OPAL news

The OPAL Reactor and its cold-neutron source continued to perform very well during the period January to April 2010, with overall reliabilities of 98% and 96% respectively when compared to the published schedule.

Bragg Institute News

We are pleased to announce that our cold-neutron instruments QUOKKA and PLATYPUS are now back in full user service and that the backlog of proposals on these instruments has been cleared. Meanwhile, we congratulate the WOMBAT instrument scientists and users for achieving the instrument’s first century of refereed research papers.

Over the period 6 June – 12 September 2014, our TAIPAN thermal 3-axis spectrometer will be taken out of service for the planned integration of the ARC-funded beryllium-filter option on which will enable molecular-spectroscopy studies on TAIPAN. Simultaneously, the shielding wall between TAIPAN and the adjacent SIKA cold 3-axis instrument will be moved and extra dance-floor panels installed to increase the Q-range available on TAIPAN.

Around the instruments

**Polarised Helium-3 and WOMBAT** (high-intensity powder diffractometer)

In mid-April, the polarised $^3$He filling station installed in the Neutron Guide Hall successfully achieved a polarisation of $^3$He atoms in excess of 70% in spin-filter cells, and the first polarisation-analysis measurements were achieved on our WOMBAT high-intensity power diffractometer.

This is the first time that such neutron experiments have been performed outside of a few laboratories in the USA, Europe and Japan. The experiment used the “Pastis” coils and the wide-angle analyser cell, provided to us by the Institut Laue-Langevin in Grenoble, France.

![Incommensurate Magnetic Structure in Multiferroics](image)

**Polarisation-analysis data from WOMBAT.**

In one sample/field arrangement, we achieved a flipping ratio between 20 and 30, and in the other a flipping ratio of 7 (which is understood in terms of a ferromagnetic component in the sample).

Congratulations to Hal Lee, Tim D’Adam, Andrew Studer and Clemens Ulrich for leading the effort to achieve these first results.

**BILBY** (Time-of-Flight SANS)

The first pulsed-neutron spectrum as measured on BILBY.
Our Institute has begun commissioning one of the world’s most advanced neutron scattering instruments for the investigation of structures at the nanoscale, BILBY, our second SANS instrument that operates in a time-of-flight mode.

The Figure above shows the first measured neutron spectrum or distribution of neutrons versus their time of arrival at the detector demonstrating the peak intensity at a wavelength of ~4 Å, reflecting the spectrum provided by the Neutron Cold Source.

The BILBY small-angle neutron scattering instrument (right) adjacent to our first SANS instrument QUOKKA (left).

Congratulations to project manager Anna Sokolova, and everyone else involved in designing, constructing and installing BILBY which is the 3rd SANS instrument at OPAL, after QUOKKA and KOOKABURRA, and our 5th small-angle instrument (when one includes the 2 SAXS instruments).

QUOKKA (SANS)

Differential Scanning Calorimetry (DSC) is a thermoanalytical technique that enables phase changes, and the energy associated with such changes, in a sample to be identified by measuring heat flow as a function of temperature. This method is very widely used, but does not provide details as to the structural changes that may occur during phase transitions. Combined with SANS, DSC can yield valuable information of a broad range of materials including polymers and liquid crystals and specifically on the correlation between structural and thermal transitions.

Our design for a combined SANS-DSC system was published in the April edition of Measurement Science and Technology in collaboration with Benjamin Day of Hobsons Instrument Services, and the device is now available for users.

SANS data on absolute scale from equimolar C_{30}H_{62}:C_{36}D_{74} mixture throughout a heating profile below, during, and at the end of, each of the three endotherms.

Binary blends of normal alkanes – unbranched hydrocarbons – are solids at room temperature. When mixtures of the general formula C_{n}H_{2n+2}:C_{m}H(D)_{2n+2} are quenched from the melt to room temperature, the mixed system may undergo at one extreme, solid-state macrophase separation (C_{20}:C_{36}) and, at the other, indefinitely stable solid solutions (C_{34}:C_{36}). In between, incommensurate lamellar structures of different periodicities are formed in which the chain-length mismatch determines both the rate and extent of phase separation.

SAXS, SANS (in which one of alkanes was deuterated) and DSC have previously been
used separately to study the influence of chain-length difference and molar composition and confinement on the dimensions of the superstructure, and the associated phase changes occurring during heating and cooling of the ‘demixed’ material. As SANS is able to detect phase separations at an earlier stage (minutes instead of days) relative to synchrotron-based SAXS experiments, since the contrast between alkanes can be increased with isotopic labelling, enabling the parent repeat of the ‘demixed’ superstructure to be observed. These materials thus form the basis for a long-standing motivation for the development of a combined SANS and DSC capability.

The Figure above shows the recently obtained time-evolution of SANS data from a demixed equimolar C₃₀H₆₂:C₃₆D₇₄ mixture that had been aged for approximately two-and-a-half years prior to conducting simultaneous SANS and DSC measurements, representing a true single shot experiment. The data shown have been collected continuously in event-mode acquisition. Three phase transitions are observed: the first endotherm is absent both on cooling (and subsequent re-heating) and is associated with the mixing transition of the demixed phase; the second and third endotherms are associated with an orthorhombic to rotator (solid-solid), Tₗₒ, and rotator to melt, Tₘ, transitions respectively.

National Deuteration Facility & ECHIDNA
(high-resolution powder diffractometer)

The arrangements of the interpenetrating nets in each MOF-14 unit cell.

Negative thermal expansion (NTE), the contraction of a material with increasing temperature, is an unusual property found in a small number of materials. This unusual behaviour has been characterised in a number of metal-organic framework materials (MOFs), and framework interpenetration is typically found to reduce the extent of NTE.

In work reported in Angew. Chem., researchers from the University of Sydney and the Bragg Institute have utilised single-crystal X-ray diffraction at the Australian Synchrotron MX-1 beamline, deuterated 4,4′,4″-benzene-1,3,5-triyl-tribenzoic acid produced by the National Deuteration Facility, and the Institute’s high-resolution neutron powder diffractometer ECHIDNA to investigate the mechanism for negative thermal expansion in Cu₃(btbtb)₂ (MOF-14, btbtb=4,4′,4″-benzene-1,3,5-triyl-tribenzoate), a MOF that exhibits an anomalously large NTE effect.

Diagram showing an exaggeration of the contraction-induced biconvex distortion of the face-to-face btbtb ligands.

Temperature-dependent structural analysis shows that, contrary to other interpenetrated materials, in MOF-14 the large positive thermal expansion of weak interactions that hold the interpenetrating networks together results in a low-energy contractive distortion of the overall framework structure, demonstrating a new mechanism for NTE.

The resulting temperature-dependent fractional unit-cell evolution of MOF-14 obtained by powder neutron diffraction (hollow diamonds), synchrotron powder X-ray diffraction (black circles) and synchrotron single-crystal X-ray diffraction (hollow triangles). Due to absolute value offsets, all values are normalized to 100 K.

Inset: Structural picture of the cubic interpenetrated structure.
Proposal Round 2014-2 Summary

Our 2014-2 proposal round closed with 258 proposals across 10 neutron instruments and both Chemical- and Bio-deuteration at the National Deuteration Facility. A total of 1325 beam days were requested across all instruments, with an overall success rate of 48%. 55% of demand was from Australian universities and CSIRO and 16% from ANSTO itself. Roughly 45% of demand in the 2014-2 Round was from overseas (Taiwan, Japan, New Zealand, UK, China, USA, Hong Kong, France, India, South Africa, Germany, Netherlands, Spain, Italy, Singapore, Sweden, Switzerland and South Korea).

Announcements

The 2015-1 Proposal Round is open for beam time between January and June 2015 and access to all 13 neutron-beam instruments, chemical- and bio-deuteration. Proposals should be submitted online by 15 September 2014 via https://neutron.ansto.gov.au.

Crystallography365

Blogging a Crystal Structure a day in 2014

To increase public awareness of the science of crystallography and its contribution to our understanding of the very basis of life, 2014 has been designated by the United Nations as ‘International Year of Crystallography’. To celebrate this proclamation, our own WOMBAT instrument scientist Helen Maynard-Casely has created the Crystallography365 blog with the ambitious aim of blogging a crystal structure a day in 2014.

New Faces

Arrivals

Natalia Davydova joins us as a Molecular biologist at the National Deuteration Facility. Natalia will be responsible for the molecular cloning of different protein constructs used for biodeuteration of molecules to be utilised for neutron experiments, NMR and other techniques.

Mark Reid is co-responsible for the KOWARI strain scanner. Mark was previously a post-doc at the University of Wollongong within the Materials Process Engineering group. His main research activities involve residual-stress analysis and texture measurement using neutron diffraction. In addition he has a strong interest in neutron imaging and in situ neutron diffraction of material processes such as thermo-mechanical processing, sintering and welding.

Andrew Manning joins the Sample Environment group. He recently completed a PhD in physics at the Australian National University.

Departures

The institute wishes to thank Brett Wheeler and Jason Christoforidis for their excellent contributions to the design and construction of the second suite of neutron instruments and wishes them all the best in their new roles at ANSTO!

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