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ABSTRACTS

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POSTERS

Monday 20 July 2015

Plenary Lecture 1

Abstract

Paper Ref: 335

NEUTRON SCATTERING FOR BATTERY MATERIALS

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Batteries are a key technology in today's society. They are used to power electric and hybrid electric vehicles and to store wind and solar energy in smart grids. Since the "lithium-ion" configuration has been widely accepted, significant efforts have been devoted to attain high energy and power densities to produce an excellent energy storage system without any safety issues. To improve battery characteristics, deep insights into the structure of the materials during the battery reactions are necessary. Neutron scattering clarifies a wide range of structures for battery materials; from local to long range structures, and these structure characteristics are related to the battery properties. New materials with high electrochemical properties are necessary to improve future battery systems. Structure and property relationships for the battery electrodes and electrolytes are important information for designing new energy storage systems. An example of new materials is solid electrolytes. An all solid-state configuration is the most promising for future devices to improve the reliability of batteries. Lithium superionic conductors, which can be used as solid electrolytes, promise the potential to replace organic liquid electrolytes and thereby improve the safety of batteries. The material, $Li_{10}GeP_2S_{12}$ shows high ionic conductivity, which exceeds the conductivity value of liquid electrolyte. Neutron scattering is one of the best methods to provide information of structure containing lithium and conduction mechanism determined by neutron scattering makes the materials design concept clear. In-situ and operando experimental techniques are another important subjects for clarifying battery reactions. An in situ technique for directly observing surface structural changes has been developed that employs thin-film model electrodes and surface X-ray and neutron scattering techniques. The surface structural changes commence with the formation of an electrical double layer, which is followed by surface reconstruction in the charge-discharge process. The structure information obtained by synchrotron X-ray diffraction and neutron reflectometry methods provides insight into the surface reaction, nano-effect of the electrode reaction, and degradation mechanism of battery reaction.

BST-1 Membranes

Abstract

Paper Ref: 189

RECONSTITUTION OF A NANOMACHINE DRIVING THE ASSEMBLY OF PROTEINS INTO BACTERIAL OUTER MEMBRANES

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Over 9.5 million people die each year due to infectious diseases caused by pathogens. Many species of pathogenic bacteria require nanomachines acting like a molecular pump that shuttle key disease-causing molecules (proteins) from inside bacteria cells to the outside surface, priming the bacteria for infections. How such proteins are assembled remains an important question in biology. If we can inhibit the nanomachines function in transporting specific violence factors, it would disable the disease process. Therefore it is crucial to understand how the proteins are transported through the nanomachines from the periplasm to the extracellular space.

Measuring the activity of the component parts of membrane-embedded nanomachines in solution is a major technological challenge.

The translocation assembly module (the TAM) is a nanomachine required for virulence of bacterial pathogens. We have reconstituted a membrane containing the TAM onto a gold surface for characterization by Quartz Crystal Microbalance with Dissipation (QCM-D) and Magnetic Contrast Neutron Reflectrometry (MCNR). We show that dynamic movements within the TamA component of the TAM are initiated in the presence of a substrate protein, Ag43, and that these movements recapitulate an initial stage in membrane protein assembly. The reconstituted system provides a powerful new means to study molecular movements in biological membranes, and the technology is widely applicable to studying the dynamics of diverse cellular nanomachines.

Abstract

Paper Ref: 66

Membrane Binding of Prion Protein N-terminal Peptides Characterised by Neutron Reflectometry

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The prion protein (PrP) is widely recognised to mis-fold into the causative agent of the transmissible spongiform encephalopathies, known as Creutzfeldt–Jakob disease (CJD) in humans, scrappie in sheep or Bovine spongiform encephalopathy in cows (BSE, “mad cow disease”). PrP has previously been shown to bind to lipid membranes with binding influenced by both membrane composition and pH. Aside from the mis-folding events associated with prion pathogenesis, PrP can undergo various post-translational modifications, including internal cleavage events. Alpha and beta-cleavage of PrP produces two N-terminal fragments, N1 and N2 respectively, which interact specifically with negatively charged phospholipids at low pH. Previous work probing N1 and N2 interactions with supported bilayers raised the possibility that the peptides could insert deeply with minimal disruption [1]. This work aimed to refine the binding parameters of these peptides with lipid bilayers. To this end, neutron reflectometry was used to define the structural details of the interactions in combination with quartz crystal microbalance interrogation and calcein release assays [2]. Neutron reflectometry confirmed that peptides equivalent to N1 and N2 insert into the interstitial space between the phospholipid headgroups but do not penetrate into the acyl tail region. In accord with previous studies, interactions were stronger for the N1 fragment than for the N2, with more peptide bound per lipid. Neutron reflectometry analysis also detected lengthening of the lipid acyl tails, with a concurrent decrease in lipid area. Overall, the data shows that the N1 and N2 peptides interact with the anionic phospholipid headgroups of supported lipid bilayers, inducing lipid ordering in the absence of significant penetration into the acyl tails or permeation of the membrane.

[1] M. P. Boland et al., *J. Biol. Chem.* **285,** 32282 (2010).
[2] A. P. Le Brun et al., *Biophys. J.* **104,** 2313 (2014).

Abstract

Paper Ref: 164

Effects of Lipid Packing on Lipid Flip-Flop Phenomena: A Neutron Reflectivity Study

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The transbilayer movement of phospholipids in cellular membranes, also known as lipid flip-flop or translocation, is an area of significant biological importance. Even though lipid flip-flop has been studied for decades, it is still far from being understood and well characterized for natural cell membranes as well as for simpler bilayer model systems. The packing state of lipid in real system, which is a consequence of multiple factors such as spontaneous curvature of lipid itself, negative charges on headgroup, and protein interaction, is potential to cause defect sites within membrane. These defects would be membrane channels which regulate many biological functions, including of lipid translocation. Since the flip-flop is a slow dynamics process, Neutron reflectivity is suitable tool to monitor the lipid composition on each layer as a function of time. We would like to investigate the effect of unsaturated lipids as 1) how the dynamics of lipid diffusion process is controlled by the membrane packing, and as 2) whether flip-flop can be induced from only one side of the membrane bilayer. By mean of Langmuir-Blodgett/Schaefer deposition methods, it is possible to prepare successive adsorbed layers resulting in a final asymmetric composition. These asymmetric bilayers were characterized by X-ray reflectivity to obtain the information of bilayer quality, complementary with Neutron reflectivity to investigate the absolute composition and the relative location of lipid molecules within the bilayer and at its interfaces. The measurement has been performed by newly developed HANARO bio-reflectometer at Korea Atomic Energy Research Institute, Korea.

Abstract

Paper Ref: 102

THE EFFECT OF BIAS POTENTIAL ON TETHERED LIPID BILAYERS

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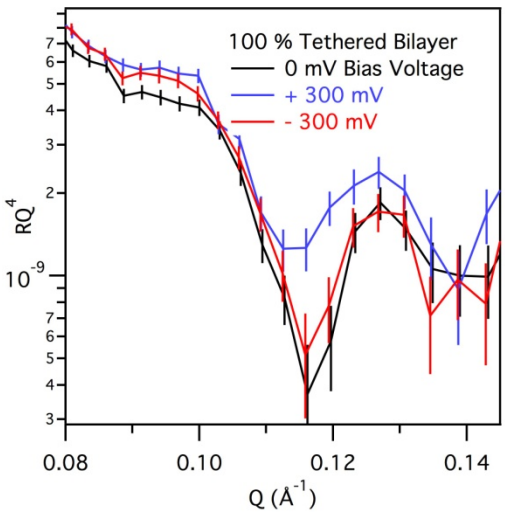
Tethered lipid bilayer membranes (tBLMs) are membranes chemically anchored to solid substrates typically employing sulphur-gold chemistry. Such membranes represent stable, convenient models of complex biological membranes and are particularly suited to physical techniques such as neutron scattering and electrical impedance spectroscopy (EIS) [1]. We have devised a tBLM architecture comprising a benzyl disulphide – O11 ethylene glycol – C20 phytanyl tether which when mixed in various ratios with a benzyl disulphide O4 – OH spacers provides a sparsely tethered tBLM possessing a hydrophilic volume between a subsequently formed lipid bilayer membrane and the tethering gold electrode surface. A critical feature of this architecture is the spacing of this layer, and the volume of water stored therein. A bias potential may be applied very simply through the application of voltage gradients between the conductive supportive gold surface and a return electrode within the bathing solution. This permits control over the potential gradients across membranes and the determination of the transport properties of membrane incorporated biological ion channels and carriers. For example the ion channel conductance (gramicidin-A in this case) may be altered by an order of magnitude in tBLMS by the application of bias voltage. This suggests the selective sequestration of ions into the reservoir space.



We have commenced simultaneous Neutron Reflection and EIS studies of the impact of bias voltage upon the properties of tBLMs where we have varied the density of the tethering layer and examined the impact of 0 & ± 300 mV upon the position of the bilayer relative to the tethering substrate. The figure shows data (March 2015) for a fully tethered bilayer. There is a small, but clear impact of bias potential on the dataset, which correlates with the EIS data. The data was collected at 0, then + 300 mV followed by a return to – 300 mV. Analysis of data and from this and other tethering density tBLMs has commenced and will be presented.

1. C.G. Cranfield, *et al.*, Langmuir, 2015, **31**, 292-298.

MSC-1 Reaction kinetics and disorder

Abstract

Paper Ref: 458

Neutron Diffraction Studies of Kinetic Systems

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The study of reaction kinetics using neutron diffraction emerged from humble beginnings when, during the 1930's and 1940's, Debye-Scherrer X-ray cameras were first fitted with heating stages. Neutron diffraction soon followed this trend in the early 1950's. Other early *in situ* environments included Isostatic Pressure, Magnetic Field and Cryogenic Temperatures. The interest in those early years was the discovery of new crystal and magnetic structures and time dependent phenomena were viewed as a great nuisance to be avoided. Over the intervening decades, large increases in available neutron (and X-ray) flux and improvements in sample environments has enabled a broadening of scope to include systems undergoing gross physical and chemical changes.

The presenter's use of *in situ* neutron diffraction experiments to study a wide range of dynamic processes with slow, moderate and rapid kinetics will be discussed. This will include early studies of *physical* kinetics in which it was demonstrated that composition and thermal history differences make different zirconia ceramics show room temperature plasticity and creep due to quite different mechanisms (ferroelasticity, Martensitic transitions and micro-cracking). The work then progresses into *chemical* or *microstructural* kinetic systems during synthesis or simulated service. At slow to moderate reaction rates, examples will be given which illustrate how a carefully planned *in situ* study can reveal transient intermediate phases during metal hydride formation and during the reactive sintering of MAX phase ceramics. Not only are new phases revealed, their role in the reaction sequence may be observed. This will be extended to a discussion of very rapid *in situ* neutron diffraction studies of combustion synthesis and spontaneous exchange reactions in which intermediate phases which last as little as a few seconds can be observed.

Emphasis will be placed not only on the ability of *in situ* experiments to reveal new mechanisms, but the rich diversity of additional data that can be extracted. Methods will be illustrated by which the *temperature* of the sample, *enthalpy of formation* of precipitating phases, *activation energy* for crystal growth and a range of microstructural parameters can be obtained.

Abstract

Paper Ref: 91

Hydration modes and mechanisms in the layered perovskite $K_{2.5}Bi_{2.5}Ti_4O_{13}\cdot H_2O$

Samuel Liu[1], Max Avdeev[2], Mark R. Johnson[3], Richard Mole[2], Andrew Studer[2], and Chris D. Ling[1]

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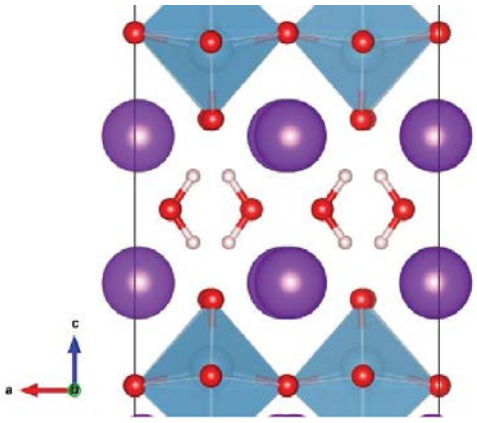
The chemical and structural flexibility of the perovskite structure, ABO_3, which has resulted in its ubiquity and application in a range of technologies, extends to layered variants such as the Ruddlesden-Popper (RP) phase. The quasi-low dimensionality of layered phases introduces a new avenue of research in creating novel multifunctional materials. A new 4-layered RP phase $K_2[Bi_{2.5}K_{0.5}Ti_4O_{13}]$ is of interest because the literature contains relatively few examples of well-ordered n = 4 layered phases.

X-ray diffraction patterns for $K_2[Bi_{2.5}K_{0.5}Ti_4O_{13}]$ change rapidly in humid atmospheres, in a manner consistent with expansion of the layer-stacking c-axis. Rietveld-refinements against neutron powder diffraction data show the incorporation of H_2O in between perovskite-type layers. However, despite significant differences between the NPD patterns of hydrated and dehydrated phases, a solution for the room-temperature hydrated structure has not been forthcoming.



The hydrated structure involves significant local disorder at room temperature, which is difficult to model crystallographically. Difference Fourier maps of refined models against low-temperature (100 K) NPD data suggest the structure shown in the figure, with intact H_2O molecules “bridging” the perovskite layers through the rock-salt interlayer gap. This model was independently supported by *ab initio* geometry optimisation calculations. Dynamic calculations suggest that these water molecules remain intact but have high mobility with increasing temperature, rotating about the layer-stacking axis.

The temperature dependence of the rotations of water were measured using quasi-elastic neutron scattering (QENS), in which we observed an increased broadening of the elastic peak as the sample approached dehydration. The temperature-dependence of the QENS signal is compared to the results of molecular dynamics calculations.

Abstract

Paper Ref: 109

A combined computational and experimental approach to understanding conduction mechanisms in apatite-type $Nd_{9.33}Si_6O_{26}$

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Materials that exhibit significant mobility of different types of charge carriers (oxide ions, protons, electrons) have a range of potential applications including solid-oxide fuel-cell membranes, electrodes, batteries and sensors. A thorough understanding of the fundamental atomic-scale mechanisms of the conduction processes in these materials is necessary to identify ways in which their local chemistry and structure can be modified to lower activation barriers and optimize pathways for conduction.

Apatite-type $Nd_{9.33}Si_6O_{26}$ is an interesting initial target system for developing this understanding by virtue of its good oxide-ionic conductivity, simple chemical compositions and scope for chemical and structural modification. $Nd_{9.33}Si_6O_{26}$ is a hexagonal apatite structure in space group $P6_3/m$ (two crystallographically distinct Nd sites) with 6.7% Nd vacancies located at the 4f site only. A thorough analysis of different arrangements of Nd vacancy positions has been performed to obtain a suitable input model for *ab initio* molecular dynamics (MD) calculations. Possible arrangements have been classified by the corresponding sum of distances between the vacancies to quantify the degree of distribution, identify energetically favored arrangements and investigate the influence of different vacancy distributions on subsequent calculations.

Here, we present the results of inelastic and quasi-elastic neutron scattering experiments at various temperatures to probe structural fluctuations that may trigger or facilitate the diffusion process. The obtained data have been verified against and interpreted with the aid of *ab initio* MD simulations to construct a detailed picture of oxide motion in $Nd_{9.33}Si_6O_{26}$. We are now using these results to develop and rigorously verify classical force fields for empirical calculations that will extend the simulations to timescales required to observe actual conduction processes. The results suggest ways in which the local chemistry and structure of materials can be modified to lower activation barriers and optimize the pathways for ionic conduction.

Abstract

Paper Ref: 230

STUDY OF NEODYMIUM OXIDE INCLUSION ON THE STRUCTURE OF A BORATE GLASS

Mr. Wilson Vaz [1], **Professor Erwin Desa [1]**, Dr. Siva Krishna [2], Mr. A B Shinde [2]

The structure of borate glass has been well studied by many different authors using a variety of techniques (1) . When other compounds are added to the glass which accommodates them, the structural environment of the inclusions will depend on the type of included elements. Given the large number of applications of borate glasses, a study of the factors that determine the surrounding structure of an included ion in a glass is thus of much relevance.

As a first step in such studies, a single ion-type viz. neodymium oxide (Nd_2O_3) was incorporated in a borate host glass at varying mole percentages of the dopant from 10% to 25%. These glasses were melt-quenched in air from 1450 ^oC. Transparent bubble-free purple glass beads were obtained and subsequently crushed into fine powders for the X-ray and neutron diffraction measurements. The glass transition temperature was approximately 750 ^oC .

An incident wavelength of 1.54 Å (CuK_α) was used in the X-ray diffraction studies. Neutron diffraction measurements were made on the High Q diffractometer at Dhruva Reactor, B.A.R.C., Mumbai in the Q range of 0.3 to 15 $Å^{-1}$. The diffraction patterns from both techniques showed some trends with concentration in the higher Q regions of S(Q). The real space correlation functions viz. T(r) and number density function N(r) gave clear indications of changes of nearest-neighbour coordination number of O around B (at 1.366 Å) as a function of Nd_2O_3 molar concentration in the glass. Thus in the first peak, the fraction due to tetrahedral BO_4 units increases from 28% to 41% with dopant level at the expense of BO_3 triangles. From an analysis of the second peak in T(r), super-structural units such as diborate and di-pentaborate units surround the Nd ion at lower dopant levels while the coordination is octahedral at higher levels of dopant. These findings are in keeping with a model that the overall structure of the host glass is affected by the concentration of the dopant ion.

1. Hannon A.C., Grimly D.I., Hulme R.A., Wright A.C., Sinclair R.N., J. Non-Cryst. Solids, 177 (1994) 299-316

Abstract

Paper Ref: 179

In-situ neutron scattering study of crystallization behaviour in ternary bulk metallic glasses and its correlation to glass forming ability

Si Lan[1], Xiaoya Wei[1], Xuelian Wu[1], Jie Zhou[2], Zhaoping Lu[2], Mikhail Feygenson[3], Jörg Neuefeind[3], Xun-Li Wang[1]

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Bulk metallic glasses (BMGs) are complex structural materials, which have potential in a variety of applications [1]. To determine the nature of excellent glass-forming ability (GFA), it is desirable to investigate crystallization kinetics [2]. Although considerable amount of works have been reported, most of them were carried out on multicomponent alloys with complex chemistry [3]. In order to access the physics of crystallization, it is essential to simplify the chemistry. In this paper we report a time-resolved neutron diffraction study of crystallization kinetics in five ternary BMGs, $Zr_{56}Cu_{36}Al_8$, $Zr_{54}Cu_{38}Al_8$, $Zr_{48}Cu_{45}Al_7$, $Zr_{45}Cu_{49}Al_6$ and $Zr_{46}Cu_{46}Al_8$, with gradually enhanced GFA. The measurements were carried out using NOMAD at the Spallation Neutron Source, Oak Ridge National Laboratory (ORNL). The structural evolution in short- and medium- range order (SRO and MRO) during the amorphous-to-crystalline transformation for these BMGs has been well resolved with a time-resolution of ~ 2 minute. According to the property of their crystalline products and their crystallization pathways, these alloys can be divided into two types: $Cu_{10}Zr_7$-type and Zr_2Cu-type BMGs. For Zr_2Cu-type BMGs, i.e. $Zr_{56}Cu_{36}Al_8$ and $Zr_{54}Cu_{38}Al_8$, there is just one crystallization temperature (e.g. ~751 K for $Zr_{56}Cu_{36}Al_8$) on their Differential Scanning Calorimetry (DSC) curves, and total neutron diffraction patterns show highly-ordered crystalline phase after divitrification. For $Cu_{10}Zr_7$-type BMGs, i.e. $Zr_{48}Cu_{45}Al_7$, $Zr_{45}Cu_{49}Al_6$ and $Zr_{46}Cu_{46}Al_8$, two crystallization temperatures (e.g. ~ 776 K and ~ 778 K for $Zr_{46}Cu_{46}Al_8$) were identified, much like that in $Zr_{52.5}Cu_{17.9}Ni_{14.6}Al_{10}Ti_5$ BMG (BAM-11) [3], an excellent glass former. The crystalline phase of devitrified $Cu_{10}Zr_7$-type BMGs, with better GFA, is poorly ordered, also similar to BAM-11. These results suggest that the unique crystallization pathway for $Cu_{10}Zr_7$-type BMGs would be a distinctive feature of alloys with excellent GFA.

[1] M. W. Chen. A brief overview of bulk metallic glasses. *NPG Asia Mater* 3: 82-90, 2011.
[2] C. Tang, and P. Harrowell. Anomalously slow crystal growth of the glass-forming alloy CuZr. *Nature materials* 12: 507-511, 2013.
[3] X. L. Wang, J. Almer, C. T. Liu, et al.. In situ synchrotron study of phase transformation behaviors in bulk metallic glass by simultaneous diffraction and small angle scattering. *Physical Review Letters* 91: 265501, 2003.

SMT-1
Compact sources, computation and deuteration

Abstract

Paper Ref: 347

RIKEN compact neutron source RANS and its application with iron and steel field

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RIKEN accelerator-driven compact neutron source (RANS) has operated since 2013 and has been proved its higher potential to be used on-site. RANS is one of the first compact neutron sources which is developed especially for such practical use as industrial use for development in the field of manufacturing, as non-destructive inspections on-site. The success of the visualization of the corrosion with wet-dry process under the film in the steel and alloy samples with RANS has clarified high potential of compact neutron source for metal and steel samples. The 3D distribution of the water and the corrosion in the steel under the film was obtained. The texture change of the steel before and after deformation was observed by the neutron diffraction experiment with RANS using TOF measurements with 2D detector, and compared with X-ray diffraction results. Large area such as $1m^2$ fast neutron detector with 1024-channel has been developed. The inside of small part of real removed bridge is observed with using its smaller version of 64-channel with the fast neutron from RANS. The one of the most important mission of our compact neutron source project is to realize the non-destructive inspection system for large scale infrastructures such as bridges. The difference of the number of the steel bars in the thick concrete slab was also detected with fast neutron. The transportable compact neutron source with the large area detector and the health diagnostic system of the infrastructure is now being developed. RANS is now moved to new building. Its development plan will be also discussed.

Abstract

Paper Ref: 373

Hokkaido University Neutron Source, HUNS, an ancient machine carving out new trend of compact accelerator driven neutron source

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We need advanced large facilities like J-PARC and JRR-3 in Japan, HANARO in Korea, OPAL in Australia and CARR in China, but also we also need a new way of feeding novel ideas for neutron instrumentation, devices, research and also researchers. Of source we can carry out such things at large facilities, but also, it is much better to have neutron sources near users who need neutrons in university, institute and industrial environments. We believe that compact accelerator driven neutron sources (CANS) based on a small-scale accelerator should be main players in this aspect and we are pretty much sure that it will change the way of conducting research and the neutron community as a whole. Number of researcher must increase far more than we are anticipating now; main users of such CANSs may not be experts in neutron field, meaning, they would use other techniques for their research and will develop new culture of using neutrons.

Hokkaido University Neutron Source, HUNS, is one of the oldest pulsed neutron sources based on a small-scale electron linac began operation in 1974 in its full energy. The linac is a 45 MeV-1 kW one operating at 50 Hz. Cold neutron sources for accelerator driven neutron sources have been developed using HUNS. It has also been a cradle of novel neutron instruments devices and detectors; the quasi-elastic neutron instrument (LANDAM) for pulsed neutron was first developed; sextuple neutron focusing magnet was first tested; a time-of-flight type focusing mirror SANS instrument, to name a few.

We are developing new ways of using CANS at HUNS by developing novel instruments or methods that have not been practical at all. We would like to show a few examples of the results obtained at HUNS, such as nanoscopic structure studies of steel and other metals using small and medium-angle scattering instrument, iANS, focusing mirror development. We would also like to talk about our accelerator and target stations upgrade plan and also an idea to improve the Bragg-edge transmission measurements at HUNS. We are aiming to develop the HUNS upgrade as the model case of CANS in future.

Abstract

Paper Ref: 216

Resolution Kernels in Time-of-flight Neutron Reflectometers

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Instrumental resolution functions must be taken into account when analysing Neutron Reflectometry data. These resolution functions are obtained by convolving the resolution kernels for the angular and wavelength components.

In the past most data reduction and analysis processes have assumed that the overall resolution kernel is Gaussian in shape. Whilst this is the case for monochromatic reflectometers this is not true for those Time-Of-Flight (TOF) Neutron Reflectometers, such as *Platypus*[1] which possess rectangular wavelength resolution distributions (a consequence of the disc chopper systems used). In such situations the resolution function is typically trapezoidal in shape.

Here we detail how a more detailed description of the instrumental resolution function is calculated[2]. In addition, we outline the situations where the differences between the detailed and approximate resolution kernels become apparent, with the main effects being observed when the width of the resolution kernel is similar to the width of the features in the reflectivity curve.

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[2] Nelson, A. & Dewhurst, C. D., *Journal of Applied Crystallography,* **2014***, 47*

Abstract

Paper Ref: 255

TERMINAL ALKYNES AS A POSITION ABSTRACTION TOOL: DETERMINATION OF THE MOLECULAR PARAMETERS BY SEMIEMPIRICAL METHOD

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Mechanosynthesis is the fabrication of atomically precise structures by formation of covalent chemical bonds with positional control of mechanical forces. A mechanosynthetic tool should have a chemically active tooltip and a chemically inert handle to which the tooltip is covalently bonded (Temelso, 2006). The suitable molecules for hydrogen abstraction tooltip include the propargyl or ethynyl radical containing two carbon atoms triple bonded together (Drexler, 1992). The unreactive regions of these molecules serve as a handle or attachment point. Hence the terminal alkynes can be used to abstract hydrogen in the production of nano materials (Musgrave et.al. 1991, Srinivasakannan, 2008). Semi-empirical methods serve as a tool in modelling and understanding the properties of molecular systems. In the present work, the structure of four terminal alkynes are optimized by AM1, PM3 & PM7 methods using MOPAC2012 (Stewart, 2013), with the available crystallographic data as the starting geometry. Calculated frequencies are compared with the FTIR spectrum to validate the results. Molecular parameters such as $E_{HOMO,}$ E_{LUMO}, the energy gap (ΔE), hardness **(η)**, electron affinity (EA), ionization potential (IE) and heat of formation (ΔH_f) are determined.

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Abstract

Paper Ref: 92

Chemical Deuteration in Neutron Scattering: Demand, Supply and Impact

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Molecular deuteration significantly increases the options in structure function investigations using neutron scattering and diffraction techniques. There have been limited global initiatives in the field of molecular deuteration where the majority of these programs focus on biological deuteration of proteins and lipids, while more complex deuterated small molecules haven't been widely available to the neutron community. This has limited the experiments that can be performed, and formed a bottle-neck for advancing the applications of neutron scattering.

In this paper we will discuss the recent advancements and the impact of deuteration on the research outcomes achieved by using deuterated molecules produced by the chemical deuteration laboratories at the National Deuteration Facility in the Bragg Institute, ANSTO. Recent high-impact case studies will be presented which reveal the exciting and diverse characterisation studies which are now available for the neutron community.

We describe here the synthesis and application of deuterated organic molecules used to investigate complex nanoscale systems in the fields of molecular electronics, structural biology, and biotechnology. The chemical deuteration of surfactants, sugars, heterocyclic and aromatic compounds has made possible a wide range of investigations. This includes the study of (i) the localisation of sugars in lipid membranes using neutron diffraction to give insights into cryoprotective mechanisms [1], (ii) the pH-responsiveness of the assembly of lipid digestion products in biologically relevant systems [2], and (iii) the structure and host-guest properties of metal-organic frameworks (MOFs) using neutron diffraction [3,4].

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CMP-1 Multiferroics

Abstract

Paper Ref: 449

Coupling of Elastic Strain with Magnetism and Interface Induced Magnetization in Complex Oxide Thin Films and Hetrostructures: a Polarized Neutron Reflectivity Study

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Nanostructured architectures with reduced dimensionality and/or enlarged interfacial areas have been used as model systems to investigate the interface effect on physical properties of complex oxides. A nanocomposite, consisting of two or more constituents can produce emergent behaviors through coupled competing order parameters (magnetic, charge, orbital, etc.). The availability of various heterostructures formed by correlated electron materials with distinct phases offers new opportunities to study competing interactions at interfaces because these interactions produce a delicate balance between states with very different properties, such as charge-ordered, ferroelectric (FE), ferromagnetic (FM), and superconducting states.

Here I will discuss measurements from polarized neutron reflectivity and other complementary techniques, which provide insight of the rich spectrum of phenomenon present at the interfaces of complex oxide films. Particularly, two recent studies, (a) unequivocal evidence for the influence of applied elastic stress on magnetic properties of a thin film of $(La_{0.4}Pr_{0.6})_{0.67}Ca_{0.33}MnO_3$ [1-5] and (b) Induced magnetization in $La_{0.7}Sr_{0.3}MnO_3/BiFeO_3$ hetrostructures [6] will be discussed.

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Abstract

Paper Ref: 115

Chemical control of magnetic anisotropy and ferroelectric polarization in multiferroic $MnWO_4$

Young-Sang Song[1], **Jae-Ho Chung**[1], Sae-Hwan Chun[2], Kwang Woo Shin[2], [2], Jürg Schefer[3], and In Hwan Oh[4]

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In the spin-current mechanism of magnetoelectricity, the electric polarizations appear via relativistic spin-orbit exchange couplings of two adjacent magnetic ions ($\boldsymbol{S}_i$ and $\boldsymbol{S}_j$). The associated polarization vector is expressed by the relation $\boldsymbol{P}_e = A(\boldsymbol{r}_i - \boldsymbol{r}_j)\times(\boldsymbol{S}_i \times \boldsymbol{S}_j)$ [1]. $MnWO_4$ is one of the representative spin-current multiferroics, in which ferroelectric polarization appears along its crystallographic *a* axis in the antiferromagnetic phase with incommensurate spiral ordering. Such nontrivial magnetic ordering without spatial inversion symmetry is stabilized by long-range exchange interactions of Mn^{2+} ions as well as by their magnetic anisotropy. The direct relation between the magnetic ordering and the electric polarization offers possibilities to control the ferroelectricity via adjusting the spatial distribution of magnetic anisotropy fields.

In this talk, we will present systematic control of the ferroelectricity in $MnWO_4$ by chemical substitutions. The adjustment of spin spirals, and their orientations in particular, was achieved by substitutions of magnetic ions with different magnetic anisotropy field distributions. For instance, the incommensurate spiral ordering and the ferroelectric phase were simultaneously suppressed when Mn^{2+} ions were substituted by Fe^{2+}. In contrast, substitution with Co^{2+} ions not only stabilized the ferroelectric phase but flopped the primary direction of polarization from *a* to *b* axis, and vice versa [2]. Furthermore, spontaneous 180° flop was achieved with Ni^{2+} substitution approximately at 16 % [3]. We will show that these seemingly complex and contrasting doping effects can be consistently explained by collective magnetic field anisotropy fields of the involved magnetic ions.

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Abstract

Paper Ref: 122

EFFECT OF Cu AND Zn SUBSTITUTION ON THE STRUCTURE AND MAGNETISM OF $NdMnO_3$ MANGANITES

Dr Megha Vagadia [1], Ms. Brinda Vyas [2], Mr. Malay Udeshi [2], Ms. Priyanka Trivedi [2], Dr. Sudhindra Rayaprol [3], Prof. D.G. Kuberkar [2]

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[3] *UGC-DAE Consortium for Scientific Research, BARC Campus*

RMnO_3 type manganite materials, where *R* is a rare-earth metal, are known to exhibit variety of interesting physical properties such as insulator to metal transition, ordering from a paramagnetic to ferromagnetic state, colossal magnetoresistance etc., which make them potential candidates for *spintronic* applications. $NdMnO_3$ exhibits antiferromagnetic ordering (T_N ~ 78 K) along with coexistence of antiferromagnetic and ferromagnetic interactions and reorientation of Mn spins at low temperatures [1, 2]. Substitution is an ideal tool for modifying the physical properties, particularly in manganite. In the present work, we have substituted Cu and Zn at Mn site to understand the influence a magnetic or a non-magnetic ion at Mn site will have on the structure and magnetism of $NdMnO_3$. The magnetic properties have been studied by magnetization measurements as a function of temperature and magnetic field. Crystallographic structural properties have been obtained from room temperature neutron diffraction studies. Temperature dependent neutron diffraction studies were carried out to understand the magnetism of this compound via the modifications observed in the magnetic structure as a function of temperature. Analysis of room temperature ND data confirms single phase formation of $NdMn_{1-x}M_xO_3$ (x = 0.05; M = Cu and Zn) compounds, crystallizing in orthorhombic perovskite structure with *Pnma* space group. Field and temperature dependent magnetization measurements show magnetic anisotropy and negative magnetization in pure and doped $NdMnO_3$. Temperature dependent neutron diffraction patterns show that the magnetic peak appears below T_N ~ 75K and becomes stronger in intensity with decreasing temperature. Detailed analysis of temperature dependent neutron diffraction and magnetization data will be presented and discussed in the light of the observation of negative magnetization and evolution of magnetic structure of $NdMn_{1-x}M_xO_3$ (x = 0.05; M = Cu and Zn) compounds below and above their respective magnetic ordering temperatures.

Acknowledgement: This work is financially supported by UGC – DAE CSR, Mumbai Center through the collaborative research project No. CRS-M-190.

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Abstract

Paper Ref: 269

Observation of electromagnons in the field-induced noncollinear ferrimagnetic phase of $Ba_2Mg_2Fe_{12}O_{22}$ by means of polarized inelastic neutron scattering

Taro Nakajima[1], Kazuhisa Kakurai[1, 2], Masaaki Matsuda[3], Youtarou Takahashi[4], Shintaro Ishiwata[4], Shuichi Wakimoto[2], Yasujiro Taguchi[1], Yoshinori Tokura[1,4], and Taka-hisa Arima[1, 5]

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Multiferroic hexaferrites have been extensively investigated because they exhibit spin-driven ferroelectricity associated with noncollinear magnetic orders in relatively low magnetic field and high temperature regions.[1,2] In fact, distinct magnetoelectric (ME) effects have been observed even up to room temperature in a Z-type hexaferrite $Sr_3Co_2Fe_{24}O_{41}$[1] and a Y-type hexaferrite $BaSrCo_{2-x}Zn_xFe_{11}AlO_{22}$[2]. Therefore, they are considered to be potential candidates for new electronic devices.

In addition to the static ME couplings, some of the hexaferrites also exhibit dynamical ME coupling, i.e., “electromagnon” excitation. Kida *et al*. performed time-domein terahertz optical spectroscopy on a Y-type hexaferrite $Ba_2Mg_2Fe_{12}O_{22}$[3], observing gigantic optical absorption associated with the magnetic ordering in the energy range of 3 ~ 5 meV.

To investigate the dynamical spin-polarization coupling in more detail, we performed polarized inelastic neutron scattering measurements on $Ba_2Mg_2Fe_{12}O_{22}$. In the present study, we focused on a field-induced noncollinear commensurate ferrimagnetic phase, in which the optical absorption is maximized. We observed inelastic scattering signals around 5 meV at the zone center. This energy is in good agreement with that of the optical absorption spectra obtained in the previous terahertz time-domain spectroscopy. Polarization analysis for the inelastic scattering signals revealed that the excitation around 5 meV is indeed of magnetic origin, and that the spins are mainly oscillating in the plane perpendicular to the magnetic field. Applying the magnetostriction model to the schematic model of the spin-wave modes obtained from the present results, we can qualitatively explain the antiferromagnetic spin-wave modes inducing oscillating electric polarization along the *c*-axis, which corresponds to the optical absorption assigned to the ‘electromagnon’ mode.

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BST-2 Proteins

Abstract

Paper Ref: 453

Protein dynamics and hydration studied by inelastic neutron scattering and molecular dynamics simulation

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Inelastic neutron scattering can measure the protein thermal fluctuations under the physiological conditions with aqueous environment. The protein dynamics should be characterized with wide time-space ranges. The inelastic neutron scattering is the powerful method to observe the low-energy protein dynamics in THz region, which, the theoretical studies have indicated, is essential for the solvent-coupled protein dynamics. The selective observation of protein and hydration water by deuteration, and the complementary analysis of hydration structure and dynamics with molecular dynamics simulation are effective methods for studying the protein hydration. Protein dynamical transition is coupled with the fluctuation of the hydration water network covered over the protein surface. The protein dynamics activated by hydration is essential for the biological function. On the other hand, it has been found recently that the vitrification of the protein accompanied by dehydration in the biological system contributes the environmental tolerance mechanism of the organism in the extremophilic environment. The hydration and vitrification of protein studied by inelastic scattering can be expected to give fruitful information not only for a basic science of protein physics but also for a molecular biology and an application for a food science. The non-destructive neutron measurement of bio-system is the effective for the examination and analysis of the bio-materials and tissues.

Abstract

Paper Ref: 214

INTERACTION OF THE CHLORIDE INTRACELLULAR ION CHANNEL PROTEIN CLIC1 WITH DIFFERENT STEROLS IN MODEL MEMBRANES

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[2] *Bragg Institute, Australian Nuclear Science and Technology Organisation, Australia*

Background and Aims: Sterols have been reported to modulate conformation and hence the function of several membrane proteins. One such group is the Chloride Intracellular Ion Channel (CLIC) family of proteins. The CLIC protein family consists of six evolutionarily conserved protein members in vertebrates. These proteins are unusual, existing as both monomeric soluble proteins and as membrane bound proteins. We now for the first time demonstrate that the spontaneous membrane insertion of CLIC1 is dependent on the presence of cholesterol in membranes. Our novel findings also extend to the identification of a cholesterol-binding domain within CLIC1 that facilitates the spontaneous membrane insertion of the protein into membranes containing cholesterol.

Methods: CLIC1 wild type (WT) and mutant proteins were purified by Ni-NTA followed by size-exclusion chromatography. Langmuir monolayer film balance experiments were carried out using 1-Palmitoyl-2-oleoylphosphatidylcholine (POPC) alone, or in a 5:1 mole ratio combination with either one of the following sterols: Cholesterol (CHOL), β-Sitosterol (SITO), Ergosterol (ERG), Hydroxyecdysone (HYD) or Cholestane (CHOS). WT CLIC1 or mutant versions of CLIC1 were then injected into the aqueous subphase under the lipid film.

Results: In lipid monolayers lacking sterols, CLIC1 did not insert. However significant membrane insertion occurred when CLIC1 was added to membranes containing cholesterol. Substitution of membrane cholesterol with either HYD, SITO or ERG, not only increased CLIC1's membrane interaction but also increased its rate of insertion. Conversely, CLIC1 showed no insertion into monolayers containing CHOS, which lacked the intact sterol 3β-OH group. CLIC1 mutants G18A and G22A, did not insert in POPC:CHOL monolayers whereas the C24A mutant showed membrane insertion equivalent to WT CLIC1. X-ray and Neutron reflectivity, along with Small Angle X-ray Scattering techniques were subsequently used to probe structural features of the membrane bound CLIC1 and CLIC1-Cholesterol complex in solution.

Conclusion: These findings confirm that the GXXXG motif within CLIC1 acts as a sterol binding site facilitating the protein's membrane interaction and insertion. Critical to this process of spontaneous membrane insertion is the presence of an intact 3β-OH group within the sterol structure itself. Furthermore, additional double bonds and methylation of the steroid skeleton enhanced CLIC1 membrane insertion.

Abstract

Paper Ref: 209

Structure and interaction among protein and nanoparticle mixture in solution: Effect of temperature

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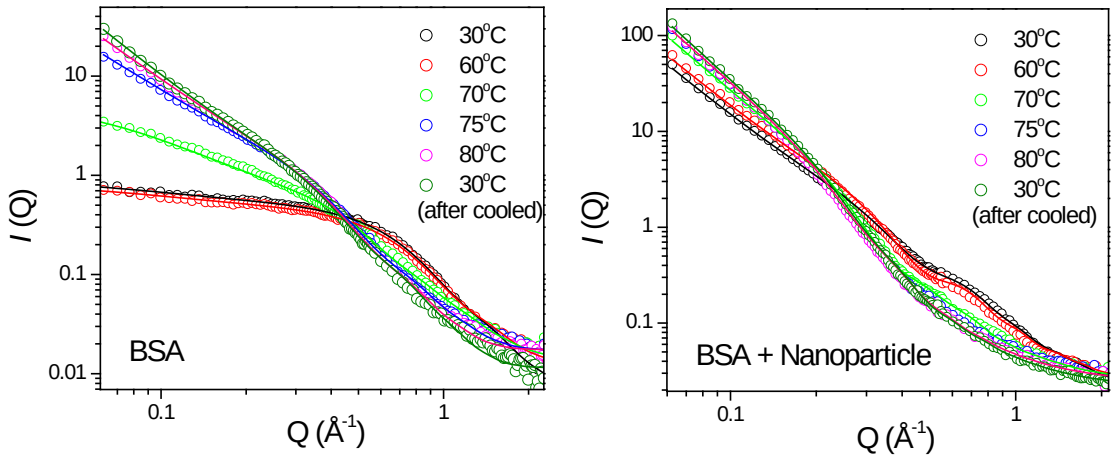
Structure and interaction among globular protein bovine serum albumin (BSA) and silica nanoparticle mixtures in solutions have been studied using small angle neutron scattering (SANS) technique by varying the solution temperature, and protein and nanoparticle concentrations. Our study shows that in absence of nanopaticles and up to a moderate temperature (≈75°C), a short-range attraction and in addition a long-range electrostatic repulsion between the protein molecules exist [1-3]. However, the attractive interaction among BSA increases with the increase of the solution temperature. Above that temperature, fractal structure forms [4] and the fractal structure exist even when the solution temperature is reduced to the initial room temperature (≈30°C). In presence of nanoparticles, fractal structures form even at room temperature by both the protein and nanoparticles. The fractal dimension increases with the increase of the solution temperature and this temperature induced structural transition is irreversible. Variations of nanoparticle concentrations show nearly the same behavior.



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Abstract

Paper Ref: 293

Using SANS to monitor the interaction of misfolding alcohol dehydrogenase with the molecular chaperone protein 14-3-3ζ

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14-3-3 is a family of acidic, dimeric proteins which are highly conserved across many species. Each monomer is approximately 30kDa in mass and contains 9 α-helices. Dimer formation is initiated at the N-terminal region of the protein as a result of the interaction between several buried polar and hydrophobic residues in this region [1]. 14-3-3 proteins interact with a wide range of proteins to regulate many cellular processes, e.g. apoptosis and mitosis [2], as well as protein misfolding associated with conformational diseases such as Alzheimer's and Parkinson's Disease [3]. A potential role of 14-3-3 in these diseases was discovered with the observation that 14-3-3ζ can act as a molecular chaperone [4], whereby it stabilises intermediately folded proteins to prevent their aggregation. The binding site and mechanism of the chaperone action of 14-3-3ζ are not known, despite being narrowed down in our NMR study [5]. We produced deuterated 14-3-3ζ and used it in SANS experiments with a model misfolding protein, alcohol dehydrogenase (ADH). Contrast variation allowed us to monitor changes in each component separately after the initiation of ADH misfolding. The R_g and D_{max} values of ADH under stress show an increase in size with time, consistent with unfolding and aggregation. In the presence of 14-3-3ζ, the unfolding of ADH is reduced and the protein maintains a globular expanded conformation consistent with an adoption of an intermediately folded (molten globule) state. 14-3-3ζ whilst chaperoning showed a reduction in size, possibly due to dissociation. *Ab initio* models were also obtained. This is the first instance where conformational changes during chaperoning of either a partly folded target protein, or 14-3-3ζ, have been observed.

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MSC-2 Structural chemistry

Abstract

Paper Ref: 15

STRUCTURALLY PRECISE COPPER HYDRIDE NANOCLUSTERS

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Encouraged by curiosity-driven chemistry in the fabrication of hydride-centered octanuclear copper clusters surrounded by 1, 1-dichalcogenolate ligands,[1] our research group has developed a new synthetic methodology, which can increase the cluster nuclearity from eight to thirty six simply by adding excess amount of borohydrides into solution containing copper salts and ligands. Herein two types of nanoscale polyhydrido copper clusters stabilized by sulfur-donor ligands are reported. The structure of $[Cu_{20}(H)_{11}\{S_2P(O^iPr)_2\}_9]$ can be expressed by a trigonal-bipyramidal unit of $[Cu_2H_5]^{3-}$ anchored within an elongated triangular orthobicupola polyhedron of eighteen copper atoms, which is further stabilized by eighteen sulfur atoms from nine dithiophosphate ligands and six capping hydrides.[2] The core unit of copper hydride nanoparticle in $[Cu_{28}(H)_{15}(S_2CNPr_2)_{12}]^+$ comprises one central interstitial and eight outer-triangular-face-capping hydrides. A further six truncating hydrides form an unprecedented bridge between the inner and outer copper atom arrays. The irregular inner Cu_4 tetrahedron is encapsulated within the Cu_{24} rhombicuboctahedral cage, which is further enclosed by a 12 dithiocarbamate ligand array which subtends the truncated octahedron of 24 sulfur atoms concentric with the Cu_{24} rhombicuboctahedral and Cu_4 tetrahedral arrays about the innermost hydride.[3] All of the hydride positions in the nanospheric clusters were determined unequivocally by the single crystal neutron diffraction. These compounds display an intriguing, if limited, room temperature H_2 evolution upon exposure of solutions to sunlight, under mild thermolysis condition, and on reaction with weak (or strong) acids to provide excellent models useful in the hydrogen storage technology.

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Abstract

Paper Ref: 370

Single Crystal Neutron Diffraction –An Authoritative Method for Chemistry

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Single crystal X-ray diffraction has become a cornerstone technique in modern chemistry. An X-ray diffraction study in a paper or manuscript is often demanded and the number of X-ray studies performed (which dwarfs the number published) has ballooned with the advent of high-throughput area detector systems coupled with reliable open-flow low temperature devices and powerful modern laboratory X-ray sources. Given the huge number of structures being determined even a small percentage of incorrect structures reaching the literature is of considerable concern.

It is desirable that new projects start from replication of results which inspire new research, this does not occur often. A particularly worrying trend in the literature is the coupling of X-ray diffraction results with theoretical calculations to “verify” or “prove” aspects of structures which cannot be demonstrated by X-ray diffraction without ambiguity – such as hydrogen atom locations ,especially adjacent to heavy atoms and in favourable cases (such as cobalt and nickel) to differentiate elements of similar atomic number. Whilst the arsenal of highly developed spectroscopic techniques, carefully applied, will provide an ensemble of data from which the answer or a set of possible answers can be derived, neutron diffraction is a powerful method which can prove the proposed structure or distinguish between a set of structures which might fit the available spectroscopic data.

Laue single-crystal neutron diffraction has brought the possibility of undertaking neutron diffraction studies within reasonable parameters of material quantity and crystal size and it is fascinating to observe the number of times the structure revealed by neutron diffraction is at variance with the model based on either the available experimental data or data supported by calculation.

It is important to ensure that the power of neutron diffraction is applied to problems where it is the experimental method capable of differentiating between multiple possibilities or proving a single structure postulated to be consistent with the available data. Our experience with KOALA at ANSTO is that it is sufficiently often the case that the result is not as “expected” or “predicted” that single crystal neutron diffraction studies remain an important method in chemical crystallography.

Abstract

Paper Ref: 261

Synthesis and Characterisation of ($M_{2-x}Fe_x$)SnO_4 (M = Mn, Zn) ternary transition metal-tin-oxygen spinel systems

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Mixed transition-metal oxide (MTMO) spinels including Mn and Zn-metal containing stannate phases have promising material properties and are known for the ability to tailor particular features for different uses. They are currently being explored as possible alternative substrates in many emerging high-tech applications such as electrode materials in lithium-ion batteries [1] and as conducting oxides in gas detector sensors [2]. The project aims to study the crystal and magnetic structures of iron and tin containing quaternary stannate to produce the novel spinel structures ($M_{2-x}Fe_x$)SnO_4 (M = Mn, Zn and $0 \leq x \leq 2$). Neutron and synchrotron X-ray powder diffraction, Mössbauer, IR and UV-Vis spectroscopy data, magnetic measurements and SEM/EDX have been performed on the ($M_{2-x}Fe_x$)SnO_4 systems to find out the exact mechanism of Fe substitution, how much Fe and in what oxidation state is substituted and the effects upon the crystal and magnetic structure. For the range of ($Zn_{2-x}Fe_x$)SnO_4 spinels, initial results support the hypothesis that there is more than one doping mechanism, which is dependent upon the amount of Fe that is doped. Diffraction results and Mössbauer data indicate that an enrichment of Fe relative to Sn is evident in the Fe-rich structures, which might be due to Fe replacing Zn followed by Sn as more Fe becomes present in the system.

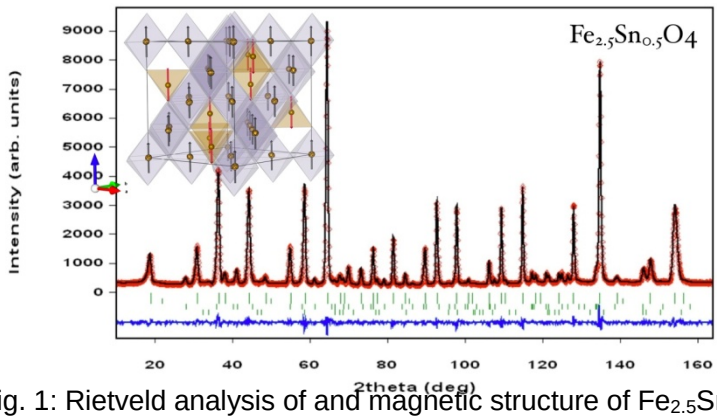

Fig. 1: Rietveld analysis of and magnetic structure of $Fe_{2.5}Sn_{0.5}O_4$

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Abstract

Paper Ref: 76

Short- and long-range modulated oxygen order in brownmillerite-type Sr_2FeCoO_5 and Ca_2FeCoO_5

Josie E Auckett[1,2], Ray L Withers[3], Andrew J Studer[2], Maxim Avdeev[2], Alison J Edwards[2], Garry J McIntyre[2], Chris D Ling[1]

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Sr_2FeCoO_5 and Ca_2FeCoO_5 belong to the perovskite-derived group of oxides known as the brownmillerites ($A_2B_2O_5$), which are characterised by layers of BO_6 octahedra alternating with layers of chain-linked BO_4 tetrahedra separated by oxygen vacancy channels. Brownmillerites have potential applications as diverse as combustion catalysts, cements, and solid oxide fuel cell cathodes. Oxide-ionic conductivity in brownmillerites is associated with the presence of oxygen vacancies adjacent to the tetrahedral chains [1,2], and is therefore influenced by the relative arrangements of oppositely twisted chains throughout the structure. Recently, a variety of modulated chain-ordering schemes have been identified in several brownmillerites that were previously thought to adopt only simple ordered or completely disordered chain arrangements.

In this work, a brownmillerite chain-ordering modulation first identified in Sr_2FeCoO_5 by electron diffraction has been confirmed by single-crystal neutron diffraction on a large crystal grown by the floating-zone (FZ) method. Although incommensurate modulation vectors are identified on the short electron diffraction length scale, the neutron diffraction data show clearly that a commensurate arrangement dominates the crystal on average. This behaviour closely follows that of the series end-member $Sr_2Fe_2O_5$, an important ionic-conductive brownmillerite, implying that Sr_2FeCoO_5 may display similarly favourable ionic-conductive properties.

We also investigated Ca_2FeCoO_5, a relatively new brownmillerite characterised in 2010 [3]. The presence of well-resolved satellite reflections in single-crystal and powder neutron diffraction data support the choice of a commensurate chain-ordered arrangement to describe the structure. However, attempts to refine this structure against either data set yielded poor results. A detailed examination of neutron and x-ray precession images obtained for FZ-grown single crystals reveal the presence of certain reflections that are forbidden by the expected *Pbma* symmetry. Consideration of the apparent pseudo-symmetry yields evidence for a micro-intergrowth of regions with slightly different chain-ordering schemes within the dominating *Pbma* matrix, similar to an arrangement reported previously for Ca_2MnGaO_5 [4].

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Abstract

Paper Ref: 64

SURVEYING THE HIGHER DIMENSIONS OF THE APERIODIC COMPOSITE NONADECANE/UREA

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We recently observed for the first time that there exist phase transitions where the structural changes correspond just to degrees of freedom hidden in the internal (super)space of an aperiodic material, here the composite nonadecane/urea [1]. A key factor in the discovery of this type of transition [2] was the examination of the diffraction pattern in 3D, only possible at the time on a four-circle triple-axis neutron spectrometer, the analyzer used in zero-energy transfer to reduce the background and improve resolution. Despite the greater accessibility in reciprocal space, the weak intensity of the superlattice reflections limited the volume of reciprocal space that could be explored. Modern neutron Laue diffractometers with large image-plate detectors permit rapid and extensive exploration of reciprocal space with high resolution in the two-dimensional projection and a wide dynamic range with negligible bleeding of intense diffraction spots [3]. Surveying nonadecane/urea with neutron Laue diffraction from 300K to 4K reveals further detail of the superspace-driven phase transition, notably a significant increase in misorientation in the plane perpendicular to the composite misfit axis, as well as a first-order transition to a new phase at lower temperature. Complementary monochromatic X-ray examination, again using a high-resolution image-plate detector, reveals that this new phase corresponds to a new ordering of the guest alkane subsystem [4]. Little more can be concluded though, since in common with other alkane-urea crystals, the paucity of unique data in the new phases prevents full structural refinement. Nevertheless these new observations shed further light on how nature uses the degrees of freedom hidden in the internal superspace to form states that cannot be envisaged in the usual 3D real space.

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EI-1 Stress and strain

Abstract

Paper Ref: 444

A Study of Lattice Elasticity from Low-entropy Metals to Medium- and High-entropy Alloys

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An equal-molar CoCrFeMnNi, face-centered-cubic high-entropy alloy system and a face-centered-cubic 304L stainless steel described as a medium-entropy system, are measured by in-situ neutron-diffraction experiments subjected to continuous tension at room and several elevated temperatures, respectively. With spallation neutron, the evolution of multiple diffraction peaks are collected simultaneously for lattice-elasticity study. Temperature variation of elastic stiffness of a single face-centered-cubic-phase Ni and a single face-centered-cubic-phase Fe are compared as low-entropy metals. The CoCrFeMnNi high-entropy alloy show distinct lattice anisotropy. The data is shown on Scripta Materialia (doi:10.1016/j.scriptamat.2015.01.011). The mechanism will be reported.

Abstract

Paper Ref: 379

RESIDUAL STRESS MEASUREMENTS IN LASER CLAD AIRCRAFT ALUMINIUM ALLOY

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Fatigue and corrosion damage of structural components threatens the safety and availability of civil and military aircrafts. There is no sign of relief from these threats as civil and military aircrafts worldwide are continuously being pushed further into and past their initial design fatigue lives in tight financial circumstances. Given fatigue and corrosion damage often initiates at the surface and sub-surface of the components, there has been extensive research and development worldwide focused on advanced aircraft repair technologies and surface enhancement methods.

The Deep Surface Rolling (DSR) is one of advanced surface enhancement technologies that can introduce deep compressive residual stresses into the surface of aircraft metallic structure to extend its fatigue life. For the development of cost-effective aircraft structural repair technologies such as laser cladding, in this study, aluminium alloy 7075-T651 specimens with simulated corrosion damage were repaired using laser cladding technology. The surface of the laser cladding region was then processed by DSR. The experimental results from subsequent fatigue testing of laser cladded baseline, DSR and post-heat treated laser cladded specimens discovered that the DSR process can significantly increase fatigue life in comparison with the as-cladded baseline. The three dimensional residual stresses were measured by neutron diffraction and the results confirmed the beneficial compressive residual stresses at the cladding surface can be achieved in depth more than 1.0 mm.

Abstract

Paper Ref: 61

The Progress of Neutron Diffraction Stress Analysis Technique at China Academy of Engineering Physics

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I. Construction of the RSND

The construction of the RSND was finished in August 2012. A standard sample 7050 aluminum alloy shrink-fit ring and plug was measured. Texture measurement device was equipped on the RSND by utilizing the Kappa goniometer in October 2014. The main specifications of the RSND are listed as follows: (i) Double focusing silicon single crystal monochromator is employed with accessible wavelengths from 0.12 nm to 0.28 nm; (ii) The uniquely designed sample table can bear loads of up to 500 kg with high position precision; (iii) Two-dimensional position sensitive detector system and radial oscillation collimator can be collaboratively used and moved; (iv) The monochromator-sample-detector distance can be adjusted to accommodate samples with different size; (v) All the adjustable units are controlled by computer automatically.

II. In-situ neutron diffraction technique

A stress rig suited for the RSND has been designed in our group to provide uniaxially tensile and compressive loads up to 20 KN, with high temperature up to 800 ºC. Microscopic mechanism for Ni-Ti alloy during tensile deformation has been measured. In the linear elasticity stage, lattice strain of austenite shows strong anisotropy. During the low strain hardening stage, the lattice strain of (110) B2 decreases rapidly while that of (002) B19' increases, which suggests that stress-induced martensite transformation occurs with appearance of <011> II type twinning and the initial martensite begins to reorient. In the high strain hardening stage, another new {20-1} type martensite twinnings appears, where the twinning deformation is considered to be the main mechanism.

III. Ni-based single crystal superalloy characterization by neutron diffraction

The superlattice structure of Ni-based superalloy during fabrication has been measured. Abundant information for Ni-based superalloy has been acquired, such as internal macroscopic stress, phase stress, equivalent stress, lattice mismatch, and diffraction peak broaden and mosaic spread of different crystal planes, which is resulted from thermal and themomechanical fatigue.

Abstract

Paper Ref: 74

EFFECTS OF WELDING PROCESS AND HEAT INPUT ON RESIDUAL STRESSES IN MULTI-PASS WELDS: APPLICATION OF NEUTRON DIFFRACTION

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High strength low alloy (HSLA) steels are widely used in oil & Gas and pressure vessel applications due to their favorable mechanical properties. The need for safe operation of the welded structures has led to an emphasis on fracture and fatigue-related failure assessments for HSLA steels. It is well known that residual stresses and particularly tensile residual stresses are potentially detrimental to the performance of the structures because they may lead to abrupt crack initiation, stress corrosion cracking, fatigue and other structural failures. Therefore controlling and minimizing the residual stress levels in welded structures are crucial for the longevity and safe operation of these structures. Controlling residual stress levels may be achieved through appropriate selection of pre-welding conditions, in-process parameters and post welding treatments. The current study employed neutron diffraction to investigate the effect of heat input and welding process on the distribution of residual stresses in multi-pass weldments. The outcomes of this study indicate that the utilization of welding procedures with higher heat input weld runs is beneficial in the reduction of the residual stresses The experimental results also indicated that the welding process itself has a significant impact on the residual stress levels. The generated experimental data may also be utilized for validation studies of computational modelling and approaches.

SMS-1 Surfaces and interfaces

Abstract

Paper Ref: 440

Aggregation States of Polymers at Non-solvent Interfaces by Neutron Reflectivity

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The aggregation states of polystyrene (PS) thin films at interfaces with non-solvents such as water, methanol and hexane were examined by specular neutron reflectivity and sum-frequency generation vibrational spectroscopy. The density profiles of the PS thin films along the direction normal to the interface with water and methanol were comparable to that in air. However, this was not the case of the film in hexane exhibiting a diffused interfacial layer due to swelling. Also, the local conformation of PS at the outermost region of the films was quite sensitive to the surrounding environment and responded consequently to a change in its environment. This was the case for typical non-solvents such as water and methanol. The extent of the conformational change might be explained in terms of the interfacial energy. Similar experiments are also performed for poly(methyl methacrylate) (PMMA). Results so obtained are compared with each other.

Abstract

Paper Ref: 295

Diffusion and Stability of Layered Structures in Organic Light Emitting Diodes

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Organic light emitting diodes (OLEDs) are used in a number of products such as mobile phones and televisions and are considered to be the next generation technology for displays and solid-state lighting. Their increasing popularity is due to their high efficiency, low power usage and the ability to be deposited onto flexible plastic substrates. OLEDs comprise multilayer structures with a typical device consisting of (at least) electrodes, adjacent organic charge-transport layers and an emissive layer. The emissive layer normally comprises a phosphorescent dopant in a host material to minimise luminescence quenching interactions.

For several years our group has studied the stability of such multilayer structures using neutron reflectometry (NR). We have shown that under thermal stress the organic layers can readily interdiffuse, leading to degradation in performance [1, 2]. NR has the benefit of giving detailed information about layer structure changes in real time, while simultaneously allowing the optical properties to be probed by photoluminescence measurements as the temperature is varied. The ability to observe interdiffusion of materials with similar structures is made possible by selective deuteration of layers, making use of the National Deuteration Facility. The factors controlling diffusion are still not totally clear, but our recent studies have shed light on how stability can be predicted and ultimately improved and we have begun to identify the factors that are critical in determining the onset temperature and direction of diffusion in complex multilayer structures [3, 4].

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Abstract

Paper Ref: 279

Interfacial Aggregation Structure of a Bio-inert Polymer Blend in Water

Toyoaki Hirata[1], Hisao Matsuno[1]**,** Daisuke Kawaguchi[2]**,** Tomoyasu Hirai[1]**,** Norifumi L Yamada[3]**,** Masaru Tanaka[4]**,** and Keiji Tanaka[1]

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To establish molecular design for blood-compatible polymers, the structure and dynamics of chains at the water interface must be understood using solid evidence as the first bench mark. Here we show how polymer characteristics at the water interface impact on the structure of the interfacial water, resulting in a change in protein adsorption and of platelet adhesion.

As a particular material, a blend composed of poly(2-methoxyethyl acrylate) (PMEA) and poly(methyl methacrylate) (PMMA) was used. Three kinds of PMEAs with the number-average molecular weight (M_n) and polydispersity index (PDI) were used; M_n of 23.5k, 53.6k and 104k and PDI of 1.09, 1.07, and 1.08, respectively. Also, PMMA with M_n of 85k and PDI of 1.09 and deuterated PMMA (dPMMA) with M_n of 93k and PDI of 1.07 were used. PMEA/PMMA and PMEA/dPMMA blend films with a bulk composition of 50/50 (wt/wt) were annealed at 353 K for 6 hours under vacuum. The composition profiles of the blend films in air and D_2O were examined by neutron reflectivity (NR) measurements.

NR analysis for PMEA/dPMMA blend films revealed that PMEA was preferentially segregated at the outermost region in the film of the blend, in which PMEA and dPMMA were completely miscible in the internal phase. The PMEA fraction in the polymer at the water interface was almost 100% and was insensitive to the M_n of the PMEA. Then, a NR measurement was also made for PMEA/PMMA blend films with a comparable thickness in deuterated water. This enables us to gain direct access to the water fraction along the direction normal to the interface, in relation to the whole, namely PMEA, PMMA and water. Also, the interface of PMEA-23.5k/dPMMA became diffuse after contacting with D_2O. These observations make it clear that the blend film was swollen by D_2O, and these results were common to all molecular weight systems. The effect of polymer dynamics at the water interface on structure of the interfacial water and on blood-compatibility will be discussed in the conference.

Abstract

Paper Ref: 80

Interactions of Solute Molecules with an Interface by Medium Angle Diffraction

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In this study we present a method for determining the profile of solute molecules confined between two interfaces with particular application to model systems for the cryobiology. The model systems in this case are stacks of oriented lipid bilayers supported on a quartz slide. When the bilayers swollen with different amounts of water and or water/solute the samples are effectively one dimensional pseudo-crystals. In this case the unit cell includes the bilayer and inter layer solution and any molecules localized within this structure. Fourier methods may be used to reconstruct the scattering length density profile of the unit cell from neutron diffraction measurements thus yielding information about the distribution of components of the solute within a unit cell. Deuteration of the sample components can be used for phasing of the Fourier reconstruction or to provide contrast between components in bilayer stacks[1]. We discuss the application of selective deuteration of various components of the lipid bilayer and the application of such methods to systems of higher dimensionality such hexagonal and cubic phases and the use of anomalous (energy dependent) diffraction as a means of obtaining phase information.

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Abstract

Paper Ref: 245

Multi-Step Swelling of Thin Nafion Films Studied by Neutron and Optical Reflectivity

Yudai Ogata[1], Daisuke Kawaguchi[2], Norifumi L. Yamada[3], Keiji Tanaka[1,4]

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Nafion has been widely used as a proton exchange film in polymer electrolyte fuel cells (PEFC). Excellent electrochemical and mechanical properties of Nafion are associated with the characteristic network structure of the hydrated water phase. Although downsizing PEFC is one of the interesting developments in the near future, it appears to us that most studies conducted so far are limited to bulk systems. In this study, water sorption behavior in thin Nafion films prepared on silver and silicon oxide (SiO_x) substrates was examined by neutron and optical reflectivity (NR and OR) measurements.

The swelling ratio for the Nafion films became larger in water with increasing time, meaning that water molecules were sorbed into the films on both substrates. The thin Nafion films thickened in three steps named here as regimes I, II and III. At first, the films asymptotically reached a swelling ratio of 1.05. Then, the films resumed thickening asymptotically up to 1.26. Finally, the swelling ratio reached 1.41. Interestingly, these asymptotic swelling ratios in regime I, II and III are coincident with the swelling ratios for the structural transition in the bulk. Thus, the three-step sorption behavior could be explained in terms of the structural evolution in the internal region of the film. While the characteristic swelling ratio at the border between different regimes was neither dependent on the substrate species nor the film thickness, the swelling kinetics remarkably slowed down and depended on the type of the substrate. This is due to the interaction between the substrate and Nafion and/or the confinement effect.

Plenary Lecture 2

Abstract

Paper Ref: 450

Neutrons for biology: Protein interactions, dynamics and hydration

Dr Kathleen Wood[1]

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Since the pioneering first protein structures in the 1960s, amazing technical advances have been made to study the structure of biomolecules. In 2014, the database of bio-molecular structures, the Protein Data Base, celebrated 100 000 entries. Biological scientists now work in the era of having at their disposal the sequences of entire genomes and a large number of the corresponding biological structures at atomic resolution. However, current challenges in structural biology have moved beyond knowledge of a single protein structure and how it relates to function. Here I will present several new challenges where neutrons have a unique role to play.

One such challenge is to put known protein structures 'back in the cell' - to understand how they function in a cellular context. In many cases a biological function is not carried out by a single protein, but by several acting together. Studies of these biologically important complexes are at a space scale well suited to small angle neutron scattering, where isotope labelling can reveal complementary information to that accessible by small angle X-ray scattering. I will highlight recent work studying biomolecular complexes with the Quokka small angle instrument at the Bragg Institute.

It is also increasingly apparent that defining a unique biomolecular structure or conformation does not inform us of all its biological complexity. Biological structures are animated by dynamics occurring over many timescales. They can be considered as nano-machines, which must interconvert between many different structures to fulfil their biological function. Neutron scattering allows us to study biomolecular dynamics on the pico to nano-second timescale. I will here discuss dynamics of the biomolecules themselves and their surrounding hydration water, which is considered by many to be a bio-molecule in its own right.

Finally, I will discuss a class of proteins being increasingly studied, which lack a well-defined folded structure, 'intrinsically unfolded proteins'. The central tenant of structural biology has been the structure-function paradigm, but these proteins challenge the paradigm, and carry out many different functions without adopting a stable static structure. Our neutron scattering work reveals both differences and similarities in dynamics across different classes of proteins.

Tuesday 21 July 2015

Plenary Lecture 3

Abstract

Paper Ref: 441

PROBING THE UNUSUAL SUPERCONDUCTING AND NORMAL STATES OF CUPRATE SUPERCONDUCTORS USING NEUTRONS

Jeffery L. Tallon[1]

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High-T_c superconducting cuprates are remarkable materials. They can support very high current densities (around 35 million Amps/cm^2) and they bear up under very high magnetic fields. As a consequence they are now being applied in many diverse areas. But a universal understanding of their behavior is still lacking. This applies to both their unusual normal state and their surprisingly less-unusual superconducting state. Because of the proximity, in these materials, of magnetically-ordered states neutron scattering has proved a powerful probe of their electronic properties as they evolve across the phase diagram as a function of doping. There is also considerable commonality with their cousins the iron pnictides. Moreover, it is possible in these materials for superconductivity and magnetic states to coexist. But do they merely coexist or compete? This talk will highlight some of the key findings from elastic scattering studies of magnetic and nuclear structure and inelastic scattering studies of the magnetic excitation spectrum. The techniques of course have their limitations and that has led to a number of complementary techniques such as resonant inelastic x-ray scattering that probes a wider fraction of the Brillouin zone.

CMP-2 Superconductivity

Abstract

Paper Ref: 442

High-Energy Spin Excitations in Electron-Doped Cuprate

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The doping-evolution of spin and charge dynamics in a doped Mott insulator is an important issue in the research field of strongly correlated electron system. The entire excitation spectra have been intensively and continuously studied with the development of spectroscopic techniques after the discovery of high-transition-temperature (high-T_C) superconductivity in a lamellar cuprate oxide. The recent state-of-the-art spectrometers at large research facilities such as J-PARC enables us to study the details of composite dynamics in the energy-momentum space arising from the interacting degrees of freedom.

Although the comprehensive study on both hole- and electron-doped systems are indispensable to extract the universal mechanism of high-T_C superconductivity, the majority of spectroscopic measurements was done on the hole-doped system. The systematic research on the electron-doped system has been lucking, mainly due to the difficulties in the crystal growth and the emergence of superconductivity through the heat treatment. Here, we introduce the results of inelastic neutron scattering measurements on the spin excitation in the electron-doped copper oxide, which was obtained after overcoming the above experimental difficulties.

First, we confirmed that the spin excitation in $Pr_{1.4}La_{0.6}CuO_4$, which is the parent compound of electron-doped superconductor, is consistent with the spin-wave excitation expected from the s=1/2 two-dimensional Heisenberg model. The evaluated nearest neighbor exchange coupling is 140±5 meV, consistent with the value for the parent compound of hole-doped superconductor, La_2CuO_4. Furthermore, the evidence of higher term of exchange coupling was confirmed from the detailed momentum-dependence of high-energy dispersion in the CuO_2 plane. It was newly found that the spin excitation tends to elongate toward the higher energy region upon electron-doping in $Pr_{1.4-x}La_{0.6}Ce_xCuO_4$ and the zone boundary energy exceeds 300 meV in the sample with $x \geq 0.08$, suggesting the steeper dispersion in the highly doped samples. This doping-evolution is in stark contrast with a negligible doping effect on the high-energy spin excitation in the hole-doped $La_{2-x}Sr_xCuO_4$, Therefore, the electron-hole asymmetry exists in the observed spin excitation against the doping.

Abstract

Paper Ref: 176

POSSIBLE INVERSE PROXIMITY EFFECT ON THE [YBCO(10nm)/LSMO (10nm)]4 SUPERLATTICE FILMS ON STO (001) SUBSTRATE

Hsiung Chou[1,*], Gopeshwar D Dwivedi[1], C.-H. Chen[2], C-Y Li[3], W. H. Li[3], S.-L. Tseng[4] and J. G. Lin[4]

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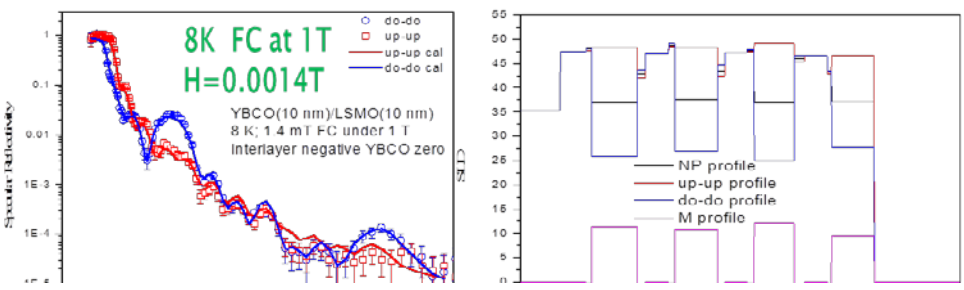
The strong coupling between the spin, orbital and lattice leads to abundant phenomena in oxide SC/FM superlattice films, such as a magnetic proximity effect, a giant magnetoresistance and a SC induced magnetic depletion in the FM layers. In this study, superlattice of [YBCO(10nm)/LSMO (10nm)]4 multilayer film were grown on $SrTiO_3$ (001) substrates by a UHV pulse laser deposition technique. To reveal the magnetic structure fluctuation at SC/FM interfaces, a polarized neutron reflectometry (PNR) measurements were carried out at 300 K (applied magnetic field 1.4mT & 1T) and at 8 K (applied magnetic field 1.4mT field cooled under 1T) in the Platypus beam line of ANSTO. The reflectivity intensities of each curve span over 5 orders of magnitude which reflect good interface quality with very low roughness. PNR data at 300 K where YBCO layers are in normal metal states shows presence of depletion layers at the SC/FM interfaces near the YBCO side. This depletion layers acts like YBCO in a normal metal state and is independent of the polarization of the incident Neutron beam. At low temperature, the depletion layer transfers to a magnetic layer with characteristics very similar both on the scattering length density and magnetization to LSMO layers. A thin interface layer near the YBCO/LSMO interface on the YBCO side shows destruction of superconductivity and induces a mall magnetic moment antiparallel to Mn moment FM layers. This phenomena can be explained on the basis of inverse proximity effect, where the suppression of superconductivity accompanying a Cu magnetic moment can be induced antiparallel to Mn moment as observed earlier by D. K. Satapathy et al., Phys. Rev. Lett. **108**, 197201 (2012).



Abstract

Paper Ref: 11

Polarized neutron scattering as a probe of spin nematicity in the iron-based superconductor $BaFe_{2-x}Ni_xAs_2$

Huiqian Luo[1] , Wenliang Zhang[1], Meng Wang[1], Chenglin Zhang[2], Louis-Pierre Regnault[3], Xingye Lu[1], Rui Zhang[1], Shiliang Li[1] and Pengcheng Dai[1, 2]

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Understanding the electronic anisotropic state associated with the nematic phase is one of the most important unresolved problems in electron correlated systems [1]. Particularly in iron-based superconductors, an in-plane electric anisotropy has been suggested as a signature of the electron nematic phase that breaks the in-plane four-fold rotational symmetry of the underlying tetragonal lattice [2], and a spin nematicity also is proved by neutron scattering experiments [3], where its physical origin remains controversial among charge, orbital [2], spin [4] or local impurity scattering [5]. Here, we use polarized neutron scattering to demonstrate that in-plane spin excitations in electron doped superconductor $BaFe_{1.904}Ni_{0.096}As_2$[6] change from isotropic to anisotropic in the tetragonal phase well above the antiferromagnetic ordering and tetragonal-to-orthorhombic lattice distortion temperatures without an uniaxial pressure [7]. Comparing with the polarized neutron results on the detwinned samples with uniaxial pressure, we argue that the anisotropic low-energy spin excitations from single ion effects are intimately connected with the spin nematic phase in iron pnictides [3], and consistent with in-plane resistivity anisotropy. These results indicate that the polarized neutron scattering is a good probe of the spin nematicity in iron pnictides [8]

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Abstract

Paper Ref: 59

Development of a ferromagnetic component in the superconducting state of Fe-excess $Fe_{1.12}Te_{1-x}Se_x$ by electronic charge redistribution

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Superconductivity in α-FeSe has received special attention due to its simple crystalline structure. It is known that the wavy layered α-FeSe structure can accommodate a significant amount of extra Fe ions. These non-stoichiometric Fe ions occupy the interstitial sites that are slightly below the center positions of the Se square sublattice. The general picture established so far for the links between superconductivity and magnetic ordering in iron chalcogenide $Fe_{1+y}(Te_{1-x}Se_x)$ is that the substitution of Se for Te directly drives the system from the antiferromagnetic end into the superconducting regime. Here, we report on the observation of a ferromagnetic component that developed together with the superconducting transition in Fe-excess $Fe_{1.12}Te_{1-x}Se_x$ crystals using neutron and x-ray diffractions, resistivity, magnetic susceptibility and magnetization measurements. The superconducting transition is accompanied by a negative thermal expansion of the crystalline unit cell and an electronic charge redistribution, where a small portion of the electronic charge flows from around the Fe sites toward the Te/Se sites. Two magnetic phases are identified. The low-temperature magnetic phase, which coexists with superconductivity, involves the ordering of the lattice Fe and interstitial Fe ions. The formation of superconducting pairs drives the electrons to flow from the Fe sites to the Te/Se sites, which alters the numbers of Fe^{2+} ions on the lattice and the excess sites. It is this change in valence of the magnetic sites that drives the system to develop an additional ferromagnetic component in the superconducting state. First-principles calculations show consistent results, revealing that the excess Fe ions play a more significant role in affecting the magnetic property in the superconducting state than in the normal state and the occurrence of an electronic charge redistribution through the superconducting transition.

Abstract

Paper Ref: 448

Understanding functional framework materials using neutron scattering and computational methods

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Coordination frameworks are a class of porous materials constructed from metal ions interconnected three-dimensionally by molecular linkers. A diversity of linker materials leads to a wide range of chemical functionalities within porous frameworks, in which pore size and topology can also be controlled. These features lead to interesting properties including high selectivity and capacity for gas adsorption, with these materials being seriously considered as sorbents for industrial applications. Further, the flexibility of these frameworks couples with their empty pore space and can lead to pronounced and sometimes highly anisotropic mechanical behaviour, such as reversible expansion and contraction known as “breathing”, auxeticity, as well as large and even negative compressibility.

In order to advance such functional materials towards application in working devices or systems, details of their molecular-level operation must be obtained, with neutron scattering making important contributions to this understanding. The neutron scattering mechanism allows information to be gained about these materials that cannot be obtained otherwise, most notably as a result of the ability of neutrons to penetrate special sample environments, to simultaneously provide information on where atoms and molecules are and how they move, and to obtain information for the atomic isotope and not only the element type. Further still, the combination of first-principles computations with neutron scattering is particularly powerful in understanding the structure/dynamics functions that underpin framework material property and performance.

This talk will present science highlights using neutron scattering and first-principle modelling methods to understand the functionality of coordination frameworks. Example studies using both neutron diffraction and spectroscopy will be presented in the area of gas separation and storage, including materials for carbon dioxide capture technologies, as well as materials exhibiting extreme mechanical properties that are of interest for the next generation of high-pressure devices.

Abstract

Paper Ref: 27

ALLUAUDITE $LiFePO_4$: NEW FE-BASED CATHODE MATERIAL FOR LI RECHARGEABLE BATTERY

Dr Jongsoon Kim [1], Mr Hyungsub Kim [2], Mr Inchul Park [2], Dr Young-Uk Park [3], Dr Jung-Keun Yoo [3], Mr Kyu-Young Park [2], Dr Seongsu Lee [1], Dr Kisuk Kang [2]

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Naturally abundant Fe and the stable covalent polyanion unit $(PO_4)^{3-}$ are considered as the important factors in the production of cost-effective, safe electrode materials for next-generation lithium ion batteries.[1-2] Since it was reported that olivine $LiFePO_4$ can have a high power capability,[2] unusual physico-chemical characteristics of $LiFePO_4$ has been focused by many researchers.[3,4] Herein, We further explore the $LiFePO_4$ chemistry by introducing a novel non-olivine $LiFePO_4$ with an alluaudite crystal structure. It is found that the alluaudite $LiFePO_4$ shows promising electrochemical properties allowing reversible extraction and insertion of ~0.8 Li^+ ions in the structure with one-phase based reaction. A novel alluaudite $LiFePO_4$ was successfully prepared, and its structure was investigated in detail through combined Neutron diffraction and X-ray diffraction analyses. The alluaudite $LiFePO_4$ has fundamentally different electrochemical behaviors from those of olivine $LiFePO_4$, while Fe^{2+}/Fe^{3+} redox reaction during charge/discharge is occurred. All of the lithium ions in $LiFePO_4$ could be reversibly extracted and inserted at a reasonably fast rate. It means that this material is another possible Fe-based cathode for lithium ion batteries. We believe that this study will provide insight into the relationship between the structure and the electrochemical activity of $LiFePO_4$ chemistry.

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Abstract

Paper Ref: 236

ELECTROCHEMISTRY AND STRUCTURE OF ELECTRODE MATERIALS FOR LI-ION BATTERIES

Wei Kong Pang[1,2], Vanessa K. Peterson[2], Sujith Kalluri[1], Hua-Kun Liu[1], Shi-Xue Dou[1], Zaiping Guo[1]

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The development of battery materials such as electrodes and electrolytes with good performance and stability is essential to emerging electric-vehicle technologies. Of serious environmental concern is that materials with these properties developed so far contain toxic and expensive elements, such as Co. Intensive studies on “green” Li-ion batteries (LIBs) have been undertaken at the Institute for Superconducting and Electronic Materials (ISEM) at the University of Wollongong. The strong collaboration of the ISEM team with the Energy materials research project at the Australian Nuclear Science and Technology Organisation (ANSTO) offers a good strategic platform for battery materials research. In this presentation we will discuss our study of the novel, Li-rich, Co-free $Li_{1+x}MO_2$ (*M* = Li, Ni, Mn, Fe) [1] composite positive electrode material as an example result from this collaboration. The electrode is prepared *via* a template-free, one-step wet-chemical method followed by conventional annealing in an oxygen atmosphere. Although isotypic to the commercial $LiCoO_2$ material, the $Li_{1+x}MO_2$ is Co free and exhibits superior cycling performance. Our collaborative studies used neutron powder diffraction (NPD) to characterize the material, connecting its electrochemical performance and function to its crystallography, yielding an atomistic-level understanding of function. High-resolution NPD studies (ECHIDNA [2]) revealed that the material has an unprecedented level of cation mixing. *Operando* high-intensity NPD studies (WOMABT [3]) revealed that the lithiation/delithiation of the electrode occurs *via* a solid-solution reaction where the lattice responds approximately linearly with cycling during the $Ni^{2+}/Ni^{3+}/Ni^{4+}$ redox transitions, differing to that observed for the iso-structural commercial cathodes containing a lower level of cation mixing. The understanding made in this work paves the way to develop new electrode materials for use in LIBs, as well as future Na-ion battery technology.

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Abstract

Paper Ref: 20

Moderate temperature oxygen ions mobility, a key feature for lowering SOFCs operation temperatures?

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Oxygen ion conductors at moderate temperatures are materials of major interest for a series of applications like fuel cells, battery electrodes and sensors. For this reason the discovery of oxygen reversible intercalation into brownmillerite-type $SrMO_{2.5}$ (M = Co, Fe) in an electrolyte at RT has been considered of paramount importance [1-3]. By means of different inelastic scattering experiments and DFT calculations we found evidence of the existence of a low energy mode related to a modification of oxygen dynamics in these compounds. To verify that the presence of oxygen mobility down to moderate temperature is not restricted to these compositions, but more general through perovskite related materials, similar findings should be established in other systems.

Here we present, together with new and well established results on iron based Brownmillerites,[2,4] interesting experimental findings on a purely ionic conductor Brownmillerite Sr_2ScGaO_5 [5] and a mixed conductor with K_2NiF_4 structure Nd_2NiO_{4+d}.[6] Combining a range of experimental techniques, including neutron scattering, with DFT and MD calculations has highlighted similarities and differences in the oxygen mobility mechanism at moderate temperature, which is however shown to be based on a modification of a specific low energy collective vibration in both cases. The clarification of the structural and dynamical characteristics that make possible oxygen mobility at moderate temperature is the fundamental step for the design of new compositions that will enhance this unconventional behavior.

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Abstract

Paper Ref: 107

Investigation of the solid-solution Sr_2YNbO_6 - $Sr_3NbO_{5.5}$

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One major challenge of the 21st century is to ensure energy security. Solid-oxide fuel cells (SOFCs) are one technology that may help to achieve this. However, the development of new solid-state ionic conductors (SSICs) is therefore mandatory to overcome their key limitation: the need to operate at high temperatures, above ~900 °C. The use of high temperatures reduces the energy efficiency of the SOFC due to the required large power input and furthermore increases the risk of material failure caused by mechanical degradation during thermal cycling.

Most of the currently used materials are based on oxide ionic conduction through vacancies in an oxide sublattice. Our approach focuses on rarely investigated materials with oxygen-excess; the presence of oxide ions in the interstitial sites of a crystalline structure; and the possible presence of superoxide species in the normal crystalline sites. Understanding these will give us insight about the structure-property relationship of O^{2-} conduction and provide a systematic route to enhanced performance and new materials, with improved ionic conduction at moderate temperatures (~500-700 °C).

To this end, we are investigating the solid solution of $Sr_{3-x}Y_xNbO_{5.5+0.5x}$ (x=0-1). The end member Sr_2YNbO_6 is an example of a double-perovskite where the corner sharing YO_6 and NbO_6 octahedra exhibit rock-salt like ordering. (1) Similar ordering occurs in $Sr_3NbO_{5.5}$ where the octahedral sites are occupied by Sr and Nb. (2) Through variable temperature neutron diffraction studies (between room temperature and 1000 °C) we have found that the average structures differ in terms of the cooperative tilting of the octahedra, with Sr_2YNbO_6 being monoclinic while $Sr_3NbO_{5.5}$ is cubic. We postulate that the oxygen vacancies reduce the need for octahedral tilting, and are now working to confirm this using a combination of diffraction and spectroscopic methods, the results of which we will be presenting.

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EI-2 Neutron imaging and cultural heritage

Abstract

Paper Ref: 354

Present Status of Energy Resolved Neutron Imaging System "RADEN" in J-PARC

Mariko Segawa[1], Takenao Shinohara[1], Tetsuya Kai[1], Kenichi Oikawa[1], Masahide Harada[1], Takeshi Nakatani[1], Motoki Ooi[1], Kosuke Hiroi[1], Yuhua Su[1], Joseph D Parker[2], Yoshihiro Matsumoto[2], Shuoyuan Zhang[2], Hirotoshi Hayashida[2], Hirotaka Sato[3], Takashi Kamiyama[3], HideoYokota[4], Toshihiro Sera[5], Koichi Mochiki[6], Kazuya Aizawa[1], Masatoshi Arai[1], Yoshiaki Kiyanagi[7]

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Energy Resolved Neutron Imaging System (RADEN) has been constructed at the beam line of BL22 in Materials and Life Science Experimental Facility (MLF) of J-PARC. RADEN is the first instrument dedicated to the pulsed neutron imaging experiments in the world. The commissioning of this instrument began from November 2014, and user operation started in April 2015.

The purpose of this instrument is to obtain energy-resolved two or three-dimensional neutron images effectively by utilizing pulsed neutron's nature. By neutron resonance imaging, Bragg-edge imaging and polarized neutron imaging, it is able to derive quantitative sample information, such as crystallographic structure, nuclide composition and magnetic fields, respectively. Therefore, it is designed to cover a wide neutron energy range from cold to epithermal neutrons of a few tens keV with good energy resolution. And also it is equipped with several neutron imaging devices with high time resolution and high spatial resolution to utilize the time-of-flight method. Moreover, because this instrument is intended to conduct a state-of-the-art neutron radiography and tomography measurements for scientific and industrial application, the thermal neutron intensity of 3×10^7 n/s/cm^2/MW, the maximum beam size of 300 mm, and the highest L/D value of 7500 are provided. In addition, a high performance computing system and new computed tomography reconstruction software is developed.

In this presentation, we show the current results of commissioning studies about the beam property and several neutron imaging devices, and discuss about the energy selective neutron tomography and prospects of the instrument.

Abstract

Paper Ref: 256

TOMOGRAPHIC AND ELASTIC STRAIN CHARACTERISATION OF AN ADDITIVELY MANUFACTURED TURBINE BLADE USING BRAGG-EDGE NEUTRON SPECTROSCOPY

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Characterisation of the residual strain state within engineering components and materials provides useful information about there deformation and in-service behavior. Non-destructively mapping this strain state however, presents significant challenges. The development of high resolution energy-resolved microchannel plate neutron detectors [1] is enabling new ways to extract material characteristics. A technique known as Bragg-edge neutron transmission [2] has recently been developed as a means to non -destructively evaluate the residual elastic strain state throughout the bulk.

The transmission spectrum of thermal neutrons through a polycrystalline material displays a series of well-defined jumps in intensity as a function of incident neutron wavelength, known as Bragg-edges. The positions of these Bragg-edges, provide a two-dimensional map of the average elastic strain in the incident beam direction when compared to the edge position in a ‘strain-free’ reference. By measuring the transmission spectrum for multiple sample orientations, two-dimensional information is built up about the three dimensional strain distribution. However, to recover the three-dimensional strain tensor from these lower order measurements presents significant challenges [3, 4].

Here, we present recent experimental results from the characterisation of a nickel turbine blade fabricated using additive manufacturing. The thermally driven additive processes during manufacture can generate significant strains throughout such components, knowledge of these strains is crucial to the development of the free-form additive building process. We discuss the development of neutron strain tomography [3,4] as a non-destructive method of efficiently mapping the elastic strain state of whole engineered components at spatial resolutions approaching tens of microns.

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Abstract

Paper Ref: 53

NEUTRON TECHNIQUES FOR CULTURAL HERITAGE

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Neutron methods are emerging as an innovative and attractive investigative approach to characterising Cultural Heritage artefacts without the need for sampling or invasive procedures.

Among Cultural Heritage collections conserved in national and private museums worldwide, metal artefacts of archaeological and historical interest, when properly studied, can reveal secrets of past human history. We are only allowed to treat them using the most delicate care and must avoid, as far as possible, accelerating any natural ageing process. Moreover, as analytical science evolves, we should not investigate these objects using invasive tools that might prevent further analytical techniques being applied to the same objects by future generations of scientists.

In particular white beam and energy selective neutron radiography, tomography, and laminography can be successfully used to characterise the structure, morphology and composition of metal artworks by providing detailed three-dimensional information through the reconstruction of macroscopic cross sections of the object under investigation.

From this wealth of data it is possible to obtain information on the conservation status of the artefact and reveal its manufacturing procedures, which in many cases may still be a mystery. In fact, the study of forging techniques, and their evolution over time, represents one of the most interesting topics in the investigation of metallic artefacts, which are considerably different within various cultures.

In the present work, a comprehensive study of the metallurgy of metal artefacts of historical and archaeological interest in museums was carried out with the aim of investigating the composition, assembly methods, and structural variations in such artefacts from different cultures and different periods of history.

The study has been developed in cooperation with several Museum Institutions and Neutron Facilities.

Abstract

Paper Ref: 393

WHAT KIND OF INFORMATION CAN BE OBTAINED FOR METAL CULTURAL HERITAGES BY USING PULSED NEUTRON IMAGING?

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Metal cultural heritages are one of important application fields of the neutron, since X-ray diffraction cannot see inside of the metal materials and they require non-destructive analysis. Pulsed neutron imaging has capability to measure the crystallographic information such as crystal phase, strain, crystallite size and preferred orientation although the information is average value along the neutron transmission. Furthermore, by using a 2-dimensional position sensitive detector it becomes possible to map such information in a real 2-dimensional space. Crystallographic or metallurgical analysis of metal cultural heritages, such as Japanese sword has been rarely performed since non-destructive analysis could not be applied, which limited the number of samples examined. Therefore, the characteristics of the pulsed neutron imaging are preferable for the study of metal cultural heritages.

In order to elucidate what kind of information is obtained by the pulsed neutron imaging we applied this method to the Japanese sword. We performed experiments at HUNS (Hokkaido University Neutron Source) and NOBORU at J-PARC/MLF. We obtained the transmission spectra of three fragments and one full sword. With HUNS data of the fragments we could obtain the crystallite size and preferred orientation, and the difference among the swords was clearly indicated. From the experimental data obtained at NOBORU we could deduce the crystallite size and the preferred orientation as at HUNS. Furthermore the quenched area around the edge was also indicated. From the images depending on the wavelength it was found that coarse-grains appeared heterogeneously and a large void was also identified. Position dependent change was observed.

Thus, it is indicated the pulsed neutron imaging has capability to deduce various information of the cultural heritages and the method is applicable to other cultural heritage materials.

Abstract

Paper Ref: 56

NEUTRON TEXTURE AND TOMOGRAPHIC STUDY OF ANCIENT GREEL INCUSE COIN PRODUCTION

Mr Scott Olsen [1], A/Prof Ken Sheedy [2], Ms Talia Knowles [2], Dr Vladimir Luzin [1], Dr Ulf Garbe [1], Dr Filomena Salvemini [1]

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In 2014 the Australian Centre for Ancient Numismatic Studies at Macquarie University teamed up with the Bragg Institute to try to solve a 2500 year old mystery. Shortly after the invention of money in Lydia in the late 7th century bc a number of Greek colonies began a unique coin minting method known as incuse coinage.

In existence from around 600 BC in the cities of Southern Italy (modern Basilicata and Calabria), incuse coins were known for the image on the front of the coin also appearing on the back, but in reverse. The incuse coins are much thinner and much more precisely aligned and struck than contemporary Greek coins.

With their abrupt disappearance after 150 years, little is known today about the minting technique used to produce these rare coins, though various theories have been proposed over the last century.

Using the Neutron Beam instrument Kowari for Neutron Texture analysis, and Dingo for Neutron Tomography a series of 27 incuse and non-incuse coins has been studied over the last 12 months with the aim of understating the manufacturing process.

Neutron Texture analysis of coinage from different Greek colonies indicates the incuse coins were made using a similar process and this process was significantly different to the standard coinage made on the Greek mainland at the same time.

Neutron Tomography also indicates this. Tomography allows scientists to have a unique insight into the internal structure of a coin and has generated details on porosity and inclusions which cannot be obtained elsewhere.

The most startling find is that a number of ancient coins believed to be pure silver are infact, mostly copper with a thin layer (0.4mm) of silver over the top. Tomography allows precise measurements of the thickness of the outer and inner layers and gives an idea as to their method of production.

SMS-2 Polymers

Abstract

Paper Ref: 7

Phase behavior of miscible block copolymer blends and baroplastic property

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The phase behavior of the multi-component polymer systems such as blends and block copolymers (BCPs) has been studied extensively, as it determines the chemical and physical properties of the polymeric materials in practical applications. Above all, polymer baroplasticity is a desirable property for the recycling and regenerating industries, since it allows the polymers to flow at relatively mild pressure. Particularly, the compressibility issue on the enhanced miscibility with pressure has been an attractive topic, because an ordered state of the baroplastic BCPs becomes a phase-mixed state (or disordered phase) between the two dissimilar blocks upon pressurizing. This is in contrast to the incompressibility that was dictated by the enthalpic effect arising from the increased unfavorable contacts between the two blocks by increasing pressure.

The phase behaviors of BCP blends composed of the weakly interacting (with no specific interaction) polystyrene-*b*-poly(*n*-butyl methacrylate) (PS-*b*-P*n*BMA) and deuterated polystyrene-*b*-poly-(*n*-hexyl methacrylate) (dPS-*b*-P*n*HMA) were investigated by Small-Angle Neutron Scattering (SANS) and Depolarized Light Scattering (DPLS) measurements. Interestingly, pressure dependence of various phase transitions for the miscible BCP blends was significantly changed, in which the blends consist of a PS-*b*-PnBMA and a deuterated dPS-*b*-PnHMA. To elucidate the origin and difference in baroplasticity of weakly interacting BCP blends, the pressure dependence of transition temperatures was evaluated using enthalpic and volumetric changes at phase transitions. We also demonstrate that the entropic compressibility for the miscible BCP blends is a baroplastic indicator, which was characterized by the negative volume change on mixing (ΔV_{mix}) at transitions.

Abstract

Paper Ref: 304

Is Osmotic Pressure Relevant in the Confinement of a Polymer Brush?

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Polymer brushes are highly dense arrays of polymers grafted to an interface and are powerful ways to control the forces of adhesion, lubrication and friction [1]. A key property of the brush that governs its behavior is how the structure of the brush responds to external forces and to confinement.

We have developed a unique new setup [2,3] that combines a surface force type apparatus with neutron reflection. Using this device, we measured the structure of polymer brushes under confinement. The brushes consisted of either the neutral poly(ethylene oxide), PEO, or the weakly charged poly(acrylic acid), PAA. Without confinement, both PEO and PAA brushes are found to be highly swollen with water, yielding the expected parabolic brush structure. Compression of the PEO brushes with as little as 0.5 bar of confining pressure is enough to reduce the brush to a polymer block of uniform density that is significantly dehydrated (<12% v/v of water) [4]. For the charged PAA brush (at pH 9), we observe the same transition from parabolic profile to nearly dehydrated block profile, but it occurs between 1 and 5 bars of confining pressure.

These experimental results are compared against a theoretical model based on numerical self-consistent field (nSCF) theory. Whilst the nSCF model correctly predicts the observed transition from a brush to a block profile, it predicts that we should need a confining pressure a factor of 100 greater than observed in our experiments. Looking also at SFA and AFM data, we will discuss further the role of osmotic pressure in a confined polymer brush.

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Abstract

Paper Ref: 243

Small Angle Neutron Scattering Analysis of Chitosan in different Phases

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Biopolymers are studied extensively due to its wide applications in the field of bio-technology, micro fluidics and lab on chip devices. Chitosan is natural biopolymer derived from chitin. It has wide applications in bio-medical engineering because of its biocompatibility and biodegradability. Also, chitosan act as reducing and stabilizing agent for the metal ions. Chitosan is also a good candidate in batteries as membranes. It is therefore important to study the conformational changes of chitosan. In this research work, we used SANS to understand the modifications in the radius of gyration (Rg) values of chitosan polymer in solution and in presence of $HAuCl_4$ and $LiClO_4$. Chitosan solution became a gel in the presence of $HAuCl_4$ enabling to study the associated conformational change in chitosan in gels [1]. We have also made films of chitosan, chitosan- Au and Chitosan- $LiClO_4$ and studied the associated conformational changes in chitosan. Gels and films were formed by varying the concentration of $HAuCl_4$. Lower concentration of $HAuCl_4$ gave films while higher concentration of $HAuCl_4$ gave gels. The Imaging was carried out for the samples using TEM and SEM. SANS shows that the chitosan solution (liquid) had greater Rg value than the chitosan film. The Rg value did not change in gels from that of solution. There was no correlation length for the fitting for chitosan in solution, however there was correlation length observed in the gels. This indicates that in gels the chitosan units are more localized than in solution phase. Interestingly, chitosan-Au-Li film shows the formation of star like structures which are not observed in case of gels. Hence we conclude that the presence of Lithium can induce conformational changes in chitosan films while $HAuCl_4$ can localize chitosan units in solution leading to formation of gels.

Reference

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Abstract

Paper Ref: 374

DISTRIBUTION OF MODIFIED FULLERENE IN A PHOTOVOLTAIC POLYMER MATRIX ALONG THE HYBRID FILM NORMAL AS REVEALED VIA COMBINED NEUTRON AND X-RAY REFLECTIVITY MEASUREMENTS

Mr Kuan-Hsun Lu[1], Mr U-Ser Jeng, Mr An-Chung Su, Mr Norifumi L. Yamada

[1] *National Tsing Hua University*
[2] *National Synchrotron Radiation Research Center*
[3] *National Tsing Hua University*
[4] *High Energy Accelerator Research Organization*

Organic solar cells based on blends of functionalized C_{70} ($PC_{71}BM$) and thieno-[3,4-b]-thiophene-alt-benzodithiophene copolymer (PTB7) matrix have long been investigated, especially at the blend ratio of 1:1.5 (PTB7:$PC_{71}BM$, w/w), because of the optimized power conversion efficiency (PCE) up to 9.2%. Taking advantage of the difference in scattering length density contrast, we have deconvoluted X-ray and neutron reflectivity (XRR/NR) profiles into a self-consistent in-depth distribution of $PC_{71}BM$ and PTB7. Combined with the results from simultaneous grazing-incidence wide/small-angle scattering (GIWAXS/GISAXS) and atomic force microscopy (AFM), we show the presence of porosity near the film surface, which may penetrate deep into the active layer. Upon addition of a minor amount (3 v%) of diiodooctane (DIO) to the blend solution, the surface morphology becomes significantly finer, with decreased porosity in bulk layer (3 v%) and increased PTB7 content near the surface. We attribute the correspondingly increased PCE to both the more evenly distributed PTB7 and the lowered porosity level in the active layer.

Abstract

Paper Ref: 204

Structure analyses of rubber-filler systems by using contrast variation SANS

Mikihito Takenaka[1], Shotaro Nishitsuji[1], Naoya Amino[2], Yasuhiro Ishikawa[2], DaisukeYamaguchi[3], and Satoshi Koizumi[3]

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Rubber-filler systems have been one of the most successful composite materials and have been widely used in industry such as tire and belts so on. Fillers reinforce rubbers by compounding and improve the mechanical and barrier properties of the rubber compounds. We need to explore the adsorption of rubber around filler particles in terms of structure analyses to clarify the correlation between the mechanical properties and the structures for rubber-filler systems. In this study, the polymer layers absorbed on silica particles in rubber-silica systems have investigated with contrast variation SANS method. Specimens were swollen by the solvents having various scattering length densities and measured their SANS intensity. We calculated the partial scattering functions by using singular value decomposition: the scattering function for polymer-polymer correlation $S_{PP}(q)$, the scattering function for silica- silica correlation $S_{SS}(q)$, and the scattering function for polymer-silica correlation $S_{PS}(q)$ as shown in Fig.1. The analyses of $S_{PS}(q)$ and $S_{SS}(q)$ explored the existence of dense polymer layers around silica aggregates. $S_{SS}(q)$ reflects hierarchical structures formed by silica particles. To characterize the adsorption layer quantitatively, we calculated the scattering functions for the model consisting of the aggregation of Silica particles, the adsorption layers on the silica particles and the matrix region. We use the mass fractal model having upper limit for the aggregation of Silica particles, and object with sharp interface for the regions adsorption layers and silica aggregates to calculate the partial scattering functions. The model can well express the experimental partial scattering functions and several characteristic parameters are estimated from the analyses, such as the size of aggregates, the thickness of layers, the volume fractions of polymer of layers and matrix, and the correlation length of the matrix network. The contrast variation SANS is found to be a powerful tool of the analyses of the structures of the rubber-filler systems.

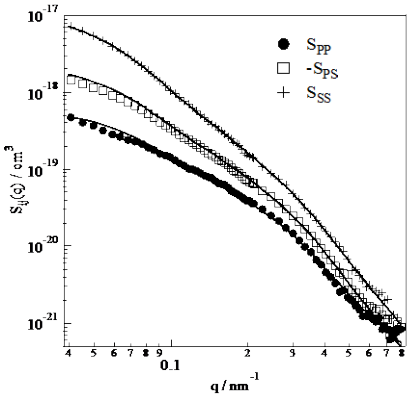

Figure 1 Partial scattering function of rubber-filler systems and their fitting results

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CMP-3 Magnetism

Abstract

Paper Ref: 455

SPIN ICE

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Spin ice is a geometrically frustrated magnetic system possessing pyrochlore lattice structure, with magnetic moments residing on the vertices of the corner-sharing tetrahedral network, and obeying the two-in two-out ice-rule Ising interactions as analogous to the water ice. The excited states of spin ice can be modeled as emergent magnetic monopoles. While a system with the additional transverse magnetic correlations to the Ising Hamiltonian term, quantum spin ice is nominated. In this talk, I will introduce our studies of the classical spin ice and quantum spin ice by using neutron scattering, μSR, and other techniques.

Abstract

Paper Ref: 362

Long-range-ordered magnetism in P2-type layered oxides with honeycomb lattices

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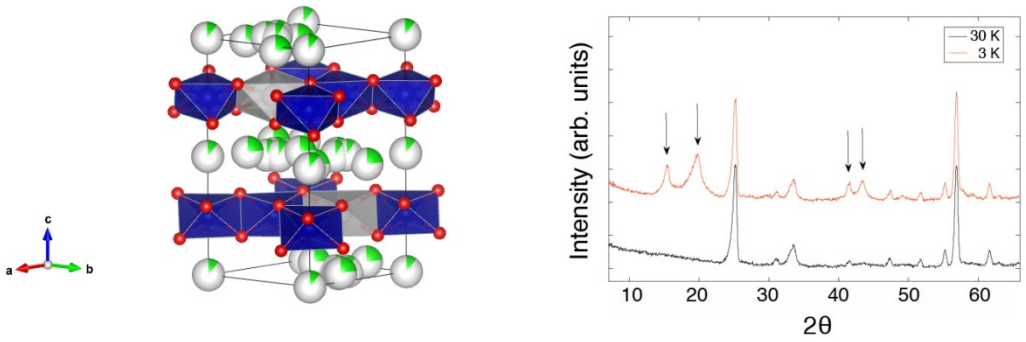
Transition metal oxides that are capable of intercalating small mobile ions such as Li^+ and Na^+ have been intensively studied for their electrochemical properties, but their magnetic properties remain relatively unexplored. Those with CdI_2-type oxide layers such as $LiCoO_2$, which are the most interesting for metal-ion battery cathode applications, have quasi two-dimensional triangular arrays of transition metals which are not conducive to long-range magnetic ordering (although they still show interesting properties, the most notable example being the superconductor $Na_xCoO_2{\cdot}yH_2O$).

When these compounds are modified so that 1/3 of the transition metal sites contain non-magnetic cations, an ordered hexagonal honeycomb lattice can result. This topology does support long-range magnetic order of various types, including antiferromagnetic (AFM) structures. A significant number of such compounds have been reported, and where magnetic properties have been measured, many of them show evidence for long-range AFM ordering. [2-3 references] Here, we present the results of the first low-temperature neutron diffraction studies of long-range magnetic structure for a series of honeycomb compounds with Te^{6+} or Sb^{5+} as the non-magnetic cation. The work presented will focus on P2-type (rather than the more common O3-type) layered compounds.



(Left) The honeycomb structure of $Na_2Co_2TeO_6$; Na = green, Co = blue, Te = grey, O = red. (Right) Neutron powder diffraction data above and below the magnetic transition temperature of 18 K reported by Viciu et al. [ref], showing long-range AFM ordering.

[1] W. Miiller *et al.*, *Journal of the American Chemical Society* **134**, 3265-3270 (2012)
[2] Z. Huang *et al.*, *Angewandte Chemie – International Edition* **53**, 3414-3417 (2014)

Abstract

Paper Ref: 258

COMPLEX MAGNETISM OF QUASI-1D MARICITE-TYPE $NaFePO_4$

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We recently reported the magnetic structure of maricite-type $NaFePO_4$ determined using neutron powder diffraction data collected at 3 K[1]. The crystal structure of this compound is derived from the olivine (Mg_2SiO_4) type by an ordered distribution of Na and Fe over the two inequivalent Mg sites in the olivine cell. This leads to a magnetically quasi-1D arrangement in which edge-sharing (FeO_6) chains are connected to each other only via phosphate groups (Fig. 1) with a shortest interchain Fe-Fe distance of ~5 Å vs. intrachain distance of ~3.4 Å.

Here we report the results of further studies using magnetometry, heat capacity, Mossbauer, and variable field and temperature powder and single crystal neutron diffraction measurements, which reveal not only an intermediate incommensurate magnetic phase existing in zero field within a very narrow interval of ~2 K, but also a metamagnetic transition around 5 T (at 2 K). We will also present and discuss the evolution of the magnetic structure of $NaFePO_4$ as a function of temperature and magnetic field in connection with the crystal structure and compared to that of other maricite type compositions such as $AgMnVO_4$[2].

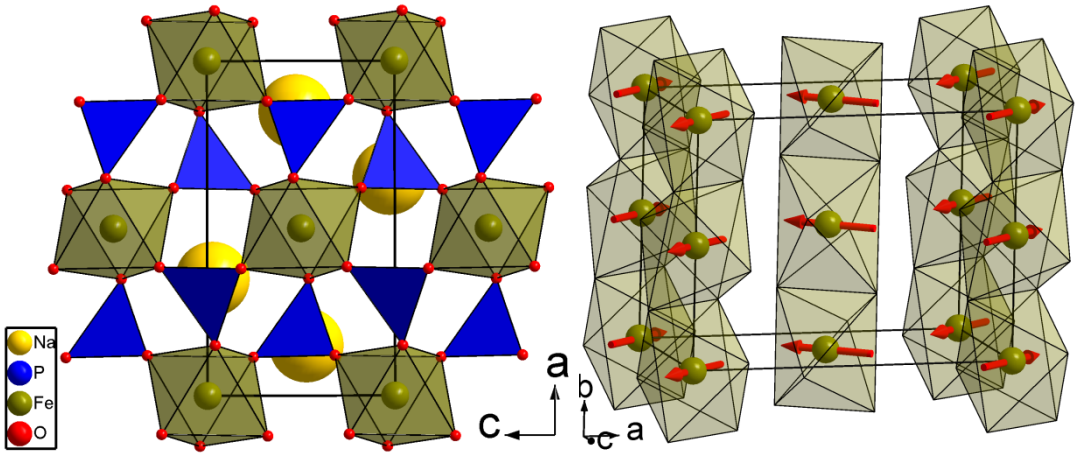

Figure 1. Crystal structure of maricite-type $NaFePO_4$ (left) and zero-field magnetic structure at 3K (right).

1. M. Avdeev; Z. Mohamed; C. D. Ling; J. Lu; M. Tamaru; A. Yamada; P. Barpanda, *Inorg. Chem.* **2013,** 52, (15), 8685-8693.
2. H. B. Yahia; M. Shikano; E. Gaudin; M. Avdeev; C. D. Ling, *J. Solid State Chem.* **2015,** 221, 306-310.

Abstract

Paper Ref: 148

A single crystal study on magnetic structure of NiS_2

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The Mott metal-insulator transition (MIT) has been at the forefront of condensed matter physics for decades. Despite this, even some of the simplest materials have a lot of things to be done. We are focusing on the cubic pyrite crystal NiS_2. It has been recognized as an insulator-metal transition material driven by pressure and chemical substitution using Se. In reported phase diagram, the magnetic structure apparently has close connection with MIT on $NiS_{2-x}Se_x$ system because the magnetic structures are different between metal and insulator phase. However, the magnetic structure of NiS_2 has not been resolved yet due to lack of knowledge about the magnetic point group even by using neutron polarization analysis technique. There are two magnetic phase transitions observed, one at T_{N1} = 39.2 K with a magnetic structure (the M1 structure) with a propagation vector of k = (0 0 0). The M1 has a non collinear magnetic ordering of the Ni fcc lattice. It was concluded that the magnetic moments of Ni at the 4a sites {(0 0 0), (1/2 1/2 0), (0 1/2 1/2), (1/2 0 1/2)} have directions of {[111], [1-1-1], [-11-1], [-1-11]} respectively with the magnitudes of ~1.0 μ_B. The other is at T_{N2} = 29.75K, NiS_2 was observed with a propagation vector of k = (1/2 1/2 1/2) (the M2 structure). We think this M2 structure is controversial.

Our recent neutron powder diffraction experiment was taken at NOMAD, SNS. At T = 2K, both M1 and M2 structures were observed. An initial determination of both magnetic structures is given. However, our powder diffraction study was restricted to measuring only 4 magnetic peaks. Of these, two were from the M1 magnetic structure, and two are from the M2 magnetic structure. To prove our magnetic structure models, now we are executing the detailed magnetic structure analysis by using single crystal sample on Wombat and Koala at ANSTO, which will be presented.

Abstract

Paper Ref: 47

EVIDENCE OF A SUPERDENSE NONMAGNETIC CO LAYER AT THE TOP OF A THIN CO FILM ON Si(111): A POLARIZED NEUTRON REFLECTIVITY STUDY

Ms. Nasrin Banu [1], Dr. Surendra Singh [2], Dr. Saibal Basu [2], Dr. Anupam Roy [3], **Professor Bhupendra Dev [1]**

[1] *Indian Association for the Cultivation of Science*

[2] *Bhabha Atomic Research Centre*

[3] *University of Texas at Austin*

A thin cobalt film (25 nm) was deposited on a HF-etched Si(111) substrate in high vacuum by e-beam evaporation. Upon exposure to air the film surface got oxidized. We have carried out polarized neutron reflectivity (PNR) experiment on this cobalt film using the experimental set up in DHRUVA, Bhabha Atomic Research Centre. Fig.1 shows the PNR results. The topmost layer (~2 nm) is cobalt oxide. Interestingly, the PNR data indicate the presence of a high density nonmagnetic material at the oxide/Co interface. The Co layer below this high density layer has magnetic moment as expected for Co. X-ray reflectivity (XRR) experiment (Fig.2) confirms the high density of this nonmagnetic layer, which is ~4 nm thick. The density of this layer is ~1.4 times that of underlying normal Co. This nonmagnetic layer cannot be a cobalt oxide layer, as the density of an oxide layer would be even smaller compared to normal Co. Rutherford backscattering spectrometry experiment has ruled out the presence of any element of higher mass than Co. XRD results have shown, in

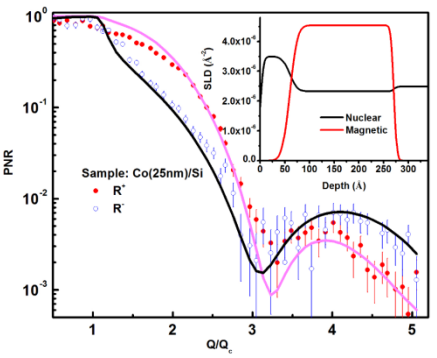

Fig.1

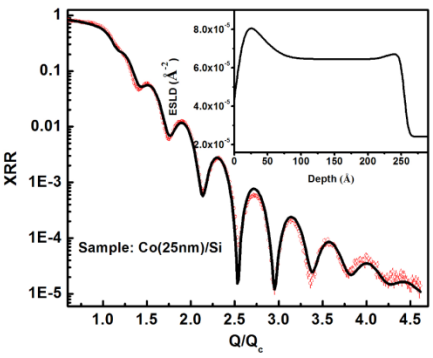

Fig.2

addition to the main Co peak, a tiny peak corresponding to ~13% smaller planar spacing. This is consistent with ~ 1.4 times density enhancement. All these results confirm that this high density layer is a Co layer with a density of about 1.4 times that of normal Co. To our knowledge, there is no experimental report on such a high density Co at ambient pressure. A DFT calculation predicts a nonmagnetic fcc Co phase with a density of about 1.4 times the normal Co density [1]. Our observed high density layer is likely to be such a superdense nonmagnetic Co layer. How could such a superdense Co layer form? Our Co film is grainy. During the growth of a grainy film, atoms could diffuse into grain boundaries producing a compressive stress [2], which may be the reason for the formation of the superdense Co layer.

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[2] E. Chason et al., Phys. Rev. Lett., 88 (2002) 156103.

MSC-4 Energy materials and spin order

Abstract

Paper Ref: 443

Local structure study of energy materials using neutron powder diffraction

Seongsu Lee[1]

[1]Neutron Science Division, Korea Atomic Energy Research Institute, Daejeon, Korea

Structure information of electrode materials obtained by neutron power diffraction is very expedient to understand the conduction mechanism of second battery. Data about a local structure change such as a position and occupation of conduction ion give us a clue for realizing the next generation second battery with high-capacity, high-efficiency and good-safety. To analyze neutron powder diffraction data, combining with Rietveld refinement and Maximum Entropy Method (MEM) is very useful to fully understand a local structure change of electrode. A local-structural change in electrode materials is observed when the electrode materials have been experienced the charging/discharging, doping, particle size control and spin ordering process etc. It is possible to design a beyond battery material with high performance by controlling a local-structure as functions of several external conditions. In this talk, we will introduce the crystal and magnetic structure studies of battery materials using high resolution neuron powder diffraction (HRPD) in HANARO. For examples, we have investigated the magnetic and crystal structure of $Na_xFe_3(PO_4)_2(P_2O_7)$ ($1 \le x \le 4$) with a mixed polyanion framework using neutron powder diffraction. The new compounds contained three-dimensional (3D)-sodium paths supported by P_2O_7 pillars in the crystal. The de/sodiation of the $Na_xFe_3(PO_4)_2(P_2O_7)$ electrode occurs via a one-phase reaction with a reversible Fe^{2+}/Fe^{3+} redox reaction and accompanies an exceptionally small volumetric change of less than 4%. Very interestingly, we have observed a very strong spin-lattice coupling behavior in this compound. Even though, the spin ordering of this material is happened at a low temperature, the trials to understand a strong correlation between spin and lattice within electrode materials are valuable to open an opportunity which realizes the new concept's second battery to be controlled the conduction property of electrode by the spin ordering.

Abstract

Paper Ref: 77

IN SITU NEUTRON DIFFRACTION STUDIES OF LITHIUM-ION BATTERIES: LITHIUM DISTRIBUTION AND TEMPERATURE DEPENDENCE

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[3] *New Energy and Materials Laboratory (NEML), Department of Chemistry, Fudan University, Shanghai 200433, China*

Electrochemical energy storage devices in the form of batteries are ubiquitous in society, used in everything from children's toys to mobile electronic devices, providing portable power solutions. There is a continuous drive for the improvement of batteries. A large proportion of the function of batteries arises from the electrodes, and these are in turn mediated by the atomic-scale perturbations or changes in the crystal structure during an electrochemical process (e.g. battery use). Therefore, a method to both understand battery function and improve their performance is to probe the crystal structure evolution *in situ* while an electrochemical process is occurring inside a battery.

Our work has utilized the benefits of *in situ* neutron diffraction (e.g. sensitivity towards lithium) to literally track the time-resolved evolution of lithium in cathode materials used in rechargeable lithium-ion batteries. With this knowledge we have been able to directly relate electrochemical properties such as capacity and differences in charge/discharge to the content and distribution of lithium in the cathode crystal structure. The first part of this presentation will cover our lithium-centred work on $Li_{1+y}Mn_2O_4$ electrodes. The second part will detail our efforts to probe battery function at different temperatures. This essentially adds another parameter that can be varied during *in situ* studies.

Such *in situ* experiments that probe the time-resolved structural evolution during charge/discharge provide unparalleled insight into how electrodes evolve. Using this information a comprehensive atomic-scale picture of battery functionality can be created and permutations can be made to the electrodes that optimize battery performance.

Abstract

Paper Ref: 361

Evaluation of the Molecular Dye Orientation in Functioning Dye-sensitised Solar Cells (DSCs) by Neutron Reflectometry

Jonathan McCree-Grey[1,2], Jacqueline M. Cole[2], Stephen A. Holt[1], Peter J. Evans[3]

[1] *Bragg Institute, ANSTO, Sydney, Australia*
[2] *Cavendish Laboratory, University of Cambridge, UK*
[3] *Institute of Materials Engineering, ANSTO, Sydney, Australia*

Since 2007, global photovoltaic energy production has increased tenfold[1] and its predicted market value is estimated to reach $155 billion by 2018.[2] However, the energy- and cost-intensive processes required for the production of conventional solid-state devices has created a demand for cheaper and more environmentally friendly alternatives, such as dye-sensitised solar cells (DSCs).

Since 1991,[3] DSCs have been intensively investigated and laboratory devices have since accomplished a conversion efficiency of 13%.[4] Typical DSCs consist of a molecular dye adsorbed onto a semiconductor, surrounded by a redox electrolyte (*e.g.* I^-/I_3^-) and positioned between two transparent conductive oxide substrates. The dye is the principal light absorber, injecting photo-excited electrons into the conduction band of the semiconductor, thus giving rise to the electrical characteristics of the cell. Even though the structure, orientation, and surface packing density of dyes at the TiO_2 interface may affect electron injection and bulk TiO_2 surface passivation processes, and thus influence the overall cell performance, details about these key parameters still remain limited.

X-ray reflectometry has already been successfully employed to measure these properties[5], however typical laboratory instruments are limited to studies at the solid-air interface. The application of neutrons by the authors, as will be presented in this talk, has allowed development of the technique to a solid-liquid environment wherein the dyes are surrounded by a solvent containing the redox electrolyte. Modifications to the sample environment have enabled measurements *in operando*, while the cell is exposed to photo-irradiation illumination, along with *in situ* measurements of the cellular I-V characteristics. Results have revealed subtle differences in dye structure at open-circuit voltage and short-circuit current and given some insight into the influence of the electrolyte on dye-layer structure, but also highlighted the current limitations of neutron reflectometry in measuring these systems.

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Abstract

Paper Ref: 180

Using quasi-elastic neutron diffraction to study positive electrode for lithium and sodium-ion batteries

James C. Pramudita[1], Neeraj Sharma[1]

[1] *University of New South Wales, Sydney, Australia*

Sodium-ion batteries has recently been proposed as the alternative for lithium-ion batteries to be the low cost energy storage system.[1] However, challenges still remains for the development of sodium-ion batteries. Optimization of electrode materials and electrolyte capable of insertion/extraction of sodium-ion in a safe and economic way under high current density is needed in order to produce commercially viable sodium-ion batteries.[1, 2] While possible positive electrode material is more prevalent than negative electrode material, many of these material still need further understanding.[1]

Quasi-elastic neutron scattering is a technique that utilize the inelastic neutron scattering that can be used to study solid-state diffusion in materials.[3] This technique can be used to study the diffusion of sodium-ion under electric field through the electrolyte and positive electrode materials in order to further understand the mechanism of sodium insertion/extraction in a working battery. This technique can also be used to study available positive electrode material for lithium-ion batteries to further understand the mechanism of lithium-ion diffusion in current working lithium-ion batteries.

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Abstract

Paper Ref: 132

ELECTRONIC STRUCTURE CHARACTERIZATION OF A SERIES OF $[Mn_3O]^{7+}$ CORE SINGLE MOLECULE MAGNETS BY INELASTIC NEUTRON SCATTERING

Dr Marc Sigrist [1,2,3], Dr Kasper Pedersen [2,4], Mikkel Sørensen [2], Dr Hannu Mutka [1], Prof. Jesper Bendix [2]

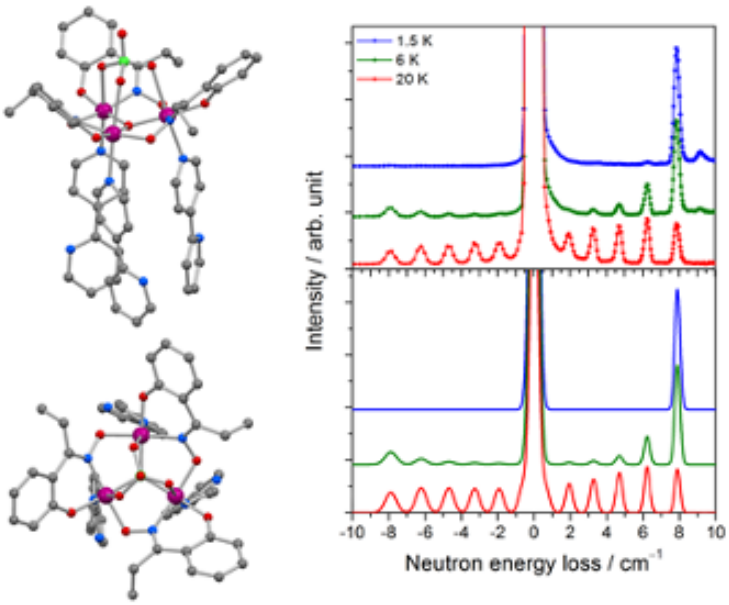
[1] *Institut Laue-Langevin, France*

[2] *Institut of Chemistry, University of Copenhagen, Denmark*

[3] *Academia Sinica, Taiwan*

[4] *CNRS, France*

Since the discovery of Mn_{12} and the characterization of its magnetic properties, synthetic chemists have put a lot of effort into the search of new single molecule magnets (SMMs). This has been accompanied by the work of spectroscopists and theoreticians who try to identify the magneto-structural correlations that lead to strong SMM behaviour. Recently several research groups were able to identify the exchange interactions in Mn_6 and Mn_3 complexes. So far the detailed spectroscopic study of the Zero-Field-Splitting (ZFS) in these SMMs has been widely neglected. In the present study we analyzed the Ground State (GS) splitting of several $[Mn_3O]^{7+}$ core SMMs by Inelastic Neutron Scattering (INS). By aligning the local ZFS axes on the manganese (III) ions with respect of the easy axis of the complex it is possible to increase the global anisotropy and thus increase the relaxation barrier. INS allowed us to measure all energy levels within the *S*=6 GS multiplet allowing not only to extract the ZFS parameter *D* but also higher order terms as well as eventual small rhombic anisotropy originating in symmetry breaking.



Left: Structure of $Mn_3O(Et\text{-}sao)_3(2,4'\text{-}bipyridine)_3ClO_4$ viewed perpendicular to and along the trigonal axis. right: INS measurement and simulation with 6.5 Å incident wavelength

EI-3 Microstructure, thermomechanical and stress

Abstract

Paper Ref: 101

Applications of Neutron Diffraction in Granular Mechanics

Dr Chris Wensrich[1], Prof Erich Kisi[1], Dr Vladimir Luzin[2], Dr Oliver Kirstein[1,3], Dr JianFeng Zhang[1]

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[2] *Bragg Institute, ANSTO, Kirrawee, Australia*
[3] *European Spallation Source, Lund, Sweden*

Granular materials represent a unique state of matter that demonstrate a range of complex behaviours depending on the situation. In dense form, the mechanical response of a particle assembly is not only dependent on the applied load, but also on the load and deformation history. At a particle level, the stochastic nature of these materials and the micro-mechanics of load-sharing give rise to significant inhomogeneity in the form of 'force chains'. These linked arrays of particles evolve with deformation, forming a skeleton that supports the majority of the applied load. This rich nature at various length scales has spawned a significant amount of interest in the scientific and engineering communities.

The penetrating power of neutrons provides the opportunity to look inside and examine the mechanical behavior of these systems. Results from a number of diffraction based strain measurement experiments within granular assemblies are presented in this paper. These results span a range of relative scales, from an examination of individual particle stress within a discrete assembly; to the bulk response of powders undergoing high-stress compaction processes. The results serve to demonstrate the potential for neutron based strain measurement in the study of granular systems.

Abstract

Paper Ref: 99

METALS UNDER THERMO-MECHANICAL PROCESSES: IN-SITU NEUTRON DIFFRACTION VERSUS SYNCHROTRON HIGH-ENERGY X-RAYS

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[1] *Australian Nuclear Science and Technology Organisation, Lucas Heights, Australia*
[2] *University of Wollongong, Wollongong, Australia*

Both neutron and synchrotron high-energy X-rays have penetrating power into metals, and intensities are competitive for ex- and in-situ studies of thermo-mechanical processes. They bear great potential in order to speed up materials design by orders of magnitude. Although it is well recognized that both neutrons and synchrotron radiation need to be combined, the user groups are still pretty distinct. The present contribution will upraise novel pioneering experiments on selected metal systems and showcase the complementarity. Neutrons bear the advantage of averaging over larger volumina and therefore, are less dependent on grain statistics, leading to good, quantitative phase analysis and texture measurements. The contrast, different to X-rays has been employed to investigate order-disorder transitions in titaniums aluminides, and dynamical theory of diffraction effects lead to the study of smallest distortions, and their kinetics at high temperature. Synchrotron X-rays allow to focus on a small number of crystallites, showing up traces of grain evolution in reciprocal space, such as grain rotation, grain growth, phase correlations, dynamic recovery and recrystallization, like in a Materials Oscilloscope.

Abstract

Paper Ref: 312

Design of the new residual stress diffractometer at CARR

Xiaolong Liu[1], Songbai Han[1], Yu Wang[1], Yuqing Li[1], Meijuan Li[1], Yuntao Liu[1], Dongfeng Chen[1], Jue ZHONG[2]

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Neutron diffraction technique is the only nondestructive method to map 3-D residual stress field in bulk component, which can help to improve the manufacturing quality of engineering components and to optimise design criteria in applications. Because of its great potential in industrial application, a new residual stress diffractometer at CARR is being built by CIAE and CNU. Its overall optics was designed and optimised by simulation using McStas and SIMRES software. It will be equipped with double focusing Si(400) monochromator, heavy weighted sample stage, large area Detector and two-layer wedges that open the shielding, which will make this instrument to be one of the best neutron stress diffractometers in the world. Also this diffractometer will be qualified with the function of texture measurement.

Abstract

Paper Ref: 233

Neutron Diffraction Method Towards Residual Stress On Stiffener–Web- Flange Joint Of Eccentrically Braced Frame (EBF) Link Element

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This research focused on the experimental study on the residual stress resulted from the welding process in and around k area stiffener joint, web and flange plates of link element. The measurement was done with the Neutron Diffraction Method using the Neutron Diffractometer DN1-M PSTBM BATAN. A number of 15 points with 45 measurement directions were conducted on the k area in normal, transversal and longitudinal directions. The result of the experiment showed 185 Mpa and 160 Mpa residual stress magnitude in longitudinal and transversal directions in the same directions with the weld toe welding between the web and flange with the extent of the stress region 8 times of the flange thickness. The perpendicular welding of the weld toe resulted in 162 Mpa and 145 Mpa residual stress in longitudinal and transversal directions with 4 times of the extent of stress region from the flange thickness. The residual stress magnitude and the width of stress region indicated the k area as a vulnerable area to the initial crack occurrence which may decrease the link performance. Creating a welding gap 4 to 5 times of the flange between the stiffener and the flange may increase the link performance because it may minimize the residual stress magnitude and distribution.

Key words: k area, residual stress, link, Neutron Diffraction, Eccentrically Braced steel Frame Structure (EBFS).

SMS-3 Polymers and surfaces

Abstract

Paper Ref: 33

PROBING FILM STRUCTURE IN ORGANIC OPTOELECTRONIC DEVICES WITH NEUTRONS

Professor Paul Burn [1], Dr Andrew Clulow [1], Prof Ian Gentle [1], Prof Michael James [2,3], Prof Paul Meredith [1], Dr Andrew Nelson [2]

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[2] *ANSTO, Australia*
[3] *Australian Synchrotron, Australia*

Controlling the structure of the active film in organic optoelectronic devices plays an important role in optimising their performance. Neutron techniques such as neutron reflectometry and small angle neutron scattering are important methods for studying the physical structures of (macro)molecules and their interactions non-destructively in solution and/or the solid state. In this presentation we will illustrate how neutron scattering measurements can be utilized to elucidate the structure of thin films typically used in organic photovoltaic devices. The presentation will focus on bulk heterojunction thin films comprised of conjugated polymer:fullerene blends. We will also show how time resolved measurements can be used to follow the evolution of the film structure during post-deposition processing. Finally, the structure of the films will be correlated to device performance.

Abstract

Paper Ref: 131

Swelling Kinetics and Structure of Thin Poly(methyl methacrylate) Films in Methanol-Water Mixture Studied by Optical and Neutron Reflectivity

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Thin polymer films have been used in a wide variety of applications in liquids such as solid electrolytes and separator films for cells, liquid filtration membranes, biochips for tailor-made diagnosis, contact lenses, stent coatings and so on. Although the liquid should be a non-solvent for the polymer, it could alter aggregation states and dynamics of polymer chains in the thin film, depending on the interaction between them. This is simply because the liquid molecule could be sorbed into the polymer film. Thus, to design highly functionalized polymer interfaces with a liquid, it is quite important to study the sorption kinetics of liquid molecules into the polymer thin films. In this study, we have examined the time evolution of thickness for poly(methyl methacrylate) (PMMA) in water-methanol mixture by optical and neutron reflectivity measurements. The former measurement indicates that the sorption of methanol or methanol rich component occurred at first, and then, the sorption of water or water rich component proceeded after the methanol sorption. The latter measurement reveals that, at a quasi-equilibrium state, the water fraction of the adsorbents in the film was higher than that of the mixed solvent contacting with the film.

Abstract

Paper Ref: 139

Controlling Hierarchical Structures in Organic Solar Cell Using π- π Interactions and Their Analysis Using SANS and PSoXS

Jongkuk Ko[1], Subhrangsu Mukherjee[2], Jiyun Song[3], Tae-Hwan Kim[4], Young-Soo Han[4], Changhee Lee[3], Harald Ade[2], Kookheon Char*[1]

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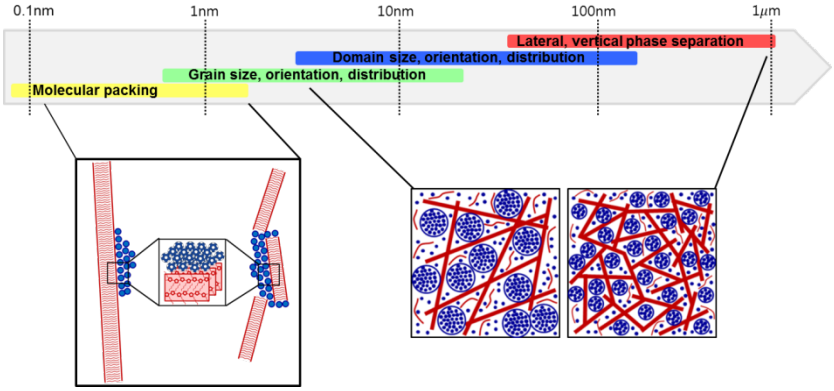
Controlling the morphology of bulk-heterojunction (BHJ) films in organic photovoltaics (OPVs) is critical in overcoming inherent drawbacks (short exciton diffusion length and low charge mobility) of organic materials and achieving optimal device efficiency. Taking into account the energy conversion process, it is important to understand the hierarchical structure in organic solar cells ranging from molecule-scale and domain-scale to device-scale structure to realize efficient charge separation, transfer, and extraction. However, most of research has so far focused on the domain-level structure such as crystallinity, domain size, and domain orientation because there lacks methods to control hierarchical structures and systematic studies on it. In the present study, we report a new approach to control hierarchical structures in organic solar cells, which was characterized by PSoXS and SANS and their relationship with device performance. We could induce highly anisotropic P3HT crystalline structures (P3HT nanowires), which were grown in a direction of pi-pi stack, facilitating charge transport, through adding poor solvents for P3HT. In addition, the anisotropy of P3HT nanowire was controlled by utilizing additives which have different affinity with PCBM. By employing controlled P3HT nanowires having different anisotropy and following thermal annealing process, we could control the molecular orientation of P3HT at PCBM interfaces (molecular-scale), domain sizes of P3HT and PCBM (domain-scale) and network structures (device-scale). Based on our new method, we could effectively control the hierarchical structures of organic solar cells with an overall view on the relationship between multiscale nanostructure and device performance.



Abstract

Paper Ref: 242

Small Angle Neutron Scattering Analysis of Chitosan- Silver Nanocomposite Films

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Recently biopolymers are extensively studied for the development of solid polymer electrolytes. Studies relating to interaction of nanoparticles with biopolymers are essential to furthering research in solid polymer electrolytes. In our research we have tried to understand the interaction between chitosan and silver nanoparticles during reduction process. Chitosan is linear polysaccharides obtained from the deacetylation of chitin. It is soluble in acidic medium and the D-glucosamine unit gets protonated leading to poly cationic polymer. It has wide potential applications in medical, water engineering, sensors, and in micro device fabrication due to its unique physiochemical properties including biocompatibility and non toxicity [1]. It also acts as both reducing and stabilizing agent for the metal ions via ion pair interaction. Silver nanoparticles are used as filler due to its well-known effectiveness in biomedical, electronic, catalytic and optical applications [2]. In this research we have synthesized chitosan - silver films. The films were synthesized by solution casting method and polymer films of thickness ~ 100 µm were obtained. The chitosan - silver films were also dipped in hydrazine hydrate for comparison. Various characterizations techniques such as XRD, FTIR, SEM, TEM and SANS were used. SEM and TEM showed structural modifications in the chitosan- silver film which is enhanced when dipped in hydrazine hydrate. Interestingly, Small angle neutron scattering (SANS) showed that fractals were formed in the chitosan- silver films both dipped and undipped in hydrazine hydrate. Electrical characterization studies showed that the conductivity was affected due to formation of silver nanoparticles. These films have potential application in solid batteries.

Reference

1. R.A.A. Muzzarelli, C. Muzzarelli (2005), Chitosan Chemistry: Relevance to the Biomedical Sciences, *Advances in Polymer Science*, **186**, 151-209.
2. Mansor Bin Ahmad, Mei Yen Tay, Kamyar Shameli, Mohd Zobir Hussein and Jenn Jye Lim (2011), Green Synthesis and Characterization of Silver/Chitosan/Polyethylene Glycol Nanocomposites without any Reducing Agent, Int. J. Mol. Sci., **12**, 4872-4884.

Abstract

Paper Ref: 210

A NEWLY DEVELOPED SIMULTANEOUS SANS/FTIR MEASURING SYSTEM FOR STRUCTURAL STUDY ON POLYMER SOLID STATES

Dr Fumitoshi Kaneko [1], Mr Naoki Seto [1], Mr Shuma Sato [1], Dr Aurel Radulescu [2], Dr Maria M Schiavone [2], Dr Jürgen Allgaier [2], Prof Koichi Ute [4]

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Small angle neutron scattering (SANS) is a powerful and convenient method to investigate the higher order structure of static polymer systems. SANS has increased its presence even in the research concerning time-dependent structural evolution. Although we have employed time-resolved SANS (TR-SANS) for the study on syndiotactic polystyrene (sPS) cocrystals so far, exploiting the large difference in molecular scattering length between fully deuterated and protonated compounds, recently we have tried to develop a new measuring system, which combine TR-SANS with the merit of FTIR spectroscopy, i.e., high potential of quantitative analysis of chemical constituents and high sensitivity to local structures such as chemical structure and molecular conformation, to enrich structural information and interpret SANS data in a more reliable manner [1]. In this paper, we would like to introduce the simultaneous time-resolved SANS and FTIR (STR-SANS/FTIR) system and its application to sPS cocrystals.

Fig 1(left) shows schematically our STR-SANS/FTIR measurement system, composed of an FTIR spectrometer and our own designed optical system, which enables the IR radiation and the neutron beam to impinge the sample film coaxially. The device was installed on a small-angle neutron diffractometer KWS2 at MLZ, as shown Fig. 1 (middle). The structural changes in a cocrystal of deuterated sPS (d-sPS) with triethylene glycol dimethyl ether (TEGDME) during the course of heating were followed with this system. Fig. 1(right) reproduces the changes in SANS 2D images and FTIR spectra measured in parallel. As for SANS images, the two lamellar reflections gradually decreased in intensity as the temperature increased and then increased slowly above 100℃. The infrared bands due to TEGDME significantly decreased in intensity, whereas the bands due to d-sPS remained almost unchanged. The information obtained by combining the SANS and FTIR data will be presented at the conference.

[1] F. Kaneko et al. Chem. Lett., 2015, in press. doi:10.1246/cl.141179.

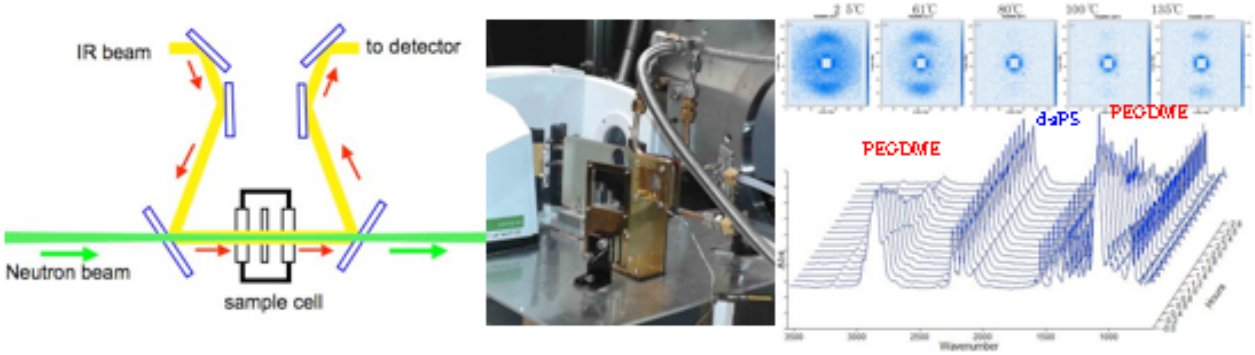

Fig. 1. Simultaneous SANS/FTIR measurement system and data obtained with this system. Left: basic design, Middle: overview, and Right: changes in SANS profile and FTIR spectrum.

Plenary Lecture 4

Abstract

Paper Ref: 457

THE NEXT GENERATION NEUTRON SOURCE ESS: WHAT IS NEW AND WHAT IS NOT

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ESS is on track to start operation in 2019 as the first long pulse spallation source in history, i.e. a bit more than 25 years after the long pulse concept was introduced. In view of the usual long lead times for funding decisions on new facilities, being based on quarter of century old concepts is commonly boasted as novelty. In contrast to most other large scale projects, ESS has in addition the key feature that it requires now new hardware technology. It makes instead radically innovative use of established technologies, offering ESS an unusually high predictability compared to projects that require(d) cutting edge developments (e.g. LHC, ITER,…). Conventional pulsed spallation sources use linear accelerators to create typically ms long proton pulses, which are then compressed by ring accelerators to less than 1 μs length, with or without further acceleration. The higher efficiency and performance of the long pulse source is achieved by suppressing the ring accelerator and related extra complexities. The savings are used to make the linear accelerator deliver more protons for producing more neutrons. Thus even with identical moderators, the ESS neutronic performance in terms of useful thermal and cold neutrons delivered to the samples is close to an order of magnitude higher at identical resolution parameters than that of the about cost equivalent SNS.. The key neutron scattering experimental capability readily achieved at ESS by reactor type mechanical chopper systems is the unrestricted potential of pulse shaping, i.e. cutting out short pulses with a great flexibility of length from the long pulses provided by the accelerator. The way to do this has been established many years ago including implementation using reactor source instruments and is now routinely utilized at one of the very successful spectrometers of J-PARC. The main recent novelty is the development of low dimensional moderators which allow us to increase the extracted cold neutron brightness by a factor of up to 4 – 6. This development follows up the far reaching innovation of using large volume para Hydrogen moderators at J-PARC. The talk will focus in more detail on the innovative use of neutron research techniques well established at neutron sources preceding ESS, prominently including those in Asia-Oceania.

Wednesday 22 July 2015

Neutron Science Facility Session

Abstract

Paper Ref: 58

Research and the User Program at OPAL, the new Australian Research Reactor

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Australian science entered a new “golden age”, with the startup of bright new neutron and photon sources in Sydney (2006) and Melbourne (2007), respectively. The OPAL reactor and the Australian Synchrotron can together be considered the greatest single investment in scientific infrastructure in Australia’s history. Fuel was loaded into the OPAL reactor in August 2006, and full power (20MW) achieved in November 2006. The formal user commenced in 2007, and fully analysed data sets have now been taken on eleven instruments. At the time of the conference, the 16th proposal round will be open. 2 further instruments are in various states of construction, and substantial additional investment is also being made in sample-environment, extra instrumental options, polarised-neutron technology, and both chemical and bio-deuteration facilities. An update will be given on the status of OPAL, the performance of its thermal and cold neutron sources and instruments, together with a selection of recent scientific results and future plans.

Abstract

Paper Ref: 454

Current Status of J-PARC MLF

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The facility has restarted since 26th February 2015 after the fire in the second experimental hall in MLF. The continuous 400-kW proton beam operation for the user operation has started from 10th March and the power will increase to 600-kW in the next summer.

The facility was suffered from the devastating disaster happened in March 2011. However, dedicated recovery work has been done and the facility started operation since February 2012. In order to mitigate the pitting on the Hg-target container, now we are injecting helium micro bubbles in the target. The Laser Doppler vibrometory showed us that vibration on the proton bombardment has been obviously reduced by the injection.

21 instruments have been already funded, 18 are operated for user program and 3 instruments are under either commissioning or construction. Now the experimental hall is almost full with instruments, leaving only 2 ports are available for instruments in the future.

World-class scientific outputs have been already created in various scientific fields, ranging from Li-battery science to bio-molecular science. Since J-PARC is internationally open for users, we have got experimental proposals from abroad more than 10% of the whole proposals. More than 30% of proposals have come from industries and a half of them are proprietary use. This fact has revealed a new horizon has come in the neutron scattering science in the 21 century.

Abstract

Paper Ref: 387

Current Status and Perspectives of HANARO

Chang-Hee Lee[1], Baek-Seok Seong[2]

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HANARO has been operated for 20 years since its first criticality on February 1995 and its cold neutron research facility (CNRF) was opened for general users in late 2011. And up to 9 instruments using cold neutron could be opened since late 2014 by opening 4 more instruments such as the cold neutron triple axis spectrometer, the disc chopper time-of-flight spectrometer, the bio-reflectometer and the cold neutron activation station comprising of a prompt gamma activation analysis unit and a neutron depth profiling unit. There are 7 thermal neutron beam instruments for users, in which the most recent user-opened instrument was the single crystal diffractometer equipped with a curved shape, large area position-sensitive detector. There was an organizational change to formulate two closely linked divisions for neutron science and technical development activity in 2014 to emphasize scientific research and user supports. The other division focuses on technical development, instrumentations, technical support and facility operation. We expect a stable capability build-up in a few years for in-house technical development and support including sample environments and on-site laboratory equipment including X-ray counterpart instruments. The user record showed a robust increase since 2011 together with marked outcomes and a remarkable progress for industrial applications by neutron techniques has been made for last few years. Discussion to bring out a joint research center with universities for long-term thesis program, joint program with national program groups, drive to make research coalition among government research institutes, planning work to construct thermal neutron guides and their instruments will be also mentioned in the presentation.

Abstract

Paper Ref: 254

National Facility for Neutron Beam Research in India

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A national facility for neutron beam research is operated at the research reactor Dhruva in BARC. It includes single-crystal and powder diffractometers, a polarization analysis spectrometer, inelastic and quasi-elastic scattering spectrometers in the reactor hall, and small-angle scattering instruments and a polarized neutron reflectometer in the neutron-guide laboratory. The National facility is utilized in collaboration with various universities and other institutions. The talk will present our facilities and discuss examples of recent work.

Abstract

Paper Ref: 445

The status of facilities at China Advanced Research Reactor

Yuntao Liu[1], Dongfeng Chen[1]

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A 60 MW research reactor, so called China Advanced Research Reactor (CARR,) was built in China Institute of Atomic Energy (CIAE), located in the southwest of Beijing and about 37 kilometers away from the central city. CARR is a tank-in-pool inverse neutron trap type reactor using D_2O reflector, the designed optimal undisturbed thermal neutron flux is 8×10^{14} $n\cdot cm^{-2}\cdot s^{-1}$. A liquid D_2 cold source will be equipped and the installation will be finished at the end of 2015. As a multipurpose research reactor, its main applications include neutron scattering, neutron activation analysis, isotope production, silicon doping, fuel element test, fundamental nuclear physics and so on. On March 13rd, 2012 CARR realized the 72 h stable operation with the full power. And the official operation license is expected to be issued at the beginning of next year.

Cooperating with the internal and international users in the first phase ten instruments complete construction and are under commissioning, which are High Resolution Powder Diffractometer, High Intensity Powder Diffractometer, Residual Stress Diffractometer, Texture Diffractometer, Four Circle Diffractometer, Reflectometer, Small Angle Neutron Scattering, two Thermal Triple Axis Spectrometers and Isotope Separator On-Line instrument . In the second phase 7 instruments were approved and are under construction now. Although the operation license was not issued, the reactor was permitted to do the testing run several times and some results were obtained during the instrument commissioning.

Abstract

Paper Ref: 9

Professor Mitsuhiro Shibayama [1], Dr. Masayasu Takeda [2], Dr. Shuichi Wakimoto [2]

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[2] *Quantum Beam Science Center*

JRR-3 was the first research reactor with 10 MW thermal power constructed by Japan and established criticality in 1962. It was refurbished in the years 1985 - 1990 to upgarde to the present 20MW JRR-3 with a liquid hydrogen cold moderator. For promotion of neutron beam science, 33 neutron instruments are installed in the reactor hall and in the neutron guide hall with two thermal and three cold neutron guides. The instruments have been installed by the neutron scattering groups of Japan Atomic Energy Agency (JAEA), and Institute for Solid State Physics (ISSP) of the Univ. of Tokyo. Two user programs run at JRR-3, i.e., the "University Users Program" run by ISSP using the ISSP instruments and the "JAEA Common-Use Facility Program" (hereafter referred as "JAEA Users Program") on the JAEA instruments. More than 600 proposals are accepted through the two users programs.

Although JRR-3 had served as an excellent vehicle for the training of a large number of Japanese and international scientists, it has not been operated since the Great Earthquake on March 11, 2011. Repair work of the reactor facility and structures was completed in FY2011. However, the Japan Nuclear Regulatory Authority (NRA) promulgated and enforced the new Regulatory Requirements for Research Reactors in Dec. 2013. For the JRR-3 restart a full legal licensing procedure under the new safety regulations, which is currently in progress, must be completed before the legal in-service inspection procedure can take place. To meet the new Regulatory Requirements, the following points must be considered additionally: (a) Seismic safety, (b) Natural disaster, such as Tsunami, Tornado, Wildfire etc. JAEA is aiming to restart JRR-3 during JFY2015.

During this outage period, several upgrade programs have been undertaking, such as replacement of neutron guides to 3Qc mirrors (C1, C3), introduction of focusing devices, installation of polarization devices. In addition, a new proposal system RING (Research Information NaviGator) has launched in 2012. On the other hand, about 10% of proposals are transferred to overseas facilities under the University Users Program to maintain the activities of neutron science in Japan. Several scientific achievements will be demonstrated.

Abstract

Paper Ref: 456

China Spallation Neutron Source

Hesheng Chen[1]

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The China Spallation Neutron Source (CSNS) is designed to provide multidiscipline research platforms with neutron scattering. The site of CSNS has been selected at Dongguan, Guangdong Province. The facility comprises an 80-MeV H- Linac, a 1.6-GeV proton rapid cycling synchrotron (RCS), beam transport lines, a solid tungsten target station, and the experimental hall for the pulsed spallation neutron applications. The RCS provides a beam power of 100 kW on the target in the phase I. In the phase II, the beam energy of the Linac will be upgraded to 250MeV, and the beam power can be further increased to 500 kW. There are three initial spectrometers of day-one in the experimental hall: general purpose powder diffractometer, small angle neutron scattering meter and magnetic reflectometer. The maximum number of the beamlines is 20 in the first target station. The civil engineering of CSNS was started May 2012, and will be finished by autumn 2015. The mass productions of the accelerator components, target system and spectrometers are under way. The installation of the Linac was started in last October. The first neutron beam to the target is scheduled by September 2017. The facility will be opened to users Spring 2018.

Abstract

Paper Ref: 451

The Development of BATAN's Neutron Beam Facilities and Their Applications

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Neutron scattering laboratory is one of the facilities utilizing neutron beam produced by the RSG-GAS reactor at Serpong for materials R&D. The laboratory is equipped with seven neutron beam instruments, i.e: Diffractometer for Residual Stress Measurement (DN1), Four Circle/Texture Diffractometer (DN2), High Resolution Powder Diffractometer (DN3), Triple Axis Spectrometer (SN1), Small Angle Neutron Scattering Spectrometer(DN2), High Resolution Small Angle Neutron Scattering Spectrometer (SN3) and Radiography Facility (RN1). The instruments have been developed to meet the requirement demanded by the users to reach their research objectives such as the replacement of some detectors in neutron diffractometer from point detector to 2D detector, the refurbishing of the 2D-Position Sensitive Detector in SANS machine and the use of neutron tomography to replace the old neutron radiography film technique. The upgraded instruments have been used to perform research and development in various topics such as materials for battery, magnetic materials and automotive industry. The current status of the instruments and their development as well as their applications for research on advanced materials and industry are presented.

CMP-4 Multiferroics, SCES, lattice dynamics

Abstract

Paper Ref: 446

Spin excitations in multiferroics and frustrated magnets

Manh Duc Le[1], Jaehong Jeong[1,2], Joosung Oh[1,2], Kisoo Park[1,2], T.G. Perring[3], P. Bourges[4], S. Petit[4], Je-Geun Park[1,2]

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[2] *Dept. of Physics and Astronomy, Seoul National University, Seoul, Korea*
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Inelastic neutron scattering (INS) is still the leading experimental probe of the spin dynamics of magnetic materials: It is not restricted to zero momentum transfer as optical spectroscopy is, and retains an order of magnitude better resolution than resonant inelastic X-ray scattering. Whilst it has been displaced by inelastic X-ray scattering as the technique of choice to measure phonon dispersion, it still retains a role in systems, such as multiferroics, where the symmetry allows an appreciable magnon-phonon coupling.

As the theory underpinning the neutron-matter interaction is well understood, one can model quantitatively not only the excitation energies and dispersions, but also the intensities of magnons, phonons and hybrid modes, given a good model of the underlying system Hamiltonian, allowing the parameters of such models, be it exchange interactions, force constants or spin-lattice couplings to be extracted. As examples of this type of work, we will discuss measurements of the magnon spectra by INS in the room temperature multiferroic compoung $BiFeO_3$ [1], and magnon-magnon [2] and magnon-phonon [3] interactions in the hexagonal $RMnO_3$ family. Finally, whilst in the past the computational machinery required to carry out these analysis have been relatively inaccessible forcing the experimentalist to devote effort into writing her own code, several open-source programs [4,5] have become available recently which may allow INS to become a more standard and widely used technique, so we will conclude with an overview of these software.

[1] J. Jeong et al., *Phys. Rev. Lett.* 113 107202 (2014)
[2] J. Oh et al., *Phys. Rev. Lett.* 111 257202 (2013)
[3] J. Oh et al., in preparation
[4] **http://www.psi.ch/spinw**
[5] **http://www.mcphase.de**

Abstract

Paper Ref: 8

Spin fluctuations in $Ce_{1-x}La_xB_6$ and $Ce_3Pd_{20}Si_6$

D. S. Inosov[1], P. Y. Portnichenko[1], A. S. Cameron[1], H. Jang[2], G. Friemel[2], P. P. Deen[3,4], S. Paschen[5], A. Prokofiev[5], J.-M. Mignot[6], N.Y. Shitsevalova[7], V. B. Filipov[7].

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[2]*Max-Planck-Institut für Festkörperforschung, Stuttgart, Germany,*
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[4]*Niels Bohr Institutet, University of Copenhagen, Denmark.*
[5]*Institute of Solid State Physics, Vienna University of Technology, Austria,*
[6]*Laboratoire Léon Brillouin, CEA-CNRS, CEA/Saclay, Gif sur Yvette, France,*
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Heavy-fermion metals exhibit a plethora of low-temperature ordering phenomena, among them the so-called hidden-order phases that in contrast to conventional magnetic order are invisible to standard neutron diffraction. The simple-cubic hidden-order compounds CeB_6 and $Ce_3Pd_{20}Si_6$ are famous for an elusive phase attributed to the antiferroquadrupolar ordering of Ce 4f moments. In the ground state, these systems also develop a more usual antiferromagnetic (AFM) order. In our recent inelastic neutron scattering experiments, we investigated the spin dynamics in these related compounds, including their dependence on magnetic field and La doping. In CeB_6, we discovered an intense ferromagnetic (FM) low-energy collective mode that dominates the magnetic excitation spectrum of CeB_6, thus placing CeB_6 much closer to a FM instability than could be anticipated. This propensity of CeB_6 to ferromagnetism may account for much of its unexplained behavior, such as the existence of a pronounced electron spin resonance, and should lead to a substantial revision of existing theories that have so far largely neglected the role of FM interactions. On the other hand, the absence of such zone-center fluctuations in $Ce_3Pd_{20}Si_6$ demonstrates that it they are unrelated to the magnetically hidden multipolar order parameter. Instead, the paramagnetic state of $Ce_3Pd_{20}Si_6$ is characterized by a diffuse quasielastic scattering pattern with a broad momentum-space structure peaked near the (111) wave vector, which represents the corner of the unfolded Brillouin zone. Its symmetry suggests that it stems from the simple-cubic Ce sublattice occupying the 8c Wyckoff site, whereas the crystallographically inequivalent 4a site remains magnetically silent in this material.

Abstract

Paper Ref: 301

Exotic ground state of a breathing pyrochlore antiferromagnet $Ba_3Yb_2Zn_5O_{11}$

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Spin-1/2 quantum pyrochlore Heisenberg antiferromagnets are known to be promising candidates for three-dimensional quantum spin liquids. Despite numerous experimental and theoretical efforts, their ground state properties have not yet been established because of the lack of a model material and the unavailability of exact solutions. A popular theoretical approach to this problem is to first decouple the full pyrochlore lattice into a set of independent tetrahedra and then reconnect them perturbatively [1,2]. Recently, Kimura *et al*. [3] have reported the new material $Ba_3Yb_2Zn_5O_{11}$ to be a model system of a quantum breathing pyrochlore lattice antiferromagnet; the breathing pyrochlore lattice consists of an alternating array of small and large tetrahedron [4]. This material crystallizes with the cubic space group *F-43m*, and intra- and inter-Yb-tetrahedron distances are ~3.29 Å and ~6.25 Å. Analysis of magnetization and heat capacity data show the formation of quantum spin-singlet state in an almost isolated small Yb tetrahedron with pseudospin 1/2. However, our inelastic neutron scattering (INS) experiment of $Ba_3Yb_2Zn_5O_{11}$ show three distinct excitation modes at T = 1.5 K instead of two modes expected from the spin-1/2 isolated tetrahedron model. In addition, at T = 10 K, INS data shows complicate additional excitation modes, which indicates the energy scheme of $Ba_3Yb_2Zn_5O_{11}$ is beyond the simple isolated tetrahedron model. We discuss the magnetic ground state of $Ba_3Yb_2Zn_5O_{11}$.

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Abstract

Paper Ref: 263

A comparison of H-bond potentials in $Rb_3H(SO_4)_2$ obtained using neutron scattering and MD simulation

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The H-bond potential of $Rb_3H(SO_4)_2$ has been studied using many techniques including deep inelastic neutron scattering[1] (DINS), inelastic incoherent neutron spectroscopy[2] (IINS), neutron diffraction[3] (ND), and nuclear magnetic resonance[4] (NMR). Of these measurements there is significant disagreement in the effective potential that the proton experiences in the short O-H-O bond. To understand this discrepancy a set of molecular dynamic (MD) simulations were performed to relate the apparently disparate results. Though the simulations used classical mechanics to model the proton motion it is argued that this does not directly affect the H-bond potential created. Instead it was found that neglect of the ground-state motion of the terminal O-atoms of the O-H-O were the dominant source of error, though one that can be corrected post-simulation. Solution of the Schrödinger equation calculated from the MD derived H-bond potential showed good agreement with new neutron diffraction results obtained using the KOALA single crystal diffractometer.

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Abstract

Paper Ref: 198

Structural and Magnetic Properties of 4d and 5d perovskites

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During the last years, the study of osmium-based oxides has attracted a lot of attention in materials science, since the properties of the 4d and particularly 5d oxides are expected to be extremely appealing. The increased spatial extent of the 4d/5d orbitals of the second/third-row transition-metal ions, with respect to the rather localized 3d orbitals of the first-row, gives rise to weaker Coulomb interactions. This leads to a greater level of splitting in the crystal field and consequently an increase in the sensitivity of these ions to lattice distortions, which present unusual electronic structures due to their characteristic spin-orbit coupling. For example, several interesting properties have been reported for pyrochlores, such as superconductivity in KOs_2O_6 (Tc = 9.6 K) and $RbOs_2O_6$ (Tc = 6.3 K) or metal–insulator (MI) transition at 225 K in the $Cd_2Os_2O_7$. Among the double perovskites, it is noteworthy to mention some examples, like the Sr_2CrOsO_6 oxide [1], which displays the highest Curie temperature (725 K) of the perovskite like-structure compounds, the recently described compounds Ca_2FeOsO_6 and Sr_2FeOsO_6 [2], which undergoes a magnetic transition from ferromagnetic to antiferromagnetic when substituting Ca by Sr, or the FCC magnetically frustrated Ba_2YOsO_6 perovskite [3].

The outstanding magnetic behavior of the osmium-based double perovskites raised the interest of searching new compounds and explores their physical properties. Therefore, in this work, we present a crystallographic and magnetic study of the solid solution $Ba_{2-x}Sr_xYOsO_6$ ($0 \leq x \leq 2$) and compare this to the behavior in the closely related series $Ba_{2-x}Sr_xYRuO_6$. Neutron powder diffraction experiments were performed at room temperature in order to investigate in detail the crystal structures. To determine the magnetic structure of the samples, NPD data were collected below the order magnetic temperature.

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MSC-5 Energy Materials

Abstract

Paper Ref: 253

Thermal Expansion and Negative Linear Compressibility in $ZnAu_2(CN)_4$: An Inelastic Neutron Scattering Measurements and Lattice Dynamical Studies

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$ZnAu_2(CN)_4$ shows negative thermal expansion behavior as well as negative linear compressibility. The linear thermal expansion behavior [1] is highly anisotropic (α_a~ 36.9×10^{-6} K^{-1}, α_c~ -57.6×10^{-6} K^{-1}). On application of hydrostatic pressure the c-axis expands along which the NTE behavior has also been reported [2]. We have performed the inelastic neutron scattering measurment using IN4C spectrometer at ILL, Grenoble France. The measurments are performed at various temperatures ranging from 150 K to 400 K as shown in Fig 1(a). From the temperature dependence measurements we observed that modes below 20 meV show significant change in energy with temperature indicating the anharmonic nature of these modes. To understand the anamolus properties from microscpic view we have been calculated in the phonon in entire Brillouin zone using DFT. Our calculation suggest that these modes mostly involve motion of $Zn(CN)_4$ tetrahedral units connected via Au atoms. The calculated pressure dependence of the phonon energies have been used for thermal expansion calculation (Fig. 1(b)) of the compound. We observe that low energy modes below 5 meV contribute negative to the total thermal expansion (α_V) behavior. However modes around 10 meV have large positive contribution to α_V and results in positive volume thermal expansion behavior. The eigen vector of a zone centre phonon mode of energy around 4 meV energy with largest negative Grüneisen parameter of -9.2 suggest the twisting motion of the -Zn-NC-Au-CN-Zn- chain along with rotational motion of ZnN_4 tetrahedra. Such motion would lead to contraction along *c* direction.

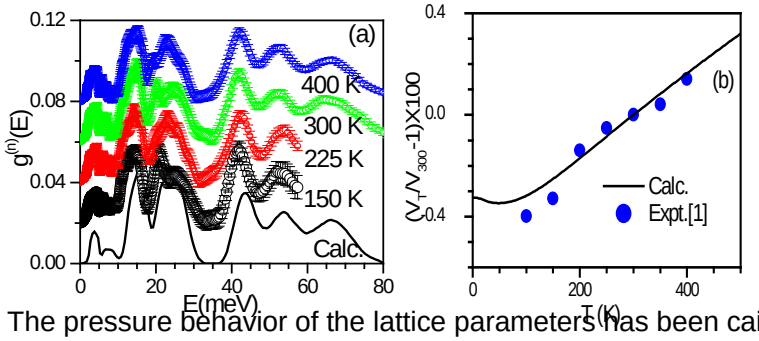

Fig 1 (a) The inelastic neutron scattering measurement with temperature. (b) The calculated volume thermal expansion behavior compared with experimental measurements.

The pressure behavior of the lattice parameters has been calculated using ab-initio calculations. NLC behavior as observed along c-axis from the experimental high pressure measurements is closely reproduced by the calculations.

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Abstract

Paper Ref: 34

Synthetic, Structural, and Electrochemical Study of Monoclinic $Na_4Ti_5O_{12}$ as a Sodium-ion Battery Anode Material

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[4] *National Institute of Advanced Industrial Science and Technology, Osaka, Japan*

Since lithium-ion batteries were commercialized in 1991[1], they have dominated the portable electronics market. In 2014, over one-fourth of the world's mined lithium was devoted to lithium-ion battery applications[2]. Increased future demand will be driven by the electric/hybrid vehicle market and load leveling solutions for renewable energy sources[3]. Therefore, the development of new, low-cost alternatives to lithium-ion batteries with comparable electrochemical properties is highly desirable. Sodium is the most obvious choice, as an inexpensive, abundant, and easily extractable element. As well as the decreased cost in starting material, sodium-ion batteries can use aluminum as the current collector for the anode because, unlike lithium, sodium does not alloy with aluminum metal. Aluminum offers both a less-expensive and lighter alternative

to the copper usually used in lithium-ion batteries[4]. However, challenges still remain in developing sodium intercalation materials capable of achieving a high reversible capacity and energy density. In particular, because of the inability of Na ions to intercalate with graphite[5], anode materials must be significantly improved.

We have investigated the monoclinic phase of $Na_4Ti_5O_{12}$ (M-$Na_4Ti_5O_{12}$) as a potential sodium-ion battery anode material. In contrast to the previously investigated trigonal phase (T-$Na_4Ti_5O_{12}$), M-$Na_4Ti_5O_{12}$ has continuous two-dimensional (2D) channels with partially occupied Na sites, providing broader pathways and more space for the intercalation of excess sodium. Neutron powder diffraction reveals the preferred sites and occupancies of the excess sodium as well as the sodium ion conduction pathway. Electrochemical measurements show that it exhibits a comparable or higher reversible capacity than T-$Na_4Ti_5O_{12}$. *In situ* synchrotron X-ray diffraction under electrochemical cycling shows that the crystal lattice undergoes strongly anisotropic volume changes during cycling.

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Abstract

Paper Ref: 367

In situ neutron and x-ray diffraction study on the thermal decomposition of MgH_2-$LiBH_4$ composite at various temperatures

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$LiBH_4$ has been a promising candidate for solid-state hydrogen storage materials due to the high theoretical hydrogen density (18 wt% H_2, 122 Kg/m^3) [1]. Nevertheless, it can release hydrogen molecules only at above 480 C due to the strong binding of H to B via covalent bonding and the ionic bonding of the $(BH_4)^-$ unit to the Li^+ ion. One of fancy ways to improve the (de)sorption properties of $LiBH_4$ is to make a composite with another metal hydride using a high energy ball-milling. In this composite, the total enthalpy change of the reaction reduces and the rehydrogenation can occur at more moderate conditions compared with that of pristine $LiBH_4$, due to a formation of new phase (metal boride) during the dehydrogenation of the composite [2]. However, it still shows high thermal stability and a sluggish kinetics, and therefore it is requested to investigate the detailed mechanism of dehydrogenation to overcome these drawbacks blocking its use as solid-state hydrogen storage materials.

In situ neutron and x-ray diffraction experiments were carried out using an isotope-labeled composite, MgH_2-$LiBD_4$, at various temperatures (T = 30 C – 360 C, P_{He} ~ 1 bar) in order to observe H-D exchange between each constituent during the dehydrogenation of MgH_2. The results indicate that the formation of $MgH_{1-x}D_x$ occurred considerably earlier than the decomposition of MgH_2. It supports the occurrence of H-exchanges at the interface of two materials, and are meaningful in suggesting the more exact point of the exchanges [3,4]. In addition, we connect this result to the desorption properties of the composite with the results of TG-MS, DSC, and Raman scattering measurements.

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Abstract

Paper Ref: 371

PHASE TRANSFORMATION STUDIES OF METHANE-PROPANE HYDRATE USING NEUTRON SCATTERING

Dr Alice Klapproth[1], Ross O. Piltz [1], Christopher J. Garvey [1], Nobuo Maeda [2], Karen A. Kozielski [3], Shane J. Kennedy [1]

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[3] *CSIRO Energy Flagship*

Gas hydrates form readily under the pressures and temperatures experienced in deep-sea oil and gas pipelines. They form large solid plugs that can block and damage pipelines and disrupt production. Consequently the Oil and Gas industry uses chemical inhibitors in deep sea fields as a preventative for gas hydrates. As a production field matures the fraction of water in the gas flow increases. The quantity of chemical inhibitors must therefore increase resulting in additional production costs. The ability to understand the processes associated with hydrate formation would allow design engineers and operators to work to tighter margins at considerable cost savings.

In-situ neutron diffraction experiments of hydrate reaction processes, using the powder diffractometer (*Wombat)*, provide insights into the kinetics of crystallisation of gas hydrates from solid and liquid water phases. We present neutron scattering studies of the phase transformations and the phase occurrence over a range of pressure/temperature (3.0 – 4.8 MPa, 263 – 288 K) of methane-propane hydrates as a simple analogue of natural gas hydrates.

Our results are mainly in accord with the thermodynamic stability of the hydrate phases, where the methane-propane mixture can produce either pure sII hydrate or a combination of sI and sII hydrates. Surprisingly of two measurements at 4.8 MPa the sI phase did not form at a temperature of 263 K but did form at a higher temperature. This result is inconsistent with the thermodynamic phase boundary of methane sI hydrate. We also demonstrated the effectiveness of two common inhibitors, monoethylene glycol and polyvinylcaprolactam.

We observe that decomposition of gas hydrates just above the ice-point results in formation of ice, which subsequently decomposes slower than the hydrate [1, 2]. Furthermore these experiments elucidate the importance of the heat flow of the reaction processes and the role of a free liquid-gas interface.

In-situ SANS experiments can probe the hydrate nucleation process. Our first SANS experiments, on *Quokka*, reveal rapid uptake of methane-propane gas on ice crystallites well below the accepted phase boundary, offering the prospect of valuable insights in planned future experiments using a gas-liquid flow loop, which is under development at ANSTO.

References:
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A. Klapproth, R.O. Piltz, S.J. Kennedy, V.K. Peterson, K.A. Kozielski and P. Hartley, Proc. 7th Int. Conf. Gas Hydrates, Edinburgh, Scotland, 2011.

Abstract

Paper Ref: 192

CONCENTRATION-DEPENDENT BINDING OF CO_2 AND CD_4 IN UiO-66(Zr): A COMBINATION OF NEUTRON POWDER DIFFRACTION AND FIRST-PRINCIPLE DFT CALCULATIONS

Hubert Chevreau[1], Weibin Liang[2], Gordon J. Kearley[1], Samuel G. Duyker[1], Deanna M. D'Alessandro[2],Vanessa K. Peterson[1].

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Over the last twenty years, tremendous progress has been achieved in the field of Metal Organic Frameworks. Among these materials, the zirconium terephthalate UiO-66(Zr) [1] has attracted a growing attention because of its interesting thermal, chemical and water stability and has shown to be a promising material for the separation of CO_2/CH_4 gas mixtures.

In order to get a better understanding of its sorption behavior towards these gases, a Neutron Powder Diffraction (NPD) investigation of UiO-66 loaded with sequential doses of CO_2 and CD_4 has been carried out on the High Resolution Powder Diffractometer instrument "Echidna" at the OPAL reactor (ANSTO, Sydney).

In total, three adsorption sites for CO_2 and three adsorption sites for CD_4 within the UiO-66(Zr) have been located by NPD then characterized by a combination of first-principles Density Functional Theory (DFT) calculations, binding energies and Quantum Theory of Atoms In Molecules (QTAIM) theory. An example of the first CO_2 adsorption site is given in figure 1.

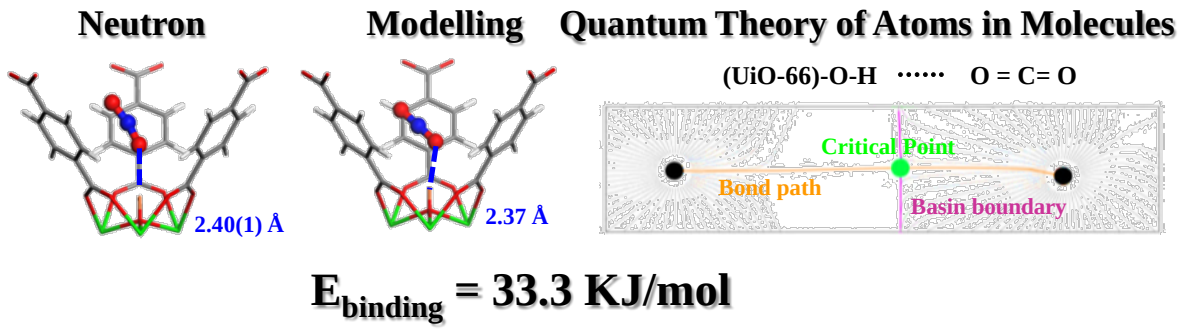

Figure 1. Structure of CO_2 at site Tc_{OH} in UiO-66(Zr) determined using NPD (a) and DFT (b). c) QTAIM analysis result showing the Gradient field (black lines), bond path (orange line), and basin boundary (purple line)

In conclusion, it could be observed that CO_2 molecules are preferentially adsorbed in tetrahedral cages over the larger octahedral cages, that underpin the material's selectivity for CO2 over CH4. [2]

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SMT-2 New instruments and devices

Abstract

Paper Ref: 28

Applications of the single crystal diffractometer at HANARO based on a large-area curved two-dimensional position-sensitive detector

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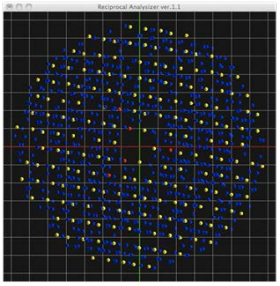
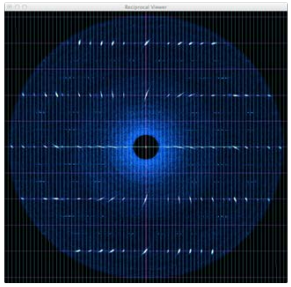
[2] *Institute of Materials Structure Science, High Energy Accelerator Research Organization, Ibaraki, Japan*
[3]*Institute of Multidisciplinary Research for Advanced Materials, Tohoku University, Sendai, Japan*

At HANARO, we developed and installed a neutron single crystal diffractometer based on a large-area curved two-dimensional position-sensitive detector (C-2DPSD) under collaboration of the Korea Atomic Energy Research Institute (KAERI) and Tohoku University. Last year, the diffractometer commissioning was completed with the development of the measurement methodology and the raw data processing software package, the 'Reciprocal Analyzer'. The diffractometer is equipped with a Ge(311) mosaic monochromator and two supermirror vacuum guide paths. Three different wavelengths can be easily selected by simply rotating the monochromator slab. The diffractometer has a customized room-temperature sample chamber and a 10K closed-cycle refrigerator-type low-temperature sample chamber to reduce the background from air scattering over the wide opening of the area detector. The calibration and corrections of the instrument are performed using a NaCl crystal. During the commissioning we carried out the experiments for various applications such as crystallographic and magnetic structure measurements, a crystallinity check on large crystals, a reciprocal space search and mapping, and a study on the composition or dopant content of a mixed crystal. The characteristics of the diffractometer, the measurement method, the calibration procedure and results, the raw data treatment and visualization, and several applications using the large C-2DPSD-based diffractometer will be introduced in this presentation.

Reciprocal space search and mapping



$Sm_{0.45}Sr_{0.55}MnO3_3$ search for diffuse scattering, H. Hiraka & Y. Endoh @Tohoku Uni.(now KEK)



$YbFe_2O_4$, search for super lattice peaks, T. Nagata & N. Ikeda@Okayama Uni.

Abstract

Paper Ref: 377

Recently commissioned time-of-flight small angle neutron scattering machine at ANSTO: Capabilities and performance

Anna Sokolova[1], Andrew Whitten[1], Liliana De Campo[1]

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Australian Nuclear Science and technology Organization (ANSTO) successfully operates one SANS instrument Quokka and recently commenced commissioning of the second SANS instrument, Bilby [1]. The Bilby is Time-of-Flight instrument built on reactor neutron source. The instrument is scheduled to be in user operation in the second half of the year 2015.

The design (in particular, set-up of four choppers which similar to that design for D33 instrument [2] at ILL) opens possibility to vary wavelength resolution in the wide range (from 4% to 30%) satisfying various scientists' requirements. Two arrays of position sensitive detectors in combination with utilizing of wide wavelength range (from ~3Å to ~20Å) provide capability to collect scattering data of wide angular range without changing experimental set-up. Offered instrument design opens possibility to collect scattering from a wide range of samples, with a unique capability to record fast kinetics data.

Additional hardware add-on will allow to use instrument in slit mode and therefore get data at very low Q (~$2 \cdot 10^{-4}$ Å^{-1}).

The presentation will be focused on general concept and unique features of the new SANS instrument, as well as first results of instrument commissioning.

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Abstract

Paper Ref: 257

Progress on the development of a polychromatic beam neutron reflectometer at NIST

C.F. Majkrzak[1], N. C. Maliszewskyj[1], A. Osovizky[1,2], Y. Yehuda-Zada[3,4], J. Ziegler[1], K. Pritchard[1], N. Hadad[1], J. Cook[1], E. Binkley[1], P. Tsai[1]

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[2]Rotem Industries Ltd., Rotem Industrial Park, Israel

[3]Ben Gurion University of the Negev, Beer Sheva, Israel

[4]Electronics & Control Laboratories, Nuclear Research Center- Negev, Beer-Sheva, Israel

Progress on the general development of the polychromatic beam reflectometer CANDOR (for Chromatic Analyser Neutron Reflectometer Or Diffractometer) being constructed at the NIST Center for Neutron Research is reported. This includes the evolution of an energy-dependent neutron detector which incorporates pyrolytic graphite analyser crystals (54 separate elements in series) in conjunction with 6LiF/ZnS(Ag) scintillation detectors and silicon photomultiplier (SiPM) devices to form an array which simultaneously detects neutrons within a 4 to 6 Angstrom bandwidth with a fractional wavelength resolution of approximately one percent. The general design of this energy dispersive detector is applicable to other instruments as well, for example, a materials diffactometer (Simmons et al. *J. Appl. Cryst.* 46, 2013). How 30 such energy-dependent detector arrays are to be configured within the reflectometer is described, particularly in regard to achieving a focusing condition in the wavevector transfer Q for specular reflectivity measurements. For non-specular reflectivity measurements, an order of magnitude gain over a conventional monochromatic beam instrument at a continuous source is in principle possible, whereas for specular reflectivity measurements -- in which a broader angular divergence of the incident polychromatic beam can be used in addition -- a gain of several orders of magnitude may be realized under certain conditions. Other components of this instrument, including polarizers, focusing optics, and filters to suppress undesirable portions of the incident spectrum are described.

Abstract

Paper Ref: 135

The effect of a velocity selector before the pyrolytic graphite monochromator on the Bamboo thermal triple-axis spectrometer

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Removing the contamination of high order neutrons from the pyrolytic graphite (PG) monochromator is one of the most critical procedures to obtain reliable data on a thermal triple-axis spectrometer (TAS). This is typically done by putting a PG filter after the sample in the fixed-E_f configuration, which results in many restrictions in the experiments. We have built the Bamboo thermal TAS at China Advanced Research Reactor (CARR), Beijng, China. By installing a neutron velocity selector before the PG monochromator, the incident neutrons are free of unwanted higher neutrons. It allows us to continuously fine tune the energy and momentum resolution in a simple manner. In addition, it provides much more choices in an experiment that may significantly improve the data quality.

Abstract

Paper Ref: 211

Current status and perspective of neutron resonance spin echo spectrometers (VIN ROSE) at J-PARC/MLF

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[3]*Institute of Materials Structure Science, KEK, Tsukuba, Ibaraki 305-0801, Japan*

Neutron spin echo (NSE) method discovered by F.Mezei [1] is very powerful tool to investigate slow dynamics. It measures directly intermediate scattering function S(q,t) with very high neutron energy resolution. In order to cover wide energy range with various sample environments, our instrument consists of two types of NSE spectrometers: NRSE (Neutron Resonance Spin Echo)[2] and MIEZE (Modulated Intensity by Zero Effort) [3]. We named these spectrometers “VIN ROSE” (VIllage of Neutron ResOnance Spin Echo spectrometers) wishing increase in the value with the passage of time and celebrating new scientific discovery with delight.

Kyoto University and KEK have constructed a new beam line for the VIN ROSE at BL06 at the Materials and Life Science Facility (MLF) of Japan Proton Accelerate Research Complex (J-PARC). There are two neutron guide tubes and polarizing system were placed at the exits of each neutron guides. The polarizing system consists of Fe/$SiGe_3$(Si) magnetic supermirrors with permanent magnets, four-quadrant slits and a long wavelength cutting filter. The polarizing supermirrors and four-quadrant slits have been installed to the shielding box in which consisted of 50 wt% B4C-containing rubber and lead to stop scattered neutrons and gamma rays. The beam sizes at exit of the polarizing system for MIEZE and NRSE were limited to 5×20, 10×10 mm^2 by the four-quadrant slits, respectively. There are two kinds of polarizing mirrors to switch wavelength band. These supermirrors were fabricated by an ion beam sputtering machine at KURRI.

In this study, we will show performance of the MIEZE and NRSE polarizing system and results of the first MIEZE experiment.

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SMS-4 Colloids

Abstract

Paper Ref: 208

SHEAR THICKENING AND ONION FORMATION OF NON-IONIC SURFACTANT SOLUTION AND THE EFFECT OF CHARGE

M. Nagao[1, 2], Y. Kawabata[3], S. Kugizaki[3], R. Bradbury[1, 2], K. Weigandt[1], K. Sadakane[4], **H. Seto[5]**

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A shear-induced lamellar to onion transition of a surfactant solution has attracted considerable attention in these decades. Diat and Roux have shown that shear flow induces a transformation from planer lamellar structure to multilamellar vesicles (onions) with a polyhedral shape, which fill all the space without excess water and lead shear thickening. [1] In a nonionic surfactant aqueous solution, pentaethylene glycol monododecyl ether ($C_{12}E_5$) and D_2O, onion formation associated with shear thickening is observed in 40 wt% $C_{12}E_5$ solution at T=55 ℃. [2] Effects of charge on the nonionic surfactant mixture were investigated by adding ionic surfactant and onion structure is induced by the suppression of Helfrich undulation of surfactant membranes. [3]

Here we investigated the rheological behavior of a dilute solution of $C_{12}E_5$ (10 wt%) and D_2O and the effect of charge. We have already shown that a disordered structure of $C_{12}E_5$ and D_2O at T=59 ℃ transforms to an ordered lamellar structure by adding an antagonistic salt such as sodium tetraphenyl-borate ($NaBPh_4$). An electrostatic interaction between surfactant membranes caused by a heterogeneous distribution of anions and cations originates the transformation as the effect of adding ionic surfactant sodium dodecyl sulfate (SDS). Figure 1 shows molar ratio between SDS and C12E5, S, dependence of viscosity as a function of shear rate. It is clear that shear thickening is observed for all the samples measured including SDS and a shoulder at 2 s-1 is observed for the sample without charged molecules. The present SANS experiment confirmed that the lamellar layers are oriented parallel to the flow direction at low shear rate, while onion structure is formed as evident by isotropic scattering pattern, which is eventually broken by further increase of shear rate. This is the first evidence of the shear thickening and the onion formation in the dilute solution of nonionic surfactant.

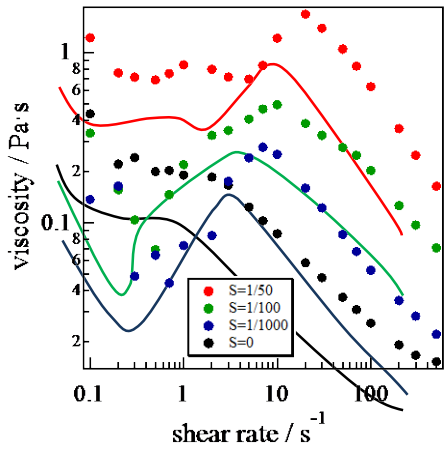

Fig. 1 SDS concentration dependence of shear viscosity against shear rate of mixtures of $C_{12}E_5$ (10 wt%), D_2O and SDS. Black: without SDS (S=0). Blue:

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Abstract

Paper Ref: 196

STUDY OF AGGREGATION IN A MICELLAR SOLUTION WITHIN BENTONITE CLAY BY SANS

Dr Reshma Raut Dessai [1], Prof. Erwin Desa [2], Dr. Debes Ray [3], Dr. Vinod Kumar Aswal [3]

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Aggregation studies of surfactant solutions exposed to different surfaces are relevant to their applications as detergents, oil recovery agents etc. The infusion of SDS micellar solutions into the pores of compacted normal bentonite clay and the clay saturated with sodium at different concentrations viz. 1, 2 and 5 molar percentages, has been studied here by small angle neutron scattering and X-ray diffraction.

In the sodium saturated bentonite, the Na^+ ion (having a positive scattering length) is held in the interlayer of the clay and on account of this, the scattering length density difference (i.e. contrast) between the bentonite matrix and the inter-layer pores is lower than the bentonite which has less sodium. The average pore size in normal bentonite clay is 13 Å while in the clay saturated with 1M Na^+ ions it is 20 Å .

For a 10% SDS solution, the micelles are prolate ellipsoids having average semi major and minor axes of 22.5 Å and 16.7 Å, respectively with an effective charge of 30 e.u. and an aggregation number of 75. Both aggregation number and effective charge increase with concentration of SDS solution.

Sodium is present in normal bentonite but is enhanced in concentration in the case of sodium saturated bentonite. Aggregation number and micellar separation are reduced for SDS loaded into raw bentonite clay and then into Na saturated clay. Likewise, there is a reduction of fractional charge on the micelle in going from raw to sodium bentonite caused by the Na^+ ions present on the surface of the clay pores which would be attracted to the negative surface of each micelle thereby reducing its net charge. The effective attraction between micelles and Na^+ ion concentration control the growth of aggregates, their volumes and aggregation numbers. Hence the sodium ion in the bentonite plays a vital role in the change of these parameters by effectively reducing charge on the micellar aggregates.

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Abstract

Paper Ref: 30

EFFECT OF N-TERMINAL AMPHIPHILIC PEPTIDE REGION ON AGGREGATION OF OVALBUMIN

Professor Mitsuhiro Shibayama [1], Mr. Yuya Okazumi [1], Mr. Takashi Hiroi [2], Dr. Ken Littrell [3], Ms. Yuri Narita [4], Prof. Naoki Tanaka [4]

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[3]*Oak Ridge National Laboratory*
[4] *Macromol. Sci. Eng. Dept.*

Aggregation of protein is widely observed in our daily life. For example, cooking is manipulation of protein state. Main cause of various human diseases such as Alzheimer's and Parkinson's diseases is also considered to be aggregation of protein. One of model proteins is ovalbumin (OVA), which is a major protein in egg white. An OVA aqueous solution aggregates at high temperature and forms gel like sunny-side up above the threshold concentration. This phenomenon has been researched thoroughly from the viewpoint of turbidity, rheology, spectroscopy, scattering and so on. Then we, as chemists, think the next step for this research is manipulation of the aggregation state by modifying the chemical structure. Kawachi *et al.* concentrated on the N-terminal amphiphilic peptide region ($pN_{1\text{-}22}$) and proved that this peptide region enhances the strength of OVA gel from the viewpoint of rheology.[1] In contrast, aggregation ability of OVA without this peptide region (pOVA) is dramatically reduced. We assume that the reason for this phenomenon originates from the amphiphilic nature of the peptide. The aim of this research is to clarify the role of $pN_{1\text{-}22}$ and the relationship between the microscopic chemical structure and the macroscopic physical properties.

To clarify the mesoscopic structure, we conducted a SANS measurement at GP-SANS, High Flux Isotope Reactor at ORNL. Samples are solutions or gels of OVA, pOVA, peptide and their mixture with various concentrations before and after heating. pH of samples was set to 7, which is common condition for the application of OVA and their derivatives. We observed a strong upturn at low-*q* region in SANS curves for pOVA solutions/gels after heating. This behavior is similar to a phase separation of well-known poly(N-isopropylacrylamide) (PNIPA) solutions. From this result, we can see that the lack of amphiphilic peptide region makes the OVA solute unstable and promotes aggregation. In contrast to this, addition of amphiphilic peptide region does not change SANS profiles noticeably even after heating. This means that the peptide enhances the strength of gels without changing the original structure and is desirable for application.

1 Y. Kawachi *et al.*, *J. Agric. Food Chem.*, **61**, 8668 (2013).

Abstract

Paper Ref: 363

Neutrons, deuteration and synchrotron X-rays for the study of biology and advanced materials: A match made in atoms…

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Together, the Australian Synchrotron in Melbourne and the OPAL research reactor, at the Bragg Institute in Sydney represent Australia's largest ever investment in scientific infrastructure. Both facilities commenced operation in 2007, have passed through their infancy and adolescence to take their place amongst the rank of top-flight international user facilities. Far from middle-aged, these two vibrant landmark facilities (each with 10 operational beamlines) and along with the National Deuteration Facility at ANSTO have provided transformational research capabilities for the Australian scientific community. Although modest in size compared to the well-established international competition, both institutions are producing excellent amounts of high-quality research with the Bragg Institute and the Australian Synchrotron generating more than 200 and 450 peer-reviewed publications per annum respectively.

At first glance both synchrotron and neutron sources show similar scientific profiles, encompassing an extremely wide range of disciplines: materials, chemistry, biology, condensed matter physics, nanotechnology, engineering, geosciences, archaeology and studies relating to cultural heritage. Common to both are advanced capabilities for the study of atomic and molecular structure, as well as operational studies of functional materials under a diverse range of extreme environments. A more forensic examination however reveals fundamental differences in their DNA. While the biological, pharmaceutical and medical research communities drive substantial capability development and research outcomes at the Australian Synchrotron, neutron scattering and molecular deuteration at the Bragg Institute provides a focus for studies in soft condensed matter, physical and inorganic chemistry, solid state physics and crystallography.

Although their respective probes are generated from different parts of the atom and interact with matter in fundamentally different ways, my presentation will highlight several case-studies where the distinct properties of neutrons, selected deuteration, and synchrotron light come together to help solve complex problems associated with cellular biology [1] and the development of a range of advanced materials [2-7].

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Plenary Lecture 5

Abstract

Paper Ref: 452

Neutrons for Chemistry – AONSA Prize Lecture July 2015

Professor John White[1]

[1] *Research School of Chemistry, Australian National University, Canberra, Australia*

The business of chemistry is synthesis, reaction mechanisms and both the structure and properties of what is made. Its scope ranges from materials science to biology. In solid-state chemistry – diffraction is “king” but for reactions in solution, a great challenge is to find out how reactions proceed between molecules on a timescale of a few picoseconds and over distances of a few angstroms. Neutron scattering in the 1960s held the potential for this (1). Now, progressive instrument development, computing and increased neutron fluxes provide a wonderful (Q, □) domain to do it.

The lecture describes, by means of examples, experiments in the last 50 years at Oxford, the ILL and in Australia to establish some things that neutrons can do in chemistry. For inelastic scattering, the calculable mode eigenvector weighting in molecular crystals (2) (now a routine molecular spectroscopic method) was a useful step and for molecular liquids, partial deuterium substitution (3) showed relative motion of molecular parts. In polymer science these methods again were useful (4) and in surface chemistry quasi- elastic scattering from thin water in clays and liquid crystals showed the effects of confinement (5) and the crystal field splitting of rotational tunnelling excitations gave local structure and dynamics at interfaces (6). In small angle scattering, deuterium substitution showed the self-assembly mechanism of templated silicates (7) and of industrial emulsions (8). Nano-toxicology-relevant protein-nanoparticle interactions (9) are now of interest – provoking, along the way, a need to better understand recombinant protein aggregation.

In all of this work, the simplification of the neutron scattering law using isotopic and other contrast variation methods has been needed. What is now available experimentally in space-time resolution will reopen much of what was found before in great depth. Our challenge is to use this by attracting such excellent young colleagues as I have been blessed with in the past.

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Thursday 23 July 2015

Plenary Lecture 6

Abstract

Paper Ref: 62

Introduction of the Reactor Source Spectrometers and Recent Research Progress in China Academy of Engineering Physics

Professor Jian Gong[1]

[1]Institute of Nuclear Physics and Chemistry, China Academy of Engineering Physics

The China Mianyang Research Reactor (CMRR) with the power of 20 MW is located in Mianyang City, Sichuan Province. It was limited open to users officially from 2012, including the thermal and cold neutron halls. The liquid hydrogen cold neutron source began to work from September 2013. The measured thermal and cold fluxes for neutron scattering experiments are 2.4×10^{14} n/cm^2·s and 10^9 n/cm^2·s, respectively.

In our institute, CMRR is devoted to both fundamental and applied research. In the first phase, eight neuron scattering instruments have been installatied and start operation from the middle of 2014. Four thermal neutron instruments were installed in the reactor hall: a high resolution neutron diffractometer (HRND), a residual stress neutron diffractometer (RSND), a thermal neutron radiography station (TNR) and a high pressure neutron diffractometer (HPDC). Four cold neutron instruments were installed in the guide hall: a small-angle neutron spectrometer (SANS), a time-of-flight and polarized neutron reflectometer (TPNR), a cold-neutron triple-axis spectrometer (CTAS) and a cold neutron radiography station (CNR). The HRPD has an optimum resolution of $\Delta d/d=0.2\%$ with a wavelength of 1.88 Å. For the RSND, the maxiumu integral neutron flux at the sample position is 4.7×10^6 n/cm^2/s. Moreover, the wavelength can be tuned from 1.2 to 2.8 Å, and its resolution is $\Delta d/d\leq0.25\%$. Recently, texture measurement was realized on the RSND by utilizing the Kappa goniometer. The PDC is equipped with a He-3 array detector containing 70 detection units. High/low temperature environment with a range of 10~1200 K and high pressure loading system with a maximum pressure of 20 GPa are also installed on the PDC. The length of the reflection room of the SANS is 13 meters, and the range of reflection vector is 1×10^{-2}~5.0 nm^{-1}. The characteristics of the TPNR are the time-of-flight and polarization modes. Relative resolution of momentum transfer of the TPNR is $\Delta q/q=(0.5\sim5)\%$ for $q=(0.05\sim3.0)$ nm^{-1}. The CTAS has an energy resolution of $\Delta E/E\leq2\%$ with a range of energy transformation of 0~10 meV.

Since the neutron scattering instruments at CMRR came into service, several experiments have been done by users from Peking University, Chinese Academy of Science, et al. and some results are published. Besides, some research experiments have also been done by ourselves and collaborators. In situ neutron diffraction measurements on zircaloy-4 alloy and NiTi alloy

have been performed on the RSND. A lot of materials have been studied by the diffraction measurements performed on the PDC, such as magnetic material $Mn_{54}Al_{46}$ (provided by Peking University), neutron irradiated Al1.0wt%B, permanent magnet material TbFeMn (RT and 500 ºC), superconducting material $FeSe_{0.2}Te_{0.8}$ and so on. Analysis of the effects of the environmental temperature and humidity on the microstructure of Estane and measurements of the precipitates in zironium alloys are performed on the SANS. Moreover, some film sample tests, such as NiFe/NiFe, Ac/Estane et al, have been done on the TPNR. The detailed analysis will be presented on the conference.

Recently, the second phase for neutron scattering instruments development has been approved. In situ loading systems will be equipped for the present spectrometers, such as high/low temperature environment, high pressure loading system, superconducting magnetic field loading system, chemical loading device (hydrogen/deuterium gas environment), et al. Furthermore, three new instruments, polarized thermal-neutron triple axis spectrometer (TTAS), ultra small angle neutron spectrometer (USANS), and neutron standard test beam-line (NSTB) will be built during the next years. Details of the progress will also be presented.

CMP-5 Magnetism

Abstract

Paper Ref: 447

A Tale of Two Kagome Magnets: From Nèel to Valence-bond Solid State

Kit Matan[1]

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Spin dynamics of two structurally similar spin-1/2 distorted kagome lattice antiferromagnets $Cs_2Cu_3SnF_{12}$ and $Rb_2Cu_3SnF_{12}$ will be discussed. Magnetic excitations in $Cs_2Cu_3SnF_{12}$, which has an ordered ground state owing to strong Dzyaloshinskii-Moriya interactions, can be qualitatively described by linear spin-wave theory. However, we found that the excitation energies are renormalized by a factor of approximately 0.6. This negative renormalization is attributed to quantum fluctuations, and is in contrast to the positive quantum renormalization observed in a quantum-spin chain. On the other hand, magnetic excitations in $Rb_2Cu_3SnF_{12}$, whose ground state is a “pinwheel” valence-bond solid state, can be understood in terms of singlet-to-triplet excitations. Recent high-intensity pulsed neutron scattering reveals a weak dispersive and “ghost” mode, which is attributed to an enlarged unit cell below a structural transition. We also observed continuum scattering at high energy, which is probably reminiscent of the fractionalized excitations recently observed in herbertsmithite. In addition, field-dependence of the singlet-to-triplet excitations will be discussed.

Abstract

Paper Ref: 197

Magnetic structure of Melilite-related centrosymmetric $K_2CoP_2O_7$: neutron diffraction and DFT study

Mr Matthew Sale [1, 2], Dr Maxim Avdeev [1], A/Prof Chris Ling [2], A/Prof Prabeer Barpanda [3]

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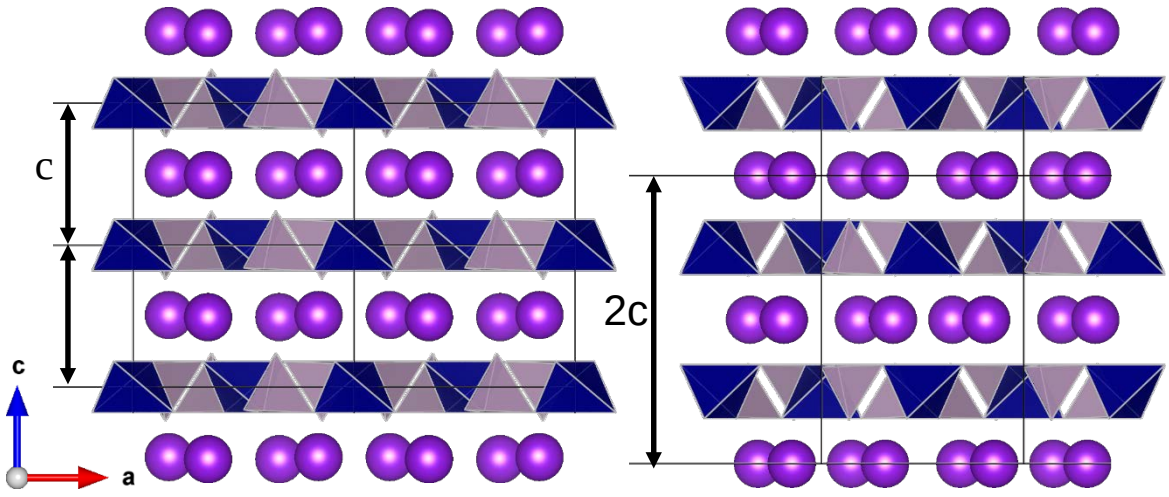
[2] *School of Chemistry, University of Sydney*

[3] *Materials Research Centre, Indian Institute of Science (IISc)*

A great number of $X_2(ZT_2)O_7$ compositions with large X=Ln, Ca-Ba, Na, K and small Z and T=Be,Al,Si,Ga,Ge,P,V adopt the melilite crystal structure type. The structure is built of tetrahedral layers of five-member rings and X is located in the interlayer space. Typically, the materials crystallize in the non-centrosymmetric P-42_1m space group. The compositions with magnetic ions (e.g. Z=Mn^{2+},Fe^{2+},Co^{2+},Cu^{2+}) are very interesting systems from the magnetic structure point of view due to the 2D character of the structure and lack of inversion center. Depending on chemistry and geometry of a particular composition, a variety of magnetic structures were reported, from simple 3D antiferromagnets to 2D spirals and multiferroics.

During our investigations of melilite-type materials a $K_2CoP_2O_7$ composition was prepared and its magnetic property and structure characterized. The material is built of melilite-type tetrahedral layers but crystallizes in centro-symmetric P4/mnm space group. The magnetic structure has been determined using neutron powder diffraction on the Echidna diffractometer (ANSTO) and further explored with DFT. The results will be presented and discussed in the context of the broad melilite structural family.



Left: Melilite $K_2MnV_2O_7$ with space group ‘P_421m’ (113), coloured grey, purple and blue respectively, ICSD: 417892, Yahia HB, 2007, Z. Naturforsch. B

Right: Melilite-type $K_2CoP_2O_7$ with space group ‘P42/mnm’ (136), coloured grey, purple and blue respectively, structure from neutron powder diffraction

Caption: As shown in figure, the second structure is similar to the first except that every second layer is rotated by 90 degrees which approximately doubles the c unit cell parameter

Abstract

Paper Ref: 145

Determination of magnetic order in nanoparticles of zinc ferrite via neutron diffuse scattering with polarization analysis

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5 Institut Laue-Langevin, 71 av des Martyrs, 38042 Grenoble, France (now at European Spallation Source, Lund, Sweden)

We have studied a series of nanostructured $ZnFe_2O_4$ samples produced by mechanical milling by neutron diffuse scattering, over the temperature range (1.5–295 K). In this study we have used polarization analysis to unambiguously differentiate between atomic and magnetic contributions. From the magnetic neutron diffraction we deduce that our samples possess tetragonal antiferromagnetic symmetry corresponding to the magnetic space group I_c222 [1, 2]. The best fit between the magnetic model and data is where moments lie in the basal plane. It is known that inversion between Zn on the A sites and Fe on the B sites of the AB_2O_4 lattice increases systematically with increased milling (as particle size decreases) [3], leading to marked changes in the magnetic correlations. Consistent with earlier reports on Zinc Ferrites [3, 4], we find that the long range magnetic order (which appears below T_N~10 K) gradually transitions from antiferromagnetic to ferrimagnetic with decreasing particle size and we have determined the relative strength of these two components. In addition we observe a marked increase in magnetic short range order above T_N. We have modeled this assuming participation only of B-site Fe atoms and find that the correlations are antiferromagnetic in nature and extend out to a distance of 9 Å. We are working on an interpretation of the change in magnetic properties from bulk to nano-particulate Zinc Ferrite through combination of crystallographic and microstructural factors based on a model with distinct core and shell contributions.

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Abstract

Paper Ref: 346

Magnetic structure and phase transition on NiO

Sanghyun Lee[1], Yoshihisa Ishikawa[1], Ping Miao[1,2] , Shuki Torii[1,3] , Toru Ishigaki[4] and Takashi Kamiyama[1,2,3]

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Neutron diffraction has been important technique to understand the magnetism. Louis Néel anticipated antiferromagnetic states theoretically and C. G. Shull *et al*. carried out neutron powder diffraction and found antiferromagnetic structure on transition metal monoxide TMO(TM=Mn, Fe, Co, and Ni). Since their pioneer works stimulated the research on various magnetic phenomena in condensed matter physics, many textbook explained TMOs as the introduction of antiferromagnetism with neutron powder diffraction [1]. Many researchers who read the textbook thought their magnetic structure was already solved completely, but actually their magnetic moment directions have been unclear for so long time and one of most delicate problem in condensed matter physics. Crystal and magnetic transition occurred simultaneously by exchangestriction and magnetostriction that causes ferroelastic domain even though it was grown as single crystal. These twin domains induce the complicated condition on experiments and analysis of magnetic moment direction. *R-3m* crystal structure and magnetic propagating vector k_m=(0 0 1.5)$_h$ are well accepted for both MnO and NiO in the research community. Their magnetic moment direction is believed inside the hexagonal plane. However, MnO is 1st order phase transition while NiO is 2nd order phase transition experimentally. To explain the difference of magnetic structure and phase transition between MnO and NiO, we carried out time-of-flight neutron powder diffraction on NiO using SuperHRPD beamline in J-PARC, Japan. Even though we couldn't observe monoclinic peak splitting which is too smaller than about $\Delta d/d$=0.02%, we constructed *C2/m* monoclinic structure with k_m=(0 1 0.5)$_m$ and assigned $\Gamma_2(B_g)$ magnetic structure on NiO by symmetry analysis [2]. We argue that MnO magnetic moment direction is along 2-fold axis while NiO is inside mirror plane in *C2/m*. This different magnetic moment direction and symmetry will be responsible to 1st and 2nd order phase transition on MnO and NiO respectively.

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MSC-6 Phase transitions

Abstract

Paper Ref: 219

In-situ neutron diffraction study for ice XV – a revisit to its stability region

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[2] *CROSS-Tokai, Tokai, Ibaraki, Japan*
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Ice crystals show the unusual structural variety – there are at least 17 forms of polymorphs (Ih, Ic, II-XVI). Most of them, apart from ice X, is found less than 300 K and 3 GPa. The appearance of many phases in a narrow pressure-temperature (*p*-*T*) region and sluggish reaction under low-*T* prevent us to understand its thermodynamical stability relations of respective phases. Here we report our recent results for ice XV [1], the ordered form of ice VI, investigated at the PLANET beamline [2] in J-PARC by using the *p*-*T* variable MITO system [3]. The high intensity with moderate resolution ($\Delta d/d$ ~ 0.6 %) of the PLANET beamline allows us to obtain cell parameters in minutes, revealing the reaction kinetics for order-disorder transition between ice VI and ice XV. Our in-situ observations at ambient to high-*p* and low-*T* revealed that ice XV is formed at ambient pressure and 128 K, whereas it diminishes at 0.4 GPa and the same temperature. These findings clearly show the necessity of slight modification for the currently accepted phase diagram of ice [1].

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[2] Hattori T., et al. (2015) Design and performance of high-pressure PLANET beamline at pulsed neutron source at J-PARC. Nucl. Instrum. Meth. A, 780, 55-67.

[3] Komatsu K., et al. (2013) Development of a new P–T controlling system for neutron-scattering experiments. High Press. Res., 33, 208-213.

Abstract

Paper Ref: 212

High-pressure transformations in sulfuric acid hydrates

Helen E. Maynard-Casely[1] Takanori Hattori[2], Shinichi Machida[3], Asami Sano-Furukawa[2], and Kazuki Komatsu[4]

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[3] Comprehensive Research Organization for Science and Society, Japan.
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The Galilean moons of Jupiter; Io, Europa, Ganymede and Callisto, have surfaces that are composed of very different materials to the silicates that make up our Earth. For Europa and Ganymede, two moons under intense investigation from past and future space missions, their surfaces are made up of ice and hydrates. Despite the apparent 'simplicity' of these materials, we still observe very complex geological formations on these moons – including subduction [1]. This means we need to understand the transformations of candidate surface materials under a range of pressure/temperature conditions in order to accurately explain the formations on these icy surfaces.

One hydrate candidate material for the surfaces of these moons are sulfuric acid hydrates, formed from radolytic sulfur (from Io) reacting with the surface ice. Sulfuric acid hydrates have already been established to have a complex phase diagram with composition [2].

We have now used the Mito cell [3] at the PLANET instrument [4] to undertake the first investigation of the high-pressure behavior of the water rich sulfuric acid hydrates. Compressing at 100 K and 180 K we see that the hemitriskaidekahydrate becomes the stable water-rich hydrate and observe some interesting relaxation behaviour in this material at pressure, which could have significant consequences for the interiors of Ganymede.

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2. Maynard-Casely, H.E., H.E.A. Brand, and K.S. Wallwork, *Phase relations between the water-rich sulfuric acid hydrates, potential markers of thermal history on Jupiter's icy moons.* Icarus, 2014. **238**(0): p. 59-65.

3. Komatsu, K., et al., *Development of a new P–T controlling system for neutron-scattering experiments.* High Pressure Research, 2013. **33**(1): p. 208-213.

4. Hattori, T., et al., *Design and performance of high-pressure PLANET beamline at pulsed neutron source at J-PARC.* Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment, 2015. **780**(0): p. 55-67.

Abstract

Paper Ref: 112

Alkali and Alkaline metal oxide materials for high temperature CO_2 sorption studies

Adriano F. Pavan[1],Peter Blanchard[1], Samuel Liu[1] and Chris D. Ling[1]

[1] *School of Chemistry, University of Sydney, NSW 2050, Australia*

In recent years, a number of novel ceramic oxide materials have emerged that are capable of absorbing CO_2 at high temperatures (>500^{O}C) while remaining stable over a large number of cycles and a wide range of temperatures [1]. The most promising are been considered for carbon capture applications – specifically, for use in combustion chambers and the smoke stacks of power plants where combustion gases which contain primarily a mixture of CO_2 and N_2 at high temperature. Compared to other CO_2 sequestration technologies, these ceramics have some advantages (eg. chemisorption at high temperatures) and disadvantages (eg. limited kinetics over time) [3].

Examples of oxides already known to show significant CO_2 absorption include Li_5AlO_4, $Li_6Zr_2O_7$, Na_2ZrO_3 and $Ba_4Sb_2O_9$. The phase formations and structural evolution of these metal oxides have been studied under environmental conditions mimicking those found in combustion chambers and power plants, over the temperature range 873–1173 K.

CO_2 absorption by these materials is believed to proceed through a layering effect of the sorbent material, explained through a core-shell model. Each phase is represented as a layer covering a particle, with the outermost layer exposed and allowed to react with the environment. Detailed studies into the mechanism of CO_2 absorption and the material layers will shed more information that can be used to fine tune the materials to increase their CO_2 absorption capacity.

Previous work has focused on the identification of phases ex situ and studies of their practical absorption capacity and kinetics. The new work we will present here uses a combination of a x-ray spectroscopy, x-ray and neutron diffraction, to understand both how the sorption process works and how the structural evolution of the phases affects the CO_2 sorption of the materials over time in-situ.

References

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[2] Choi, S., Drese, J. H., Jones, C. W., Chem.Sus.Chem, 2009, 2, 796
[3] Favre, E. Journal of Membrane Science, 2007, 294, 50–59

Abstract

Paper Ref: 85

High temperature magnetic and structural studies of $Ba_XSr_{1-X}TcO_3$

Emily M Reynolds[1], Brendan J Kennedy[1], Gordon J Thorogood[2], Ken R Czwerwinski[3], Frederic Poineau[3]

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Of all the 4d transition metals, the solid-state chemistry of technetium is by far the most mysterious. This is a result of the challenges associated with the synthesis and characterization of radioactive materials. Beyond the intrigue of the unknown, Tc-chemistry is important in the development of the nuclear fuel process as it is a major constituent of high-level nuclear waste, and so the phases it forms with other fission products such as Sr-90 is of great interest. Furthermore, technetium oxides have been shown to exhibit exciting magnetic behavior; the perovskite $SrTcO_3$ has the highest magnetic ordering temperature in a compound without 3d transition elements.[1, 2] The distorted perovskite retains G-type antiferromagnetic order up to a remarkable temperature of 1000 K.

Following computational results[3], in order to increase the Néel temperature we have attempted to dope the larger Ba cation onto the *A*-site. The series $Ba_xSr_{1-x}TcO_3$ was successfully synthesized with x = 0.1, 0.2, 0.3, 0.4. The ambient and high temperature structures of the samples have been determined using synchrotron X-Ray diffraction, collected on the powder diffraction beamline at the Australian synchrotron. The structures vary with composition from *Imma* to *I4/mcm*, as expected from the temperature induced phase transitions.

Unlike electrons, neutrons have a magnetic moment, which allows them to interact with the magnetic structure, and so we require neutron diffraction to probe the magnetic structure of these materials. Of course, a consequence of low neutron flux is the requirement of a relatively large sample size, and this was a non-trivial challenge to overcome for Tc-containing compounds. Neutron diffraction data was collected on the Echidna instrument at ANSTOs Bragg institute, at low, ambient and high temperatures. The results of the Rietveld refinements of the magnetic and crystallographic structures will be presented.

1. Thorogood, G.J., et al., *Structural phase transitions and magnetic order in SrTcO3.* Dalton Transactions, 2011. **40**(27): p. 7228-7233.
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3. Borisov, V.S., et al., *Magnetic exchange interactions and antiferromagnetism of ATcO(3) (A = Ca, Sr, Ba) studied from first principles.* Physical Review B, 2012. **85**(13).

Abstract

Paper Ref: 126

High Temperature Structural Phase Transformations in Strontium Uranium Oxides

Gabriel L. Murphy[1], Brendan Kennedy[1], Zhaoming Zhang[2]

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[2] *Australian Nuclear Science and Technology Organisation, Sydney, Australia*

The renaissance that is currently enveloping the nuclear power industry has reinvigorated research attempts into optimising UO_2 based fuels. Despite the first electricity being supplied from a power reactor occurring over 60 years ago, little progress has been made on the fundamental solid state chemistry of the oxide fuel matrix and its interaction with fission daughters. This has resulted in a potential nuclear waste epidemic as no long term viable solution is yet available for medium to high level waste.

We have extensively investigated the AUO_4 monouranate material type where *A* is Ca, Sr and Ba with focus on $SrUO_4$ since Sr, a common and very hazardous fission product within fuels in the case of Sr-90. Initial investigation using *in situ* synchrotron X-ray diffraction (SXRD) and thermogravimetric analysis (TGA) revealed $SrUO_4$ exists in two structural types, a metastable rhombohedral (α) and a stable (but sensitive to oxygen vacancies) orthorhombic (β) form. The structural transformation was found to occur in an anomalous manner, seemingly facilitated via the generation of reduced forms of uranium through a rapid ejection of coordinating oxygens by a first order mechanism (figure 1.)

To further elucidate this peculiar structural behaviour, *in situ* neutron powder diffraction (NPD) measurements in conjunction with X-ray absorption near edge structure (XANES) measurements were undertaken using the echidna diffractometer at the Australian Nuclear Science and Technology Organisation's Opal nuclear reactor and at the Australian Synchrotron respectively. These complementary experiments demonstrated that along with the required generation of reduced forms of uranium, the transformation to the orthorhombic structure is dependent upon reabsorption of atmospheric oxygen in order to generate a stoichiometric oxide. Without this reabsorption the material cannot obtain thermodynamic stability, continuing as a structurally metastable material with extensive oxygen vacancies at high temperature.

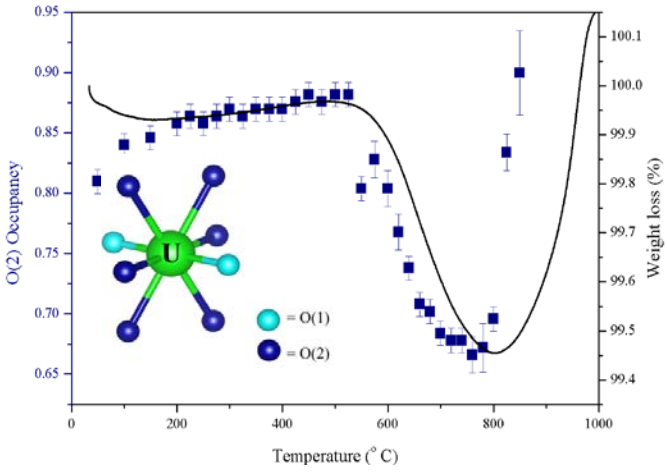

Figure 2. Refined O(2) occupancy for rhombhohedral-$SrUO_4$ transformation from SXRD and TGA weight changes between 25 and 1000 °C. The fully coordinated rhombohedral UO_8 is shown as reference to the transformation mechanism.

SMT-3 In-situ and polarization methods

Abstract

Paper Ref: 52

In-Situ Neutron Reflectometry during Thin Film Growth by Sputter Deposition

W. Kreuzpaintner[1], S. Mayr[1], J. Ye[1], B. Wiedemann[1], A. Paul[1], T. Mairoser[2], A. Schmehl[2], A. Herrnberger[2], J. Stahn[5], J.-F. Moulin[4], P. Korelis[5], M. Haese-Seiler[4], M. Pomm[4], B. Hjörvarsson[6], P. Böni[1], and J. Mannhart[3]

[1] *Technische Universität München, Garching, Germany*
[2] *Universität Augsburg, Augsburg, Germany*
[3] *Max-Planck--Institut für Festkörperforschung, Stuttgart, Germany*
[4] *Helmholtz-Zentrum-Geesthacht Zentrum für Material- und Küstenforschung GmbH, Geesthacht, Germany*
[5] *Paul Scherrer Institut, Laboratory for Neutron Scattering, Villigen PSI, Switzerland*
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Thin magnetic layers and heterostructures thereof are the basic building blocks of a large number of magneto-electronic devices. Their performance strongly relies on the magnetic properties of the layers they consist of. These are functions of the layers' morphology and microstructure and on the coupling between them. Since these parameters can change during the process of growth, it is important for the understanding and optimisation of magnetoelectronic devices to not only accurately monitor the structural but also the magnetic properties during the process of growth.

While the structural characterisation of thin films during growth by various techniques is common practice (as e.g. commonly done by RHEED/LEED, STM or synchrotron radiation), the *in-situ* measurement of the magnetic properties of films using (polarised) neutron reflectometry is a challenging task. Within a collaboration of TU München, University Augsburg and MPI Stuttgart, we operate a mobile sputtering facility for the growth and *in-situ* monitoring of magnetic multilayers, which can be installed at suitable neutron beamlines. In our contribution, the setup and first proof of principle polarised *in-situ* neutron reflectivity measurements on *in-situ* grown Fe/Cr carried out at the ToF reflectometer REFSANS at the FRM II neutron source and at the AMOR beamline at PSI will be presented. At the latter, use of the Selene neutron optical concept allows very fast polarised neutron reflectivity measurements to be performed within only 15min per spin direction.

Abstract

Paper Ref: 26

FIRST APPLICATION OF SIMULTANEOUS SANS AND DIFFERENTIAL SCANNING CALORIMETRY: MICROPHASE SEPARATED ALKANE BLENDS

Mr Stuart Pullen [1], Mr Scott Olsen [1], Mr Benjamin Day [2], Mr Ferdi Franceschini [1], Mr David Mannicke [1], Mr Norman Booth [1], **Dr. Elliot Gilbert** [1]

[1] *Australian Nuclear Science and Technology Organisation (ANSTO)*
[2] *Hobsons Instruments Services*

For almost 30 years, it has been possible at synchrotron facilities to perform small-angle x-ray scattering experiments whilst simultaneously measuring phase transitions using differential scanning calorimetry (DSC). However, a range of challenges exist to enable the collection of simultaneous small-angle neutron scattering (SANS) and DSC data associated not only with intrinsic flux limitations but also scattering geometry and thermal control. The development of a DSC (temperature range ca. −150 C to 500 C) suitable for SANS is detailed here which, to our knowledge, is the first and only one of its kind. An example study is presented from the 40 m SANS instrument, QUOKKA, at the OPAL reactor at ANSTO (Figure 1), concerned with phase transitions in a binary blend of normal alkanes in which one component has been deuterium labelled[1]. The ability to conduct simultaneous DSC and neutron scattering studies allows investigators to use these two complementary techniques to provide insight into structural and thermal changes and opens up the opportunity for SANS to make significant new contributions to a range of systems in which either scattering contrast is insufficient for SAXS studies or where neutron scattering is essential or inherently desirable (e.g. isotope effects).

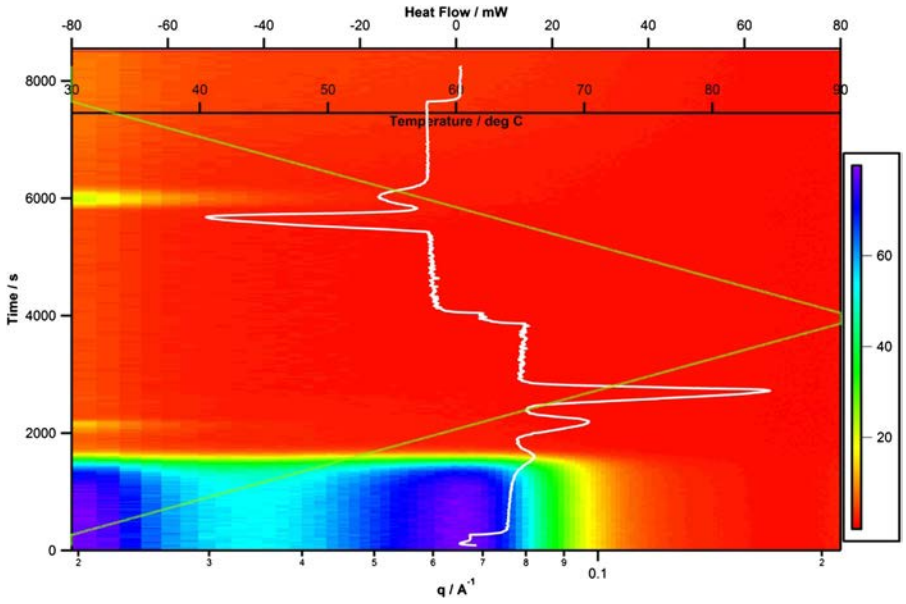

Figure 1 - Time-evolution of SANS from demixed equimolar $C_{30}H_{62}$:$C_{36}D_{74}$ blend during heating and cooling at 1 K min^{-1}; DSC data, collected simultaneously, are superimposed. Intensity is shown on a linear colour map (right). DSC data are shown as a solid curve and the temperature profile of the DSC experiment is shown as a dotted line.

References

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Abstract

Paper Ref: 48

Advances of High Pressure *in-situ* Neutron Diffraction at CMRR

Leiming Fang[1], Xiping Chen[1], Lei Xie[1], Guangai Sun[1], Bo Chen[1], and Duanwei He[2]

[1] *Institute of Nuclear Physics and Chemistry, China Academy of Engineering Physics, Mianyang, China*

[2] *Institute of Atomic and Molecular Physics, Sichuan University, Chengdu, China*

High-pressure in-situ neutron diffraction technology, which developed very quickly especially in advanced countries, has many unique technology advantages in many important fields over the range from based scientific researches to national security strategy. A reactor neutron source (China Mianyang Research Reactor, CMRR), which is one of the only three neutron sources for neutron scattering in China, was newly built and operated in our institute (China Academy of Engineering Physics, CAEP). Base on it, we have been developed and established high pressure extreme environment on our neutron diffraction spectrometer. The aim of our plan is to make the pressure up to 20 GPa, the temperature exceed 1500 K, as well as the sample size larger than 2 mm. By using a newly designed large-volume panoramic-type opposed anvil cell, we have been realized the high pressure condition of *in-situ* neutron diffraction up to 10 GPa recently. Details of the progress and the related researches by using high pressure *in-situ* neutron diffraction will be presented on the conference.

Abstract

Paper Ref: 277

Advances in Neutron Polarisation Analysis Capability for Material Sciences Research on OPAL Instruments

Wai Tung Hal Lee[1], Tim D'Adam[1] , David Jullien[2], Ken Andersen[3], Pierre Courtois[2]

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[2] *Institut Laue Langevin, Grenoble, France*
[3] *European Spallation Source, Lund, Sweden*

We are opening to the user community a new material sciences research capability of using neutron polarization analysis on the instruments in the OPAL reactor at the Australian Nuclear Science and Technology (ANSTO). Polarised neutron scattering is a powerful technique that can cleanly separates the magnetic scattering of magnetic moments and magnetic excitations from the nuclear scattering of the chemical structure and structural dynamics. We can identify the location, strength, and direction of the magnetic moments to atomic resolution and the strength and polarization of magnetic excitations with this technique. In addition, it allows us to separate the structural signal and the hydrogen background signal in hydrogen-rich materials which has long been a challenge in studying organic materials with neutrons. At ANSTO, polarisation analysis has previously been available on the reflectometer PLATYPUS for thin film and multilayer studies. The operation of a 3He polarising station has now provided this capability for more instruments. We have now measured the magnetic structure of multiferroic single-crystals and giant-magnetocaloric powder samples on the WOMBAT diffractometer and the TAIPAN triple-axis spectrometer. We have also measured the polarization and location of magnon excitation in a multiferroic single-crystal on the TAIPAN triple-axis spectrometer. These capabilities are now opened to users in the research community. Commissioning tests have been done for polarised off-specular scattering capability on PLATYPUS to study lateral magnetic surface structure. It will soon be followed by polarization analysis on the cold neutron chopper spectrometer PELICAN for magnetic excitation measurements and polarised SANS on QUOKKA for magnetic nanostructured material and hydrogen-rich material studies. This presentation will highlight the material sciences measurements done using this new capability and present the main characteristics of this technique.

SMS-5 Polymers and surfaces

Abstract

Paper Ref: 439

Polymer/Water Interfaces Revealed by Neutron Reflectivity

Hideaki Yokoyama[1]

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A layer of polymer chains tethered by one end to a surface is called polymer brush and known to show various unique properties such as prevention of protein adsorption and anti-fouling activity. However, the characterization of polymer brush is still not straightforward since the brush layer is embedded between solid and water interface. One of limited number of analytical methods to reveal solid/water interface structures is neutron reflectivity (NR). We have been applying NR to reveal the problems related to polymer brush at solid/water interfaces and here present two subjects related to polymer brush.

The first subject will be dynamic polymer brush which utilizes the surface segregation phenomena of copolymers with surface-active blocks for preparing polymer brush in spontaneous process. We have reported hydrophilic polymer brushes formed at the interface between water and hydrophobic elastomer by the segregation of amphiphilic diblock copolymers blended in the elastomer. In this system, while the hydrophilic block with high surface energy avoids air surface, upon contact with water the hydrophilic block segregates to cover the interface between hydrophobic elastomer and water. Surprisingly high density dynamic polymer brush at D_2O/polymer interfaces was observed by NR.

The second subject will be evaluating inclusion kinetics of polyrotaxane formation using NR. A polyrotaxane is composed of a polymer chain and cyclic molecules such as polyethylene glycol (PEG) and cyclodextrin (CD). Inclusion complex formation of a polymer chain with cyclic molecules is an important step to synthesize polyrotaxanes. However, inclusion complex formation induces gelation or precipitation of the complex, and hence makes detailed observation of the reaction difficult. We fixed polymer chains on a substrate, which is polymer brush, and conducted in-situ time slice NR measurement. NR results showed that brush layer thickness gradually increases and reaches its plateau by inclusion complex formation, of which time scale depends on the concentration of cyclic molecules and other characteristics of the CDs.

Abstract

Paper Ref: 84

Distribution of Glass Transition Temperatures and Dynamic Heterogeneity of Polymer Thin Films

Toshiji Kanaya[1], Hiroki Ogawa[1], Rintaro Inoue[1], Norifumi Yamada[2] and Thomas Proksha[3]

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[2]*High Energy Accelerator Research Organization, Tsukuba, Ibaraki-ken 305-0801, Japan*

[3] *Laboratory for Muon-Spin Spectroscopy, Swiss Muon Source SμS, Paul Scherrer Institut, 5232 Villigen PSI, Switzerland*

Polymer thin films are utilized in many industrial fields including lithography, coating, lubricant and so on, and the extensive studies have been performed to reveal that thermal and mechanical properties of polymer thin films were quite different from those of bulk. Thickness dependence of glass transition temperature T_g and thermal expansivity, ultra-slow expansion and contraction process, and the dewetting at a temperature lower than bulk T_g are some of the representative examples for the anomalous properties of polymer thin films.

In this work we have studied the distribution of glass transition temperature T_g in polystyrene (PS) thin films consisting of alternatively stacked deuterated polystyrene (d-PS) and hydrogenated polystyrene (h-PS) layers ~20 nm thick using neutron reflectivity by SOFIA in J-PARC/MLF. The result clearly showed very heterogeneous dynamics in PS thin films.

In addition, we also investigated the T_g distribution of the alternatively stacked film as well as non-labeled PS thin films using low energy muon spin relaxation (mSR) technique, which clearly showed the deuterium labeling had no large effects on T_g in PS thin films. On the basis of the results, we will discuss the heterogeneous dynamics of polymer thin films in the conference.

Abstract

Paper Ref: 57

CHARACTERISATION OF HIERARCHICALLY-STRUCTURED CELLULOSE HYDROGELS BY SMALL ANGLE NEUTRON SCATTERING

Dr Marta Martinez-Sanz [1, 2], Dr Patricia Lopez-Sanchez [2], Dr Deirdre Mikkelsen [2], Si-Qian Chen [2], Dr Bernadine Flanagan [2], Prof. Michael J. Gidley [2], Dr Elliot P. Gilbert [1]

[1] *Bragg Institute, Australian Nuclear Science and Technology Organisation*
[2] *ARC Centre of Excellence in Plant Cell Walls, University of Queensland*

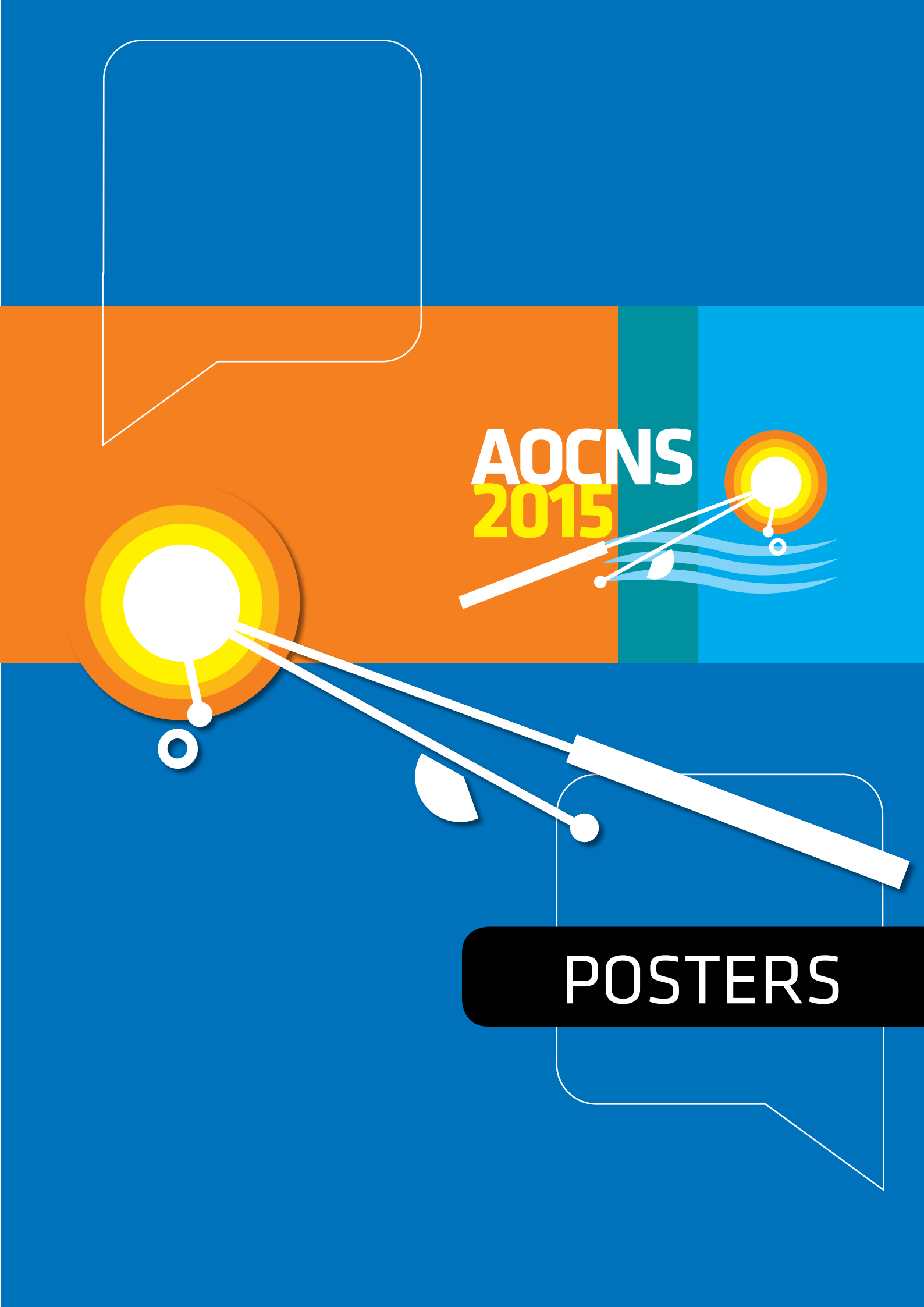
This work reports on the characterisation of cellulose hydrogels by means of small angle neutron scattering (SANS), combined with complementary techniques such as small angle X-ray scattering, X-ray diffraction, NMR spectroscopy and electron microscopy. Pure cellulose hydrogels were synthesized by cultivation of *Gluconacetobacter xylinus* strains in glucose-containing media. Composites were also produced by incorporating polysaccharides typically found in plant cell walls (PCW) into the culture media.

The application of a multi-technique characterisation approach enabled elucidation of the complex hierarchical architecture of cellulose hydrogels. Cellulose ribbons, typically modelled as solid one-phase structures, were proven to consist of a sub-structure of cellulose microfibrils interacting with each other and with solvent by means of a dense hydrogen bonding network. The existence of such sub-structure led to the creation of regions with different solvent accessibility within the ribbons, as indicated by the SANS data of pure and composite cellulose hydrogels. Based on this, a core-shell cylinder model combined with an interfacial scattering term was applied to fit the SANS contrast variation data. The fitting results suggested a different effect on the ribbons' solvent exchange for the diverse composite hydrogels and, supported by additional characterisation, highlighted the distinct interaction mechanisms between cellulose and PCW polysaccharides.

Furthermore, the production of partially deuterated cellulose hydrogels by using a deuterated glucose-based feedstock was seen to effectively enhance the neutron scattering length density contrast, opening new possibilities to selectively match the different components in composite hydrogels. The structure of the deuterated cellulose was compared with the native protiated cellulose and SANS contrast variation experiments confirmed the presence of solvent trapped within the cellulose ribbons, behaving differently to the bulk solvent surrounding them.

The ability of diverse bacterial strains to produce cellulose hydrogels with different yields and properties was also investigated. The distinct solvent accessibility suggested by the SANS results was related to the different structures synthesised by the various bacterial strains.

These results evidence the ability of SANS, combined with additional techniques, to provide valuable insights on cellulose biosynthesis and interactions with PCW polysaccharides.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Monday 20 July 2015

Poster Session 1 – BST, SMS and SMT

Abstract

Paper Ref: 422

PO01

Deuterated recombinant protein production: A high yield, robust and reliable method using *Escherichia coli*

Anthony P Duff[1], Karyn L Wilde[1], Agata Rekas[1], Natalia Davydova[1], Peter J Holden[1]

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In support of neutron scattering studies of proteins, we use a highly reliable method for the deuteration of a broad range of proteins by recombinant expression in *Escherichia coli* BL21. Typical biomass yields are 40-80 g/L wet weight, yielding 50-500 mg/L purified protein. This method uses a simple, relatively inexpensive defined medium, and routinely results in a high yield expression without need for optimisation. The key elements are: very tight control of expression, careful starter culture adaption steps, media composition and strict maintenance of aerobic conditions ensuring exponential growth. Culture temperature is reduced as required to prevent biological oxygen demand exceeding maximum aeration capacity. The defined medium has glycerol as the sole carbon source and we have not encountered an upper limit for the size of proteins that can be expressed, achieving excellent expression for proteins from 11-154 kDa. The quantity produced at 1L scale ensures that no small angle neutron scattering (SANS), nuclear magnetic resonance (NMR) or neutron crystallography experiment is limited by the amount of deuterated material available. Where difficulties remain, these tend to be cases of altered protein solubility due to high protein concentration and a D_2O-based environment. This method is also applied to the recombinant multiple labelling (^{13}C, ^{15}N, ^{2}H) of proteins for NMR investigations.

Abstract

Paper Ref: 104

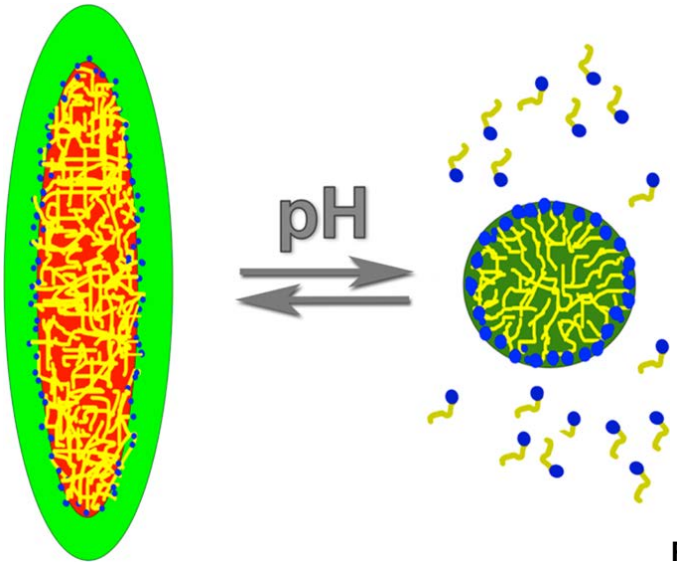
PO02

STRUCTURAL INVESTIGATION INTO THE INFLUENCE OF LIPOLYSIS PRODUCTS ON THE STRUCTURE OF BILE SALT MICELLES

Ms Stephanie Phan, Dr. Stefan Salentinig, Prof. Ben Boyd, Dr. Nigel Kirby, Dr. Tamim Darwish, **Dr. Elliot Gilbert**

1 Monash Institute of Pharmaceutical Sciences,
2 Australian Synchrotron,
3 National Deuteration Facility, ANSTO
4 Bragg Institute, ANSTO

Free fatty acids play a vital role as fuel for cells and in lipid metabolism. During lipid digestion in the gastrointestinal tract, triglycerides are hydrolyzed resulting in the amphiphilic products free fatty acids and monoglycerides. These components, together with bile salts, are responsible for the transport of lipids and poorly water soluble nutrients and xenobiotics from the intestine into the circulatory system of the body. In this study we show that the self-assembly of digestion products from medium chain triglycerides (tricaprylin) in combination with bile salt and phospholipid is highly pH responsive. Individual building blocks of caprylic acid within the mixed colloidal structures are mapped using a combination of neutron scattering combined with both solvent contrast variation and selective deuteration as well as synchrotron-based small angle X-ray scattering (1). Modelling of the scattering data shows transitions in size and shape of the micelles in combination with a transfer of the caprylic acid from the core of the micelles to the shell or into the bulk water upon increasing pH. The results help to understand the process of lipid digestion with a focus on colloidal structure formation and transformation for the delivery of triglyceride lipids and other hydrophobic functional molecules.



Reference: Stefan Salentinig*, Stephanie Phan, Tamim Darwish, Nigel Kirby, Ben Boyd, Elliot Gilbert*, Langmuir 30 (2014) 7296-7303.

Abstract

Paper Ref: 213

PO03

Small angle scattering examination of structures self-assembled during human breast milk digestion

Stefan Salentinig[1], Stephanie Phan[1], **Adrian Hawley[2]**, Ben J Boyd[1]

[1] *Monash Institute of Pharmaceutical Sciences, Parkville, Australia*
[2] *Australian Synchrotron, Clayton, Australia*

Human breast milk is the critical part of an infant's diet and the complete diet for a baby's first months of life. As such, breast milk contains both water soluble and water insoluble components all of which are made bioavailable during digestion to provide everything needed for growth and development. Following the recent discovery of self-assembled structures as bovine milk is digested,[1] a similar progression through a range of different self-assembled structures has been studied during the in-situ digestion of human breast milk.[2] These highly organized structures were studied using small angle scattering to follow the formation and progression of the structures in real time during digestion. Cryo-TEM was also used to study the phases formed. The duration of the digestion, pH and bile salt concentration were found to act together to gradually shift the lipophilic environment inside breast milk fat globules to more hydrophilic surfaces in highly ordered structure with high internal surface area. The formation and transitions in self-assembled structures are likely to be key to making water insoluble species bioavailable in the gastrointestinal tract of the infant. Further small angle scattering studies will allow a greater understanding of how individual components impact the digestion and whether additional species, e.g. adding sparingly soluble vitamins, changes the digestion stages. Understanding the different stages and structures of digestion offers scope to develop improved nutritional supplements or controlled release drug delivery systems for infants.

[1] S Salentinig, S Phan, J Khan, A Hawley, BJ Boyd "Formation of highly ordered nanostructures during the digestion of milk" (2013) *ACS Nano* 7:10904-10911

[2] S Salentinig, S Phan, A Hawley, BJ Boyd "Self-Assembly Structure Formation during the Digestion of Human Breast Milk" (2014) *Angewandte Chemie (International Edition)* 127:1620-1623

Abstract

Paper Ref: 404

PO04

Kumatori Deuteration Station (KIDS) Project

Rintaro Inoue[1], Masaaki Sugiyama[1]

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It is quite well known that small-angle neutron scattering (SANS) is a powerful tool to study the quaternary structures of protein in solution state. One of the most fascinating characteristic features in SANS technique is its ability to discern hydrogen (H) from deuterium (D) due to the difference of scattering lengths. Taking advantage of intrinsic feature contrast variation SANS (CV-SANS), which modulates the both the contrasts of solvents and solutes make the detailed structural analyses on complex system possible. In addition deuteration labeling on protein samples offer little perturbation on their structure and functionality compared to other labeling method such as site-directed mutations. However the structural analyses on protein through SANS is quite limited compared to small-angle X-ray scattering (SAXS) despite of its unique property.

It is considered that one of the obstacles for SANS study is the difficulty for the preparation of enough amounts of deutertaed proteins. In order to overcome such a situation we launched KumatorI Deuteration Station (KIDS) project, which dedicated to the preparation of deuterated (or controlled-deuterated) protein. At the poster session we would like to present our recent activity and fruitful results from KIDS project.

Abstract

Paper Ref: 69

PO05

The Structure of Lipopolysaccharide from Rough Strains of *Escherichia coli* at the Air – Liquid Interface

Anton P. Le Brun[1], Luke A. Clifton[2], Candice E. Halbert[3], Binhua Lin[4], Mati Meron[4], Peter J. Holden[1], Jeremy H. Lakey[5] and Stephen A. Holt[1]

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The outer membrane (OM) of the Gram-negative bacterial cell envelope is an important biological interface as it initiates bacterial cell adhesion, activates innate immunity during infection, withstands environmental stresses and acts as a gate keeper to nutrients and toxins. The OM is a complex structure consisting of an asymmetric bilayer with an inner leaflet of phospholipids and an outer leaflet of lipopolysaccharide (LPS) with LPS consisting of approximately 75 % of the Gram-negative bacterial surface. Due to the complexity of LPS there are very few OM models that include this important molecule and thus little is known about LPS structure at interfaces. In this work we used a truncated LPS from *E. coli* (Rc-LPS, consisting of Lipid A and the first seven sugars of the core polysaccharide) to form stable monolayers at the air – liquid interface. Neutron and X-ray reflectometry were used to probe the vertical structure of the monolayers, with the neutron reflectometry enhanced by the production of a deuterated Rc-LPS. The lateral structure was studied with grazing incidence X-ray diffraction and Brewster angle microscopy. It was found that at surface pressures above 20 mN/m the Rc-LPS monolayers could be resolved as hydrocarbon tails, inner headgroup, and outer headgroup of polysaccharide with increasing solvation from tails to outer headgroup. The lateral organisation of the hydrocarbon tails was of an oblique hexagonal unit cell across all surface pressures with only the tilt angle of the chains changing with surface pressure [1]. The study of the monolayer structure provides the first complete analysis of a realistic *E. coli* OM surface model.

[1] A. P. Le Brun et al. *Biomacromolecules* **14**, 2014 (2013)

Abstract

Paper Ref: 181

PO06

Small Angle Neutron/X-Ray Scattering Study of Microtubules and Polycations

Juncheol Lee[1], Jimin Lee[1], Chaeyeon Song[2], Herbert P. Miller[2], Kyuhan Kim[1], Leslie Wilson[2], Cyrus R. Safinya[2] and Myung Chul Choi[1]

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[2] *University of California, Santa Barbara, California, USA*

Microtubules (MTs) are hollow cylindrical protein nanotubes with 25 nm diameter, composed of α/β-tubulin heterodimers with surface charge density ~ e/nm^2. They are involved in many cellular functions such as cell division, maintaining cell shape, and intracellular trafficking. There have been studies about higher-order assemblies of MTs (e.g. Hexagonal bundle of MTs, inverted tubulin tubes, etc) in the presence of multivalent cations (e.g. Ca^{2+}, spermine, etc). We show our recent findings on the assembly structures of MTs and cationic polymers, which have different structures as the concentration of the polycations changes. Various assemblies are studied both in real and reciprocal spaces using small angle Neutron/X-ray scattering and transmission electron microscopy.

Abstract

Paper Ref: 288

PO08

STRUCTURAL STUDIES OF LIPOSOMES USING SANS

Dr Kathleen Wood[1]

[1]Bragg Institute - ANSTO

Membrane formation is often listed as one of the key steps in the origins of life on this planet, since this enabled the first organisms a certain control over the environment around their genetic material. Unilamellar liposomes are one of the simplest models available of cellular membranes, and small angle neutron scattering is an effective technique to characterize their structure.

Bacterial and eukaryotic cells differ in their lipid composition, notably in an increase in charged lipids for bacterial membranes. We are currently studying charged and uncharged liposomes.

New Sources, Methods and Techniques

Abstract

Paper Ref: 424

PO09

DEVELOPMENT OF HIGH-PRESSURE SINGLE-CRYSTAL NEUTRON DIFFRACTION ON KOALA

Mr Jack Binns [1], Prof Garry McIntyre [2], Prof Simon Parsons [3], Dr Stephen Moggach [3], Dr Konstantin Kamenev [3]

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[2] *ANSTO*
[3] *University of Edinburgh*

Hydrogen bonds are one of the most important classes of intermolecular interaction, and accurate H-atom positions are critical for analysis of the energy terms which determine the thermodynamic stability of molecular crystals. At ambient pressure and low temperatures, H atoms can often be located by X-ray diffraction, and X-ray data can provide an accurate picture of the intermolecular contacts.

High-pressure experiments do not afford this luxury. The high systematic errors introduced by the pressure cell and low completeness mean that H-atom positions are not revealed in X-ray Fourier maps. In some compounds H-atom positions can be inferred from the positions of other atoms, but this is not possible in other cases.

Neutron diffraction data are much more sensitive to H than are X-ray data, and they are essential in cases where accurate H-atom location is important. Neutron powder patterns of complex molecular systems suffer from extensive peak overlap, and single-crystal diffraction therefore has a huge advantage; there is also no need to deuterate.

Initial developmental experiments using a high-pressure cell on the KOALA Laue diffractometer at ANSTO have shown the feasibility of this technique for single-crystal studies at high pressure. The implications of the high-pressure sample environment for Laue diffraction will be explored and the results from experiments on hexamine will be presented.

Abstract

Paper Ref: 55

PO10

Design and Implementation of a DSC for the Simultaneous Measurement of SANS

Stuart A Pullen[1], **Norman Booth[1]**, Scott R Olsen[1], Benjamin Day[2], Ferdi Franceschini[1], David Mannicke[1] and Elliot P Gilbert[1]

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[2]Hobsons Instruments Services, 1/6 Sherbourne St, Essendon Vic 3042, Australia

The combination of small-angle x-ray scattering experiments with simultaneous measurement of phase transitions using differential scanning calorimetry (DSC) has become common-place. However, no such facility existed, till recently to enable the simultaneous measurement of DSC and small angle neutron scattering (SANS). DSC data complements the SANS data by providing first and second order phase transition temperatures and, with appropriate calibration, the magnitude and sign of the associated enthalpy changes due to these phase transitions. A range of challenges have been addressed to enable the collection of simultaneous SANS and DSC data associated not only with intrinsic flux limitations but also scattering geometry, thermal control and synchronisation of the DSC and SANS data. The development of a DSC suitable for SANS is detailed here which, to our knowledge, is the first and only one of its kind. The resulting instrument has a temperature range of -50ºC to 500ºC and a furnace geometry that allows access to the full q range of QUOKKA, at the OPAL reactor to be reached. The DSC-SANS was first used to investigate the behaviour of binary mixtures of normal alkanes, a subset of data collected is presented. Several successful experiments have been carried out since the trial presented here.

References[1] Pullen S.A., Booth N., Olsen S.R., Day B., Franceschini F., Mannicke D. and Gilbert E.P.*, Measurement Science Technology, 25 (2014) 055606.

Abstract

Paper Ref: 100

PO11

Status of Neutron Beam Facilities at HANARO and A Thermal Neutron Guide Project of KAERI

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After successful installation of cold neutron facilities at HANARO such as neutron guides, cold neutron source including cold neutron instruments, now 14 cold and thermal neutron spectrometers are operating, and 5 instruments are under commissioning.

The neutron guides with complicated shapes placed in the beam plug and the main shutter also in the curved part were delivered by a guide provider but the rest guides such as the guides in the guide bunker and the guide hall area were fabricated by KAERI. All the guides are coated with M=2 supermirror having different cross-sections and curvatures were operating with a high performance, where 10 cold neutron spectrometers will open to outside users.

For a planning of a new project called ‘thermal guide facilities development’, the neutron guide system design started late last year, which was carried out to optimize the layout of the instruments and to calculate the neutron flux at sample position.

At this meeting, the simulation results of the thermal neutron guide beam lines, status of in-house neutron guide development and specifications of some instruments will be presented.

Abstract

Paper Ref: 284

PO12

EMU, THE COLD-NEUTRON BACKSCATTERING SPECTROMETER AT THE BRAGG INSTITUTE

Dr Nicolas de Souza[1], Dr Gail Iles[1], Dr Alice Klapproth[1]

[1]*Australian Nuclear Science and Technology Organisation*

The Bragg Institute is presently commissioning a cold-neutron backscattering spectrometer in the ANSTO OPAL research reactor neutron guide hall. First neutron spectra from its commissioning are presented.

The spectrometer, called EMU, is based on Si (111) crystal backscattering and extracts neutrons from a cold neutron guide via a double HOPG (002) crystal premonochromator setup. Backscattering is realized through implementation of spherical focusing between the Si (111) crystal monochromator and analyser arrays, so as to achieve a high FWHM energy resolution, in order of 1.2 μeV.

EMU also features a unique 7-meter long focusing guide between its two HOPG premonochromators, allowing measurements over the ± 31 μeV energy transfer range enabled by its Si monochromator Doppler drive, all the while maintaining high count rates at its 3He linear-position sensitive detector arrays.

EMU is well suited to characterizing diffusion and tunneling processes as occurring in e.g. polymer chains, membranes, proteins, molecular crystals, between interstitial crystal lattice sites, as well as observing hyperfine splitting of nuclear energy levels. Relaxation times from a few 10 ps to over 1 ns are accessible for momentum transfers up to 1.95 Å^-1, and over temperatures ranging from 50 mK up to 800 K. Other sample environments such as pressure, magnetic fields, controlled gas delivery, etc. are also available upon request.

Abstract

Paper Ref: 93

PO13

SIKA --- the Cold-Neutron Triple-Axis Spectrometer with Multiplexing Analyzer at Bragg Institute

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[2] Neutron Group, National Synchrotron Radiation Research Center, Taiwan

[3]Hahn-Meitner Institut - Berlin, Germany

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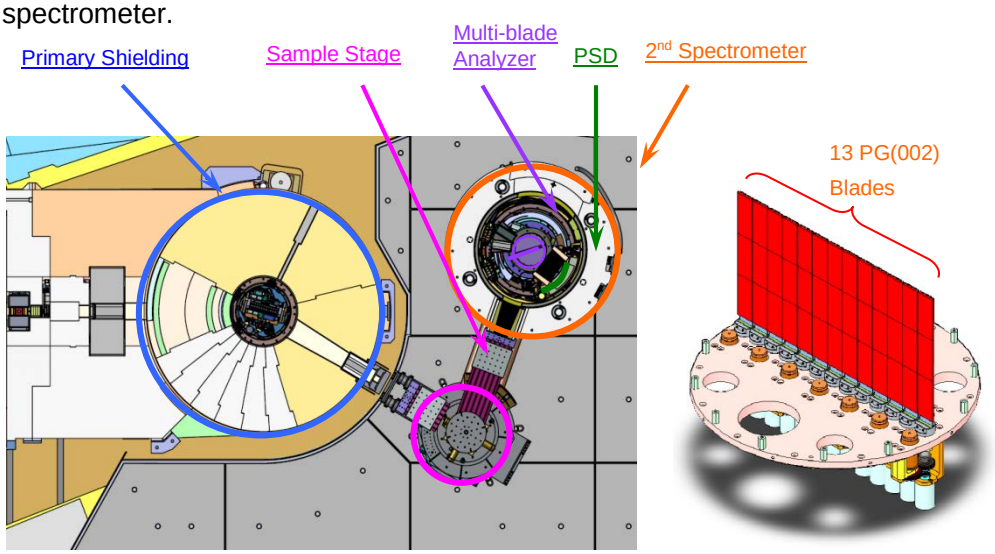
SIKA is a high-flux cold-neutron triple-axis spectrometer funded by Ministry of Science and Technology of Taiwan and currently being operated by National Synchrotron Radiation Research Center. It locates at the OPAL reactor face at the Australian Nuclear Science and Technology Organisation (ANSTO). Its incident energy ranges from 2.5meV to 30meV with the highest flux at ~8meV. With an advanced design, SIKA is equipped with an analyzer array of 13 PG(002) blades (Fig. 1), a multi-wire detector, and a separate diffraction detector. Such a design allows SIKA to run in a traditional step-by-step mode or in various mapping (or dispersive) modes by changing the configuration of analyzers and detectors. Several typical mapping modes are analyzed and simulated using Monte Carlo ray-tracing package SIMRES of RESTRAX. [1] The performance of different mapping modes are demonstrated and evaluated, providing the dispersion relations of these operation modes as references for experimental studies. In hot-commissioning, a multiplexing mode with constant E_f was used to measure the phonon dispersion in a Pb single crystal. The simulation and experiment results demonstrate the flexibility and fast data-collecting potential of SIKA as a next generation cold neutron triple-axis spectrometer.



* Corresponding author: guochu.deng@ansto.gov.au

Fig. 1. Left: top view of SIKA; Right: the SIKA analyzer stage consisting of 13 individually controlled PG(002) blades.

[1] J. Šaroun, J. Kulda, "Neutron ray-tracing simulations and data analysis with RESTRAX",SPIE proceedings 5536 (2004), 124-133

Abstract

Paper Ref: 390

PO14

Design and study of sample table of neutron spectrometer

Wu Yunxin[1], **Gong Hai[1]**, Li Yang[1], Chen Kanghua[1],Deng Hua[1],Zhu Changjun[1],Hu Zhigang[1],Yang Liu[1],Li Qunming[1],Xie Jinghua[1]

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A sample table was designed for neutron diffraction experiment.The mian parameters:maximum l oad is 1000 kg;four degrees of freedom movement; measurable space l 800 mm*800 mm*400mm;high accuracy in positioning samples is 30μm.
Due to the height limits for the incident position of neutron source, the unique multi stage telescopic structure was adopted to ensure that the height of the entire table was agreed w ith the requirement of measuring position. The path planning and optimization methods were studied to avoid that sample collide with slit system and to achieve the goal of shortening the locating time and reducing the path error.
The digital filter of sensor signal was studied to improve the performance of the control system. The gas lubrication technology of large load and high precision were studied to make the sample table move conveniently and accurately and to reduce the vibration and noise.

Abstract

Paper Ref: 125

PO15

NEUTRON AND X-RAY RADIOGRAPHY: A NATURAL SYNERGY

Dr Chris Hall[1], Dr Joseph Bevitt[2]

[1] *Australian Synchrotron*
[2]*ANSTO*

It is understood in the radiography community that images of objects made using penetrating electromagnetic waves (x-rays) and particles (neutrons) provide complementary information on the composition of the object. This fact has been exploited to great effect in many radiographic studies in the past. The opportunity to use both of these imaging techniques within Australia is now available. The Australian Synchrotron x-ray source is running an imaging beam line and the Opal reactor neutron source has recently opened a radiography facility too. Both facilities allow computed tomographic imaging to provide 3-D information on the object. IMBL images with a beam that can be up to 400 mm wide and 40 mm high. Monochromatic x-rays are available in beams from 20 KeV up to 200 KeV. Dingo provides a 200 mm by 200 mm thermal neutron beam and optional energy selection using a velocity selector.

This presentation will discuss the technical aspects of the two facilities and how these tools may be used in, particular for archeological and paleontological science.

Abstract

Paper Ref: 321

PO16

Single-crystal neutron diffraction data analysis using *LaueG* and *Crystals*

Gail N. Iles[1], Ross Piltz[1], Alison Edwards[1]

[1] *Australian Nuclear Science and Technology Organisation, Sydney, Australia*

The Laue diffractometer, KOALA, at the Bragg Institute offers the capability to measure large portions of reciprocal space in a single image. The data reduction process is handled by an in-house programme, *LaueG*, developed from the precursor programme, LaueGEN, from the 1980's. Given the lattice parameters of the sample and a space group, *LaueG* can be used to treat the Bragg reflections, generating an *hkl* file for use in an analysis programme. We present here the data treatment process using *LaueG* in order to minimise *R*-factors in the *hkl* file, and the data analysis process that turns this *hkl* file into a structure. To facilitate the discussion we will use the example of a Cu_2ZnSnS_4 sample which has site defects at the Cu and Zn positions, and for data analysis, the free-to-use software, *Crystals*.

Abstract

Paper Ref: 262

PO17

The International Society for Sample Environment (ISSE)

Paolo Imperia[1], Koji Kaneko[2], Seiko Ohira-Kawamura[3], Klaus Kiefer[4], Yamali Hernandez[5]

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[2] *Quantum Beam Science Directorate (JRR-3),JAEA, Tokai, Japan*

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[5] *NIST, Washington, USA*

Sample environment, which includes the fine control of physical parameters at the sample position, performing experiments at extreme conditions, and enabling complementary in-situ measurements, plays a crucial role in the success of almost every scattering experiment. Sample environment has shifted in the last decade, from purely technical infrastructure to a key component, critical to the scientific success of the experiments and the efficient use of precious beam-time at large scale facilities.

During the 8th International Workshop on Sample Environment, held in October 2014 at Eynsham Hall in the UK, representatives of 10 major facilities around the world founded the International Society for Sample Environment, with the aim of globally consolidating collaborations for all themes related to sample environment. The major goal of the Society, besides providing a stable home for the series of sample environment workshops and technical schools, is to provide a unique information exchange platform for the benefit of the international scientific community. The Society is open to anyone interested in sample environment and individuals and organisations are invited to join. In this presentation the newly formed Society goals, statutes and participation criteria will be illustrated.

Abstract

Paper Ref: 294

PO18

The Sample Environment and Support Available for User Experiments at the Bragg Institute

Paolo Imperia[1], Gene Davidson[1], Norman Booth[1], Stanley Lee[1], Andrew Manning[1], Timothy D'Adam[1], Nicholas Timperon[1]

[1] *ANSTO, Sydney, Australia*

The Bragg Institute, funded in 2002, operates a suite of 13 instruments dedicated to neutron scattering. In the short time since foundation, the Bragg Institute has become a significant user facility open to domestic and international researchers, competing and collaborating with neutron scattering facilities worldwide. To complete and complement the instruments, the Bragg Institute builds, procures and commissions a continuously evolving suite of sample environment equipment, able to determine and manipulate the physical conditions under which a neutron scattering experiment is performed. The sample environment equipment, therefore, plays a crucial role in the success of an experiment; the very role of sample environment today has shifted from purely technical infrastructure to an invaluable component, critical to the success of our user community. The sample environment equipment at the Bragg Institute is operated by a dedicated support team whose mission it is to help our users to achieve the maximum possible scientific outcome from experiments performed at the Institute. The range of equipment available spans from cryostats and cryofurnaces, superconductive magnets, high temperature, high pressure, gas and vapour delivery systems to more specialised equipment tailored to specific necessities.

Abstract

Paper Ref: 432

PO19

Recent progress of J-PARC MLF data analysis environment: instrument status data and remote access

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[3] *Computing Research Center, KEK, Tsukuba, Ibaraki, Japan*
[4] *Institute of Materials Structure Science (IMSS), KEK, Tsukuba, Ibaraki, Japan*

We report the recent progress on two fields of data analysis environment of J-PARC (Japan Proton Accelerator Research Complex) MLF (Materials and Life Science Experimental Facility). One is a utilization of event data of instrument status, another is a development of remote data access.

Event data of instrument status

TrigNET[1], which is a universal signal readout module developed in KEK, can acquire various instrument status, such as a tensile load or a goniometer position, as time-tagged event data in the same manner with a neutron detection signal. It allows us to treat both events of neutron detection and instrument status on an identical time-line. In the other words, instrument status can be handled at same time precision as the neutron detection event. In order to implement this function, our data analysis software has been upgraded. For example, this upgrade made flexible to analyze data under cyclic load-unload deformation or continuous sample rotation.

Remote data access

The remote data access is an key service for a large user facility such as J-PARC MLF. Developments of two components for the remote data access are on-going. The MLF experimental database system will provide users a search and a download for raw data and meta-data of their experiments through a web browser. The remote data analysis service will provide data analysis environment on a web browser utilizing computing resource at MLF.

[1] T. Seya, S. Muto and S. Satoh, MLF Annual Report 2010, 102 (2011)

Abstract

Paper Ref: 36

PO20

Installation of cold neutron triple-axis spectrometer at HANARO

Kwangpyo Hong[1], **Byoungil Jeon[1]**, Sungil Park[1]

[1]*Korea Atomic Energy Research Institute, Daejeon, Republic of Korea*

This paper presents a recent installation of a cold neutron triple-axis spectrometer (Cold TAS) at the 30 MW HANARO research reactor at the Korea Atomic Energy Research Institute (KAERI). Because of the importance of neutron science, a cold neutron facility construction project had been started in July 2003 and was finished in 2010. The Cold TAS was primary installed at HANARO during the project. After primary installation, some technical problems had been occurred, Cold TAS improvement was initiated in 2013. The basic design of the Cold TAS was inspired by other triple-axis spectrometers, but was modified slightly to improve reliability and efficiency of the instrument. Some features of the instrument are (a) access to a high-flux neutron beam with a neutron velocity selector (NVS) as a high order filter (HOF), which can also increase the signal-to-noise ratio by eliminating neutrons of the wrong wavelength upstream of the monochromator, (b) a custom tailored and efficient electronics control system with the SPICE interface, and (c) an in-house design and fabrication of a compact single-unit multi-analyzer and detector shielding body system that surrounds an array of pyrolytic graphite (PG) crystals. The Cold TAS was also developed to be flexible enough to facilitate different experimental resolutions and requirements. In its present setup, the instrument is capable of measuring the momentum and energy transferred to the sample in a range physically accessible by the spectrometer and is expected to help instrument users perform sophisticated experiments for future studies of the dynamic energy landscape in strongly-correlated electronic materials.

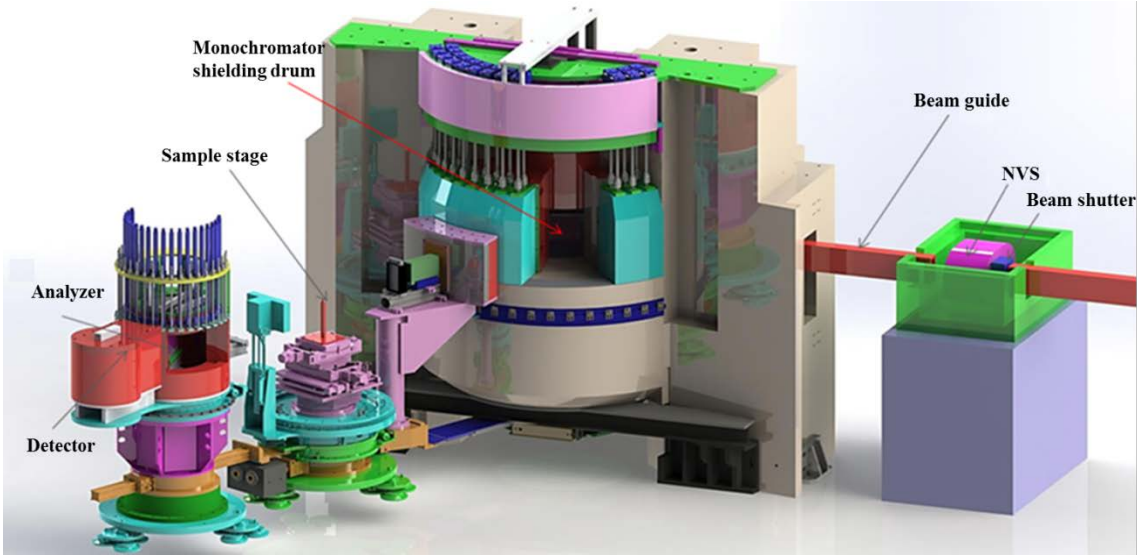

Fig. 1. A design layout of the Cold-TAS.

Abstract

Paper Ref: 163

PO21

Design study of Be-target for Proton Accelerator Based Neutron Source with 13MeV Cyclotron

[1,2]Jeong ho Kim, [1]Ohsung Oh, [1]Youngju Kim, [1]Hanseong Jeong, [2]Myungkook Moon, [2]Jongyul Kim, [3]TaeJoo Kim and [1]Seung Wook Lee

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[2] *Neutron Instrumentation Division, Korea Atomic Energy Research Institute, Daejeon, Republic of Korea*
[3] *Neutron Science Division, Korea Atomic Energy Research Institute, Daejeon, Republic of Korea*

There is a cyclotron named KIRAMS-13 in Pusan National University, Busan, Korea, which has the proton energy of 13MeV and the beam current of 0.05mA. Originally, it was developed for producing medical radioisotopes and nuclear physics research. To improve the utilization of the facility, we are considering the possibilities of installing a neutron generation target in it. The Beryllium target has been considered and neutrons can be generated by $^{9}Be(p,n)^{9}B$ reaction above the threshold proton energy of 2.057MeV.

In this presentation, we suggest candidate materials and structures, thicknesses, metal layers and cooling systems of target, which is optimal for the KIRAMS-13. We chose the Beryllium material of 1.14mm thick, which is calculated by stopping power of Beryllium, based on PSTAR, NIST. As for the cooling system, we chose to use water as a coolant, which will also act as a moderator. As protons pass through the target, hydrogen ions continue to pile up in the material and this makes the material brittle. To solve this problem, we chose Vanadium material because it has high hydrogen diffusion coefficient and short half-life isotope after being activated by neutrons. We simulated the neutron characteristics by the Monte Carlo simulation code, Geant4, CERN and performed thermal analysis on the target.

The design of target system is very important to produce neutrons for the desired purposes. There are several other existing facilities in Korea, in addition to the cyclotron facility considered in this study, where new neutron target system can be installed and neutrons can be generated. Two prominent facilities are KOMAC, Gyeongju and RFT-30, Jeongeup and we are planning to do study on the possibilities of utilizing the accelerators for neutron generation.

[1] B.N.Lee et al., Design of Neutron Targets with the 4 MeV Cyclotron for BNCT, Journal of Korean Physical Society, Vol. 59, No.2, pp. 2032-2034, D. Busan, Book Title, Publisher, 2011.

[2] Y. Yamagata et al., Neutron and Precision Engineering, Journal of the Japan Society for Precision Engineering, Vol. 79, No. 9, pp. 805-808, 2013.

Abstract

Paper Ref: 326

PO22

IN-SITU GAS SORPTION-DESORPTION AND VAPOUR DELIVERY SYSTEMS

Mr Stan Lee[1], Dr Norman Booth[1], Dr Hubert Chevreau[1], Dr Samuel Duyker[1], Dr Vanessa Peterson[1], Dr Paolo Imperia[1]

[1] Bragg Institute, ANSTO

Two systems are described which separately enable neutron scattering experiments with in-situ vapour delivery or multicomponent gas environments at programmable pressures. The gaseous environments are delivered to the sample through a capillary line and valve located centrally between the two sections by virtue of a gas delivery platform such as the Hiden IMI for gas desorption, or Hiden XCS for vapour control. All delivery lines are thermostatted to ensure no pressure errors or condensation.

Firstly, the gas delivery system is able to operate from vacuum (10^{-9} bar) to high pressure (200 bar) in both static and flowing gas delivery modes. The gas delivery system includes a sample positioning system for use in a top loading cryostat. The sample positioning system is separable into two sections with an independently sealable lower section able to be inserted into a glove box for sample cell attachment without risk of contamination. The IMI is capable of programming sequential in-situ sorption/desorption measurements with up to five gas species (including Helium) connected to the sample positioning system simultaneously. Absorption/desorption of gas on a sample is calculated volumetrically using molar balance expressions.

Secondly, the vapour delivery system is available in static or dynamic form. The dynamic system can deliver three gases (two of which may transport vapour) up to 200ml/min. The static system controls the vapour pressure above the sample (i.e. no flow of vapour) such that the sample is exposed to only the vapour as no carrier gas is required.

Both the gas delivery and dynamic vapour delivery systems can be used in conjunction with a portable quadrupole mass spectrometer, enabling trace analysis of the downstream gas composition.

Abstract

Paper Ref: 375

PO23

Progress on vertical neutron reflectometer upgrade at HANARO

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Polarised neutron reflectometry as an ideal tool to investigate the magnetic structure of thin films and interfaces, has been an essential instrument in neutron facilities. In HANARO (High-flux Advanced Neutron Application Reactor), we have been expanding polarized neutron reflectometry capabilities since the completion of the cold neutron guide in 2008. Polarised neutron reflectometry REF-V was utilized to study exchange coupling and magnetic anisotropy in layered magnetic thin film heterostructures. Recently we started vertical neutron reflectometer upgrade to meet user demands, and a new set of polarisers/spin flippers/analyser will be installed along with updated sample environments. We will present the upgrade status and discuss the future development.

Abstract

Paper Ref: 309

PO24

PHASE RESOLVED METHODS FOR INVESTIGATIONS OF ELECTRO-MECHANICAL ACTUATION

Miss Lisha Liu [1], Dr. Andrew Studer [2], Dr. John Daniels [1]

[1] *University of New South Wales*

[2] *Australian Nuclear Science and Technology Organisation*

When an electric field (static or dynamic) is applied to a bulk ceramic electro-mechanical material, transient states often exist which are not observed by static or ex-situ experiments. These transient states are critical for understanding electro-mechanical actuation mechanisms. Stroboscopic data acquisition approaches have allowed for detailed investigations of time-resolved structural changes under electric field when large changes to the structure occur. However, this technique is not sensitive to relatively small changes and often requires very long acquisition times, which lead to sample fatigue and failure. The event streaming mode data acquisition system of the Wombat neutron diffractometer at ANSTO involves the recording of each individual neutron event with its position and absolute time-stamp. This allows the post-processing of data in the most flexible way possible, for instance, applying phase sensitive demodulation of the data. This work explores the use of phase sensitive data analysis methods for creating the most sensitive time-resolved scattering experiments possible. The application to electro-mechanical materials where in-situ electric fields are applied will further our understanding of the very subtle structural response of these systems.

Abstract

Paper Ref: 259

PO25

Effective Transverse Coherent Extent of a Neutron Wave Packet

C.F. Majkrzak[1], C. Metting[1,2], B. Maranville[1], J.A. Dura[1], S. Satija[1], T. Udovic[1], and N.F. Berk[1,2]

[1]*Center for Neutron Research, NIST* Gaithersburg, MD 20899, USA
[2]*Department of Materials Science and Engineering, University of Maryland* College Park, MD 20742

Progress on measurements of the effective coherent extent of the neutron wave packet transverse to its mean propagation vector *k* is presented, expanding upon previous work [1]. There are two principal motivations for such an investigation, one being a fundamental physical interest in the characteristics of a free neutron as a quantum object whereas the other is of a more practical nature, relating to the understanding of how to interpret elastic scattering data when the neutron is employed as a probe of condensed matter structure on an atomic or nanometer scale. For instance, knowing the neutron's effective transverse coherence can dictate how to properly analyze specular reflectivity data obtained for material film structures that possess some degree of in-plane inhomogeneity. Determination of the effective transverse coherence length of the neutron wave packet via specular reflection from a series of diffraction gratings, each with a different period, is described.

[1] C.F. Majkrzak, C. Metting, B. Maranville, J.A. Dura, S. Satija, T. Udovic, and N.F. Berk, *Physical Review A* **89**, 033851 (2014).

Abstract

Paper Ref: 194

PO26

Advanced Workflow for Experimental Control

David Männicke[1], Norman Xiong[1], Nick Hauser[1]

[1] *ANSTO, Sydney, Australia*

Gumtree is a software product developed at ANSTO and used for experimental control as well as data visualization and treatment. In order to simplify the interaction with instruments and optimize the available time for users, a user friendly multi sample workflow has been developed for Gumtree. Within this workflow users follow a step by step guide where they list available samples, setup instrument configurations and even specify sample environments. Users are then able to monitor the acquisition process in real-time and receive estimations about the completion time. In addition users can modify the previously entered information, even after the acquisitions have commenced.

The presentation will focus on how ANSTO integrated a multi sample workflow into Gumtree, what approaches were taken to allow realistic time estimations, what programming patterns were used to separate the user interface from the execution of the acquisition, and how standardization across multiple instruments was achieved. Furthermore, the presentation will summarize the lessons learned during the development iterations, the feedback we received from our users and what future opportunities our approach enables.

Abstract

Paper Ref: 297

PO27

CALIBRATION OF HIGH-PRECISION TEMPERATURE SENSORS AND CONTROLLERS

Andrew Manning[1], Paolo Imperia[1], Norman Booth[1], Gene Davidson[1]

[1]*Bragg Institute, Australian Nuclear Science and Technology Organisation, Lucas Heights, Australia*

The ability to precisely and accurately measure and control the temperature of a sample is pivotal to providing a sample environment for neutron scattering experiments. The diversity of sample environments required in experiments, such as high or low temperatures and strong magnetic fields, necessitate the use of a variety specialised temperature sensors. These sensors must not only be carefully calibrated, but also have their calibration verified regularly to ensure that accuracy is maintained. The temperature controllers used to measure these sensors, such as the Lake Shore 336 and 340 models or the Oxford Mercury iTC, must also have their calibration verified so that their readings can be trusted.

We have developed procedures for ensuring that both the temperature sensors and controllers remain within the tolerances of their calibration. First, we present a simple, robust and flexible method to verify the calibration of temperature controllers using a calibrated resistor/voltage source and a laptop computer. This procedure can simulate commonly used sensors such as PT-100 resistance temperature detectors, DT-670 silicon diodes and K-type thermocouples, and is performed in-situ to avoid the down-time and cost associated with sending the devices away for calibration, as well as giving a better indication of the behavior of the devices as part of a running experiment. The results of this calibration check were then used to compare the performance of several Lake Shore 336 and 340 devices, as well as several Oxford Mercury iTCs.

A purpose-built calibration rig has also been designed to determine the correspondence between a calibrated sensor and other sensors which require their performance to be checked. Using a cold head and cartridge heater setup (similarly to other cryofurnaces), a wide temperature range can be accessed. Several features of this rig have been designed to ensure that the calibration of sensors can be performed quickly and accurately for a variety of sensor types. This setup could also allow new calibration curves to be composed for sensors which have drifted out of calibration.

Abstract

Paper Ref: 291

PO28

QUOKKA - THE 40 M PINHOLE SANS INSTRUMENT AT THE OPAL REACTOR

Dr Jitendra Mata[1], Dr Katy Wood[1], Dr Chris Garvey[1], Dr Elliot Gilbert[1]

[1]*Bragg Institute, ANSTO, Locked Bag 2001, Kirrawee DC, NSW 2232*

Quokka [1] is a conventional pin-hole reactor based small angle neutron scattering (SANS) instrument installed on the CG1 guide from the OPAL reactor at ANSTO (see Figure 1). Quokka is used to study a range of materials: polymers, food, proteins, metals, ceramics, rocks, magnetic clusters, and superconductors. Quokka receives neutrons from a 20 L liquid-deuterium cold source operating at approximately 20 K. Neutrons from the cold source pass through a velocity selector and a variable system of collimation and guides before interacting with the sample position. Recent installation of variable length (bellows) collimation pipe ensures minimum air travel for neutrons and also reduce alignment time significantly. A two-dimensional detector is situated at variable distance from the sample for measurement of the scattered intensity which can be optimized according to the demands of angular resolution or range. Data acquisition may be made in number of modes, including standard time or detector integral counts, stroboscopic, event mode which allows post binning of kinetic data and the summation of multiple time series binned according to an external trigger signal. Calibrated absolute intensity scattering measurements may be made from isotropic scatterers over a standard range of scattering vectors, q (6×10^{-4} Å^{-1} < q < 0.7 Å^{-1}) where the lowest value is achieved by use of focusing optics. Non-standard measurements may be made using a range of collimations, neutron wavelength and wavelength spread, incident beam polarization and subsequent analysis and incident beam dimensions. In the sample position Quokka supplies the user community with a range of measurement environments both standard and unique in the small angle scattering community. Some of these sample environments are, a Rapid Heat Quench Cell enabling a sample to be studied in situ following a thermal shock (-120°C to 220°C) [2]; The neutron Rapid Visco Analyser (nRVA) [3] enables SANS to be measured simultaneously with viscosity via an RVA – an instrument widely used within the food industry; In-situ Differential Scanning Calorimetry (DSC) [4]; A stopped flow cell, and a RheoSANS.

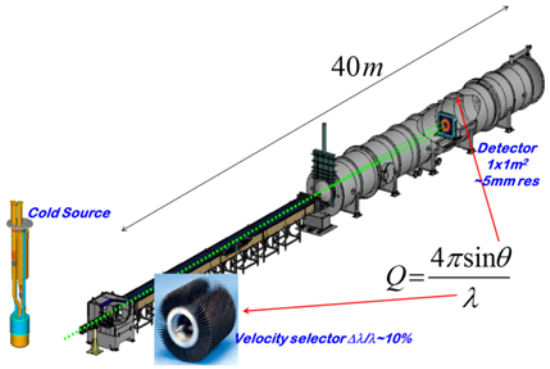


Figure1- Schematic view of the Quokka-SANS instrument at ANSTO

[1] E.P. Gilbert *et al.*, Physica B, 385-386 (2006) 1180.
[2] Stewart A Pullen *et al.*, Measurement Science and Technology, 19 (2008) 65707.
[3] James Doutch *et al.*, Carbohydrate Polymers, 88 (2012) 1061.
[4] S.A. Pullen *et al.*, Measurement Science and Technology, 25 (2014) 055606.

Abstract

Paper Ref: 240

PO29

Recent status of a cold neutron disk chopper spectrometer AMATERAS

Kenji Nakajima[1], Seiko Ohira-Kawamura[1], Tatsuya Kikuchi[1], Yukinobu Kawakita[1], Yasuhiro Inamura[1], Ryoichi Kajimoto[1], Mitsutaka Nakamura[1], Kazuhiko Soyama[1], Masahide Harada[1], Kenichi Oikawa[1], Shinichi Takata[1], Hiromichi. Tanaka[1], Takaaki Iwahashi[1], Wataru Kambara[1], Yasuhiro Yamauchi[1] and Kazuhiro Aoyama[1]

[1] *Materials & Life Science Division, J-PARC Center, Tokai, Ibaraki 319-1195, Japan*

AMATERAS (Fig. 1) is a cold-neutron multi disk-chopper spectrometer at Materials and Life Science Experimental Facility (MLF) in J-PARC [1]. By using a pulse shaping chopper and owing to the high peak intensity from a coupled moderator source at MLF, AMATERAS is designed to realize high intensity and fine and flexible energy resolution measurements in quasielastic and inelastic neutron scattering experiments from cold to thermal neutron energy region. The spectrometer has had the first neutron beam in May 2009 and had the first user in December 2009. The user program on AMATERAS is running successfully for 5 years. Until June, 2014, experiments of 61 general proposals have been carried out, which have resulted more than 20 papers, 5 theses and nearly a hundred of presentations at scientific meetings. In parallel to the user program, machine study and commissioning work to improve spectrometer performance have been continuously done. Recently, we have finished refurbishment of the beam transport section. Also, there is a progress in background reduction work, development of data analysis method and preparation in sample environments.

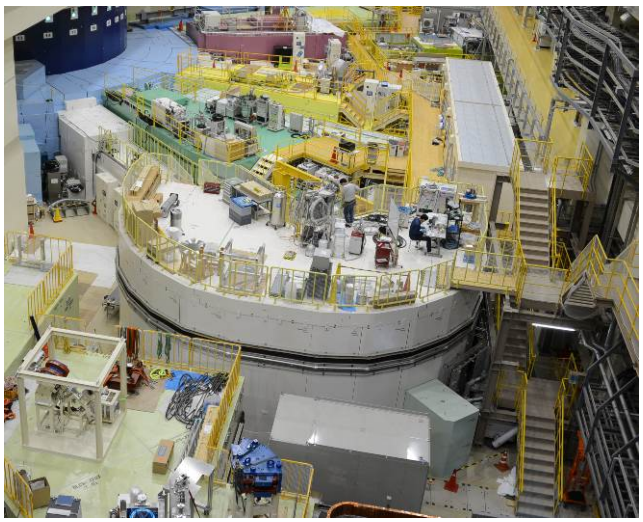


Figure 1. Recent photo of AMATERAS.

In this presentation, we will present the current status of AMATERAS together with ideas of future upgrade plans.

Reference

[1] K. Nakajima *et. al.,* J. Phys. Soc. Jpn., 80 (2011) SB028.

Abstract

Paper Ref: 376

PO30

Automatic Measurement Software in J-PARC, MLF

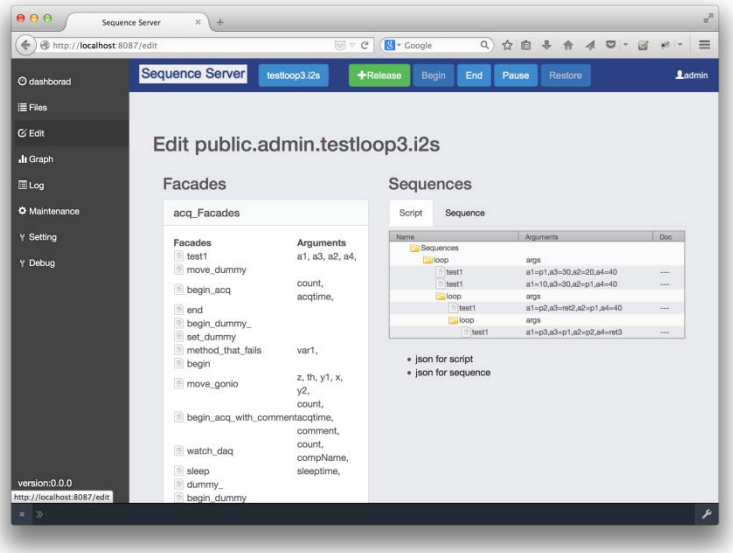
Takeshi Nakatani[1] , Yasuhiro Inamura[1] , Takayoshi Ito[2] , Toshiya Otomo[3]

[1] *Materials and Life Science Division, J-PARC Center, Tokai, Ibaraki, Japan*

[2] *Research Center for Neutron Science and Technology, Comprehensive Research Organization for Science and Society (CROSS), Tokai, Ibaraki, Japan*

[3] *Institute of Materials Structure Science (IMSS), KEK, Tsukuba, Ibaraki, Japan*

A neutron scattering experiment in Materials and Life Science Experimental Facility (MLF) of Japan Proton Accelerator Research Complex (J-PARC) is done while changing measurement conditions. Variations of measurement conditions are automatically done by the control software of the experimental instrument in MLF. In 2013, we have upgraded our standard control software framework of the instruments. The new framework, named “IROHA2” is more user-friend, available, flexible and scalable than the original IROHA. Last year, we have developed the sequence management server based on IROHA2 for an automatic measurement. The sequence management server has the functions which are not only sequential control but also feedback control with exchanging the parameters between control commands.



In addition, the software components of IROHA2 have the web user interfaces. The sequence management server also has the web user interfaces. The figure shows one of the web user interfaces of the sequence management server. This user interface is to edit the sequence of the automatic measurement. We can make a control sequence which has loop structure with a drag-and-drop operation on this user interface.

In this presentation, we will show the details of our sequence management server based on IROHA2.

Abstract

Paper Ref: 267

PO31

DEVELOPMENT OF A CRYOSTAT FOR PHOTO-CRYSTALLOGRAPHY ON A TOF SINGLE CRYSTAL DIFFRACTOMETER SENJU

Takashi Ohhara[1], Takayasu Hanashima[2], Koji Munakata[2], Taketo Moyoshi[2], Ryoji Kiyanagi[1], Akiko Nakao[2], Tetsuya Kuroda[2]

[1] *J-PARC Center, Japan Atomic Energy Agency, Tokai, Ibaraki, Japan*
[2] *Research Center for Neutron Science and Technology, Comprehensive Research Organization for Science and Society, Tokai, Ibaraki, Japan*

Photo-induced crystalline-state properties such as light-emission and magnetism of organic molecular crystals have been actively investigated because they can derive new photo-functional organic materials. In recent investigations of those molecular crystals, *in-situ* light-exposure X-ray diffraction is one of the most important techniques to determine the photo-induced chemical species. On the other hand, neutron diffraction is a powerful technique to observe a hydrogen atom which is hardly observed by X-rays. If visible light is induced in a closed-cycle cryostat and *in-situ* light-exposure neutron diffraction measurement of those crystals under low temperature becomes possible, we will be able to trap the photo-induced metastable species produced by the proton transfer and determine its structure including the transferred proton, which is hard to determine by X-ray. Consequently researchers of photo-functional materials will obtain a new powerful analysis technique.

In this research, we developed a new closed-cycle cryostat with 2-axes goniometer in which visible white light can be induced to carry out an *in-situ* single crystal neutron structure analysis of a photo-induced species in a single crystal on SENJU, a TOF-Laue single crystal diffractometer at MLF/J-PARC.

The cryostat is composed from a 4K pulse-tube refrigerator, a fixed-□ type 2-axes goniometer using piezo-rotators and an optic fiber light guide. There are several service ports at the interface between the vacuum part and atmospheric part and a vacuum connector in which the light guide is embedded is set on one of the service ports. The forefront of the light guide is covered with super-insulation film and fixed on an optical port of the 4K radiation shield beside the goniometer. Visible light is led into the vacuum part of the cryostat through this light guide and exposed to the sample crystal. The Xe light source for the cryostat, MAX-303 (ASAHI SPECTRA Co.), has some wavelength selecting filters and the optimum wavelength region for each experiment can be chosen. The lowest temperature at the sample position is 8 K without photo-irradiation, 11 K with a 400 nm band-path filter, 18 K with a 400 nm long-cut filter and 66 K with no filter.

Abstract

Paper Ref: 71

PO32

TAIPAN THERMAL TRIPLE AXIS SPECTROMETER UPGRADES

Mr Scott Olsen[1], A/Prof Kirrily Rule[1], Mr Steven Pangelis[1], Dr Sergey Danilkin[1], Mr Tunay Ersez[1], Mr Scott Randall[1], Mr Andrew McGregor[1], Mr Adrian Ogrin[1], Dr Anton Stampfl[1]

[1]*Australian Nuclear Science and Technology Organisation*

The thermal triple axis spectrometer, TAIPAN, has been operational since 2010 and employs a graphite monochromator to access neutron energy transfers up to 80meV. User feedback indicated that an increase in energy transfer is required. An initial project was approved in 2013 to increase the accessible energy and momentum transfer range by increasing the floor space available to the sample, analyser and detector modules/drums.

The heavy-concrete shielding wall between the thermal triple axis TAIPAN and the cold neutron triple axis spectrometer SIKA was removed and a new, angled wall was installed. Building the wall from a combination of steel and lead allowed for a more compact design which improved instrument access while increasing the radiological shielding capabilities by ~4 times. The dance floor was extended by ~3m2, leading to a 30% improvement in accessible momentum transfer (at higher energy transfers).

A second project was approved in 2014 to purchase and install a copper monochromator and produce a translation mechanism for the sapphire filter. This was both for the triple axis instrument and for the recently commissioned Beryllium Filter option.

The copper monochromator, to be mounted back to back with the existing graphite monochromator, will allow an increase in the energy transfer up to 160 meV and improve the energy resolution, at the cost of lower neutron flux.

The sapphire filter cuts out higher energy neutrons (E>80meV) as a means to lower the background radiation when the graphite monochromator is used. A mechanism to translate this filter in and out of the beam will be important for maximizing the high energy flux of the neutrons from the new copper monochromator. This project is in the early stages of design and will be completed next year.

A key challenge is the radiation change due to the removal of the filter. The shielding wall is subject to seismic calculations and the entire reactor beam hall has floor loading limits as it is a suspended floor. ANSTO operates under the ALARA principle and doses outside of the enclosure walls are to be kept below 3uS/hr wherever possible.

Abstract

Paper Ref: 398

PO33

NEUTRON OPTICS UPGRADE FOR THE KOWARI RESIDUAL STRAIN SCANNER

Mr Scott Olsen [1], Dr Vladimir Luzin [1], Dr Anna Paradowska [1], Dr Mark Reid [1], Mr Steven Pangelis [1], Mr Adrian Ogrin [1], Mr Tai Nguyen [1], Dr Hiroshi Suzuki [2], Mr Mark New [1]

[1] *Australian Nuclear Science and Technology Organisation*
[2] *Japanese Atomic Energy Agency*

The thermal neutron residual strain scanner, Kowari, has been operational since 2007. In the original design of the neutron beam input slit system was statically fixed, difficult to align and optimized for a limited range of gauge volumes. Gauge volumes for complex shapes were difficult to achieve. Changing input and output optics was a slow and difficult manual handling process.

A project commenced in 2010 to design and manufacture a new neutron optics exchange system , to improve the slit system and to allow much faster change over between configurations of the neutron optics (currently, three option: slit system, radial collimator, open detector). The new system allows for rectangular gauge volumes of quite a large size range, has extended toolkit for accurate alignment and built-in anti-collision system.

In 2013 a project commenced to design and purchase a series of collimators for the diffracted beam. Tests using collimators on loan from the JRR-3M reactor in Japan had shown the promise of a collimation system. The set of radial collimators for KOWARI was accomplished in 2013 and involves the radial collimators with gauge sizes 2, 3 and 5 mm for stress measurements as well as 10 mm gauge for texture measurements. Commissioning tests were performed to confirm spec characteristics of the collimators, neutron optics properties (transmission, resolution) in comparison with traditional slit system. Results of the comparison will be presented.

Abstract

Paper Ref: 10

PO34

ABOUT THE VERSATILITY OF THE STATE-OF-THE-ART USANS INSTRUMENT KOOKABURRA

Christine Rehm[1], Liliana de Campo[1]

[1]*Bragg Institute, ANSTO, Locked Bag 2001, Kirrawee DC, NSW 2232, Australia*

Being opened for external users in 2014 the ultra-small-angle neutron scattering (USANS) instrument KOOKABURRA has since then proven to be a state-of-the-art instrument for the determination of large-scale structures. In our contribution we will discuss the instrument layout and highlight its performance as evidenced by successful USANS experiments on a variety of sample materials like, e.g., sulfide ore minerals, opals, and other complex systems.

Abstract

Paper Ref: 352

PO35

Development of a New Excusive Function with a Center-of-Gravity Calculation for the 2012 Model ^{6}Li Time Analyzer Neutron Detector System

Setsuo Satoh[1]

[1] *High Energy Accelerator Research Organization, Tsukuba, Japan*

A 2012 model ^{6}Li time analyzer neutron detector (LiTA12) system has been developed as a high-count-rate neutron detector. An exclusive function with a center-of-gravity calculation which not only prevents over-counting due to cross-talk, but also obtains fine position resolution, has been included in this version.

As the high-count-rate neutron detector, this device can detect neutrons with a 3 mm pixel size in a detection area of 5 × 5 cm^2, and arranged as a 16 × 16 matrix with a detection efficiency of approximately 40% of that of a ^{3}He detector. A maximum count rate of 50 million counts per second (Mcps) was obtained.

The exclusive function [1] which prevents cross-talk between pixels allows the detector to use a no-cut scintillator. The function expands the scintillator selection flexibility. Furthermore, the function with a center-of-gravity calculation allows the detector to measure neutrons with 0.4 mm pixel size. Fig. 1 shows the data of the function. Fig. 1(a) is a “KENS” character of Cd on the front of the detector, (b) is a two-dimensional (2D) graph with 3 mm real pixel size, and (c) is a 2D graph with 0.4 mm pixel size, which are compensated by the data without the Cd character. The shadows of the holding tapes including hydrogen are seen at the top and bottom of the Cd character.

This new function is useful for high position-resolution experiments involving a high-intensity neutron source, such as those performed at J-PARC. In this presentation, I will report the LiTA12 system in detail.

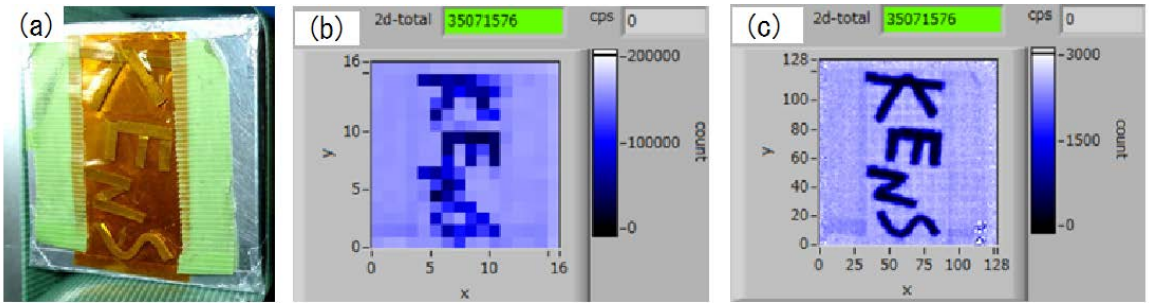


Figure 1. (a) Cd “KENS” character, (b) 2D graph with 3 mm pixel size, (c) 2D graph with 0.4mm pixel size, which are compensated by the data without the Cd character.

References

S.Satoh, Development of New Exclusive Function for a 2012 model for the ^{6}Li time analyzer detector system, to be published in JPSJ, 2nd International Meeting of J-PARC symposium (J-PARC Symposium 2014), July 2014, Tsukuba Epocal.

Abstract

Paper Ref: 372

PO36

ANSTO Research Portal – ANSTO's Next Generation User Portal

Jamie Schulz[1], Bob Beazley[2], Adam Barry[2], David Carroll[1] and Joseph Bevitt[1]

[1] *Bragg Institute, Australian Nuclear Science & Technology Organisation, Sydney, Australia*
[2] *Information Technology Services, Australian Nuclear Science & Technology Organisation, Sydney, Australia*

ANSTO has a mature software platform to which allows us to manage all aspects of the neutron beam operations at the OPAL reactor. The suite of applications consist of a web based portal and online training system, a scheduling application, a bar-coded sample management application and a facility status monitor. At the heart of the platform is a single database which allows data to be entered once and propagated throughout the software suite.

ANSTO is presently developing its next generation user portal. The vision for the new user portal is to extend the proposal management system to other facilities such as the Centre for Accelerator Science and the Australian Synchrotron and provide a single entry point for external users of all ANSTO facilities.

An overview of the existing user portal and the features of the new portal will be given.

Abstract

Paper Ref: 382

PO37

Managing Operational Risk & Safety in a Neutron Scattering Facility

Jamie Schulz[1] and Cassie Haley[1]

[1] *Bragg Institute, Australian Nuclear Science & Technology Organisation, Sydney, Australia*

Managing operational risk and safety in a neutron scattering facility can be a challenging task. The balance between regulatory compliance and the demands of a user program requires good systems and practices to be employed.

Nurturing a strong safety culture is key to minimising risk and ensuring a safe work environment for staff and users, along with a rigorous process for proposal assessment, approval, and completion.

An overview of the practices and systems employed to manage operational risks and safety of neutron beam instruments and experiments at the OPAL Neutron Scattering Facility will be presented along with trends in the data collected to date.

Abstract

Paper Ref: 274

PO38

Developing Neutron Scattering Instrument Software : DC-ToF Case

Ji-Yong So[1]

[1] *Korea Atomic Energy Research Institute, Daejeon, Korea*

One of key component of neutron scattering instrumentation is the software which coordinates and controls the physical components such as detectors, monochrometer, sample environments and finally produce the raw data. In addition, experimental data are treated by another kind of software - data reduction and analysis software.

I’ve developed a Disk Chopper Time-of-Flight Spectrometer (DC-ToF) in HANARO research reactor and also developed instrument control software and data reduction software. In this presentation, the detail of DC-ToF instrument control software and other software for the instrument management will be introduced with the short description of DC-ToF instrumentation.

Abstract

Paper Ref: 231

PO39

A BE-FILTER-BASED NEUTRON SPECTROMETER FOR VIBRATIONAL SPECTROSCOPY

Anton P.J. Stampfl[1], Andrew Eltobaji[1], Anthony Kafes[1]

[1]Australian Nuclear Science and Technology Organisation, Lucas Heights, Australia

A low energy neutron band-pass filter spectrometer is currently in the last phase of commissioning at the Bragg Institute and will be ready for use on TAIPAN in the second half of the year. TAIPAN will therefore have two measurement modes available for use, the existing triple-axis spectrometer and the Be-filter spectrometer. The filter spectrometer is based on a NIST design that employs a number of filter materials to select scattered neutrons less than or equal to 1.8 meV. The heart of the spectrometer consists of curved banks of graphite and beryllium blocks arranged in an arc of 80° and a bank of 30 ^{3}He detectors placed directly behind the filter blocks that collect the energy filtered neutrons. A vibrational spectrum is built up by collecting neutrons at discrete incident neutron energies, e_i, on the sample. As the final scattered energies collected are fixed at $e_f \leq 1.8 meV$, a spectrum, in terms of energy transfer, $e_n=e_i-e_f$, is obtained representing the vibrational density of states of the material under investigation. A 0.9 sr solid angle of scattered neutrons is collected by the spectrometer and integrated into one single vibrational spectrum.

Measurement is typically performed on powder samples with potentially rapid acquisition times as short as 20 minutes for one full spectrum from 20-80 meV. Significant impact will be made in the areas of:

[3] Molecular spectroscopy (energy systems involving hydrogen storage, hydrogen bonding, carbon-black like materials, such as C_{60}),
[4] Catalysis (fuel cell catalysts, methane-based catalysts, low temperature CO oxidation)
[5] Zeolite science
[6] Mineral science (hydration)

Results of commissioning experiments as well as a detailed description of the spectrometer will be presented.

Abstract

Paper Ref: 397

PO40

The Small and Wide Angle Neutron Scattering Instrument TAIKAN of J-PARC

Jun-ichi Suzuki[1], Shin-ichi Takata[2], Kazuki Ohishi[1], Hiroki Iwase[1], Taiki Tominaga[1], Takenao Shinohara[2], Takayuki Oku[2], Takayoshi Ito[1], Takeshi Nakatani[2], Yasuhiro Inamura[2], Toshiaki Morikawa[1], Masae Sahara[1], Toshiaki Hosoya[2], Kentaro Suzuya[2], Kazuya Aizawa[2], Masatoshi Arai[2], Toshiya Otomo[3], and Masaaki Sugiyama[4]

[1]*Comprehensive Research Organization for Science and Society (CROSS), Tokai, Ibaraki, Japan*
[2]*J-PARC Center, Tokai, Ibaraki, Japan*
[3]*High Energy Accelerator Research Organization (KEK), Tsukuba, Ibaraki, Japan*
[4]*Kyoto University Research Reactor Institute, Kumatori, Osaka, Japan*

The small and wide angle neutron scattering instrument TAIKAN has been developed and utilized at the Materials and Life Science Experimental Facility (MLF) of the Japan Proton Accelerator Research Complex (J-PARC) for studying nanostructure and hierarchical structure of materials and its related dynamics with high structural and time resolution. For the study TAIKAN has a detector system, which is composed of small-, medium-, and high-angle detector banks and a backward detector bank with arrays of one dimensional ^{3}He position detectors. 1,216 pieces of detector were installed in total at the banks so far. A high-resolution area detector and a magnetic lens for focusing neutrons on the area detector have been under commissioning. The q range of TAIKAN will be $5\times10^{-4} \sim 20$ A^{-1} with both such detectors and neutrons in the wide wavelength range of 0.5 ~ 8 A. A neutron spin polarizing cavity with Fe/Si magnetic supermirrors was installed and a neutron spin analyzing filter ban be tentatively used for separation of coherent scattering and incoherent scattering from hydrogenous materials. For user program, various kind of equipment can be used. Those are a sample changer, 4K cryostat, 0.2Tesla magnet, 1Tesla magnet, 4 Tesla magnet, 10Tesla magnet, laser furnace, tensile load cell, etc. In this paper, current status of TAIKAN is introduced with scientific results obtained with TAIKAN.

Abstract

Paper Ref: 174

PO41

The Design of CSNS Online Software System

Haolai Tian[1], Ming Tang[1], Lili Yan[1], Ming Li[1], Junrong.Zhang[1]

[1]China Spallation Neutron Source, Institute of High Energy Physics, Chinese Academy of Sciences, Dongguan, China

Block-based online data processing software system is being developed at the China Spallation Neutron Source (CSNS) for the manipulation and analysis of time-of-flight event data from the neutron scattering instruments.

This software is implemented with MVC pattern in C++, and CMake as a cross-platform build system. It can run on typical Linux/Windows/Mac cross-platforms. It provides Python APIs to other related systems, such as data acquisition, slow control and central data management system, etc.

The online data processing software uses block-based design, which consists of several featured modules: middle-level framework, event-data parallel processing algorithms, data I/O component, web interface and real-time visualization system.

This contribution introduces the design and implementation of CSNS online software system.

Abstract

Paper Ref: 400

PO42

Status Report of Super High Resolution Powder Diffractometer in J-PARC

Shuki Torii[1,2], Ping Miao[1], Sanghyun Lee[1], Yoshihisa Ishikawa[1], Masao Yonemura[1,2] and Takashi Kamiyama[1,2,3]

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[3] *Sokendai, The Graduate University for Advanced Studies, Tsukuba, Ibaraki 305-0801, Japan*

The super high resolution powder diffractometer, SuperHRPD suffered damage by the Great East Japan Earthquake on the 11th of March 2011. We could return to the state before the earthquake of instrument over the past three years. In this restoration work, the vacuum jacket of the guide tube and the guide tube support system were improved to lighter and simply. The iron vacuum jackets of the curved guide section in the upstream were replaced with aluminum jackets. At the straight guide section without vacuum jacket in the downstream, about 50 m of the guide tube support system was also changed to aluminum, which made it lighter. The beam line shield of the straight guide section was completely redesigned to concrete containing boron. The new design beam line shield has a ten-odd cm clearance around the guide tube, and then re-alignment of the beam line became easy.

As part of the upgrade of the instrument, a new detector system of 8 mm diameter position sensitive detectors (PSDs) were fully installed at the backward detector bank. The resolution experiments using NIST standard silicon sample (SRM640d) carried out, we were able to obtain a very sharp diffraction peak with $\Delta d/d$ = 0.08%. By limiting the detector area, it was possible to achieve less than 0.05%.

Abstract

Paper Ref: 287

PO43

SCIENTIFIC HIGHLIGHTS FROM QUOKKA, THE 40M PINHOLE SANS INSTRUMENT, AT OPAL

Dr Kathleen Wood[1], Dr Jitendra P. Mata[1], Dr Christopher J. Garvey[1], Dr Elliot P. Gilbert[1]

[1]*Bragg Institute - ANSTO*

Quokka is the 40 m pinhole SANS instrument at the OPAL reactor serving the growing needs of both domestic and international users [1]. In 2014, over 200 days of user experiments were run. Outputs from Quokka have been published that cover such diverse fields as magnetism, structural biology, mineralogy, polymers, food science and soft matter. We present here a selection of recent scientific highlights.

[1] www.ansto.gov.au/ResearchHub/Bragg/Facilities/Instruments/Quokka/

Abstract

Paper Ref: 133

PO44

Taiwan's High Resolution Cold Triple-Axis Spectrometer at ANSTO

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[2] *Bragg Institute, ANSTO, Lucas Heights, NSW, Australia*

SIKA , the cold neutron triple-axis spectrometer at ANSTO, is funded by the Ministry of Sciense and Technology of Taiwan. We will report on the current status of SIKA which is currently being commissioned by the National Synchrotron Radiation Research Center and opening to users in the next proposal round N-2015-2.

SIKA has a view of the cold source from the reactor beam hall in OPAL, providing it with a good neutron flux in the cold range. As a state-of-the-art triple-axis spectrometer, SIKA equips with mutiblade analyser system which allows for the simultaneous data collection over a specified range in (Q, E) by operating in a flat or in a multiplexing mode when coupled to the one-dimensional PSD consisting of 48 wires. This analyser system can also operate in a horizontal focusing mode that directs the scattered neutrons into a single-detector. The entire analyser-detector system is packed into a single, well shielded secondary spectrometer housing to provide a low background. SIKA is also equipped with a fully automated sample stage and a series of collimations (both sölar and radial).

The combination of high neutron flux, good energy and momentum resolution, low background and a large dynamic range will make SIKA ideally suited to study of spin and lattice dynamics in single crystal samples. The low-q capability of SIKA especilly suitable for biological study on the topic of lipid bilayer structure. Relaxation phenomena, critical scattering, soft-modes, magnetism, ionic conductors, catalysts, H storage and functional energy-related materials will also play an important role in the science output of SIKA.

Abstract

Paper Ref: 187

PO45

Python Scripting for Instrument Control and Online Data Treatment

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Scripting is an important feature of instrument control software. It allows scientists to execute a sequence of tasks to run complex experiments, and it makes a software developers' life easier when testing and deploying new features. Modern instrument control applications require easy to develop and reliable scripting support.

At ANSTO we provide a Python scripting interface for Gumtree. Gumtree is an application that provides three features; instrument control, data treatment and visualisation for neutron scattering instruments. The scripting layer has been used to coordinate these three features. The language is simple and well documented, so scientists require minimal programming experience.. The scripting engine has a web interface so that users can use a web browser to run scripts remotely.

The script interface has a numpy-like library that makes data treatment easier. It also has a GUI library that automatically generates control panels for scripts. The same script can be loaded in both the workbench (desktop) application and the web service application for online data treatment. In both cases a GUI will be generated with similar look and feel.

* Gumtree T. Lam, N. Hauser, A. Gotz, P. Hathaway, F. Franceschini, H. Rayner, GumTree. An integrated scientific experiment environment, Physica B 385-386, 1330-1332 (2006)

** Gumtree http://docs.codehaus.org/display/GUMTREE/Home

*** numpy http://docs.scipy.org/doc/numpy/reference/

Abstract

Paper Ref: 311

PO46

DEVELOPMENT OF ELLIPTICAL FOCUSING MIRROR AND HIGH EFFICIENCY DETECTOR WITH HIGH SPATIAL RESOLUTION FOR NEUTRON REFLECTOMETER SOFIA IN J-PARC

Dr Norifumi Yamada [1], Takuya Hosobata [2], Masahiro Hino [3], Takeshi Fujiwara [4], Shin Takeda [5], Jiang Guo [2], Setsuo Sato [1], Tomohiro Seya [1], Shin-ya Morita [2], Tatsuro Oda[3]

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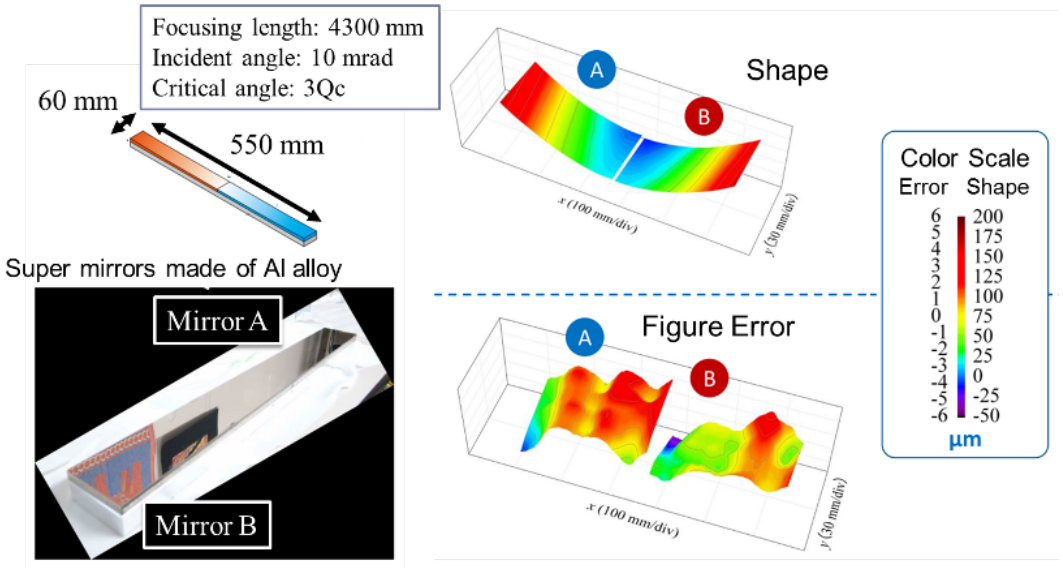
[2]*RIKEN Center for Advanced Photonics*
[3]*Kyoto University*
[4]*The University of Tokyo*
[5]*Hokkaido University*

SOFIA is a horizontal-type neutron reflectometer constructed at Beamline 16 (BL16) of the Materials and Life Science Experimental Facility (MLF) of the Japan Proton Accelerator Research Complex (J-PARC). Owing to the high-flux beam of J-PARC, less than one hour is needed for taking a full *Q*-range data and only a few seconds for a limited *Q*-range data in the case of a sample with 3 inches (76 mm) in diameter; even though the beam power is still one-third of the planned value, 1 MW. However, several hours are still needed for a small samples such as 10 mm × 10 mm, which is a typical size of a sample for X-ray reflectometry.

For further upgrade of the SOFIA reflectometer, we plan to install an elliptical focusing mirror and a high efficiency detector near future. The focusing mirror enable us to illuminate a sample with a neutron beam with a large beam divergence. In the case of a conventional double slit collimation, the optimal beam divergence decrease with the sample size. Hence, the focusing optics with the mirror has an advantage on the beam flux especially for small samples. On the other hand, diverged beam causes less angular resolution. To avoid this problem, position sensitive detector is needed because an incident angle can be evaluated from the detection position under a specular condition. The angular resolution in this case is, however, limited by a spatial resolution of the detector. Therefore, a requirement of a detector is not only high counting efficiency but also a high spatial resolution for reflectometry with focusing optics. The goals of the specification of the focusing mirror and detector are a beam size of 0.1 mm at a sample position with the divergence of 2.5 mrad. and counting efficiency of 50% for thermal neutrons with the spatial resolution of 0.1 mm, respectively.

In this presentation, we will report a development of the focusing mirror and one dimensional position sensitive detector in detail.


Focusing length: 4300 mm
Incident angle: 10 mrad
Critical angle: 3Qc
60 mm
550 mm
Super mirrors made of Al alloy
Mirror A
Mirror B
Shape
A
B
x (100 mm/div)
y (50 mm/div)
Figure Error
A
B
x (100 mm/div)
y (50 mm/div)
Color Scale
Error Shape
6 200
5 175
4 150
3 125
2 100
1 75
0 50
-1 25
-2 0
-3 -25
-4 -50
-5
-6
μm

Abstract

Paper Ref: 168

PO47

A New Online Data Processing Software Framework: SNiPER

Lili Yan[1], Jiaheng Zou[1], Haolai Tian[1], Ming Tang[1], Ming Li[1], Junrong Zhang[1]

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In the presentation, a new implementation of the framework SNiPER will be introduced, and the design of kernel will be given in detail.

SNiPER is an internal development framework providing standard interfaces for common software components and offering Python APIs. The goal of SNiPER is to build a framework which can be applied to the CSNS online data processing applications. These applications transform the data collected from the detector and control system into experimental data based on NeXus. It adopts a data centered architectural style and separates between "data" and "algorithm", which allows algorithms do not directly access the data objects in the data file. SNiPER provides a hierarchical transient data store, which is designed to operate not only on event-based data for event data collecting and filtering, but also on matrix-based data for histogram manipulation.

Abstract

Paper Ref: 276

PO48

CURRENT STATUS OF TOF NEUTRON DIFFRACTION DATA PROCESSING SOFTWARE FOR iBIX

Dr. Naomine Yano [1], Dr. Takashi Ohhara [2], Dr. Takaaki Hosoya [3], Dr. Taro Yamada[1], Dr. Ichiro Tanaka [3], Dr. Katsuhiro Kusaka [1]

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[2]J-PARC Center, JAEA, 3 Faculty of Engineering, Ibaraki University

STARGazer has been developed as the software to process Time-of-flight (TOF) neutron diffraction data measured at IBARAKI Biological Crystal Diffractometer (iBIX) in J-PARC. This software is mainly composed of two parts, data processing part and data visualization part. The former is to make HKLF list from raw data and the latter is to display the raw data with predicted positions of reflections. We are developing user friendly software and have been able to distribute STARGazer and its manual to iBIX users for 2 years.

In order to improve the quality of integrated intensity of weak reflections, we are aiming to implement software component of profile fitting to STARGazer. Profile fitting method has already been adopted by X-ray single crystal diffraction data processing software such as Mosflm and Denzo. Because the diffractometer was installed on a coupled moderator which has more intense peak and integrated intensity but more asymmetric and wider pulse shape than a decoupled moderator, it is difficult to realize profile fitting method actually. We tried to seek existing functions representing asymmetry intensity distribution, such as the 2 back-to-back exponentials convolved with a Gaussian, Ikeda-Carpenter function convolved with a Gaussian from GSAS program and verified usefulness of these functions by using RNase A single crystal diffraction data. We explain concrete results in the poster.

In the future, accelerator power of J-PARC will be improved at 1000kW and the diffraction data of larger unit cell crystals (~135 $Å^3$) can be measured. The wide pulse and large unit cell cause overlap of the adjacent reflections. The method to deconvolute overlapping reflections is essential. If the development of profile fitting method is realized, this method can be applied to solve this problem.

Abstract

Paper Ref: 228

PO49

CROSS-Tokai Promoting Science at the MLF

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CROSS-Tokai is an independent agency appointed by the Japanese government in April 2011 to administer, support and promote the user program on the public-access neutron beamlines at J-PARC. Core functions of CROSS-Tokai are to administer proposal selection and beamtime allocation on the public beamlines, to select and review contract beamlines, to support user experiments on the public beamlines and facilitate positive research outcomes, to provide expert advice and develop information resources for users and potential users of the facility and to undertake outreach and facility utilization promotion activities.

This paper will present the CROSS-Tokai activities; the organization, the proposal selection system, the strategic research programs, the beamtime distribution on the public beamlines, results of the trial use, examples of user support such as sample preparation, data acquisition, data analysis and discussion.

Abstract

Paper Ref: 306

PO50

Development of *in operand* Techniques for Battery Study using Special Environment Neutron Powder Diffractomter, SPICA

Masao Yonemura[1], Takashi Kamiyama[1,2], Kazuhiro Mori[3], Toshiharu Fukunaga[3], Yoshihisa Ishikawa[1], Shuki Torii[1], Ryoko Tomiyasu[1], Koichi Kino[4], Dyah Sulistyanintyas[5], Yohei Onodera[3], Takahiro Naka[5], Makoto Morishima[5], Masaya Shiotani[5], Toshiyuki Matsunaga[5], Hideyuki Komatsu[5], Kouji Kitada[5], Keiji Shimoda[5], Satoshi Taminato[6], Masaaki Hirayama[6], Ryoji Kanno[6], Naoya Ishida[7], Naoto Kitamura[7], Yasushi Idemoto[7], Yoshio Ukyo[5], Hajime Arai[5], Yoshiharu Uchimoto[8], Zempachi Ogumi[5]

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[2] *The Graduate University for Advanced Studies (Sokendai), Tokai, Japan*
[3] *Research Reactor Institute, Kyoto University, Kumatori, Japan*
[4] *Faculty of Engineering, Hokkaido University, Sapporo, Japan*
[5] *Office of Society-Academia Collaboration for Innovation, Kyoto University, Uji, Japan*
[6] *Interdisciplinary Graduate School of Science and Engineering, Tokyo Institute of Technology, Yokohama, Japan*
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SPICA, a new special environment powder neutron diffractometer was built at BL09 in the Material and Life science Facility (MLF) of the Japan Proton Accelerator Research Complex (J-PARC). This is the first instrument dedicated to the study of next-generation batteries in J-PARC and is optimized for in situ measurements to determine the structural changes of battery materials at the atomic level. To make in situ measurements of real batteries more fruitful, we designed the high $\Delta d/d$ resolution with wider d ranges to detect many phases during chemical reaction, high neutron intensity to know the specific reaction process in high speed charge/discharge, low background and a dedicated chemistry area to carry out long-term scheduled experiments with many sets of on-beam measurements and off-beam charge-discharge measurements. In the commissioning stage, the *in operand* diffraction and *in situ* transmission imaging experiments were carried out. The structural changes of the electrode materials, which are dependent on the lithium content in a commercialized Li-ion battery, were clearly observed. Position dependences in the battery cell were also determined. In this presentation, the current status of SPICA will be reported.

Acknowledgement
This work was supported by the Research and Development Initiative for Science Innovation of New Generation Batteries (RISING) project of the New Energy and Industrial Technology Development Organization (NEDO). This work was carried out as the S-type project of KEK (Proposal No. 2009S10, 2014S10).

Abstract

Paper Ref: 266

PO51

Update on PELICAN – a multi-purpose time of flight cold neutron spectrometer

Dehong Yu[1], Richard A Mole[1]

[1] *Bragg Institute, Australian Nuclear Science and Technology Organisation*

Pelican, a direct geometry multi-purpose cold neutron spectrometer, combines the state of the art triple monochromators [1] and Fermi chopper systems to perform inelastic and quasi-elastic neutron scatterings on a variety of materials of powder, polycrystal, single crystal, glass and liquid, covering fields of physics, chemistry and biology. The provision of a cryogenic vacuum from sample to detector maintains the scattered intensity, minimises the background from air scattering and surrounding materials such as from sample environment. The instrument is designed to accommodate various sample environments like high magnetic field, low and high temperatures, etc. [2, 3].

Polarization analysis is an option of the Pelican instrument. The compact incident neutron polarization system is an integration of a solid-state bender-type supermirror polarizer with a gradient radio frequency (RF) spin flipper. The supermirror polarizer consists of three sections with 1 degree inclination between the outer sections relative to the central section in order to match with the three individual beams from the triple monochromators. The polarization analysis is achieved by ^{3}He polarization filter system containing a wide-angle ^{3}He polarization filter and a *Pastis*-type magnetic coil. The whole polarization analysis system is installed inside the high vacuum sample chamber through a dedicated vacuum adaptor flange. *In-situ* refilling of pre-generated polarized ^{3}He has been implemented.

Update on the progress and performance test will be demonstrated by several experiments of quasi-elastic and inelastic neutron scatterings, including the first test on the polarization analysis system. Problems encountered and lessons learnt so far will also be presented, including the identification and reduction of fast neutron generation from Be-filter. The extension of dynamic range with optimising phase delays between two Fermi choppers will be demonstrated.

[1] A. K. Freund and D. H. Yu, Nucl. Instr. and Meth. A 634, S75-S80 (2011).

[2] D. H. Yu, R. A. Mole, T. Noakes, S. Kennedy and R. Robinson, J. Phys. Soc. Jpn. 82, SA027 (2013).

[3] Dehong Yu, Richard Mole and G.J. Kearley, Performance Test on Pelican – a mulit-purpose Time of Flight Cold Neutron Spectrometer, EPJ Web of Conferences 83, 03019 (2015).

Abstract

Paper Ref: 178

PO52

The Challenges of Data Analysis and Management Software at CSNS

Junrong Zhang[1], Haolai.Tian[1], Ming.Tang[1], Lili Yan[1], Ming Li[1], Fangwei Wang[2]

[1]*China Spallation Neutron Source, Institute of High Energy Physics, Chinese Academy of Sciences, Dongguan, China*
[2]*Institute of Physics, Chinese Academy of Sciences, Beijing, China*

The overall design of data analysis & management system at China Spallation Neutron Source (CSNS) is not so much different with those in other big-science facilities. Now the software is under development, and will be deployed in the end of 2017.

Although the software implementations are very similar for the neutron sources so far, recent innovations in software and computer might provide new ways to play neutron. So users will benefit from the new technologies, as well as facility will.

The data management system of CSNS, is based on open source metadata management project (ICAT). The data portal, user program and analysis interface will be integrated too. Here the clustered file system (CEPH) and cloud database are used. Once the interfaces among instruments and facilities are built, big data analytics could be utilized to extract correct and full information from all the complex data.

The online and offline data processing are implemented in the big-data framework (SNiPER), which will work on both local beam sites and central portal. And we also will collaborate with Mantid project in data reduction. The virtual cloud computing based on OpenStack and web app based on AJAX framework, will share the site sources: data, computer and software, etc. So user can have quick experiment preview, data analysis and scientific simulation, anytime, anywhere.

To make scientists happy to use the system, the neutron software developers have to learn more and think deeply on new technologies.

Soft Matter Systems

Abstract

Paper Ref: 159

PO53

MORPHOLOGICAL STUDY ON FLUORESCENCE PROPERTIES OF BLOCK COPOLYMER MICELLES FOR FAST, SENSITIVE, AND LABEL-FREE CHEMICAL SENSORS

Mr Hyungmin Ahn [1], Dr Moon Jeong Park [1,2]

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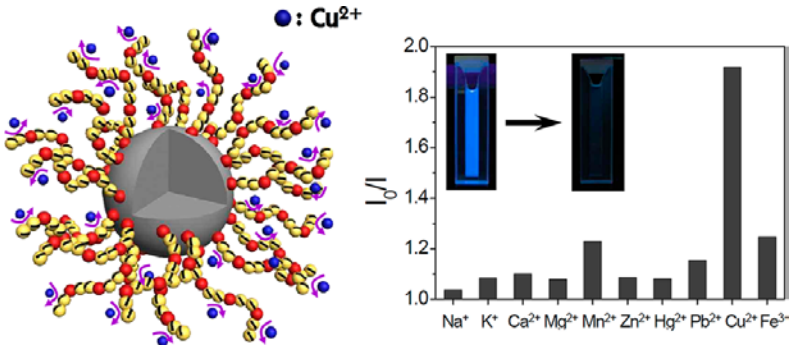
[2] *Division of Advanced Materials Science, POSTECH*

Relationship between the morphologies and fluorescence properties of non-conjugated block copolymer micelles possessing partially sulfonated polystyrene (PSS) chains has been studied. Significantly enhanced fluorescence intensities were observed for PSS-containing block copolymer micelles upon stabilizing the light-emitting PSS chains within nanosized micellar core phases, which is in sharp contrast to the weak fluorescent PSS homopolymer solutions. High fluorescent quantum yield (QY) up to 37 % was achieved by controlling the type of solvent, contrary to the low QY of 5% for PSS homopolymer solutions lacking organization regardless of the type of solvent. We demonstrated that fluorescence intensities of PSS-containing block copolymer solutions are largely depending on the micellar morphologies in terms of core/shell sizes and the location of PSS chains in the micelles, as studied by solution scattering methods. Based on this knowledge, both the fluorescence emission wavelengths and intensities of PSS-containing block copolymer micelles were fine-tuned to yield the various emission colors ranging from blue to green. Notably, the fluorescence properties of PSS-containing block copolymer micelles were highly sensitive and selective to Cu^{2+} ions with a rapid quenching response time within 1 min, indicating the potential applications of our systems towards heavy metal-ion sensors. The spatial matching derived from morphological factors and fast collision kinetics for Cu^{2+} through high binding affinity with $–SO^{3-}$ were found out to be an origin for the observation.



Scheme 1. Micellar morphology of blue-emitting PSS-containing block copolymer micelle and its selective sensing of Cu(II) ion against other metal ions.

Abstract

Paper Ref: 79

PO55

Dynamic and structural heterogeneity in red blood cells – neutron scattering investigations of cell physiology

Christopher J. Garvey[1]

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The highly penetrative and non-ionizing nature of neutrons can provide an ideal probe of structure and dynamics in cellar systems in a near physiological context. The low information content of neutron scattering data can provide an important barrier to extracting meaningful information from experiments. While many cellular systems can be quite complex the red blood cell provides a simple system in which useful quantitative information can be extracted. Investigations with SANS[1] and QENS[2] from cell suspensions and concentrated haemoglobin solutions have shown the power of the technique. Here we discuss the analysis of scattering from red blood cell and define a simple scattering problem where it is the intra-cellular solution of haemoglobin which provides the only resolvable component to the neutron scattering. In particular we discuss ultra-high resolution small angle neutron scattering (USANS) collected from suspensions of red blood cells (RBCs) at rest (not flowing) in D20 saline solutions on the S18 instrument (Institute Laue Langevin, Grenoble, France). We discuss the approximation that the contrast in USANS from suspensions of RBCs is due to the difference in haemoglobin concentration inside and outside the cells. This conclusion allows us to use USANS as a simple probe of cell volume in collections of metabolically active cells.

[1] C. Garvey, R. Knott, E. Drabarek, P. Kuchel, *European Biophysics Journal With Biophysics Letters* **2004**, *33*, 589-595; A. M. Stadler, R. Schweins, G. Zaccai, P. Lindner, *Journal of Physical Chemistry Letters* **2010**, *1*, 1805-1808.

[2] A. M. Stadler, C. J. Garvey, J. P. Embs, M. M. Koza, T. Unruh, G. Artmann, G. Zaccai, *Biochimica et biophysica acta* **2014**, *1840*, 2989-2999; A. M. Stadler, C. J. Garvey, A. Bocahut, S. Sacquin-Mora, I. Digel, G. J. Schneider, F. Natali, G. M. Artmann, G. Zaccai, *Journal of the Royal Society Interface* **2012**, *9*, 2845-2855.

Abstract

Paper Ref: 103

PO56

ORGANOGEL FORMATION VIA SUPRAMOLECULAR ASSEMBLY OF OLEIC ACID AND SODIUM OLEATE

Dr. Constantinos Nikiforidis [2,3], Dr. Elke Scholten [2,3], **Dr. Elliot Gilbert** [1]

[1] *Bragg Institute*

[2] *Top Institute Food & Nutrition*

[3] *Wageningen University*

To create materials with novel functionalities, the formation of gels within hydrophobic media has become popular. This is often accomplished through the assembly of low molecular weight organogelators into a variety of complex phases through intermolecular interactions. In the case of edible materials, the assembly of saturated fatty acids to form fat crystal networks is often used for structuring. Here, the first example of structuring with unsaturated fatty acids is reported, namely mixtures of oleic acid and sodium oleate, to structure edible lipid phases. Small-angle neutron scattering demonstrates that the resultant structures, which vary with oleic acid and sodium oleate molar ratio, comprise either inverse micellar or lamellar phases, combined with the formation of crystalline space-filling networks. Network formation was found for filler concentrations above 8.0 wt%. Rheological measurements show that gel strength depends on the ratio of oleic acid to sodium oleate, and is greater when only oleic acid is used. The addition of up to 1.5 wt% of water enhanced the strength of the organogels, probably through supplementary hydrogen bonding but, for concentrations greater than 2.0 wt%, the assembly was inhibited leading to collapse of the gel. Polarized microscopy along with rheology, differential scanning calorimetry, and scattering techniques were used to investigate the assembly structures to provide insight into the gelation mechanism.

Abstract

Paper Ref: 272

PO57

Gelation Process of Tetra-Armed Polymer in Ionic Liquid Studied by Time-Resolved Small Angle Neutron Scattering

Kei Hashimoto[1], Kenta Fujii[2], Kengo Nishi[1], Takamasa Sakai[3] and Mitsuhiro Shibayama[1]

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[2]*Graduate School of Science and Engineering, Yamaguchi University, Yamaguchi, Japan.*
[3]*Department of Bioengineering, School of Engineering, The University of Tokyo, Tokyo, Japan.*

Ion gels composed of ionic liquid (IL) and polymer have been attracted attention as gas separation membranes because of CO_2 absorption selectivity, thermal and chemical stabilities. For application, mechanically tough ion gels with low polymer content are required. We have recently developed tetra-arm poly(ethylene glycol) (TetraPEG) ion gel which shows excellent mechanical properties even with low polymer concentration (3-6 wt%).[1] We investigated the gelation mechanism to point out that its mechanical toughness deeply depends on the gelation process[2]. In this work, we investigated the gelation process using time-resolved small angle neutron scattering (SANS).

Fig. 1 shows time-resolved SANS profiles during the gelation for Tetra-PEG ion gel (M_w = 10000, c = 20 g/L) in 1-ethyl-3-methylimidazolium bis(trifluoromethanesulfonyl)amide ([C_2mIm$^+$][TFSA$^-$]).The scattering intensity rapidly increased as soon as the reaction started and gradually increased as the reaction proceeded, which can be explained by second order reaction based on the reaction kinetics. To evaluate the network structure quantitatively, curve fitting analyses were performed for the observed SANS functions. As shown in Fig. 1, Ornstein-Zernike functions (corresponding to the solid lines) well reproduced the experimental data. The correlation length (ξ) and the scattering intensity at q = 0 (I_0) were estimated by fitting. It was found that both of them increased with reaction time in the $\phi < \phi^*$ region, where ϕ^* is the chain overlap polymer fraction. In contrast, neither of them scarcely changed in the $\phi > \phi^*$ region, indicating the evolution of the network structure deeply depends on the polymer volume fraction in ionic liquid system.

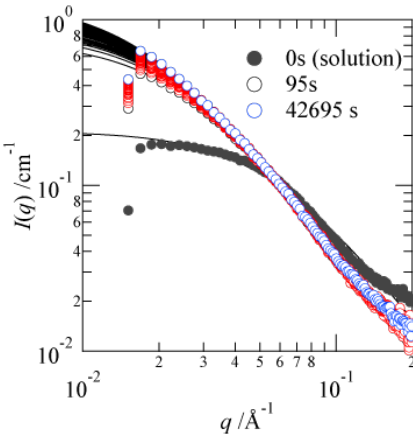

Fig.1 Time-resolved SANS results during gelation process of Tetra-PEG ion gel for every 600 sec. The solid lines indicate the results of curve fit with Ornstein-

REFERENCE: [1] K. Fujii et al, *Soft Matter*, 2012, [2] K. Hashimoto et al, *Journal of Physical Chemistry B*, 2015.

ACKNOWLEDGEMENT: The SANS experiment was performed by using 40 m SANS at HANARO, KAERI, Daejeon, South Korea.

Abstract

Paper Ref: 408

PO58

Hydration State Analyses of Poly(sulfobetaine) Brushes by Neutron Reflectivity

Yuji Higaki,[1,2] Ai Takenaka,[1] Norifumi L Yamada,[3] Atsushi Takahara[1,2]

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[2] *Institute for Materials Chemistry and Engineering, Fukuoka, Japan*

[3] *Institute of Material Structure Science, High Energy Accelerator Research Organization (KEK), Ibaraki, Japan*

Hydration state of high-density poly(sulfobetaine) brushes was investigated by neutron reflectivity. Polyzwitterions have been investigated for the specific physical character in contrast with polycations and polyanions. The zwitterionic side groups lead the aggregation through the interzwitterion pairing, whereas the added salt and thermal fluctuation break the interactions to make chains swelled. The salt and heat induced hydration state surely correlate with the material performance in the wetting state including lubrication, adhesion, and anti-fouling properties. Recent progress in polymer brush preparation via surface-initiated controlled radical polymerization affords high-density polyelectrolyte brushes with precise molecular weight control. Poly(sulfobetaine)s are zwitterionic polyelectrolyte with quaternary ammonium cation and sulfonate anion, and is well known to show upper critical solution temperature (UCST). The UCST and the salt concentration dependence depend on the alkyl chain spacer length. Three kinds of polymer brushes were prepared by surface-initiated atom transfer radical polymerization of sulfobetaines, which has ethyl [(3-(*N*-2-methacryloyloxyethyl-*N,N*-dimethyl) ammonatoethanesulfonate), MAES], propyl [(3-(*N*-2-methacryloyloxyethyl-*N,N*-dimethyl) ammonatopropanesulfonate), MAPS], or butyl [(3-(*N*-2-methacryloyloxyethyl-*N,N*-dimethyl) ammonatobutanesulfonate), MABS] spacer. Neutron reflectivity experiments were carried out at BL16 SOFIA in J-PARC (Ibaraki, Japan). Volume fraction profiles for the poly(sulfobetaine)s were calculated from scattering density profile obtained through curve fitting of the neutron reflectivity profiles. The high-resolution reflectivity profiles for the swollen poly(sulfobetaine) brushes afforded the detailed insight of the inhomogeneous swelling state. The thickness of the poly(sulfobetaine) brushes under deuterium oxide (D_2O) increased twice the poly(sulfobetaine) brush thickness in the dried state, whereas the cutoff thickness of the diffusive swollen layer is quite low. The sulfobetaine units are hydrated by D_2O to lead the polymer brush thickness increment but the interzwitterion paring prevents the formation of diffusive swollen interface. On the other hand, addition of salt and heating break the aggregation structure of the hydrated zwitterions to make the interface much diffusive. Correlation of the hydration state and the macroscopic material character in lubrication, adhesion, and anti-fouling property was clearly demonstrated through the swelling state investigation.

Abstract

Paper Ref: 386

PO59

SMALL-ANGLE NEUTRON SCATTERING STUDY ON UNIQUE PHASE SEPARATION OF THERMO-RESPONSIVE POLYMER IN IONIC LIQUID

Mr Kazu Hirosawa [1], Dr Kenta Fujii [2], Dr Takeshi Ueki [3], Dr Yuzo Kitazawa [4], Dr Kenneth Littrell [5], Dr Masayoshi Watanabe [4], Dr Mitsuhiro Shibayama [1]

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[4] *Yokohama National University*
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Recently, it was reported that poly(benzyl methacrylate), PBnMA and its derivatives show a lower critical solution temperature, LCST-type phase separation in a hydrophobic IL, 1-ethyl-3-methylimidazolium bis(trifluoromethanesulfonyl)amide, [C_2mIm$^+$][TFSA$^-$].[1-2] Interestingly, slight modification on chemical structure of polymers or ILs results in a drastic variation of T_c in polymer-IL systems. For example, poly(2-phenylethyl methacrylate), PPhEtMA shows phase separation at 315 K in [C_2mIm$^+$][TFSA$^-$], which is about 60 K lower than T_c of PBnMA (375 K). However, structural information about the mechanism of phase separation in polymer-IL solution system is not clear at the present stage.

We carried out small-angle neutron scattering (SANS) experiment for PPhEtMA in deuterated ionic liquid, d_8-[C_2mIm$^+$][TFSA$^-$] at High-Flux Isotope Reactor in Oak Ridge National Laboratory, USA. d_8-[C_2mIm$^+$][TFSA$^-$] was prepared by the procedure established in our research group. The deuteration rate of the synthesized d_8-[C_2mIm$^+$][TFSA$^-$] was confirmed to be higher than 90 %. Fig. 1 shows the SANS profiles of 3 wt% PPhEtMA solution of d_8-[C_2mIm$^+$][TFSA$^-$] at various temperatures, 288 K – 310 K. As can be seen from the figure, steep upturn was observed when temperature is larger than T_c (~ 307 K in d_8-[C_2mIm$^+$][TFSA$^-$]), indicating the appearance of large aggregates in this region. Here, note that the scattering profile changes discontinuously when temperature increase across T_c. Such a behavior is completely different from poly(N-isopropylacrylamide), PNIPAm aqueous solution which also shows a LCST-type phase separation. It is reported that the scattering profile of PNIPAm aqueous solution varies continuously with increasing temperature, resulting in divergence of correlation length.[3] Such a difference in the scattering

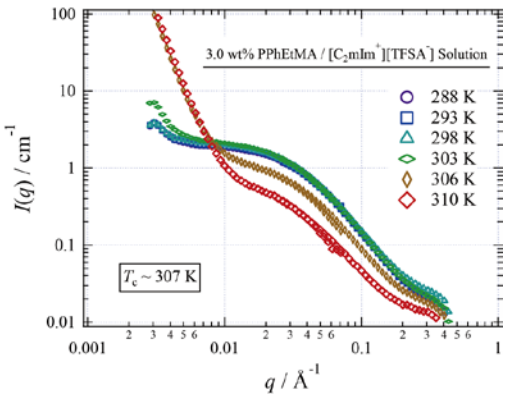

Fig. 1 SANS profiles of PPhEtMA / d_8-[C_2mIm$^+$][TFSA$^-$] at various temperatures in 288 K – 310 K.

profile indicates the fundamental difference in the mechanism of phase separation of polymer in IL and aqueous solution.

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2. Kodama, K. *et al.*, *Langmuir* **2009**, *25*, 3820
3. Shibayama, M. *et al.*, *J. Chem. Phys.* **1992**, *97*, 6829

Abstract

Paper Ref: 22

PO60

WATER PERMEABILITY STUDY OF ORDERED PS-B-PMMA BLOCK COPOLYMER FILMS

Mr Yeongsik Kim [1], Mr Hoyeon Lee [1], Ms Hyerin Yoon [1], Mr Jaseung Koo [2], Mr Jeong Soo Lee [2], Mr Du Yeol Ryu [1]

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Block copolymers (BCPs) can be used as nano-template and scaffolds, since dissimilar polymers linked together covalently self-assemble into the periodic arrays of lamellar, cylindrical, spherical, and gyroid morphologies with typical feature sizes of tens of nanometer and either one component can be selectively tuned to generate the nanostructures. To optimize the BCP films for practical use as templates, the perpendicular orientation of microdomains are preferred and the several techniques are developed such as the surface modification, solvent evaporation, and graphoepitaxy. Polystyrene-*b*-poly(methyl methacrylate) (PS-*b*-PMMA) is the one of widely used BCPs to produce the perpendicular orientation under a neutral confinement along with the similarity of surface energy between the two blocks.

In this study, we controlled the cylindrical microdomain orientation of PS-*b*-PMMA arrays by adjusting the surface energy on substrate. A parallel microdomain orientation of cylinders would be favorable with preferential interactions of the substrate with one block, and/or the difference in surface energies of the two blocks, while the perpendicular orientation of cylinders can be achieved by neutralization of surface using PS-*r*-PMMA.

A homemade chamber was used and the water permeability for the BCP films was measured. Using neutron reflectivity (NR), we investigated the permeability of BCP films with deutrated water to investigate the orientation dependence of cylindrical microdomains, indicating that the water permeation is favorable with the perpendicular orientation rather than parallel orientation.

Abstract

Paper Ref: 95

PO61

Structure and Dynamics of Polymer Thin Films at an Attractive Graphene Oxide Surface: Neutron Reflectivity Studies

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We investigated a mobility of polymers near a graphene oxide surface from dewetting dynamics and diffusion dynamics of polymer thin films on GO monolayers. Langmuir-Schaefer (LS) technique was applied to deposit the GO monolayer on substrate surfaces. Neutron reflectivity and atomic force microscopy (AFM) were utilized to measure the film structure of deposited GO sheets, revealing that the GO was deposited with single layer thickness without coagulation. From dewetting and diffusion dynamics results, we found that the polymer mobility can be restricted near GO surfaces. GO sheets significantly stabilized the PS/PMMA bilayers against dewetting above T_g. This reduced mobility of polymers in thin films was also seen from diffusion dynamics of the PMMA in thins films near the GO surface from the neutron reflectivity measurement. This can be attributed to favorable interaction between the GO sheet and PMMA layer due to the function of GO sheets as "amphiphilic membranes". This amphiphilic property was supported by the measurement of assembled structure of PS-*b*-PMMA thin films, where microdomains of copolymer were perpendicularly orientated on GO monolayer.

Abstract

Paper Ref: 225

PO62

Self-assembled Structures of Amphiphilic molecules induced by external Conditions: Small Angle Scattering Studies

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Amphiphilic molecules (i.e. block copolymers, surfactants and lipids) self-assemble into various nanostructures and represent rich phase behaviors according to their geometrical molecular shape in water. The geometrical molecular shape of amphiphilic molecules can be readily changed by the external conditions such as temperature, pH and additives without a complicated chemical reaction, which controls their self-assembled structures. Therefore, the amphiphilic molecules have been fascinating for a wide range of potential applications such as nanobuilding blocks, nanotemplates or drug delivery. Even though the phase behavior of amphiphilic molecules under different external conditions is fundamental information for their practical use, there have been still remaining to study it it has not been fully exploited yet. Here, we report the various self-assembled structures of block copolymers under different external conditions, which have been investigated by using the small angle neutron scattering (SANS). Depending on the external conditions, the SANS intensity showed that the block copolymer formed a variety of nanostructures including sphere, cylinder micelle and vesicle in aqueous solution.

Abstract

Paper Ref: 403

PO63

Neutron Reflectivity Investigation for Segment Distribution of Short Diblock Chain in Block Copolymer Binary Blend Having Hydrogen Bonding

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[3] *University of Texas, Austin, United States*

A binary mixture of two block copolymers whose blocks are capable of forming the hydrogen bonding allows one to obtain various microdomains that could not be expected for neat block copolymer. For instance, we reported that the binary blend of asymmetric polystyrene-block-poly(2-vinylpyridine) copolymer (PS-b-P2VP) and polystyrene-block-polyhydroxystyrene copolymer (PS-b-PHS) blends where the hydrogen bonding occurred between P2VP and PHS showed highly asymmetric lamellar microdomains. To explain the unexpected results, one should know the exact location of short block copolymer chains in the interface. For this purpose, we synthesized deuterated polystyrene-block-polyhydroxystyrene copolymer (d-PS-b-PHS) and prepared a binary mixture with PS-b-P2VP. We investigate, via small angle X-ray scattering (SAXS) and neutron reflectivity (NR), the exact location of shorter dPS block in the mixture near the interface of the microdomains.

Abstract

Paper Ref: 191

PO64

Complementary studies of SANS and SAXS on structure of Pluroinc F108 micelle in citric acid solution

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Effects of citric acid on the micellar structures of Pluronic F108 (PL F108) in 0, 5, 10, 20 and 24% citric acid (CA) D_2O solutions were investigated by small-angle neutron and X-ray scattering (SANS and SAXS). The different origins of scattering length densities (SLD) of neutron and X-ray probe the morphological changes of PL F108 and distribution of CA in this system. The cylindrical micelle of self-assembled PL F108 was observed in D_2O and CA solutions with different dimensions depending on CA concentrations. Protonated D_2O ($D_2(H)O^+$), CA and $citrate^-$ molecules would decorate on PEG shell of the polymeric micelle through hydrogen bonds. The micellar radius in 5% CA solution was larger than that in D_2O, while the micellar radii slightly decreased in 10-24% CA solutions. A stretch PEG shell could be obtained in 5% CA solution due to the electrostatic positive force provided by $D_2(H)O^+$. However, the CA and $citrate^-$ molecules would screen the positive charges of $D_2(H)O^+$ on the PEG shell as CA concentration increased resulting in less stretched PEG chains. In addition, the decoration of $D_2(H)O^+$, CA, and $citrate^-$ molecules on PEG shell increased the surface area of PEG segments. Thus, a spherical-like shaped PL F108 micelle were preferred to form due to the increase of curvature of micellar surface. The large-scale structure of PL F108 in CA solutions, mainly contributed from the anisotropically distributed $D_2(H)O^+$, CA, and $citrate^-$ molecules could be identified by SAXS measurements as well.

Abstract

Paper Ref: 24

PO65

INTERFACIAL WIDTH BETWEEN DEUTRATED PS FILMS AND PS BRUSHES WITH VARIABLE GRAFTING DENSITY

Mr Hoyeon Lee [1], Miss Yeongsik Kim [1], Mr Yeseul Shin [1], Mr Toyoaki Hirata [2], Mr Norifumi L. Yamada [3], Mr Keiji Tanaka [2], Mr Du Yeol Ryu [1]

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[3] *Neutron Science Laboratory, High Energy Accelerator Research Organization*

In polymer thin film system, the type of interfacial interaction is a critical parameter to determining the thermal and physical properties of polymer films. Polymer brushes using end-functionality were studied for modifying substrate to control interfacial interactions between the substrate and melt polymer layer on it. With the different chemical structures of modified polymer brushes, the thermal dynamics of polymer films are dynamically change, such as adhesion, lubrication, slip, and the wettability. Interestingly, the interfacial energy of brush layer is remarkably altered by simply controlling brush's grafting density, which has been referred to as *autophobic behavior*. This behavior was turned out to be more significant at densely grafted layer, indicating the high surface tension of a brush layer on polymer films with the same chemical identity. Many efforts have been devoted to modulating the interfacial interactions with changing the condition of modified polymer brushes, such as chemical identity, chain length and grafting condition.

In this study, we controlled the molecular weight of hydroxyl-functionalized polystyrene (HO-PS) to modify the surface in a standard Si wafer. Grafting HO-PS to a native oxide layer was performed by thermal annealing, and followed by rinsing with toluene to thoroughly remove the un attached polymer chains. Deutrated polystyrene (dPS) was prepared on the PS brush layer to enhance the neutron contrast between the brush layer and top polymer films. The interfacial widths between layers were calculated by Neutron Reflectivity (NR) experiments to define the interfacial interactions between the PS brush layer and top dPS polymer films. The detail calculation of the interfacial interaction will be discussed in the presentation.

Abstract

Paper Ref: 389

PO66

Small Angle Neutron and X-ray Scattering Study for Binary 1D Nanoparticle Superlattice

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[2] European Synchrotron Radiation Facility, Grenoble, France.

One-dimensional (1D) nanoparticles exhibit novel optical, mechanical and electrical properties which originate from anisotropic shape and size-dependent properties, making it an excellent building block for a broad spectrum of applications. Synthesis of binary nanoparticle superslattices, which may provide new emerging properties through collective coupling of two different types of nanoparticles, has been of great interests. While synthesis of binary superlattices of spherical nanoparticles have been intensively investigated and reported suing various approaches including slow evaporation, DNA-programming and so on, binary superlattice of 1D nanoparticles has been rarely reported. Recently our group reported a highly ordered binary superlattice of 1D nanoparticles where a hexagonal array of one type of 1D nanoparticles were embedded in a honeycomb lattice of another 1D nano-objects. In this presentation, the binary superlattices of 1D nanoparticles with different hierarchical structures depending on the diameter and mixing ratios, which were investigated by small angle neutron and x-ray scattering techniques, will be discussed. The understanding obtained in this study provides new insights for designing highly ordered binary superlattice of 1D nanoparticles.

[1] S.-H. Lim, H.-S. Jang, J.-M. Ha, T.-H. Kim, P. Kwasniewski, T. Narayanan, K. S. Jin and S.-M. Choi, *Angew. Chem. Int. Ed.*, 53, 12548-12554 (2014)

Abstract

Paper Ref: 313

PO67

COMPARISON OF INTERACTIONS OF TWO BIOPOLYMERS WITH A DESIGNED PROTEIN SURFACTANT AT THE AIR-WATER INTERFACE BY NEUTRON REFLECTION STUDY

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[2] *Bragg Institute, Australian Nuclear Science and Technology Organisation*

Protein-biopolymer mixtures find common applications in food and pharmaceutical processes where biopolymers (such as cellulose and polysaccharide) are used to enhance formulation for protein-based foam and emulsion. The interactions between protein and biopolymer molecules play a critical role in the interfacial structure and stability. Studies have been focused on interactions of protein and small-molecular-weight surfactants in protein-surfactant mixed systems. However, very limited research has paid attention to protein-biopolymer mixed systems.

In this work, neutron reflectometry was employed to compare the different interactions between protein and two types of biopolymer molecules at the air-water interface. Hydroxypropylmethyl cellulose (HPMC) and pectin were chosen as representative biopolymers to mix respectively with a designed protein – DAMP4. HMPC represents polymers with high surface activity (significantly reducing surface tension); and Pectin represents polymers with less surface activity (not significantly reducing surface tension). Two contrast variations were used for each mixture system: deuterated DAMP4 – hydrogenated polymer and hydrogenated DAMP4 – hydrogenated polymer.

It is found that at a high bulk polymer concentration (0.1%wt.), DAMP4 adsorption was inhibited by both HPMC and Pectin. In the DAMP4-HPMC mixed systems, HPMC inhibited DAMP4 adsorption by dominating the surface, while Pectin did by slowing down DAMP4 adsorption kinetics in the DAMP4-Pectin mixed systems. At lower bulk polymer concentrations (10^{-2} – 10^{-5} %wt.), DAMP4 surface excess increased with the decrease in HPMC bulk concentration in DAMP4-HPMC mixed systems. For DAMP4-Pectin mixed systems, DAMP4 adsorption kinetics was significantly speeded up at very low pectin bulk concentration (10^{-4} & 10^{-5} %wt.). These findings are fundamental and critical to understand the relationship between interfacial properties and functions of foams and emulsions, and will guide the design of tailorable foaming and emulsion systems for widespread applications.

Abstract

Paper Ref: 273

PO68

Probing the structures of a pH and salt responsive polymer brush

Timothy J Murdoch[1], Joshua D Willott[1], Ben A Humphreys[1], Grant B Webber[1], Steve Edmondson[2], Robert Barker[3], Andrew Nelson[4], Robert M Richardson[5], **Stuart W Prescott[6]**, Erica J Wanless[1]

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We report the swelling and deswelling behaviour of polymer brushes synthesised from the weakly basic poly((2-diethylamino)ethyl methacrylate). The brushes are responsive to changing pH and KNO_3 concentration as demonstrated by *in situ* ellipsometry and a quartz crystal microbalance with dissipation [1]. Cycling the pH showed that the kinetics of switching between a collapsed and swollen state was significantly faster than the reverse process. Our currently proposed mechanism for pH induced swelling and collapse is that the brush swells from the top and collapses from the base upwards [2]. Time resolved neutron reflectivity has allowed the structure normal to the surface to be tracked during these transitions.

At low salt (<10 mM), the brush was found to swell over a narrow concentration range (~1 mM), whereas theory predicts a weaker power law dependence due to the increased charging of the brush (osmotic brush regime). At higher salt concentrations where the charge in the brush is maximised, the brush collapses with increasing salt concentration due to electrostatic screening (salted brush regime). Neutron reflectivity was used to elucidate the ionic strength dependent structure of the brush. Additionally, a recently developed cell was used to study the effect of physical confinement on the ionic-strength dependent swelling of the brushes at pressures up to 7.0 bar [3].

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Abstract

Paper Ref: 410

PO69

Structures of Dendrimer-Surfactant Complexes Studied by Small Angle X-RAY Scattering

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[1]*Department of Chemical Engineering*, *National Tsing Hua University, Hsinchu, Taiwan*

Dendrimer-surfactant complexes have gained increasing attention due to their potential applications as templates to nanoparticle synthesis and building blocks for ordered nanostructures. These systems may also be used to simulate dendrimer-hydrophobic drug conjugates for controlled release. In this study, the self-assembled structures of the electrostatic complexes formed by dodecylbenzenesulfonic acid (DBSA) with poly(amidoamine) (PAMAM) dendrimers of generation four (G4) and six (G6) were studied by means of small-angle X-ray scattering (SAXS). For DBSA-G4 dendrimer complexes, the complexation caused the deformation of dendrimers from spherical to disklike shape at low surfactant-to-amine molar ratios (x). At high x, the complexes precipitated out of the aqueous medium. The self-assembled structure in the condensed state transformed from a hexagonal columnar phase to a lamellar structure as x increased from 0.1 to 1.0. The lamellar structure was formed by a monolayer of dendrimers incorporated between the DBSA bilayers with tail-to-tail arrangement. The DBSA-G6 dendrimer complex with x = 1.0 also formed a lamellar structure with a interlamellar distance about 34 Å, which was obviously smaller than the diameter of neat G6 dendrimer. The results thus revealed that the binding with the surfactant induced drastic deformation of dendrimer molecules. Moreover, it is the packing of the DBSA molecules that governs the characteristics of the lamellar structure rather than the stiffness of dendrimers. Small angle neutron scattering (SANS) experiment is underway to resolve the deformation of dendrimer molecules upon binding with DBSA.

Abstract

Paper Ref: 396

PO70

AGGREGATION IN MIXED SYSTEM OF NONIONIC AND CATIONIC CLASSIC SURFACTANTS STUDY BY SANS METHOD

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[1]National Centre for Nuclear Research

The mixed system of nonionic classic surfactant $C_{14}E_7$ (heptaethylene glycol monotetradecyl ether) and cationic surfactant CTABr (cetyltrimethylammonium bromide) in heavy water solutions was investigated for concentration of nonionic surfactant c=0.17% (dilute regime) at temperatures t= 20º and 35 ºC with two methods - tensiometric and small-angle neutron scattering (SANS) on SANS spectrometer ("YuMO") of the IBR-2 on pulsed neutron source at FLNP, JINR in Dubna (Russia). Measurements have covered Q range from $8x10^{-3}$ to 0.4 $Å^{-1}$. The micellar solutions were prepared in D_2O since the contrast between the micelles and the solvent in neutron experiments is better with D_2O than with H_2O. It was obtained as the result that the shape of micelles changes depending on surfactant concentration and temperature. At lower concentrations and temperatures micelles are ellipsoids but at higher concentrations and temperature are rather cylinders. For calculation and approximation results from SANS experiment was used program PCG 2.0 of Glatter O. and co-workers from University of Graz (Austria).

Abstract

Paper Ref: 137

PO71

BEHAVIOR OF ROD-LIKE PARTICLES IN DEFORMED NANOCOMPOSITE HYDROGEL

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[2] *Korea Atomic Energy Institute*

Nanocomposite materials has been developed to confer high performed mechanical property, and one of the efforts is preparation of inorganic particle induced hydrogel. In this research, the gamma-ray irradiated imogolite-acrylic acid hydrogel was investigated with small angle X-ray scattering (SAXS) and small angle neutron scattering (SANS) in terms of particle concentration and deformation. SAXS and SANS were performed to probe the polymer contrast matched system and the ternary system, respectively. The SAXS analysis results present polymer dominant elongation, particle orientation and elongation of inter-particle distance as structural response against the external deformation. The neutron scattering analysis also supports the elongation of internal structure through the increase in correlation length of inhomogeneity of the system according to the magnitude of deformation

Abstract

Paper Ref: 207

PO72

CHARGE EFFECTS ON STRUCTURE OF NON-IONIC SURFACTANT SOLUTION

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Electrostatic interaction plays an important role to stabilize nano-scale structures of soft matter. We have recently shown that the electrostatic interaction due to the heterogeneous distribution of cations and anions of an antagonistic salt, sodium tetraphenylborate ($NaBPh_4$), induces the ordered lamellar structure in the water-rich mixture of D_2O and 3-methyl pyridine (3MP). [1] This phenomenon is understood as the interfacial tension between D_2O and 3MP is reduced by the addition of $NaBPh_4$, a microphase separation of D_2O and 3MP domains is induced, and the lamellar structure is stabilized by the electrostatic force between 3MP domains. This evidence suggests that the antagonistic salt behaves as a co-surfactant when added to an aqueous solution of non-ionic surfactant.

Figure 1 shows the SANS profiles of the water-rich mixture of non-ionic surfactant $C_{12}E_5$, D_2O and an antagonistic salt, $RbBPh_4$, at 59 ˚C. Here the total water content is fixed at 90 wt%. The profile of the surfactant solution without salt (*S*=0 where *S* the molar ratio of a salt against non-ionic surfactant) indicates that a disordered structure is formed. By adding the antagonistic salt to the binary mixture, a Bragg peak indicating an ordered lamellar structure is emerged, and it grows with increasing the salt concentration, *S*. This experimental result shows that only a few amounts of ions, whose molar ratio against surfactants is an order of 1/1000, has a large influence on the nano-scale structure of the surfactant solution. This tendency is almost the same as we added other antagonistic salt or ionic surfactant, sodium dodecyl sulfate (SDS), to the binary mixture. These results could be interpreted that that the effect of adding antagonistic salt is the same as adding ionic surfactant, which increase the electrostatic interaction between non-ionic surfactant membranes.

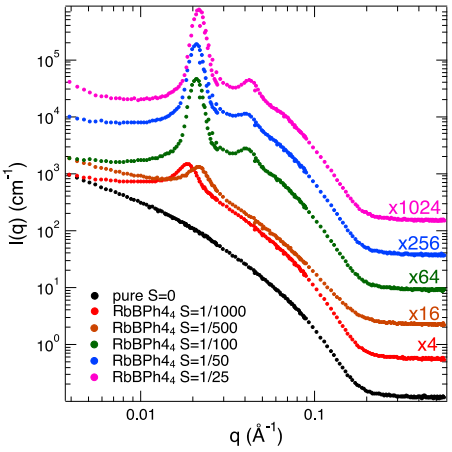

Fig. 1 SANS profiles observed from the mixtures with $C_{12}E_5$, D_2O and $RbBPh_4$.

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Abstract

Paper Ref: 35

PO73

Neutron Reflectometry on Interface Structure in Nano-scale TiAlN/CrAlN Bilayers

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Thin films of transition metal nitrides, such as TiN, CrN, CrAlN and TiAlN, exhibit remarkable physical and chemical properties of high hardness, low friction coefficient, high thermal stability and good oxidation resistance. These thin films have found numerous applications, i.e. cutting tools, high temperature thermal barrier coatings and tritium permeation resistant coatings for nuclear reactor. In particular, recent advance on nano effect of the nano-scale thin films has driven the study of nitride ceramic thin films onto a new level, in which the interface structure in nano-scale thin film is a critical factor in delivering a nano effect.

Reflectometry is the most applicable technique for characterisation of interface structure of thin films. However, the interaction between light elements and X-ray is reletively weak, whereas neutron is particularly sensitive to nitrogen, so that neutron reflectometry become a suitable probe in study of interface structures involving nitrogen element. Neutron reflectometry has been theoretically and experimentally developed in the past decades [1], and the interface structures of some ceramic thin films have been successfully revealed by neutron reflectometry [2,3].

TiAlN/CrAlN and CrN/TiN bilayer films with a whole thickness of around 150 nm have been fabricated on Si substrates by reactive magnetron sputtering. The neutron reflectometry (NR) measurements are performed to investigatge the thickness and interfacial roughness of the bilayers and element diffusion on interfaces of the films. Fig.1 presents the non-polarized specular neutron reflectivity curves and SLD profiles of the bilayers. The reflectivity curves exhibit characteristic Kiessig fringes associated with multiple reflections, and are successfully fitted using a bilayers model. The average thicknesses of the TiAlN and CrAlN layers in a bilayer of the sample, obtained from the unpolarized NR measurements, are 71.8nm and 84.5nm, respectively. The measured thickness is within 10% of the expected value (~ 150nm). The interface roughness of the TiAlN/CrAlN interfaces is 1nm. The SLD profiles in Fig.1(a) (inset) also reveal very thin diffusion interfaces with the thicknesses of about 2.3nm and 4.7nm in CrAlN/TiAlN and TiAlN/Si interfaces, respectively. It is indicated that the interfaces in bilayers are sharp. These NR results have been discussed and compared with those obtained by X-ray reflectometry on the same samples (data not shown).

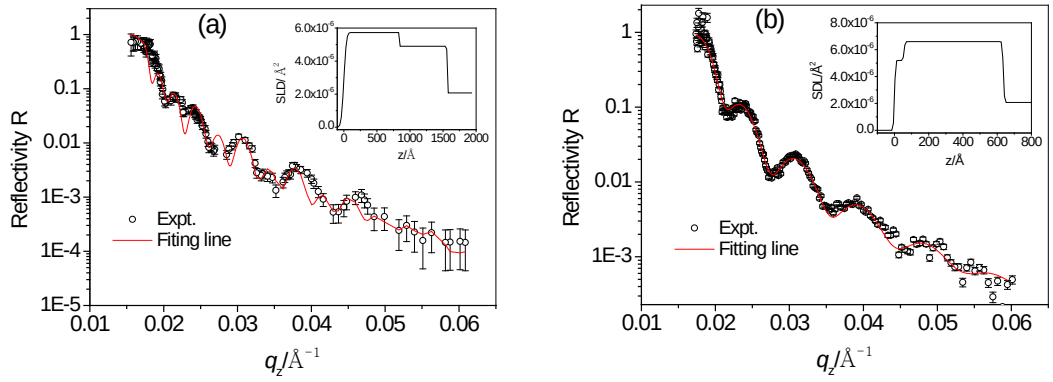

Fig. 1. Observed unpolarized specular neutron reflectivities of (a) CrAlN/TiAlN and (b) CrN/TiN films together with model fit.

Acknowledgements

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Tuesday 21 July 2015

Poster Session 2 – CMP, EI and MSC

Condensed Matter Physics

Abstract

Paper Ref: 160

PO75

NEUTRON SCATTERING STUDIES OF $YbMn_2Si_2$ - MAGNETIC EXCITATIONS

Richard A. Mole[1, 4], Michael Hofmann[2, 4], Jianli Wang[3, 4] and **Stewart J. Campbell[4]**

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Ternary intermetallic RT_2X_2 compounds (R - rare earth, T - transition metal and X - p-block element e.g. Si or Ge) with the $ThCr_2Si_2$ structure continue to attract attention. RMn_2X_2 compounds are of particular interest as Mn atoms carry a magnetic moment [*e.g.* 1]. The magnetic states in RMn_2X_2 systems are sensitive to chemical composition and intraplanar Mn-Mn spacing thereby allowing control of the interplay between R-Mn and Mn-Mn exchange interactions. More recently RMn_2X_2 compounds have attracted attention due to their potential for applications that exploit different coupling mechanisms such as the magnetocaloric effect, magneto-elasticity and induced magnetostriction [2, 3].

While the overall structural and magnetic behaviour of RT_2X_2 compounds are increasingly well understood, less attention has been paid to the magnetic and crystal field excitations. Our recent neutron scattering investigations of $YbMn_2Si_2$ provided insights to magnetic structures (collinear antiferromagnetic AFil structure below T_{N1} = 526(4) K and doubling of the magnetic c-lattice below T_{N2} = 30(5) K; [4]) and crystal field excitations to ~ 25 meV [5]. Above T_{N2} the latter results are described by the Yb crystal field while additional peaks are observed below T_{N2}, consistent with a perturbation of the crystal field by the ordering of the Mn sublattice.

Energy level diagrams were determined for Yb^{3+} ions in the different sites above (single site) and below T_{N2} (two sites). The excitation energies for both sites are temperature independent with

the temperature dependences of the transition intensities described well by a Boltzmann model. Detailed insight to the excitations occurring in $YbMn_2Si_2$ at low temperatures (T<~ 30 K) in the low energy region below ~5 meV have recently been obtained using the cold neutron t-o-f spectrometer Pelican at the OPAL reactor. The continuing analysis of the additional and unexpected features observed using Pelican will clarify further the complex behaviour exhibited by $YbMn_2Si_2$.

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Abstract

Paper Ref: 319

PO76

Metamagnetism of Single Crystal $FeTiO_3$ Investigated by High Field Neutron Diffraction

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Metamagnetism refers to a magnetic material which at a critical applied field undergoes a magnetic phase transition [1], causing the magnetisation of the specimen to increase significantly. Ilmenite (FeTiO3) is a hexagonally layered antiferromagnet exhibiting strong uniaxial anisotropic behaviour of the magnetic susceptibility, and a metamagnetic transition with fields applied along the c-axis. While the crystal properties of bulk ilmenite are reasonably well understood, the thin film form of this material continues to raise a lot of questions. The overreaching aim of this research is to establish whether thin films of FeTiO3 possess the adaptability to become a competitive material with applications in spintronics due to a possible switchable metamagnetic transition.

Initial studies towards this goal focused on powdered and single crystal samples which will later be used to synthesise epitaxial $FeTiO_3$ thin films. The nuclear and magnetic structure of both $FeTiO_3$ powder and a c-cut 10x5x5 mm^3 single crystal, prepared by the floating zone method, were investigated on the high-intensity neutron diffractometer (WOMBAT) at ANSTO. Diffraction experiments at 0 T showed a magnetic phase transition to an antiferromagnetic (AFM) state at ~ 64 K. Experiments on this single crystal in the (h0l) alignment at 0 T also revealed a doubling of the magnetic unit cell at low temperature which is characteristic of an AFM ground state. In addition, the antiferromagnetic spin-canting structure of $FeTiO_3$ was directly confirmed with the occurrence of the (00l) reflections. Furthermore, the intensities of the (00l) and (h0l) type magnetic reflections, revealed that the spin-canting angle of $FeTiO_3$, below the magnetic ordering temperature, is approximately temperature independent. Under the application of high fields along the a-axis, magnetorestrictive effects were found to increase the intensity of both magnetic and atomic reflections at low temperature. In an attempt to observe the metamagnetic transition of $FeTiO_3$, external fields were then applied parallel to the crystals c-axis. Such investigations revealed that a complex magnetic structure in the ab-direction collapsed, as predicted, to a simple ferromagnetic state under the application of a high field along the c-axis.

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Abstract

Paper Ref: 114

PO77

Magnetic ordering and excitations in frustrated tetragonal spinel $ZnMn_2O_4$

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Spinel oxides (AB_2O_4) are one of the best known systems for geometrical frustrations to be observed. The geometrical frustrations in this system are primarily ascribed to the antiferromagnetic interactions among the B site ions. These are particularly strong under cubic symmetry, and stabilization of long-range ordering often requires symmetry-breaking lattice distortions. In $ZnMn_2O_4$ the B sites are occupied by Mn^{3+} ($3d^4$, S=2) ions, which stabilize the tetragonal (c > a) lattice symmetry due to the Jahn-Teller effect [1]. Even though simple antiferromagnetic ordering has been expected, the nature of magnetic structure is elusive experimentally [2-3].

In this work, we studied magnetic ordering and excitations of polycrystalline $ZnMn_2O_4$ using magnetization and neutron scattering measurements. Whereas magnetic susceptibility did not reveal clear onset of antiferromagnetic transitions, neutron powder diffraction showed two successive magnetic orderings below 50 K. The magnetic structure below 35 K is best described with the model involving cell-doubling-type modulations of the antiferromagnetic chains. These modulations are observed equivalently along two high-symmetry directions in the *c* planes. Inelastic neutron scattering in the same phase showed magnetic excitations with an anisotropy gap of 7 meV and the band width of 20 meV. Using linear spin wave calculations, we show that the cell-doubling-type magnetic ordering is stabilized by next-nearest-neighbor exchange interactions.

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Abstract

Paper Ref: 166

PO78

ANOMALOUS MAGNETIC INTERACTION AND CYCLOIDAL SPIN DESTRUCTION IN $BI_{0.9}CA_{0.1}FeO_3$ AND $Bi_{0.9}Pb_{0.1}FeO_3$

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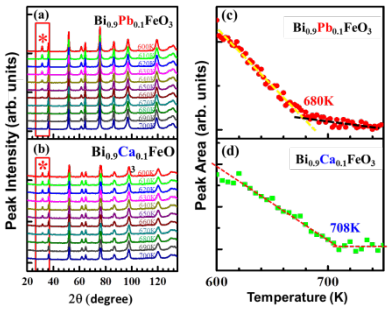
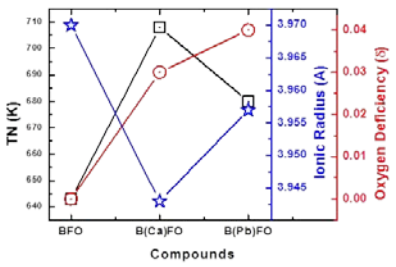
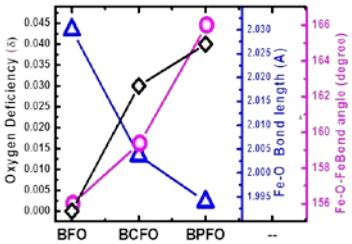
BiFeO3 (BFO) is a multiferroics that can operate above room temperature and, therefore, has a great potential to be used in spintronics devices to manipulate the magnetic state by indirectly controlling electric field. The antiferromagnetic cycloidal spin structure of BFO weakens the coupling strength at the FM/BFO interface and increases difficulties in control the magnetic state of the FM layer by applying electric field. Cation doping at Bi sites have shown improving in multiferroic characteristics with mechanism not clearly understood. In this study, temperature dependent neutron diffraction patterns of Ca and Pb doped samples, $Bi_{0.9}Ca_{0.1}FeO_3$ and $Bi_{0.9}Pb_{0.1}FeO_3$, show their Neel temperature (T_N) has been increased to 710 K and 720 K. This anomalous increase in T_N have been investigated in detail by probing X-ray absorption spectroscopy, X-ray photoemission spectroscopy techniques and structural refinement of neutron and X-ray diffraction patterns. Room temperature X-ray absorption spectroscopy clearly shows that there is no evidence of mixed valence states on Fe ions despite of the divalent cations doping in trivalent Bi-sites. Instead of that, divalent dopants have introduced oxygen vacancies in the system. Structural refinement of neutron diffraction patterns also support the presence of oxygen vacancies in $Bi_{0.9}Ca_{0.1}FeO_{3-}$ and $Bi_{0.9}Pb_{0.1}FeO_{3-\square}$. Oxygen deficiency ($\square$) plays a pivota role in reducing Fe-O bond length in FeO_6 octahedra and hence increasing the Fe-O-Fe bond angle in $Bi_{0.9}Ca_{0.1}FeO_3$ and $Bi_{0.9}Pb_{0.1}FeO_3$. This decreased Fe-O bond length and increased Fe-O-Fe bond angle favors the Goodenough-Kanamori-Anderson (GKA) coupling that enhances the magnetic interaction between the spins and hence increases the T_N. Additionally, stronger magnetic coupling between the spins stables the magnetic structure resulting in the destruction of cycloidal spin structure and formation of a simple but minor canted antiferromagnetic (AFM) structure.







Abstract

Paper Ref: 25

PO79

Coexistence of long-range magnetic ordering and singlet ground state in the spin ladder superconductor $SrCa_{13}Cu_{24}O_{41}$

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$Sr_{14-x}Ca_xCu_{24}O_{41}$ is the only known high temperature superconductor system with a quasi-one-dimensional spin ladder structure. Beside its superconductivity, many exotic physical properties, such as charge transfer, hole crystallization, etc. were observed in this compound. With extended high pressure single crystal growth technique, a series of highly Ca-doped $Sr_{14-x}Ca_xCu_{24}O_{41}$ single crystals were grown,[1] which allow us to investigate the physics in these materials with neutron scattering. With the highest Ca-doped single crystal sample $SrCa_{13}Cu_{24}O_{41}$, a long range magnetic order was discovered by susceptibility, specific heat and neutron diffraction experiments. The temperature dependence of the magnetic Bragg peak intensity could be well fitted to the power law with a transition temperature T_N = 4.23K and a critical exponent β = 0.28, indicating a three-dimensional phase transition for a low dimensional magnet. A computer program was developed and found two possible magnetic structure models fitting best with all the observed magnetic peaks. These models suggest the spin ladder sublattice is magnetically ordered with Cu moments aligning along the *a* axis. The spin interactions are primarily antiferromagnetic along rungs and legs while there are ferromagnetic clusters along legs. Surprisingly, the singlet-triplet spin gap excitation is observable above and below T_N,[2] indicating a coexistence of the spin singlet ground state and long range magnetic ordering state in this compound.[3]

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Abstract

Paper Ref: 385

PO82

Synthesis and Characterization of Magnetic Nanoparticle Superstructure

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Magnetic nanoparticles, which show unique magnetic properties depending on its size and shape, have been of great interests for various applications including contrast agent for magnetic resonance imaging, drug delivery, hyperthermia treatment of cancer, data storage, and green catalyst for environmental remediation. Recently, the magnetic nanoparticle superlattices with various symmetries have been of great interests as model systems to investigate dipolar magnetism. While simple magnetic nanoparticle superlattice with FCC symmetry has been readily synthesized and rather extensively investigated, synthesis of magnetic nanoparticle superlattices with various controlled symmetries still remains challenging. In this presentation, the synthesis and functionalization of monodisperse magnetic nanoparticles, and their self-assembled superstructures with different symmetries will be discussed.

Abstract

Paper Ref: 414

MAGNETIC PHASE TRANSITION AND MAGNETIC STRUCTURES IN $SmBaMn_2O_6$

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The materials represented as $ReBaMn_2O_6$ (Re = trivalent rare-earth metal) exhibit variety of orderings due to the competition between charges, orbitals and spins, as is seen in $ReMnO_3$ type materials. $ReBaMn_2O_6$ has a double perovskite-type structure with the A-sites alternately occupied by Re and Ba ions along *c*-axis. In the case of Re = Sm, a charge-orbital ordered phase (COO1) emerges below 380 K followed by a re-arrangement of COO (COO2) around 200 K accompanied by an antiferromagnetic phase transition. In addition, an anomaly in the magnetization around 10 K has been reported. Although the structure and the charge-orbital (CO) ordering pattern have already been understood, the magnetic structure and the relation between the re-arrangement of the CO ordering and the magnetic ordering around 200 K have not been clarified, yet. In this study, neutron magnetic diffraction technique was utilized in order to study the magnetic structure and the details of the phase transitions.

The experiment was conducted on the single crystal neutron diffractometer, SENJU, installed at MLF/J-PARC. A single crystal of $SmBaMn_2O_6$ with the size of ~ 2.0 x 4.0 x 1.2 mm^3 was mounted on a cryostat equipped with two rotating axes.

The observed diffraction pattern clearly revealed the existence of two types of magnetic superlattice reflections, one is at (0 1/2 1/2) and the other (1/2 1/2 1/2). These propagation vectors coincide with the CO ordering patterns of Mn ions. The propagation vectors were found to be unchanged below 10 K, while the changes in intensities of the magnetic superlattice reflections were observed. This suggests that only the orientations of the spins are changed below 10 K. As for the phase transitions around 200 K, the temperature dependences of the superlattice reflections of COO1 and the magnetic reflections clarified that the transformation of COO1 to COO2 occurred just above the antiferromagnetic phase transition.

Abstract

Paper Ref: 218

PO84

Strain-induced magnetic phase transition in $SrCoO_{3-\delta}$ thin films measured by neutron diffraction

Dr. Sara Callori [1,2], Mr. Songbai Hu [3], Dr. Joel Bertinshaw [1,2], Mr. Zengji Yue [4], Dr. Sergey Danilkin [1], Prof. Xiaolin Wang [4], Prof. Nagarajan Valanoor [3], **Prof. Frank Klose [1,5]**, A/Prof. Jan Seidel [3], A/Prof. Clemens Ulrich [2]

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It has been well established that both in bulk at ambient pressure and for films under modest strains, cubic $SrCoO_{3-\delta}$ ($\delta < 0.2$) is a ferromagnetic metal [1]. Recent theoretical work, however, indicates that a magnetic phase transition to an antiferromagnetic structure could occur under large strain accompanied by both the onset of ferroelectricity and a metal-insulator transition [2]. We have observed a strain-induced ferromagnetic to antiferromagnetic phase transition in $SrCoO_{3-\delta}$ films grown on $DyScO_3$ substrates, which provide a large tensile epitaxial strain (2.8%), as compared to ferromagnetic films under lower tensile strain on $SrTiO_3$ substrates (1.7%) [3]. Magnetometry results demonstrate the existence of antiferromagnetic spin correlations at high temperature. Neutron diffraction experiments were performed at the TAIPAN triple axis spectrometer at ANSTO and provide direct evidence for a G-type antiferromagnetic structure with Néel temperatures between T_N ~135 ±10 K and ~ 325 ± 10 K depending on the oxygen content of the samples (see Fig. 1). Our data experimentally confirm the predicted strain-induced magnetic phase transition to an antiferromagnetic state for $SrCoO_{3-\delta}$ thin films under large epitaxial strain. Therefore, the results presented here present the first step in obtaining new functionalities in $SrCoO_{3-\delta}$.

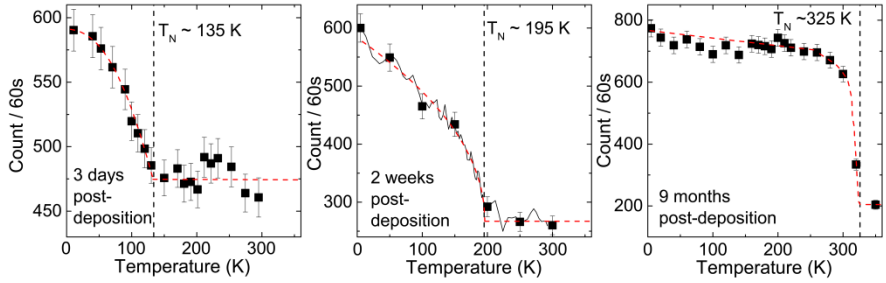

Fig. 1. Intensity of the antiferromagnetic neutron diffraction peak for a 20 nm $SrCoO_{3-\delta}$ film on $DyScO_3$ as a function of time post-deposition. An increase in Néel temperature as well as scattering intensity with aging is observed.

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Abstract

Paper Ref: 300

PO85

INVESTIGATION OF SPINTRONIC MATERIAL SYSTEMS USING POLARIZED NEUTRON REFLECTOMETRY ON PLATYPUS

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As the demand for highly functional magnetic storage materials grow, the need to understand the basic physical properties behind these materials has become key to engineering storage devices. Many recent approaches to this challenge have focused on spintronic devices, which enhance memory capabilities by adding spin as a degree of freedom to conventional semiconductors and insulators. Such systems are often built using thin films and layers of magnetic materials and the properties of the constituent materials, the interactions between them, and the interfaces in the system all contribute to the overall properties of the devices. As such, polarized neutron reflectometry (PNR) is an ideal tool for the study of such systems, as this technique allows both a nuclear and a magnetic depth profile of thin films and layered systems to be obtained. The PLATYPUS reflectometer at ANSTO provides an environment for users to perform PNR studies on a wide variety of materials. The instrument is able to perform both spin-flip and non-spin-flip, PNR, allowing the full in-plane magnetization vector of magnetic layers to be resolved, which is ideal for understanding the dynamics of magnetic reversal. Highly customizable sample environments allow the response of systems to variables, such as temperature and magnetic and electric field, to be probed.

Here, we will review recent PNR experiments on PLATYPUS which have focused on spintronic materials and highlight the capabilities of the instrument. These include the study of perpendicular magnetic coupling in multilayer spin valves [1], the depth dependence of doping in dilute magnetic oxide semiconductors [2], and hybrid ferroelectric/magnetic oxide heterostructures.

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Abstract

Paper Ref: 130

PO86

Field-dependent magnetic ordering and magnetic anisotropies in Zn_2Y-type hexaferrite $Ba_{0.5}Sr_{1.5}Zn_2(Fe_{1-x}Al_x)_{12}O_{22}$

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Hexagonal ferrites containing Fe^{3+} (S = 5/2) ions are attracting much attention because their high ordering temperatures offer positive prospect for realizing high-temperature low-field multiferroics. Y-type $Ba_{0.5}Sr_{1.5}Zn_2Fe_{12}O_{22}$ exhibits proper-screw antiferromagnetic order in zero field, which does not allow ferroelectric polarization according to the inverse Dzyaloshinskii-Moriya mechanism. Finite electric polarization can, however, be induced when a strong external magnetic field (~ 0.3 T) is applied perpendicular to its *c* axis. The field-response of this compound is known to become greatly improved via Al substitution on Fe sites which is ascribed to the formation of conical spin structures upon reduction of planar anisotropy. The largest magnetoelectric susceptibility has been observed for the sample with 8 % substitution.

In this work, we report the magnetic phase transitions in Zn_2Y-type hexaferrite $Ba_{0.5}Sr_{1.5}Zn_2(Fe_{0.92}Al_{0.08})_{12}O_{22}$ under external magnetic field applied perpendicular or parallel to the *c* axis. When the applied field was perpendicular, the pitch of conical spiral did not change significantly. Instead, an abrupt first-order incommensurate-to-commensurate ordering was observed [1]. When the applied field was parallel to the *c* axis, however, spiral pitch was gradually reduced and the longitudinal conical phase was stabilized regardless of the zero-field ordering. The incommensurate ordering persisted up to the maximum field of 3 T. These behaviors suggest that the field response of its magnetic ordering is strongly affected not only by the competing exchange interactions but also by coexisting multiple magnetic anisotropies fields.

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Abstract

Paper Ref: 12

PO87

Magnetic structure of $(Tb_{1-x}Mn_y)MnO_{3-\delta}$ studied by neutron diffraction

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Eight samples with the nominal formula $(Tb_{1-x}Mn_y)MnO_3$ (x=0.040, y=0.005 for S1; x=0.056, y=0.024 for S2; x=0.073, y=0.044 for S3; x=0.089, y=0.063 for S4; x=0.106, y=0.082 for S5; x=0.122, y=0.102 for S6; x=0.139, y=0.121 for S7; and x=0.155, y=0.140 for S8) have been characterized using neutron diffraction and magnetic measurements.

Similar behaviors as $TbMnO_3$ have been found for S1 and S2. Figure 1 shows the temperature dependent neutron diffraction data of S1. It is found that the diffraction patterns collected at 43 to 290K shown in Fig.1c are similar to each other. For the data collected at 10K to 42K, new reflections are found, which are attributed to the sinusoidal antiferromagnetic ordering of Mn with a wave vector ***q***= (~0.283, 0, 0) in the space group *Pna2₁* setting as that reported for $TbMnO_3$[1,2] This magnetic structure is noted as magnetic phase A. And for the data collected at 3.5 to 8K, round reflections are found, which can be attributed to the sinusoidal antiferromagnetic ordering of Tb with a wave vector ***q***= (~0.415, 0, 0) in the space group *Pna2₁* setting as that reported for $TbMnO_3$,[1,2] and noted as magnetic phase B. For S3, it is similar to S4. The magnetic diffraction peaks corresponding to the magnetic phase B are not observed. S5 is similar to S6. New magnetic diffraction peaks around 25° appear as shown in Figure 2, which are attributed to the antiferromagnetic ordering of Mn and Tb, and noted as magnetic phase C. In addition, the diffraction peaks corresponding to the magnetic phase A also appear in the neutron diffraction data collected at low temperature. As for S7 and S8, it is found that only magnetic diffraction peaks related to the magnetic phase C appear.

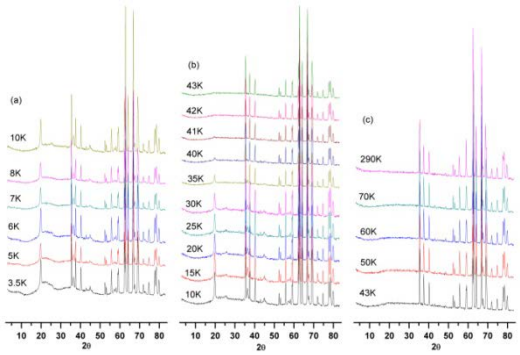

Figure 1 Neutron diffraction data of S1.

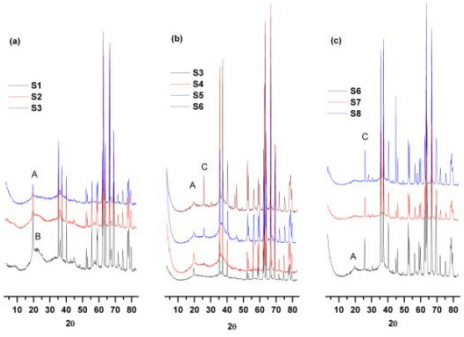

Figure 2 Neutron diffraction data for S1, S2, S4, S5, S6, S7, and S8 at 3.5K, and for S3 at 6K.

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Abstract

Paper Ref: 235

PO88

THE INTERPLAY BETWEEN EXCHANGE BIAS AND EXCHANGE SPRING IN PERMALLOY/HEMATITE BILAYERS PROBED BY POLARIZED NEUTRON REFLECTOMETRY

Dr. Sara Callori [1,2], Ms. Grace Causer [3], Dr. David Cortie [4], **Professor Ko-Wei Lin** [5], Professor Frank Klose [6]

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Magnetic thin films or multilayer structures often use neighboring antiferromagnetic (AF) and ferromagnetic (FM) components in order to utilize exchange bias [1], which occurs when the FM layer magnetization is pinned through interactions with the AF layer near the interface. In addition, the control over the magnetization distribution in the FM layer can be achieved through an exchange spring design [2]. Polarized neutron reflectometry (PNR) provides an ideal experimental platform to measure exchange spring/bias behaviour, as the technique is capable of measuring a depth profile of the in-plane magnetization vector within thin films and layered systems. Here we demonstrate that, by varying the thicknesses of FM $Ni_{80}Fe_{20}$ (Permalloy) and AF Fe_2O_3 (Hematite) as well as by magnetic field cooling combined with sample rotation procedures, either (1) a fan-like structure associated with exchange-spring behaviour in a $Ni_{80}Fe_{20}$ (~ 14 nm) / Fe_2O_3 (~ 16 nm) bilayer was created or (2) typical exchange bias behaviour in a $Ni_{80}Fe_{20}$ (~ 11 nm) / Fe_2O_3 (~ 8 nm) bilayer was found, as probed by PNR. X-ray diffraction and transmission electron microscopy show that the $Ni_{80}Fe_{20}$ / Fe_2O_3 bilayer fabricated by using a dual ion-beam sputtering deposition technique [3] consisted of fcc Permalloy (top layer) and hcp Hematite (bottom layer) structures, consistent with our previous work [1]. The average surface roughness in the $Ni_{80}Fe_{20}$ / Fe_2O_3 bilayer interface was determined to be ~ 0.1 nm, as measured by atomic force microscopy. PNR data have shown that a fan-like structure was created by rotating the sample 90° under an applied field with respect to the field-cooling direction. The perpendicular components of the exchange spring structure lead to a strong PNR spin-flip signal. Our results indicate that the turning angles of the magnetization within the Permalloy layer are related to the competition between the Permalloy/Hematite interfacial pinning and applied magnetic field.

The research was supported by MOST, Taiwan and ANSTO, Australia.

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Abstract

Paper Ref: 409

PO89

New type of symmetry breaking coupling with metal-insulator transition

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Metal-insulator Transition (MIT) is a kind of phenomenon demonstrating huge resistivity changes, and has been widely observed in strongly correlated systems. It is often found accompanied with breaking of the charge, spin and/or orbital degrees of freedom. In cobaltites, the competition between crystal field (CF), on-site Coulomb correlations, and the intra-atomic exchange energies leads to an additional degree of freedom for Co^{3+} ions in terms of three possible spin states: the low spin state (LS, $t_{2g}^6e_g^0$), the intermediate spin state (IS, $t_{2g}^5e_g^1$), and the high spin state (HS, $t_{2g}^4e_g^2$). The MIT in $RBaCo_2O_{5.5+x}$ can be induced by temperature, pressure and also hole doping. The temperature induced MIT was observed in the paramagnetic phase of $RBaCo_2O_{5.5}$ in conjunction with a concurrent anomaly in the inverse magnetic susceptibility that is generally attributed to the spin-state transition (SST). However, no satisfactory mechanism has been established for elucidating the correlation between the spin-state degree of freedom and the MIT. Moreover, consensus hasn't been reached on the detailed crystal structure of $RBaCo_2O_{5.5+x}$. To investigate the nature of the MITs in $RBaCo_2O_{5.5+x}$, we have carried out a multi-probe study on $PrBaCo_2O_{5.5+x}$ ($0 \le x \le 0.24$) polycrystalline samples using neutron, electron and X-ray beams.

Through careful structural study, we found that the MITs in $PrBaCo_2O_{5.5+x}$, induced by both temperature and hole doping, occurs simultaneously with the structure transition from *Pmmm* ($a_p \times 2a_p \times 2a_p$) to *Pmma* ($2a_p \times 2a_p \times 2a_p$). This symmetry breaking is caused by the spin-state ordering (SSO) along *a* axis, which is a new type of symmetry breaking that coupling with MIT. Furthermore, parallel behaviour was also observed in electronic states by x-ray absorption spectroscopy (XAS), indicating a common nature for the MITs. We believe both carrier number (*n*) vanishing by this new type of symmetry breaking and the transfer parameter (*t*) change by SST should be accounted for the MITs.

Abstract

Paper Ref: 433

PO90

Neutron Scattering Studies of $Co_3(OH)_2(C_4O_4).xH_2O$

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Cobalt hydroxy squarate, $Co_3(OH)_2(C_4O_4).xH_2O$, is a porous coordination polymer with complex magnetic behavior. We have shown that the magnetic behavior is influenced by both the frustrated exchange topology and the large single ion anisotropy associated with the unquenched orbital angular momentum of the octahedral Co(II) ions. Further the low density framework and the resulting large separation of ions by the squatate dianion give rise to a one-dimensional magnetic exchange topology. Neutron diffraction experiments have revealed that the magnetic properties are hydration dependent suggesting that the small changes in structure can give rise to significant changes in the magnetic properties. Neutron diffraction has also shown that there are multiple ordered phases including the so called idle phase whereby one of the magnetic sites does not order. Our recent work has focused on inelastic neutron scattering using the PELICAN time of flight spectrometer. The key result has been the discovery of a gapped, dispersionless, excitation associated with the idle spin. This excitation persists well above the magnetic ordering temperature and the gapped nature indicates the quantum behavior of this J = ½ ion. In this contribution I will summarize or recent results on this system.

Abstract

Paper Ref: 157

PO91

Structural and Magnetic Studies of the Uranium Oxides: $CrUO_4$ and $MnUO_4$

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Transition metals such as manganese and chromium have been shown to play an integral part in the optimized performance of uranium based nuclear fuels where they act as dopants, often taking the structural form of AUO_4 (*A* = Cr or Mn). Whilst such compounds were of interest in the pioneering stages of nuclear fuel development, their solid state chemistry and in particular magnetic properties have been largely neglected over subsequent decades.

Given what can be described as a renaissance of nuclear fuel based research, we have revisited the structural and magnetic properties of the materials $CrUO_4$ and $MnUO_4$. Initial analysis using synchrotron X-ray powder diffraction (SXRD), and X-ray absorption near edge structural spectroscopy (XANES) undertaken at the Australian Synchrotron, identified both oxides to form orthorhombic structures, $CrUO_4$ in space group *Pbcn* and $MnUO_4$ *Imma.* In addition XANES measurements revealed the valance states of the cations as U^{5+} and Cr^{3+} in $CrUO_4$ and U^{6+} and Mn^{2+} in $MnUO_4$, all magnetic ions except U^{6+}.

To further explore the structural and magnetic properties, room and low temperature neutron powder diffraction (NPD) measurements were taken using the echidna diffractometer at the Australian Nuclear Science and Technology Organisation's OPAL nuclear reactor, in addition to *in situ* heat capacity and magnetic susceptibly measurements. It was found that upon cooling below 44 K $CrUO_4$ undergoes an antiferromagnetic transformation where the U^{5+} spins and Cr^{3+} spins anti-align along the b axis. Magnetic susceptibility measurements suggested the onset of a second magnetic anomaly, however further NPD and heat capacity measurements dismissed this as a second magnetic sublattice, rather, it was attributed to crystal field splitting between the two lowest lying Kramer doublets of the U^{5+} ground state. A similar antiferromagnetic transformation was identified in $MnUO_4$ at temperatures below 14 K, forming a pyrolusite type structure, where MnO_6 polyhedra align antiferromagnetically along the b-axis. Further magnetic susceptibility analysis highlighted the peculiar hysteresis behavior in $MnUO_4$.

Abstract

Paper Ref: 68

PO92

Commensurate to incommensurate magnetic phase transition in the multiferroic $YBaCuFeO_5$

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The simultaneous existence of magnetic and ferroelectric ordering is a characteristic of multiferroic materials. The search for new multiferroics is partly motivated by the need for non-volatile random access memories for which the electric polarization (magnetization) is controlled by magnetic field (electric field) or vice versa. The use of such materials would be extremely beneficial for the next generation of electronic devices (mobile phones, tablets etc) whereby compact size constraints are important as well as optimizing reading/writing speeds and power consumption. $YBaCuFeO_5$ has been classified as a type-II multiferroic due to its complex magnetic interactions and low temperature feroelectricity [1]. Two magnetic phase transitions of antiferromagnetic nature have been found near T_{N1} = 450 K and T_{N2} = 170 K [2]. The first represents the ordering of the Fe^{3+} ions into a commensurate antiferromagnetic state, while the second represents the ordering of the Cu^{2+} ions giving an overall incommensurate antiferromagnetic ground state. For the first time, using modified traveling solvent floating zone growth method, we have been able to grow a centimeter-sized, high-quality, single-crystal of $YBaCuFeO_5$. Magnetisation and neutron diffraction results indicate a complex magnetic phase diagram in this material with a strong hysteresis effect and a chiral magnetic ground state.

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Abstract

Paper Ref: 435

PO93

Initial Magnetization Process of Nanocrystalline Nd-Fe-B Permanent Magnets

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Permanent magnets, the most familiar applications of magnetic materials, are often regarded as just strong ferromagnets among physicists, yet they still have some physics to be unveiled inside them such as initial magnetization and magnetic reversal processes. Here, we present the initial magnetization process in nanocrystalline Nd-Fe-B permanent magnets observed with small angle neutron scattering (SANS). We have carried out SANS experiments at QUOKKA, ANSTO with three samples; standard nanocrystalline Nd-Fe-B magnet, Nd-Cu and Pr-Cu infiltrated nanocrystalline Nd-Fe-B magnets. Cu alloys are infiltrated by soaking standard nanocrystalline Nd-Fe-B magnet into the melted alloys to isolate $Nd_2Fe_{14}B$ grains and reduce magnetic interaction between the grains.

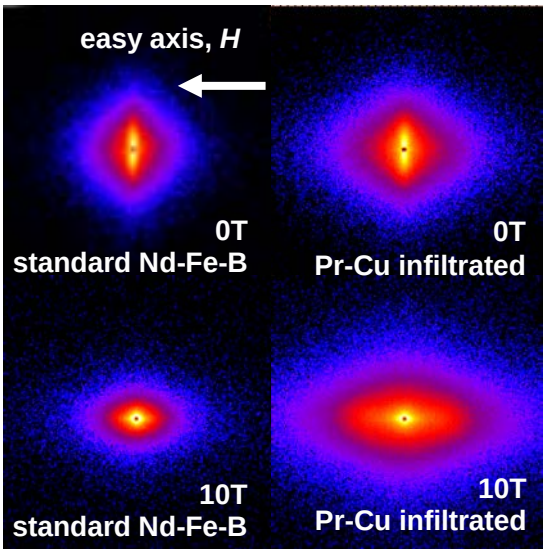

Figure 1: SANS patterns of standard Nd-Fe-B magnet and Pr-Cu infiltrated magnet. The result of Nd-Cu sample is similar with that of Pr-Cu sample.

Two-dimensional SANS patterns for the thermally demagnetized state at 0 T show strong scattering intensity along the direction perpendicular to the magnetization easy axis of the samples (upper part of figure 1). Magnetic field dependence of these intensities for three samples indicates that the origin of the intensities is magnetic scattering from domain walls (not shown here). When the magnetization is saturated at 10 T, magnetic scattering intensity is suppressed and nuclear scattering intensity becomes dominant (lower part of figure 1). Our previous study have shown that infiltrated Cu alloy reduces magnetic interaction between $Nd_2Fe_{14}B$ grains, which results in the increase in the coercivity[1]. Present SANS results show that Cu alloy infiltration changes initial magnetization process by disconnecting domain walls of adjacent $Nd_2Fe_{14}B$ grains. Details will be discussed with magnetic field dependence of SANS intensity for different Q ranges.

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Abstract

Paper Ref: 89

PO94

DETERMINATION OF COMPOSITION AND MAGNETIZATION OF ALLOY THIN FILMS AND INTERFACES USING POLARIZED NEUTRON REFLECTOMETRY

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Polarized neutron reflectometry (PNR) is a powerful technique used to investigate the nuclear and magnetization profiles of surfaces and interfaces in thin films and multilayers. Its high degree of surface (and interface) sensitivity to differences in refractive indices and thereby associated scattering length densities of adjacent layers in a stratified structure, enables determination of composition with depth. Another advantage of this technique used in structural characterization is the detection of small magnetic moments with excellent depth resolution. PNR in specular mode at Dhruva reactor, India, has been used to determine stoichiometry and magnetic structure of: co-sputtered Ni-Zr alloy thin films with varying composition and thermal treatments, ultra thin alloy layers formed due to room temperature diffusion at interfaces of trilayers Si/Co/Si and Si/Ni/Si. A combination of x-ray reflectivity and PNR data analyses was used to generate depth dependent chemical and magnetic structures averaged over lateral dimensions of each sample. This included quantitative estimation of thickness, roughness and density of the constituent and interfacial layers in case of the trilayer systems and also provided an excellent method to obtain stoichiometry of the alloy films.

Ni-Zr alloys although well known to have high glass-forming ability can be tuned to the crystalline phase, which enhances their applicability in various fields such as hydrogen storage and nuclear reactor engineering. The thermodynamic stability of the crystalline phase was studied by systematic annealing for different durations followed by characterization through reflectometry techniques with nanometer resolution. Ferromagnetic/non-magnetic semiconductor layered structures are potential candidates for the development of magnetic random access memory devices. Interfacial mixing across the metal/Si interfaces can occur in these as-deposited samples due to free energy conditions governing the system. This can lead to the formation of interfacial alloys that may or may not be magnetic.

Abstract

Paper Ref: 147

PO95

SUPERCONDUCTIVITY-INDUCED MAGNETIC MODULATION IN A FERROMAGNET THROUGH AN INSULATOR

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Coexistence of ferromagnetic and superconducting orders and their interplay in ferromagnet-superconductor heterostructures is topic of intense research. While it is well known that proximity of a ferromagnet suppresses superconducting order in the superconductor, there exist few studies indicating the proximity of a superconductor suppressing ferromagnetic order in a ferromagnet. Superlattices of complex oxides of superconducting and ferromagnetic, in particular $YBa_2Cu_3O_7$ (YBCO) and $La_{2/3}Ca_{1/3}MnO_3$ (LCMO), has shown a range of interesting effects, like a giant magnetoresistance, a transient photo-induced superconductivity, a magnetic proximity effect. These phenomena are thought to be collective interactions between charge, spin, orbital and lattice degree of freedom in these oxides. Here we show a rare observation of the suppression of ferromagnetic order in a LCMO layer separated from an YBCO layer by a thin insulator, $SrTiO_3$ (STO) below superconducting transition temperature. Polarized neutron reflectivity measurements on LCMO/STO/YBCO trilayer deposited on [001] $SrTiO_3$ single crystal suggest the modulation of magnetization and emergence of a thin magnetic “dead” layer in LCMO adjacent to the insulating layer below its superconducting transition temperature. Further, the dead layer grows in thickness when the insulating layer is thinner. This indicates tunneling of the superconducting order parameter through the insulating $SrTiO_3$ inducing modulation of magnetization in $La_{2/3}Ca_{1/3}MnO_3$.

Abstract

Paper Ref: 430

PO97

CRYSTAL STRUCTURE DETERMINATION OF THE HIGH T_C SUPERCONDUCTOR $Sr_2Ca_2Cu_3O_Y$

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We have studied the structure of the novel high-T_c superconductive cuprate $Sr_2Ca_2Cu_3O_y$ ($0^{(Sr)}$ 223) above and below the T_c. High-T_c superconductive cuprates have a layered crystal structure with CuO_2 superconducting layers, rock salt planes and charge reservoir blocks. However $0^{(Sr)}$223 and $Ba_2Ca_2Cu_3O_y$ ($0^{(Ba)}$223) have unique crystal structures which do not have any charge reservoir blocks and have two rock salt planes facing each other. Water molecules can intercalate between the rock salt planes when the samples are exposed to humid air at room temperature or placed in D_2O. In the $0^{(Ba)}$223 crystal, the T_c value decreases from 126 to 90 K when water molecule intercalation occurs (1), however, the irreversibility field properties increase. Water molecules are neutrally charged so the net charge change in the structure caused by the water molecule intercalation is zero. Therefore it is thought that the field effect caused by the intercalation of water molecules in $0^{(Ba)}$223 crystals affects the charge distribution. This in turn affects the T_c and the irreversibility of the field (2). These changes upon water intercalation have been proposed empirically but no experimental data exists (3). Massive experimental efforts have been made to explore novel high-T_c superconductors by conventional inorganic synthesis routes and there are few elements remaining to modify the crystal structure. It is possible to produce other structures via a combination of other molecules an example of which is H-m223 (1). We have performed high-resolution neutron diffraction to determine the precise coordinates of the oxygen atoms in the crystal structure as well as calculate valences of Cu atoms in the CuO_2 planes via the bond-valence-sum (BVS) approach. Of note is that only 20 mg of sample can be synthesised at a time therefore the results we present are the result of neutron diffraction on 100mg of sample or five equivalent pieces.

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Abstract

Paper Ref: 270

PO98

Spin-reorientation and spin wave excitations of $PrFeO_3$

Dehong Yu[1*], Shixun Cao[2*], Dehui Sun[1], Richard A Mole[1], Guochu Den[1], V O Garlea[3], Barry Winn[3], Mark Hagen[4]

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Rare-earth orthoferrites $RFeO_3$ (R = rare earth), as a series of classical magnets, have been continually attracting great interests because of their complicated phase diagram at low temperature and multiferroic properties appearing in several systems. Due to the strong antiferromagnetic coupling at a wide temperature range, rare earth orthoferrites have recently attracted great interests in ultrafast spin manipulation in the THz regime [1]. This offers promising applications in many areas such as wireless communication, optomagnetic memory, quantum computing, and spintronics technologies based on ultra-fast spin switching using femtosecond laser pulses. The general structure of $RFeO_3$ is an orthorhombically distorted perovskite structure. Interesting magnetic properties arise from the competitive magnetic interactions of Fe–Fe, R–Fe and R–R. At high temperatures the strong Fe–Fe interactions lead to an antiferromagnetic ordering with Néel temperatures ranging from 600 to 700 K [2]. In the range of 100 – 200 K, the competition of the Fe–Fe and R–Fe interactions [3] leads to a so-called spin-reorientation transition of the ordered Fe^{3+} magnetic moments [1]. At even lower temperatures the rare earth ions may become ordered.

We recently focus on the studies of $PrFeO_3$, one of the orthoferrites whose crystal structure and magnetic structure have been studied a while ago [4]. Though the general orthorhombically distorted perovskite crystal structure with Pbnm space-group and G_x magnetic structure has been established, there are still some open questions: At what temperature does spin reorient? How do the spins evolve with temperature along with the spin reorientation process? We have carried out a series of systematic studies on both powder and single crystal PrFeO3. Neutron diffraction on powder samples reveals for the first time that the spin reorientation starts around 10 K and this is consistent with magnetization measurements on single crystals along different crystallographic directions. Crystal field and spin wave excitations at low energy transfer range have also been measured with inelastic neutron scatterings.

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*corresponding authors

Engineering and Industrial

Abstract

Paper Ref: 329

PO99

Dinosaurs, Egyptian Mummies and Early Hominins: Application of Neutron Tomography to Paleontology and Archaeology

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Conventional paleontological examination and preparation of fossilised material requires the chemical or mechanical extraction of fossils from the surrounding matrix. Such preparative methods are conducted blind, i.e. without an exact prior knowledge of the number of location of fossils within the matrix; do not retain a full record of the spatial relationships between material, orientation and articulation, and leads to the destruction of non-skeletal fossilised material such as mineralised soft tissue.

Excessive absorption of X-rays prevents tomographic reconstruction of bulk rock samples, while conventional microtomographic imaging using X-rays requires that fossilised material be extracted from its embedded rock matrix, through either mechanical or chemical methods.

Neutron micro-tomography using the ANSTO DINGO Radiography/Tomography/Imaging Station provides a non-destructive alternative to X-rays for imaging fossils and archaic human material. Compared to X-rays, neutrons have a far greater ability to penetrate dense material, enabling the imaging of fossilised material inside conglomerate rock matrix and avoiding the damaging extraction process. This retains all contextual information provided by the rock matrix, such as spatial relationships between material, orientation and articulation.

This presentation will provide an overview of DINGO's technical capabilities, as well as demonstrate the applicability of DINGO toward the fields of palaeontology, archaeology and cultural heritage using recent investigations of Australian dinosaurs, disease and medical treatment in Greco-Roman Egypt, hominin cave sites and ancient coins as examples.

Abstract

Paper Ref: 308

PO100

NEUTRON RESONANCE TRANSMISSION METHOD AT HOKKAIDO UNIVERSITY NEUTRON SOURCE

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Neutron resonance transmission method is a technique that obtains the information of nuclide by analysis of resonance dips in a transmission spectrum. Since resonances appear at specific energies for each nuclide, we can distinguish the elements and isotopes by use of neutron resonance. In addition, neutron resonance transmission is used to estimate atomic density with high sensitivity because of its large cross section. At present, neutron resonance transmission method is proposed as a non-destructive analysis for various fields, for examples, industrial materials, archaeological study and nuclear material quantification.

Hokkaido University neutron source (HUNS) is a pulsed neutron source which is based on an electron accelerator. Neutron resonance transmission experiments can be performed at thermal neutron beam-line in HUNS. To analyze resonance dips quantitatively, the pulse function should be reproduced since the dips are expressed by the convolution of the theoretical resonance and the pulse function. Therefore, we have carried out simulation calculations of the pulse function of HUNS and then found that the Ikeda-Carpenter function [1] was the function expressing the pulsed function of HUNS. We have developed the neutron resonance analysis system based on the resonance analysis code “REFIT” [2].

To estimate the quality of densitometry by neutron resonance transmission at thermal neutron beam line in HUNS, we measured a neutron transmission spectrum of some metal foils containing resonant element (Tantalum, gold and indium). We analyzed few-eV resonance dips by our analysis system and succeeded in estimating the density of samples within an error of about 5%.

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Abstract

Paper Ref: 90

PO101

Visualization of magnetic field direction in an electric motor using polarized pulsed neutron imaging

Kosuke Hiroi[1], Takenao Shinohara[1], Hirotoshi Hayashida[2], Joseph D. Parker[2], Kenichi Oikawa[1], Masahide Harada[1], Tetsuya Kai[1], Masatoshi Arai[1]

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A magnetic field imaging technique using polarized pulsed neutron enables us to obtain information about field distribution in a bulk sample and a surrounding space. Recently, we have been developing an imaging technique for quantitative evaluation of magnetic field [1] in industrial equipment, e.g. an electric motor and an electric transformer, etc. To reduce the power loss of these products, it is important to quantitatively evaluate not only strength of an internal field but also directional distribution. In this study, we attempt to evaluate direction of a field in the gap between the rotor and the stator of a model electric motor.

Magnetic imaging experiment was performed at BL10 “NOBORU” in Materials and Life science experimental Facility (MLF) of J-PARC. In this experiment, we changed the quantized axis of a neutron spin using spin-rotators to obtain polarization images with various neutron spin directions. Polarization degree P of polarized neutron that passed through a magnetic field can be expressed as follows,

$$P / P_0 = 1 - \left(1 - < \cos\varphi >\right) \cdot \sin^2\theta = \cos^2\theta$$

where P_0 is polarization of a incident beam, φ is a Larmor precession angle and θ is an angle between the magnetic field and the quantized axis of a neutron spin. While the oscillation term, which depends on the angle φ, was smeared out due to the coarse wavelength resolution, information about field direction, which corresponds with θ, was still observable in the polarization image taken at each spin-direction. Fig.1 shows the result of field-direction distribution in the model motor. The field in the left and right side of the rotor was aligned along the Y direction and the top and bottom side was along the X direction. This tendency almost agrees with that expected from the position of magnetic poles in the rotor. In the presentation, we will discuss details of this analytic method and compare the experimental results with computational simulation.

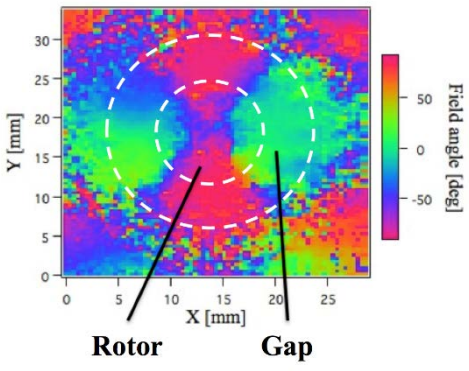

Fig.1 Distribution image of the field angle from Y-axis in the model motor

We would like to appreciate Mr. Takao Imagawa of Hitachi, Ltd. providing the model motor. This work was supported by Photon and Quantum Basic Research Coordinated Development Program from the Ministry of Education, Culture, Sports, Science and Technology, Japan.

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Abstract

Paper Ref: 438

PO102

Status of Resonance Absorption Imaging in RADEN, J-PARC

Tetsuya Kai[1], Yoshihiro Matsumoto[2], Joseph D Parker[2], Mariko Segawa[1], Takenao Shinohara[1], Kosuke Hiroi[1], Yuhua Su[1], Shuoyuan Zhang[2], Hirotoshi Hayashida[2], Kenichi Oikawa[1], Yoshiaki Kiyanagi[3]

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The first neutron beam was provided to the energy-resolved neutron imaging system, RADEN, of J-PARC in November 2014. Neutron resonance absorption imaging is one of the techniques to take full advantage of pulsed neutron characteristics. Test/ demonstration measurements of the resonance absorption imaging carried out so far will be presented.

Neutron energy-dependent transmission rate of 1-euro and 2-euro coins and a copper foil of 1 mm in thickness were measured by a gas electron multiplication (GEM) neutron detector. The enhanced images of copper and zinc were clearly obtained at resonance energies of these elements. For a demonstration of practical applications, a commercially available lithium-ion battery (110 x 92 x 14 mm) was also measured. Spatial distributions of cobalt, manganese and tungsten were obtained even though no structure inside the battery was visualized by conventional neutron radiography due to the absorption and scattering by lithium and hydrogenous material.

The resonance absorption imaging was also performed by using a high-speed camera which accumulates time-of-flight dependent digital images coupled with a neutron color image intensifier. The enhanced images of cobalt, gold and cadmium foils were successfully obtained without the loss of measurement time associated with data transfer from camera to computer. Further feasibility studies of this system are to be performed.

We carried out a test measurement of neutron thermometry which was an important application of the neutron resonance absorption imaging. Neutron resonances of copper, manganese and platinum were measured at different temperatures by using the GEM neutron detector. Doppler broadenings of the resonances were recognized. Such basic parameters of this technique as reliability, required time and spatial resolution are to be studied.

This work was partially supported by a Grant-in-Aid for Scientific Research (S) from Japan Society for the Promotion of Science (No. 23226018) and the “Photon and Quantum Basic Research Coordinated Development Program” from the Ministry of Education, Culture, Sports, Science and Technology (MEXT).

Abstract

Paper Ref: 183

PO103

Remote Temperature Measurement by Neutron Resonance Absorption Spectroscopy using Plural Nuclides

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Neutron resonance absorption spectroscopy (N-RAS) is a method which measures the dynamics of atoms by analyzing the Doppler broadening of their resonance absorption peaks. The method enables us to investigate motions of a particular element, because of the resonance energies are peculiar to the nuclide. Such information about dynamics can be translated to the parameter related to its temperature, which called effective temperature, T_{eff}. T_{eff} has a trend to coincide with the real temperature of the sample above sufficiently high temperature. In the past studies, we tried to measure the temperature distribution inside a sample object with computer tomography (CT) method. For the CT reconstruction, we need the position and direction depend spectrum series, that means a long measurement time is required. In this study, we have simplified the procedure of obtaining the temperature distribution. We set each nuclide to the respective positions where take the temperature information. We can obtain the each temperature by analyzing the resonance absorption peaks of nuclides at each position.

The measurements were carried on the neutron resonance absorption spectrometer at Hokkaido Univerity neutron source (HUNS) in Japan. The sample was made with a Ta foil, Ag foil and Sm_2O_3 powder. Each sample had its thickness 0.05 mm, and the area 100 mm by 100 mm. The sample was set in a furnace to hold its temperature. The temperature range of the experiment was 298 to 543 K.

The Doppler broadening analysis of resonance absorption peaks for each nuclide was carried out with the convolution of a neutron pulse shape which was determined by the

Monte Carlo simulation of HUNS thermal neutron source. The experimental temperature results analyzed from each nuclide the showed a good agreement with the kept temperature. In the presentation, the detailed experimental conditions and analysis procedure will explain.

Abstract

Paper Ref: 412

PO104

Evaluation of crystal structural change of the carbon anode in a lithium-ion battery product by neutron transmission Bragg-edge analysis

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Non-destructive methods to evaluate crystalline structure of functional materials in a product, such as a graphite anode contained in a lithium-ion battery (LIB), are needed for in-situ test of the product. While charging a LIB electrically, the crystal lattice spacing of graphite {002} becomes larger than the discharged one, because the lithium ions intercalate into the graphite layers of the anode. The lattice spacing of graphite {002} can be related to the charge level of the LIB. Using pulsed neutron Bragg-edge imaging, which is one of methods to evaluate microstructure nondestructively, we may obtain the spatial distribution of charge level in the LIB product. However, it is not clear that Bragg-edge analysis can be applied to the LIB product, which consist of some materials having large neutron cross sections. Therefore, at first we investigated feasibility to measure the Bragg-edge spectrum and second to analyze lattice spacing of the graphite anode by this method to evaluate the charge level of the LIB product for future spatial dependent measurements.

We measured neutron transmission spectra of the LIB in different charge levels at Hokkaido University Neutron Source (HUNS). We were able to confirm that Bragg-edge of graphite {002} in the LIB product was observed by this method. We analyzed the Bragg-edge of the carbon anode in the neutron transmission spectrum of the LIB product, and evaluated the lattice spacing of the graphite anode, depending on each charge level.

As a result, it was found that the lattice spacing of graphite {002} in the discharged level, 0.338 nm, was smaller than that of the half-charged level, 0.355 nm and that of the full-charged level, 0.353 nm. These structural changes of graphite arise from the stage structure of graphite intercalation compounds, and we succeeded to observe the transition of graphitic stage 4 to stage 2. Moreover, it was found that the dispersion of the lattice spacing of graphite {002} in the half-charged level was larger than the others. That is because the LIB product in the half-charged level contains several stages of graphite. It is evident from these results that we can measure the spatial distribution of lattice spacing, which corresponds to charge level, in the LIB product by pulsed neutron Bragg-edge imaging.

Abstract

Paper Ref: 237

PO105

BRAGG INSTITUTE INDUSTRIAL LIAISON OFFICE

Dr Anna Paradowska [1], Dr Nicholas Hoye [1,2]

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[2] *University of Wollongong, Australia*

The OPAL research reactor at ANSTO has a number of neutron instruments available for science and engineering applications. The instruments have a unique non-destructive ability to determine critical aspects of a wide variety of material systems. Over time, the Bragg Institute has built an exceptional body of skills, experience and technical expertise, which is now on offer to support industrial research and development.

The Bragg Institute Industrial Liaison Office (ILO) was set up in April 2014 to promote the use of the Bragg Institute's facilities for applied industrial research and to manage technology transfer.

This presentation will focus on the challenges and highlights of first year of operation.

Abstract

Paper Ref: 238

PO106

RESIDUAL STRESSES AND EFFECTS ON STRUCTURAL INTEGRITY IN GAS PIPELINE HOT-TAP JUNCTION

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[2] *ANSTO*
[3] *University of Wollongong*

The residual stresses were measured in a thick spherical tee, both in as-built and in post-weld heat treated (PWHT) condition. The measurements were made by using non-destructive neutron diffraction technique at the OPAL reactor.

As-built, high residual stresses were found at weld intersections, particularly at the weld toes and root, but were less than code-based estimates. The residual stresses were significantly reduced after PWHT, and are also lower than code-based estimates for PWHT welds.

The critical defect size was estimated for both stress states. The use of measured residual stress values increased the critical crack depth.

Abstract

Paper Ref: 186

PO107

Design of Magnetic Field Imaging using Compact Neutron Source

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Neutrons may be utilized for many applications that is feasible with compact neutron source compared with high flux neutron sources such as research reactors and spallation neutron sources. We are expecting that non-destructive inspection, imaging(Structural analysis), small angle neutron scattering, re-flectometor and so on may be able to be implemented with our neutron source. Among them, magnetic structure was analyzed by interaction between the nuclear magnetic moment μ of neutron and a magnetic field inside and/or around the magnetic sample since neutron has also a spin of ±1/2. The strength of the interaction depends not only on the size of the electronic magnetic moments, but also on their relative orientations. Therefore polarized neutron scattering techniques are very powerful tools in the investigation of magnetic structure. In this paper, we have studied the feasibility of magnetic field imaging based on the compact neutron source. We have done a basic conceptual design of magnetic field imaging with the neutron source. The designed system consist of a super-mirror polarizer(neutron spin filter), a spin flipper, solenoid coil, a collimator, an analyser, and a detector. A Super-mirror polarizer based on thin silicon wafers coated one side with Si-FeCo polarizing super-mirrors and the other side with Gd. A spin flipper was designed by adiabatic fast-passage (AFP) method and was simulated maxwell 3D program. The solenoid coil was designed 1mT, 3mT, 5mT, 7mT and 10mT on plastic bobbin of 15 mm x 10 cm for measurement of magnetic field. We plan to conduct experiments with continuous neutron source of HANARO or J-PARC and will produce data for the detailed design of the system suitable for compact neutron source.

Abstract

Paper Ref: 437

PO108

Measurement of the neutron imaging Bragg-edge broadening distribution depending on the neutron transmission direction in a quenched steel rod.

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The pulsed neutron imaging and Bragg-edge analysis offer a non-destructive method to reveal internal crystallographic structure distribution in a bulk metal. We are capable of quantitative evaluation of microstructural information such as crystal lattice plane spacing, crystalline orientation, crystallite size and crystalline phase by analyzing the Bragg-edges [1] with RITS program [2]. Additionally, We are promoting the analysis of Bragg-edge broadening that is include the microstrain information (Intergranular strain and dislocation density) and the small crystallite size information as is the case with the peak broadening of Bragg diffraction [3].

In this study, we will introduce the neutron imaging measurement of a quenched steel rod, and the Bragg-edge broadening distribution depending on the neutron transmission direction. Neutron transmission measurement of a steel rod was carried out using MCP neutron detector at BL10 / MLF / J-PARC. A clear broadening was observed on the outside of rod, and broadening distribution has been overlapped in hardness distribution, due to the martensite structure with the high dislocation density, or with the small crystallite size. However, the Bragg edge depends on direction of a material structure, and it is considered that edge broadening has dependence of the neutron transmission direction, according to the situation of the micro-strain or of the small crystallite size. Therefore, we compared the rod axis direction and the vertical axis of Bragg-edge broadening in this steel rod to study the direction dependency. They were examined for the homogeneity (or the inhomogeneity) of the material structure distribution relative to the transmission direction. Such a direction dependency of structure distribution would be useful for CT technology of structure information in future.

This work was supported by a Grant-in-Aid for Scientific Research (S) from Japan Society for the Promotion of Science (No. 23226018).

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Abstract

Paper Ref: 129

PO109

Time-of-Flight Neutron Bragg-edge Imaging for Microstructure of Bent Steel Plates

Yuhua Su[1], Kenichi Oikawa[1], Masahide Harada[1], Takenao Shinohara[1], Tetsuya Kai[1], Shuoyuan Zhang[2], Hirotaka Sato[3], Yoshinori Shiota[4], Yo Tomota[5]

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Bending is a common and vital process in manufacturing industry, which simultaneously includes tension and compression deformation. Recently, the Bragg-edge transmission imaging using pulsed neutron has been developed to visualize the spatial distributions of crystalline structure in relatively thicker materials. We applied this technique to clarify the distribution of microstructure in the bent steel plates. Neutron imaging experiment on four kinds of bent steel specimens was carried out at NOBORU, BL10 of MLF at J-PARC. A Gas Electron Multiplier (GEM) detector was used for two-dimensional transmission spectra using time-of-flight method. 2D distribution of lattice strain, crystalline phase ratio, crystallite size and texture were determined by Bragg-edge spectrum analysis using RITS (Rietveld Imaging of Transmission Spectra) code [1]. The local texture and microstructure of the specimens were also characterized by electron backscatter diffraction (EBSD) analysis.

Figure 1(a) shows a measured pattern of a ferrite (α-Fe, BCC structure) steel. Figure 1(b) shows three transmission spectra extracted from the indicated regions in Fig. 1(a). These Bragg-edge patterns show clear difference between the compression and tension regions due to the change of texture. The detailed results obtained by the RITS code will be presented at the conference.

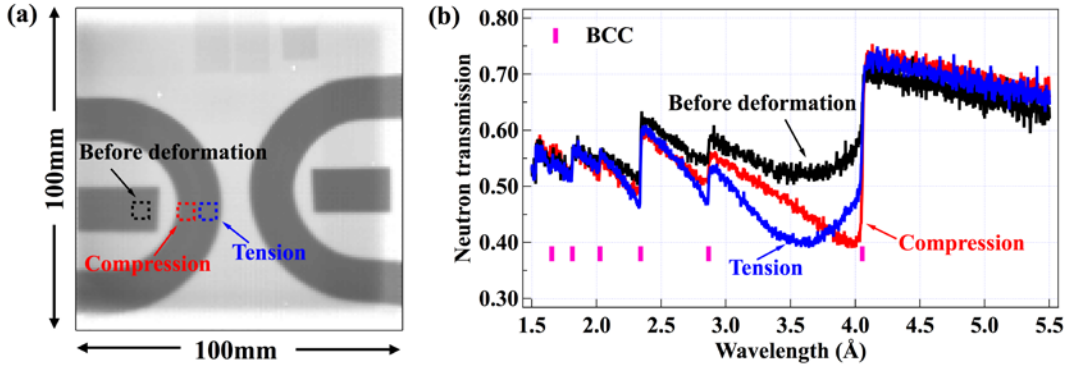

Fig. 1. Neutron imaging of a ferrite steel: (a) 2D image; (b) Bragg-edge spectra of each region indicated in (a).

This work was supported by Photon and Quantum Basic Research Coordinated Development Program from the Ministry of Education, Culture, Sports, Science and Technology, Japan.

[1] H. Sato et al.: Physics Procedia. 43, 186 (2013).

Abstract

Paper Ref: 407

PO110

MPISI: New material science diffractometer at SAFARI-1

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A world-class neutron strain scanner and texture instrument has been established at the SAFARI-1 research reactor. This new generation instrument has been developed based on experiences gained from the use of the previous instrument, as well as from the use of a number of instruments at leading international centres and positive information exchange with the respective instrument scientists.

MPISI, Materials Probe for Internal Strain Investigations (Zulu name for Spotted Hyena), has been completely modernised by addressing all aspects of the neutron beam conditioning, sample handling and detection. The diffractometer is designed to be equally applied to texture or residual stress analyses. The system compromises a double focused Popovi type bent Si-multiwafer monochromator, sturdy high-precision diffractometer with integrated sample manipulation stages, adjustable apertures interchangeable with radial collimators, and a 300 x 300 mm^2 multiwire detector. The data acquisition and control system was sourced from ANSTO, configured and implemented to comply with site-specific requirements. Amongst others it is equipped with a Wi-Fi based remote hand-held instrument control module, running on Android, to aid with sample setups. In addition a number of advanced procedures are being developed to improve the overall sample setup times and adapted data acquisition to improve the overall instrument efficiency.

The instrument has been benchmarked against the VAMAS shrink-fit ring and plug specimen that confirmed its sensitivity and accuracy in capturing the essential features presented by this sample [1]. The instrument is now available for routine operation and acess as a User Facility. We shall present details of instrument performance and results from the first projects.

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Abstract

Paper Ref: 128

PO111

TEXTURE DEVELOPMENT IN POTASSIUM STRONTIUM NIOBATE ($KSr_2Nb_5O_{15}$) PIEZOCERAMICS PREPARED BY TEMPLATED GRAIN GROWTH

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Induction of crystallographic texture via the templated grain growth (TGG) approach has been extensively used to improve the electromechanical response of ferroelectric materials. Understanding the bulk texture evolution during the TGG process is of critical importance for optimising the process and facilitating the preparation of highly textured ceramics. In this work, the texture development during the TGG of $KSr_2Nb_5O_{15}$ (KSN) lead-free piezoceramics was investigated using *in situ* neutron diffraction. Rietveld texture refinements were performed to quantify the texture evolution of the green samples produced by tape casting during the high-temperature sintering. The samples in the green state were found to present a strong (00*l*) fiber texture having the pole maxima of 8.2 multiples of the random distribution (mrd) along the tape casting direction. The formation of this fiber texture before sintering was attributed to the preferred alignment of acicular KSN templates during tape casting. The initial fiber texture was only slightly enhanced before the temperature increased to 1250 °C from room temperature at a ramping rate of 4 °C/min. At 1250 °C, the magnitude of (00*l*) texture was rapidly increased and reached a maximum of 12.4 mrd within 10 min. The observation of this rapid texture development indicates the epitaxial grain growth of the KSN matrix powders on the template particles was efficiently accomplished at 1250 °C. This work improves the understanding of the kinetics of the texture development in the KSN system during the TGG process and provides important guidelines for the further development of textured lead-free electroceramic materials.

Materials Science and Chemistry

Abstract

Paper Ref: 161

PO112

SIMULTANEOUS MEASUREMENTS OF AE SIGNAL AND NEUTRON DIFFRACTION FOR ANALYSIS OF STRAIN IN ROCK MATERIALS

Dr Jun Abe [1], Dr Kotaro Sekine [2], Dr Stefanus Harjo [3], Dr Wu Gong [3], Dr Kazuya Aizawa [3]

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In order to utilize crustal environment as storage space (e.g. the geological isolation of high-level nuclear waste, CO_2 safety storage in underground) and to exploit underground resources (e.g. metals, hydrocarbons and geothermal energy), understanding of the mechanical behavior of rock materials is essential.

Commonly, strain measurements of rock specimens have been performed using strain gauge. Recently, diffraction techniques with neutrons for investigating strain in engineering materials have been developed and is being applied to the rock materials. To investigate deformation mechanism of rock materials, we have developed a measurement system simultaneously obtaining neutron diffraction pattern and AE sinals of a rock specimen under uni-axial compressive loading condition. Acoustic emission (AE) signal measurement is a useful tool for identifying crack initiation and propagation, phase transition, and slip. The combination of these techniques will provide us new insights into strain accumulation and fracturing mechanisms in the rock materials.

Two types of rock materials were examined in this study: sandstone and carbonate rock. The former is mainly composed of quartz (SiO_2), and the latter, a carbonate rock, is mainly composed of calcite ($CaCO_3$) with minor apatite. Both rock specimens were prepared in cylindrical shape with 25 mm diameter and 50 mm length. Sandstone was compressed up to 35.6 MPa with two-cycle compression. Carbonate rock specimen was compressed until the specimen fractured at 16.4 MPa. In situ neutron diffraction measurements were performed using the Engineering Materials Diffractometer “TAKUMI” in J-PARC.

Macroscopic strain (measured by strain gauge) was greater than lattice strain (measure by neutron diffraction), and AE signals that would be generated by grain slip and pore collapse in rock specimens were detected. Characteristics of AE signals, such as amplitude and frequency, depend on rock type. This difference between the two rock specimens suggests that the dominant deformation mechanism is a function of rock type. The combination of neutron diffraction and AE signal measurements is a promising tool for investigating the deformation mechanisms of rock materials.

Abstract

Paper Ref: 83

PO113

The Thermal Expansion of Li and Na intercalated ZrW_2O_8

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Thermal expansion has been associated with many factors limiting the functionality and lifetime of various devices. Zirconium Tungsten Oxide, ZrW_2O_8, is known for its isotropic Negative Thermal Expansion (NTE) from 0.3 to 1050 K. In this study we report a novel approach to controlling the thermal expansion of this material. Li- and Na-ion batteries were constructed with ZrW_2O_8 used as an anode into which Li and Na intercalates. The main advantage of using batteries is the ability to precisely control the amount of Li and Na that is inserted. Electrochemical analysis shows that ZrW_2O_8 exhibits higher first discharge capacity of 463 mAh/g as an anode for Li-ion batteries compared to 114 mAh/g in Na-ion batteries. In principle, this suggests that more Li can intercalate into ZrW_2O_8 than Na. *In situ* synchrotron powder X-ray diffraction (XRD) data shows that Li and Na intercalates into ZrW_2O_8 as the batteries are discharged. While ZrW_2O_8 maintains its stability (or structure) as Na was inserted to the maximum capacity, it breaks down into ZrO_2 and WO_3 as Li is inserted. Interestingly, in both cases no shifts were observed in the ZrW_2O_8 reflection positions as Li and Na are inserted which may suggest that ZrW_2O_8 is a zero-strain material for Li and Na insertion.

Neutron powder diffraction (NPD) has the advantage that it is not greatly influenced by the large difference in the atomic number of the elements present in Li/Na-intercalated ZrW_2O_8 compared to XRD. *In situ* NPD will be used to find out the mechanisms through which Li and Na intercalates into ZrW_2O_8. Furthermore, *in situ* variable temperature NPD will be used to quantify the effect of the amount of intercalated Li and Na into ZrW_2O_8 on its thermal expansion and whether that may result in a zero thermal expansion material.

Abstract

Paper Ref: 271

PO114

Exploring the Properties of Mixed Cobalt/Iron/Manganese-Tin Clusters

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Layered transition metal oxide materials containing third row elements are popular candidates for the study of strongly correlated-electron system materials with novel multiferroic properties. The parent compound for this study, $Fe_4Si_2Sn_7O_{16}$ [1], provides a novel situation in oxide compounds. It is described as a composite of intermetallic ($FeSn_6$) oxide clusters and Sn-doped olivine-type layers within one structure. SiO_4 tetrahedra separate these layers by about 7 Å resulting in two nearly perfectly 2D oxide systems in the one structure. Despite large amounts of high spin Fe^{2+} in the structure, strong spin-orbit coupling prevents magnetic ordering above 1.45K. In this presentation we will describe the replacement of Fe with Co and/or Mn and the resultant changes of electronic interactions between the two layers. Magnetic susceptibility measurements show that these substitutions can lower geometric frustration of the metal oxide layer allowing the layer to magnetically order albeit at low temperatures. Refinements against neutron powder diffraction data show that the MSn_6 octahedra in these materials contain both Fe and Co. Room temperature ^{57}Fe-Mössbauer (Fig. 1) and x-ray absorption spectroscopy experiments confirm our proposed model and show that Mn has an overwhelmingly strong preference to occupy the olivine-type layers. We will discuss the crystal-chemistry behind these features and in the context of their effects on the magnetic and electronic properties, with particular reference to spin-orbit coupling.

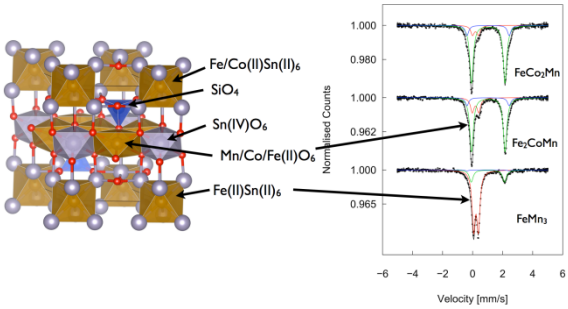

Figure 1. Crystal structure (left) and ^{57}Fe-Mössbauer spectra of $Fe_{4-x-y}Mn_xCo_ySi_2Sn_7O_{16}$.

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Abstract

Paper Ref: 17

PO115

Thermal Expansion of Monoclinic Natrojarosite: A Combined Time-of-Flight Neutron and Synchrotron Powder Diffraction Study

Helen E. A. Brand[1], Nicola V. Y. Scarlett[2], Ian E. Grey[2] and Kevin S. Knight[3,4]

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Jarosites and related minerals are of great interest to a range of mineral processing and research applications. In some industrial settings jarosite formation is encouraged; for example to aid the removal of iron species from solutions in hydrometallurgical processes. In other environments such as bioleaching, jarosite formation can hinder the process by creating a kinetic barrier, in the form of a passivation layer, to the desired reaction. There has been a recent resurgence in interest in jarosite minerals since their detection on Mars by the MER rover Opportunity. In this context, the presence of jarosite has been recognised as a likely indicator of the presence of water on Mars in the past. It is hoped that study of their formation mechanisms, stability and thermoelastic properties will provide insight into the environmental history of Mars as well as informing terrestrial industrial concerns. To this end we are engaged in a program to study jarosites, their formation and stability behaviour, over a range of conditions.

This contribution describes *in situ* powder diffraction experiments to determine the thermal expansion of a deuterated natrojarosite. Data were collected on the HRPD beamline at the ISIS spallation source where the natrojarosite sample was heated from 10–700K, and at the powder diffraction beamline at the Australian synchrotron where the sample was heated from 80-700K.

Analysis of the lattice parameter variation with temperature shows that all cell-edges increase smoothly to ~500 K where there is a discontinuity. This discontinuity represents the initially non-stoichiometric monoclinic jarosite converting to a stoichiometric, rhombohedral phase, shortly after which $FeOHSO_4$ peaks become visible. Details of the combined neutron-synchrotron analysis approach will be discussed.

Abstract

Paper Ref: 111

PO116

Effect of removal method of a boron rich layer on Al_2O_3-passivated boron emitters for solar cell application

Joo Yeon Choi[1], Young Joon Cho[1], and **Hyo Sik Chang[1]**

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We reported that low boron emitter saturation current achieved with Al_2O_3/SiN stack passivation using conventional BBr_3 diffusion process. N-type Si wafers are less sensitive to impurities that are usually present in silicon feedstock and do not suffer from LID (Light Induced Degradation) caused by the simultaneous presence of boron and oxygen in the wafers. Consequently, n-type wafers with a high electrical quality can obtain high solar cell efficiency potential. Therefore, we investigated that the change of the carrier lifetime and open circuit voltage according to removing method of a boron rich layer (BRL) that is formed during BBr_3 diffusion process. We used in-situ oxidation method during BBr_3 diffusion process and chemical etching method to remove BRL. In this study, we report that the removal method of BRL had a strong effect on passivation performance for boron emitter passivated with Al_2O_3/SiN stacks. And the film thickness and interference were examined with a neutron scattering.

Abstract

Paper Ref: 411

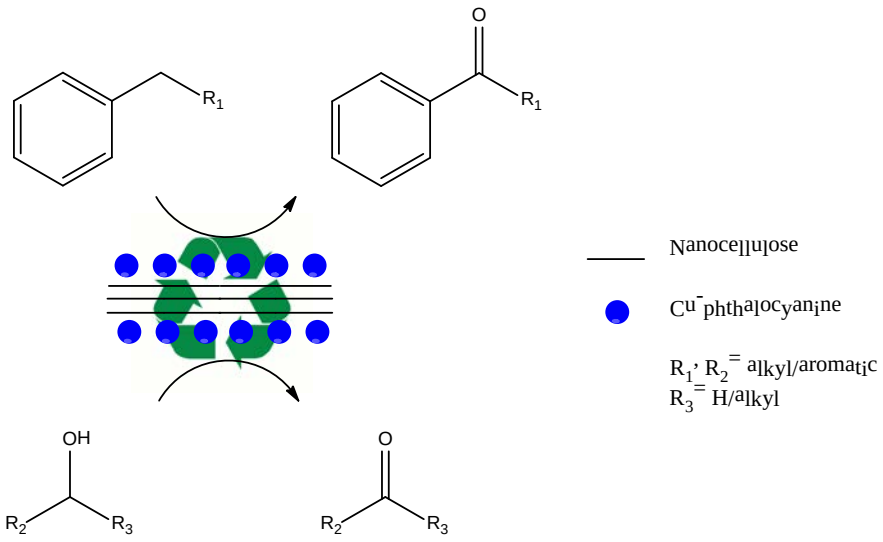
PO117

Nanocrystalline cellulose grafted phthalocyanine: a heterogeneous catalyst for selective aerobic oxidation of alcohols and alkyl arenes at room temperature in green solvent

Prashant Chauhan[1] and Ning Yan[1]

[1] *University of Toronto, Canada*

The nano-crystalline form of cellulose, "**NCC"**, being world's abundant, biodegradable and cost effective biopolymer on earth; has recently drawn acute interest in different applications due to its outstanding physical and chemical properties.[1] Wherein, recently some notable examples in the different fields have been explored, such as : drug delivery, photodynamic therapy, electo-magnetic, sensors etc. Recently, we have demonstrated A Cu-tetrasulfonate-phthalocyanine was conjugated to cationic cellulose nanocrystals which serves as heterogeneous based catalyst for aerobic oxidation of alcohols and alkyl arenes at room temperature in aqueous medium.[2] The major advantages of this bio- nano- catalyst are as follows: inexpensive nanocellulose as the solid support, ease in synthetic process of catalyst, heterogeneous nature of the catalyst, in water as green solvent reaction condition, single step workup process, high catalytic activity at room temperature, and no need for any base requirement. The catalyst also displayed ease in recovery and reusability features with no significant loss in activity.



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Abstract

Paper Ref: 264

PO118

Studying Structuring in Organic Solar Cell Casting Solutions using SANS

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Organic solar cells (OSCs) have received much attention in recent years due to their potential to provide a flexible and lightweight power source. The light-absorbing active layers of OSCs often comprise a blend of polymer and fullerene deposited from solution, allowing integration with printing techniques such as roll-to-roll fabrication. The efficiency of the resulting OSCs is intimately related to the polymer/fullerene distribution in the thin film active layers, which has received much attention in the literature.[1] More recently the structures formed in solution prior to film deposition have been recognised to be important. The molecular weight and crystallinity of the polymer can have dramatic effects on the rheological properties of the processing solutions and this has also been found to influence the efficiency of resulting devices.[2] Thin films have also been prepared from polymer/fullerene nanoparticles in eco-friendly solvent systems such as alcohols or water.[3]

In this presentation we will report our recent results from studies on solution structuring in OSC casting solutions containing conjugated polymers blended with fullerenes. Small angle neutron scattering experiments were performed on QUOKKA at Australia's OPAL research reactor utilising solvent contrast the complementary neutron scattering length densities (SLDs) of the polymers and fullerenes to highlight their structures.[4] Polymer clustering in nominally good solvents will be related to casting solution rheology and the efficiency of operational OSC devices. The internal structuring of polymer/fullerene nanoparticles prepared in alcoholic solvents without surfactant stabilisers will also be shown to be different to their stabilised counterparts.

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Abstract

Paper Ref: 203

PO119

STRUCTURES OF VITREOUS AND PARTIALLY DEVITRIFIED NEODYMIUM ALUMINO-PHOSPHATE GLASS

Mr. Benedict Soares [1], **Professor Erwin Desa [1]**, Dr. Siva Krishna [2], Mr. A B Shinde [2]

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The structure of a glass is determined by not only its composition but also by the particular thermal history that is responsible for its formation. In the process of cooling rapidly from the super-cooled state to form a glass, certain structural correlations are set up in the melt as it freezes into the vitreous state. The resultant continuous random network may be generated from quasi-crystalline regions in which local crystal-like correlations are progressively broken until a random structure is achieved over a correlation length – typically a few Angstroms (1).

In order to choose a viable quasi-crystalline region from which the random network can be generated, we report here an attempt to identify the preferred crystalline structures that are generated within a glass when it de-vitrifies. The objective of these measurements was to quantify the crystalline structures formed on de-vitrification and use these in a quasi-crystalline model of the glass.

The system chosen for these studies was an alumino-phosphate host glass with neodymium oxide added to the host at the level of 20 mole % of the additive. The glass transition temperature was measured to be about 850 ^{0}C. In order to obtain the devitrified sample, the glass was held at temperatures of 1050 ^{0}C and 950 ^{0}C. The glass and devitrified samples were examined using the High Q neutron diffractometer (0.3 Å^{-1} to 15 Å^{-1}) at the Dhruva Reactor of B.A.R.C., Mumbai.

The neutron diffraction pattern of the Nd phosphate glass sample have the main features expected of a phosphate glass. The diffraction patterns of the devitrified samples clearly demonstrate both crystalline and glassy character in that well-defined crystalline reflections are superimposed on the background of the diffraction pattern of the glass. The real space correlation functions show the devitrified samples as having the same local nearest-neighbor correlations as the glass. Higher correlations in the devitrified samples pertaining to next-nearest neighbor distances were more pronounced than in the glass. The generation of the appropriate quasi-crystalline model for this particular doped phosphate glass is reported here.

1. Wright A.C., Advances in Structure Research (1974)

Abstract

Paper Ref: 65

PO120

Study of Structural and Magnetic Properties of $BaFe_{1-x}Bi_xO_{3-\delta}$

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A series of tetragonal perovskite $BaFe_{1-x}Bi_xO_{3-\delta}$ ($0.09 \leq x \leq 0.35$) has been synthesized by traditional solid state method. A combinational use of powder X-ray, synchrotron X-ray and neutron diffractions results that they all crystallize in *P4/mmm* space group (with *a* = 4.0759(1) Á, *c* = 4.0782(1) Á for x=0.15). The magnetic susceptibility measurements showed that the antiferromagnetic transition for these materials occurs from 64 to 50 K. Neutron powder diffraction data collected at 3 K, 55 K and 300 K for BFB15 ($BaFe_{0.85}Bi_{0.15}O_{3-\delta}$) to understand the magnetic properties of $BaFe_{1-x}Bi_xO_{3-\delta}$. It is found that the main reflections are very similar and can be fitted well with the same structural model used for whole series at room temperature. However, with a fine comparison among these neutron diffraction patterns, some little additional satellite peaks are observed in range of 35-40 degree 2θ value for neutron diffraction data at 3 K and 55 K as shown in Figure 2(c and d). These additional reflections are due to incommensurate magnetic ordering of Fe ions with q = (0.195, 0.150, 0.000) and μ_{eff} = (3.30, 4.40, 0.00)μ_B at 3K and q = (0.195, 0.153, 0.000) and μ_{eff} = (2.69, 3.95, 0.00)μ_B at 55K in the space group *P4/mmm*. These reflections are similar to that of Cubic $BaFeO_3$[1] at low temperature and can be attributed to antiferromagnetic ordering of Fe ions. The corresponding Rietveld refinement data at 3K and 55K are listed in Figure 2.

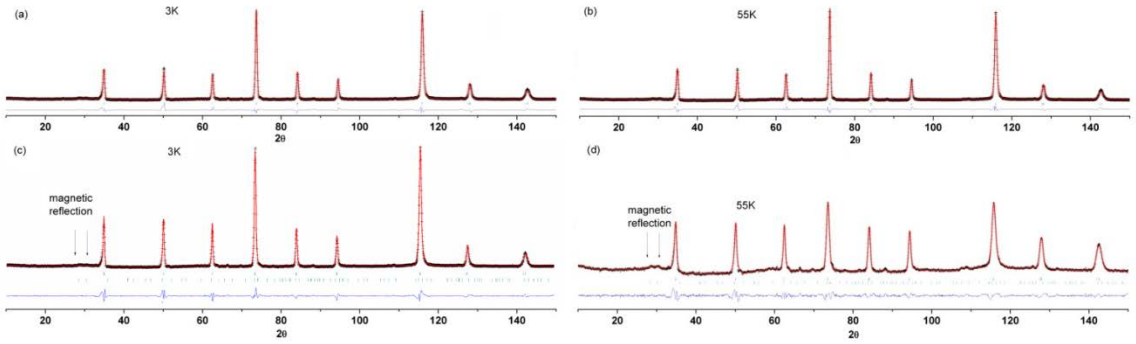

Figure 2: Rietveld refinement of the neutron data of BFB15 ($BaFe_{0.85}Bi_{0.15}O_3$) at 3K with nuclear structure (a), at 55K with nuclear structure (b), at 3K with both nuclear and magnetic structure (c) and at 55K with both nuclear and magnetic structure (d).

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Abstract

Paper Ref: 97

PO121

$Li_7La_3Zr_2O_{12}$ AS AN ELECTROLYTE FOR NEXT-GENERATION SOLID-STATE BATTERIES

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Rechargeable lithium-ion batteries already appear ubiquitously throughout many portable electronic devices, where high power density, high specific capacity, and reliable charge/discharge characteristics are required. However, conventional lithium-ion batteries rely on a liquid electrolyte, which not only presents a possible safety hazard, but also restricts miniaturisation of batteries for smaller devices. All solid-state batteries are able to overcome these drawbacks and also offer higher capacities, higher operating temperatures, and the ability to withstand shock or vibrations during operation. Solid-state electrolytes also make possible the development of significantly smaller thin-film batteries, which enables the production of new microelectronics, such as implantable medical devices or microelectromechanical systems.

The primary limitation in the development of all solid-state batteries is a lack of electrolyte materials, however garnet-structured $Li_7La_3Zr_2O_{12}$ (LLZ)[1] is unique in that it is currently the only oxide material reported to exhibit the desirable properties for a solid-state electrolyte: good stability against air, moisture and lithium metal while also having a wide electrochemical window. LLZ delivers the highest lithium ion conductivity (5×10^{-4} Scm^{-1} at 25°C) of any garnet structured material. The major focus of this work is to introduce new dopants into the LLZ structure in order to make higher ionic conducting garnets and to fundamentally understand the correlation between the type of dopants and the resulting ionic conductivity. We will be looking at doping Bi for La in both crystal structures, and then evaluating the influence of the lone pair of $6s^2$ electrons found on Bi^{3+}on the conduction properties. Our investigation will also be extended by the substitution of Na for Li.

In order to characterise the newly developed materials, we will make extensive use of ex-situ high resolution neutron diffraction and X-ray diffraction to determine the crystal structure of the solid-state electrolyte. In addition, we will conduct in-situ diffraction studies, which allow the crystallographic changes that occur within the battery materials to be monitored in real-time, and should provide unparalleled insight into how the lithium ions influence the electrolyte lattice during battery operation. Solid-state Li^7 NMR and XPS will also be used to gain a better understanding of the environment of intercalated ions.

1. R. Murugan, V. Thangadurai, W. Weppner, Angewandte Chemie International Edition 2007, 46, 7778.

Abstract

Paper Ref: 87

PO122

Strain mechanisms in actuators: in operando investigation of functional materials

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The function of materials widely depends on the crystalline structure and the modification of that structure during the application. In the special case of piezoelectric actuators an applied electric field couples elastic strain and polarization. Among the various types of piezoelectric devices only actuators rely on high electric fields to generate high strains and forces. Prominent examples for actuators are multilayer stack actuators used for nanopositioning or in modern combustion engines for automobiles to control injection cycles. Despite extensive studies and elaborated measurement techniques, the correlation between macroscopic strain and structural response is still not fully understood.

Most of the relevant systems found up to now are compositions close to phase boundaries linking highly correlated phases. This results in major challenges for structural analyses due to overlapping reflections. Apart from the well-known field induced structural responses such as domain switching and the piezoelectric effect we recently identified field induced phase transitions in different systems as an additional poling mechanism [1,2]. The development of a structural analysis of *in operando* powder diffraction data allows resolving all three involved poling mechanisms within only one experiment. The key to obtain enough information for such an analysis is a combination of high resolution and a broad Q-range, together with different sample orientations. This can be done at the WOMBAT Neutron powder diffraction instrument at ANSTO. The results not only separately reveal the contributions of each poling mechanism to the macroscopic strain, but also different behaviours of the individual phases.

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Abstract

Paper Ref: 364

PO123

THE CURRENT STATUS AND A TRIAL NEUTRON DIFFRACTION MEASUREMENT FOR SMALL QUANTITIES SAMPLES IN IMATERIA

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iMATERIA ((IBARAKI Materials Design Diffractometer) [1] is a high throughput-versatile neutron diffractometer in J-PARC, build by Ibaraki prefecture, the local government of the area where J-PARC sites). It covers the d in range 0.18 < d (Å) < 5 with Δd/d = 0.16 % at high resolution bank, and 5 < d (Å) < 40 with the resolution changing gradually at two detector banks of 90 degree, and low angle. So, this diffractometer covers very wide d-range (0.18 < d (Å) < 40). If the small angle detector bank will be available, which is currently under commissioning, iMATERIA will cover wider d-range (0.18 < d (Å) < 800). It takes several minutes to obtain a 'Rietveld-quality' data for the X-ray laboratory sized sample measured at 1MW. Currently, the beam power is limited for tuning the accelerator (~400kW), so that the measuring time is about 10 to 20 min for about 1g of standard oxide samples. The automatic sample changer system is most important sample environment for high throughput experiments. iMATERIA is equipped the automatic sample changer system[2]. This system can change more than 600 samples continuously at room temperature without breaking a vacuum of sample chamber. Recentry, we modified drive system of automatic sample changer. As a result, measurements became speedier. The sample exchange time was shortened from 5 minutes to 2 minutes in this modification.

The beam power became higher than before, it is possible to measure the mg samples for about one day. Small sample measurement is very important for the research for practically materials. We are making some trial neutron diffraction measurement for small quantities samples. Foe example, we have measured 25.4mg YBa2Cu3Oy sample for 18.5h at 300kW beam power. The occupation parameters for deficient oxygen site are almost same with the result of 1g sample measurement. This result supported that the neutron diffraction experiment with mg sample can be performed. The result of another small quantities samples will shown in presentation.

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Abstract

Paper Ref: 434

PO124

Intermediate range ordering in molten noble-metal halides

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Noble-metal halides exhibits physical properties intermediate between ionic and covalent bonding of cation-anion, which causes superionic conduction in some of them. We are now interested in their molten state, because bonding nature is much emphasized in molten state without any restriction of long-range periodicity. Among them, AgI and CuI are famous materials extensively investigated experimentally and theoretically on relationship between partial structures and ionic conduction. Our previous studies revealed that intermediate-range order is involved in cation-cation partial structure which results in a first sharp diffraction peak (FSDP), cation-cation pair correlation penetrates deeply into the nearest neighboring shell formed by unlike-ion pairs and cation exhibits chain-like fragments in molten state. We have recently found by X-ray diffration that pseudo-binary mixture of AgCl and RbCl whose component doesn't exhibit a FSDP individually has a FSDP. A molecular dynamics (MD) simulation using polarizable ion model (PIM) was performed to obtain its partial structures. It reproduced successfully the FSDP with showing positive contribution of Ag-Ag and Rb-Rb partials to it and negative contribution of Ag-Rb one. This means that Ag and Rb has clusters individually with arranging alternative between Ag and Rb clusters. The used potentials and the effect of PIM may give an insight that the origin of intermediate-range chemical ordering is a coexistence between ionic bond of Rb-Cl and covalent bond of Ag-Cl. In this presentation, we will review medium-range ordering in molten noble-metal halides and pseudo-binary molten salt mixtures between noble-metal halide and alkali halide.

Abstract

Paper Ref: 113

PO126

Electrohydrodynamic-jet-printed metal-grid transparent electrode thermal-pressed into flexible substrate and its application to organic solar cells

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We have demonstrated the EHD (Electrohydrodynamic) printing of Ag nano-particle ink to prepare Ag-grid transparent electrodes. Small Angle Neutron Scattering (SANS) has been used for determining the sizes and shapes of Ag nanoparticles used in the ink. SANS spectra confirmed that the nano-Ag has an Ag core diameter of 50nm and a 0.6 nm thick shell in solvent. The width of the printed grid is less than 10 um, which cannot be identified by the naked eyes. The Ag-grid was pressed into plastic substrates during heat treatment. The Ag-grid was well embedded inside the plastic substrates, which leads to a smooth surface of the transparent conducting films. The electrical and optical properties of the EHD-printed Ag-grid electrodes are investigated. Ag-grid is carefully designed in order to maximize both conductivity and transparency of the EHD-printed transparent electrodes. Depending upon the grid pitch (distance between adjacent Ag lines), the resistivity varies accordingly. We introduce the concept of “figure of merit (FM)”. The calculated FM is used to find out the optimized grid pitch for their future application to optoelectronic devices. The optimized Ag-grid transparent electrode is employed to the fabrication of organic solar cells.

Abstract

Paper Ref: 158

PO127

IN-DEPTH STUDY OF NAPHTHOQUINONE-DERIVATIVES ORGANIC MATERIALS FOR LITHIUM ION BATTERY WITH HIGH CAPACITY AND STABILITY

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In energy storage systems, there are growing needs for developing new active materials with high capacity and good electrochemical stability. Organic compounds, such as quinone-based materials have shown promising possibilities for future cathode materials of high energy density, eco-friendly and cost-effective rechargeable lithium ion batteries (LIBs). In particular, naphthoquinone (NQ), especially vitamin K compounds have extensively been investigated as one of promising candidate materials for such purpose. Unfortunately, these organics compounds have commonly exhibited low reduction potentials, compared to conventional transition-metal oxides-based LIB cathode materials, hampering the achievement of high power densities from the batteries. In addition, stable electrochemical performance was rarely obtained from the organic cathode materials owing to their high solubilities into liquid electrolytes. This stimulated synthesis and in-depth characterizations of new organic cathode materials beyond NQ to improve the battery performance. Here, we report a facile synthesis of a new set of naphthoquinone-derivatives through simple organic substitution reactions. In particular2- and 3-positions of NQ ring were substituted with various functional groups, i.e., electron-donating (-NH_2, -OH, -CH_3) and/or electron withdrawing (-Cl, -F) groups, enabling us to control the discharge potential plateaus of organic materials systematically. The optimized NQ-derivatives demonstrated high discharge potential plateaus over 2.3 V, which is significantly higher than conventional NQ-based systems. Theoretical examination on electronic structures of various NQ-derivatives using DFT calculation indicated good agreement with our experimental results. In order to underpin the improvement, various intermediates of organic clusters of 2-, 3-substitued NQ materials formed during the lithiation/delithiaion process were analyzed by combining small and wide angle scattering methods. Structural changes of organic electrodes upon contacting with liquid electrolytes were further investigated by diffraction experiments.

Abstract

Paper Ref: 96

PO128

$[Cu_{32}(H)_{20}\{S_2P(OiPr)_2\}_{12}]$: A RECORD-BREAKING TWENTY HYDRIDES RECORDED ON A MOLECULAR METAL CLUSTER BY NEUTRON DIFFRACTION

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[1] *Department of Chemistry, National Dong Hwa University*
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After successful synthesis of the air- and moisture-stable hydride-encapsulated copper complexes $[Cu_8(H)L_6]^+$ and $[Cu_7(H)L_6]$ stabilized by dichalcogen (S, Se) donor ligands (L),[1] we have extended the synthetic protocol by forming a larger dithiolate-stabilized nanospheric copper(I) polyhydride cluster, $[Cu_{20}(H)_{11}\{S_2P(O^iPr)_2\}_9]$,[2] and $[Cu_{28}(H)_{15}\{S_2CNPr_2\}_{12}]PF_6$.[3] Herein we report a novel air- and moisture-stable nanoscale polyhydrido copper cluster $[Cu_{32}(H)_{20}\{S_2P(O^iPr)_2\}_{12}]$ (**1_H**). The molecular structure of **1_H** exhibits a hexacapped pseudo-rhombohedral core of 14 Cu atoms sandwiched between two nest-like triangular cupola fragments of (2 x 9) Cu atoms in an elongated triangular gyrobicupola polyhedron, which is stabilized by 12 dithiophosphate (dtp) ligands and a record number of 20 hydride ligands (Figure 1). The hydride positions were located by high resolution single-crystal neutron diffraction to exhibit tri-, tetra- and penta-coordinated hydrides in capping and interstitial modes. This result was further supported by ^1H, ^{31}P NMR spectroscopy, ESI-mass spectroscopy and elemental analysis.

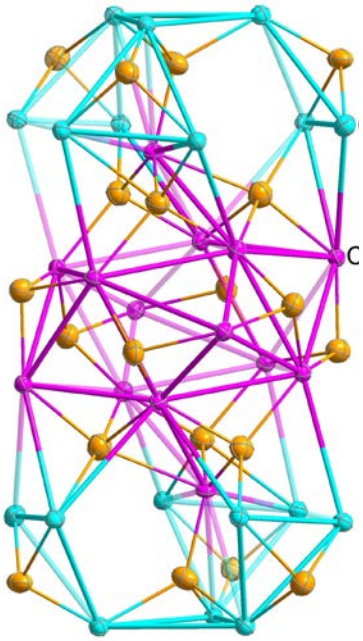

Figure 1. $Cu_{32}H_{20}$ core of **1_H** (30% thermal ellipsoid).

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Abstract

Paper Ref: 29

PO129

Pressure-induced valence transitions in geometrically frustrated perovskites

Chris D Ling[1], Brendan J Kennedy[1] and Max Avdeev[2]

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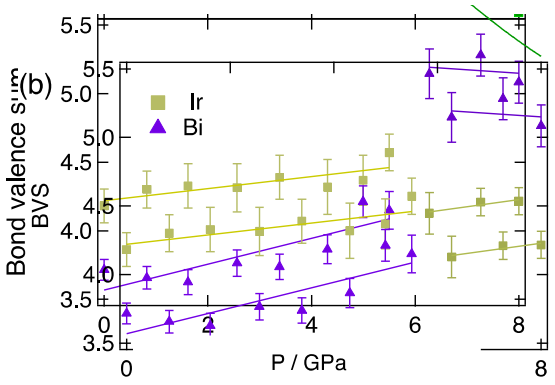
Solid-state compounds are generally thought of as consisting of ions with well-defined oxidation states. While ionic bonds always have some degree of covalent character, the ionic approximation is usually sufficient to understand their “crystal chemistry” in conjunction with concepts such as bond valence sum (BVS) and effective ionic radius (IR). IR predicts that an atom will shrink as its oxidation state increases. This occurs gradually as electrons are removed within a shell (*e.g.*, IR(Ir^{3+}) = 0.68, IR(Ir^{4+}) = 0.625, IR(Ir^{5+}) = 0.57 Å in 6-fold coordination), but removing the last electron of a shell produces a more pronounced change (*e.g.*, IR(Bi^{3+}) = 1.03, IR(Bi^{5+}) = 0.76 Å). For a compound with a suitable combination of cations, it should therefore be possible to effect a net reduction in volume by transferring an electron from one to the other. A change in temperature and/or pressure could make such a valence state transition favourable; but in practice, such transitions are extremely rare.

Using *in situ* high-pressure neutron powder diffraction, we have observed such a pressure-induced charge transfer, from Bi to Ir (or Ru), in a series of hexagonal perovskites $Ba_{3+n}BiM_{2+n}O_{9+3n}$ (n = 0,1; M = Ir, Ru) [1,2]. They all show ~1% first-order volume contractions at room temperature above 5 GPa, due to the large reduction in the IR of Bi when the 6s shell is emptied on oxidation. These are the first such transitions involving 4d and 5d compounds, and double the total number of cases ever observed. *Ab initio* calculations suggest that magnetic interactions through very short (~2.6 Å) *M–M* bonds contribute to the finely balanced nature of their electronic states.



(Left) Bond valence sums for Bi/Ir vs. pressure for $Ba_3BiIr_2O_9$ from NPD data. (Right) Structure of $Ba_3BiIr_2O_9$; O are red, Ba are green, BiO_6 are purple and IrO_6 are gold.

[1] W. Miiller *et al.*, *Journal of the American Chemical Society* **134**, 3265-3270 (2012)

[2] Z. Huang *et al.*, *Angewandte Chemie – International Edition* **53**, 3414-3417 (2014)

Abstract

Paper Ref: 418

PO130

Neutron Study on Li_3PO_4 Solid Electrolyte Prepared by Wet Chemical Reaction

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Safety is the main problem on developing the lithium ion battery. The combustion is mainly due to the leakage or shortcut of the electrodes, caused by the liquid electrolyte and polymer separator [1]. For this reason, the research on solid electrolyte for replacing the existing liquid electrolyte is very important. Li_3PO_4 has been proved to be a good candidate for solid electrolyte, due to its easy in preparation, low cost, high melting temperature and good compatibility with the electrode materials. So far, Li_3PO_4 has been applied in thin film battery [2]. In the present work, Li_3PO_4 has been prepared by wet chemical reaction, a simple method with the advantage of recycling a waste product H_3PO_4. The crystal structure of Li_3PO_4 has been measured by using high resolution powder diffraction (HRPD) at the Neutron Scattering Laboratory, National Nuclear Energy Agency (BATAN), Indonesia. In addition, the x-ray diffraction was also carried out to the same sample, besides several measurements on its thermal and electrical properties [3]. The neutron results show the crystal structure of orthorhombic phase P m n 21 (31), that belongs to the β-Li_3PO_4 [4], with the lattice parameters are a = 6.116819, b = 5.249803, c = 4.872359. The conductivity of β-Li_3PO_4 was around 10^{-8} S/cm, and no phase transition β-to γ-Li_3PO_4 was detected on heating the materials up to 900^{o}C.

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Abstract

Paper Ref: 70

PO131

Molecular Ordering Around the Solid-Liquid Interface – Ordering in the Liquid State (Poster Presentation)

Catherine J Onie[1], Richard A Mole[2], Aditya Rawal[3], James M Hook[3], Nageshwar R Yepuri[4], Tamim A Darwish[4], John A Stride[1,2]

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Despite being a fundamental state of matter, the liquid phase remains poorly understood. The field of condensed matter consists of much literature on the solid state, including solid-solid phase transitions,[1-2] whilst evidence for transitions between liquid states in single-component substances remains relatively little-known, although is increasing.[3-4] We have examined the supercooled liquid state of pentamethylbenzene (PMB), a material composed of small low-symmetry molecules. PMB has a thermal hysteresis of 10 – 14 K between its solidus and liquidus, due to a liquid-liquid transition from the isotropic to a semi-ordered state prior to crystallisation on cooling. The highly anisotropic molecular shape[5] is conducive to liquid ordering, but to date no evidence of such a phenomenon has been reported. Quasielastic neutron scattering of selectively labelled isotopic variants of PMB have been performed in order to understand the molecular diffusion in PMB. Complementary physical measurements were also undertaken, to verify and characterise the anomaly across a wide range of time- and length-scales, including: solid-state NMR, powder X-ray diffraction, polarising optical microscopy and differential scanning calorimetry. These measurements were performed across a broad range of temperatures around the solid-liquid boundaries, and it was concluded that there is evidence of liquid ordering prior to crystallisation on cooling from the melt, consistent with the delayed onset of crystallisation. Also confirmed is the long-known but poorly characterised solid-solid phase transition of PMB[6] on heating, mirrored by a delayed onset of 3 – 4 K on cooling. Whilst these findings are specific to PMB, it is foreseen that the phenomenon is universal, demonstrating the roles of molecular dynamics and symmetry in ordering within supercooled liquids, providing greater insight to liquid-liquid phase transitions.

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Abstract

Paper Ref: 220

PO132

CHARACTERISATION OF ELECTRODE MATERIALS FOR LI-ION BATTERIES USING OPERANDO NEUTRON POWDER DIFFRACTION

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Secondary rechargeable Li-ion batteries (LIBs) are the primary choice of power source for portable electronic devices, including mobile phones, laptops, as well as electric vehicles. The working principle of a LIB is to store electric energy in chemical form by using charge-balancing Li ions that reversibly insert into the electrodes. The structure and chemistry of the electrodes are closely related and determine its functional mechanism. The removal and insertion of Li ions from the electrodes can significantly affect phase and structure, sometimes with a complex evolution during battery charge and discharge, with these processes underpinning function and electrochemical performance of the whole battery. Therefore, a mechanistic understanding of the reaction pathways, i.e. the atomistic and molecular-scale origin of battery performance [1-5], will enable the rational improvement of electrode materials and pave the way for entirely new battery systems. The suitable elemental contrast, relatively-large penetration depth, and non-destructive interaction with matter that neutrons offer couple with the fast-detection ability of modern instrumentation to enable neutron powder diffraction to probe in real time the bulk crystallographic changes of electrodes in functioning batteries, with such experimental approaches known as *operando* studies. In this presentation, examples of *operando* neutron powder diffraction studies of electrode materials performed using WOMBAT [6], the high-intensity neutron powder diffractometer at the Australian Nuclear Science and Technology Organisation, will be presented, demonstrating the importance of diffraction techniques in battery research.

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Abstract

Paper Ref: 23

PO133

Spectroscopical characterisation of high surface area carbons through a multitechnique approach

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High surface area carbons are industrially relevant materials whose properties depend on morphology, texture and surface features. The detailed characterization of functional group on the surface of this class of materials is a fundamental step for understanding their potential in a variety of applications. Unfortunately IR, the widely used laboratory spectroscopy method, is difficult to be applied on carbons due to the intrinsic strong absorption.

We present here an multitechnique approach, based on the synergic combination of three vibrational spectroscopies: i) FT-IR diffuse reflectance spectroscopy (DRIFT), that, limiting the strong absorption of the transmitted light is effective in evidencing vibrations with change in the dipole; ii) back-scattering Raman spectroscopy, which is sensitive mostly to carbon bulk vibrational modes; iii) Inelastic Neutron Scattering (INS) that, eliminates the problem of radiation interaction and is sensitive to vibrations involving hydrogen including species, highly abundant on carbons surface. The three technique are applied to two classes of activated carbons, subjected to specific chemical treatments. The whole set of experimental data, interpreted with the help of DFT calculations, allow us to point out their structural and surface properties, and to clarify some controversial information present in the specialized literature, where conclusions are done on the basis of the data obtained by a single technique.

Abstract

Paper Ref: 49

PO134

A neutron diffraction study of structural properties of a half-Heusler alloy MgAgSb-based thermoelectric material

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The continuous increase of energy consumption is a major societal issue for the 21th century. It becomes more and more attractive to improve the energy efficiency that electricity is generated by unused waste heat in industrial processes, home heating, and automotive exhaust by using thermoelectric generators. However, the application of thermoelectric generators is currently hampered by their low efficiency, i.e. a low figure of merit (ZT), especially working around room temperature. Recently, high thermoelectric performance has been found in the MgAgSb-based half-Heusler alloys, their ZT values are close to 1 at room temperature with a maximum of ZT =1.4 at 475 K[1], making them as possible candidates of room-temperature thermoelectric generators. Here we present the crystallographic structure of $MgAg_{0.97}Sb_{0.99}$ at 3K and 300K studied by high resolution neutron powder diffraction using the Echidna instrument at ANSTO, Australian. Rietveld structure refinement of the neutron data indicates that the sample is pretty single phase with tetragonal structure (space group: *I-4c2*). No detectable structural transition is consistent with our heat capacity data recorded by PPMS. Because of the remarkable crystal grain size effect, an isotropic size parameter of Gaussian character is refined. An average grain size of 18.9 nm is calculated from this refined parameter, in agreement with the values of 10-20 nm estimated by the TEM [1]. The tiny deviation from the stoichiometric ratio of the start raw materials are difficult to refine due to the close neutron scattering lengths of Mg, Ag, and Sb. The overall average isotropic thermal factor B_{over} increases from 0.32 at 3 K to 1.05 at room temperature, which seemingly indicate the large atom relaxation in nano crystallites and/or between them at room temperature.

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Abstract

Paper Ref: 405

PO135

Neutron Diffraction Study of Short-range Order of Deuterium in Laves Phase Alloy $Ti_{0.68}Zr_{0.32}CrMn$

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Ti-Zr-based AB_2 type Laves phase alloys are studied extensively for hydrogen storage because of their high storage capacity. In neutron diffraction studies of deuteride of these alloys, diffuse scattering has been noted by some researchers, and attributed to the existence of a short-range correlation between nearest deuterium atoms in the lattice of the deuterides [1-3]. Hydrogen storage capacity is thought to be closely related to the short-range correlation of hydrogen in these alloys. However, apart from deuterium in a pure metal of vanadium [4], the diffuse scattering and associated short-range order of deuterium in alloys have not been reported. Among Laves phase alloys, the $Ti_{0.68}Zr_{0.32}MnCr$ alloy exhibits excellent performance [5]. This is also a null matrix alloy for neutron diffraction, which allow us to study the short range correlations of deuterium without contamination from diffraction of the metal matrix .

Neutron diffraction measurements of deuterated $Ti_{0.68}Zr_{0.32}MnCr$ alloy were performed on Wombat (λ = 0.878 Å) and D4 (λ = 0.498 Å) diffractometers at ANSTO and ILL, respectively. The alloy was charged at different pressures at room temperature and at 40 K, and the diffraction patterns were collected in situ.

The neutron diffraction shows strong diffuse scattering increasing in intensity with increasing pressure and reducing temperature. Thus, the diffuse scattering can be attributed to short-range order of the absorbed interstitial deuterium. In addition to diffuse scattering, Bragg peaks associated to the long-range order of interstitial deuterium are observed. Rietveld refinement of these peaks shows that deuterium partially occupies the 6*h*, 12*k* and 24*l* interstitial sites. However, with increasing diffuse scattering intensity, the relative occupancy of deuterium in the 24*l* sites is reduced, indicating formation of short-range order clusters.

Fourier tranformation of the neutron diffraction patterns indicates that the nearest neighbour distance of deuterium is close to the critical value of 2.1 Å, the proposed limiting distance of D-D. This distance appears to decrease slightly with increasing deuterium, suggesting that the D–D repulsion is strong at these pressures and temperatures. The relevant local deuterium arrangement under such separation distances and the associated interstitial routes for diffusion of deuterium will be discussed.

Acknowledgements

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Abstract

Paper Ref: 420

PO136

Deuteration of Arylamines and their Use as Precursors in the Synthesis of Organic Light Emitting Diodes (OLED)

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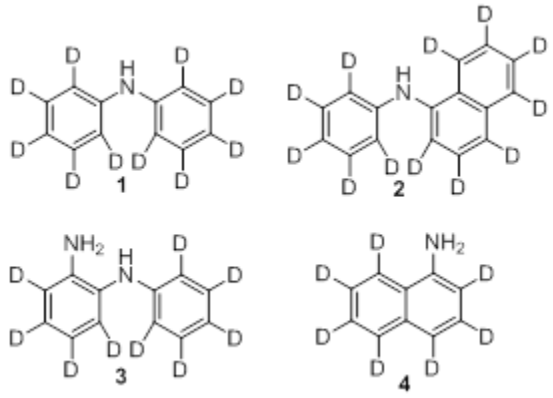
Organic optoelectronics is a rapidly growing area of interdisciplinary science and technology, with investigations focused on small molecules, conjugated polymers, and dendrimers, that can be used as the light-emitting layers in organic light-emitting diodes (OLEDs). A recent investigation with deuterium substituted polymer based OLEDs has shown improved spin transport, magnetoconductance and magnetoelectroluminescence.[1]

Chemical Deuteration activities, where catalysed $^{1}H/^{2}H$ exchange is followed by custom chemical synthesis, have led to exciting diverse neutron scattering and NMR studies previously hampered by the lack of relevant labelled compounds, some of which are found in OLED devices.[2] These compounds made possible a diverse range of neutron reflectometry (NR) investigations including the study of the morphology, diffusion and interfacial behaviour in thin-film OLEDs.[3] We present an overview on the deuteration of aryl amines and their use in the synthesis of poly-aryl amine compounds which are found in optoelectronic applications. Here we also report a general simple method and reaction conditions such as reaction scale, time, temperature, catalyst ratio and co-solvent were optimized for each molecule. Deuterium levels are measured based on mass spectral analysis and levels at individual sites were calculated using ^{1}H, ^{2}H, and ^{13}C NMR technique. Deuterium labelling was performed either by H/D exchange on the protonated form of the desired compound or precursor using deuterium oxide, catalysed by Pt/C and Pd/C under hydrothermal conditions.



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Abstract

Paper Ref: Here

PO137

Cryostat with dilution insert and Polarised ^{3}He Neutron Spin Filter on Pelican Instrument

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The Pelican instrument (cold neutron time-of-flight polarisation analysis spectrometer) has been designed to use a new closed cycle refrigeration cryostat with a dilution insert, providing temperatures ranging from 300 K to 40 mK. This is provided in conjunction with a polarised ^{3}He analyser cell with 120° of coverage in the x-y plane. *In-situ* refilling of pre-generated polarised ^{3}He gas has been implemented to maintain the analysing efficiency. A highly uniform 3-Dimensional magnetic field utilising a Pastis coil arrangement (Stewart et al., 2006) for X and Y-axis and a modified Tetracoil set (Gottardi et al., 2003) for the Z axis provide a static holding field; preserving the polarisation of ^{3}He gas. The Z-axis Tetracoil has also been designed to provide a guide field for incoming polarised neutrons.

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Abstract

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PO138

HIERARCHICAL SOFT MATTER SYSTEMS: STRIPED VESICLES, CUBOSOMES AND HEXOSOMES

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Cubosomes and hexosomes are hierarchically organized materials, where sub-micron sized dispersed particles contain an internal liquid crystalline nanostructure. [1] This nanostructure is usually based on lipids that self-assemble due to their amphiphilic character, i.e., they have two immiscible parts linked together in the same molecule.

As an extension, we have been developing star-polyphiles, which are small lipid-like molecules, but with three mutually immiscible parts (a hydrophilic, hydrocarbon and fluorocarbon chain) attached to a common center. [2-4]

Here we present our combined USANS/SANS/SAXS study of vesicles, cubosomes and hexosomes based on star-polyphiles, which show hierarchical ordering on three length scales: submicron-sized particles with internal liquid crystalline phase, in which the membrane of the liquid crystals consists of hydrocarbon and fluorocarbon stripes.

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