Layer Deposition (ALD) method at extremely low temperature (60-120°C) with use of the very reactive metalorganic zinc precursor (diethylzinc, DEZn).

The obtained polycrystalline ZnO films are characterized by good crystallographic quality as confirmed by X-ray diffraction. Atomic Force Microscopy (AFM) study confirms good structural quality of these low temperature ZnO films. Root Mean Square (RMS) achieve 0.1 nm for film obtained for growth temperature 60°C.

The room temperature photoluminescence (PL) studies show strong emission of excitonic origin ("edge" PL) observed even for very low temperature grown ZnO. Our studies also show that ZnO thin films obtained at extremely low growth temperature are characterized by low free carrier concentration below $5 \times 10^{17} \text{ cm}^{-3}$ at room temperature.

Our results show that the ALD growth ZnO with DEZn precursor performed at extra-low temperature resulted in unexpectedly high quality material.

The work was supported by Polish grant SPUB No. 180/6.PR UE/2006/7 (6 Framework Programme – priorytet 2 (IST)) and European project with an acronym VERSATILE

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Advanced nanostructural zirconia ceramics for optical oxygen sensorsJanusz D. Fidelus¹, Witold Lojkowski¹, Donats Millers², Krisjanis Smits², Larisa Grigorjeva²**1.** Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland **2.** Institute of Solid State Physics, University of Latvia, 8 Kengaraga, Riga LV-1063, Latvia

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Our studies on ZrO₂ nanocrystals has shown that the intensity of luminescence is dependent on the oxygen partial pressure and that repeatability of the luminescence signal was within 6% [[1],[2]]. This result would be promising for the development of a solid state luminescent oxygen sensors which could be used for environmental monitoring, technological process control, monitoring of oxygen content. The possible advantages of the proposed optical oxygen sensors versus electrical and electrochemical sensors are presented.

The exchange of oxygen between nanocrystals and surrounding gasses strongly depends on the nanocrystals' properties. Therefore the methods of synthesis of selected advanced nanostructured zirconia powders have been studied and their density, specific surface area, morphology, phase composition and grain size distribution have been characterised. The luminescence of ZrO₂ nanocrystals at room and higher temperature is defect related and for its excitation an UV LED (Light Emitting Diode) can be used. The luminescence intensity dependence on oxygen partial pressure is suggested as being due to a change of defect concentration in the nanocrystal oxygen sub-lattice. The contribution from two kinds of luminescence excitation – direct excitation of the luminescence centre and its excitation via energy/charge transfer – will be shown. The comparison of luminescence from undoped and doped ZrO₂ nanocrystals under different excitation energy (within band gap and band-to-band) was used to determine the efficiency of each luminescence excitation. The model of the luminescence centre is proposed. We made an attempt to explain on the experimental basis why the luminescence intensity dependence on the partial pressure of oxygen.

The work supported in part by project NaMiC.

References:

- [1] Fidelus J. D., Lojkowski W., Millers D., K. Smits, Grigorjeva L., Piticescu R.R.: "Zirconia-based nanomaterials for oxygen sensor - generation, characterisation and optical properties", Solid State Phenomena Vol. 128 (2007) pp. 141-150, online at <http://www.scientific.net>.
- [2] Fidelus J. D., Millers D., Lojkowski W., Grigorjeva L., Smits K.: "Zirconia based nanomaterials for luminescent oxygen sensors", Proceedings of COE Conference, Zakopane 2006, przyjęty do druku w SPIE (The International Society of Optical Engineering).

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Optical and Spectroscopy of nanosized system on Si base after implantation and thermal treatment under enhanced hydrostatic pressureAndrzej Misiuk¹, M. Prujarczyk¹, Rostyslav V. Shalayev², Evgeny Shemchenko², Boris M. Efros², Anatoly Prudnikov², Alexander Yakovec²**1.** Institute of Electron Technology (ITE), al. Lotników 32/46, Warszawa 02-668, Poland **2.** National Academy of Sciences of Ukraine, A.Galkin Donetsk Institute for Physics & Technology (DonPTI NASU), Roza Luxemburg 72, Donetsk 83114, Ukraine

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Recent developments in the fields of integrated circuits, technology of CDs, and large volume information transmission resulted in still growing interest in optical and spectral properties of silicon - related materials, in particular produced basing on single crystalline Czochralski grown silicon, Cz - Si. Electronic states related to the presence of structural defects and impurities in Si:O,H,... - related materials [1] are still intensively investigated.

In the presented work, Si:O,H,... samples prepared by implantation technique, and processed at up to 1400 K (HT) under enhanced hydrostatic pressure in argon atmosphere (HP, up to 1.2 GPa) have been investigated. It has been shown that the HT - HP treatment of implanted samples leads to the formation of nano - clusters. The nano - sized structure formation in the samples results in essential changes of optical and spectral properties, being dependent on the HT - HP processing parameters.

The nature and mechanism of nano - sized structure formation in implanted single crystalline silicon, especially in Si:O and Si:H, subjected to complex processing [2, 3], and the effects of HT – HP treatment on spectral characteristics this materials are discussed. Usefulness of such materials for producing of sensors has been demonstrated.

1. A. Misiuk, B.M. Efros. Pressure-induced transformations during annealing of silicon implanted with oxygen: Physics and Technology High Pressure, 16 (2006) 49.
2. A.M. Prudnikov, A. Misiuk, I.E. Tyschenko, B.M. Efros. Solid state transitions investigations in DAC by spectroscopy methods: Defect and Diffusion Forum, 208-209 (2002) 315.
3. W. Wieteska, W. Wierzchowski, W. Graeff, A. Misiuk, A. Barcz, L. Bryja, V.P. Popov: Acta Phys. Polon. A, 102 (2002) 239.

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Study of undoped and doped zirconia nanocrystals luminescenceKrisjanis Smits¹, Donats Millers¹, Larisa Grigorjeva¹, Janusz D. Fidelus², Witold Lojkowski²**1.** Institute of Solid State Physics, University of Latvia, 8 Kengaraga, Riga LV-1063, Latvia **2.** Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland

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The study of time-resolved luminescence of undoped as well as europium and praseodymium activated zirconia free standing nanocrystals was carried out. The different excitation sources (e-beam, x-ray, lasers 4.66eV and 3.67eV, deuterium lamp) were used in experiments. The intrinsic defects related and activator luminescence bands were

observed. The luminescence dependence on activators concentration is shown. The excitations in zirconia are mobile, so with increasing of activator concentration the intrinsic defects related luminescence band intensity decreases and activator luminescence intensity increases until it reaches the saturation. The thermal treatment of ZrO₂ nanocrystals in vacuum resulted in the change of luminescence. It is proposed that the ZrO₂ nanocrystals recombinative luminescence intensity reduction is due to the oxygen loss from nanocrystals in vacuum. The relative intensities of luminescence bands due to transitions ⁵D₀ - ⁷F₁ and ⁵D₀ - ⁷F₂ of Eu³⁺ activated zirconia nanocrystals shows that significant fraction of Eu³⁺ is incorporated in low symmetry sites. The Eu³⁺ luminescence excited by a laser showed that this luminescence decay kinetics depends on both activator and intrinsic defects concentration in nanocrystals. The difference of intra-centre and recombinative luminescence was observed. The use of different excitation sources allow to determine role of direct excitation of luminescence centers, energy transfer by excitons to these centres and band carriers trapping involved in the creation of excited states of luminescence centres. The mechanisms of excited state creation and the possible models of luminescence centers in the nanocrystals will be discussed.

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Luminescence properties of zinc oxide nanopowders doped with Al ions obtained by the hydrothermal and vapour condensation methods.

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In this work we analyze the influence of Al ions doping on light emission efficiency and microstructure of zinc oxide nanopowders obtained by the hydrothermal and vapour condensation method. Vaporization of the precursor nanopowders was caused by high solar energy and subsequent deposition. The structure, crystallinity, luminescence properties were investigated using a range of techniques, including SEM, XRD, spectrofluorimeter and photomultiplier for the PL detection. We report an increase of lattice constant with increasing level of Al ions doping as well as large emission enhancement, which we relate to surface passivation of recombination mechanism and plasmon mechanism of photoluminescence stimulation.

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Fast luminescence in ZnO structures

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The results of the photo- and cathodoluminescence studies in ZnO nanopowders and ceramics were reported. The luminescence due to exciton annihilation was studied in undoped and Al, Co, Eu doped ZnO nanopowders obtained by sol-gel, microwave-driven hydrothermal, plasma synthesis methods and vaporization-condensation in Solar reactor. Luminescence depends on nanopowder morphology. The results of luminescence studies were compared with those obtained for commercial ZnO powder. The ZnO ceramics was sintered from different raw powders. The grain size of obtained ceramics was estimated from SEM images. The luminescence properties of ceramics obtained with different sintering parameters was studied. The decay times and light yield was analyzed. It is shown that fast scintillating ceramics will be prepared from nanostructured raw material.

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Luminescence properties of cerium doped Y₃Al₅O₁₂ nanopowders and nanoceramic

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Nanostructured Y₃Al₅O₁₂ (YAG) doped by various rare-earth ions is perspective raw material for transparent scintillating and/or laser ceramics. The nominally pure as well as cerium doped YAG nanocrystals (nanopowders) with the grain size ~20 nm and higher were obtained by co-precipitation methods. Translucent cerium doped YAG nanoceramic samples were sintered from nanopowders by means of high pressure and low temperature technique. Nominally pure and doped nanopowders and nanoceramics have been studied by means of time-resolved luminescence spectroscopy. Luminescence measurements were carried out using three different excitation sources: pulsed synchrotron radiation at the SUPERLUMI station at HASYLAB (DESY, Hamburg), the pulsed electron beam (270 keV, 10ns, 10¹² el/pulse), pulsed lasers (266 nm, 337 nm and 532 nm).

It was shown that nanoceramic's sintering processes have a strong influence on luminescence properties of the material. In fact it was detected that nanostructured cerium doped YAG ceramic has worse luminescence intensity comparing to luminescence intensity for the starting nanopowder. Decay kinetics of cerium related emission could be approximated by sum of two exponents (fast and slow) in all nanoobjects studied. It was shown that the intensities ratio of fast and slow components is significant parameter. This parameter shows a relative concentration of impurity ions on the nanoparticle's surface. The model proposed is based on the assumption that the distribution

of impurity ions is different in a single crystal, nanopowders and nanoceramics and it is suggested that quenching processes on grain borders of nanopowders and nanoceramics play a key role.

The comparison of luminescence properties for nano- and single-crystals shows that nanosized YAG may offer an advantage in respect to single crystal and further investigation and production of nanocrystalline YAG are perspective.

15:50	Poster	40

Optical properties of nanocrystalline YAG:Ce

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Due to its high quantum efficiency (QE) for luminescence, conventional coarse-grained YAG:Ce finds widespread use in light conversion and scintillator applications. Preparing YAG:Ce in nanocrystalline form may impart modified optical properties of advantage to technological applications, but the latter depend on highly efficient energy conversion. In this work, the effect of the particle size on quantum efficiency as well as on the optical lifetime of the YAG:Ce emission will be characterized and discussed.

Nanocrystalline YAG:Ce with an average particle size of 20 to 50 nm is synthesized by the chemical vapor reaction (CVR) method and subsequently analyzed by different techniques. Photoluminescence (PL) is used to investigate the relative quantum efficiency, emission properties and excitation behavior of different samples, while lifetime measurements of the luminescence intensity reveal information about the relaxation mechanisms of the excited Ce states. X-ray diffraction (XRD) is performed to examine the structure and grain size of the material. Furthermore, analysis of the chemical composition and doping concentration is performed by energy dispersive x-ray analysis (EDX) in a scanning electron microscope.

Comparing the nanocrystalline samples to a coarse-grained reference sample, we find a significant influence of the particle size on quantum efficiency. It turns out that the investigated nanocrystalline samples exhibit a lower QE at ambient temperature than the coarse-grained reference. The results of the optical lifetime measurements are discussed in relation to this reduction in QE.

15:50	Poster	41

Nanomaterials in dental applications

Malgorzata Lewandowska, Joanna Siejka-Kulczyk, Mariusz Andrzejczuk, Krzysztof J. Kurzydłowski

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Nowadays, nanopowders and nanocomposites reinforced with nanofillers are the most rapidly developing group of materials with excellent perspectives for application in a wide range of industry and medicine. The aim of the paper is to present several examples of nanomaterials developed at the Faculty of Materials Science and Engineering of Warsaw University of Technology which can be used as dental materials.

Ceramic-polymer composites are the most popular materials used for dental fillings. The conventional composites are reinforced with ceramic powders with an average particle size ranged from a half to tens micrometers. Beside unquestionable advantages of composite materials, their properties, such as polymerization shrinkage and

mechanical resistance, still need to be improved. The paper will discuss the influence of nanofiller addition on relevant properties of ceramic-polymer dental composites.

The other group of nanomaterials applied as dental materials is nanostructured yttria stabilized zirconium oxide ceramics. This ceramic is widely used for fabrication of crowns, bridges, inlays and other dental elements for which high strength, durability and esthetical appearance are required. The effect of nano-grain size of ceramic powders on the sintering parameters, microstructure and properties of zirconia ceramic will be discussed.

Finally, possibility to use of nano-titanium for the most loaded dental parts will be also presented.

15:50	Poster	42

Nanostructure formation in austenitic stainless steel

Agnieszka T. Krawczyńska, Malgorzata Lewandowska, Krzysztof J. Kurzydłowski

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Nano-metals are prospective materials for applications in a wide range of industries due to their properties which are significantly improved in comparison to the conventional counterparts. One of the ways of producing nanomaterials is hydrostatic extrusion (HE). This process, carried out either at low or high temperatures, allows for severe plastic deformation of metals at very high strain rates, which result in profound grain refinement down to the nano-meters scale. The aim of this work is to show the possibilities of grain size reduction by HE in an austenitic stainless steel.

Samples of 316L stainless steels were hydrostatically extruded, in a multi-step process with the total true strain of 1.84, and subsequently treated at different annealing temperatures. The changes taking place as a result of HE and annealing were observed using transmission electron microscope. Microhardness was also measured under the load of 200g.

The results show that the process of hydrostatic extrusion results in uniform microstructure consisting of nanotwins. The subsequent annealing at 600°C results in the partial transformation into nano-grained structure with the average grain size of 54 nm. At slightly higher temperature, at 700°C, a totally recrystallized uniform microstructure with nanograins of 68 nm in diameter is formed. It should be noted, that the temperature 700°C, is much lower than that for micro-grained samples of the same chemical composition.

The observed microstructural changes during the annealing have significant effect on mechanical properties. The microhardness after HE is 421 HV_{0.2}. It increases with the increase of the annealing temperature up to 484 at 500°C. Further temperature rise, up to 800°C, results in a drop of mechanical properties indicating a rapid grain growth.

15:50 Poster 43

Microstructure and mechanical properties of HE-treated titanium depending on its initial stateKrzysztof Topolski¹, Halina Garbacz¹, Wacław Pachla², Krzysztof J. Kurzydłowski¹**1.** *Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland***2.** *Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland**e-mail: ktopolski@materials.pl*

The study was concerned with examining the properties of titanium subjected to hydrostatic extrusion (HE) which is classified among the methods of severe plastic deformation (SPD). In our earlier studies we used hydro-extrusion for producing nano-crystalline titanium with the mechanical properties comparable with those of the binary Ti6Al4V titanium alloy [1-3]. The aim of the present work was to find how the microstructure of titanium in its initial state (prior to HE) would affect the effectiveness of the hydroextrusion process. The measure of this effectiveness was taken to be the disintegration degree of the microstructure and the mechanical properties of the titanium rods thus produced. The results obtained will permit optimizing the process of hydrostatic extrusion of titanium. The titanium rods examined had average grain sizes of 15µm and 160µm and differed in their mechanical properties. The rods were subjected to many-stage extrusion so as to achieve a true strain of 3.79. The mechanical properties of the materials were estimated by examining their microstructure and subjecting them to static tensile tests. The grain size was analyzed by scanning electron microscopy. It has been found that, in the materials examined, the microstructure of titanium in the initial state does not affect the course of the hydrostatic extrusion process. Irrespective of their initial microstructure, the HE-treated titanium rods had similar grain sizes of about 60nm and were characterized by similar mechanical properties.

1) Kurzydłowski K.J.: Materials Science Forum, 503-504 (2006) p.341-348. 2) Garbacz H., Lewandowska M., Pachla W., Kurzydłowski K.J.: Journal of Microscopy. Vol.223, Pt 3 September 2006, p.272-274. 3) Topolski K., Garbacz H., Pachla W., Kurzydłowski K.J.: Advances in Materials Science No.4 (2007)-in press.

15:50 Poster 44

Nanoscale nickel-free austenitic stainless steelMaciej Tuliński¹, Karolina Jurczyk², Mieczysław Jurczyk¹**1.** *Poznań Technical University, pl. Marii Skłodowskiej-Curie 5, Poznań 60-965, Poland* **2.** *Univ. of Medical Sciences in Poznań, Dept. of Conservative Dentistry, Poznań, Poland**e-mail: maciej.tulinski@doctorate.put.poznan.pl*

Recently, a new manufacturing process of nickel-free austenitic stainless steels with nitrogen absorption treatment has been developed [1]. In this method, small devices can be precisely machined in a ferritic phase and then during nitrogenization of their surfaces in nitrogen gas at temperature approx. 1200°C they become nickel-free austenitic stainless steels with better mechanical and corrosion resistance properties.

In this work, a nanocrystalline nickel-free stainless steels has been synthesized by the combination of mechanical alloying (MA), heat treatment and nitrogenation of elemental microcrystalline powders. The process parameters, morphology and EDX-analysis of obtained powders were determined. Phase transformation from ferritic to austenitic was confirmed by XRD analysis. Changes of size and shape of

the mechanically alloyed powder mixtures as a function of milling time was done by the SEM and AFM techniques. Human normal diploid fibroblasts were cultured onto each disk of nanostructured steels.

The microhardness of the final bulk material was studied using Vickers method. The result is almost two times greater than in austenitic steel obtained by conventional methods.

Mechanical alloying is a very effective technology to improve also the corrosion resistance of stainless steel. Decreasing the corrosion current density is a distinct advantage for prevention of ion release and it leads to better cytocompatibility.

According to existing conceptions, decreasing of material's crystallites size to nanometric scale allows to achieve much better mechanical properties (e.g. microhardness) compared to conventional materials [2]. With regard to austenitic stainless steels it could help to obtain better biomedical implants (e.g. stents) with better mechanical properties, corrosion resistance and biocompatibility.

[1] M. Sumita et al.: Materials Science and Engineering C 24 (2004) 753

[2] Edelstain A.S., Murday J.S., Rath B.B.: Prog. Mater. Sci., 42 (1997) 5

15:50 Poster 45

Microstructure and mechanical properties of 7475 aluminium alloy processed by SPD methodsKinga Wawer¹, Małgorzata Lewandowska¹, Arkadiusz K. Wieczorek², Michael J. Zehetbauer², Krzysztof J. Kurzydłowski¹**1.** *Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland***2.** *University of Vienna, Institute of Materials Physics, Wien, Austria**e-mail: kiniaac@o2.pl*

SPD techniques are capable of achieving ultrafine grain structures being the basis for outstanding mechanical properties. The aim of this work was to compare the efficiency of two different severe plastic deformation (SPD) techniques, i.e. hydrostatic extrusion (HE) and high pressure torsion (HPT), with respect to the microstructure and mechanical properties of 7475 aluminium being processed from two different initial conditions: (i) solution heat treated and (ii) solution heat treated and aged. It was found that SPD processing leads to a significant grain refinement down to nanometer scale. However, the kinetics of microstructure evolution depends on the initial microstructure and processing route. In the case of HE processing the samples deformed immediately after quenching exhibit higher mechanical strength whereas for HPT processing this is true for aged samples. The results are discussed in terms of the influence of both processing parameters and initial microstructure on mechanical properties of SPD processed 7475 aluminium alloy.

15:50 Poster 46

Influence of hydrostatic extrusion parameters on the microstructure and mechanical properties of 6082 aluminium alloyPaweł Widlicki, Piotr Wieciński, Halina Garbacz, Krzysztof J. Kurzydłowski*Warsaw University of Technology, Faculty of Materials Science and Engineering (InMat), Wołoska 141, Warszawa 02-507, Poland**e-mail: pwidlicki@inmat.pw.edu.pl*

Aluminium alloy 6082 combines a good toughness, excellent corrosion resistance with a very high strength. As a result it is widely used in structural applications in the building, marine and transport industries as well as for machined parts in the automotive industry. High strength of the 6082 alloy is achieved via: precipitation hardening, strain hardening and grain refinement which can be in economic way achieved by severe plastic deformation (SPD) methods, especially hydrostatic extrusion (HE) [1-4].

The aim of this work was to optimise the hydrostatic extrusion parameters in terms of their influence on the microstructure and mechanical properties of 6082 aluminium alloy. The investigations focused on the fabrication of nano- and ultrafine-grained form of 6082 aluminium alloy. In particular, multi-stage process was compared with single-stage process at the same cumulated true strain of 3.75.

Both multi- and single-stage extrusions resulted in profound grain refinement of 6082 aluminium alloy. The microstructure consisted of grains with 195 nm and 239 nm average size, respectively. The grain refinement induced significant improvement of mechanical properties. It should be noted that a slightly higher increase of the microhardness and tensile strength was observed for the multi-stage one.

Genetic algorithms for beginners

Welcome

Genetic Algorithms can be applied to solve global optimization tasks across a broad range of scientific disciplines. These methods mimic the evolutionary processes that occur in biological systems. The one-day workshop **Genetic Algorithms for Beginners**, is intended:

- to provide a basic course on genetic methods of global optimization aimed at those who are not yet familiar with these techniques and those who wish to systematize or extend their basic knowledge of these techniques, and

- to promote such methods among materials scientists.

Twelve lectures will be given on 16th September 2007, mainly by materials scientists, physicists, chemists and crystallographers (typically Symposium-G lecturers) who already have implemented such computational methods within their own fields. Some of the lectures will be directed towards the presentation of simple instructive examples of applications in materials sciences (catalysis, clusters, proteins, ...) in order to show the simplicity and power of the genetic algorithm approach.

Topics:

- explanation of basic ideas and concepts underlying the construction of genetic algorithms
- design and implementation of genetic algorithms
- genetic operators (mutation, selection, crossover, ...)
- variables: discrete or continuous ?
- coding methods
- parameter values: universal or problem-specific ?
- convergence criteria
- advantages of combined methods (hybrid methods, combination with neural networks, etc ...)
- multi-objective methods
- parallel computation
- ready-to-use software (in particular, to be presented by the software authors, suppliers or users)
- real implementations of genetic algorithms using open-source software
- materials scientist's views on the genetic approach
- simple examples of applications in materials science

Organisers

Wojciech Paszkowicz, *Institute of Physics, Polish Academy of Sciences, Warsaw, Poland*

Kenneth D.M. Harris, *School of Chemistry, Cardiff University, Wales*

PROGRAMME COMMITTEE:

Chairmen: Wojciech Paszkowicz (*Institute of Physics, Polish Academy of Sciences, Warsaw, Poland*), Kenneth D.M. Harris (*School of Chemistry, Cardiff University, Wales, UK*), members: Nirupam Chakraborti (*Indian Institute of Technology, Kharagpur, India*), Pierre Collet (*LSiIT Laboratory, University of Strasbourg, Strasbourg, France*), Roy L. Johnston (*School of Chemistry, University of Birmingham, Birmingham, UK*)

Programme

Sunday, 16 September

Opening

Sunday morning, 16 September, 10:15

Genetic algorithms: The method

Sunday morning, 16 September, 10:30

10:30

Invited

Genetic algorithms: Explanation of basic ideas (tutorial lecture part I)

Nirupam Chakraborti

Indian Institute of Technology, Kharagpur (IIT-KGP), Kharagpur 721302, India

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The proposed tutorial on Genetic Algorithms is designed to provide the basic exposure in Genetic Algorithms to graduate students and practicing engineers at large. The lectures will be decomposed into three sessions, and the organization for this session will be as follows:

Session 1 (The single objective approach)

Evolutionary Computation and its historical perspective. The American and German schools of thought. Simple Genetic Algorithms and its major operators: Reproduction, Crossover, Mutation and their variants. Genetic Algorithms for obtaining a sequence Mathematical Construction of Genetic Operators. Schema Theorem of John Holland. Variants of Binary Encoded Genetic Algorithms: Micro Genetic Algorithm, Messy Genetic Algorithm, Greedy Genetic Algorithm etc. Hamming Cliffs. Real Coded Genetic Algorithms. Differential Evolution. Elements of Evolutionary Strategies. Uni-modal vs. Multi-modal problems in Genetic Algorithms.

11:00

Invited

Evolutionary Algorithms: operators and parameters

Pierre Collet

University Louis Pasteur, Strasbourg, France

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This talk will mainly focus on genetic engine operators and parameters, that are essential in obtaining any good results on a particular problem. The No Free Lunch theorem will be evoked, to underline that off the shelf algorithms simply do not work.

An evolutionary algorithm must be finely tuned in order to solve a specific problem, so a selection of operators will be presented with their pros and cons, while also addressing beginner's questions concerning parameters settings.

11:30 Invited

Gentle but rigorous introduction to genetic algorithms or what makes them tick?

Marek W. Gutowski

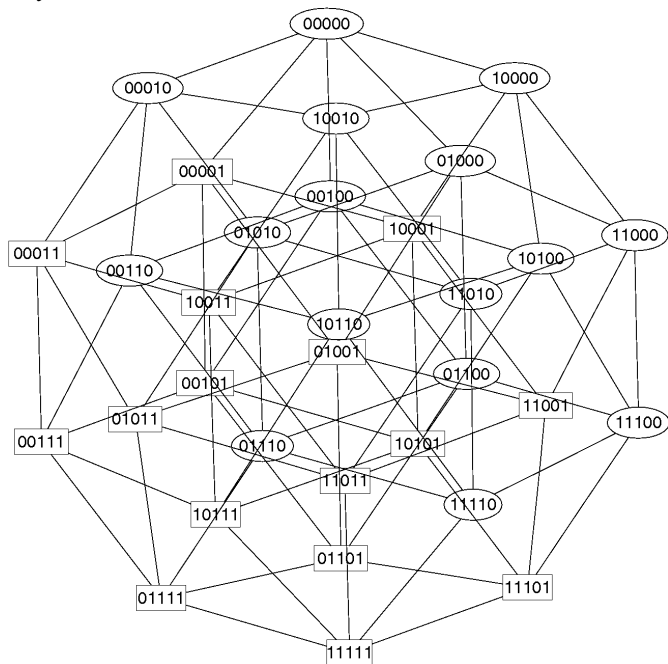
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The idea of genetic improvements of trial solutions seems quite natural and obvious. Yet, when it comes to translation of the original (optimization) problem into "genetic language", i.e. to practical realization - then the majority of potential users get stuck. In this introductory talk I am going to present not only the general idea of those algorithms but also some very simple (!) mathematics behind them. The purpose of my talk is twofold:

- showing that overwhelming majority of practical problems may be expressed in a unified way,
- pointing that the so called "tuning parameters", like population size, mutation ratio, and mating probability among others, need not to be problem-specific.

More precisely, only some of the tuning parameters are really important and their values should satisfy pretty simple *necessary* conditions (inequalities) in order to work correctly and efficiently. These conditions are completely unrelated to the specific problem under consideration. Small-world view of the complete space where 5-bit long chromosomes may live.



The figure accompanies the talk "Gentle but rigorous introduction to genetic algorithms" by Marek W. Gutowski.

Coffee

Sunday afternoon, 16 September, 12:00

Genetic algorithms: The method (I).

Sunday afternoon, 16 September, 12:15

12:15 Invited

Evolutionary algorithms: From design to implementation

El ghazali Talbi

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e-mail: talbi@lil.fr

In this talk, we will present the main search mechanisms in designing evolutionary algorithms: encodings, genetic operators, selection, replacement, diversification. We will tackle the hybridization aspect with other metaheuristics, the solving of multi-objective optimization problems and the design of parallel models for evolutionary algorithms.

We will also present a software framework to implement any evolutionary algorithm: the PARADISEO software (see <http://paradis-eo.gforge.inria.fr>).

12:45 Invited

Genetic algorithms: Multi-objective methods (tutorial lecture part II)

Nirupam Chakraborti

Indian Institute of Technology, Kharagpur (IIT-KGP), Kharagpur 721302, India

e-mail: nchakrab@iitkgp.ac.in

The proposed tutorial on Genetic Algorithms is designed to provide the basic exposure in Genetic Algorithms to graduate students and practicing engineers at large. The lectures will be decomposed into three sessions, and the organization of this session will be as follows:

Session 2 (The Multi-objective Approach)

The dominating and non-dominating solutions, Strong and weak optimality. Some recent Multi-objective algorithms. Distance Based Genetic Algorithms, Strength Pareto Evolutionary Algorithm, Pareto Converging Genetic Algorithms, Non-dominated Sorting Genetic Algorithm, Predator-Prey Algorithm, Generalized Differential Evolution.

Lunch

Sunday afternoon, 16 September, 13:15

Genetic algorithms: The method and its applications.

Sunday afternoon, 16 September, 14:30

14:30 Invited

Genetic Programming

Pierre Collet

University Louis Pasteur, Strasbourg, France

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After the previous presentations on genetic and evolutionary algorithms, this talk will present Genetic programming and its specificities. Once the basics are presented, typical GP problems will be addressed (such as bloat, or overfitting) and practical guidelines will be given that will help users design efficient GP algorithms.

15:00

Invited

The genetic algorithm-neural net combination (tutorial lecture part III)Nirupam Chakraborti*Indian Institute of Technology, Kharagpur (IIT-KGP), Kharagpur 721302, India**e-mail: nchakrab@iitkgp.ac.in*

The proposed tutorial on Genetic Algorithms is designed to provide the basic exposure in Genetic Algorithms to graduate students and practicing engineers at large. The lectures will be decomposed into three sessions, and the organization for this session will be as follows:

Session 3 (Combination of Genetic Algorithms and Neural Nets)

Preliminaries of Neural Networks. Training of neural Networks with Genetic Algorithms. Combination of Neural Networks, Genetic Algorithms and Gradient based methods. Evolving Neural Nets of flexible architecture. Neural Network evolution with single objective Genetic Algorithms, Neural Network evolution with Multi-objective Genetic Algorithms.

15:00

Invited

Application of genetic algorithms in the development of catalytic inorganic materialsManfred Baerns*Fritz-Haber-Institute of Max-Planck-Gesellschaft, Faradayweg 4-6, Berlin D-14195, Germany**e-mail: MBaerns@aol.com*

This workshop contribution deals with various aspects of catalyst discovery and development by high-throughput experimentation. First, a randomised first generation of catalytic materials of different compositions is prepared; the choice of chemical elements leading to the various compositions is based on fundamental knowledge and intuition as well as accounting for the unexpected. The first generation is then tested for catalytic performance. Mainly those materials that meet a set of objective functions, specifically activity, selectivity and/or yield for an individual catalytic reaction, are chosen for designing the second generation by applying a genetic algorithm (GA); the experimental procedure is then iteratively repeated until no further improvement of the objective functions is observed. In general, at least 5 to 10 generations each consisting of approximately 50 to 100 different catalytic materials are required for finding an optimal composition of the catalytic material.

By relating the output data of the objective function to the descriptors of the materials (i.e., composition and other properties) via an artificial neural network (ANN) insights in the relationships between catalytic performance and descriptors may be obtained. Furthermore, visualization of the results is facilitated.

Besides these basic principles in the application of a GA for catalytic-materials development also aspects of high-throughput experimentation are discussed, which are required for preparing and testing the large number of materials needed in the application of GA.

Coffee

Sunday afternoon, 16 September, 16:00

Inspirations and applications. Implementations and software.

Sunday afternoon, 16 September, 16:15

16:15

Invited

Global optimization and protein folding studiesJooyoung Lee*Korea Institute for Advanced Study (KIAS), 207-43 Cheongnyangni 2-dong, Dongdaemun-gu, Seoul 130-722, Korea, South**e-mail: jlee@kias.re.kr*

In this lecture, I will cover the basics of stochastic global optimization methods, such as simulated annealing, Monte Carlo with minimization, genetic algorithm, and generalized ensemble methods. The advantages and disadvantages of the existing methods will be discussed. The concept of annealing in the phase space (i.e., conformational space) is introduced to illustrate the philosophy/advantage of a new method, conformational space annealing (CSA). Examples of successful applications of CSA to various hard combinatorial optimization problems including traveling salesman problems, structural optimization of atomic clusters, model protein studies, and peptide/protein structure optimizations will be presented. The intrinsic difficulty of protein folding studies will be discussed in the light of the interplay between the accuracy of a score function and the difficulty of global optimization. Finally, a guideline to implement CSA method to outstanding problems of your interests will be provided. If time allowed, the parallel implementation of CSA will be also discussed.

16:45

Invited

Genetic algorithms for cluster optimization: general aspectsRoy L. Johnston*School of Chemistry, University of Birmingham, Edgbaston, Birmingham B152TT, United Kingdom**e-mail: r.l.johnston@bham.ac.uk*

In this lecture, I will start by giving a short overview of the field of cluster science and the reasons for using genetic algorithms for global optimization in this field. I will then present a brief history of the application of genetic algorithms to various types of clusters. Finally, I will describe the development of our own genetic algorithm (the Birmingham Cluster Genetic Algorithm) for cluster geometry optimization - giving examples of its application to a variety of different types of clusters.

During the lecture, I will emphasize how the nature of the problem or system being studied influences our choice of genetic algorithm methodology and operators and I will introduce a number of ways in which the basic genetic algorithm can be improved - for example by developing hybrid search methodologies to enable local as well as global searching. I will also talk about potential pitfalls and possible future directions.

17:15

Invited

Implement your Genetic Algorithms with a mouse

David Farrusseng

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The objective of the tutorial consists at the implementation of Evolutionary Algorithms in the frame of material screening. It will include:

- material encoding for Genetic Algorithms and Evolutionary approach
- implementation of evolutionary algorithm workflows
- design algorithms with appropriate diversity/selective pressure ratio

Case studies from literature will be presented and used for training purposes. The training will be carried out on the open source software “OptiCat”. Attendees can download OptiCat prior to the workshop at <http://a.farrusseng.free.fr/>

17:45

Invited

Hands-on guide for breeding structures using GULP

Scott M. Woodley

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General Utilities Lattice Program, GULP, is a program for performing a variety of types of simulation on materials using boundary conditions of 0-D (molecules and clusters), 1-D (polymers), 2-D (surfaces, slabs and grain boundaries), or 3-D (periodic solids). The focus of the code is on analytical solutions, through the use of lattice dynamics, where possible, rather than on molecular dynamics. A variety of force fields can be used within GULP spanning the shell model for ionic materials, molecular mechanics for organic systems, the embedded atom model for metals and the reactive REBO potential for hydrocarbons. Analytic derivatives are included up to at least second order for most force fields, and to third order for many.

Here I will briefly explain the methodology implemented within the evolutionary module of GULP and show example input files when GULP is used to predict the atomic structures adopted by dense or microporous, ionic compounds.

The latest version of the code and scientific details about GULP can be found at <https://www.ivec.org/gulp>.

COST Action D30 Working Group Meeting

20 and 21 Sept. in conjunction with Special Acta Materialia Gold Medal Symposium Perspectives of Nanotechnology. Further information will appear soon.

Programme

Unscheduled abstracts

Author alphabetical order

Oral

Stability, instability, metastability and grain size in nanocrystalline ceramic oxide systems

Giora Kimmel

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The following nanocrystalline binary oxide systems were studied: Mg-Ti, Zn-Ti, Fe-Al, Fe-Ti, Ni-Ti, Mg-Ti, Zr-Al, Zr-Mg and Y-Ln (Ln = rare earth element). Some pure or doped unary oxides were also studied. The xerogels were heated at constant T (200 to 1600 °C) for 3 to 6 hours. There was a threshold T for oxide formation and in many cases the products were metastable nanocrystalline phases, depending on grain size and composition, including doping. The oxide phases of Ni-Ti, Mg-Ti, Zr-Al, and Ln-Y formed at T < 900 °C are different from those formed at high T. New ranges of solid solution and formation of higher temperature structures were found. A transition phase can be defined as a structure formed at relative low T, irreversibly transforming higher T into an equilibrium phase of the same elemental composition. Some low T transition phases have a structure similar to that of a high T equilibrium phase, e.g., (equilibrium phase given in parentheses) tetragonal ZrO₂ (monoclinic) and low-T qandilite-like solid solutions (qandilite + geikielite). Others are unique with no representation in the equilibrium phase diagram, e.g., gamma-like aluminas (corundum) and anatase (rutile), which are formed as nanocrystalline oxides due to small growth rate caused either by low-T of calcination or due to additives. To assess the importance of crystal size in the stabilization of transition phases, we conducted the following studies: (a) XRPD analysis of all unary, doped and binary compositions; (b) evolution of transition phases in HT XRPD of the Mg titanates; (c) at temperatures where mixtures of transition and equilibrium phases were found, phase evolution was studied with time; (d) the retention of pure tetragonal ZrO₂ on quenching Al-Zr oxides after calcinations at high T; (e) additional evidence from HRTEM, SEM and DTA experiments is also being collected. A model correlating the size effect with the unusual phases and structures is proposed.

Oral

High pressure-low temperature synthesis of new nanostructured compounds

Roxana M. Piticescu

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Mild thermodynamic conditions (in terms of pressure and temperature parameters) specific to hydrothermal or solvothermal reactions are able to initiate through chemical bonding directly from solutions new developments in inorganic and molecular chemistry. The enhanced reactivity at the solution/nuclei or inorganic/organic interfaces is one way able to improve chemical bonding between different species. Such chemical bonds can be used to produce in soft conditions nanostructured oxide compounds or hybrid inorganic/organic molecules. More over, the addition of external electric field is able to produce oriented grain growth deposition of thin films starting from these nanocompounds.

The paper presents the opportunities offered by the methods developed by our research group in obtaining different new materials. Examples presented are doped nanocrystalline zinc oxide – maleic acid copolymers with controlled nanostructure and electronic properties, hydroxyl apatite – polysaccharides and layered silicates-polymer compounds - calcium phosphate-polymer hybrids for tissue engineering and regenerative medicine.

Acknowledgment: Romanian Agency for Scientific Research for financial support in the frame of Research for Excellence contracts 46-ReteBdent, 69-SINAPS and 16-TECOREMED and COST D30 – High pressure Tuning of Materials.

Oral

Microwave technique applied on the hydrothermal synthesis and sintering of calcia stabilized zirconia nanoparticles

Antonino Rizzuti

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Calcia as well as di- or tri-valent oxides stabilized zirconia have received the most attention because of their high oxygen-ion conductivity which combined to their high thermochemical stability makes them as the best candidate for electrochemical devices, such as oxygen sensors, solid oxide fuel cells and oxygen pumps which operate at high temperatures. Main preparation methods of these compounds consist of solid state reaction and also sol-gel route, which require drastic or complex conditions. Hydrothermal treatment appears to be the more simple and less expensive method for the preparation of zirconia nanopowders characterised by high purity and high degree of compositional homogeneity which ensure an appropriate sinterability. The hydrothermal synthesis of calcia zirconia powders has been reported in few papers, never mentioning the possibility to have a microwave-assisted technique. This work is focused on synthesis zirconia nanopowders stabilized by 6 mol% calcium oxide prepared under hydrothermal conditions in combination, for the first time, with microwave technology. Microwave technology is, as diffusely reported, an advantageous tool for the efficient obtainment of inorganic oxides nanoparticles. Hydrolysis of calcium and zirconium nitrates followed by microwave irradiation to reach 200°C for 90 minutes was sufficient to obtain calcia stabilized

zirconia as white powders. XRD analysis has evidenced the formation of nanocrystallites of tetragonal symmetry as the main phase with a size of ca 8 nm. The sintering of these nanopowders was performed under microwave irradiation on the sample in the form of a pressed pellet which contains polyvinyl alcohol as binder at a temperature of 1300°C in air. SEM has confirmed that the sample was successfully sintered and the grains have maintained their nanometric size very narrowly dispersed, ranged between 100 and 150 nm.

List of Participants

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