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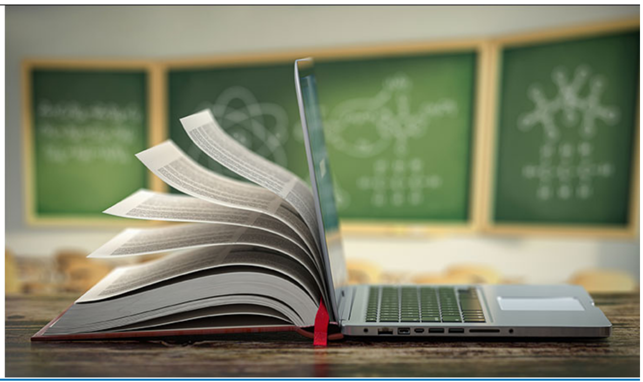

Neutron scattering at the OPAL research reactor

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Abstract. The current suite of 14 neutron scattering instruments at the multipurpose OPAL research reactor is described. All instruments have been constructed following best practice, using state-of-the-art components and in close consultation with the regional user base. First results from the most recently commissioned instruments match their design performance parameters. Selected recent scientific highlights illustrate some unique combinations of instrumentation and the regional flavour of topical applications.

1. Introduction

Australian neutron scattering leapt into the 21st century with the start up of the 20 MW OPAL nuclear research reactor at the Australian Nuclear Science and Technology Organisation (ANSTO) in 2006. OPAL is unique amongst modern high-power research reactors in that it is multipurpose, being used to produce medical isotopes and exploited for neutron transmutation doping and neutron activation analysis, as well as being used to provide beams for neutron scattering. While the multipurpose nature led to some compromises in the core design for the neutron-beam instruments, it brought with it valuable political and economic security. Both OPAL and its cold-neutron source, the running of which is not linked intimately to the operation of the reactor unlike at most research reactors, now operate consistently for around 300 days per year - an impressive statistic amongst modern research reactors.

The major part of the initial success at OPAL has been in crystallography, carrying on the excellent tradition established since the late 1950s at the HIFAR reactor at Lucas Heights. HIFAR had an illustrious career in itself. As well as providing a materials research capability for the (then) projected nuclear power programme in Australia, it was also the training ground for many young scientists that went on to distinguished careers in Australia and abroad. Hugo Rietveld and Alan Hewat of neutron powder diffraction (NPD) fame were just two of these, although it is noteworthy that their first neutron experiments were in single-crystal diffraction [1] and triple-axis spectrometry [2] respectively, since the power of NPD in materials discovery was yet to be realised, much less appreciated.

As befits a national facility, the neutron-scattering science at OPAL is largely driven by the external user programme, through a peer-review proposal system for access to the neutron-beam instruments. Fee-for-service commercial use is growing with the present target being 5% averaged over the full suite. Certain scientific themes, where neutrons offer distinct advantages as a research tool, are also promoted within the Bragg Institute, the entity that runs the neutron-scattering programme. These themes currently include energy materials, thermo-mechanical processes, magnetism, food science, biomolecules, and cultural heritage.

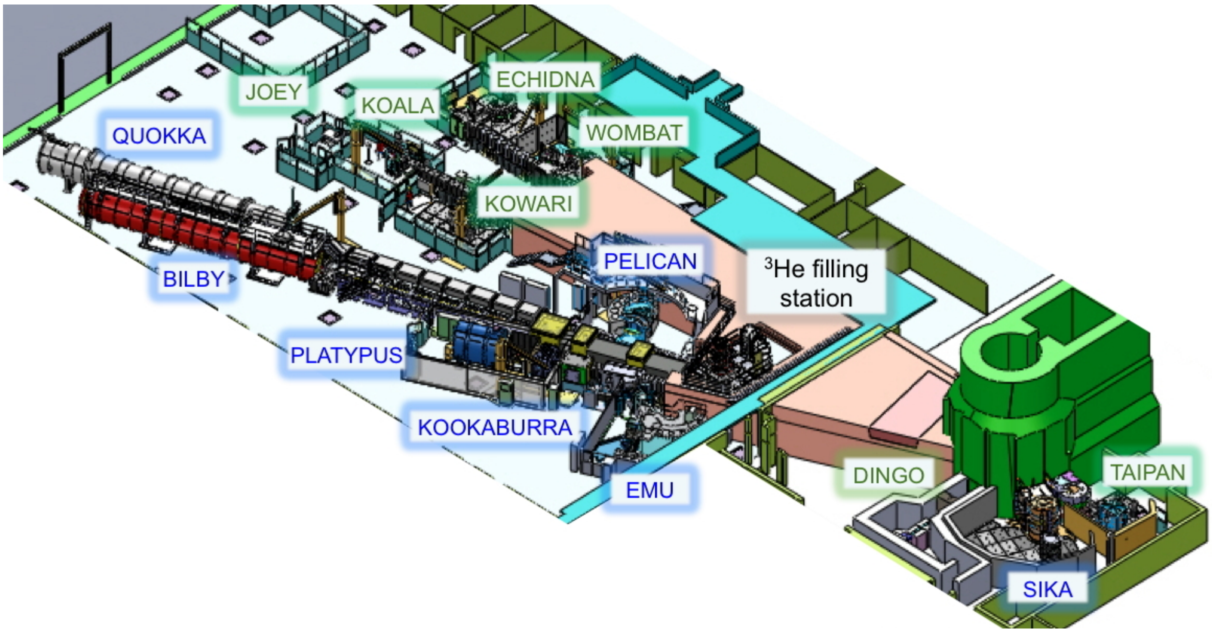

Figure 1: Schematic of the layout of the neutron-beam instruments at OPAL. DINGO, SIKA, and TAIPAN are located close to the reactor face, the other instruments are on neutron guides. Green text denotes the instruments on thermal neutron beams, blue text those on cold neutron beams. Also indicated is the convenient location of the ^{3}He spin-filter filling station atop the neutron guide bunker.

Here we introduce the OPAL neutron-beam facility, show the first results from the instruments most recently brought on-line, and describe recent scientific highlights allied closely to the new or unique instrumentation.

2. Neutron-beam instruments at OPAL

The construction of the neutron-beam instruments at OPAL has proceeded in three main tranches. The first, called *Neutron Beam Instrument Project - 1* (NBI-1), produced the first seven instruments [3] as part of the full funding for the reactor construction. The second, denoted *Major Capital Works* with funding from various internal and external sources, yielded four further instruments. Most recently, the *Neutron Beam Instrument Project - 2* (NBI-2) funded as part of the Super Science Initiative of 2009, has added three more [4], to bring the total number of instruments to 14. Figure 1 shows the layout of the current suite of neutron-beam instruments at OPAL, and table 1 describes each instrument with its key features and the year that it entered user operation.

Based largely on user wishes, the suite of instruments is fairly conventional and constructed using proven components following best practice elsewhere, to produce state-of-the-art instruments, but occasionally in unique combinations of components and with unique applications. One example is the time-of-flight spectrometer PELICAN, which has been designed from the outset to offer a full polarisation analysis capability using a supermirror polariser before the sample, a PASTIS insert [5] around the sample, and a wide-angle ^{3}He spin-filter cell after the sample, a worthy successor to the long-wavelength polarised-neutron spectrometer LONGPOL [6], which gave HIFAR a unique capability for several decades.

The cold-neutron triple-axis spectrometer, SIKA, constructed by National Central University, Taiwan, and just recently awarded its operating licence, has been presented elsewhere at

Table 1: Neutron-scattering instruments at OPAL in 2015.

Instrument	Description	Features	In user program	Reference[a]
Diffraction				
ECHIDNA	High-resolution powder diffractometer	128-element multi-tube detector, robot sample changer	2008	[7]
JOEY[b]	Neutron Laue camera for single-crystal alignment	Fast-read-out CCD detector; precision sample table that supports 200 kg	-	[8]
KOALA	Thermal neutron Laue diffractometer	Sample volumes of typically 0.1 mm^3	2008	[9]
WOMBAT	High-intensity (powder) diffractometer	Large area detector, optional radial oscillating collimator	2008	[10]
Inelastic scattering				
EMU	High-resolution backscattering spectrometer	Nearly exact backscattering at low Q	2016[c]	
PELICAN	Time-of-flight spectrometer	Designed at the outset for optional polarisation analysis	2014	[11]
SIKA	Cold-neutron three-axis spectrometer	Choice of single or multi-analyser	2015	
TAIPAN	Thermal-neutron three-axis spectrometer	Optional Be-filter mode	2010	[12]
Small-angle scattering				
BILBY	Time-of-flight small-angle neutron-scattering (SANS) instrument	Large Q range for kinetic studies	2016[c]	
KOOKABURRA	Ultra-small-angle neutron-scattering instrument	Bonse-Hart geometry with choice of two wavelengths	2014	[13]
QUOKKA	Pinhole SANS instrument	Large area detector; refractive lens; polarised-neutron options	2009	[14]
Nano- to macro-scale imaging				
DINGO	Neutron imaging instrument	High-flux and high-resolution modes	2014	[15]
KOWARI	Strain scanner	Large-volume samples up to 1 t; texture studies	2008	[16]
PLATYPUS	Neutron reflectometer	Polarised-neutron options	2009	[17] [18]

[a]Additional details of each instrument can be found at [19]; [b]Accessible on an *ad hoc* basis; [c]Anticipated

ECNS 2015 by Dr Jason Gardner. SIKA is now operated by the National Synchrotron Radiation Research Center (NSRRC), Taiwan. The NSRRC team [20] at ANSTO is not just an outstation but is in many respects an integral part of the neutron-scattering program at ANSTO. A polarised-neutron capability using ^{3}He spin filters is currently being introduced on five instruments - PELICAN, QUOKKA, SIKA, TAIPAN, and WOMBAT - and added as an option to the capability already possible using polarising super-mirrors on PLATYPUS. Observation of the first scattered neutrons is an exciting time for the instrument construction teams, and figure 2 shows the first results from the most recently commissioned instruments or capabilities.

The high-resolution powder diffractometer, ECHIDNA, with 51 papers in 2014, has now achieved a publication rate amongst the world's best comparable instruments, and several other OPAL instruments are not far behind in such comparisons.

There is space for several additional instruments in the present guide hall, mostly by installation or extension of one each of additional thermal and cold neutron guides, the exact number of instruments depending on the types finally installed. There is also space directly opposite the present guide hall for a second guide hall, and for installation of a hot source if user demand warrants it. Just as for the first suite of instruments, the choice of additional instruments will be made in close consultation with the potential user base [21]. Such an approach has been fully vindicated by the consistent over-demand for beam time on the existing instruments.

The Bragg Institute currently includes the National Deuteration Facility (NDF), which produces a range of deuterated molecules, including lipids, proteins, biopolymer, aromatics, detergents, and sugars, principally for diffraction, SANS, and reflectometry experiments. The NDF also produces samples for other research techniques, such as NMR, and to study the effect of isotopic substitution itself on properties [24]. There is also a small computing cluster and limited atomistic-modelling support, as well as X-ray small-angle scattering and reflectometry instruments to complement the neutron user program, and most recently a Physical Properties Measurement System (Quantum Design Inc.).

The main commercial use of neutron beam time is for strain scanning of large pipes, rail components, and welds in general, with a growing interest in strain analysis of additive manufacturing, and radiography of cement samples. An interesting observation from our industrial program is that smaller companies are keen to advertise their use of the neutron-beam instruments to validate their work or process. The Bragg Institute Industrial Liaison Office [23] coordinates industrial activities, including products like deuteration at the NDF and software, in addition to neutron beam time.

3. Recent scientific highlights

Each set of authors of overviews of the OPAL neutron-beam instruments brings their own slant. Being the current research leaders in the Bragg Institute we focus on a small selection of the science that the new capabilities explore with emphasis on the instrumentation where OPAL is at the forefront and on topical regional applications.

3.1. 'Dynamic' sample environment

WOMBAT was the scene for the world's first *in situ operando* experiments on a functioning battery just five years ago [25], and ANSTO continues to play a leading role in *in situ* neutron diffraction on Li-ion batteries, producing 46% of the publications worldwide in this area since then.

'Dynamic' *in situ* experiments of a softer kind have also been pioneered by adapting a Rapid Visco$^{\mathrm{TM}}$ analyser to QUOKKA to study simultaneously structural and viscosity changes during cooking of starch[26], diffraction studies of crystallisation of the triglyceride, tripalmitin, under shear force using a rheometer on WOMBAT, and simultaneous differential scanning calorimetry

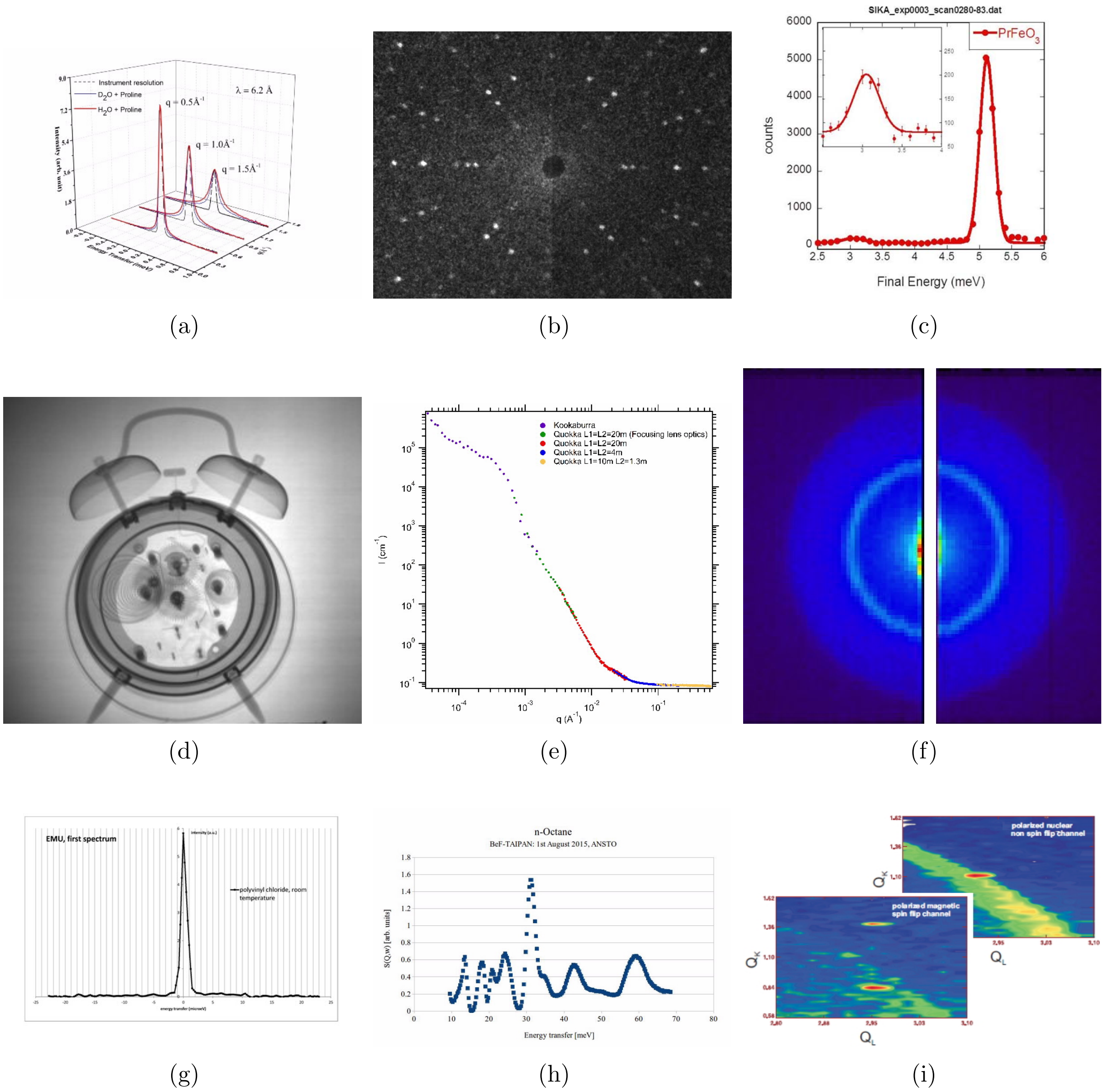

Figure 2: First results from the latest tranche of neutron-beam instruments or capabilities at OPAL: a) Quasi-elastic neutron scattering from 9.1% proline in H_2O/D_2O on PELICAN [22]; b) Laue pattern from $TbMn_2O_5$ on JOEY; c) Crystal-field excitation from $PrFeO_3$ recorded on SIKA; d) Radiograph of an alarm clock on DINGO; e) Extension of the Q range in small-angle scattering to 4 x 10^{-5} Å^{-1} enabled by KOOKABURRA; f) Small-angle scattering from silver behenate on BILBY; g) Energy-transfer resolution on EMU determined by an energy scan from polyvinyl chloride; h) Vibrational density-of states spectrum for octane from the Be-filter option on TAIPAN; i) Polarisation analysis in the magnetic phase of a multiferroic single-crystal sample using a ^{3}He spin filter on WOMBAT.

(DSC) and SANS on QUOKKA [27]. The signal in the last experiments, which monitored the periodicity of generally incommensurate lamellar structures of a binary blend of alkane molecules of different length, was enhanced by deuteration of one of the alkanes to give a considerable contrast advantage over similar studies with X-rays.

3.2. Chemical crystallography

With the regrettable withdrawal of the pioneering thermal-neutron Laue diffractometer, VIVALDI [28], from the user program at the Institut Laue-Langevin, KOALA, essentially a clone of VIVALDI, is now the sole reactor-based thermal-neutron Laue diffractometer accessible to external users, yet is leading the world in application of neutron diffraction to the study of challenging hydrides [9]. One surprising recent example is the 28 copper 15 hydride *Chinese puzzle molecule* found to form when certain copper compounds are mixed with a hydride of boron [29]. This new structure has alternating layers of hydride and copper wrapped in an outer shell of protecting dithiocarbamate molecules, and is much more stable than many other hydride compounds, which has promising implications for nanoparticle formation.

3.3. Spintronics

Not surprisingly, magnetism is a common theme on the majority of instruments, with transition-metal and rare-earth magnetism in bulk samples being strong favourites. There is also a growing effort in studies of thin-film magnetism, exploiting the tunability allowed by doping, strain, and proximity effects, of properties of importance for real applications. Reflectometry on PLATYPUS and diffraction on TAIPAN are the favoured techniques, often aided by structural characterisation using the in-house X-ray reflectometer. Recently, diffraction experiments on TAIPAN revealed that thin films of $SrCoO_{3-\delta}$ on a $DySrO_3$ substrate, which provides large epitaxial linear strain, undergo a strain-induced ferromagnetic-to-antiferromagnetic phase transition unlike similar films on the better-lattice-matched $SrTiO_3$ substrates [30]. Films as thin as 20 nm, a new record for successful neutron diffraction experiments, were necessary to maintain the strain through the film.

3.4. Granular materials

Granular materials are a unique form of matter that can act in a similar manner to a solid, liquid, or gas, depending on the circumstances. In quasi-static systems their ability to flow and support static shear stress provides for interesting physical behaviour. The constitutive response of granular materials is highly non-linear and incremental (historical) and has provided an active area of study for physicists and engineers for hundreds of years. Far from being of purely academic interest, the ability to model the response of granular materials is of high relevance to the handling of bulk commodities such as coal (4.72 tonnes 'flow' through Newcastle in New South Wales, Australia, each second), cereal grains, and mineral sands.

The full triaxial stress state within individual particles in a monodisperse spherical granular assembly has recently been measured for the first time [31]. This was made possible by neutron imaging and computed tomography on DINGO combined with neutron diffraction strain measurement techniques and associated stress reconstruction using KOWARI. The assembly in question consisted of 549 precision steel ball bearings under an applied load of 85 MPa in a cylindrical die. Clear evidence of force chains was observed in terms of both the shape of the probability distribution function for normal stresses and the network formed by highly loaded particles.

3.5. Imaging ancient Australian fauna

One could be forgiven for thinking that Australian fauna still hark back to the days of dinosaurs, but it is little appreciated that real dinosaurs did roam the southern continent and that there exist several quite important dig sites. The most significant of these are around Winton in outback Queensland, where the first bones were unearthed just 12 years ago. Ada Klinkhamer and David Elliot of the *Australian Age of Dinosaurs* Museum in Winton have been working with the DINGO scientist, Joseph Bevitt, to explore the possibilities of neutron radiography for non-invasive examination of rocks from the Winton area to detect ancient fossils. It can be hit or miss, but occasionally beautiful images like that in figure 3 can be teased out.

4. Instruments of communication: Public outreach

ANSTO is very conscious of the need to communicate our scientific and commercial activities to the general public. The external-communications group and our Discovery Centre are very proactive in outreach to the general public, having stalls at local fairs and celebration days, sponsoring groups from ANSTO in local charity sporting events, and conducting tours of the site including visits to the viewing platform overlooking the reactor guide hall. Figure 4 shows a recent billboard advertisement, and similar advertisements were posted on local bus shelters and on buses themselves.

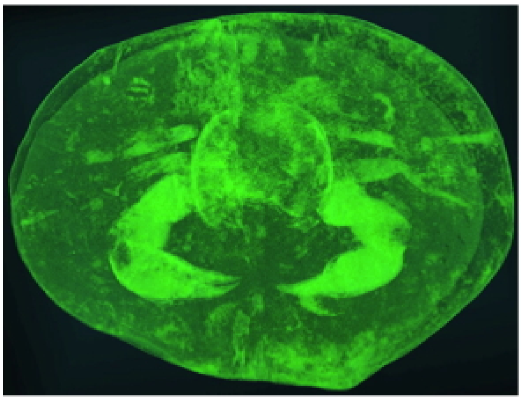

Figure 3: Non-invasive neutron radiography on DINGO reveals the outline of *Torynomma quadrata*, a cretaceous crab.

Figure 4: A Sutherland Shire billboard inviting the general public to visit ANSTO and the neutron-beam facilities.

Several groups of school classes, or community groups such as senior citizens, visit the site each week day, and the general public can register for guided tours on Saturdays. Annually, there are over 15,000 such visitors, with 11,000 in school excursions. The net result is that the nuclear-based activities at ANSTO are predominantly seen in a favourable light by the general public, and with considerable pride by the citizens of Sutherland Shire, where ANSTO is located.

The communications group also aids ANSTO scientists to create media and demonstration tools, such as the recent video that shows how EMU works [32] and a LEGO robotics model of TAIPAN [33], the latter in collaboration with Macquarie University.

5. Conclusions

The combination of state-of-the-art instrumentation, support facilities, expert scientific staff, and enthusiastic users of OPAL has yielded an impressive series of scientific results, as well as a fledgling industrial programme. The Bragg Institute's scientific projects are effective interfaces with the user community, have international impact, and often local public appeal. With currently 13 neutron-scattering instruments in the user programme, including a radiography/tomography instrument, all run by very expert, talented scientists and engineers, ANSTO is well on the way to achieving its commitment to make OPAL one of the top three research reactors for neutron scattering in the world.

Acknowledgments
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