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**NEUTRON SCATTERING
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Book of Abstracts

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Plenary / 88

The present and future of neutron scattering for the characterisation of key functional materials

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Properties and functions of materials are determined by the structure and its evolution on every relevant time, length, field and energy scale. To understand these, neutrons play a critical role in providing important insights into the structure of polar functional materials as it responds to stimuli. In this talk, I will present a summary of recent progress in neutron scattering studies of piezoelectric, anti/ferroelectric and multiferroic materials, with a special focus on the investigation of neutron diffraction conducted under different fields (e.g. temperature, magnetic/electrical field and pressure). I will also report structurally dynamic behaviours of zeolites and metal-organic frameworks (MOFs) investigated by using time-of-flight inelastic neutron scattering and comment on the role neutrons play in gaining new insights into the properties of a broad range of key materials for practical application. I will then discuss overcoming challenges and technical difficulties to attain precision neutron analyses, and give my personal perspective as a user on the continuous development of instruments and methods needed for neutron scattering studies in the future.

Topic:

Topical Session 1: Advanced Materials / 91

Magnetic Interface Phenomena in Nano-Architectures and their Applications

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Interfaces between heterostructure components in nanoscale films play important roles in communicating low-dimensional phenomena and act as anchor points for the direct control and tunability of device performance. In this talk I will give an overview of our group's recent investigations into the occurrence of magnetic interface phenomena in low-dimensional thin-film systems which have conceivable utility in future condensed-matter technologies. First, the magnetic interface quality of an FePt3 nano-magnet formed via ion-induced chemical disorder will be analysed [1]. Here, neutron and electron measurements used in combination with density functional theory calculations reveal a rather counterintuitive result which could prove beneficial towards the development of ultra-high density magnetic recording devices. In a second study, the layer-averaged static magnetisation and macroscopic magneto-dynamic behaviours of a Co/Pd bilayer during hydrogen-gas cycling are analysed. To perform this characterisation, we first had to develop and commission an original sample environment which innovatively combines polarised neutron reflectometry and microwave spectroscopy [2]. The Co/Pd interface is found to feature tailorable magnetic surface anisotropy in the

presence of hydrogen gas – the mechanism of which could act as a safety switch in next-generation vehicles powered by hydrogen.

[1] G. L. Causer, D. L. Cortie, H. Zhu, M. Ionescu, G. J. Mankey, X. L. Wang and F. Klose. *ACS Appl. Mater. Interfaces*, 10, 16216-16224 (2018)

[2] M. Kostylev, G. L. Causer, C.-H. Lambert, T. Schefer, C. Weiss, S. J. Callori, X. L. Wang and F. Klose. *J. Appl. Cryst.* 51, 9-16 (2018)

Topic:

Advanced Materials

Topical Session 1: Advanced Materials / 7

Theoretical Study of a Family of Lanthanoid-Dioxolene Single-Molecule Magnets

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Lanthanoid Single-Molecule Magnets (SMMs) are molecular materials that exhibit slow relaxation of the magnetization of molecular origin, thus making them promising targets for the development of spintronic devices and molecular memories. Since the electronic and magnetic properties of lanthanoid-based SMMs are strongly dependent on the characteristics of the electrostatic crystal field induced by the ligands on the lanthanoid ion, a thorough understanding of such magneto-structural correlations is crucial to develop molecules displaying SMM behavior at sufficiently high temperatures to warrant commercial applications. For this reason, *ab initio* calculations have proven to be valuable tools to elucidate the details of the electronic structure of SMMs and improve the understanding of their effect on magnetic properties and relaxation mechanisms.

In this work, we have performed a set of *ab initio* calculations on the family of molecules [Ln(bpy)₂(Cl₄Cat)(Cl₄CatH)](M) (Ln = Tb, Dy, Ho), employing the CASSCF/RASSI-SO method, and we have compared the predicted electronic and magnetic properties with the experimental data. These molecules, recently synthesized, are expected to display SMM behavior due of their structural similarity to other SMMs previously described in literature, with their low-lying energy spectrum determined with Inelastic Neutron Scattering (INS) for Ln = Tb, Ho. We show that there is a good agreement between computational and experimental results, thus confirming the validity of theoretical predictions of electronic and magnetic properties of lanthanoid-based SMMs.

Topic:

Physics

Topical Session 1: Advanced Materials / 85

Investigation of phonon dynamic in single crystal lead-halide perovskites by inelastic neutron scattering

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The lead halide perovskite materials have recently risen to prominence for remarkably high photovoltaic efficiencies in polycrystalline materials that are highly defected [1]. Some of the reasons for this good defect tolerance are the very low exciton binding energy and consequent highly delocalized electrons and holes leading to high mobilities in these materials, coupled to low thermal conductance. Other recent work by our group has shown long lifetimes for hot carriers in a range of perovskites with organic lead iodide perovskites having the longest lifetimes [2]. In order to explain those rather promising physical properties, a closer investigation of phonon dynamics is needed. Although ab initio simulations (DFT) can predict phonon dispersions to a reasonably accurate extent (comparison between different phonon modes) [2, 3], scaling their energies to actual phonon energies (particularly at high momenta near the zone edge) can be rather inaccurate. In order to obtain a detailed phonon dispersion to overcome the limits of the ab initio methods, inelastic neutron scattering techniques can be used (as they offer full Brillouin zone mapping and are suitable for large single crystal samples).

The thermal triple axis spectrometer (TAS) on TAIPAN at OPAL reactor at ANSTO was used with the aim to map phonon dispersion of single crystal Methyl Ammonium Lead Halide Perovskites, CH₃NH₃PbBr₃ and CH₃NH₃PbI₃ [4]. The alignment was performed so that we could scan through [h k l] and [0 k l] planes in reciprocal space for MAPbBr₃ and MAPbI₃ samples, respectively. Assuming the cubic space group, ABX₃, for the MAPbBr₃ sample, we were able to perform transverse and longitudinal scans along each high symmetry direction in the Brillouin zone (Γ -X and Γ -M) which would ensure the mapping of all phonon modes in the h k plane of the Brillouin zone.

The full phonon dispersions that will be obtained with this method can be of great significance as by adding up to the already simulated data we will be able to have a deeper insight into the underlying physics that is responsible for the previously observed properties (such as significantly extended hot carrier lifetimes) where phonon dynamics will play a significant role.

Topic:

Advanced Materials

Topical Session 1: Advanced Materials / 40

Solid Ionic Conductors for Energy Applications: Developing a Complete Picture from Structure and Dynamics

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There has been renewed interest in solid state sodium-ion batteries (SIBs) as a safe, sustainable and cost-effective alternative system for large scale energy storage applications.[1] This, in turn, has motivated many studies on the development of materials that facilitate high ionic conductivity over

multiple charge-discharge cycles. Layered sodium manganates and the NASICON family of compounds are promising candidate sodium electrode and solid-state electrolyte materials respectively. In both cases, it has been shown that the overall performance of these materials for their respective functions is significantly improved through structural modifications, including by hydration or chemical doping.[2-8] However, the characterisation of these materials are typically limited to techniques which only offer a macroscopic picture, such as electrochemical impedance spectroscopy. As such, direct links between conductivity and structure, particularly with reference to the effect of chemical doping on the microscopic properties of materials are rarely investigated.

We have selected candidate materials which have been shown to be amongst the best performing for their purpose and use high resolution diffraction data to solve their average structure. In parallel, we use quasielastic neutron scattering spectroscopy to gain insight into the diffusion mechanisms at an atomic level. We consequently aim to form a fuller picture of the effects that structural modifications have on the ionic conductivity and hence overall performance of these materials.

Palomares, V., et al., Na-ion batteries, recent advances and present challenges to become low cost energy storage systems. *Energy & Environmental Science*, 2012. 5(3): p. 5884-5901.
Han, M.H., et al., High-Performance P2-Phase $\text{Na}_{2/3}\text{Mn}_{0.8}\text{Fe}_{0.1}\text{Ti}_{0.1}\text{O}_2$ Cathode Material for Ambient-Temperature Sodium-Ion Batteries. *Chemistry of Materials*, 2016. 28(1): p. 106-116.
Han, M.H., et al., Moisture exposed layered oxide electrodes as Na-ion battery cathodes. *Journal of Materials Chemistry A*, 2016. 4(48): p. 18963-18975.
Jolley, A.G., et al., Improving the ionic conductivity of NASICON through aliovalent cation substitution of $\text{Na}_3\text{Zr}_2\text{Si}_2\text{PO}_{12}$. *Ionics*, 2015. 21(11): p. 3031-3038.
Khakpour, Z., Influence of M: Ce^{4+} , Gd^{3+} and Yb^{3+} substituted $\text{Na}_{3+x}\text{Zr}_2\text{-xM}_x\text{Si}_2\text{PO}_{12}$ solid NASICON electrolytes on sintering, microstructure and conductivity. *Electrochimica Acta*, 2016. 196(Supplement C): p. 337-347.
Samiee, M., et al., Divalent-doped $\text{Na}_3\text{Zr}_2\text{Si}_2\text{PO}_{12}$ sodium superionic conductor: Improving the ionic conductivity via simultaneously optimizing the phase and chemistry of the primary and secondary phases. *Journal of Power Sources*, 2017. 347: p. 229-237.
Song, S., et al., A Na^{+} Superionic Conductor for Room-Temperature Sodium Batteries. *Scientific Reports*, 2016. 6: p. 32330.
Nam, K.W., et al., Critical Role of Crystal Water for a Layered Cathode Material in Sodium Ion Batteries. *Chemistry of Materials*, 2015. 27(10): p. 3721-3725.

Topic:

Advanced Materials

Topical Session 1: Advanced Materials / 14

COMBINING X-RAY AND NEUTRON DIFFRACTION AND MODELLING FOR BETTER UNDERSTANDING ADVANCED MATERIALS

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Many advanced materials, such as thermoelectrics, phosphors for light emitting diodes, electrodes and solid electrolytes for batteries, etc. are difficult objects for stand-alone crystal structural analysis based on diffraction techniques due to intrinsic high disorder of one of the sublattices. Traditional diffraction data analysis based on atom-centric models with explicitly declared atomic positions is often unstable or unable to fully capture all the details due to correlations between variables. Additional difficulties arise from the limitations of X-ray diffraction in locating light elements and distinguishing elements with close atomic numbers (e.g. Mn/Ni/Ci). Combining X-rays with neutrons and traditional diffraction data analysis with other approaches, such as Maximum Entropy Method, and atomistic modelling and theoretical symmetry analysis allows to paint a more complete picture. I will illustrate the point using our recent studies of several such structurally complex systems, such as NASICON and P2-types and phosphor polyanion frameworks. All of them have been studies for

decades and yet complementing experiment with theory and modelling revealed new features which help understand and improve properties.

Topic:

Advanced Materials

Topical Session 2: Chemistry / 62**Structural Design of Ionic Liquids for Biomass Processing**

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Ionic liquids (ILs) are molten salts at room temperature. ILs possess remarkable physio-chemical properties including high solubility of compounds that are otherwise insoluble in molecular solvents. One important application is the dissolution of lignin during pretreatment of biomass. Previous studies have shown low-cost and biocompatible ILs containing cholinium as the cation and an amino acid as the anion. Cholinium lysinate (ChLys) demonstrates the highest potential as a lignin processing solvent, with low viscosity and high selectivity at ambient temperature. The treatment is effective with up to 20% water.

Time-of-flight neutron diffraction directly measures the structure of ILs and its mixtures. Using isotopic substitution of hydrogen by deuterium, we simultaneously fit a series of diffraction patterns into a simulation method called Empirical Potential Structure Refinement (EPSR). This method experimentally refines a simulation box to describe liquid structure at the nano- and atomic scale. Our study investigates the structure of amino-acid ILs, with and without the presence of water. We use guaiacol (2-methoxy-phenol) as a model aromatic residue of lignin to identify key solvent-solute interactions. Bio-based ILs can be nanostructured. Results show pronounced nanostructure of ChLys before and after the addition of water. The added water molecules form domains at the nanoscale, without disrupting the amphiphilic nanostructure of ILs. Based on key intermolecular interactions, we present a framework for the design of IL structure to minimize competition and to enhance driving forces for aromatic extraction.

Topic:

Chemistry

Topical Session 2: Chemistry / 70**Structural investigation of the disordered Cu_{3-x}SbO_{5.5+y} ternary oxide**

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Many different electrolytes with perovskite and fluorite-type structures have been extensively explored and characterised. Minimal focus has been given to potential Bixbyite-type electrolytes. A ternary copper antimony oxide previously referred to as Cu₉Sb₄O₁₉ with an unresolved crystal structure looked like a promising candidate to study. This was synthesised using classic solid state techniques under high temperature and oxygen pressure. Refinement of lab X-ray diffraction data strongly indicated that this material was isostructural with Cu₃TeO₆ with a curiously large degree of oxygen vacancies. Refinement of neutron diffraction patterns revealed the system was much more complicated than first anticipated, confirming the under occupancy of oxygen and revealing the mixed occupation of Cu²⁺/Sb⁵⁺ sites in the cationic sub-lattice. The final composition can be refined to Cu_{3-x}SbO_{5.5+3x/2}. X-ray absorption experiments confirmed the local coordination significantly differs from Cu₃TeO₆ complimenting the diffraction data. This is an interesting material in a good position for many potential applications like; oxygen membranes, sensors or as a cathode/electrolyte material warranting further investigation.

Topic:

Chemistry

Topical Session 2: Chemistry / 33**Drug-induced morphology transition of self-assembled glycopolymers: Insight into the drug-polymer interaction**

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It is often assumed that a hydrophobic drug will be entrapped in the hydrophobic environment of a micelle. Little attention is usually drawn to the actual location of the drug and the effect of the drug on properties. In this publication, we show how the chosen drug curcumin is not only unexpectedly located in the shell of the micelle, but that the accumulation in the hydrophilic block can lead to changes in morphology during self-assembly. A block copolymer poly(1-O-methacryloyl-β-D-fructopyranose)-b-poly(methyl methacrylate), Poly(1-O-MAFru)₃₆-b-PMMA₁₉₂, was loaded with different amounts of curcumin. The resulting self-assembled nanoparticles were analyzed using TEM, SAXS, and SANS. Initial microscopy evidence revealed that the presence of the drug induces morphology changes from cylindrical micelles (no drug) to polymersomes, which decreased in size with increasing amount of drug (Figure 1). SAXS and SANS analysis, supported by fluorescence studies, revealed that the drug is interacting with the glycopolymer block. The drug did not only influence the shape of the drug carrier, but also the level of hydration of the shell. Increasing the amount of drug dehydrated the nanoparticle shell, which coincided with a lower nanoparticle uptake by MCF-7 breast cancer cells and non-cancerous Raw-264.7 cells. As a result, we showed that the drug can influence the behaviour of the nanoparticle in terms of shape and shell hydration, which could influence the performance in a biological setting (Figure 1). Although the depicted scenario may not apply to every drug carrier, it is worth evaluation if the drug will interfere in unexpected ways, for example, when the drug locates on the surface and affects the internal structure of the nanocarrier.

Topic:

Chemistry

Topical Session 2: Chemistry / 71**Synthesis of Deuterated Molecules Using Enzyme Catalysis**

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Analysis of hydrogen-containing molecules via neutron scattering analyses often benefits from the substitution of hydrogen with deuterium atoms, since hydrogen (protium) and deuterium have different scattering length densities (SLDs). Selective replacement of protium for deuterium in multi-component systems allows the scattering from individual components to be observed in isolation. For this reason, neutron scattering facilities such as the European Spallation Source (ESS) are invested in furthering techniques for producing deuterated molecules.

Current methodologies fall broadly under the categories of ‘chemical’ – using H/D exchange reactions, or deuterated reagents, to exchange or install deuterons – or ‘biological’ – growing organisms in D₂O, often with a deuterated carbon source, followed by extraction and purification of the molecules of interest. The chemical deuteration laboratory at ESS is aiming to establish a combined chemical-biochemical approach to deuterated molecules exploiting enzyme catalysis. Enzyme catalysis is advantageous because it is safer than and operates under milder conditions than conventional chemical synthesis and because it shows excellent chemo-, regio- and enantioselectivity, greatly increasing efficiency.

We have successfully applied this approach to the synthesis of enantiopure deuterated D- and L-lactic acid-d₄; current work focuses on applying this method to tail-deuterated mixed-acyl phospholipids such as 1-palmitoyl-2-oleoyl-sn-glycerol-3-phosphocholine (POPC). Recent results will be presented.

Topic:

Chemistry

Topical Session 3: Earth & Environment / 56

Neutrons Illuminate the Muddy World of Clay – Water Dynamics

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Hydrous clay minerals (smectites) have complex interactions with water that both define them as separate from other layer silicates (e.g., micas) and also impart their unique suitability as environmental barriers. Cation hydration, surface adsorption and matrix suction are processes by which water is explained to interact with clay mineral surfaces. The hydration energies of cations held within the interlayer space of montmorillonite have long been viewed as key to clay mineral hydration. Many experiments have shown that the charge and hydration energy of the interlayer cation imparts differences in the way the clay mineral hydrates, orders and interacts with its surroundings. Water adsorption to the surfaces of clay minerals are also important: halloysite contains interlayer water without the presence of interlayer cations; instead the residual charge at the gibbsite-like surface is thought to enable fairly strong hydrogen bonding. As fine-grained materials, the structure of the pore-network within a particle of clay mineral (containing perhaps many thousands of individual layers) can be shown to control water uptake and release. During either of these processes, the pore-structure can change in smectites, making complete characterization difficult.

Various approaches can be used to study the above processes and this paper serves to highlight applications of neutron scattering, particularly elastic fixed window (EFW) and quasi-elastic neutron scattering (QENS) methods to study the mobility of water within bulk pores, the interlayer space, and surrounding the interlayer cation of montmorillonite. A simple experiment will be detailed that

enables full quantification of water in various hydration states within a well-characterized montmorillonite sample, and further how this can be related to applied engineering problems in determining the unfrozen water content of a bentonite (a smectite enriched rock) used as an environmental barrier for site remediation work by the Australian Antarctic Division at Casey Station, Antarctica.

Topic:

Earth & Environment

Topical Session 3: Earth & Environment / 20**Thermal expansion and phase changes of methane and nitrogen at Pluto temperatures**Helen Maynard-Casely¹ ; Helen Brand¹¹ ANSTO**Corresponding Author(s):** helenmc@ansto.gov.au

Some of the most striking images from the New Horizons fly-by of Pluto were of the towering mountains surrounded by seemingly flowing glacial terrain [1]. The explanation for this terrain has its basis in crystallography, where at 44 K the strength of the hydrogen bond endows water ice with the resilience to build such mountains, while the rotational disorder in the solid structures of methane [2] and nitrogen [3] allow these materials to flow plastically even at 44K. These interpretations have been strengthened by the spectral observations that correlate these materials to the respective terrains [4].

In order to undertake accurate modelling of the geological features on Pluto the physical properties of the constituent materials must be well constrained. Although water-ice has been the subject of intensive laboratory studies, the same cannot be said of methane and nitrogen. The thermal expansions of these materials have been investigated [5], but only to a lowest temperature of 40 K and using techniques which were largely insensitive to, in particular, the hydrogen positions within methane. Thus, the aim of the experiments reported here is to investigate the structures and determine the thermal expansion of methane and nitrogen over temperatures relevant to Plutonian processes using neutron diffraction.

References: [1]. Moore, J.M., et al., *Science*, 2016. 351(6279): p. 1284-1293. [2]. Press, W., *Journal of Chemical Physics*, 1972. 56: p. 2597. [3]. Press, W., B. Janik, and H. Grimm, *Zeitschrift für Physik B Condensed Matter*, 1982. 49(1): p. 9-16. [4]. Grundy, W., et al., *Science*, 2016. 351(6279): p. aad9189. [5]. Heberlein, D., E. Adams, and T. Scott, *Journal of Low Temperature Physics*, 1970. 2(3): p. 449-463.

Topic:

Earth & Environment

Topical Session 3: Earth & Environment / 89**Neutron micro-CT as a non-destructive tool for Palaeontology in Australia**Joseph Bevitt¹¹ Australian Nuclear Science and Technology Organisation**Corresponding Author(s):** joseph.bevitt@gmail.com

The physical extraction of fossilised remains from rocks enables quantitative physiological investigation of bonedimensions, volume, and porosity, however leads to the destruction of valuable contextual information and soft-tissue remains within the matrix.

Conventional and synchrotron-based X-ray computed tomography (XCT) have been utilised for many years as critical tools in uncovering valuable 3-D internal and surface renderings of scientifically important fossils, however poor contrast and X-ray penetration often prevents thorough tomographic analysis.

DINGO, Australia's first and only neutron micro-computed tomography (nCT) instrument, located at the OPAL nuclear research reactor, is being used to obtain unprecedented reconstructions of extraordinary fossilised anatomical features not visible with conventional imaging techniques. This presentation will outline the physical capabilities of DINGO, the limitations and results to-date in the field of palaeontology. Drawing upon specimens scanned from across Australia, North America, New Zealand, and China, this presentation will demonstrate the complementarity of nCT to classic XCT methods for certain geological formations and fossil localities.

nCT has yielded unprecedented contrast and detailed-reconstructions of fossilised soft tissue in a Jurassic cynodont. The stomach contents and digestive function of herbivorous and carnivorous dinosaurs, and a Cretaceous Australian crocodylian have been revealed, providing insights into ancient environments and food chains. In this way, a new species of Australian dinosaur has been discovered.

Topic:

Earth & Environment

Keynote / 68

Measurement of Magnetic Exchange in Asymmetric Lanthanide Dimetallics

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We have been investigating the magnetic interactions between lanthanide ions in a series of isostructural asymmetric dimetallic complexes of dysprosium(III), erbium(III) and ytterbium(III). Using a range of techniques including electron paramagnetic resonance spectroscopy, inelastic neutron scattering, and complete active space self-consistent field calculations, we have determined the highly anisotropic magnetic coupling matrix within the low-lying manifold spanned by the ground Kramers doublets of each ion.

In all cases the magnetic interaction is not solely dipolar in origin, indicating a measurable superexchange component. We find a unique orientation for the magnetic interaction matrix, corresponding to a common elongated oxygen bridge for the erbium(III) and ytterbium(III) analogues, suggesting a microscopic physical connection to the magnetic superexchange. These results are vital for building and validating model microscopic Hamiltonians to understand the origins of magnetic interactions between lanthanides and how they may be controlled with chemistry.

[1] E. Moreno Pineda, N. F. Chilton, R. Marx, M. Dörfel, D. O. Sells, P. Neugebauer, S.-D. Jiang, D. Collison, J. van Slageren, E. J. L. McInnes and R. E. P. Winpenny, *Nature Commun.*, 2014, 5, 5243.

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Topic:

Chemistry

Topical Session 4: Physics / 39**Fabrication of Sub-Stoichiometric Ti₂O₃ for Room Temperature Thermoelectric Energy Regeneration: Tuning of Structural and Electronic Properties via Defects Engineering**Jack Yang¹ ; Yichen Liu² ; Dehong Yu³ ; Sean Li²¹ *School of Materials Science and Engineering, UNSW*² *School of MSE, UNSW*³ *ANSTO***Corresponding Author(s):** yichentaichi@gmail.com

Titanium oxides has drawn extensive attention as functional electronic materials in the past few years, due to their unique layered structure and physical properties. Sub-stoichiometric titania are particularly interesting non-toxic materials for thermoelectric applications because of their high electrical conductivity with possible low phonon thermal conductivities originated from phonon scatterings at ordered defect planes. Hereby, layered sub-stoichiometric Ti₂O₃ material has been successfully fabricated by densifications of the ball-milled precursors with spark plasma sinterings. The experiments were performed on densified Ti₂O₃ samples with 0.5, 3 and 10 h ball-milling times to compare the changes in PDOS. The application of high-energy ball milling could significantly decrease the grain size in the SPS-densified bulk sample, and thus affect the phonon behaviours. The XRD results showed with the increasing of ball milling hours, the percentage of Ti₃O₅ increased while Ti₂O₃ is still the main phase. Measurements of phonon density-of-states (PDOS) were performed with the PELICAN time-of-flight neutron spectrometer in the energy-gain mode at ANSTO, at 200, 300, 500 and 650 K, respectively. The overall shapes of the GDOS are very similar across the three samples, with three peaks located at around 20, 40, and 60 meV and matched well with the calculated PDOS of Ti₂O₃, indicating the dominate phase for three samples are still Ti₂O₃. With the temperature increasing, the peak intensity at around ~20 meV increased, however, the red-shifts and intensity decreases were observed at the 40 and 60 meV phonon DOS peaks (as indication of anharmonic effects). This suggested that the acoustic phonons response differently to temperature increase compared to optical phonons. The intensities at between 50 to 60 meV increases for the 10H spectrum, compared to the other two. This is probably because of the excitation of the phonon states in Ti₃O₅, as the increased Ti₃O₅ percentage in 10H sample. Our results suggested the measurement matched well with the theoretical study, which indicates the structural changes could have played significant roles in determining the phononic structure of sub-stoichiometric Ti₂O₃ based material.

Topic:

Physics

Topical Session 4: Physics / 16**Structure and dynamics in photovoltaic metal hydrides****Author(s):** Gail Iles¹**Co-author(s):** Katherine Chea² ; Kirrily Rule³ ; Richard Mole⁴ ; Margaret Elcombe⁴¹ *RMIT*² *RMIT University*³ *Australian Nuclear Science and Technology Organisation*⁴ *ANSTO***Corresponding Author(s):** s3542118@student.rmit.edu.au

Solar cell technology is an active area of research with the quest to improve the efficiency of solar cells to above the current value of 44% [1]. Hot carrier solar cells are particular types of cells which may enable higher efficiencies to be obtained. However, these are only feasible where there is a sufficiently large band gap in the phonon dispersion of the bulk material to minimise energy loss to from thermalisation, thus keeping the electrons 'hot'. Binary compounds with a large mass difference between the two constituent atoms, and high level of crystal symmetry such as titanium hydride, can have such a gap in their phonon dispersion.

Titanium hydride is an interesting photovoltaic material with a broad range of properties, which vary depending on the proportion of hydride present. Theoretical studies show TiH₂ has a phonon band gap of 95 meV in the bulk phase [2], however, there is little experimental data to confirm this. TiH_{1.65} has been measured using X-ray powder diffraction and inelastic neutron scattering whereby it was found that this sample had a phonon band gap of 65 meV [3].

We present here further X-ray powder diffraction and inelastic neutron scattering data on powder samples of TiH₂ and TiH_{1.5} whereby we show the correlation of phonon band gap with hydrogen content.

[1] Solar cell efficiency graph <https://phys.org/news/2016-02-solar-cell-efficiency-nrel.html> (last accessed 15/09/2018)

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Topic:

Physics

Topical Session 4: Physics / 32

Neutron spectroscopy of α -Fe₂O₃ nanorods: Direct detection of long-range spin wave excitations

David Cortie¹ ; Gilberto Casillas-Garcia¹ ; Andrew Squires¹ ; Richard Mole² ; Xiaolin Wang¹ ; Yun Liu³ ; Yen-Hua Chen⁴ ; Dehong Yu²

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We present time-of-flight neutron spectroscopy data from PELICAN on α -Fe₂O₃ nanorods with an average length of 300 ± 100 nm and diameter of 60 ± 10 nm. A strong quasi-elastic neutron signal is associated with absorbed water on the nanoparticle powder, which can be removed through heat treatment. After suppressing the QENS signal, it is possible to observe weak spin wave excitations originating from the antiferromagnetic structure of the α -Fe₂O₃ nanocrystals. The excitations are directly compared with measurements conducted on larger microscale α -Fe₂O₃ particles at various temperatures to highlight differences in mode intensity and width. The interchanged spectral intensities in the nanorod are a consequence of a suppressed spin orientation, and this is also evident in the neutron diffraction which demonstrates that the weak ferromagnetic phase survives to 1.5 K. The main magnon features are similar in bulk and nanoforms and can be explained using a model Hamiltonian considering interactions up to fourth nearest-neighbors. Complementary scanning transmission electron microscopy data is presented in order to clarify the atomic-scale structure and morphology of the rods. Finally, the implications are discussed for technological devices based on magnonic transmission at surfaces and through nanowires [2].

References

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[2] A. V. Chumak et al., Magnon transistor for all-magnon data processing, *Nature Communications* 5 4700, (2014)

Topic:

Physics

Topical Session 5: Biology / 60**Using neutrons to elucidate the molecular details of enzyme isoform selectivity by small molecule inhibitors.****Author(s):** Zoë Fisher¹**Co-author(s):** Robert McKenna²; Wolfgang Knecht³; Tobias Schrader⁴; Andreas Ostermann⁵; Matthew Blakeley⁶; Cynthia Okoh²; Brian Mahon⁷; Katarina Koruza³¹ *European Spallation Source ERIC*² *University of Florida*³ *Lund University*⁴ *JCNS*⁵ *Technical University of Munich*⁶ *Institut Laue Langevin*⁷ *Princeton University***Corresponding Author(s):**

Human carbonic anhydrase IX (CA IX) expression is activated by hypoxic condition in aggressive, metastatic tumors. Cancer patients positive for CA IX have generally a poor prognosis. CA IX has emerged as an important cancer target, but efforts to develop isoform selective inhibitors are complicated by the presence of 14 other CA isoforms that share high sequence and structural similarity. This leads to off-target inhibitor binding and side effects. Recent studies showed that saccharin (SAC) already shows some isoform discrimination, and that conjugating SAC to a glucose molecule (Saccharin-Glucose Conjugate, SGC) further improves the K_i against CA IX by 2-fold.

Ligand binding to proteins are mediated through numerous interactions, including: H-bonding directly and/or through intervening waters, electrostatic interactions with charged or polar amino acid side chains, metal coordination, energetic changes through water displacement, aromatic ring stacking, or other hydrophobic interactions. As neutrons scatter strongly from atomic nuclei of light atoms ¹H (Hydrogen), and its isotope ²H (Deuterium), it is possible to use neutron protein crystallography (NPX) to “see” the light atoms and any interactions they are involved with. (e.g. H-bonds). We used joint X-ray and neutron crystallography methods to determine the crystal structures of a CA IX mimic alone and in complex with SAC and SGC, respectively. Our analyses reveal the molecular details of solvent displacement upon ligand binding, the H-bonding between the ligands and the proteins, involvement of water-mediated H-bonds, and the remodeling of H-bonds to accommodate ligand binding. The structures and analysis also provide an explanation for the observed CA isoform selectivity of the ligand under study.

Topic:

Biology

Topical Session 5: Biology / 26**Understanding the interaction between the *Proteus mirabilis* Scs proteins using neutron scattering****Author(s):** Emily Furlong¹**Co-author(s):** Andrew Whitten²; Hassanul Choudhury¹; Fabian Kurth¹; Anthony Duff²; Jennifer Martin¹

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Correctly forming disulphide bonds is critical to the folding of a wide variety of proteins. Bacterial virulence factors are one class of proteins containing disulfide bonds, thus, an approach to disarm virulent bacterial might involve shutting down the machinery involved in the formation of disulfide bonds. The suppressor of copper sensitivity (Scs) proteins form part of the disulfide bond forming machinery in bacteria, and it is hoped that determining the structure of molecules such as this may lead to the development of new classes of antibiotics. There are four Scs proteins (ScsA, B, C and D) present in numerous Gram-negative bacteria, and few have been structurally characterised. In this work, we have created cysteine variants of PmScsC and PmScsB to produce a stable complex and using small-angle X-ray and neutron scattering with contrast variation, we have determined the low-resolution structure of the PmScsC–PmScsB complex.

Topic:

Biology

Topical Session 5: Biology / 100

Bismuth-NSAIDs as colorectal cancer chemopreventives

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To date, epidemiological studies, animal studies and clinical trials have indicated the potential of non-steroidal anti-inflammatory drugs (NSAIDs) for the chemoprevention of colorectal cancer (CRC) [1]. Unfortunately, the use of NSAIDs for CRC chemoprevention is significantly limited due to the severe gastrointestinal (GI) side effects that have been associated with their long term use [1]. It is hypothesised that the coordination of NSAIDs to bismuth, a heavy metal with proven gastrointestinal sparing properties [2], may allow the use of NSAIDs as chemopreventives for CRC while also combating their associated GI side effects. The present study investigates the interactions of bismuth-coordinated NSAIDs (BiNSAIDs) with eukaryotic membrane mimics with the aim of establishing the possible uptake mechanisms of these compounds. This knowledge will be extended by investigating the behaviour of BiNSAIDs in more complex systems, including CRC cells and a CRC animal model.

QCM-D studies involving biological membrane mimics composed of POPC or POPC/cholesterol demonstrated that BiNSAIDs and their parent NSAIDs interact with biological membranes [3]. Neutron reflectometry was also used to study the membrane interactions of BiNSAIDs and provided further evidence of the membrane interactions of BiNSAIDs, suggesting that passive diffusion is a likely method of uptake of these compounds [3]. These strength of these membrane interactions was an indicator of BiNSAID cytotoxicity against CRC cells.³ A CRC animal study has recently been completed with aspirin, which has promising preliminary results. In conclusion, the aforementioned studies continue to highlight the potential of BiNSAIDs as candidates for further investigations into their potential for the chemoprevention of CRC.

References

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Topic:

Topical Session 5: Biology / 29

HIERARCHICAL ARCHITECTURE OF CELLULOSE AND ITS INTERACTION WITH OTHER PLANT CELL WALL POLYSACCHARIDES

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Plant cell walls (PCWs) are extremely complex structures in which cellulose microfibrils are hierarchically assembled and embedded in a multi-component matrix. While the cellulose microfibrils represent the basic building unit providing mechanical strength [1], the matrix components are able to tune the properties of each specific tissue [2-3], increasing the flexibility or limiting the transport of moisture, for instance.

The synthesis of cellulose hydrogels by means of bacterial fermentation is an efficient approach to mimic the cell wall biosynthesis process and investigate the interactions established between cellulose and matrix polysaccharides by incorporating the latter into the culture medium. We have characterised cellulose hydrogels and their composites with PCW polysaccharides by means of SANS and SAXS, combined with complementary techniques such as X-ray diffraction, spectroscopy and microscopy. Furthermore, the production of partially deuterated cellulose hydrogels by using a deuterated glucose-based feedstock is presented as a strategy to enhance the neutron scattering length density contrast [4].

The application of a multi-technique characterisation approach enabled elucidation of the complex hierarchical architecture of cellulose hydrogels and led to the development of a multi-scale model based on core-shell structures [4-8]. The model describes the multi-phase structure of cellulose microfibrils and ribbons, as well as the essential role of water at the different structural levels. In addition, USANS experiments are presented as a promising method to characterise the structure of native cellulose in the longitudinal direction, providing information on the microfibril length and ribbon twisting periodicity.

PCW polysaccharides such as xyloglucan, arabinoxylan, mixed linkage glucans and pectins during cellulose synthesis have a distinct structural role and interaction mechanism with cellulose (interfering with the crystallisation process and strongly interacting with the cellulose microfibrils, or establishing interactions at the ribbons' surface level).

These results highlight the ability of small angle scattering techniques to provide valuable insights on cellulose biosynthesis and interactions with PCW polysaccharides.

Topic:

Biology

Topical Session 6: Engineering & Industry / 94

Bragg-Edge Neutron Strain Imaging and Tomography

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For more than 10 years, time-of-flight detectors at pulsed neutron sources have been capable of providing high-resolution images of strain fields through Bragg-edge analysis [1]. With a geometry akin to a traditional radiograph, these images represent a projection of the full 3D tensor strain distribution to a scalar field. This poses a rich tomography problem based on a generalised version of the Radon transform known as the Longitudinal Ray Transform (LRT). The solution to this Bragg-edge strain tomography problem promises a new approach by which the full triaxial elastic strain (and hence stress) distribution could be observed within crystalline solids over the scale of centimetres. This presentation will provide an overview of Bragg-edge imaging and strain measurement before outlining recent work by the Authors focused on solving the associated tomography problem. The central issue that rendered the problem ill-posed will be discussed before introducing a range of approaches based on equilibrium constraints.

A two dimensional experimental demonstration based on data from the RADEN energy-resolved imaging instrument (at J-PARC in Japan) will be presented with comparisons to detailed constant-wavelength strain scans from the KOWARI diffractometer (ANSTO). The generalisation of this approach to three-dimensions will also be discussed.

[1] Tremsin et al. "High-resolution strain mapping through time-of-flight neutron transmission diffraction with a microchannel plate neutron counting detector", *Strain*, v48 pp296-305, 2012.

Topic:

Engineering & Industry

Topical Session 6: Engineering & Industry / 92

Neutron measurements for Additively Manufactured components

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Additive manufacturing is an exciting new approach to produce components that are not manufacturable using traditional methods. Coupled with its short CAD-to-product time, this method had been gaining significant attention. In particular, the application of Selected Laser Melting, which can process metals, is gaining popularity in application on aerospace components. Due to the numerous rapid thermal cycling in the melting process, strong residual stresses develop within the component being built, which leads to deformation and cracking. Neutron diffraction has sufficient penetration and measurement resolution to map the residual stresses variations within the component. Furthermore, the dimensional accuracy of the produced components is particularly difficult to measure due to the complex geometry. For example, the application of topology optimisation for weight reduction leads to a more organic shape as well as internal cavities but the component becomes more sensitive to geometric variations. Neutron CT is able to capture the geometry, particularly of internal cavities, as well as possible defects present. The combination of these two neutron

measurement techniques provide a strong basis for improving the understanding of the SLM process and aid in the maturing of this process towards serial production.

Topic:

Engineering & Industry

Topical Session 6: Engineering & Industry / 75**Neutron diffraction residual stress determinations on Intermetallic alloy components produced by wire-arc additive manufacturing (WAAM)**

Chen Shen^{None} ; Yan Ma¹ ; Mark Reid^{None} ; Anna PARADOWSKA^{None} ; Huijun Li^{None}

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Intermetallic alloys such as aluminides of titanium, nickel and iron exhibit an attractive combination of physical and mechanical properties such as high melting point, low density, high strength, good oxidation, and creep resistance, due to their strong internal order and mixed bonding. However, the properties of these materials are often obtained at a cost in terms of ease of manufacturing. In recent years the WAAM process has been successfully applied to in-situ produce TiAl and Fe₃Al intermetallic components with designed chemical compositions. One of the major concern is residual stresses (RS) distribution in the WAAM fabricated components as it not only influences the part tolerance but also cause premature failure.

The neutron diffraction technique has been recognised as the most precise and reliable method of mapping sub-surface RS in components for both academic and industrial-economic relevance. Considering the outstanding capability of obtaining RS non-destructively deep within the interior of components, our study utilised neutron diffraction technique to conduct RS measurement by the angular scanning instrument KOWARI. Furthermore, an averaging method has been developed for the WAAM multi-bead buildup intermetallic alloys with large grain size. With the averaging method applied during experimental setup and data processing, reasonable residual stress results have been obtained from the acquired neutron diffraction data.

Topic:

Engineering & Industry

Topical Session 6: Engineering & Industry / 43**Structural properties of elastomer thin films bound to model filler interfaces**

Koichiro Hori¹ ; Norifumi Yamada² ; Tomomi Masui¹ ; Hiroyuki Kishimoto¹ ; Hideki Seto²

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The interfaces between polymers and inorganic materials are of pivotal academic and industrial interest. An example of this is the interface in nanocomposites, a class of materials composed of polymers (matrix) and inorganic nanoscale powders (fillers). In general, the performance of a nanocomposite strongly depends not only on the physical properties of the matrix but also on the interaction

between the matrix and the filler material. It is noteworthy that the physical response of a composite can be modulated via the interactions resulting from the attachment/detachment of polymers to the fillers. This means that an understanding of the interfaces between the polymer matrix and inorganic fillers is essential to construct highly functionalized nanocomposites.

A specific example is tire materials; because the rubber used for tires consists mainly of a polymer elastomer matrix and carbon/silica fillers, an understanding of the interface between the polymer and the filler is important for improving quality. In the case of a carbon filler, a nanometer-thick layer called the bound rubber layer (BRL), is typically formed on the carbon surface and is resistant to be dissolved even in a good solvent. In theory, the interactions between polymers and carbon material surfaces restrict molecular motion, which correlates with increased resistance to mechanical deformation compared to free polymers that are located away from carbon material surfaces.

Significant effort has been made in industry to manufacture high-performance tires using surface-modified fillers. Since the structure and/or thermal molecular motion of polymers at the filler/polymer interface is affected by the interactions with the fillers, it is believed that surface modification changes the properties of the BRL, resulting in an improvement in the tire's performance. However, this is still hypothetical, because there is insufficient evidence and empirical data, e.g., on the influence of surface modification on the BRL. The relationship between the BRL and tire performance needs to be evaluated to develop guiding principles for improving tire performance. Although the number of reports on the general BRL framework is increasing, it is still unclear how the surface modification of fillers affects the structural and mechanical properties of BRLs.

In this study, we have investigated the distribution of rubber polymer on model filler surfaces (carbon or silica) using neutron reflectometry (NR) at SOFIA reflectometer in J-PARC. The experimental results show that the heterogeneous distribution of rubber polymer near the carbon surface depending on the surface energy. Additionally, we investigated the distribution change depending on the kind of coupling agent at the silica surface.

Topic:

Engineering & Industry

Topical Session 7: Neutron Facilities / 46

Recent Progress and Scientific Activities at Materials and Life Science Experimental Facility, J-PARC

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Materials and Life Science Experimental Facility (MLF) at J-PARC is a user facility providing the world highest class neutron and muon pulsed beam. As a neutron part of MLF [[1]], we are serving 20 neutron instruments to user program, which are covering various type of measuring techniques, i.e., direct/indirect geometries and spin-echo spectrometers for inelastic and quasielastic neutron scattering, single-crystal, powder, engineering and high-pressure dedicated diffractometers, a total scattering instrument, reflectometers in horizontal and vertical geometries, a small and wide angle scattering instrument, an energy resolved imaging facility, beamlines for fundamental physics studies, and so on. Also, a polarized neutron dedicated chopper spectrometer is its commissioning phase and will be on line soon. These instruments are realizing research in wide range of fields, such as fundamental physics, solid state physics, biology, chemistry and industrial applications, which are carried out by users (we had 950 unique users visited MLF in 2017) and facility staff. We are also devoting some of efforts to promoting or to enhance scientific outcome. One of examples is a deuteration laboratory program which is under way with strong help of Australian friends.

In this presentation, I will overview current status of our neutron source, neutron instruments at MLF with selected topics of recent scientific output from the facility.

Reference

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Topic:

Neutron Facilities

Topical Session 7: Neutron Facilities / 17

The CMRR neutron scattering instruments and application

Author(s): Guang-ai Sun^{None}

Co-author(s): Jian Gong

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The China Mianyang Research Reactor (CMRR) with the power of 20 MW is located in Mianyang city, Sichuan Province. The thermal and cold fluxes for neutron scattering experiments are 2.4×10^{14} n/cm²·s and 109 n/cm²·s, respectively. The reactor has run 120 days in 2016, and run more than 150 days in 2017. Now it has been included in the website: Neutronsources.org. Eight neutron scattering instruments have been operated normally from the middle of 2014. Four thermal neutron instruments were installed in the reactor hall: a high resolution neutron diffractometer (HRND), a residual stress neutron diffractometer (RSND), a thermal neutron radiography station (TNR) and a high pressure neutron diffractometer (HPDC). Four cold neutron instruments were installed in the guide hall: a small-angle neutron spectrometer (SANS), a time-of-flight and polarized neutron reflectometer (TPNR), a cold-neutron triple-axis spectrometer (CTAS) and a cold neutron radiography station (CNR). Intensive work has also been done for instrument improving during recent years. The texture measurement was realized on the RSND by utilizing the Kappa goniometer. The HPDC is has been upgraded with 6.7m focusing neutron guide to improve the flux, which has reached to 3×10^6 n · cm⁻² · s⁻¹. The high pressure loading has realized maximum pressure 20GPa. The HRPD will be upgrade with high resolution and high intensity modes. SANS has changed the velocity selector with GE products and adjust the collimation system, thus the cold neutron flux at the sample position is 2×10^7 n·cm⁻²·s⁻¹. The characteristics of the TPNR are the time-of-flight and polarization modes. Relative resolution of momentum transfer of the TPNR is $\Delta q/q = (0.5-5)\%$ for $q = (0.05-3.0)$ nm⁻¹. The CTAS has an energy resolution of $\Delta E/E \leq 2\%$ with a range of energy transformation of 0~10 meV.

Based on the neutron scattering instruments at CMRR, some interesting research experiments have also been done. RSND provides a unique tool for accessing the internal stress distribution and deform mechanism inside components. Neutron powder diffraction pattern gives the degrees of structure distortion and changes of bond lengths between Co/Fe and O in Co_{0.708}Fe_{0.292}WO₄, CoWO₄ and FeWO₄, which has been published on Nano Energy. The finding of the localized NH₄⁺ and totally disordered H⁺ in the battery of (NH₄)₃Zr(H₂/3PO₄)₃ has been published on 2018. Some results of soft matter using SANS has been published on Macromolecules and Adv. Mater. etc. Some magnetic film study by TPNR has been published on Sci. Report. and Phys. Rev. Appl. etc.

The second phase for neutron instruments construction has been started from 2017. Three new instruments will be built within next 3-5 years, which are a polarized thermal neutron triple-axis spectrometer (TTAS), an ultra small-angle neutron spectrometer (USANS), and a neutron standard-test beam line (NSTB) for single crystal and texture measurement, respectively. More sample environment systems will be equipped for the present spectrometers, such as high/low temperature, high pressure, superconducting magnetic field, chemical loading (hydrogen/deuterium gas environment), and so on. Recently, we also get the funding for neutron spin echo spectrometer. The inelastic mode (LNRSE) and small angle mode (SESANS) are both under designed by split the C3 cold neutron guide using bend guide. The relative polarized ³He signal was firstly observed by FID NMR in May 4 2018. So it could be expected that more contributions for the neutron scattering will be made by our institute in China.

Topic:

Neutron Facilities

Topical Session 7: Neutron Facilities / 90**NSRRC neutron scattering group at ACNS****Author(s):** Shin-ichiro Yano¹**Co-author(s):** Chin-wei Wang²; Hanz Peng³; Chun-Ming Wu¹¹ *National Synchrotron Radiation Research Center*² *ANSTO*³ *NSRRC***Corresponding Author(s):**

The National synchrotron radiation research centre (NSRRC) in Taiwan has a group of scientists stationed at the Australian Centre for Neutron Scattering (ACNS), ANSTO, Australia. Three Instrument Scientists were hired to operate neutron scattering instruments and to serve users; Chun-Min Wu works on Small angle neutron scattering instruments, Chin-wei Wang is a Powder diffractometer scientist and Shinichiro Yano works on the cold triple axis spectrometer SIKA. In addition, one staff member is a software engineer.

Under this arrangement, we have built the cold triple axis spectrometer SIKA in the OPAL reactor beam hall at ANSTO. SIKA was designed to study dynamics in magnetic materials in the energy range of meV and below with high resolution. The Taiwan investment adds particular value to the area of materials science whereby Taiwan and Australia both benefit. Following 10 years of construction, SIKA has been in the user program since 2015 July with the number of scientific publications growing steadily since then.

Taiwanese users now occupy about 20 percent of international proposals submitted to ACNS in the 2018-2 proposal round whilst the Taiwanese neutron scattering community is also growing. We hope for continued good relations between NSRRC and ANSTO with a view to expanding our collaboration. We are currently in the process of hiring an additional instrument scientist who would be working on a reflectometer at ACNS. In this talk, we will present scientific outcomes from the NSRRC neutron group and updates from SIKA.

Topic:

Neutron Facilities

Keynote / 87**Behaviour of Single Transmembrane Peptides During In Meso Crystallization from the Contrast-Matched Lipidic Cubic Phase of Monoolein**

Leonie van 't Hag¹; Liliana de Campo²; Nhiem Tran³; Anna Sokolova⁴; Raphael Trenker⁵; Matthew Call⁶; Chris Garvey²; Anna Leung⁷; Tamim Darwish²; Anwen Krause-Heuer²; Robert Knott²; Thomas Meikle⁸; Sally Gras⁹; Calum Drummond⁸; Raffaele Mezzenga¹⁰; Charlotte Conn⁸

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In meso membrane protein crystallization within a lipidic mesophase has revolutionized the structural biology of integral membrane proteins (IMPs). High-resolution structures of these proteins are crucial to understanding fundamental cellular processes at a molecular level, and can lead to new and improved treatments for a wide range of diseases via rational drug design. However, overall success rates of the promising in meso crystallization technique remain low because of a fundamental lack of understanding about factors that promote crystal growth. In particular, to date, two decades from invention of the method, the protein-eye-view of the in meso crystallization mechanism had not been solved. We have investigated this for the first time using small-angle neutron scattering (SANS).

Contrast-matching between the scattering of the lipid membrane formed by MO and the aqueous solution was used to isolate and track the scattering of single-transmembrane peptides during the growth of protein crystals in meso. No peptide enrichment was observed at the flat points of the diamond cubic QIID phase of MO in contrast to suggestions in several modeling studies. During in meso crystallization of the DAP12 peptide a decrease in form factor and a transient fluid lamellar $L\alpha$ phase could be observed providing direct evidence for the proposed crystallization mechanism. Synthesis of fully deuterated MO was required for this purpose and scattering of this new material in various solvents and under a range of conditions will be described, specifically regarding the effect of the relative scattering length densities (SLD) of the headgroup, acyl chain and solvent, which can advance the use of neutron scattering with other self-assembly materials.

Topic:

Biology

Topical Session 8: Soft Matter / 9

The Internal structure of a NIPAM brush layer

Author(s): Ben Humphreys¹**Co-author(s):** Edwin Johnson¹ ; Grant Webber¹ ; Erica Wanless¹ ; Stuart Prescott² ; Andrew Nelson³ ; Elliot Gilbert³¹ University of Newcastle² UNSW³ ANSTO**Corresponding Author(s):** c3131868@uon.edu.au

The internal structure of a thermoresponsive poly(N-isopropylacrylamide) (PNIPAM) brush coating has been investigated via neutron reflectometry and small angle neutron scattering (SANS).[1] PNIPAM is a thermoresponsive polymer with an entropically driven lower critical solution temperature (LCST). Below the LCST, the polymer will hydrogen bond with H₂O, solubilising the chains. Above the LCST, the entropic penalty from solubilising the hydrophobic regions of the polymer becomes too great, and PNIPAM becomes insoluble. When tethered to a surface in the polymer brush regime, the brush layer is swollen at low temperatures and collapsed above the LCST. Furthermore, this abrupt LCST broadens into a temperature transition range spanning 10-20 °C.[2]

We have examined the influence of molecular weight, ionic strength and salt identity on the temperature induced swelling/collapse transition of PNIPAM brushes using ellipsometry, QCM-D, AFM, contact angle measurements and DLS.[1-2] These techniques provided a detailed understanding of bulk and surface properties of the PNIPAM systems investigated. The use of neutrons to interrogate these systems, however, has enabled subtle variation in the brush volume fraction profile normal to the substrate to be elucidated (Fig. 1). This highlights any variations related to surface curvature, brush thickness, surface confinement, ionic strength and salt identity.

- [1] T. J. Murdoch, B. A. Humphreys, et. al., *Macromolecules* 2016, 49, 6050-6060.
[2] (a) B. A. Humphreys, et al., *JCIS* 2018, 516, 153-161; (b) B. A. Humphreys, et. al., *PCCP* 2016, 18, 6037-6046; (c) T. J. Murdoch, B. A. Humphreys, et. al., *JCIS* 2018, 526, 429-450.

Topic:

Chemistry

Topical Session 8: Soft Matter / 53**Nanoplastics – protein interaction: A scattering study of transition from soft and hard corona**Shinji Kihara¹; Ingo Koeper²; Chris Seal¹; Duncan McGillivray¹; Jitendra Mata³¹ *School of Chemical Sciences, The University of Auckland, Auckland 1010, New Zealand*² *Flinders University*³ *ANSTO***Corresponding Author(s):** skih331@aucklanduni.ac.nz

There is growing concern about plastic waste in the environment, and its impact on biological organisms. While bulk plastics are thought to be non-toxic, when the plastics break down to a sub-micron length scale (i.e. nanoplastics), they obtain extra mobility inside living things, and may cause various adverse effects [1,2]. This, coupled with a lack of knowledge surrounding the dangers from different types of plastics, prevents well-designed responses to the problem. Hypothetically, the potential adverse effects are caused by protein denaturation, oxidative stress and/or cellular membrane damage. However, the inherent complexity of biological systems makes it challenging to gain a mechanistic understanding. Adding complexity to this problem, the potential adverse effects are highly dependent on the nature of nanoparticles (NPs) – the contributing factors could include elemental composition, chemistry of the plastic surface, and/or size of the plastic particle [3,2,4].

When in biological systems, nanoplastics are surrounded by various types of proteins⁵. The structure of proteins surrounding nanoplastics are important parameters to understand the interaction of nanoplastic/protein composite. We carried out light scattering and small angle neutron scattering (SANS) experiments to explore the structure of the protein corona on monodisperse polystyrene spheres using a model protein human serum albumin (HSA). The geometry of the PS/HSA complex was investigated with a contrast matching method. The transition from a “soft” to a “hard” interaction between the nanoparticle and the protein was observed when pH is lowered from 7.4, and the implications of this on nanoplastic toxicity is discussed.

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Topic:

Soft Matter

Topical Session 8: Soft Matter / 34

Examining the structural and mechanical implications of surfactants on neutral polymer brushes through neutron reflectometry

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Layers of densely-tethered polymers (polymer brushes) are of interest due to their potential applications as nano-actuators, biocompatible coatings, and switchable lubricating or antifouling surfaces. These applicable properties are dependant on the structure of the polymer interface, so it is important that the structural effects of common compounds and relevant environmental variables be understood. Neutron Reflectometry (NR) is the only technique capable of providing detailed structural resolution of solvated multi-component polymer brush systems due to its penetrating power and the possibility of isotopic substitution.

Here we present a Neutron Reflectometry study on the effects of surface-active molecules (surfactants) on two neutral polymer brushes, poly(ethylene oxide) (PEO) and Poly(N-isopropylacrylamide) (PNIPAM), focusing on small, single tail surfactants. PEO is a widely used biocompatible polymer with a range of medical and commercial applications, whilst PNIPAM is a well known thermoresponsive polymer, undergoing a swollen to collapsed transition over its critical solution temperature (CST) of 32°C. We show that these two polymers exhibit similar yet distinct interactions with surfactants, with the observed differences having implications for the mechanism of brush-surfactant interaction. The presence of surfactants was found to raise the CST of PNIPAM; we show that this effect is dependent strongly on surfactant identity and concentration. As part of this work we have developed new modelling techniques for the analysis of NR data from polymer brush interfaces. These advancements will be explained in the context of the data at hand, and their applicability to other soft diffuse interfaces will be briefly discussed.

Topic:

Soft Matter

Topical Session 8: Soft Matter / 22

Hierarchical structure of solid lipid nanoparticles

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Suspensions of solid lipid nanoparticles (SLNs) stabilized with emulsifiers have been extensively investigated as drug carriers since the 1990s, although details of their ultrastructure are poorly defined. Previously, our group reported a novel microwave-assisted microemulsion-based technique

to prepare SLNs. Ultrastructure generally relates to interior of the particle and can relate to internal partitioning through, for example, a core-shell structure. It can also relate to the formulation itself, including the particle itself, but can also include structures such as micelles, which may be simultaneously present.

Our previous investigations revealed that SLNs were prepared by the novel microemulsion technique have size of 200-300 nm. Preliminary multi-angle SLS/DLS studies indicated core-shell type of SLNs. To understand the detailed ultrastructure of these SLNs, ultra-small angle neutron scattering (USANS) and small angle neutron scattering (SANS) experiments were conducted on suspensions of hydrogenated stearic acid SLNs stabilized with hydrogenated Tween 20 surfactant in D2O. Together, SANS and USANS gave a combined Q range of 4.7×10^{-5} to $6 \times 10^{-1} \text{ \AA}^{-1}$ (corresponding to a size range of $\sim 1 \text{ nm} - 15 \text{ \mu m}$). This extended Q range allows a comprehensive understanding of the hierarchical structure of SLNs. The SANS/USANS data are consistent with the multi-length scale structure of SLNs having polydispersed large particles at the microscale level, intermediate between spheres to rod, with roughened surfaces. At the nanoscale level, the results are consistent with the SLNs solution having an ellipsoidal shape intermediate between spheres and rods, with a crossover from mass fractals to surface fractals. The elucidation of this structure is particularly important given that the structure influences the stability and drug release properties of the nanoparticles. These results will assist in the development of systems with desired shape and properties.

Topic:

Soft Matter

Sponsors / 99**Scitek - A word from our sponsors.****Corresponding Author(s):** ken@scitek.com.au**Topical Session 9: Neutron Instruments & Techniques / 11****SPATZ: The Second Time-of-Flight Neutron Reflectometer at the OPAL Research Reactor****Author(s):** Anton Le Brun¹**Co-author(s):** Paris Constantine¹; Stewart Pullen¹; Marcus Trapp²; Roland Steitz²¹ ANSTO² HZB**Corresponding Author(s):** abn@ansto.gov.au

Neutron reflectometry is a powerful technique for studying the structure of surfaces and interfaces at the nanometer. The useful properties of neutrons allows for isotopic contrast variation in multi-component systems and being able to investigate phenomena under a wide variety of sample environments. At the OPAL Research Reactor there is currently one operating neutron reflectometer – PLATYPUS, however demand is sufficient that a second is needed. In September 2015, an agreement was signed between HZB and ANSTO to transfer the V18 ‘BioRef’ time-of-flight neutron reflectometer [1], previously situated at the 10 MW BER-II Research Reactor, to the OPAL Research Reactor. During 2016, a joint team of ANSTO and HZB personnel carefully disassembled BioRef and packed it into shipping containers for transport to ANSTO. BioRef arrived at ANSTO in early 2017 and is known as SPATZ (German for Sparrow) and will be the 15th neutron-scattering instrument at OPAL. SPATZ has a vertical sample geometry, which complements PLATYPUS with its horizontal sample geometry. The vertical sample geometry will allow for use of sample environments which cannot be currently used on PLATYPUS due to geometry constraints and allows for wide-angle diffraction

from multilayers and lamellar stacks. SPATZ will also be equipped for simultaneous infra-red spectroscopy and reflectometry experiments.

The instrument views the OPAL cold neutron source (CNS) by taking the end position of the CG2B guide, which has recently been installed.

SPATZ is scheduled to start hot commissioning in October/November 2018 and start user experiments in early 2019. This presentation will provide an overview of the project, its current status, and future direction. Feedback from the neutron user community is encouraged.

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Topic:

Neutron Instruments & Techniques

Topical Session 9: Neutron Instruments & Techniques / 23

AUSTRALIAN CENTRE FOR NEUTRON SCATTERING: SAMPLE ENVIRONMENT REPORT

Andrew Manning¹ ; Deborah Wakeham¹ ; Gene Davidson¹ ; Norman Booth¹ ; Paolo Imperia¹ ; Rachel White¹ ; Stan Lee² ; Timothy D'Adam³

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In the past 2 years since the 2016 AANSS symposium, the sample environment group of the Australian Centre for Neutron Scattering (ACNS) has continued to facilitate neutron experiments and expand sample environment capability. This report will present the current sample environment and laboratory facilities and recent developments.

We have made progress in light irradiation and spectroscopy developments; on a new temperature controlled multi-sample changer with tumbling capability; on a rotational PE Cell; and on new sample probes made from composite materials. Other improvements include new high pressure couplings for helium compressors and modifications to a dilution insert to allow larger samples and use in other cryostats extending the capability.

Ongoing major projects are a new superconducting split-coil magnet dedicated to SANS and TOF-PAS, two new cryostats (1.5 K to 800 K temperature range) with the aim of halving the system and sample cooling time and a new dilution fridge that will allow top-loading of samples and the ability to take much larger samples than the existing dilution insert.

There have also been staffing changes with previous Sample Environment Group Leader, Paolo Imperia moving into the Operations Manager position. The new group leader, Rachel White, was recently appointed. Our Laboratory Manager, Deborah Wakeham, joined us in July 2017.

Topic:

Neutron Instruments & Techniques

Topical Session 9: Neutron Instruments & Techniques / 96

Rheo-ND: Temperature and shear induced crystal transformation of a model triglyceride observed using neutron diffraction.

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Rheo-SANS (rheology and small angle neutron diffraction) is now a well-established technique to probe the shape and size of particles under different shear and temperature regimes [1]. Recent work on WOMBAT [2], the high intensity neutron diffraction instrument at the Australian Centre for Neutron Scattering, has successfully combined rheology and neutron diffraction. This was used to follow the crystalline phase transformations in a model (deuterated) triglyceride.

The initial impetus for this work was part of a forensic investigation [3] linking the crystallisation of triglycerides under high shear rates that are encountered in motor vehicle accidents. However now the technique has been demonstrated it may be of interest to investigate crystallization due to shear in other organic systems.

Figure 1 - Diffraction data collected from the shear cell during cooling and shearing at 1000 s⁻¹, colours give the intensity of the diffraction pattern (left axis) and the black squares chart the measured viscosity (right axis). This demonstrates the ability to track crystallisation in situ under high shear rates.

The high neutron flux and detector efficiency available at WOMBAT is instrumental in allowing diffraction patterns to be collected from the small sample volumes available in the Couette cell geometry. The Couette cell used has a sample gap of 1mm and was mounted tangentially to the neutron beam. The shear rate applied to the sample can be varied over a range of 10-2500 s⁻¹ and the temperature can be controlled using an external water jacket from between -10 and 80 °C. We invite other researchers who are interested in this capacity to contact us about possible experiments.

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Topic:

Neutron Instruments & Techniques

Topical Session 9: Neutron Instruments & Techniques / 95**New Contrast Options - Diversity and Specificity of Deuteration**

Peter Holden¹ ; Marina Cagnes¹ ; Natalia Davydova^{None} ; Anthony Duff¹ ; James Howard¹ ; Anwen Krause-Heuer¹ ; Rao Yepuri¹ ; Tamim Darwish¹

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The Australian National Deuteration Facility (NDF) continues to develop new capabilities in molecular deuteration. The tailoring of deuteration approach to solve specific contrast problems has greatly increased the range of systems that can be investigated and recent capability development and new applications will be described.

The common problem of how to obtain good contrast during SANS of membrane proteins solubilised in detergent micelles has been solved by specific deuteration of detergent head groups and hydrophobic tails to different levels to contrast match at 100% D₂O buffer. Likewise, in investigating the location of a purely hydrophobic transmembrane peptide in bicontinuous cubic phase liquid crystals

suitable for drug delivery, a mixture of deuterated and hydrogenated phytanoyl monoethanolamide was used to perfectly contrast match out either gyroid or diamond phase lipids to pinpoint the peptides location.

In Soft Matter, investigation of structure of the ligands in the shell layer of self-assembled monolayer-protected nanoparticles has been made possible by use of various combinations of immiscible deuterated or hydrogenated Phenylethanethiol (PET) and Dodecanethiol (DDT) and MONSA modelling of SANS data showed the janus and belt like distribution of the ligands on the surface and the effects of nanoparticle core size and ligand ratio on the patterning. Other case studies will include use of deuterated ionic liquids and new previously unavailable deuterated molecules will be highlighted. This work was supported by the National Collaborative Research Infrastructure Strategy.

Topic:

Neutron Instruments & Techniques

Topical Session 9: Neutron Instruments & Techniques / 15

ECHIDNA: a decade of high resolution neutron powder diffraction at OPAL

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The ECHIDNA high-resolution neutron powder diffractometer at the 20MW OPAL research reactor produces high-quality data for a broad spectrum of crystal and magnetic structural studies. The poster presents an overview of the current status of the hardware, latest developments in data reduction software, statistics of the instrument usage and user programme, and instrument limitations.

Topic:

Neutron Instruments & Techniques

Poster Session / 61

DEMAX: Deuteration and Macromolecular Crystallization Support labs for the European Spallation Source.

Author(s): Zoë Fisher¹

Co-author(s): Wolfgang Knecht² ; Hanna Wacklin-Knecht¹ ; Anna Leung¹ ; Arno Hiess¹

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In soft matter and life science neutron scattering experiments, access to full or partially deuterated materials is critical. Such molecules may include proteins, lipids, fatty acids, small molecules, membranes and so on. These classes of molecules are of intense interest to the neutron research community, however they are often not used due to various reasons, including: deuterated molecules are not commercially available, or deuterated molecules are simply too difficult and expensive to produce. For small angle neutron scattering (SANS), neutron reflectometry (NR), and neutron protein crystallography (NPX), using deuterated samples has numerous benefits. For SANS, NR deuteration

is most commonly used to enable contrast variation, allowing scientist to selectively “match out” components of complexes. In NPX deuteration is used to boost weak signal-to-noise ratios, reduce the incoherent background due to Hydrogen, improve neutron scattering length maps, and enable direct visualization of Hydrogen bonds and solvent networks.

DEMAX is the Deuteration and Macromolecular Crystallography support lab for soft matter and life science users of the European Spallation Source (ESS). DEMAX has three support pillars and will be available to all users of ESS instrument: chemical deuteration, biological deuteration, and large crystal growth. We have fully equipped chemistry and life science labs and during operations will offer service for specific classes of deuterated inorganic or organic small molecules (e.g. lactic acid), deuterated biomass/crude lipids/proteins, and access to our crystallization labs for large crystal growth. For biological deuteration and protein crystallization we have established a partnership with Lund University’s Lund Protein Production Platform (LP3), a cross-faculty support lab for the production and crystallization of proteins, both unlabeled and labeled (^{13}C , ^{15}N , ^2H) using bacteria, insect cells, and yeast. DEMAX and LP3 are co-located in the Biology Department of LU. Access to DEMAX will be granted on a proposal, peer-reviewed basis. We aim to solicit requests for support services and expressions of interest to help grow our competence and develop methods in early 2019. ESS is also the organizing node for DEUNET, a network of deuteration facilities around Europe, more information and news can be found at: <http://www.deuteration.net>.

Topic:

Neutron Facilities

Poster Session / 72

Investigation of Residual Stresses Distribution in High Strength Steel Beams Using Neutron Diffraction

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Residual stresses induced in the fabrication of steel components may lead to distortion and a significant loss of strength in a steel structure. Accordingly, research on the residual stress distribution in steel sections is becoming an important consideration in steel structural analysis. The characteristics of residual stresses in hot-rolled and welded mild steel sections have been extensively reported and integrated in national design standards. However, it is questionable as to whether such patterns are applicable for high strength steel. Currently, very high strength low-alloy steels produced by quenching-tempering and thermo-controlled processes offer yield strengths as high as 1000 MPa with good weldability, and because of this they have been receiving much attention in mega-structure applications such as long-span bridges or high-rise buildings. To promote the use of such materials, knowledge of welding residual stresses is vital.

This study presents the measurement of residual stresses in I-beam sections welded from Australian BISPLATE80 and BISPLATE100 high strength steel using neutron diffraction technique. Tensile coupon testing shows the base plate materials have corresponding yield stresses of 851 MPa and 1003 MPa respectively, with the weld material yield strength as high as 810 MPa. The residual stress measurement was taken on the KOWARI strain scanner at ANSTO. Due to the complexity and the large size of samples the SSCANSS software and a virtual instrument of KOWARI were utilised to position a sample and optimise the measurement procedure. Important features of the residual stress distribution in welded high strength steel I-sections have been obtained across the beam and within the weld. These results are the essential in order to validate a residual stress model able to be used in future high strength steel structural analysis, design and assessment.

Topic:

Engineering & Industry

Poster Session / 74**Residual stress measurements and weld defects investigations by neutron diffraction**Huijun Li^{None} ; Anna PARADOWSKA^{None}**Corresponding Author(s):** huijun@uow.edu.au

Welding is a foundational aspect of all large-construction industries, ensuring strong, sustainable connections. Due to its relative operational simplicity, welding has become the most important industrial process used for manufacturing of metal parts. Also, the insertion of welding in the context of additive manufacturing (AM) make the welding knowledge to be a great importance for the development of new technologies.

In welded structures residual stresses (RS) are formed primarily as the result of differential contractions which occur as the weld metal solidifies and cools to ambient temperature. Generally, RS are considerably large in layer by layer AM parts. RS have a significant effect on fracture resistance, creep and corrosion/fatigue performance and a full understanding of these stresses is desirable. Therefore, experimental measurements are essential to establish a quantitative understanding of the sign, magnitude and distribution of RS in both traditional welding and AM process. Neutron Diffraction (ND) is outstanding in its ability to obtain RS non-destructively within the subsurface and interior of components. Therefore, the angular scanning instrument KOWARI has been selected in our studies for RS measurements on many big components from industries.

Welding introduces inhomogeneity and defects such as bad joint preparation, lack of fusion, solidification cracking and distortion, which have great influences on weld integrity, must be investigated and understood. With the use of DINGO neutron tomography instrument, our research conducted 3D defect measurements on welded structures. A full assessment on weld quality has been obtained.

Topic:

Engineering & Industry

Poster Session / 63**Scientific computing support for neutron scattering experiments at ANSTO**Ramzi Kutteh¹¹ ACNS - ANSTO**Corresponding Author(s):** rku@ansto.gov.au

The purpose of the scientific computing support at ANSTO is to aid in the interpretation of both structural and dynamical data from the neutron scattering instruments using atomistic modelling calculations. Most of these calculations are done with ab initio scientific software packages based on Density Functional Theory, including VASP, WIEN2K, ABINIT, SIESTA, PHONON, and QUANTUM ESPRESSO, although some are performed with packages based on classical force fields, such as LAMMPS, DL_POLY, NAMD, and GULP. Analysis of the results of these calculations exploits tools such as VMD, NMOLDYN, XCRYSDEN, and ISAACS, in addition to in-house code. Calculations and analysis are carried out locally on a scientific computing Linux cluster comprising 624 ACNS dedicated cores and 1416 ANSTO shared cores, with jobs managed by PBS. We give a brief overview of all of the above capabilities and an example of a typical calculation/analysis.

Topic:

Neutron Facilities

Poster Session / 45**QUOKKA a 40 m small angle neutron scattering (SANS) instrument**Chris Garvey¹; Elliot Gilbert¹; Jitendra Mata¹; Kathleen Wood²; Chun-Ming Wu³¹ ANSTO² Australian Nuclear Science and Technology Organisation³ National Synchrotron Radiation Research Center**Corresponding Author(s):** cjg@ansto.gov.au

QUOKKA is a versatile small angle neutron scattering (SANS) instrument in operation at the Australian research reactor, OPAL [1]. SANS is a powerful techniques for structural characterisation require a minimum of special preparation. As a conventional 40 m pinhole instrument operating with a neutron velocity selector the instrument1 is capable of providing a statistical and non-destructive perspective on structure over length scales from 10's to 1000's of Ångstrom. QUOKKA has a large flexible sample area, capable of accommodating a variety of sample environments including: standard 20 position sample changer for automated measurements on solids, liquids, pastes and powders; Couette geometry rheometer for in situ rheometry/SANS; a stopped flow device; and a differential scanning calorimeter. QUOKKA is available for general use through a biannual proposal system.

[1] Wood, K.; Mata, J.; Garvey, C. J.; Wu, C. M.; Hamilton, W. A.; Abbeywick, P.; Bartlett, D.; Bartsch, F.; Baxter, P.; Booth, N.; Brown, W.; Christoforidis, J.; Clowes, D.; d'Adam, T.; Darmann, F.; Deura, M.; Harrison, S.; Hauser, N.; Horton, G.; Federici, D.; Franceschini, F.; Hanson, P.; Imamovic, E.; Imperia, P.; Jones, M.; Kennedy, S.; Kim, S. J.; Lam, T.; Lee, W. T.; Lesha, M.; Mannicke, D.; Noakes, T. J.; Olsen, S. R.; Osborn, J. C.; Penny, D.; Perry, M.; Pullen, S. A.; Robinson, R. A.; Schulz, J. C.; Xiong, N.; Gilbert, E. P., QUOKKA, the Pinhole Small-angle Neutron Scattering Instrument at the OPAL Research Reactor, Australia: Design, Performance, Operation and Scientific Highlights. J App Crys 2018, 51.

Topic:

Neutron Instruments & Techniques

Poster Session / 55**EMU - the high-resolution backscattering spectrometer at ANSTO**Nicolas de Souza¹; Alice Klapproth²¹ ANSTO - Australian Centre for Neutron Scattering² ANSTO**Corresponding Author(s):** nrlsouza@gmail.com

EMU, the high-resolution neutron spectrometer installed at the OPAL reactor, ANSTO [1] delivers 1 μeV FWHM energy transfer resolution for an accessible $\pm 31 \mu\text{eV}$ energy transfer range. The spectral resolution is achieved by neutron backscattering from Si (111) on the primary and second flight paths, which also determines the accessible 0.35 to 1.95 \AA^{-1} momentum transfer range.

Two years of user operation document strong demand for QENS characterization of microscopic diffusion processes in energy materials such as solid-state electrolytes, and increasingly in bio-related soft materials [2,3]. Over the same time frame most experiments were carried out with standard cryo-furnaces (2 to 800 K temperature range). Spectrometer beam-time access is merit-based, thus welcoming experiments beyond the first two-year 'sample', and including experiments that may require other ancillary equipment such as (existing) controlled-gas delivery, pressure, applied fields,

etc.

Examples of the spectrometer capabilities will be shown, with an emphasis on QENS line shape and mean-square displacements analyses.

Scientific support is presently focused on enabling data analysis of the collected data, and on the instrumental side reaching the design 0.1 \AA^{-1} minimum momentum transfer range and growing signal-to-noise ratio beyond its current $\sim 1650:1$ value.

[1] N.R. de Souza et al., Neutron News, 27, 20 (2016).

[2] D.L. Cortie et al., J. Phys. Chem. C, 121, 18762 (2017).

[3] M.K. Rasmussen et al., accepted EPJ Special Topics (2018).

Topic:

Neutron Instruments & Techniques

Poster Session / 77

Upgrades and improvements to the Kowari residual strain scanner

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This poster highlights some recent upgrades to the Kowari instrument. These include: A wireless cable free remote control for the sample stage allowing freer movement of the operator around the stage when positioning samples; A group of lasers for aligning the primary and secondary/radial collimators as well as allowing accurate positioning of the sample with respect to the primary neutron beam; A redesign of the detector shroud to greatly reduce the time taken to switch between the adjustable slit system and radial collimators.

Topic:

Neutron Instruments & Techniques

Poster Session / 4

Which glue to choose? A neutron-scattering study of various adhesive materials and their effect on background scattering.

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We have investigated the background scattering from numerous, frequently used sample adhesives to determine the background contribution of these glues when used for inelastic-neutron-scattering measurements. Starting with a bare Cu sheet, we have trialled different glues such as GE-varnish, Teflon tape, Fomblin oil and two-component epoxy glue. Measurements were collected using the cold-neutron Time of Flight (TOF) spectrometer, PELICAN which is capable of collecting data over a wide range of Q - ω space simultaneously. Results indicate that those glues containing hydrogen gave much higher background signals, while those that did not contain hydrogen had a much

smaller impact on the background signal. This was observed for both elastic and inelastic neutron scattering.

While it is widely understood that mechanical fastening with copper or aluminium is often the best method for reducing additional and unwanted neutron scattering, it is not always the most practical method. Thus this study has been performed on numerous glues to investigate their effect on both elastic and inelastic background. By keeping all other variables constant (eg instrument configuration, sample environment, copper-sample plate), a direct comparison can be made between each sample. By performing measurements on the TOF spectrometer, PELICAN, we have been able to visualise the data in a number of ways to extract scattering in the elastic channels, inelastic channels and also for diffraction, thereby showing relevant scattering profiles for many commonly used neutron scattering techniques.

Topic:

Neutron Instruments & Techniques

Poster Session / 37

Neutron Polarisation Analysis Capability of PELICAN – A Time-of-Flight Cold Neutron Spectrometer

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The implementation of polarisation analysis on a conventional time-of-flight spectrometer has been a major instrumental goal for some time. Here we present our recent results describing our successful test of the polarisation analysis option on the PELICAN spectrometer. The compact incident neutron polarisation system is an integration of a solid-state C-bender supermirror polariser with a gradient radio frequency (RF) spin flipper. Analysis is achieved by using a polarised ³He neutron spin-filter that covers a span of 120 degrees. The ³He analyser is installed inside the high vacuum sample chamber through a dedicated aluminium vacuum adaptor flange. In-situ refilling of pre-generated polarised ³He gas has been implemented. The supermirror polariser and spin-flipper have been characterized with a Heusler crystal as the analyser for a neutron wavelength of 4.68 Å. 99% and 97% efficiencies have been obtained for the polariser and spin flipper, respectively. Further tests with the ³He analyser on a non-magnetic alumina sample achieved overall polarisation efficiency of 90%, this gives a 94% efficiency for the ³He analyser filled with 1.2 Bar of polarised gas. The T1 lifetime of the ³He cell is approximately 100 hours. Nuclear-spin incoherent scattering measurements of Vanadium have been performed and the 2:1 ratio between the spin-flip and non-spin-flip signal has been observed. The energy resolution of the conventional spectrometer was maintained and approximately 80% of the detector area is still useable.

Topic:

Neutron Instruments & Techniques

Poster Session / 48

Vibrational spectroscopy using the BeF spectrometer on TAIPAN

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Vibrational spectroscopy using neutrons as a probe, as opposed to electromagnetic radiation, is a relatively new capability in Australia. A number of instruments at the Australian Centre for Neutron Scattering are capable of measuring vibrational density of states. In particular, the so-called Beryllium Filter Spectrometer, BeF, located on TAIPAN, is devoted to the measurement of vibrational density of states. Even though most scientists are familiar with techniques such as IR, Raman, NMR etc, vibrational spectroscopy using neutrons poses somewhat of a mystery as the equipment, technique, and analysis, are very different to those techniques using well-known electromagnetic probes.

Unlike light that strongly interacts through the electromagnetic force with the electronic structure, neutron radiation interacts through that part of the residual strong force directly with the atomic nucleus. As a consequence, thermal neutrons probe directly vibrational motion: all excitations are observable because of the lack of selection rules, and scattering cross sections are isotopically sensitive. In particular, scattering from hydrogen allows molecular-like, or localised, modes to be probed. A weighted density of vibrational states is directly measured that can be compared to calculation. In this way the dynamics and mechanical interaction of molecular units may be directly investigated.

For this presentation an overview of the BeF instrument is given, as well as the theory explaining how measured spectra are in fact the weighted density of vibrational states of the system under investigation. Analysis steps are given that take the raw spectra and turn them into spectra suitable for comparison to calculation.

Topic:

Neutron Instruments & Techniques

Poster Session / 42

KOOKABURRA, the ultra-small-angle neutron scattering instrument at ansto: design and recent applications

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The double-crystal ultra-small-angle neutron scattering (USANS) diffractometer KOOKABURRA at ANSTO was made available for user experiments in 2014. KOOKABURRA allows the characterization of microstructures covering length scales in the range of 0.1–10 μm . Use of the first- and second-order reflections coming off a doubly curved highly oriented mosaic pyrolytic graphite premonochromator at a fixed Bragg angle, in conjunction with two interchangeable pairs of Si(111) and Si(311) quintuple-reflection channel-cut crystals, permits operation of the instrument at two individual wavelengths, 4.74 and 2.37 \AA (Figure 1). This unique feature among reactor-based USANS instruments allows optimal accommodation of a broad range of samples, both weakly and strongly scattering, in one sample setup [1,2]. The versatility and capabilities of KOOKABURRA have already resulted in a number of research papers, including studies on hard matter systems like rocks and coal [3,4], as well as soft matter systems like hydrogels or milk [5,6]. This clearly demonstrates that this instrument has a major impact in the field of large-scale structure determination. Some of the recent examples will be presented here.

[1.] Rehm, C. et al, J. Appl. Cryst., 2013, 46 1699-1704.

[2.] Rehm,C.et al, J. Appl. Cryst., 2018, 51, 1-8.

[3.] Blach, T.et al, Journal of Coal Geology, 2018, 186, 135-144

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[5.] Whittaker, J. et al, *Int. J. Biol. Macromol.*, 2018, 114, 998-1007
[6.] Li, Z. et al, *Food Hydrocolloid*, 2018, 79, 170-178.

Topic:

Neutron Instruments & Techniques

Poster Session / 24**Recent Developments in Sample Environment at ACNS**

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Here we present some recent developments on sample environment equipment at ACNS, which markedly improve the existing capabilities for a range of beamline instruments, leading to greater scientific outcomes and operational efficiency.

A six-position thermalised sample changer with tumbling capability, which avoids sedimentation and particle size separation in samples suspended in a medium, has been designed for use on US-ANS. To date, USANS experiments have successfully been completed with a prototype applying both independent temperature control and tumbling. An equivalent thermalised sample tumbler for use on SANS is currently undergoing development.

A new sample positioning probe made from composite materials has been designed to reduce the sample cooldown time in top-loading cryostats. Fabricating probes from thin-walled carbon fibre, which is both lightweight and a poor thermal conductor, has resulted in a two-thirds reduction in the sample cooling time when compared with standard stainless steel sticks. Future work will look to incorporate these features in other applications such as for gas delivery probes.

The Rotational Paris Edinburgh Cell, capable of providing both high pressure and controlled high shear to investigate in areas of physics, chemistry, earth and planetary sciences, has been commissioned at ACNS. It is believed to be one of only two high pressure/shear Paris Edinburgh presses worldwide. This added sample environment capability opens up new experimental possibilities in high pressure – high shear regimes for users. A stretch goal is to add a high temperature capability to the ROPEC, creating a rare trio of extreme temperature – pressure – shear.

Topic:

Neutron Instruments & Techniques

Poster Session / 21**Past, present, and future plans for SIKA, the cold-neutron triple-axis spectrometer at ANSTO**

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For many years the triple-axis spectrometer has been utilised by for neutron scatters to study many areas of magnetism and condensed matter physics. With cold neutrons, a triple-axis spectrometer has the capability to investigate physical phenomena with high energy and momentum resolution. Whilst time-of-flight spectrometry, such as PELICAN, is advanced, the cold triple-axis spectrometer has advantages of scanning $S(Q, \omega)$ space at each reciprocal point, measuring critical scattering, availability of a number of sample environments, and so on.

A cold triple-axis spectrometer SIKA was installed in ANSTO, OPAL reactor. The components, capabilities, sample environment, software, and statistics will be presented in this presentation. We will also give some scientific examples to help users to write proposals for their own scientific project.

Plans for future progress will be also discussed. SIKA has a plan to multiplex monochromator. With using position sensitive deflectors, the instrument will be more efficient to collect neutron scattering data. The other improvement will be ^3He polarization analysis system which is under commissioning. Polarized neutron can help user to study magnetic excitations in detail.

The authors look forward to welcoming users to the cold triple axis spectrometer SIKA.

Topic:

Neutron Instruments & Techniques

Poster Session / 19

WOMBAT – High Intensity powder diffractometer at OPAL

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Wombat is a high intensity neutron diffractometer located in the OPAL Neutron Guide Hall. It is primarily used as a high-speed powder diffractometer, but has also expanded into texture characterisation and single-crystal measurement, particularly diffuse scattering. The high performance comes from the combination of the best area detector ever constructed for neutron diffraction with the largest beam guide yet put into any research reactor and a correspondingly large crystal monochromator, all combine to provide an instrument which is unique in its capabilities within the Southern hemisphere.

Wombat has been used to explore a broad range of materials, including: novel hydrogen-storage materials, negative-thermal-expansion materials, methane-ice clathrates, piezoelectrics, high performance battery anodes and cathodes, high strength alloys, multiferroics, superconductors and novel magnetic materials.

Topic:

Neutron Instruments & Techniques

Poster Session / 67

Gapped Magnetic Excitations in $153\text{EuMn}_2\text{Ge}_2$

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Layered RT₂X₂ compounds (R = rare-earth, T = 3d, 4d transition metal, X = Si, Ge) of tetragonal body centred ThCr₂Si₂ –type structure (I4/mmm) are among the most widely studied systems in condensed matter science [e.g. 1]. Yb- and Eu-based intermetallics are of particular interest due to their intermediate valence character and unusual physical and magnetic properties [e.g. 2, 3]. While EuMn₂Si₂ exhibits lattice behaviour at room temperature characteristic of a trivalent state, EuMn₂Ge₂ indicates a divalent state [4, 5]. Previously we established that the Mn sublattice of EuMn₂Ge₂ has an axial antiferromagnetic structure of space group I4'/m'm'm below the Néel temperature T_N ~ 667(9) K of Mn moment $\mu(0) = 3.43(4) \mu_B$ [4] while Ryan et al. [5] established that the Eu sublattice orders below 9.8(1) K.

Our recent inelastic neutron scattering measurements of isotopically enriched ¹⁵³EuMn₂Ge₂ (~1.5 – 50 K; PELICAN) have confirmed magnetic order in the Eu sublattice below ~ 10 K with the associated spin waves revealing that a gap opens in the excitation spectrum. Aided by a prospective neutron diffraction measurement at ~1.8 K on ECHIDNA, we present an overview of the fascinating magnetic behaviour exhibited by EuMn₂Ge₂ with particular emphasis on modelling the spin wave spectrum and related gap. Such a gap could be the origin of the unusual temperature dependence reported for magnetic ordering of the Eu sublattice in EuMn₂Ge₂ [5].

[1] A. Szytula and J. Leciejewicz, Handbook of Crystal Structures and Magnetic Properties of Rare Earth Intermetallics (CRC Press, Boca Raton, 1994)

[2] K. Kummer, Yu. Kucherenko, S. Danzenbächer, C. Krellner, C. Geibel, M. G. Holder, L. V. Bekenov, T. Muro, Y. Kato, T. Kinoshita, S. Huotari, L. Simonelli, S. L. Molodtsov, C. Laubschat, and D. V. Vyalikh, Phys Rev B 84, 245114 (2011)

[3] R.A. Mole, M. Hofmann, D.T. Adroja, O. Moze, S.J. Campbell, J Magn Magn Mater, 347, 86 (2013)

[4] M. Hofmann, S.J. Campbell and A.V.J. Edge, Phys. Rev. B 69, 174432 (2004)

[5] D. H. Ryan, Rasa Rjali, J.M. Cadogan, R. Flacau and C. D. Boyer. J. Phys.: Condens. Matter 28, 166003 (2016)

Topic:

Physics

Poster Session / 27

Linear Spin Wave Theory and TOF spectrometry: the perfect match

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Inelastic neutron scattering has long been the tool of choice for the study of spin waves and magnons, with the first reports stemming from the seminal work of Brockhouse. In the intervening decades time-of-flight spectrometers have become the tool of choice for the study of complex magnetic materials due to the large volume of reciprocal space investigated. While in more recent times the measurement of spin waves in powders has become possible due to the implementation of large position sensitive detectors, which allow for much more detailed spectra to be determined. Until

recently, obtaining the spectra was much more straightforward than modelling it – resulting in a bottleneck for data analysis. However the implementation of codes for spin wave theory [1,2] has meant that this is now fairly straightforward even for complicated cases such as low symmetry structures, incommensurate magnetic structures and powder samples. In this contribution we will summarise some recent successes from work performed at ANSTO in this field such as the use of powder averaging and the study of quantum spins.

[1] S. Toth, B. Lake *J. Phys. Cond. Mat.*, 27, 2015, 166002

[2] <http://www-llb.cea.fr/logicielsllb/SpinWave/SW.html>

Topic:

Physics

Poster Session / 28

Inelastic Neutron Scattering and Thermoelectric Materials – Examples from PELICAN

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Research on thermoelectric (TE) materials have been an active field for the past decade as TE material can potentially be used to capture and recycle waste-heat into electricity. Significant developments have been achieved in the search for the next-generation of advanced thermoelectrics that could play an increasing role in sustainable technology. One of the strategies in improving the performance of a thermoelectric material is to decrease the thermal conductivity, which is directly related to the lattice dynamics of the materials. Measurement of phonon density of states and phonon dispersion as a function of temperature can provide fundamental understanding of the thermal conductivity in terms of, for example, anharmonic vibrations and low energy rattling modes. PELICAN has been actively used for measuring phonon density of states for many systems, such as, CuSe₂, Ca₃Co₄O₉, BiCuSeO, BiCuTeO and Ti₂O₃. The capability of the PELICAN instrument in this field will be demonstrated with these outcomes.

Topic:

Physics

Poster Session / 52

Role of competing magnetic interactions and anisotropies in determining the ground states in magnetoelectric/multiferroic honeycomb M₄Ta₂O₉ (M= Mn,Co,Fe and Ni)

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In magnetoelectrics (ME) magnetic ordering and electric polarization (EP) coexist. The ME effect is larger, if in these materials EP is of spin origin and the coupling is strong. This coupling can be utilized in applications such as MRAMs. Although technically important, the coupling mechanism is complicated. Two types of materials fulfill this requirement: (i) Type II multiferroics (ii) Materials which lack spontaneous EP in the ground state and it is induced by an external magnetic field. The magnetic order parameter should break both time reversal and inversion symmetries [1-4]. M₄A₂O₉ (M=Mn,Co,Fe and Ni and A=Nb,Ta) are a rare family of materials where depending on M, either (i) or (ii) are induced below the magnetic ordering [5-8]. The structure is built from an alternative stacking of two different honeycombs leading to a competition between various magnetic interactions, anisotropy and dimensionality. In order to elucidate the emergence of (i) or (ii) in M₄A₂O₉, a comprehension of these competing interactions is essential, which in turn also necessitates the investigation of its electronic structure. In this work, we combined neutron powder diffraction, inelastic neutron scattering and theoretical methods including density functional theory to determine the magnetic structures, magnetic excitations and electronic structure of M₄Ta₂O₉. These investigations led to a discovery of a variety of fundamental spin systems (easy-axis vs easy-plane) and excitations (gapped vs ungapped) in this family of materials, which exhibit a variety of ground states [9].

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[6] N. D. Khanh et al., PRB 93, 075117 (2016).

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[8] S. N. Panja et al., PRB 98, 024410 (2018).

[9] N.Narayanan et al., accepted to be published in PRB.

Topic:

Physics

Poster Session / 80

The Magnetic Exchange Pathways of Fe₄Si₂Sn₇O₁₆

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Below $T_N = 3.0$ K, the bi-layered $\text{Fe}_4\text{Si}_2\text{Sn}_7\text{O}_{16}$ possesses a frustrated Kagome lattice of Fe sites within its $\text{FeO}_6/\text{SnO}_6$ oxide layer¹. This magnetic structure contains canted AFM chains whose sites are AFM w.r.t. neighbouring chain sites along y and xy (bridged by NM Sn and frustrated Fe sites) and along z (~ 9 Å apart and through a non-magnetic FeSn_6 cluster layer).

DFT calculations of the electronic and magnetic structure of $\text{Fe}_4\text{Si}_2\text{Sn}_7\text{O}_{16}$ are used to investigate the mechanisms behind the intralayer and interlayer magnetic ordering. Initial bonding analysis of the oxide layer show possible separate AFM super-exchange pathways along x and y, with an additional pathway proposed in 2017¹ also being investigated alongside (an AFM 'super-super' exchange between diagonally opposite sites along xy and y).

The question of the exact state bonding and charge distribution within the cluster layer has also been investigated computationally as this would be a critical factor affecting the interlayer ordering.

Similar structural trends were seen in computational models with Ru substitution into the cluster layer and with Mn doping into the oxide layer, the latter is in line with NPD studies of $\text{Fe}_{1.45}\text{Mn}_{2.55}\text{Sn}_7\text{O}_{16}$ which shows the same magnetic ordering as $\text{Fe}_4\text{Si}_2\text{Sn}_7\text{O}_{16}$ ². Future NPD studies of Ru and simultaneous Ru/Mn substituted compounds are being pursued to compliment this theoretical work.

Topic:

Physics

Poster Session / 73

Investigations into the Magnetic and Crystal Field Excitations of the Orthorhombically Distorted Perovskites TbVO_3 and CeVO_3

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Inelastic neutron scattering experiments have been performed on a series of vanadates, in particular TbVO_3 and CeVO_3 , to categorise the crystal field and magnetic excitations. The vanadates possess a configuration with corner sharing, distorted VO_6 octahedra (space group Pbnm) with a collinear C-type antiferromagnetic structure occurring below Néel temperatures of $T_N = 110$ K and 124 K respectively [1-4]. Data from neutron scattering experiments reveal a hitherto unobserved shift of

crystal field excitation energy in TbVO₃ and CeVO₃. Point-charge model calculations have confirmed this shift by theoretically calculating the crystal field excitation spectrum. We propose that the mechanism behind the effect is the onset of local magnetism caused by the ordering of the vanadium sublattice at the magnetic phase transition. This magnetic exchange field from the vanadium ions polarises the spins of the rare-earth ions located at the centre of the unit cell. This results in a Zeeman-like splitting of crystal field energy levels. As a result, crystal field transition energies demonstrate a linear shift as a function of internal magnetic field strength.

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Topic:

Physics

Poster Session / 50

Investigation on the Nature of the Verwey Transition in Cu-doped Fe₃O₄

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Magnetite (Fe₃O₄), the oldest known magnet, is still a hotly debated material in scientific research, due to its complex magnetic, electronic and transport properties. One of the most interesting physical phenomena associated with Fe₃O₄ is the occurrence of a metal-insulator transition at ~120 K (TV), the so-called Verwey transition, which is associated to a charge ordering below TV, accompanied with a structural transition from the cubic phase to the monoclinic phase. However, due to the twinning of crystal domain, the detailed crystallographic structure is not fully solved yet and different charge ordered and bond-dimerized ground states have been proposed. In order to overcome this problem, we have investigated Cu-doped Fe₃O₄ and have determined the stability range of the Verwey phase in the phase diagram of Fe_{1-x}Cu_xFe₂O₄. Using neutron diffraction and high-resolution X-ray synchrotron diffraction we have investigate both the crystallographic and magnetic structure of Cu-doped Fe₃O₄ (Cu_xFe_{3-x}O₄ with x = 0 to 1.0) in order to elucidate the effect of doping on the Verwey transition. The obtained data indicate that the Verwey transition remains unchanged up to highest doping levels of 75% Cu-substitution. This large stability range of the Verwey phase is surprising and did require a systematic investigation. The analysis of our high-resolution diffraction data did allow us to extract detailed information on the precise doping mechanism, for example if the Cu-ions are placed on tetrahedral or octahedral sites in the spinel structure. The obtained data therefore provide valuable information on the charge order, i.e. the Verwey transition.

Topic:

Physics

Poster Session / 49

Vibrational modes of n-octane: a theoretical comparison to neutron spectroscopy results

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A number of different calibration samples are used on the Beryllium Filter Spectrometer on TAIPAN. Normal-octane is used extensively as a calibration sample because of its well known vibrational modes that are clearly and quite regularly spaced apart over the entire energy range that is covered.

On the other hand alkane chemistry presents itself as an interesting solid-state physics problem. Alkanes exist in several different condensed phases, for example for the low-temperature phase for chain lengths between 8 to 21 carbons, even numbers of carbons produce a triclinic phase whilst odd numbers may be either triclinic or orthorhombic. Behaviour and hence crystallography is dependent on both the electronic structure and the dynamics involved between the different possible vibrational modes and excitations.

Neutron spectroscopy is a wonderful tool to study the dynamical nature of materials and in particular alkanes and similar organic compounds. The short-range nuclear interaction introduces isotopic sensitivity to the measurement, which is governed by the weak nucleon-nucleon interaction, and which gives a very large scattering cross section in the case of hydrogen of 82 barn. In this way a weighted vibrational density of states that is highly sensitive to hydrogen in a material may be measured from polycrystalline (powdered) material. Here, neutron spectra are compared directly to force-field calculations, semi-empirical quantum chemistry calculations, and solid-state density functional calculations. Any overview of the theory is given along with the steps required in analysis.

Topic:

Physics

Poster Session / 81

Nanoparticles in lyotropic liquid crystals: the structural and rheological effects due to size and surface chemistry

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Lamellar liquid crystals consisting of water, para-xylene, and Triton X-100 were formulated and were examined as silica nanoparticles of differing size and surface chemistry were doped in. The particles, regardless of size, were found to likely migrate to the liquid crystal domain boundaries, where they influence the structural and rheological properties of the mesophase. Consequently, phases with doped-in silica were not only found to be stiffer, but also had differing relaxation properties dependant on the type of silica nanoparticle present, and certain sizes and chemistries favoured the presence of a previously undiscovered mesophase in the phase diagram of the system. These results allow for a more comprehensive understanding of how liquid crystal systems act as solvents and host systems, which in turn allows for more sophisticated uses in catalysis, microfluidics, and protein crystallisation media.

Topic:

Soft Matter

Poster Session / 38

The Relationship of Li⁺ Displacement and Temperature in the Garnet Oxides Li_{7-x}La₃Zr_{2-x}TaxO₁₂

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The lithium containing garnet oxides of Li₇La₃M₂O₁₂ (M = Ta, Nb) were reported to have Li⁺ conducting abilities back in 2003.¹ Since then lithium containing garnet oxides have been identified as a electrolyte candidate for lithium ion batteries as they exhibit the physical and chemical properties desired for a solid-state electrolytes.² The most notable garnet oxide material is the cubic phase (Ia-3d) Li_{6.4}La₃Zr_{1.4}Ta_{0.6}O₁₂ which has a reported Li⁺ conductivity of 1×10^{-3} S cm.^{1,3} Here the structures and Li⁺ site occupancies of garnet series of Li_{7-x}La₃Zr_{2-x}TaxO₁₂ (x = 0, 0.25, 0.50, 0.75, 1.00) was further explored. A one step heating synthesis was employed for garnet preparing. Successful cubic phase garnet synthesis was confirmed with X-ray powder diffraction. Determination of lithium occupancy at the Li₂₄ and Li₉₆ sites was achieved with high-resolution neutron powder diffraction. NPD data were collected at room temperature, 200°C, 400°C and 600°C.

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Topic:

Advanced Materials

Poster Session / 76

Structural investigation of doped quaternary antimonates

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Copper containing oxides hold a widespread research interest in inorganic fields due to the fascinating electronic and magnetic properties the compounds exhibit. In the Cu-Sb-O ternary system, CuSb₂O₆ is the most intensively studied compound [1], owing to its unusual structural and magnetic behaviour. Jahn-Teller distortions from the Cu²⁺ cause an axial elongation of the Cu-O octahedra to give rise to a monoclinic structure (s.g. P2₁/n) [2]. At high temperatures, this material undergoes a second order phase transition to the tetragonal phase (s.g. P4₂/mnm), isostructural to room temperature structures of CoSb₂O₆ and NiSb₂O₆ [3]. This modification may only be possible through an intermediate orthorhombic modification in Pnma as defined through systematic symmetry reduction[4]. Through the doping of CuSb₂O₆ with Co and Ni, this structural transition can be investigated.

Neutron, lab X-ray and synchrotron single crystal and powder diffraction have been used to study phase transitions in both solid state solutions. In the Cu_{1-x}CoxSb₂O₆ system, it was found that two phases exist between compositions x = 0.2 and 0.5, with a Cu-rich monoclinic phase and a Co-rich tetragonal phase. The magnetic susceptibility for all compounds match closely to CoSb₂O₆,

even at low doping levels. This indicates a change from the 1D magnetic behaviour of CuSb₂O₆ to 2D. By contrast, the Cu_{1-x}Ni_xSb₂O₆ system exhibits a single phase region from $x = 0.4$, where only the tetragonal phase remains. This has been attributed to a reduction of Cu²⁺ due to the high temperatures used in synthesising these compounds.

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Topic:

Chemistry

Poster Session / 5

Interaction of cinnamic acid and its analogues with Pluronic® micelles

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The interaction of cinnamic acid (CA) and its analogues viz. p-coumaric acid (PCA) and caffeic acid (CFA) with core-shell micelles of a moderately hydrophobic Pluronic® P123 has been investigated using cloud point (CP), viscosity, dynamic light scattering (DLS), small-angle neutron scattering (SANS) and steady-state fluorescence measurements. These solubilizates alter micellar behaviour of copolymer solution dependant on their hydrophobicity. P123 micelles exhibit time-dependent restructuring and growth processes and at different rates which is pH dependent and responsive to the presence of a salt. Observed results demonstrate that restructuring and growth of polymer micelles can be tuned with ease upto a substantial extent just by changing the concentration of the additives and the pH of the solution. Considering the medical applications of cinnamic acid and its analogues and of Pluronic® in drug delivery systems, the present study can provide important insight of the possible time-dependant delivery mechanism of drugs.

Topic:

Biology

Poster Session / 30

Probing the Structure and Function of Tethered Bilayer Membranes by Neutron Reflection.

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Tethered Bilayer Membranes (TBMs) are a versatile system for studying models of the cell membrane bilayer architecture and form the basis of a number of medical nano-sensing devices. These systems are typically tethered to a conductive substrate to enable the electrical signal, indicating the

functionality, of the device to be recorded. The fact that these devices operate fully immersed in solution and rely upon organic/biochemical molecules (low electron density) against a gold surface (high electron density) rule out many structural experimental probes, including x-rays. Fortunately neutrons provide both the penetrating power to enable the interface to be probed and the appropriate contrasts to allow one to study submerged biomolecules on a gold surface at high resolution.

This presentation will give a taste of two current projects, firstly fundamental studies of bilayer architecture and the impact of solution pH and hence hydrogen bonding, on the bilayer porosity and thickness. Secondly I will also outline work undertaken on producing a bilayer that mimics the structure of the Gram Negative Bacteria (GNB), including interactions with currently used antibacterial drugs. Understanding the mechanism of antibacterials gives confidence in applying this platform for screening of new drugs.

Topic:

Biology

Poster Session / 12

Using Neutron Reflectometry to Understand Antibiotic Resistance in Gram-negative Bacteria at the Outer Membrane

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With bacteria increasingly becoming resistant to common antibiotics, we are currently heading for a post-antibiotic world, where treatable common ailments are suddenly untreatable. This means that there is now considerable research effort in understanding how antibiotic resistance arises, and in creating a new generation antibiotics. The outer membrane is the first line of defence against antibiotics for Gram-negative bacteria. Being able to penetrate the outer membrane is essential to designing effective antibiotics and antimicrobial peptides. The outer membrane is an asymmetric bilayer consisting of phospholipids on its lower leaflet and lipopolysaccharides on its environment-facing outer leaflet. This work will present on creating model outer membranes from *Pseudomonas aeruginosa*, a bacterium that is normally harmless, but infections from which can prove to be problematic for those that are immunocompromised. Worryingly, *P. aeruginosa* is showing increasing signs of becoming resistant to Polymixin B, an antibiotic of last resort. Certain biochemical modifications to lipid A (a component of lipopolysaccharides) can confer resistance to Polymixin B. Model *P. aeruginosa* outer membranes using lipid A with different modifications were created on silica surfaces using Langmuir-Blodgett and Langmuir-Schaeffer deposition techniques. Model outer membranes created this way are ideal tools for studying the binding antimicrobial peptides because: a) they reflect the lipid composition of the membrane, b) reflect the fluidity of the membrane, and c) maintain the asymmetric nature of the outer membrane. The nanoscale structures of the membranes were determined using neutron reflectometry and it was observed that Polymixin B was unable to penetrate into bilayers that consist of de-acylated lipid A. New drug targets Octapeptin A3 [1], and modified Polymixins FADDI-019 and FADDI-020 [2] were tested and found to disrupt membranes composed of modified lipid A which confer resistance to Polymixin B.

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Topic:

Biology

Poster Session / 93

Electrochemical fabrication of mesoporous gold-electrode towards the ultrasensitive detection of microRNA

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Nanostructured gold electrodes have attracted immense interest for a broad range of applications, especially in biosensing due to their unique biocompatibility, stability, sequence dependent adsorption of nucleic acids, and high throughput optical and electrochemical responses. Integration of tuneable mesopores into gold electrodes offers high surface area with voluminous active sites for releasing and uptaking guest biomolecules, thereby remarkably increasing electrochemical responses[1].

Herein, we report an electrochemical preparation of a new class of mesoporous gold (film) electrode (MPGE) using diblock copolymer-micelles (polystyrene-block-polyoxyethylene; PS-b-PEO) as a soft-temple. The PS-b-PEO spherical micelles were formed through the interaction of aqua-AuCl₄⁻ ions with hydrophilic EO blocks near the outer layer providing PS cores. Under the optimal applied potential, the composite micelles were deposited to the working electrode surface. After that, the PS-b-PEO micelles were completely removed. The SEM images exhibited the presence of uniformly sized (average size 25±5 nm) mesopores. In-depth electrochemical characterization of the resultant electrode were also carried out using popularly used [Fe(CN)₆]^{3-/4-} redox biomarker for nucleic acid (microRNA) sensing. The MPGE exhibited around 10 folds-higher activity than that of planar gold electrode. This highly active MPGE shows great potential towards the direct adsorption of miRNA and subsequent electrochemical (differential pulse voltammetry-DPV) interrogation of adsorbed miRNA in presence of [Fe(CN)₆]^{3-/4-} system.

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Topic:

Advanced Materials

Poster Session / 8

Strong synergism in oppositely charged mixed surfactant systems: self assembly from surface tension, rheology and SANS studies

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Micellization and microstructure of mixed micelles from mixtures of an anionic fluorocarbon surfactant sodium perfluorooctanoate (NaPFO) and conventional anionic surfactant sodium oleate (NaOl) with cationic alkyl methyl-imidazolium based non-amphiphilic and amphiphilic ionic liquids (ILs) with different alkyl chain length (C4, C6, C8 and C10) and anions viz. chloride (Cl⁻), tetrafluoroborate (BF₄⁻), hexafluorophosphate (PF₆⁻), octylsulphate [C₈SO₄⁻] and trifluoromethane sulphonate (CF₃SO₃⁻) were examined by tensiometry, rheometry and small angle neutron scattering (SANS) measurements. Low critical micelle concentration (CMC) and large negative value of interaction parameter (β) showed synergism in all the mixed systems and it depends on molecular characteristics

of ILs. Higher negative value of β for NaPFO+ILs compared to NaOI+ILs shows strong synergistic interaction in perfluoro anionic surfactant system. Thermodynamic parameters for mixed monolayers and mixed micelles were evaluated and discussed. Micellar growth, shape changes and micelle to vesicle transitions obtained from these oppositely charged mixed systems depend on the surface activity of ILs (alkyl chain and counterion). The data for size, shape and aggregation number for aggregates formed by these mixed systems evaluated from the fitted SANS data are reported and discussed.

Topic:

Soft Matter

Poster Session / 10**Advancing the reflectometry cause at ANSTO - updates and upgrades to the time-of-flight Platypus Neutron Reflectometer****Author(s):** Andrew Nelson¹**Co-author(s):** Stephen Holt²; Frank Klose; Anton Le Brun¹; Wai Tung Lee²¹ ANSTO² Australian Nuclear Science and Technology Organisation**Corresponding Author(s):** andrew.nelson@ansto.gov.au

Since the first suite of neutron scattering instruments was commissioned in 2008 the Australian Nuclear Science and Technology Organisation (ANSTO) has invested in instrumentation for the analysis of thin interfacial films. The horizontal time-of-flight reflectometer, Platypus, has now been joined by an X-ray reflectometer and a variable angle spectroscopic imaging ellipsometer. The high quality science possible on these instruments has led to a large oversubscription rate on Platypus. Here, we outline the key developments and upgrades we have made to Platypus that have led to this success. These include the development of event mode acquisition for studying kinetic processes, new sample environments (confinement cell, vapour delivery systems), as well as projects to upgrade its performance by installing new collimation systems and detectors.

Topic:

Neutron Instruments & Techniques

Poster Session / 18**Structure and picosecond dynamics of liposomes****Author(s):** Kathleen Wood¹**Co-author(s):** Richard Mole²; Michihiro Nagao; Robert Knott²; Paul Butler¹ Australian Nuclear Science and Technology Organisation² ANSTO**Corresponding Author(s):** kwo@ansto.gov.au

Membrane formation is often listed as one of the key steps in the origins of life, since this enabled the first organisms control over the environment around their genetic material. Unilamellar liposomes are one of the simplest models available of cellular membranes, and we have used small angle neutron scattering and neutron spectroscopy to characterize their structure and dynamics [1,2]. Biomolecules exhibit dynamics over a wide range of time scales [3] and the concept of a hierarchy

of motions from larger scale slower motions and faster smaller motions provides a theoretical framework [4]. Which timescales are crucial to biological function, and how motions on the different timescales correlate with each other remain open questions in membrane biophysics. Spin echo spectroscopy results on liposomes showed that above the lipid transition temperature fluctuations in the bilayer thickness could be observed on the order of 100 ns [1]. It is frequently assumed that such larger scale motions are 'lubricated' by faster motions. After characterization using small angle neutron scattering, we have studied the dynamics of liposomes on the ps timescale accessible on the PELICAN spectrometer at the Australian Centre for Neutron Scattering [2]. We observe a correlation between the bilayer fluctuations at 100 ns and the ps timescale.

Bacterial and eukaryotic cells differ in their lipid composition, notably in an increase in charged lipids for bacterial membranes. We therefore studied charged and uncharged liposomes using pure DMPC and mixtures of DMPC/DMPG. Small angle scattering shows that including charge in the membrane increases the bilayer thickness, while quasielastic neutron scattering indicates an increase in membrane mobility with increasing charge.

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Topic:

Biology

Poster Session / 25

Recent highlights from the PELICAN spectrometer

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Here we present some recent developments on sample environment equipment at ACNS, which markedly improve the existing capabilities for a range of beamline instruments, leading to greater scientific outcomes and operational efficiency.

A six-position thermalised sample changer with tumbling capability, which avoids sedimentation and particle size separation in samples suspended in a medium, has been designed for use on US-ANS. To date, USANS experiments have successfully been completed with a prototype applying both independent temperature control and tumbling. An equivalent thermalised sample tumbler for use on SANS is currently undergoing development.

A new sample positioning probe made from composite materials has been designed to reduce the sample cooldown time in top-loading cryostats. Fabricating probes from thin-walled carbon fibre, which is both lightweight and a poor thermal conductor, has resulted in a two-thirds reduction in the sample cooling time when compared with standard stainless steel sticks. Future work will look to incorporate these features in other applications such as for gas delivery probes.

The Rotational Paris Edinburgh Cell, capable of providing both high pressure and controlled high shear to investigate in areas of physics, chemistry, earth and planetary sciences, has been commissioned at ACNS. It is believed to be one of only two high pressure/shear Paris Edinburgh presses worldwide. This added sample environment capability opens up new experimental possibilities in high pressure – high shear regimes for users. A stretch goal is to add a high temperature capability to the ROPEC, creating a rare trio of extreme temperature – pressure – shear.

Topic:

Neutron Instruments & Techniques

Poster Session / 47

Small molecule profiles in stacks of lipid bilayers by neutron diffractionChris Garvey¹ ; Gary Bryant² ; Robert Georgii³¹ ANSTO² Centre for Molecular and Nanoscale Physics, School of Applied Sciences, RMIT University³ Technical University Munich**Corresponding Author(s):** cjg@ansto.gov.au

While well established for studying the internal structure of bilayers and orientation of peptides and proteins with respect to bilayers, neutron lamellar diffraction has a powerful insight the average locus of solubilisation of small molecules in stacks of lipid bilayers. The approach has provided unique insights into important issues in anhydrobiology and cryobiology, where previously only molecular dynamics simulations (MD) had been able to provide molecular scale insights. Currently we use the methodology to understand the interaction of cryoprotectants with bilayers, with the aim of providing important validation of MD parameters to further enhance the utility of the method. Here we discuss the experimental approach, both in terms of the use of contrast variation and the use of selective deuteration to simplify the extraction of scattering length density profiles within the bilayer.

Topic:

Soft Matter

Poster Session / 65

Multiferroic thin films investigated by neutron diffractionClemens Ulrich¹¹ The University of New South Wales**Corresponding Author(s):** c.ulrich@unsw.edu.au

Artificially grown thin film heterostructures of transition metal oxides by far exceed the capabilities of current semiconducting technology as they offer additional functionalities such as metal-insulator transitions, magnetism, superconductivity, or multiferroicity. Bismuth ferrite (BiFeO₃) is the rare case of a room temperature multiferroic material and offers as such the most promising pathway for spintronics applications. The existence of a spin cycloid, which is mandatory for magneto-electric switching, is hindered in thinnest films due to the large epitaxial strain. Our neutron diffraction experiments have demonstrated that we were able to realize a spin cycloid in thinnest films through improved electrostatic and epitaxial constraints [1] and the use of Co-doping. This cycloid, despite its out-of-plane propagation vector, can be stabilized in films as thin as 25 nm, a length smaller than the cycloid period itself. The cycloid expands significantly for thinnest films and as a function of temperature close to TN and showing a distinct systematic scaling behaviour.

A further fascinating example are SrCoO₃ thin films. Theoretical calculations have predicted ferromagnetic to antiferromagnetic phase transitions induced by epitaxial strain. With the proper choice of substrate material we were able to confirm the FM-AFM transition by neutron diffraction [2]. As such, SrCoO₃ would constitute a new class of multiferroic material where magnetic and electric transitions can be driven through external strain. This opens new avenues for fundamental research and technical applications in spintronic or magnonic devices.

In collaboration with: J. Bertinshaw, S. J. Callori, S. R. Burns, D. Sando, B. Xu, B. Dupé, L. Russell, G. Deng, L. Bellaiche, J. Seidel, and N. Valanoor.

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Topic:

Advanced Materials

Poster Session / 84

Photo-Responsive Lyotropic Liquid Crystals using Unconventional Solvents

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Liquid crystals combine the short to medium range order of crystalline structures with the macroscopic flow properties of liquids. They are of interest because of these and other properties, including optical birefringence. The azobenzene functional group readily forms liquid crystalline phases, and is of interest because of its ability to reversibly switch between trans and cis (E/Z) conformations when irradiated with UV and blue light respectively.

A range of azobenzene molecules based on the 4((4 hydroxyphenyl)diazenyl)benzoic acid motif, with varied amide coupling to the benzoic acid functional group have been synthesized for lyotropic liquid crystalline systems, allowing for a facile route to induce conformational change in the azobenzene and structural change in the aggregation of the molecules in the liquid crystal.

While azobenzenes typically have poor solubility in conventional solvent systems, they display good solubility in many more unconventional solvents including DMSO and ammonium containing ionic liquids (ILs). Ionic liquids generally have near zero vapour pressure and can be non-flammable, which makes them ideal candidates for a range of commercial applications.

The phase behaviour of these photo-stimuli responsive, lyotropic LCs has been determined using characterization techniques including polarizing light microscopy, SAXS, and SANS. Further development of these systems aims to create a range of responsive optical and optoelectronic materials as smart sensors and actuators.

Topic:

Soft Matter

Poster Session / 54

Position Resolved Profiling of Flocculated Sediment Structure with USANS

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Gravity thickeners operate by mixing slurry with polymer to produce flocculated aggregates which settle much faster than isolated particles. Applying low shear rates through rakes and sloped walls, flocculated aggregates densify, making them settle even faster. Direct observations of aggregate densification have been limited to light scattering and video analysis. However, current analysis methods pre-suppose a fractal structure which becomes invalid as aggregates densify.

USANS (Kookaburra) experiments were conducted to quantify the impact of shear and compression on the structure of flocculated calcite aggregates through in-situ shear. Due to difficulties with maintaining constant shear, a novel new method was developed and trialed to spatially resolve the scattering of a sedimented sample as a function of vertical height by moving the sample relative to a rectangular aperture (nominally 4 mm high and 15 mm wide). Inspired by a spatially resolved SANS method (Harti, Strobl et al. (2017)), an adaption of this method with a modification of the USANS detector location has the potential for significantly improved spatial resolution. Promising preliminary results have recently been obtained by dark field imaging from sedimenting flocs at a test beam line at the Paul Scherrer Institute (Villigen Switzerland).

Current research seeks to produce vertically resolved data (1-D imaging) from the USANS Kookaburra instrument to quantify the impact of shear and compression on the vertically resolved structure of sediments formed from flocculated calcite aggregates subject to different shear histories, thus providing a basis for understanding how to best identify, quantify and exploit the aggregate densification phenomenon for large scale industrial processing.

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Topic:

Neutron Instruments & Techniques

Poster Session / 35

Effects of Nb-doping on phase evolution and electrochemical performance of Li-rich $x\text{Li}_2\text{MnO}_3 \cdot (1-x)\text{Li}[\text{Ni}_a\text{Cob}_b\text{Mnc}_c]$ cathode materials

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Li-rich $x\text{Li}_2\text{MnO}_3 \cdot (1-x)\text{Li}[\text{Ni}_a\text{Cob}_b\text{Mnc}_c]\text{O}_2$ ($a+b+c=1$) cathode materials are promising candidates for lithium-ion batteries owing to their high practical capacity (250-280 mAh/g). But its practicality is hindered due to the intrinsic voltage fade, large irreversible capacity loss, low rate capability, and structural instability issues. Elemental doping strategies are already shown to be effective in enhancing the rate capability and limiting the voltage decay in these materials. [1] Nb⁵⁺ doping is already tried in Li-rich layered oxide particles are proven to be useful in stabilizing the structure and thereby enhancing the electrochemical performance. [2, 3]. However, an in-depth analysis regarding the effect of Nb⁵⁺ doping in the crystal structure, as well as its mechanistic reactions, of Li-rich layered oxides is lacking. In this work, we combine high-resolution neutron powder diffraction and in-operando synchrotron X-ray powder diffraction to study the detailed crystallographic structure of such materials and the corresponding mechanistic behaviours upon lithiation and delithiation, under the effects of Nb⁵⁺ doping. Nb⁵⁺ ions doped into the transition metal ions sites will not only expand the layered structure, improving Li-ion diffusion and hence the rate capability,[2], but also,

more importantly, Nb⁵⁺ ions may tune the lattice oxygen activities, accompanied phase transition and structural degradations which are believed to be the root cause of discharge voltage fading in Li-rich layered oxides.

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Topic:

Chemistry

Poster Session / 69

Surface Characterization of Insect Olfactory Receptors based Bio-electronic Nose

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The ability to express and purify insect odorant receptors (OrXs) and reconstitute them into artificial membranes such as liposomes and nanodiscs has paved the way for their use as recognition elements in biosensors and also to aid in structural and functional experiments. In this study, we have used surface characterization techniques including ellipsometry, quartz crystal microbalance with dissipation monitoring (QCM-D) and electrochemical impedance spectroscopy (EIS) to understand the various properties of an insect odorant receptor (D.melanogaster Or22a) integrated into the lipid bilayers of liposomes when immobilized on a gold surface. Neutron reflectivity studies under three contrasts (D₂O buffer, H₂O buffer and gold matched water) were carried out at various stages of an Or22a based biosensor fabrication to further investigate the properties of these receptors when covalently attached on gold wafer and also their interaction with an odorant (methyl hexanoate). These studies confirmed that Or22a/liposomes can be successfully immobilized on gold surfaces while preserving the receptor function as well as maintaining the integrity of the membrane. After the binding of methyl hexanoate to Or22a, EIS indicated a decrease in resistance, QCM-D indicated a decrease in mass, and Neutron reflectivity indicated a decrease in thickness; all of these changes can be related to a structural and/or conformational change in the Or22a protein which will require more detailed investigations in the future.

Topic:

Chemistry

Poster Session / 78

Investigating Long-range Magnetic Ordering in Li₃Co₂SbO₆ a layered oxide honeycomb lattice

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Over the last decade layered-honeycomb oxides have come to increasing prominence as materials that exhibit an array of interesting properties, including use as electrodes for Li-ion batteries, high electrical conductivity, low temperature magnetic phases, and spin-glass transitions. Layered oxides with the general formula $A_xM_2XO_6$ ($A = \text{Li, Na}$; $M = \text{transition metals eg. Co, Cu, Ni}$; $X = \text{Bi, Sb, Te}$; $0 \leq x \leq 3$) are a form of 'honeycomb' lattice where one third of the transition metal sites are doped with high charge cations such as Sb^{5+} and Te^{6+} . More recently honeycomb lattices have been investigated as Kitaev lattices and potential Quantum Spin Liquids (QSL).(1)

My project involves the conventional solid-state synthesis of $\text{Li}_3\text{Co}_2\text{SbO}_6$ and the $\text{Na}_{3-x}\text{Li}_x\text{Co}_2\text{SbO}_6$ solid solution. The focus is on investigating the magnetic behaviour of these systems, including antiferromagnetism below the Néel temperature(TN) and magnetic frustration within the 2D honeycomb layers. In addition, the Na analogue $\text{Na}_3\text{Co}_2\text{SbO}_6$, possible QSL properties, as: is predicted to be a material which is a QSL.

Low-temperature neutron powder diffraction provides the means to investigate the structure of these materials and the magnetic lattice below TN. The spins are predicted to align in an antiferromagnetic 'zig-zag' arrangement.(2) Inelastic neutron scattering provides a means to verify if these materials do in fact become the unusual QSL as some have predicted.

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Topic:

Chemistry

Poster Session / 83

Novel Light-Responsive Surfactants for Formation of Single Component Photo-Rheological Fluids

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Research into the development and understanding of photo-rheological fluid formulations has gained significant momentum in recent years due to their potential for widespread application in fields such as biomedicine, nanotechnology and oil drilling. Imparting of photo-controlled viscosity to a solution can be achieved by formation and destruction of elongated worm-like micelle surfactant aggregates. While many successful photo-rheological fluid formulations have been developed using this method the overwhelming majority are quite complex, using multiple components each required in specific concentrations.

We are currently developing and synthesising a library of novel Azo-Betaine surfactants, with the primary goal of using them to create simple single component photo-rheological fluid formulations. Additionally, using this library, we hope to better understand the relationship between a surfactant's chemical structure and its physical properties, both in the bulk solution and at the interface. These simplified systems will allow more widespread application of photo-rheological fluids, while the surfactant library will further our understanding of their structure-property relationship.

Currently we have a completed synthesis and testing of a small five molecule library of photo-surfactants, using small-angle neutron scattering to determine their aggregation structure in aqueous solution. Results from these initial surfactants show elongated rod-like micelles that revert to almost entirely spherical micelles on irradiation with UV light. This change in aggregation geometry exceeds any previously observed within our group when using single component photo-surfactant formulations. Additionally, we are beginning to understand the relationship between tail group length, bulk and the resulting surfactant aggregation properties for this novel class of surfactants.

Topic:

Chemistry

Poster Session / 31**Hydration mechanisms and proton conduction in the mixed ionic-electronic conductors Ba₄Nb₂O₉ and Ba₄Ta₂O₉**Chris Ling¹ ; Julia Wind¹ ; Richard Mole² ; Dehong Yu³ ; Max Avdeev⁴¹ University of Sydney² ANSTO³ Australian Nuclear Science and Technology Organisation⁴ Australian Nuclear Science and Technology Organisation, Australian Centre for Neutron Scattering**Corresponding Author(s):** chris.ling@sydney.edu.au

Mixed conductors – materials that exhibit significant mobility of more than one type of charge carrier such as oxide ions, protons and electrons – have a range of important applications including solid oxide fuel cell membranes, electrodes, batteries and sensors. We recently studied the behaviour of hydrogen in the mixed ionic-electronic conductors γ -Ba₄Nb₂O₉ and 6H-Ba₄Ta₂O₉, using a combination of experimental (neutron diffraction and inelastic neutron scattering) and computational (*ab initio* molecular dynamics) methods. While these compounds have isostructural low-temperature polymorphs, they adopt distinct forms in the high-temperature conducting regime. We found that they also have distinct mechanisms for hydration and ionic conduction. Hydration of γ -Ba₄Nb₂O₉ is localised to 2-D layers in the structure that contain a 1:1 ratio of isolated but adjacent NbO₄ and NbO₅ polyhedra. OH⁻ and H⁺ ions combine with two polyhedra respectively to form complete layers of NbO₄OH polyhedra, giving rise to a stoichiometric hydrated form γ -III-Ba₄Nb₂O₉.1/3H₂O. Protons then diffuse through these 2-D layers by “hopping” between oxygen atoms on adjacent polyhedra. In the case of 6H-Ba₄Ta₂O₉, hydration occurs by intercalating intact water molecules into the structure up to a maximum of ~0.375 H₂O per formula unit. This explains the unusual local and long-range structural distortions in the hydrated form observed by neutron diffraction. Diffusion then occurs by water molecules moving between neighboring symmetry equivalent positions. These fundamentally different hydration and proton conduction mechanisms explain why 6H-Ba₄Ta₂O₉ has the less well-defined and higher maximum water content, while γ -Ba₄Nb₂O₉ has the higher proton conductivity.

Topic:

Advanced Materials

Poster Session / 41**Investigation into the AlF₃ coating effect on the electrochemical behaviours in the LiNi_{0.5}Mn_{1.5}O₄//Li₄Ti₅O₁₂ full batteries by in-operando neutron powder diffraction and electrochemical characterization**

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Key words: lithium ion batteries, battery safety, output voltage, LiNi_{0.5}Mn_{1.5}O₄/Li₄Ti₅O₁₂ full battery, AlF₃ coating, in-operando, neutron powder diffraction, electrochemical performance

Lithium ion batteries (LIBs) with graphite anodes and LiCoO₂ cathodes have been widely used in our daily life. Since the operating voltage of graphite is close to that of the metal lithium dendrite formation, lowering the battery operating safety, Li₄Ti₅O₁₂ (LTO) anode is regarded as the promising alternative due to its excellent safety originating from its high working potential (~1.55V vs. Li/Li+) and superior structural stability ascribed to zero-strain lithium insertion. In order to enhance the output voltage of full batteries with LTO anode, the so-called 5V spinel LiNi_{0.5}Mn_{1.5}O₄ (LNMO) cathode is chosen. In the LNMO//LTO full battery, the LTO anode shows poor rate performance because of low intrinsic electrical conductivity and lithium-ion diffusion coefficient.

In this work, AlF₃ coating was applied on LTO to improve its rate capability in full batteries. The modified LTO anode exhibits significantly improved rate performance because of AlF₃ coating layer and parasitic Al doping (~3% onto the 16c crystallographic site of LTO structure) indicated by the joint refinement of X-ray diffraction (XRD) and neutron powder diffraction (NPD) data. In-operando NPD data also exhibits a smaller lattice parameter change of the modified LTO during lithiation and delithiation process. More interestingly, the AlF₃ coating on the anode LTO could also promote the rate of structural response of LNMO cathode shown by the in-operando NPD data of LNMO, suggesting faster delithiation and enhanced Li diffusion. We contribute this cathodic improvement to the protection of AlF₃ against HF and greater availability of lithium in the LNMO//AlF₃-coated LTO battery. Further details will be brought and discussed in the presentation.

Topic:

Advanced Materials

Poster Session / 64

Texture and its Impact on Thermal Conductivity in Miscibility Gap Alloys

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Miscibility Gap Alloys (MGA) are a new type of latent heat Thermal Energy Storage (TES) material that takes advantage of a miscibility gap between two elements or compounds, often metals or semi-metals. MGA encapsulate a distributed fusible phase for latent heat storage inside a matrix phase. Their storage capacity is dominated by the included phase but their high thermal conductivity is mainly a function of the matrix.

Many MGA are made with a graphite matrix due to its excellent thermal properties and its chemical stability with a host of materials. However, graphite crystals are highly anisotropic, so bulk graphite samples are susceptible to anisotropy, or "texture". This can occur naturally, or through the deliberate manipulation of the material in the manufacturing technique (e.g. using powder metallurgy techniques). It is possible to obtain much higher or lower properties (such as thermal conductivity)

in a desired direction; however, the perpendicular direction(s) will experience the opposite effect. When a material is only required to perform a function in one or two directions (e.g. radial heat transfer), anisotropy can be beneficial.

This work investigates the presence, effect and manipulation of texture in MGA samples with a graphite matrix based on varying manufacturing techniques, and its impact on thermal conductivity. Neutron diffraction is used to measure the texture within graphite matrix MGA. Direct measurement of thermal conductivity will then allow us to validate current texture to thermal conductivity correlations. Finally, this knowledge of anisotropic thermal conductivity within the MGA will allow for optimal placement of heat transfer piping and layout of the material within storage blocks, among other advantages.

Topic:

Advanced Materials

Poster Session / 66**Confinement effect induced high magnetic moment above room temperature of Co clusters in TiO₂****Author(s):** Xiang Ding¹**Co-author(s):** Jiabao Yi¹; Wai Tung Lee²¹ UNSW² ANSTO**Corresponding Author(s):** xiang.ding@student.unsw.edu.au

5% Co-TiO₂ film has been deposited using pulsed laser deposition. By delicately controlling deposition parameters, nanoclusters of Co are formed in the interface, confirmed by transmission electron microscopy, energy dispersive spectroscopy and X-ray absorption near edge spectroscopy. The film exhibits a very high saturation magnetization measured by magnetometer, equivalent to 6.54 μB/Co, given that the magnetic moment is all contributed from Co dopant. However, magnetic measurement by magnetometer can only give the overall information of the entire sample. To identify the surface dead layer and the magnetization in the interface where secondary phase or precipitation can easily be formed, we employed polarized neutron reflectometry (PNR) to identify the magnetic profile of the sample. From the peak of magnetic scattering length density, we can obtain that the highest saturation magnetization at the peak area is around 106 emu/cm³. The work indicates that very small nanoclusters under confinement environment can exhibit a very large magnetic moment above room temperature, which is promising for designing artificial high magnetization materials.

Topic:

Advanced Materials

Poster Session / 79**Magnetic Phase Transitions of New Sodium-ion Battery Cathode Na₄Ni₇(PO₄)₆****Author(s):** Qingbo Xia¹**Co-author(s):** Chris Ling²; Chun-hai Wang¹; Maxim Avdeev³; Siegbert Schmid¹¹ The University of Sydney² University of Sydney

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Sodium-ion batteries (NIB) are of intense current interest as alternatives to lithium-ion batteries for large-scale applications in which kinetics and weight are not the primary consideration. However, the jury is still out on the ultimate competitiveness of NIB versus other energy storage solutions, with one major uncertainty being a lack of Na electrode materials. [1] Monoclinic Na₄Ni₇(PO₄)₆ has demonstrated a great potential to be a new electrode candidate in our recent study. In addition, its complex magnetic properties also attracted our interest. [2] In this presentation, we mainly focus on investigation of the magnetic phase structures of Na₄Ni₇(PO₄)₆. According to the magnetic susceptibility analysis and variable temperature neutron diffraction measurements, Na₄Ni₇(PO₄)₆ presents three successive antiferromagnetic (AFM) ordered phases (Phase I, Phase II and Phase III) at 9.1-17K, 4.6-9.1K and <4.6K respectively, with the magnetic ordering vector [0, 1, 1/2], [0, 2/3, 1/2], and [0.076, 2/3, 1/2], refer to the nuclear unit cell. The magnetic ordering shows distinct ferromagnetic (FM) Ni²⁺ strips and antiferromagnetic arrangements between FM strips. The moment amplitude of all strips is equivalent in Phase I but varies in Phase II. Phase III is an incommensurate structure and should have a similar spin arrangement with Phase II.

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Topic:

Advanced Materials

Poster Session / 86

Magnetocaloric Mn(Co_{1-x}Ni_x)Ge - Structural and magnetic transitions

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The structural and magnetic properties of MnCoGe-based alloys have been studied extensively in recent years due to their potential application as magnetic cooling materials based on the magnetocaloric effect (MCE). The Mn(Co_{1-x}Ni_x)Ge series is of particular interest as magnetic transitions in the range 275 K to 345 K generally coincide with a martensitic structural transition T_M , with such an overlap then allowing scope for the formation of a magneto-structural transition (ferromagnetic orthorhombic to paramagnetic hexagonal) and hence an associated large MCE [e.g. 1].

Neutron diffraction, magnetisation and x-ray experiments on Mn(Co_{1-x}Ni_x)Ge compounds ($x = 0.12$ to 1.00) have demonstrated magnetic structures ranging from ferromagnetic for $x < 0.50$ to non-collinear spiral antiferromagnetic for $x > 0.55$ at low temperature (e.g. 5 K). T_M is found to decrease initially with increasing Ni content and then increase. First-order magneto-structural transitions are observed in Mn(Co_{1-x}Ni_x)Ge samples for $\sim 0.20 < x < \sim 0.65$ with the presence of ferromagnetic and antiferromagnetic structures in Mn(Co_{1-x}Ni_x)Ge allowing investigation of both direct and inverse magnetocaloric effects. Our results (including the magnetic phase diagram for Mn(Co_{1-x}Ni_x)Ge) are

discussed in terms of the increase of valence electron concentration on substitution of Ni ($3d^8 4s^2$) for Co ($3d^7 4s^2$) in the orthorhombic phase, leading to expansion of the unit cell and redistribution of the valence electrons [2].

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Topic:

Advanced Materials

Poster Session / 36

Multiple scattering in neutron Laue diffraction

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Apart from the specialised but important application to phase determination, multiple scattering in single-crystal neutron diffraction is usually an undesirable source of error. The presence of multiple scattering for a particular Bragg reflection can be verified by performing an azimuthal scan, if the diffractometer offers that possibility, or by varying the wavelength. Either of these checks is generally straightforward on a monochromatic diffractometer, where attention can be easily focused on a single reflection. Correction for multiple scattering ranges from rejection of the reflections affected in longer-wavelength experiments to subtraction of an empirically determined constant contribution from all data in shorter-wavelength experiments.

Little consideration has thus far been given to the contribution of multiple scattering to neutron Laue diffraction, due primarily to multiple scattering being less likely to occur in the samples best adapted to the technique. In addition, scanning a single reflection to detect the presence of multiple scattering is not the forte of Laue diffraction where many reflections are excited and detected simultaneously, and the broad waveband further complicates the possibility to correct for multiple scattering.

Here we show that the multiple scattering can occur in a neutron Laue experiment by using two single crystals (in a diamond-anvil high-pressure cell) to highlight the effect in the Laue pattern. The (few) observations in this unconventional experiment allow estimation of the magnitude of the effect in standard neutron Laue experiments both at reactor and spallation sources.

Topic:

Neutron Instruments & Techniques

Poster Session / 44

Combining USANS and SANS for micro and nanoscale structural characterisation

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Ultra-small and small angle neutron scattering (USANS and SANS) are versatile techniques for investigating the micro and nanoscale structure of materials such as food, surfactants, polymers, colloids, metals, minerals, and emulsions. These techniques have been exceptionally useful for studying complex materials of industrial importance in recent years. Various examples have emerged where combining USANS and SANS data have provided valuable knowledge.

Australia is the home of state of the art reactor based USANS and SANS instruments at the ACNS, ANSTO. Combining data from QUOKKA-SANS/BILBY-SANS with KOOKABURRA-USANS instruments provide a tool to study structures from 1 nm to >10 μ . In this talk we discuss some of the recent examples where combined USANS and SANS data have provided valuable understanding for various complex systems.

Topic:

Neutron Instruments & Techniques

Poster Session / 51**Small Angle Neutron Scattering instrument Bilby: typical experiments and scientific highlights**Anna Sokolova¹ ; Liliana de Campo² ; Andrew Whitten²¹ *Dr*² *ANSTO***Corresponding Author(s):** anna.sokolova@ansto.gov.au

ANSTO successfully operates one Small Angle Neutron Scattering instrument QUOKKA and in January of year 2016 commenced user operation of the second SANS instrument, BILBY. The Bilby is the utilizing both, Time-of-Flight (ToF) and monochromatic capabilities.

The design (in particular, set-up of four choppers which uses idea of that for D33 instrument [2] at ILL) opens possibility to vary wavelength resolution in the wide range (from 4% to 30%). Two arrays of position sensitive detectors in combination with utilizing of wide wavelength range (from ~3Å to ~18Å) provide capability to collect scattering data of wide angular range without changing experimental set-up (the most common settings used by now allow simultaneous data collection in the range between 1·10⁻³ Å⁻¹ and 1.8Å⁻¹). Offered instrument design opens possibility to collect scattering from a wide range of samples, with a unique capability to record fast kinetics data.

The presentation will be focused on two aspects. At first, some specific features in utilizing ToF mode comparing to monochromatic set-up for softmatter samples will be presented. At second, the most interesting of the scientific results obtained on Bilby withing last years will be shown.

Topic:

Neutron Instruments & Techniques

Poster Session / 97**Protein-polysaccharide complex coacervates as carriers of bioactive molecules**Sunandita Ghosh¹ ; Duncan McGillivray² ; Laurence D Melton^{None} ; Chris Seal^{None} ; Ivanhoe Leung^{None} ; Aiqian Ye^{None}¹ *School of Chemical Sciences, University of Auckland, Auckland*² *School of Chemical Sciences, The University of Auckland, Auckland 1010, New Zealand*

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Protein-polysaccharide complexes have been used in various food applications such as fat replacers, meat analogues [1], encapsulation of bioactive ingredients [2], and for enhancing foam and gel stability [3]. Studying these complexes at the microstructural level is important as a way to understand the origin of their functional and physicochemical characteristics [4]. β -Lactoglobulin (Blg) and pectin has been selected as model protein and polysaccharide respectively. Blg has been chosen as it is a major component of whey protein, its molecular properties are well-known and due to its ability to bind to various ligands. Pectin is selected as it is widely used as a functional ingredient in food products [5]. The microstructure and interactions of the complex coacervates formed by the Blg and pectin interactions has been previously established by isothermal titration calorimetry (ITC), circular dichroism (CD), intrinsic fluorescence spectroscopy, small angle X-ray scattering (SAXS) and small angle neutron scattering (SANS) [4,6]. In this study, curcumin is used to study the entrapment in these coacervate systems. The binding constant of curcumin to Blg has been determined by intrinsic fluorescence spectroscopy and confirmed by induced circular dichroism. SAXS and SANS methods will be employed to investigate these complexes formed at the microstructural level. The understanding of the structural data and interactions can then be related to the active release of the compounds and ultimately aim at seeing if these complex coacervates can be effectively used as carriers for bioactive molecules for targeted delivery in the human gut.

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Topic:**Poster Session / 98****Interaction of Antimicrobial peptides and cell membrane**

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Antimicrobial peptides (AMPs) have been widely investigated because of their potential solution to the encroaching 'antibiotic crisis' but information about mechanism of action of the peptides is still scarce [1]. Bacterial cell membranes will always be the initial point of contact for AMPs due to electrostatic interactions between the anionic molecules on the bacterial cell surface and the cationic residues within the AMPs [2]. The interaction can be investigated using model system such as liposomes. Liposomes are made from glycerophospholipids, with a similar ratio to that found in real membranes, through sonication or extrusion. Both techniques can produce liposomes but at different sizes.

Small angle X-Ray and Neutron scattering techniques (SAXS and SANS) can be used to investigate the effect of AMPs on the thickness, curvature of the membrane and also identifying the location of the peptide between the membranes [3,4]. However, the weakness of the model system is the high ratio of peptide to vesicles that is required to rupture the membrane, which may not represent the actual system [5]. In the future, a comparison between model and real system will be made in scattering measurement to assess this problem.

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Topic:

Poster Session / 102

Effect of Glycerol on the Structure of an Intrinsically Disordered Protein

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Intrinsically disordered proteins (IDPs) are defined by their lack of secondary structure in dilute aqueous solution. One of the best characterized plant IDPs is Cold Regulated (COR) 15A from the model plant *Arabidopsis thaliana*. This COR15A protein is highly hydrophilic and predominantly unstructured in solution, but fold into mainly amphipathic α -helices during drying or in the presence of high concentrations of low-molecular-mass crowding agents such as sucrose or glycerol. Due to its high conformational flexibility, no crystal structures are available for COR15A and there are also no reported NMR spectra. We investigated structural information of the COR15A protein based on the presence of a crowding agent (glycerol in this case) in order to induce the folding of the protein. The structural properties of the protein COR15A was studied by small angle neutron and X-ray scattering, wide angle scattering, circular dichroism (CD) spectroscopy and dynamic light scattering (DLS). The obtained results from these measurements showed that the structure of the protein COR15A has transferred into a more compacted structure upon increasing the concentration of glycerol (0, 20, 40, 60, 80%v/v). At the high Q-range there is a significant change in the value of the power law scaling coefficient from 1.7 to 3 when the protein COR15A dissolved in a solvent contains 80%(v/v) glycerol comparing with in a pure buffer solution. Furthermore, both small angle scattering and DLS measurements suggested that an oligomer formed during this folding process which can be described as a molecular complex comprised of a few monomers.

Topic:

Poster Session / 103

Completion of polarised neutron equipment deployment and development of methodology and data reduction in using polarised ^3He on ANSTO instruments

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We report the completion of the deployment project to incorporate neutron polarisation analysis capabilities on 6 ANSTO instruments. The instruments include high-intensity diffractometer WOM-BAT, SANS QUOKKA, thermal triple-axis spectrometer TAIPAN, cold triple-axis spectrometer SIKA, cold neutron chopper spectrometer PELICAN, and reflectometer PLATYPUS (for off-specular scattering). We can now provide user research with neutron polarisation analysis option to study materials in length scales from Ångström to micrometers and in energy transfer from 1meV to 50meV.

The use of polarised ^3He comes with its unique challenges. The trade-off between neutron polarisation and neutron transmission needs to be taken into account when planning an experiment. Regular monitoring measurements and proper data reduction must be applied to account for the neutron spin filter efficiency change over time. We have implemented a reliable measurement methodology and data reduction procedure to correct the data.

Some user experiments have already been carried out on instruments that acquired the capabilities early in the deployment. We will use some of the measurements to illustrate both our measurement capabilities and the reliability of our methodology and data reduction procedures.

Topic:

Neutron Instruments & Techniques