OPAL and Bragg Institute News

The OPAL reactor operated successfully for 289 days in 2013, and has achieved 196 days of almost flawless cold-neutron operation since 18 July 2013. Over the next year, we are doubling the number of instruments available to users, and the Commonwealth Government has made funds available for us to operate the new instruments, as part of its $180M scientific infrastructure package announced in mid-2013. We are currently in the process of hiring the extra staff.

So we are pleased to announce that the PELICAN time-of-flight spectrometer, the KOOKABURRA ultra-SANS instrument and the DINGO radiography/tomography/imaging station are now available for user proposals via the OPAL on-line portal, bringing the total number of available neutron instruments to ten. All three instruments are currently in commissioning with neutrons, and have taken publication-quality data. Award of beam time is contingent upon regulatory approval to move from commissioning to operations.


Around the instruments

Kookaburra (ultra-small-angle scattering)

In November, our new KOOKABURRA Bonse-Hart-type Ultra-Small-Angle-Neutron-Scattering instrument took its first scattering data, from a suspension of standard latex spheres, in a water-heavy-water solution. The 5-bounce silicon (111) crystals were used at a wavelength of 4.74 Å, and standard polystyrene spheres have a diameter of 5 microns. We have yet to install all of the background-reducing shielding around the analysers, so we expect to achieve further improvements. KOOKABURRA can reach down to $Q \sim 3 \times 10^{-5} \text{ Å}^{-1}$, extending the range of conventional pinhole SANS, as performed on QUOKKA and BILBY, into the micron size range, overlapping with optical microscopy and direct neutron imaging, as can be done on DINGO.

Kowari (strain scanner)

Researchers from CSIRO, ANSTO and Swinburne and Monash universities have been using KOWARI to investigate the residual stresses in additively manufactured, wedge-shaped samples of H13 tool steel. H13 is the most widely used tool steel for forging and die-casting dies, due to its high strength and excellent mechanical properties at elevated temperatures.

“Additive manufacturing” is the practice of melting together and solidification of powder particles by either laser or electron beam on a very fine scale down to a 50 μm spot size, to produce structures/components not possible with conventional manufacturing technologies: for instance, complex spline shapes, lattice structures and thin-walled structures (as shown below), allowing for the optimisation of component design with respect to the in-service stresses experienced. This in turn results in weight reduction, an important aspect for automotive and aerospace applications.
To date, there have been measurements of wall structures, showing the tensile stresses during the build and subsequent cooling. But the issue is that typical additively manufactured components have complex geometries. The wedge-shaped sample for this study was chosen to evaluate the role of geometry and section thickness in residual-stress formation; at its thinnest section the cooling rates will be fast, while the thicker sections will cool at lower cooling rates. These cooling rates can be correlated with the final stress distribution allowing insight into the effect of section size on residual stress.

It was anticipated that the thicker end would cool more slowly than the thinner end, producing a different stress magnitude, however the results reveal that the residual-stress profiles are characteristic of relatively slow cooling from the outside inwards, similar to that of a slow quench. Therefore the residual stress produced is relatively independent of section thickness and more dependent on the cooling rate after the processes has finished. This is exciting because it shows that if the parts are cooled slowly after manufacturing, the tensile residual-stress distribution can be reduced or eliminated, irrespective of the section size of the component.

**Koala** (Laue diffractometer)

Two isostructural series of compounds NH₄[Ln(cdm)₆(H₂O)₁₈c₆·3H₂O] (1Ln) and [K(18c6)(H₂O)₂][Ln(cdm)₆(H₂O)₁₄]·H₂O (2Ln) were synthesised and structurally characterised (Ln = Gd, Dy, Er; cdm = C(CN)₂(CONH₂)⁺). These two classes of compounds are shown to be essentially isostructural to each other, despite the change in counter cation of an NH₄⁺ cation in 1Dy, which forms hydrogen bonds to the crown ether, with a coordinating K⁺ cation in 2Dy, ensuing changes in coordination and hydrogen bonding of water molecules in the structure.

The hydrogen bonding ‘belt’ around the [Dy(cdm)₆(H₂O)₁₄]⁻ anion, taken from the structure of 1Dy.

Structural data for 1Dy and 2Dy were obtained on KOALA, allowing the precise location of all hydrogen atoms to be determined using fully anisotropic models.

For more information: CrystEngCom (2014); DOI: 10.1039/C3CE42031K.
In Biomacromolecules we report experiments conducted at the Australian Synchrotron’s Infrared beamline to study phase separation in biopolymer blends biosynthesised at the National Deuteration Facility (NDF), using deuterated precursors from the Chemical Deuteration Labs at the NDF.

A major limitation in the use of infra-red microspectroscopy has been its reliance on overlapping peaks in the spectra to differentiate between polymers of similar chemical compositions in blends. This investigation demonstrates that biodeuteration of one mixture component provides the desired contrast in phase separated materials.

Deuterated poly(3-hydroxyoctanoate) was produced via microbial biosynthesis from deuterated substrates, and the characteristic C-D stretching vibrations provided distinct signals completely separated from the C–H signals of protonated poly(3-hydroxybutyrate) (PHB).

Phase separation was observed in 50:50 blends, as domains up to 100 microns through the film cross sections, consistent with earlier reports of phase separation observed by scanning electron microscopy of freeze-fractured protonated polymer blends. The presence of deuterated phases throughout the film suggests there is some miscibility at smaller length scales, which increased with PHB content. These investigations demonstrate that biodeuteration combined with IR represents a useful tool for mapping the phase behaviour of polymer blends. Further experiments will investigate the role of nanoparticles on polymer miscibility and suitability in biomedical applications.

The results of this collaborative work between the Institute and the University of New South Wales were accepted by Biomacromolecules (2013); DOI: 10.1021/bm4017012.

Sika (cold-neutron 3-axis spectrometer)

The first inelastic scattering data from SIKA of a crystal-field excitation in PrFeO₃.

In late January, the SIKA cold-neutron 3-axis spectrometer obtained its first inelastic neutron scattering spectrum of a crystal-field excitation in PrFeO₃. A fixed incident energy of 5.11 meV was used and the spectrum shows a scan of the final energy, with the excitation at an energy transfer of around 2 meV. Construction of SIKA has been funded in full by the National Science Council of Taiwan and will be operated by the National Synchrotron Radiation Research Centre. The next step is to submit the application to operate SIKA to the regulator, ARPANSA.

Joey (neutron camera)

The first successful crystal alignments have been performed on JOEY, our new neutron Laue diffraction camera. The purpose of JOEY is single-crystal characterisation and alignment prior to an experiment on a high-flux instrument, such as TAIPAN, KOALA, WOMBAT or SIKA, enabling users to optimise their experiments and thus help to save precious beamtime on their scheduled experiments. JOEY is designed in...
such a way that sample environments, such as standard cryostats or furnaces, can be placed on the instrument. This will allow for scientific data acquisition for standard diffraction experiments in addition to sample characterisation and alignment.

Neutron Laue diffraction patterns obtained on the crystal-alignment station, JOEY. Terbium Gallium Garnet: \( \text{Tb}_3\text{Ga}_5\text{O}_{12} \), along a) the (100) direction and b) the (110) direction; multiferroic \( \text{TbMn}_2\text{O}_5 \) along the c) a-axis and d) c-axis.

JOEY is equipped with a neutron camera for back scattering Laue diffraction and a 260 x 200 mm\(^2\) 6LiF:ZnS:Ag scintillating area detector. The optical signal is guided through tapered fibre-optics directly onto two Peltier cooled high resolution optical CCD cameras (1680 x 1320 pixels). The measured signal reflects the real diffraction pattern without any geometrical distortion of the optical beam. This allows for full diffraction analysis of the measured pattern. A Huber goniometer stage allows full x, y, z positioning and a 360° rotation. For sample alignment, a two-axis goniometer is installed which allows for tilt angles of up to 200°. The load capacity on the goniometer stage is 200 kg and can thus host standard sample environments available at the Bragg institute such as cryostats or furnaces.

**New Faces**

**Arrivals**

Anwen Krause-Heuer has joined the National Deuteration Facility with responsibility for the synthesis of deuterated molecules and the running and maintenance of our API 4000 mass spectrometer, an essential tool for determining deuterium levels in the synthesised molecules.

Chris Mehl rotates into the Institute from ANSTO-Engineering as part of the ANSTO graduate program. He will be working with Jamie Schulz assisting him in his role as operations manager for the four-month rotation period.

Nicholas Timperon joins us from ANSTO Health, as Laboratory Manager - he will be working to ensure that the laboratories are in top condition for visiting users. He will also be performing safety assessments on proposals.

Andrew Wildes is visiting the Bragg Institute on a 3 month sabbatical from the Institut Laue-Langevin in Grenoble, France. He is Australian and is familiar with ANSTO, having spent a good deal of time using the neutron instruments at HIFAR during his Ph.D at Monash University. He is carrying out neutron experiments while he is here and collaborating with our Institute scientists on two research projects: the magnetic structure and dynamics of two-dimensional honeycomb antiferromagnets; and the thermodynamics of the melting transition in DNA.

Britta Willenberg joins us from HZB Berlin and TU Braunschweig in Germany for 3 months. She will be working with Kirrily Rule, investigating the magnetic properties of Cu-O mineral linarite. Neutron experiments have been schedules, as well as synthetic crystal growth.

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