

In-situ TEM studies of the radiation tolerance of ceramics

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For the nuclear industry ion-irradiation has been used to simulate recoil damage from alpha decay, and exposure to neutrons in fission and fusion reactors [1]. The particular focus of this research is on the crystalline to amorphous transition facilitated by exposure to accelerated ions.

The complex ceramic oxides chosen for this study, Ln_2TlO_5 (Ln = lanthanides and yttrium), have several uses within the nuclear industry. The compound Dy_2TlO_5 has been used within WWER type reactors due to its good resistance to irradiation induced swelling and structural failure [2]. Of particular interest are the cubic symmetry compounds with defect fluorite structure, which gives good radiation response. Previous studies show the series of Ln_2TlO_5 compounds may take on a variety of crystal symmetries depending on the lanthanide size and fabrication conditions used [3, 4].

The study of ion-irradiation response was carried out using the in-situ approach where the test materials were exposed to 1 MeV Kr^{2+} ions and monitored for their transition from crystalline to amorphous state. This was carried out using the intermediate voltage electron microscope (IVEM)-Tandem facility at Argonne National Laboratory. The critical dose of irradiating ions, D_c , required to render the Ln_2TlO_5 completely amorphous was determined by monitoring selected area electron diffraction patterns for loss of diffraction spots (Bragg maxima) and replacement with diffuse rings.

Within the Ln_2TlO_5 series of compounds studied a linear relationship appears between the radii of the lanthanide and the radiation response. This follows a similar trend found in related pyrochlore compounds. Possible explanations for this trend are investigated.

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Online Education and Training for Microscopy and Microanalysis: MyScope™

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MyScope is an online educational site that provides modules used to train in the areas of scanning and transmission electron microscopy, scanning probe and atomic force microscopy, confocal microscopy, X-ray diffraction and microanalysis (<http://www.ammr.org.au/myscope/>). The MyScope site is popular across the globe, and Google Analytics tracks about 15,000 sessions per month. Growing demand for advanced research techniques, limited staff time, and demand for specialist machine access create pressures in core microscopy and microanalysis facilities. The modular tools accelerate learning so that new users coming into laboratories can be better prepared. Once familiar with technical language and theory specific to the desired research technique as well as experience on a virtual machine, users are able to undertake practical instruction in the laboratory in a shorter than usual time. Improvement in core knowledge has also been quantified. There are a variety of ways that MyScope is used in training and education. Modules can be tailored to requirements, for those with a specific learning aim in mind, and a drag and drop function allows educators to create a personalized, new site, restructured and edited specifically for class needs. This function is gaining more popularity but many users are still unaware of its flexibility. This lunchtime workshop/ information session will update attendees on MyScope in its present form. We will demonstrate how educators are currently using the features available and address the gains that core facilities and educators find from its use. The session will provide information on current oversight and plans for the platform in 2016.

Geomaterials in the age of megapixel imaging

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Geological samples are extremely diverse and share a tendency for heterogeneity and complexity. This is especially true for ores and for environmental samples, which result from complex processes in dynamic environments. In recent years, a number of tools that enable imaging element distribution in geological samples at 1-50 μm -resolution and over cm^2 areas have seen rapid development and have become readily available. The application of synchrotron-based X-ray fluorescence mapping has been limited to addressing key questions because of low availability and high cost. However, recent advances in X-ray fluorescence detector technology are bringing new possibilities to petrology. Millisecond dwell times allow collection of thin-section-size maps in hours, and improvement in data analysis produces quantitative elemental maps. The technique can be combined with XANES imaging to provide additional information about element speciation (e.g., As oxidation state).

We illustrate applications of M(egapixel)- μXRF for ore petrology (commodities: Au, Pt, U, Cu, Ge, Ti, REE, Nb), coal petrology, and environmental samples. Examples of outcomes include: (i) the distribution of μm -sized Pt-rich grains and Ti-mobility during schistosity formation at the Fifield Pt prospect (Australia); (ii) confirmation of the two-stage Ge-enrichment in the Barrigão deposit (Portugal), with demonstration of the presence of Ge

in solid solution in the early chalcopyrite; (iii) enrichment of U during late dissolution-precipitation reactions in the Cu-rich ores of the Moonta and Wallaroo IOCG deposits (Australia); (iv) history of REE-Ti-Nb-(As) mobility during amphibolite to greenschist facies metamorphism in the Binntal Valley, Switzerland; (v) contrasting distribution of As, Ge and W in Ge-rich coals across the Globe; and (vi) evolution of the distribution and speciation of Cu upon aging of biosolids.

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Atom probe tomography in the geosciences

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Atom probe tomography (APT) combines 3-dimensional atomistic spatial reconstruction with time-of-flight mass spectrometry to provide chemical information in three dimensions down to sub-nanometre scales. Traditionally applied to metallurgical research, over the past ~10 years this technique has been expanded to include the analysis of semiconductors, non-conductive materials, device structures, and even some biological specimens.

More recently APT has found its first applications of significance within the geoscience research community. Though only a handful of publications have so far appeared, this is expected to become a growth area as previous nano-scale characterization studies are extended, providing further insights into existing problems, and new areas of investigation are made accessible via the high spatial resolution provided by APT.

This presentation will review work to date on the application of atom probe tomography in geoscience research. Recent results will be presented from the newly-established Geoscience Atom Probe Facility located at Curtin University; including the analysis of trace element distributions in zircon, a mineral widely used in U-Pb isotopic dating. The high resolution of the technique provides new opportunities in geochronology development. More foundational studies will also be reported, which lay the groundwork for the application of this powerful technique to new geological material systems.

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Correlative atom probe tomography, NanoSIMS and Maia Mapping of gold in arsenopyrite (FeAsS)

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Arsenopyrite (FeAsS) is found in many precious metal deposits and commonly contains gold up to several thousand parts per million. The distribution of the gold in such minerals is critical in understanding the geological development of the deposit and optimising gold extraction during mineral processing. However, the primary distribution of gold remains controversial with competing models including gold homogeneously distributed within the crystal lattice or hosted as nanoparticles. To address this controversy, arsenopyrite from the giant Obuasi gold deposit, Ghana, has been studied by correlative atom probe tomography (CAMECA LEAP 4000X HR), sub-micron resolution ion microprobe imaging (CAMECA NanoSIMS 50) and quantified synchrotron X-ray fluorescence element mapping using the Maia detector array at the Australian Synchrotron. Each technique provides different, nonetheless complementary information at various scales and resolution. The large synchrotron maps (several mm²) indicate that the arsenopyrites are zoned, with gold-rich rims hosting up to 2000 ppm of gold and nickel-rich overgrowths, dissecting the auriferous domains. The high-resolution NanoSIMS data yield 100x100um² reveal that gold is distributed in alternating, submicron-scale concentric bands (up to 100 in individual grains) with sharp boundaries. Atom probe tomography of these different bands reveals the distribution of gold at the nanoscale and provides the data required to differentiate between competing models of gold distribution in arsenopyrite. The workflow developed in this study allows to investigate the microchemistry of ore minerals at the smallest atom probe scale within a well characterised framework.

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Recovering from Extreme Shock: Trace element migration in shock deformed zircon revealed by atom probe tomography

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The mineral zircon (ZrSiO₄) is widely used in the geosciences to date geological events. Recent work has documented that the plastic deformation of zircon can modify the distribution of trace elements within the zircon lattice, thereby raising the possibility of being able to date deformation of Earth's crust. However, the processes responsible for element migration during zircon deformation are unclear. Here we combine atom probe tomography (APT), electron backscatter diffraction (EBSD) and transmission Kikuchi diffraction (TKD) to address this problem.

The Stac Fada Member of the Stoer Group, NW Scotland represents ejecta from a meteorite impact event about 1.2 billion years ago. EBSD data from this rock unit reveals the presence of the extremely rare high-pressure polymorph of ZrSiO₄, reidite, within the host zircon. This provides unambiguous evidence of shock pressures in excess of ~30 GPa. In addition, the host zircon and reidite lamellae both contain low-angle boundaries, which are interpreted to represent recovery and the migration of shock-induced dislocations into lower energy configurations in the latter stages of the impact event. TKD analysis of one of these low-angle boundaries, captured within a FIB-milled atom probe needle, reveals a lattice disorientation of 2° across a zone of ~20 nm width. Atom probe analysis reveals elevated concentrations of Y, Al, Be and Mg within the low-angle boundary, which we interpret to reflect trace element migration within the cores of mobile dislocations during recovery. The prospect of a dynamic