

## OPAL news

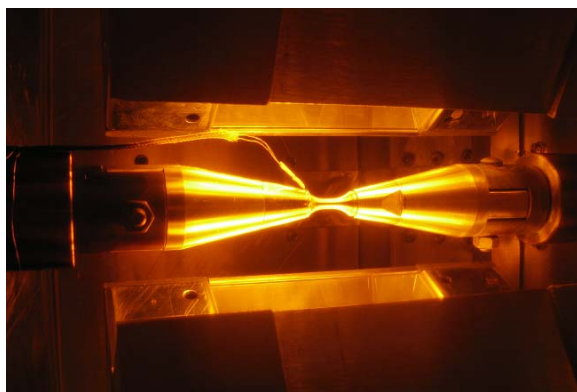
The OPAL Reactor has provided good thermal-neutron reliability throughout the first three months of 2011, and 84% of this beam time has been used for user experiments on our [5 operating thermal-neutron instruments](#). However, the cold neutron source has not returned to service, due to continuing difficulties with the helium gas system that drives the cryogenic system. ANSTO has been working with all the relevant contractors to understand and ameliorate this situation, and we are hopeful that cold-neutron service will resume on 14 May after the next planned shutdown. Given the present non-operation of OPAL's cold-neutron source, and a large backlog of approved user experiments, the QUOKKA and PLATYPUS instruments have been withdrawn from the 22 May proposal round.

## Bragg Institute news

After the **next proposal deadline of 22 May 2011**, we will move to fixed and regular proposal deadlines of 15 September and 15 March each year, for beam time allocations in the January-June and July-December windows respectively. The next beam-time allocation period will hence be for the shorter window of 3 months between October and December 2011, inclusive. This move has been made after extensive consultation with our [Program Advisory Committee](#), which includes formal representation from both [AINSE](#) and the [Australian Neutron Beam Users Group](#) with our staff.

## Around the instruments

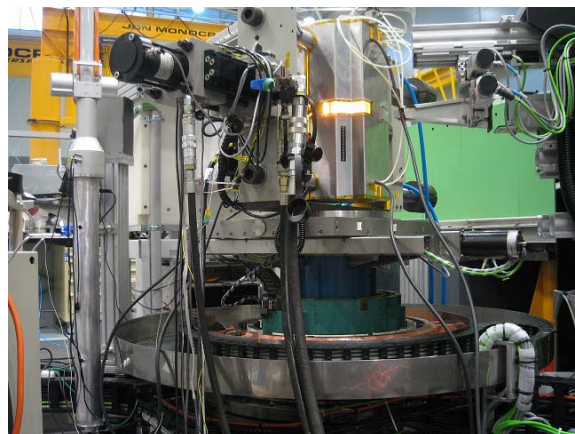
### *Kowari* (strain scanner)



*The specimen under heating and load environment.*

In April, researchers from ANSTO's Institute of Materials Engineering and the Bragg Institute conducted strain measurements on a Zr-2.5%Nb alloy using our KOWARI Strain Scanner at 900 °C, equipped with a 100 kN uniaxial INSTRON load frame and infra-red heating lamps.

Zr-2.5%Nb alloy, a material used for structural components of nuclear reactors including the OPAL reflector vessel, has the right combination of strength, ductility, corrosion resistance and neutron economy. Although zirconium-based alloys have been extensively studied and engineered over the last four decades, new insights are being revealed through use of advanced diffraction based techniques, which may lead to better optimization of these alloys for use in the next generation of reactors.



*The thermomechanical load frame and sample on the KOWARI strain scanner.*

Previous experiments conducted on WOMBAT showed that various phase transformations occur between 500 and 865°C, above which the Zr-2.5%Nb alloy exists almost exclusively in the  $\beta$ (bcc) phase. As this alloy is heated above 865°C, the peak intensities of all  $\beta$  peaks show an anomalous drop by up to 70%. This phenomenon was not observed during X-ray experiments which show neither peak intensity changes nor texture changes under identical conditions. The more deeply penetrating neutrons, however, show changes in peak intensity during heating, and these are correlated to primary extinction effects within the [dynamical theory of diffraction](#), reflecting the evolution of  $\beta$  crystallite quality with temperature.

KOWARI was used to induce mosaicity (mostly dislocations at these temperatures) via plastic

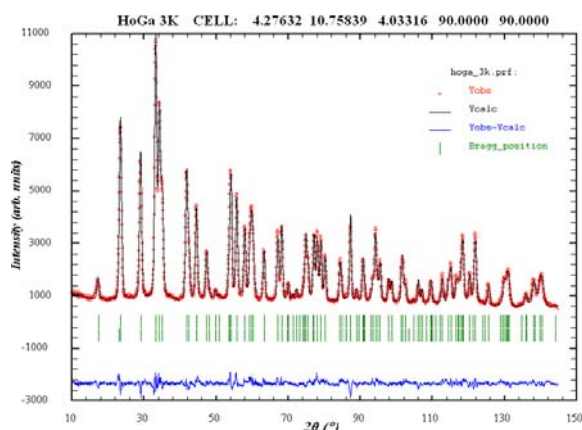
deformation. By observing changes in peak intensities upon plastic loading, the researchers were determining whether peak intensities recover, and thereby validate the extinction model.

The long-term goal of this method is to determine kinematic processes occurring in the sample such as dynamic recovery, nucleation and growth of secondary phases in these alloys.

**Echidna** (high-resolution diffractometer)

A major research area being pursued by Australian expatriate Sean Cadogan at the University of Manitoba (Canada), in collaboration with colleagues at McGill University and the University of Alberta, involves the study of the intrinsic magnetic behaviour of intermetallic compounds containing rare-earth (R) elements. International partners include researchers from Brazil and India. A common feature of the compounds of interest is the role played by crystal-field interactions in determining their intrinsic magnetic behaviours.

In a recent series of experiments on ECHIDNA the magnetic structure of Nd<sub>2</sub>Al was determined, showing that this ferromagnet has a strongly crystal-field quenched neodymium magnetic moment. Speculation that the reduction in magnetization is associated with antiferromagnetic components in the magnetic order, or short-range magnetic clusters, was ruled out. A paper on this work was presented at the *Joint European Magnetic Symposium* held last year in Poland and will appear soon in *J. Phys. Conf. Series*.

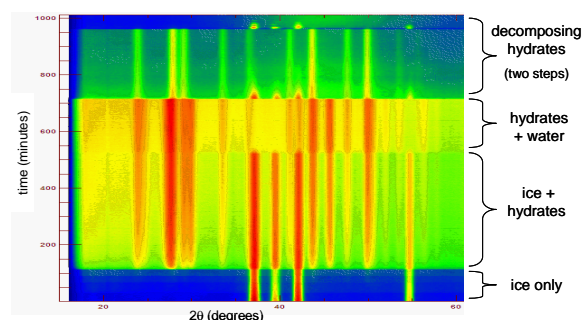


Neutron powder diffraction of HoGa, obtained at 3 K on Echidna

The magnetic ordering and spin-reorientation in a number of rare earth-gallium intermetallics have also been determined. These compounds enjoy some interest at the moment due to their potential use in low-temperature magnetic refrigeration applications. HoGa is a uniaxial ferromagnet which undergoes a spin-reorientation away from the easy axis due to the effect of higher-order crystal-field terms (Fig.1). Aspects of this work will be presented at the *Rare-Earth Research Conference* to be held in Santa Fé in June and the *ECNS Conference* to be held in Prague in July.

Finally, the magnetic ordering of the Nd and Cr sublattices in NdCrGe<sub>3</sub> have been investigated. The RCrGe<sub>3</sub> compounds are metallic and adopt a hexagonal perovskite structure, which in itself is unusual for intermetallics. Our measured Cr magnetic moment is significantly lower than that expected for either Cr<sup>3+</sup> or Cr<sup>4+</sup> and confirms the metallic, itinerant nature of the magnetism in these phases.

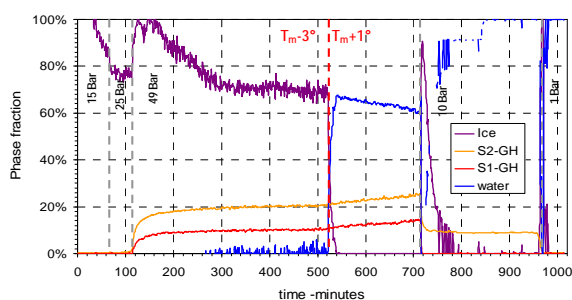
**Wombat** (high-intensity diffractometer) **AND QUOKKA** (SANS)



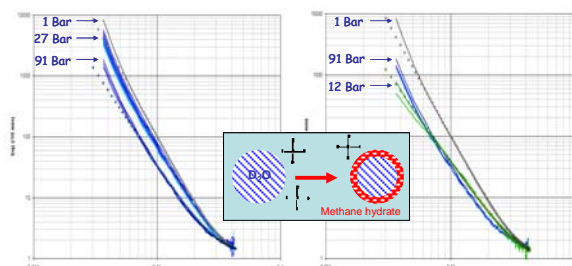
Selected region of the Wombat diffraction patterns from hydrate formation by heavy water (D<sub>2</sub>O) in methane-propane gas at pressures up to 49 Bar.

Researchers from CSIRO and ANSTO are investigating gas hydrates. These inclusion compounds form in deep ocean sediments and in permafrost from the action of water molecules encapsulating gases such as methane. They are of particular interest in the energy sector, as a potential energy source, and to environmental science as a geo-hazard. As gas-hydrate formation and dissociation processes are poorly understood, the natural gas industry takes extreme precautions to minimise the risk of their formation in pipelines.

*In-situ* experiments monitoring the reaction between heavy water ( $D_2O$ ) and high-pressure methane-propane gas on WOMBAT provide insights into the kinetics of hydrate crystallite formation and dissociation and the thermodynamics of the reaction processes. Diffraction patterns obtained on WOMBAT demonstrate that gas-hydrates initially form on the surface of ice crystallites and later at the interface between water and gas. Finally, dissociation of gas hydrate occurs on depressurisation, with a concomitant reformation of ice crystallites due to local cooling.



*Kinetics phase map of Ice, Type I & Type II hydrates and water, extracted from the neutron diffraction patterns above.*



*Log-log plots of the SANS patterns during methane hydrate formation & dissociation in  $D_2O$ . (left) Below the ice point, the slope of SANS decreases from  $Q^{-3.7}$  (grey dotted line) at 1 bar gas pressure to  $Q^{-3.1}$  (blue dotted line) after 6 h at 27 bar and 1 h at 91 bar; indicating formation and thickening of a crust of methane hydrate on near-spherical ice particles, and (right) Above the ice point, the slope decreases further to  $Q^{-2.9}$  (green dotted line) after 2 h at 91 bar; indicating further thickening of the methane hydrate crust. The insert is a schematic of the formation of a methane hydrate crust on a spherical  $D_2O$  core, consistent with the changing slope of the SANS pattern.*

Hydrates of [Type I](#) and [Type II](#) structure were observed in this experiment, and the kinetics of change of both forms were more sensitive to

changes in temperature than to changes in pressure.

The effect of addition of either a thermodynamic inhibitor (mono-ethylene-glycol), or a kinetic inhibitor (Luvicap EG) were tested on gas hydrate formation, and demonstrate that the kinetic inhibitor was more effective at low concentrations in retarding the growth of crystallites, even at concentrations of 0.5%.

*In-situ* SANS experiments on QUOKKA were also conducted to probe the hydrate nucleation process, which precedes the crystallisation processes observed in the WOMBAT diffraction experiments. QUOKKA revealed rapid uptake of methane gas on ice crystallites well below the accepted phase boundary and formation and thickening of a crust of methane hydrate on near-spherical ice particles. Also, the QUOKKA data provided an independent measure of the growth rate of methane hydrate and the diffusion rates of hydrocarbon gases through the solid and liquid water and through solid hydrate material.

## Italian-Australian Workshop on New Scientific Techniques in Cultural Heritage



*Delegates outside the Rydges Hotel in Cronulla.*

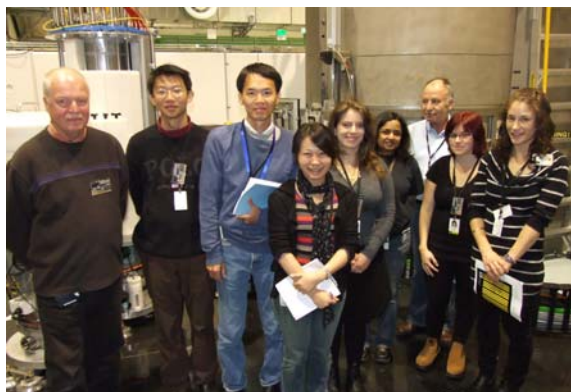
In mid-March, ANSTO co-hosted the Italian-Australian workshop at Rydges Hotel in the seaside suburb of Cronulla. 14 Italian, 37 Australian and two Indonesian experts came together to discuss the use of modern scientific techniques such as, accelerator mass spectrometry, DNA analysis, X-ray, neutron and ion-beam analysis, to solve current problems in cultural heritage. Work on the origins of human beings was highlighted along with chronological investigations. Also given were general studies of archaeological objects, ancient environments and the analysis of modern objects of cultural significance, such as works of art.

Much honest and at times lively debate was given regarding the applicability and suitability of particular techniques, their limitations, and future capabilities. Likewise the context in which the artefacts and localities under investigation were produced, used and established was continually stressed as being absolutely vital for meaningful analysis to be made.

News on publications and outcomes as they result from this workshop is available at [http://www.ansto.gov.au/talks\\_and\\_workshops/italian-australian\\_archaeology\\_and\\_cultural\\_heritage\\_workshop](http://www.ansto.gov.au/talks_and_workshops/italian-australian_archaeology_and_cultural_heritage_workshop)

## Announcements

### 2011 ANSTO-AINSE Neutron School



*A group from the 2010 Neutron School with the TAIPAN three-axis spectrometer.*

This year's [ANSTO-AINSE Neutron School](#) on **Order and Disorder** will be held 14-19 August 2011 at ANSTO.

Thermal neutron techniques currently available at OPAL will be covered in the school, i.e. powder diffraction, single-crystal diffraction, strain scanning and inelastic scattering using the TAIPAN three-axis spectrometer. Candidates will participate in experiments to further enhance their understanding of the relevant techniques, attend lectures and spend significant time analysing data.

Applicants should submit an abstract outlining their scientific interests with relation to neutron scattering. The template can be downloaded [here](#).

**Neutron School abstracts are due Friday 20 May 2011.**

## New Faces

### Arrivals



Ben Kent is a new postdoctoral researcher at the Institute. He completed his PhD at [RMIT University](#) in Melbourne, where he investigated the membrane preservation mechanisms of sugars during desiccation and freezing. He will be using SANS techniques to model membrane systems in the context of cryo- and anhydro-biology.



Sebastian Brück arrives from the [Max Planck Institute for Metals Research](#) where he investigated interface coupling phenomena in magnetic or superconducting thin films. During his PhD he set up the UHV soft X-ray reflectometer end station at a synchrotron and was involved in the development of [ReMagX](#), an associated data processing tool. Sebastian holds a joint postdoctoral fellowship with the [University of New South Wales](#).



Renee Rose has joined us as the User Office Administration and Liaison Officer and in this role will be a primary point of contact for users both before and during their visits to the Institute.

### Departures



Oliver Kirstein left us in February, after eight and a half years, to take up a project leadership positions with the [European Spallation Source](#) (ESS) project in Sweden. The Institute wishes Oliver all the best in his new role.

### Contact us

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