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Neutron scattering studies of biochar for catalysis applications

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A combination of infrared, Raman and inelastic neutron scattering (INS) was carried out to achieve a complete vibrational assignment of biochar (BCR) obtained from the pyrolysis of palm oil empty fruit bunch. The CH–OH ratio in BCR is ~5, showing that the hydroxyl functional groups are a minority species. There was no evidence for any aromatic C–H stretch modes in the infrared, but they are clearly seen in the INS and are the majority species, with a ratio of sp3–CH:sp2–CH of 1:1.3. The hydrogen bound to sp2–C is largely present as isolated C–H bonds, rather than adjacent C–H bonds. The Raman spectrum shows the characteristic G band (ideal graphitic lattice) and three D bands (disordered graphitic lattice, amorphous carbon, and defective graphitic lattice) of sp2 car- bons. Adsorbed water in BCR is present as disordered layers on the surface rather than trapped in voids in the material and could be removed easily by drying prior to catalysis. Catalytic testing demonstrated that BCR was able to catalyse the hydrodeoxygenation of guaiacol – a lignin model compound, yielding phenol and cresols as the major products. Phenol was produced both from the direct demethoxylation of guaiacol, as well as through the demethylation pathway via the formation of catechol as the intermediate followed by deoxygenation.

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In situ SANS analysis to investigate the electric field alignment of Silver-based Solvate Ionic Liquid (SIL) and Silver Nanoparticles (AgNPs) in nanocomposites

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The structural design of composite matrices is necessary to optimize electrical, mechanical, and thermal properties for smart applications in various industries, including aerospace, defence, and MedTech. The induced electrical or magnetic field alignment of anisotropic fillers (e.g., Ag, Graphene) or SIL has drawn attention since they can boost composites' performance. For example, the enhance- ment in bulk electrical conductivity of aligned Ag nanoparticles (AgNPs) in a composite matrix can

be five orders of magnitude, converting the sample conductivity from polymer-dominated to silver- dominated.

Our work presents a comprehensive study of the impact of alignment changes of Ag (I) and AgNPs in various composite matrices under the externally applied electric field and assess the alignment's sta- bility after the electric field is permanently removed. We studied the effect of directional alignment on creating the high crosslinked density regions in composite matrices. Furthermore, We evalu- ated the contributions of the high crosslinked density segments and the hierarchical structure of the aligned complexes and composite matrices' properties and performance.

In addition to the utilization of the Infrared macrospectroscopy beamline to evaluate the directional alignment using the four-angle approach, we used SANS to study the kinetic effects under external impulse influence since SANS data helped identify the clusters of Ag under an external electric field in various composite matrices. We also applied computational modeling based on quantum mechanics to validate investigation outcomes and reveal the uniqueness of the composite structure responsible for the superior properties.

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The cutting edge of archaeological inquiry: Antique swords and neutron techniques

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Until recently, little has been known about swords from the historical Kingdom of Dahomey in the Bight of Benin in West Africa, an area that was otherwise known as the Slave Coast because of its unique role in the trans-Atlantic slave trade. The kingdom, which existed from about the mid-17th century to 1894, when it was conquered and subjugated by France, was one of the most intriguing and unique centralized states of pre-colonial West Africa – not least because of the all- female warrior corps that garnered it rapt worldwide attention. In what has been a world first, with a grant from ANSTO six Dahomean swords were examined using neutron techniques that were able to delve beneath the surface of the metal, uncovering insights into their manufacture that helps answer hitherto open questions on whether these swords were made with imported European iron, or not. While it is known that iron from Northern Europe was imported in significant amounts into the Gulf of Guinea throughout the period, the relative importance of those imports has been a mystery. In fact, this study suggests that European imported iron has been overvalued in the perceptions and assessments of European observers for the last 150 years, and that local African smelted iron was of far greater importance to local weapons manufacture. In addition, the research uncovered hitherto unknown data on some of the unique construction techniques employed in the manufacture of these rare and hitherto understudied historical items.

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The Effect of Size Reduction Induced by Mechanical Milling on Rare Earth Manganites

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The perovskite manganites with chemical formula $AMnO_3$ are known for strong coupling of spin, electronic, and structural degrees of freedom. These couplings manifest as extreme sensitivity in the electronic and magnetic properties with variations in structure and temperature; the colossal magnetoresistance effect being the best-known example. In particular, the phase diagram of the $RE_{1-x}(Ca_{1-y}Sr_y)_xMnO_3$ (RE = trivalent rare-earth) system is diverse, containing insulating and metal- lic phases with orbital and magnetic ordering as hole doping (x), and the structural tolerance factor,
(t) varies.

This study focuses on how mechanical milling affects the magnetic and structural properties of the manganite series $Nd_{1-x}(Ca_{1-y}Sr_y)_xMnO_3$ (NCSMO, nominally: $x = 0.4$, $0 \leq y \leq 0.3$), as it is near to a magnetic phase boundary, around which an antiferromagnetic insulating state with charge and orbital ordering , and two ferromagnetic states, one metallic and one insulating with orbital ordering occur.

The magnetic and structural properties of bulk NCSMO powder have been compared against me- chanically milled NCSMO powder using X-ray diffraction, PPMS, and neutron powder diffraction (WOMBAT and ECHIDNA) techniques. It has been observed that reducing the size of the crystallites via mechanical milling changes the magnetic properties of the manganite materials, even though no noticeable structural changes occur. This work presents possible mechanisms which can lead to the observed deviation to the magnetic structure.

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In-Situ Structural Analysis of the Oxygenation of YBaCo4O7+

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In this work, the oxygenation of $YBaCo_4O_{7+\delta}$ is further explored through neutron diffraction studies. The 114-compound material of $YBaCo_4O_7$ is a mixed cobalt valence system that exists in a hexagonal arrangement (space group $P\ 6_3\ mc$). Mixed $Co^{2+/3+}$ valance allows the structure to accommodate additional oxygen through a structural transition to an orthorhombic arrangement. Neutron powder diffraction patterns revealed the evolution of the oxygenated $YBaCo_4O_{7+\delta}$, $\delta = 1$ phase when exposed to oxygen at 320 °C. This is paired with a decrease in the $YBaCo_4O_{7+\delta}$, $\delta = 0$ phase due to the one formula equivalent of Co^{2+} fully oxidising to Co^{3+}. It results in a bulk mixture containing both $\delta = 0$ and 1 phases. After 1 hour of exposure to oxygen at 320 °C, more than 97 % of the phase mixture is now $\delta = 1$. The diffraction results here show that thermogravimetric studies, while convenient, do not give the complete picture of what is happening. And understanding what exact phases of $YBaCo_4O_{7+\delta}$ are present can help with potential oxygen storage applications.

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Advanced Diffraction and Scattering beamlines (ADS-1 and ADS- 2): The closest thing to a neutron beam at the Australian Syn- chrotron?

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One of the advantages of neutron radiation over X-ray radiation for diffraction, spectroscopy and imaging experiments is its superior penetration in bulky and/or high-Z samples (and sample en- vironments). By contrast, materials characterisation experiments using typical hard X-ray beams (~8–30 keV, λ = 0.4–1.5 Å) in transmission mode are usually limited to samples ≤1 mm thick, even with very high-flux synchrotron radiation sources.

The Australian Synchrotron is currently building two Advanced Diffraction and Scattering (ADS) beamlines that will offer high-energy X-rays (45–150 keV, λ = 0.08–0.27 Å) for a wide range of diffraction, imaging and tomography experiments. These X-ray beams will penetrate up to 1 cm of aluminium or steel with 70% or 25% transmission, respectively, paving the way for more “neutron- like” experimental setups, such as strain mapping in large objects and *in situ* sample environments with metallic windows.

ADS-1 and ADS-2 are designed to be highly versatile beamlines, offering flexible instrument con- figurations to support diverse structural and microstructural characterisation experiments in mate- rials science, engineering and mineralogy. The two endstations will feature multi-axis positioning systems for polycrystalline, single-crystal and monolithic samples, along with in-built gas delivery facilities and space for user-supplied equipment.

I will present an overview of the planned capabilities of the ADS beamlines and the expected timeline for their implementation, with emphasis on the complementarity of ADS with instruments such as WOMBAT, KOWARI, KOALA and DINGO.

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Disorder and ionic conductivity in Ba3Nb(M)O8.5

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Understanding atomic-scale structure and local defect distributions in functional materials, such as ionic conductors, is essential for rationalising their behaviour and tuning them for better per- formance. We have grown the first single crystals of the oxide-ionic conductors Ba3NbMoO8.5 and Ba3NbWO8.5 in order to investigate the relationship between the highly disordered average Ba3Nb(M)O8.5 structure and the arrangements of ions on the local scale. Our neutron scattering experiments on single-crystal and polycrystalline samples yield several important insights into the local cation and anion arrangements, which are deduced after taking into consideration both chem- ical and geometric constraints. Our data also inform the interpretation of calculated bond valence energy landscape (BVEL) maps, which point to a high degree of oxide ion disorder along two- or three-dimensional conduction pathways. These findings highlight the need for a more flexible de- scription of oxide ion distributions in the Ba3Nb(M)O8.5 structure than is conventionally used in the literature.

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Top-down patterning of topological edge states using ion beam and characterization with neutron reflectometry

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Amorphous states in topological insulators have recently emerged as a point of intense discussion, and whether they are topological or trivial is currently of great interest. This question is crucial as it determines whether they behave as a “topological vacuum” to enable surface lithographic patterning via spatial control over $\mathbb{Z}2$. Recent theories predict that the collapse of the topological states in the glass is not guaranteed. Instead, solid-state amorphous topological insulators do exist in exceptional cases, at least in numerical simulations. Therefore, it is crucial to understand how amorphization modifies the strong TIs in the crystalline chalcogenide family (Bi2Se3, Sb2Te3 and Bi2Te3). Here it is experimentally shown that targeted modification with an ion beam at the surface of Sb2Te3 locally switches the strong 3D crystalline topological insulator into an insulating amorphous state with negligible bulk and surface conductivity. This is attributed to a transition from $\mathbb{Z}2=1 \rightarrow \mathbb{Z}2=0$ at a critical disorder strength that drives a topological transition, as supported by density functional the- ory from ab initio molecular dynamics and model Hamiltonian calculations. This allows for the pat- terning of lateral heterostructures, and remarkably, the boundary of the amorphous and crystalline region gives rise to additional edge state conductivity that can be detected at room temperature. The ability to drive the topological transition and modulate the surface conductivity in real space with a phase-change material is useful: it allows for inverse lithography to write topological surfaces, edges, and corners which are the future building blocks of scalable topological electronics.

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In-house design and domestic fabrication of cryofurnace and in- duction furnace

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As part of the Australian Government's research infrastructure and investment plan (RIIP), ACNS has acquired several new bespoke sample environments for high and low temperature experiments. We have designed from scratch a 4K to 800K cryofurnace and a 300K to 1273K induction furnace, which are currently being commissioned.
By designing these systems from the ground up we are able to tailor them to our specific operational needs and gain the ability to freely source spare parts and make future modifications.

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DINGO, the neutron imaging instrument at ANSTO

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Since receiving first neutrons in October 2014, the thermal-neutron radiography/tomography/imaging instrument DINGO [1] has achieved a high subscription rate from a broad national and international research community. The high demand for DINGO is based upon the different insights achieved when looking into objects because of the different contrast achieved with neutrons compared to X- rays, the high sensitivity of neutrons to light elements, and the dedicated expertise of the instrument staff.

DINGO has shown real value in industrial applications, particularly in the structural analysis of cracks and defects in concrete, metals and complex composite materials. Originally designed to achieve a true spatial resolution of 54–200 um for samples up to 35 cm in height, a combination of in- strument upgrades now enables ANSTO to offer a globally unique, parallelised and high-throughput scanning capability for drillcores and samples up to 1m in length, improved signal-to-noise and spatial resolution, a larger field-of-view and the flexibility to incorporate sample environments for kinetic and real-world studies.

With a strong and research-active support team, DINGO is the global go-to facility for the appli- cation of thermal-neutron imaging in palaeontology, cultural heritage, mining, energy storage and commercial applications, exemplified through high-impact research outputs, major museum exhibi- tions, and reports in the media.

This poster presents an overview of the DINGO instrument and its technical capabilities, highlights a selection of research achievements from the collaborative, merit-based user access program. Come, meet our team and give neutron tomography a try!

[1] Garbe, U; Randall, T; Hughes, C; Davidson, G; Pangelis, S and Kennedy, SJ, A New Neutron Ra- diography / Tomography / Imaging Station DINGO at OPAL, Physics Procedia 69, 27 (2015).

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The Invisible Revealed: Cultural Forensics with Neutron Beam Science

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The Powerhouse Museum in Sydney houses more than half a million objects of cultural significance, spanning 3000+ years of human technological advancement. “The Invisible Revealed” exhibition (Nov 2021 - Sep 2022) evolved through a partnership between the Museum, ANSTO and the Ex- panded Perception and Interaction Centre at UNSW. It is the first major museum research exhibi- tion for ANSTO, and internationally, the first to showcase neutron imaging. It presents 26 of the Museum’s objects alongside digital 3D models produced using innovative neutron- and synchrotron- X-ray tomography, photogrammetry, Digital Twin technology, Artificial Intelligence, and other in- sights achieved with nuclear methods.

This talk provides an overview of neutron imaging and complementary methods applied in this national-award winning exhibit, and demonstrates the hidden mechanisms, repairs, degradation and insights gleaned in miniature spy cameras, a T’ang Dynasty statue, early coinage and more.

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Spin Ice Behaviour in Spin Crossover Materials

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Metal-Organic Frameworks (MOFs) are a class of compounds that have received increased interest over the last few decades due to the multitude of properties they exhibit. This work primarily fo- cussed on the suspected spin ice system, Fe(Ag(CN)2)(dps)2.2EtOH, (dps = 4,4'-dipyridylselenide). In this structure elastically coupled Fe(II) ions are mapped on a triangular Kagome-like array, lead- ing to a deviation from the conventional low spin-high spin (LS-HS) structure seen in mixed spin- state (multi-step) spin crossover systems. Here, competing long-range ferroeleastic interactions (governed by the volume change associated with HS-LS or LS-HS transitions) and opposing anti- ferroeleastic interactions (arising from the geometric restraints of the Kagome lattice) inhibit the formation of an ordered spin state. In pursuit of possible emergent spin ice behaviour, temperature and magnetic field dependent neutron scattering experiments were performed with the aim of iden- tifying pinch points in magnetic diffuse scattering. Coupled with this, computational modelling of the powder averaged magnetic diffuse scattering is used to aid in locating these features in experi- mental data. Further investigation into the complex nature of these kagome structures may lead to the direct experimental observation of a spin ice in a 3-dimensional framework system.

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Deuteration of cholesterol for neutron applications

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Cholesterol is an essential component in animal cell membranes and a feature in lipid nanoparti- cle technologies. The National Deuteration Facility (NDF) at the Australian Nuclear Science and Technology Organisation (ANSTO) has recently experienced increased interest in access to deuter- ated cholesterol for neutron studies of biological systems, and lipid nanoparticles as drug and gene delivery vehicles.

The NDF has a reliable and robust method to produce deuterated cholesterol from bioengineered yeast in high purified yield. Different levels of deuteration are also possible through adjustment of growth media, and so can be tuned according to the application.

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Understanding and controlling the formation of photonic crys- tals from polydisperse colloidal systems

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The fundamentals of crystallisation and glass formation are not yet fully understood. Colloidal sus- pensions have been shown to be promising model systems for understanding these processes. As colloidal motion is Brownian, rather than ballistic, kinetics and dynamics can be studied in real-time. It is well documented that colloidal suspensions can "successfully crystallise" when the particles in the system have sufficiently low polydispersity.[1,2] If a system is highly polydisperse, this will hinder the solidification process.

Nanodiamonds are a topic of interest in many material studies due to their variety, and unique mechanical and optical properties.[3,4] Detonation nanodiamonds (DNDs) are of particular interest due to their unique fabrication process. Due the detonation synthesis method, the particles are small (several nm) and faceted, but in solution self-assemble into highly irregular fractal shapes.[5] Despite this high polydispersity, when centrifuged, these types of DNDs can produce iridescent colours which are reminiscent of photonic crystals – this is highly surprising given the highly irregular structures of these materials. This phenomenon was first discovered by Grichko et al.,[5] however, the mechanism behind this process is still unknown. With a combination of lab techniques and experiments at the Australian Synchrotron and the Australian Centre for Neutron Scattering, we will systematically investigate these nanodiamond photonic crystals, and examine their structure and formation kinetics. We recently conducted experiments for SANS and USANS at ANSTO and investigated the structure of nanodiamonds to better characterise the structure of the nanoparticles, in particular the particle surface (non-diamond carbon plus functionalised layer). The DND particles were probed at three different contrasts to define the structures of the individual particles as well as their conglomerates.

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Deuterium labelling at the National Deuteration Facility: Updates on Demand and Supply

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The use of deuterated molecules in neutron scattering experiments for investigating the relation- ship between molecular structure and function expands the range and complexity of science that can be conducted at reactor and spallation-source neutron facilities. There have been increasing global demand and initiatives in the field of molecular deuteration in the recent years; however, the complexity of the required molecules for new experiments has also increased. This has limited the experiments that can be performed and formed a bottle-neck for advancing the applications of neu- tron scattering.

In this paper we will discuss the recent advancements and the impact of deuteration on the research outcomes achieved by using bespoke deuterated molecules produced by the National Deuteration Facility. Recent high-impact case studies will be presented which reveal the exciting and diverse characterisation studies which are now available for the neutron research community. We will de- scribe the synthesis and application of new deuterated molecules which are unavailable from com- mercial sources, such as deuterated cholesterol, proteins, phospholipids with deuterated oleic and linoleic chains, detergents for membrane proteins, and deuterated ionic liquids and metal-organic frameworks (MOFs) precursors. Such molecules were used to investigate complex nanoscale sys- tems in the fields of structural biology, biotechnology and molecular electronics and storage using neutron scattering techniques.

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Using a computer game engine to teach neutron reflectometry alignment

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Neutron reflectometry is a technique that probes the structure of thin films with nanoscale thick- ness. To produce an accurate reflectometry measurement, the sample must first be carefully aligned. This requires positioning the sample within 10 micrometres of its optimum position in the beam and ensuring the sample angle is aligned with a precision of at least 0.01 degrees. In practice, this is achieved using motorised translation and tilt stages, controlled using an iterative procedure of position and angle scans. Many users find the initial alignment process challenging.

In this poster, I will discuss a browser/Android app that has been developed to help teach users the process of alignment. The goal of this app is to build user confidence ahead of neutron reflectometry experiments. The app also covers some other useful skills including using the Swiss Instrument

Command System (SICS) and the procedure for mounting/dismounting samples including the basic radiation surveying step. The app covers the alignment protocol for both the Spatz and the Platypus reflectometers available at the ACNS.

I will discuss two different free game engines that can be used for rapidly developing this type of simulation-game, and show how the source code can be adapted to demonstrate different virtual neutron/X-ray instruments and techniques.

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Solution structure characterisation of the complex of ubiquitin and its conjugating enzyme E2 by deuteration and small angle neutron scattering.

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This structural study exploits the possibility to use modular protein deuteration to facilitate the study of ubiquitin signalling, transfer, and modification. A protein conjugation reaction is used to combine the protonated E2 enzyme with deuterated ubiquitin for small angle neutron scattering with neutron contrast variation. The combined biomolecules stay as a monodisperse system during data collection in both protonated and deuterated buffers indicating long stability of the E2~Ub conjugate. With multiphase *ab initio* shape restoration and rigid body modelling we reconstructed the shape of a E2~Ub conjugated complex. Solution x-ray and neutron scattering data for this E2~Ub conjugate in the absence of E3 jointly indicate an ensemble of open and backbent states in solution, with a preference for the latter in solution. The approach of combining protonated and labelled proteins can be used for solution studies to assess localization and movement of ubiquitin and could be widely applied to modular ubiquitinated systems in general.

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KOALA decommissioned October 2022 what has been achieved? and what remains to be done KOALA2 implementation under- way

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KOALA entered the Bragg Institute (now ACNS) user program in 2009 with the first chemical crystal- lography publication appearing in 2010. Since then 82 papers have appeared (56 chemical-crystallography).
This is the first time single-crystal neutron diffractometer output has been dominated by publications in chemical-crystallography. By the time all KOALA output appears this balance may shift even fur- ther. In the context of chemical crystallography, the major achievement with KOALA has been that the single-crystal neutron diffraction experiments and results have been obtained in time for them to be included in the high profile and high impact publications. A literature survey 2009 revealed that the earlier instruments had only rarely produced structures which were reported with the im- portant chemistry for which the determinations were made, appearing years later in stand-alone crystallographic papers which were disconnected from the high-profile work and rarely cited. The stated aspirational aim of the Bragg Institute was to match the world leading neutron facilities for impact of publication and an initial target to achieve an average JIF for papers of 3. With the outstanding proposals that our community has made and our significant achievements in data re- duction and analysis KOALA became sufficiently successful that it will now be the first of the initial Bragg Institute instruments to be renewed.

KOALA was the only instrument purchased entire from a “supplier” and the discipline of standard- ised motion control, electronics etc was not applied. This has rendered the original instrument ob- solete and the achievements of our community have been recognised with the building of KOALA2 which will be introduced in this presentation.

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Strengthening Oxygen Lattice to Achieve Better-Performance Cobalt- free Li-Rich Layered Oxide Cathodes

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Li-rich layered oxides are one of the most promising cathode candidates for the next-generation high-energy-density lithium-ion batteries. However, their real-world application is hindered by dramatic capacity fading and voltage decay on cycling. This work comprehensively studies the mechanistic behaviour of cobalt-free Li1.2Ni0.2Mn0.6O2 and demonstrates the positive impact of Ru introduction for suppressing voltage decay and improving cycling performance. A mechanistic transition from the monoclinic to the hexagonal behaviour is found for the structural evolution of Li1.2Ni0.2Mn0.6O2, via in operando synchrotron-based X-ray powder diffraction. The enhancement mechanism of Ru doping is completely understood using Neutron-based pair distribution function analysis, neutron powder diffraction, synchrotron-based near-edge X-ray absorption fine structure and X-ray absorption characterizations. The Ru doping improves the structural reversibility in the first cycle and effectively restrains structural degradation during cycling by stabilizing oxygen redox and reducing Mn reduction, thus enabling high structural stability, an extraordinarily stable voltage (decay rate < 0.45 mV per cycle), and a high capacity-retention rate during long-term cycling. The fundamental understanding of the structure-function relationship of Li1.2Ni0.2Mn0.6O2 sheds light on the selective doping strategy and rational materials design for better-performance Li-rich layered oxides, which is extendable to other types of electrodes for use in metal-ion batteries.

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Diffuse Scattering from a Martensitic Fe-Pd Alloy

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Fe-30at%Pd exhibits two martensitic transformations on being cooled from room temperature to 100 K, attributed to phonon softening. A preliminary study of a large single crystal of this composition on the Koala neutron Laue diffractometer at the OPAL reactor, not only showed evidence for these transformations but also revealed interesting and unexpected satellite reflections around certain Bragg reflections.

In a subsequent experiment at the Sika triple-axis spectrometer these satellite reflections were shown to involve elastic scattering to within 1 meV and the propagation vector around the (200) reflection of 0.180, [g0.186, [g0.034. During a parallel experiment to the Sika one, an ideally small piece of the crystal, electro-discharge machined from the large one, was studied on Koala but the interesting satellites found for the large crystal were not present. As a result, in a further experiment on the large crystal at Koala, completed in early 2021, diffraction patterns were collected with the aim of surveying the whole of the large crystal, particularly in the vicinity of the edge from which the ideally small crystal piece had been extracted. Each of these latter diffraction patterns showed the satellite reflections, an observation which raises the question as to why electro-discharge machining has affected the microstructural state of the large crystal.

It is now proposed to undertake further research at the Taipan triple-axis spectrometer using polar- ization analysis for one experiment and an applied magnetic field for the second, with the aim of solving the microstructural origin of the satellite reflections.

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Thermodynamics and local environment in liquid gallium under static magnetic fields

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Gallium is a fascinating element that melts as liquid metal at around room temperature (Tm = 302.9 K) and keeps the liquid state till supercooled to 270 K, which endows it with complex solid and liquid characteristics. Its extraordinary physicochemical properties have resulted in gallium-based materials being applied in many fields and have attracted significant research attention. Unlike the diamagnetic in its solid state, the liquid gallium shows a slight paramagnetic attribute. Our research aims to reveal the effects of static magnetic fields (SMF) on liquid gallium on atomic scales. The study will provide the principle support for future liquid gallium-based materials applications under mag- netic fields. We performed time-of-fight neutron scattering spectroscopy (Pelican, ANSTO), utilising a superconducting magnet, to study the atomic diffusion behaviours and local order profiles of liquid gallium (320 K) under SMF (0 – 4 T). The inelastic neutron scattering results are cross-validated and consistent with our previous high-energy X-ray diffraction experiments (under 0.2 T SMF). Those results of experiments and simulations indicate that SMF will influence atomic diffusion and drive short-range orders to form lower coordination numbers in local clusters.

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ACNS – Scientific Computing Services

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The Scientific Computing team supports researchers by performing numerical simulations that com- plement experimental research at the Australian Centre for Neutron Scattering. In particular, we use state-of-the-art software to perform computational quantum mechanical modelling, molecular dynamics simulations, lattice dynamics calculation, data analysis and visualisations.

In this poster, I will present the Scientific Computing Support service for the ACNS experiments. In particular, I will describe the techniques and give an overview of the software and hardware available for our researchers and users.

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Status, statistics, and recent research highlights from Echidna

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The Echidna high-resolution powder diffractometer remains a reliable and productive ACNS instru- ment contributing annually to about 50 published studies done on a wide range of topics, from magnetic, energy and planetary materials to cultural heritage and additive manufacturing. We will discuss how Echidna has recovered from COVID-19 impacts, latest and planned developments, user programme statistics, and recent research highlights.

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Interrogation of the mechanism of Li-mediated ammonia electrosynthesis by in situ neutron reflectometry

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To truly harness renewable energy, it is imperative we solve the issue of intermittent availability. Excess renewable energy can be stored as carbon free chemical fuels such as H2 and NH3. These energy dense battery alternatives offer the ability to transport renewable energy off site, without reliance on an electrical grid. Currently NH3 is primarily produced for implementation as a fer- tiliser via the anthropogenically expensive Haber Bosch process. To realise its use as a chemical fuel a sustainable production method is required, such as electrochemical reduction of N2 into NH3. Currently, the only electrochemical process able to achieve this is the lithium mediated nitrogen reduction reaction(Li-NRR).1 At Monash, we have developed Li-NRR methods capable of achieving a world leading 100% faradaic efficiency. 2,3 We have now turned our sights toward decreasing the energetic demands and increasing the overall sustainability of the process. This requires intelligent catalyst and electrolyte design, both of which can be facilitated by advancing our knowledge of the solid electrolyte interface (SEI). Blair et al. utilised in situ neutron reflectometry (NR) in an attempt to prove the state of the electrode during the Li-NRR but failed to obtain meaningful results as this work produced no ammonia.4 We are in the process of using NR, combined with our insights from the development of highly efficient Li-NRR to describe the mechanism occurring at the electrode interface, and define the SEI environment responsible for high faradaic efficiency.

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Reflecting Neutrons with Platypus

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PLATYPUS is the initial neutron reflectometer at the Australian Centre for Neutron Scattering with a capability to study surfaces and interface systems with applications from biomolecules, soft matter through to magnetic thin films [1-3]. There have been a number of significant improvements to both the instrument and data reduction and treatment software [4] over the last two years. On the hardware front the original detector has been replaced [5] enabling higher count rate capabilities, greater detection efficiency at shorter wavelengths and significantly lower background. The slits which define the neutron beam have been replaced with upgraded positioning mechanisms enabling greater flexibility in experimental setup. Most recently new slit blades have been installed resulting in a substantial reduction in instrument background. These changes have significantly enhanced the instrument performance with improved reproducibility.

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Revealing composition and conformation of liposomes using in- tegrated methods of HPLC/AF4, UV-Vis absorption, Refractive In- dex, MALLS, DLS, and SAXS-WAXS

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Liposomes are increasingly better developed as efficient drug carriers. Structural characterization of the functional liposomes with and without drug-uptake and the consequent drug-transport and conditional drug-delivery, is of interest in biomedical applications. Here, we report an established combined methods using high-performance liquid chromatography (HPLC), asymmetric flow field- flow fractionation (AF4), UV-Vis absorption, refractive index (RI), multi-angle laser light scattering (MALLS), dynamic light scattering (DLS), and small- and wide-angle X-ray scattering for structural characterization of liposome solutions. We demonstrate an example of using the integrated system to successfully determine hydrodynamic radius and its distribution, molecular mass, lipid aggregation number, of a model liposome system of unilamellar vesicles (ULV). The radius of gyration Rg of 50 nm, hydrodynamic radius Rh of 50 nm, bilayer thickness, and lipid aggregation number of the ULV, with and without drug molecule loading, are determined using simultaneous small- and wide- angle X-ray scattering, incorporated with HPLC/UV-vis/RI, at the high-flux 13A BioSAXS undulator beamline of the 3.0 GeV Taiwan Photon Source. Moreover, a chain-chain packing peak of the ULV bilayers could be observed at q = 1.488Å-1 (d = 4.28 Å); reversible changes of the peak position and peak broadening with temperatures between 20 – 50C, providing a direct evidence of the correlation

of the lipid chain-chain packing with the commonly observed gel-ripple-fluid phase transitions of multilamellar vesicles.

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Reflecting Neutrons with Spatz

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Spatz is the newest reflectometer at the Neutron Beam Facility of the OPAL Research Reactor. It has been in the ACNS User Program since November 2020 and has completed a number of experiments. Spatz operates using the time-of-flight principle and has a vertical sample geometry (horizontal scat- tering geometry). A wide variety of sample environments can be used on the instrument including solid-liquid cells and atmospheric chambers and heating stages. This presentation highlights the recent advances with Spatz including instrument developments and scientific highlights.

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Thermal Treatment on Vertical Morphology of PffBT4T-2OD: ITIC Non-fullerene Bulk Heterojunction Thin Films

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Solution-processable organic solar cells are extensively attractive in academic and industrial fields with the advantages of roll-to-roll processing and flexible and lightweight large-area devices. Bulk- heterojunction (BHJ) is a device architecture comprising electron donors and acceptors in the mix- tures. The donor-acceptor mixed phases in the BHJ morphology aid in dissociating the photogen- erated excitons and transporting the charges to the respective electrodes through the individual phase segregation. Therefore, optimizing BHJ nanomorphology for efficient exciton dissociation and charge extraction highly determines the device performance. Neutron reflectivity is conducive to probing the vertical morphology in a blend film. In this work, PffBT4T-2OD and ITIC are used as donor and acceptor materials in a BHJ layer. The study aims to (1) profile the vertical distribution of donor and acceptor phases in the blend film. Reveal the effect of donor-acceptor composition and solvents on the morphology change of the BHJ layer. (2) understand the changes in vertical morphology through thermal annealing. The elevated temperature possibly initiated the preferred molecular interaction to generate a morphology change in the BHJ layer. These results will com- plement the other characterization techniques, such as AFM, GIWAXS, and molecular dynamics simulation, assisting in developing high-efficient non-fullerene organic solar cells.

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Residual stress in multilayer laser cladding depositions on light rail components

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A shift towards sustainable practices places an increasing demand on Australia's public transport networks. This results in growing rates of rail wear and fatigue due to high passenger loads, increas- ing operation speeds and service frequency. Light rail is particularly susceptible to these damage modes as components such as switches are manufactured from lower steel grades. As rail replace- ment procedures are both expensive and disruptive, new maintenance techniques are required to efficiently recondition light rail components.

Laser cladding is an additive manufacturing-based repair strategy which utilises a high energy laser to melt a metallic powder at the rail surface to form a metallurgically bonded coating. To rebuild worn rail profiles, multiple layers of cladding are required which exposes the underlying substrate to cycles of melting and solidification. This alters the microstructure with each successive layer, result- ing in internal stress which may increase the susceptibility to fatigue failure. Accurately measuring the residual stresses after multi-layer laser cladding is critical to ensure repairs are safe for railway operation.

To determine the stress generation after muti-layer depositions, double-layer laser cladding has been carried out on 600 mm sections of ex-service tram rail using Stellite 21. The triaxial residual stresses were measured on the Kowari strain scanner using through thickness line scans that traverse the cladding, heat affected zone and rail substrate. Comparison of these results with single depositions will provide information on the tempering effects of repeated cladding cycles and how this may affect the rail lifetime. These findings will accompany microstructural and mechanical property analysis to determine the optimal cladding parameters for light rail repair to be implemented by the railway industry.

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EMU cold-neutron backscattering spectrometer at ACNS, ANSTO

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EMU is the high-resolution neutron spectrometer installed at the OPAL reactor, ANSTO, which de- livers 1 μeV FWHM energy transfer resolution for an accessible ±31 μeV energy transfer range. The spectral resolution is achieved by neutron backscattering from Si (111) crystals on the primary and secondary flight paths, allowing up to 1.95 Å-1 momentum transfer range. The spectrometer is well for suited quasi-elastic and inelastic neutron scattering studies, notably in the field of soft-condensed mater including biophysics and polymer science, chemistry and materials science, and geosciences. Most experiments are carried out with standard cryo-furnaces (2 to 800 K temperature range). Spec- trometer beam-time access is merit-based, thus welcoming as well experiments in other materials research areas, and including experiments that may require e.g. other ancillary equipment such as existing controlled-gas delivery, and potentially pressure, applied field set-ups, etc. Examples of the spectrometer capabilities are shown through select case studies.

8

Experimental and numerical investigation of fracture in welded connections

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The steel structures are built from beams and columns that may fail in a steel frame because of local yielding, buckling or fracture, and our project focuses on one of the most important failure modes in the structural analysis, the fracture in welded connections. The rapid heating and uneven cool- ing of different parts during the welding process will cause significant residual stresses. And the high temperature also locally changes the microstructure of the base metal and leads to altering of the mechanical properties in the neighbouring areas of the weld and the heat-affected zone. These welding-related properties, such as tensile residual stresses and change in mechanical properties in and around welds, play a significant role in the behaviour of steel connections and need to be char- acterised and optimised to minimise their effects on welds fracture.

Triaxial residual stress generated by different types of welds are systematically investigated through the neutron diffraction approach using the strain scanner Kowari at ANSTO. The detailed 3D distri- bution of the residual stress in the welded cruciform specimens and end-plate connection provide a more comprehensive understanding the complex steel connections. Based on these measurements, the fracture behaviour of welded connections will be analysed, and the effect of welding-related properties will be discussed.

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Multiscale structure parameters affecting phase transitions in PbZrO3-based antiferroelectric ceramics

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Antiferroelectric (AFE) materials are considered as promising candidates for energy conversion and storage devices. The main origin of these potential applications arises from the distinctive structural transition induced by external stimuli such as electric-field (E-field), temperature and mechanical force. Thus, it becomes pressing to unveil the structure parameters affecting the critical fields and phase transitions. This study systematically investigates the structural and crystallographic texture change as a function of the E-field and temperature in the prototype PbZrO3-based AFE materials. It is found that in addition to the crystal structure parameters such as antiparallel atomic displace- ments and octahedral tilting, the microscopic crystallographic texture can also influence the critical E-field triggering the AFE-ferroelectric (FE) phase transition. The degree of the crystallographic tex- ture can be adjusted via the external E-field and temperatures, which directly modifies the energy barrier between the AFE and FE phases. It is found that the aligned state presents a lower critical E-field compared with that of the random state. Our results demonstrate that multiscale structure

parameters can be used to tune the properties of the AFE materials. Moreover, these parameters can be not only modified by the chemical compositions and synthesis conditions but also controlled by the history of the applied E-field and temperatures, which introduces a new strategy for improving the performances of AFE materials.

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A 3-D Reconstruction of neutron activation in samples irradiated at DINGO

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Non-destructively characterising the interior elemental composition of bulk samples (larger than a few hundred µm) is essential across many fields (e.g. archaeometry, palaeontology, mining, etc.) for obtaining a holistic picture of an object and its context. Traditional neutron activation analy- sis (NAA) techniques are utilised to identify elemental distributions within bulk samples, however, these techniques do not yield the 3-D spatial information needed to determine where in the object these elements originate. Alternatively, advanced imaging techniques such X-ray and neutron to- mography yield 3-D structural information, but the tomographic reconstructions cannot often be accurately extrapolated to yield internal elemental distributions.

To address this issue, a novel method that combines neutron scattering and medical imaging tech- niques has been proposed. Using ANSTO's thermal-neutron imaging instrument (DINGO), samples are irradiated with thermal neutrons, during which, a fraction of the nuclei within these samples will become activated via neutron capture. Similarly to NAA, as the resulting radionuclides de- cay, the characteristic energies of emitted gamma photons can be measured to uniquely identify the isotopes created. By performing single-photon emission computed tomography (SPECT) of the neutron-irradiated samples over an extended period, the spatial distribution of elements, via their radioisotopes, can be determined. To validate this method, 3-D reconstructions of Cu-64 in copper samples have been achieved, with reconstructions of other elemental samples pending. Given these results, post-neutron-tomography SPECT imaging has the potential to produce 3-D isotopic recon- structions of bulk objects which can be further combined with existing tomography data to create rich multidimensional datasets about a sample.

1

Recent Achievements in Neutron Backscattering

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Crystal spectrometers such as IN16B at the Institut Laue-Langevin in Grenoble, France, operating in backscattering provide high energy resolution down to sub-micro-eV. While the spectrometer IN16B has been serving its international user community for about a decade, we continuously strive to ex- tend the capabilities of these spectrometers well beyond today's limits. In the presentation, we focus on two recent major achievements.
A first upgrade of IN16B aimed at the installation of a ToF opportunity into the primary spectrometer necessitating a very fast chopper system. This initiated the fabrication of novel chopper disks made from oriented carbon fibers. The resulting new BATS option (Backscattering And ToF Spectrometer) offering an energy transfer range enhanced by more than one order of magnitude has been brought into routine user recently.
At present, we are installing a variable guide focusing element in the vicinity of the choppers which will enhance the neutron flux at the sample up to a factor of 6.
In addition, we have demonstrated the feasibility of a future ultra-high energy resolution backscatter- ing spectrometer by replacing the Si 111 reflection generally used today by the GaAs 200 reflection featuring a significantly smaller Darwin width. The established energy resolution is 76 neV, one order of magnitude superior than available in standard backscattering setups.

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Neutron Polarisation Capabilities at ACNS

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By controlling the spin of neutrons during scattering experiments, unique information about samples can be found that would not be available otherwise. Manipulating and filtering the neutron spin both

before and after its interaction with a sample allows not only more detail about magnetic structures to be found, but also enables coherent and incoherent scattering signals to be distinguished.

An overview of the neutron polarisation capabilities offered at ACNS will be summarised here, in- cluding details of the six neutron scattering instruments which are compatible with polarisation ex- periments, as well as some examples of the types of measurements which can be undertaken.

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How ionic liquid composition and structure affect micelle mor- phology

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A common method of making viscoelastic, wormlike micellar systems in water is to combine a cationic surfactant with a salt featuring a strongly bonding or hydrotropic counterion, such as sodium salicylate. Despite their inherent high ionic strength and evidence for cylindrical or worm- like micelles, viscoelastic micellar systems have yet to be reliably observed in ionic liquid solvents.

In this work, micellar structures of a series of alkyltrimethylammonium and alkylpyridinium sali- cylate surfactants were examined by small-angle neutron scattering (SANS) in the protic ionic liq- uids ethylammonium nitrate (EAN), ethanolammonium nitrate (EtAN), ethanolammonium formate (EtAF), their mixtures, and aqueous solutions.

Results reveal the conditions under which wormlike micelles form and elucidates how solvent struc- tural features such as ionic strength, H-bond connectivity and the inherent liquid amphiphilic nanos- tructure affect micelle morphology. In turn, learning about these subtleties provides a greater under- standing of how amphiphilic self-assembly occurs in neoteric solvents, allowing for more directed design of such systems for a variety of applications.

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Understanding nanoplastic toxicology using neutron scattering techniques.

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In nature, most plastics physically and chemically degrade to form minute particles on the micro- and nanometer scale. Multiple international agencies have recently raised environmental and health con- cerns regarding these micro and nano plastics, but there is insufficient knowledge of their properties to allow for an accurate risk assessment to be conducted and any risks managed. For this reason, research into the toxicity of nanoplastics has focused strongly on documenting their impacts on biological organisms. In this work we summarise the recent findings on the adverse effects on bio- logical organisms and strategies which can be adopted to advance our understanding of nanoplastic properties and their toxicity. Identifying the chemical and biological bases behind the observed bio- logical effects (such as in vitro cellular response) is a major challenge, due to the intricate nature of nanoparticle-biological molecule complexes and an unawareness of their interaction with other bio- logical targets, particularly at interfacial level. An exemplary case includes protein corona formation and ecological molecule corona (eco-corona) for nanoplastics. We also highlight recent findings and importance of coronae formation and structure characterisation especially using neutron scattering techniques. Finally, we discuss the opportunities provided by model system approaches (model pro- tein corona and lipid bilayer) to deepen the understanding of the above-mentioned perspectives and corroborate the findings from in vitro experiments.

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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 mi- crostructures covering length scales in the range of 0.1–20 µm. Use of the first- and second-order re- flections coming off a doubly curved highly oriented mosaic pyrolytic graphite pre-monochromator 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 Å. This unique feature among reactor based USANS instruments allows optimal accom- modation 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 sys- tems 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.

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Deuteration as a tool for structure determination of membrane proteins

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Membrane proteins are important structures for cellular function. Around a third of all human proteins are membrane-bound, and they account for about a half of all drug targets.[1,2] Despite their obvious importance, determination of the structure of membrane proteins is still a significant challenge. This mainly pertains to the difficulty of preserving the native conformation of the protein once it has been removed from its endogenous environment. The National Deuteration Facility, ANSTO, has a large catalogue of specifically deuterated materials that are able to facilitate a suite of methods to obtain structural information about membrane proteins.[3] Deuteration allows for small-angle neutron scattering (SANS) of membrane proteins without contribution from contrast- matched membrane protein carrier systems: detergents, lipid nanodiscs and lipid mesophases.[4,5,6] To obtain satisfactory SANS signal-to-noise of the membrane protein structure, the detergents or

lipids are synthesised with deuteration levels such that they have the same neutron scattering length density as the surrounding aqueous buffer. Herein, we disclose an overview of the synthetic methods used to access important deuterated detergents and lipids and provide examples of their recent use in structural biology.

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Supermirror focussing guide for Pelican

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Pelican is a cold-neutron time-of-flight spectrometer at the Australian Centre for Neutron Scattering. It has applications in a diverse range of scientific disciplines. One fundamental limitation of nearly all neutron spectroscopy is the requirement for relatively large samples due to signals (and thus measurements) being flux limited. To address this we are designing a system of focusing supermirror neutron guides that will allow a factor of ten increase in the beam intensity at on a 10 mm x 10 mm beam spot at the sample position. In this contribution we will demonstrate the design concept and ray tracing simulations that demonstrate that this will be possible. We will also give an overview of the increased scientific capabilities that will be possible using this neutron optics upgrade, this includes but is not limited to small single crystal samples, use of high-pressure cells with small sample spaces and the measurement of difficult to synthesise samples.

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Neutron scattering measurements on rare earth single molecule magnets.

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Single molecule magnets are a class of materials which show a magnetic bistability and hysteresis on a molecular level. Such properties have meant they are potential candidates for spintronics ap- plications, data storage and as qubits. Inelastic neutron scattering has been shown to give unique information on these materials as it allows the ground state splitting to be probed directly. In recent years we have demonstrated that combined with *ab initio* calculations the structure property rela- tionships can be understood. More recently it has been demonstrated that spin-phonon coupling can also be measured directly using this technique. Finally new sample environment has meant that it is possible to probe these effects in the presence of a static magnetic field. In this contribution I will give a summary of the work that has been completed on these fascinating materials at the ACNS.

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Recent highlights from the Pelican spectrometer

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Pelican is a cold-neutron time-of-flight spectrometer at the Australian Centre for Neutron Scattering (ACNS). Pelican has been in operation since 2014 and has been used for a diverse range of applica- tions, including single molecule magnets, barocaloric materials, solid oxide conductors for quantum magnets. In this contribution we will present some recent highlights from the Pelican spectrometer demonstrating the varied capabilities of the instrument.

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Neutron Diffraction at the start and end of the Nuclear Fuel Cy- cle

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Uranium oxides are the foundation of the nuclear fuel cycle, presenting as complex chemical species in both uranium ore concentrates, to phases of interest in the development of stable waste forms at the end of the nuclear fuel cycle. Australia is a major exporter of uranium and has a responsibility to contribute to the long-term management of nuclear materials. This requires an in-depth structural understanding of materials present in the nuclear fuel cycle, and how they respond to changes in the environment.

Two such examples of uranium oxides at the start and end of the nuclear fuel cycle are $[UO_2(\eta^2\text{- }O_2)(H_2O)_2]$ (metastudtite), and $CaUNb_2O_8$, a scheelite-type oxide.

The precise structure of metastudtite is controversial with low resolution structural studies sup- ported by DFT calculations suggesting that the trans-uranyl group is not linear. Non-linear uranyl groups are uncommon, with a recent study using metastudtite sourced from the Beverley mine in Australia showing that the trans-uranyl groups are linear in conflict with the DFT study. We hope to resolve these issues using high resolution neutron diffraction to establish an accurate and precise structure of $[UO_2(\eta^2\text{-}O_2)(H_2O)_2]$ in a combined S-XRD+NPD study.

$CaUNb_2O_8$ is a rare example of a scheelite-type oxide containing U^{4+} on the 8-coordinate site. In situ variable temperature synchrotron X-ray powder diffraction confirmed a reversible phase trans- formation from fergusonite to scheelite. With our upcoming in situ neutron powder diffraction mea- surements, the nature of the fergusonite to scheelite transformation in $CaUNb_2O_8$ will be revisited and the thermal stability of the scheelite explored.

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Experimental and Computational Studies of Sulfided NiMo Sup- ported on Pillared Clay: Catalyst Activation and Guaiacol Ad- sorption Sites

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We report on intermediate (oxysulfides) and sulfided structures of NiMo supported on aluminium pillared clay (Al-PILC) during the catalyst activation process and the prefered guaiacol adsorption sites on the sulfided catalyst. In situ X-ray absorption fine structure (XAFS) together with density functional theory (DFT) calculations confirm the existence of ill-defined suboxides (MoOx, NiOx) and the well-known subsulfides (Mo2S9, Ni3S2) at the first stage which, at a later stage in the pro- cess, transform into MoS2 with oxygen-decorated Mo and Ni edges. The freshly sulfided NiMoS2 catalyst under sulfiding agents is mainly terminated by Mo-edge surface with 50% sulfur coverage (Mo-S50) with a disordered Ni-edge surface that can be assigned as NiMoS ($1\bar{1}010$). When exposed to an inert atmosphere such as He gas, the Mo and Ni edges evolved partially into new structures of Mo and Ni edges with zero sulfur coverage, labelled as Mo-Bare and Ni-Bare. Guaiacol is often used as a model compound for lignin and a series of calculations of guaiacol on the structural edges of a sulfided NiMoS2 catalyst show relatively good agreement between the observed and calculated inelastic neutron scattering (INS) spectra for Mo-S50, Ni-Bare, and NiMoS ($1\bar{1}010$) where guaiacol weakly chemisorbed via oxygen atom of OH group. The results also confirm that guaiacol is ph- ysisorbed on the basal plane of NiMoS2 in a horizontal (flat-lying) configuration via van der Waals interaction at a separation of about 3.25 Å.

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Update on the progress of ACNS Industrial Liaison Office and fu- ture opportunities

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Abstract

The instruments at ACNS have a unique non-destructive ability to assist academia and industry in the characterisation of the materials and improvements of the manufacturing processes. ACNS ILO versatile team has established a strong record in assisting Australian and international researchers and engineers across a wide range of projects. Over time, we have built a team with unique expe- rience and technical expertise, which is now on offer to support industrial research and develop- ment.

This presentation will focus on the resent expansion of the team, showcase industry highlights and opportunities to further enhance the industry engagement. Our collaborative approach help indus- try find the competitive edge via neutrons and in consequence improve safety, efficiency, quality and often reduce the cost of materials or manufacturing process.

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Topologically stabilized spin configurations in rare- earth based systems using polarized neutron scat- tering

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Structural and magnetic correlations can be accessed by various neutron and x-ray scattering techniques at low angles. Topologically stabilized spin configurations like helices
in the form of planar domain walls with magnetic functionalities in transition metal/rare-earth and rare-earth/rare-earth systems are readily investigated using polarized neutron
reflectivity (PNR). Recently, using magnetization and susceptibility as a function of temperature along with thermo-remanent magnetization measurements we have confirmed the superspin-glass (SSG) type of behavior within the rare-earth/rare-earth systems of Dy/Tb, and also other transition-metal/rare-earth systems. Interestingly, a detailed analysis of the polarized neutron reflectometry profiles has established evidence of superimposed helical magnetic configurations within both rare-earth materials of Dy and Tb associated with spin-frustrated interfaces. Such coexistence of SSG and helical phases is due to the spin frustrated interdomain magnetic interaction as indicated by polarized off-specular neutron scattering. The quantized energy spectrum within finite magnetic chains results in stabilizing topologically protected configurations of helices. Since both materials are anisotropically hard and their respective helical and ferromagnetic ordering
temperatures are also significantly different in bulk form, it was hardly expected for them to establish exchange-coupled helices. Thus, our work realizes a novel state of spin configuration even within such unusual candidates. Realization of such spin configurations opens up the opportunity for scientific and technological applications in increasing the energy density for all-spin-based systems and eventually controlling the chirality in such systems, proving an extra degree of freedom in spintronics.
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Structures of photocycle intermediates of the orange carotenoid protein studied by small angle scattering using partial deutera- tion

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The orange carotenoid protein (OCP) plays a pivotal role in photoprotection in cyanobacterial pho- tosynhtesis. OCP undergoes significant structural changes upon illumination. The photoprotective function of OCP is terminated by complex formation with the fluorescence recovery protein (FRP). The individual steps of the OCP-FRP interaction are currently a matter of intensive research efforts. We used small-angle neutron scattering coupled in part with size exclusion chromatography to un- ravel the solution structures of two variants of OCP lacking the N-terminal extension (OCP-ΔNTE) and its complex formation with FRP. The dark-adapted, orange form OCP-O is compact and fully photoswitchable. Its complex with FRP consists of a monomeric OCP component, which closely re- sembles the compact structure expected for the OCP ground state. In contrast, the pink form OCP-P is mostly non-switchable. The latter pink OCP form appears to be characterized by a significant separation of the NTD and CTD protein domains. Therefore, OCP-P can be viewed as a prototypical model system for the active state of OCP. The dimeric structure of OCP-P is retained in its complex with FRP. Small-angle neutron scattering using partially deuterated OCP-FRP complexes reveals for the first time that FRP has to undergo significant structural changes upon complex formation with both OCP forms, a feature not visible in complementary small angle X-ray experiments. We con- clude that both OCP-FRP structures determined resemble individual intermediate complex states of the OCP photocycle, one resembling the complex of the active form of OCP with FRP, while the other one is similar to a step in the back-conversion of OCP from the active state to the dark-adapted ground state.

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Diverse Deuterated Lipids for Neutron Applications

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Deuterated small molecules are an important tool in structure-function investigations using neu- tron scattering and diffraction techniques. Where the availability of deuterated small molecules is insufficient in terms of quantity or diversity a bottleneck is created which limits the advancement of neutron techniques.

We will discuss recent advancements in and the impact of deuteration upon the research outcomes achieved by using bespoke deuterated lipids produced by the National Deuteration Facility of the Australian Nuclear Science and Technology Organisation. We will describe the synthesis and appli- cation of new deuterated molecules which are unavailable from commercial sources, as well as the

challenges and opportunities associated with expanding the menu of deuterated lipids available for neutron applications.

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Recent upgrades to the Kowari strain scanner

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This poster will highlight some recent upgrades to the Kowari residual strain scanner. These include, implementation of an oscillation system for the radial collimators on the detector, as well as improve- ments to the general motorisation of the detector. In addition the new ATOS5 GOM structured light scanner will also be described.

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The role of the solvent in specific ion effects: PNIPAM brushes in non-aqueous electrolytes

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Stimulus-responsive polymers can uniquely alter their conformation as a function of external fac- tors.1 When these polymers are densely tethered to a surface (forming a polymer brush), the confor- mational change can be used to modify interfacial properties. One such system is the thermorespon- sive poly(N-isopropylacrylamide) (PNIPAM) brush, which undergoes a phase transition from well- to poorly-solvated over a broad temperature range, thereby profoundly changing the interaction of the surface with biological media (e.g., cellular adhesion, protein adsorption). The phase transition of polymer brushes can also be moderated by the inclusion of ions, where the change to higher or lower temperatures is dependent on the identity of the ion: specific ion effects (SIE).1

Natural environments are composed of complex mixtures of aqueous and non-aqueous electrolytes, which poses both a challenge and an opportunity for the deployment of responsive polymer systems. The challenge is that the behaviour of these polymers in complex solutions must be understood, the opportunity is that certain solution components may be used as additional stimuli. To probe the behaviour of the brush in these complex electrolytes, we investigate changes in the behaviour of a PNIPAM brush in mixed dimethyl sulfoxide/aqueous electrolytes using neutron reflectometry (NR). NR (Platypus) is the only technique capable of resolving the subtle yet detailed changes within the internal structure of the brush. However, significant advances in modelling NR data were required to deconvolute these subtle changes in brush structure (including describing the diffuse brush with interpolating polynomials);2 unveiling the complex relationship between the solvent, solute (i.e., ions) and substrate (i.e., polymer brush).

1Gregory, Elliot, Robertson et al., 10.1039/D2CP00847E
2Gresham, Murdoch, Johnson, Robertson and et al., 10.1107/s160057672100251x

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refnx + refellips: Analysing neutron/X-ray/light scattering from surfaces in Python

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At ACNS we are fortunate to have a wide variety of tools for characterising interfacial structures using neutrons, X-rays and light, via neutron and X-ray reflectometry (NR + XRR), and ellipsometry. The complementarity of these techniques improves understanding of complex interfaces as each of the techniques are sensitive to different aspects of the system. For example, NR and XRR are very sensitive to the thickness of a polymer film, which can then be used as a constraint when modelling

its optical dielectric function from ellipsometry data.
Unfortunately many proprietry ellipsometry analysis packages are typically tied to specific hard- ware, not designed for reproducible research, and can't be combined with modelling packages for other techniques.
Here we present a new package, *refellips*, an open-source package for the analysis of ellipsometry data, that is designed to address all these issues. Moreover, *refellips* is integrated with the neutron and X-ray reflectometry analysis software, *refnx* (also developed at ACNS). This integration makes it possible to model share and co-refine neutron and X-ray reflectometry, and ellipsometry data, for the first time.
Here we outline the main points of *refellips*, how ellipsometry can be used to inform NR and XRR experimental design, and demonstrate its use in a corefinement process.

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Development of the yeast platform in the NDF for expanding the range of lipids available to researchers.

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The expansion of biosynthetic capability of the NDF has provided a greater range of molecules for neutron and other research techniques. Yeast and other microbes have been selected for expression of deuterated sterols including cholesterol and campesterol, that have been used in neutron scatter- ing investigations of SARS-CoV-2 infection and mRNA vaccine development.1,2 We have recently formed a collaboration with the ARC Centre of Excellence in Synthetic Biology at Macquarie Uni- versity to create new yeast strains for deuterated sitosterol that may be used in the development of next generation of mRNA vaccines.3
The yeast platform is also providing new deuterated lipids like squalene that will facilitate neutron research in new areas. Other lipids are in development originating from singe cell marine protists, that produce a complex mix of triglycerides, saturated and polyunsaturated fatty acids, etc in order to survive in challenging environmental conditions. Complex purifications are required to achieve high purity compounds that can be isotopically labelled using heavy water or labelled substrates in the growth medium. These challenges are being taken on by the team of bio- and organic chemists to meet the growing demand from researchers in neutron scattering, and providing reagents and standards for mass spectrometry techniques.

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3. Engineering of Phytosterol-Producing Yeast Platforms for Functional Reconstitution of Down- stream Biosynthetic Pathways https://pubs.acs.org/doi/10.1021/acssynbio.0c00417

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Update from the Australian Centre for Neutron Scattering

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The Australian Centre for Neutron Scattering (ACNS) utilises neutrons from Australia's multi-purpose research reactor, OPAL, to solve complex research and industrial problems for Australian and inter- national users via merit-based access and user-pays programs. An update will be given on the OPAL reactor and its neutron beam facilities, the status of the neutron beam instruments and supporting capabilities, user program, and future plans.

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Cubosomes: A polytherapy based approach to combat antimicro- bial resistance

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An imperative emerging problem in global healthcare is antimicrobial resistance. New antimicro- bial therapeutics are urgently needed, including innovative nanotechnologies to fight multidrug- resistant bacterial superbugs. My team discovers (Lai et al. Nature Communications 2022) that a polytherapy based approach using lyotropic liquid crystalline nanoparticles (cubosomes) is far su- perior than loading the antibiotic within the lipid nanoparticles as is commonly done.

This work exploits a range of cross-disciplinary approaches, including the use of confocal microscopy and neutron reflectometry to mechanistically study the interactions between nanoparticles, antibi- otics and bacterial membranes. Neutron reflectometry suggest the superior polytherapy activity was achieved via a two-step process. Firstly, electrostatic interactions between polymyxin and lipid A initially destabilize the outer lipid bilayer of the model membrane leaflet. Subsequently, influx of cu- bosomes into the bilayer result in significant further membrane disruption of the bilayer structure via a lipid exchange process. Our finding offers novel insight to the nanotherapeutics field whereby the 'one-size-fits-all' approach, i.e., focusing only on the conventional antibiotic-loaded nanoparticles, is not sufficient for the identification of optimal regimens with maximal bacterial killing.

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Investigating Allergenicity of Pollen Allergens in Thunderstorm Asthma Using Neutron Reflectometry

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Epidemic thunderstorm asthma (ETSA) is a rare geographically dependent phenomenon that results in a dramatic outbreak of asthma attacks within a short span of time during seasonal thunderstorms. The high load of submicronic thunderstorm-borne aeroallergens proteins owing to their submicronic size can penetrate the narrow lower airways such as alveoli to trigger inhibition of adequate gaseous exchange during breathing. The inhalation of pollen allergens has been associated with the down- stream hypersensitive immune reactions, however, the underlying physicochemical mechanism of pollen allergen interactions with the lung surfactant monolayer and the epithelial bilayer of the alveoli is not well-understood.

In this talk, we demonstrate how a simplistic biomimetic model system can aid in the investigation of biointerfacial interactions such as allergen-lung surfactant (LS) monolayer and allergen-epithelial bilayer interactions. The LS monolayer and the bilayer was constituted of *dipalmitoylphospahtidyl- choline* (DPPC) and *phosphatidylcholine* (POPC), respectively. To mimic the atmospheric activity of thunderstorm, we generated the plasma activated water (PAW) containing reactive oxygen nitrogen species (RONS) for treating allergens. Our experimental data analysis revealed that model allergen protein, *Wisteria floribunda* Lectin (WFL) fetches RONS from the environment, ionizes strongly as well as form large aggregates in an aqueous solution. It was also observed that PAW-treated WFLs intensely adsorb on the hydrophobic LS monolayer and hydrophilic bilayer and undergo conforma- tional alterations to maximize adsorption and cause insertion into the monolayer and bilayer, which

could contribute towards its enhanced allergenicity. The key findings of this study will enable effec- tive diagnostics and therapeutics for the treatment of ETSA.

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Small Angle Neutron Scattering instrument Bilby: capabilities to study mainstream and complex systems

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ANSTO for more than ten years successfully operates the Small Angle Neutron Scattering (SANS) instrument Quokka[1] and in 2016 commenced the user operation of the second SANS instrument, Bilby[2]. The Ultra-small angle scattering instrument Kookaburra[3] is completing the set of the SANS instruments at ANSTO.

Bilby exploits neutron Time-of-Flight (ToF) to extend the simultaneous measurable Q-range over and above what is possible on a conventional reactor-based monochromatic SANS instrument. In ToF mode, choppers are used to create neutron pulses comprising wavelengths between 2 and 20 Å of variable wavelength resolution (~3% – 30%). In addition, Bilby can operate in monochromatic mode using a velocity selector.

Two arrays of position sensitive detectors in combination with utilizing the wide wavelength range provide the capability to collect scattering data of a wide simultaneous angular range without chang- ing the experimental set-up (maximum accessible Q on the instrument is 0.001-1.8Å^-1).

Additionally, there is a range of sample environment available allowing to change sample condi- tions in situ, which is priceless for the study of a wide variety of samples ranging from colloids and hierarchical materials to metals. Here we present some recent examples.

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Magnetic non-uniformity of antiferromagnetic FeRh film from polarized neutron reflectometry

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B2-phase FeRh exhibits a metamagnetic first-order phase transition from antiferromagnetic (AFM) to ferromagnetic (FM) around 370 K. It accompanies a volume expansion of about 1% and variation of electrical resistivity due to the increasing density-of-state at the Fermi level. Unlike bulk, unex- pected FM characteristics have been observed in a thin-film form, even in the AFM state.
This presentation shows the detailed origin of the residual FM state by examining temperature- dependent physical properties. First, the residual ferromagnetic induces thermomagnetic irreversibil- ity (e.g., spin-glass-like behavior) and negative magnetoresistance. Second, the non-uniform distri- bution of the magnetic profile at low temperature (i.e., below the transition temperature) is observed from the temperature-dependent polarized neutron reflectometry. Interestingly, the bottom and top interfaces exhibit an FM state, while the middle region is an AFM state. Furthermore, the bottom (top) interface shows a temperature-independent (-dependent) FM state, indicating that the FM ori- gin of the interface between substrate and film (bottom interface) and capping layer (Al) and film (top interface) are due to structural distortion and non-stoichiometry respectively.

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Phonon Engineering Thermoelectrics with Inelastic Neutron Scat- tering

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The challenge of designing a highly performing thermoelectric material lies in the requirement to de-couple the electronic and thermal transport; creating a material that has the electronic proper- ties of a highly conducting crystal, while simultaneously having the thermal properties of a poorly conducting glass. The phonon-glass electron-crystal concept has contributed to significant research on the "phonon engineering" of materials. One exceptionally useful tool for aiding this engineer- ing process is inelastic neutron scattering (INS). Information such as phonon linewidth, dispersion, and density of states (DOS), directly obtained by INS, can all be related back to the thermal prop- erties of materials. In this talk, I will give an overview of the usefulness of INS as a tool to study phonons in thermoelectric materials by presenting some of our recent INS data on chalcogenide- based thermoelectric materials obtained at ACNS. In our work, we explore how INS can be used to understand the thermal conductivity of SnTe by studying the evolution of phonon modes as a function of temperature using Pelican (time-of-flight) and Sika (triple-axis). We also compare data obtained by each instrument and highlight significant and unexpected differences. Additionally, we study the phonon DOS of carbon nanomaterials, such as nanodiamond, to understand their intrinsic phonon lattice dynamics, and compare them with thermoelectrics to understand phonon scattering in nanocarbon doped thermoelectric composites due to thermal boundary resistance.

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McStas Simulations of Taipan

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McStas is a well-known ray-tracing program for the design, development, investigation of neutron instruments as well as a potential learning and teaching tool for those that wish to understand or show how a neutron instrument works.

Here we build the thermal neutron triple-axis spectrometer on Taipan in McStas, configuring it to determine the resolution function in R(ω,$\underline{Q}$) under a number of conditioning states. We are initially

interested in how the lengths of the arms of the triple-axis alter the resolution function, in-particular how close we can place the analyser to the sample before losing resolution. We are wishing to potentially increase scattered flux without degrading resolution to the point of being unusable. We also wish to explore how reducing the thickness of the sapphire filter in the pre-optics affects the ultimate resolution of the spectrometer. We discuss our results to-date.

This study is the result of a Macquarie University PACE (Professional and Community Engagement) program where third/fourth year Physics students work for a number of weeks with a company or government institution on a research, development, or industry project.

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Taipan: The Thermal Triple-Axis and Be-Filter Spectrometers

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Two thermal spectrometers are available on Taipan, a single ^{3}He detector triple axis spectrometer and a so-called Be-filter spectrometer that consists of an array of 30 ^{3}He detectors. The latter is predominately used to easily measure the vibrational density of states of powders while the former allows a wide range of measurements to be performed on single crystals, thin films, and powders, from phonon mapping, magnetic structure determination, and low energy excitation measurements. Both spectrometers are used to extract fundamental information about the magnetic and vibrational properties of a material under investigation.

Each spectrometer shares a common monochromator-set and pre-optics (virtual source, sapphire filters, and collimators). There are two monochromators available, a doubly focussed, PG(002) or Cu(002) monochromator, which gives access to incident neutron energies from ~5-200 meV and both of which may be separately driven into the neutron beam. When the triple-axis is in use, the Be- filter analyser spectrometer is hoisted out of the instrument dance floor and stored in an unoccupied space of the reactor beam-hall. While when the Be-filter analyser is in use the triple-axis analyser, detector, and sample stage is placed in the corner of the instrument dance-floor.

Here we describe the details of each spectrometer and report on some interesting recent studies.

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Rational Design of Advanced Nuclear Fuel Materials: New Path- ways from In Situ Neutron Diffraction.

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Uranium nitride (UN) is a high-density nuclear fuel, under consideration as a drop-in replacement for existing light water reactor fuel assemblies. High uranium density fuels are desired because they allow increased fuel burnup for a given enrichment, meaning more energy can be extracted from the fuel before removal from the core. However, UN is known to corrode in steam i.e., conditions that would present during a loss of coolant accident. Doping with alloying compounds is known to affect the corrosion properties of UN, yet the rational design of advanced fuels requires an in-depth understanding of the corrosion mechanisms on the atomic scale.

Here, at the Australian Centre for Neutron Scattering, our team has developed sovereign capabili- ties for studying nuclear fuels under off-normal reactor conditions. Using UN-20vol%CrN and UN- 20vol%ZrN as case studies, I will demonstrate how *in situ* neutron diffraction can provide real-time insight into when and how corrosion occurs under realistic accident-like conditions – results which cannot be obtained from conventional *ex situ* post-corrosion studies. The mechanistic insights gained lead to a new pathway for the design of advanced nuclear fuel materials with enhanced corrosion resistance in steam.

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Difference the Spot with Wombat

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Over the life of the Wombat diffractometer, there has been a steady interest in using the instrument for experiments on single crystals. With the implementation of the "Int3D" package (1) the capability now extends to full automated peak integration. This has already proven very useful; several examples will be reviewed.

1 Katcho *et.al., Crystals* **2021**, *11*, 897

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Wombat – the high intensity 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 characteri- sation 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 monochro- mator, all combine with the centre's polarisation capability to provide an instrument which is unique 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 perfor- mance battery anodes and cathodes, high strength alloys, multiferroics, superconductors and novel magnetic materials. Our poster will highlight both the capacity of the instrument, and some recent results.

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Investigation of the residual stress of steel after laser cleaning

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Key words: residual stress, neutron diffraction, laser ablation, metal and alloys

Laser cleaning is a cutting-edge technique for the removal of contaminated and painted layers from solid surfaces by irradiating the surface with a laser beam. It is a non-contact process, which can be carefully controlled to minimize damage to the original substrate beneath a contaminated or corroded surface. The nanosecond or femtosecond laser treatment, in comparison to other cleaning techniques, significantly reduces the heat output to the structure, lowers the energy consumption, and optimises the control of the characteristics of the material surface, providing better long-term conservation of the steel structures such as the Sydney Harbour Bridge which is the focus of this project.

This research investigates residual stresses and mechanical properties of nanosecond and femtosec- ond laser-cleaned steels. Non-destructive X-ray and neutron diffraction residual stress measure- ments were performed with the team at the Australian Centre for Neutron Scattering and at Nuclear Fuel Cycle at ANSTO. X-ray diffraction was used for near-surface residual stress measurements. Neutron diffraction strain measurements on the Kowari instrument, were used to enable internal residual stress analysis.

The results of the residual stress measurements indicated that the femtosecond laser cleaning process has no detrimental effect on ablated steels. However, the nanosecond laser induces residual stresses in the substrate.

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Interrogation of the mechanism of Li-mediated ammonia electrosynthesis by in situ neutron reflectometry

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Ammonia has promising applications as a low-carbon fuel. However, its production via the Haber- Bosch process contributes 1.4% of all anthropogenic CO2 emissions.1 Alternatively, ammonia can be produced sustainably from N2 and H2O directly using electrosynthesis powered by renewable energy.
The lithium-mediated nitrogen reduction reaction (Li-NRR) is the only known electrochemical pro- cess that can enable the conversion of N2 to NH3.2 The hypothesised Li-NRR mechanism includes the following steps:
Li+ + e- [g Li0, 6Li0 + N2 [g 2Li3N, and Li3N + 3H+ [g NH3 + 3Li+.
However, the potential required to drive the lithium reduction induces other electrochemical trans- formations such as the unproductive reduction of protons to H2 or hydrides, which decreases the faradaic efficiency. The solid electrolyte interface (SEI) is a thin layer of electrolyte degradation products which functionalise the electrode surface and is crucial to the Li-NRR effectiveness. Our ex situ X-ray photoelectron spectroscopic studies suggest that fluorides and sulphites are components of the SEI, but to elucidate its structure and formation whilst the surface is active, in situ characteri- sation is required.
At Monash, the current best-performing Li-NRR system has been recently developed3 and improved to achieve near 100% faradaic efficiency and high performance at 1 bar N2 pressure. These develop- ments enabled preliminary in situ NR experiments and allowed the detection of initial SEI formation. These experiments provide a guide for our future work which will provide the first, unique insights into the mechanism of the Li-NRR.

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Sample Environment Developments from the Australian Centre For Neutron Scattering

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Sample environment at ACNS has had a productive period in 2021 and 2022. We received funding to replace equipment to maintain and improve our existing capabilities and undertaken development projects to create new capabilities.
We are currently commissioning a new Anton-Paar rheometer; Cobra+ cryostream; potentiostats and new cryofurnaces. Our funding has also allowed us to regain a capability we had in the past with the purchase of a Universal Testing Machine (UTM) for our diffraction and strain scanning neutron instruments. This project was conducted with close consultation with the user groups and

will be commissioned over the next year.
We have undertaken two significant design developments in the last 12 months. One is our own 4K-800K cryofurnace and the other is an induction furnace. The choice to create a cryofurnace is to offset some of the challenges we have faced with distance to suppliers and improve our future turn- around on repairs. The induction furnace will bring high temperature capability to the backscatter instrument, Emu, and the Be-filter used on the Taipan triple-axis spectrometer. It will also allow us greater flexibility for a growing demand with gas and vapour experiments at elevated temperatures. Both projects have been designed in-house and construction and commissioning will begin soon.
A Direct Laser Melting (DLM) deposition system, co-funded by a state grant, was designed and created by the team with assistance from other operations groups at ACNS. This system allows for in situ studies of residual stress during the additive manufacturing process and was recently commissioned on the strain scanner instrument, Kowari.

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Modelling Small-Angle Neutron Scattering of concentrated worm- like micelles

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Wormlike micelles are a self-assembly of surfactants that have solution state properties akin to poly- mers but are not covalently bound together, meaning the micelles can break and reform in a dynamic fashion. Industrially, wormlike micelles are utilised in personal care products such as shampoo and liquid soaps to provide viscosity (as well as to clean). The surfactant concentrations utilised in these formulations is usually greater than 10% by mass, whereby micelles are strongly interacting and entangling with one another. Small-angle neutron scattering is uniquely appropriate for in situ investigation of such soft colloidal materials, but for concentrated wormlike micelles, small-angle neutron scattering data is strongly dominated by a volume exclusion structure factor due to inter- micellar interactions. This describes the position of the micelles relative to each other due to the high concentration. Micellar shape and size (form factor) in such systems is typically modelled using a flexible cylinder model, but is only unambiguously obtained from dilute (non-interacting) systems that are far from the concentrations used in real products.

In standard analysis software, there is no option to reliably analyse these concentrated materials. We now have the ability to model this structure factor for polymers and wormlike micelles using the polymer reference interaction site model (PRISM), an extension of the flexible cylinder model. This model has been shown to reliably extract parameters for wormlike micelles such as those formed by the prototypical sodium laureth sulfate and cocamidopropyl betaine.

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Polycation radius of gyration in a polymeric ionic liquid (PIL): the PIL melt is not a theta solvent

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Polymeric ionic liquids (PILs) are functionalised ionic liquids (ILs) that covalently attach one of the IL ions to a polymer backbone, with the other free ion acting as the counter ion. Due to their macromolecular architecture, PILs form viscous gels that are ionically conductive, making them ideal for use in solid-state electrochemical devices. The conformation of the polycation in the pro- totypical PIL poly(3-methyl-1-aminopropylimidazolylacrylamide) bis(trifluoromethylsulfonyl)imide (poly(3MAPIm)TFSI) was probed using small-angle neutron scattering (SANS) and ultra-small-angle neutron scattering (USANS) at 25 ℃ and 80 ℃. Poly(3MAPIm)TFSI contains microvoids which lead to intense low q scattering that can be mitigated using mixtures of hydrogen- and deuterium-rich materials, allowing determination of the polycation conformation and radius of gyration (Rg). In the pure PIL, the polycation adopts a random coil conformation with Rg = 52 ± 0.5 Å. In contrast to conventional polymer melts, the pure PIL is not a theta solvent for the polycation. The TFSI− anions, which comprise 48% v/v of the PIL, are strongly attracted to the polycation and act like small solvent molecules which leads to chain swelling analogous to an entangled, semi-dilute, or concentrated polymer solution in a good solvent.

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Quokka, the Monochromatic Small Angle Neutron Scattering in- strument at ANSTO: Planned Upgrades & Scientific Highlights

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Quokka is a 40 m pinhole Small Angle Neutron Scattering instrument, operating with a neutron velocity selector, an adjustable collimation system providing source-sample distances of up to 20 m and a high-count rate 1 m2 detector, capable of measuring neutrons over a flight path up to 20 m. Quokka routinely operates with focusing lens optics, capable of measuring down to a minimum Q value of 6 x 10-4 Å-1. The instrument is also equipped with incident beam polarisation and analysis capability and features a unique detector capable of countrates in excess of 5 MHz.

Quokka is designed as a general-purpose SANS instrument, with a large sample area, capable of accommodating a variety of sample environments, suitable for in-situ studies in both hard and soft matter. Grazing incidence geometry is also available through a project funded by the Taiwanese National Synchrotron Radiation Research Center.

We present current planned upgrades, including replacement of our guide system, along with a selection of recent research highlights.

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GISANS to Study The Mechanism in Perforated Layers of Blend Films of a Symmetric Polystyrene-block-Poly(methyl methacry- late)

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The distributions of dPS in PLs // can be probed by grazing-incidence small-angle neutron scattering (GISANS) and time-of-flight neutron reflectivity (ToF-NR). In this study, by finely tuning the compo- sition (ϕPS+dPS = 63.8 vol%) of the total PS/dPS component and annealing temperature (230 and 270

℃), P(S-b-MMA)/dPS blend films mainly form perforated layers with parallel orientation (hereafter PLs //). Where basically follow up our previous studied segmental distributions of polymer chains in blend films of a weakly-segregated polystyrene-block-poly(methyl methacrylate) [P(S-b-MMA)] and deuterated polystyrene (dPS). The GISANS and ToF-NR results offer evidence that dPS chains are preferentially located at the free surface and within the PS layers for blend films that were an- nealed at 230 ℃. Upon annealing at 270 ℃, dPS chains distribute within PS layers and perforated PMMA layers. Nevertheless, dPS chains still retain a surface preference for thin films. In contrast, such surface segregation of dPS chains is prohibited for thick films when annealed at 270 ℃.

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Investigation of anomalous thermal and pressure-induced distor- tion in spin crossover materials

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Metal-organic framework (MOF) materials display sensitive responses to external stimuli, which are an emerging class of materials with functionality spanning many fields.1 It remains a challenge to develop synthetic strategies for the rational design of MOFs that incorporate components with con- trollable properties. One approach to tuning the functionality of MOFs is to generate mixed-linker MOFs (MIXMOFs), where two or more ligands of similar structural geometry and solubility are in- corporated into a single framework.2 Herein, two Fe-based stimuli-responsive MOFs were generated and one of which consists of one ligand and another with mixed ligands. These two materials present reversible electronic state switching behaviour, known as spin crossover (SCO) phenomenon,3 in the response to the change of temperature and pressure. Structural properties were characterised by using powder X-ray diffraction and neutron powder diffraction under pressure to demonstrate the correlations between lattice motions. Both materials display anomalous thermal-induced struc- tural distortion showing scissor-type lattice flexing which is the colossal negative thermal expansion in one lattice direction and positive thermal expansion in another direction. Variable temperature magnetic susceptibility was conducted on these materials to demonstrate their SCO behaviours. The different extent of lattice motions and SCO behaviours under temperature and pressure of both ma- terials was demonstrated and compared to reveal the ligand field and inter- or intramolecular effect on their properties.

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The updates from the cold neutron axis spectrometer SIKA

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We would like to give you updates about the cold-neutron triple-axis spectrometer SIKA that is on the CG4 beam port at the OPAL reactor, ACNS, ANSTO. We have reported the capabilities and status of SIKA in the last several user's meetings. In this meeting, we discuss the recent scientific achieve- ments and technical developments of polarized neutron scattering experiments on SIKA focusing on the results from 2022.

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Vibrational Properties Beyond Debye Model

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For over a century, the Debye model has served the fundamental law for our understanding of the vibrational properties of bulk solid materials which show a low energy relationship of g(ω) ∝ ω2, where ω is the frequency and g(ω) is the number of modes within an energy/frequency interval. However, recent discoveries show that under certain sceneries the Debye law failed. Upon the tran- sition from solid to liquid phase, the low energy scaling changes to a linear relationship, g(ω) ∝ ω [1]. Under 2D confinement, this relationship changes to an ω^3 behaviour for solid in both crystalline and amorphous phases [2].

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Pressure Cells for Quasielastic and Inelastic Neutron Scatterings

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Neutron diffraction under high pressure has become a routine technique to study material structure evolution verses pressure, however, it is still a challenging task to perform quasielastic (QENS) and inelastic neutron scattering (INS) under high pressure for material dynamics investigation. This is mainly attributed to the intrinsic several orders of magnitude lower cross section for the QENS and INS processes than elastic scattering. Consequently, a large amount of sample and minimum background contribution from the sample cell is essential for a successful high pressure QENS and INS. Two clamp pressure cells for QENS and INS have been developed. One is a hybrid CuBe/NiCrAl cell which is for relative high pressure up to 1.5 GPa and another one is made from high strength aluminium alloy (mesolite NA723) with pressure up to 0.5 GPa. The sample volume is 0.3 mL and 1 mL respectively. The pressure cells have been thoroughly calibrated and tested. In addition, the contribution to phonon density of states from the pressure cells and transmission medium has been evaluated. The applications of the pressure cells for INS and QENS is demonstrated by studies of pressure induced phase transition of plastic crystals.

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Structural characterization of niosome nanoparticle for ocular drug delivery

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Drug delivery to the posterior segment of the eye to treat wet-type Age-related Macular Degenera- tion (AMD) is challenging due to ocular barriers and low bioavailability of drug molecules. Nanopar- ticles hold great promise for treating posterior diseases (AMD) because of their good bioavailability, ability to deliver hydrophilic and hydrophobic drugs with a controlled and sustained release rate, enhanced residence time, and low administration frequency. Here, we demonstrate the formula- tion of drug loaded niosome based lipid nanoparticles from non-ionic surfactants and cholesterol molecules to treat AMD. Niosome nanoparticles have a bilayer structure and encapsulate the hy- drophilic drug in the aqueous core. To improve the efficacy of the treatment, the formulation of niosome nanoparticle is optimised based on the size, surface potential, stability, drug loading and re- lease profile. The result shows high drug loading efficiency (over 80%) of slightly negatively charged niosome nanoparticles of size 350 nm. To investigate the structural characteristics using small angle neutron scattering techniques, we formulated the niosome nanoparticles from deuterated choles- terol. The distribution of cholesterol within the bilayer and the membrane structural changes within the vitreous environment are investigated at different time interval, providing fundamental insights of niosome degradation and drug release profile. This knowledge is imperative to develop highly efficient therapeutic nanoparticles for sustained delivery of active biomolecules, such as antibody, RNA, and genes for treating ocular diseases.