

User Meeting 2018

Thursday, 22 November 2018 - Friday, 23 November 2018

Australian Synchrotron



Book of Abstracts

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Organisational Update

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UAC Town Hall Meeting

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2018 Australian Synchrotron Stephen Wilkins Thesis Medal

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Plenary 4 / 10

Plenary 4: Dr. Cathy Foley, Deputy and Science Director of CSIRO Manufacturing

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Welcoming & Opening Address

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Concurrent Session 3

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A high-resolution synchrotron-based diffraction technique for in-situ characterisation of deformation behaviour in magnesium alloys

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To promote the accurate lattice strain measurement and twinning observation during in-situ deformation of age-hardenable lightweight magnesium alloys, a high-resolution X-ray diffraction technique was applied using medium energy synchrotron X-rays (21 keV) coupled with a fast Mythen strip detector. This technique allows data collection in transmission geometry with sufficient grain sampling statistics achieved by rocking the samples during each measurement under step-wise uniaxial tensile/compressive loads. The capabilities of the method are demonstrated on a model age-hardenable Mg–Sn based alloy in compression. The measurements confirm that this technique offers high angular resolution and wide angular range- minimising the problem of peak overlap which is advantageous for accurate lattice strain determination of both the -Mg matrix and strengthening precipitate phases. The absolute strain resolution is approximately $\pm 2 \times 10^{-4}$. Lattice strain partitioning and anisotropy in the -Mg phase reveals the occurrence of micro-plasticity due to the activation of basal dislocation slip in Mg alloys and provides experimental information for characterising the plastic anisotropy of the materials. The initiation and growth of {10-12} tension twins are identified and quantified from the changes of integrated intensities of 10-10/0002 reflections as a function of stress. The critical resolved shear stresses (CRSS) for the activations of basal slip and tension twin modes in both non-aged and aged materials were estimated. Results reveal that after the ageing treatment, the CRSS value for basal slip increases from 18 to 33 MPa, an increase of ~83%; and the CRSS value for tension twinning increases from 32 to 52 MPa, an increase of ~63%. The methodology also enables further microstructural data to be probed in an in-situ manner. This includes the apparent area-weighted twin size and dislocation density during twin onset and the precipitate volume fraction.

Parallel Session 2 / 14

Localised Synchrotron Radiation In Mice Induces Persistent Systemic Genotoxic Events Mediated By The Functional Immune System

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The discovery of the radiation-induced bystander effect (RIBE) (1) has expanded knowledge of radiobiological mechanisms beyond the scope of the central dogma of radiation biology, i.e. that only cells that absorbed a dose of ionising radiation (IR) are affected and the response is dose-dependent. The RIBE is now a well-established phenomenon comprising cyto- and genotoxic effects in out-of-field cells associated with irradiated cells. A counterpart in vivo phenomenon, a change in an organ or tissue distant from the irradiated region, was termed the radiation-induced abscopal effect (RIAE) (2). The mechanisms of the RIAE are only beginning to be understood, however the immune system has been proposed as the main mediator.

It is not known how radiation settings affect non-targeted normal tissues and therefore the risk of radiation-related adverse abscopal effects. At the Imaging and Medical Beamline (IMBL), the Australian Synchrotron, we examined systemic effects of microbeam radiotherapy (MRT) and broad beam (BB) configurations, in mice that were locally exposed to a very short pulse of a high dose-rate X-ray synchrotron beam (49 Gy/sec). We determined how radiation volume and dose impact the RIAE. We associated the propagation of these systemic effects with the induction of innate and adaptive immune effector responses and with modulations of plasma cytokine concentrations. Finally, we compared the RIAE in mice with the functional immune system and in immune-deficient mice. C57BL/6 mice were irradiated with 10 or 40 Gy incident dose of MRT or BB in an 8x8, 8x1, or 2x2-mm area of the right hind leg. For irradiation with MRT, a collimator produced beam widths of 25 μ m and microbeam centre-to-centre spacings of 200 μ m. The absorbed doses of incident and scattered radiation were measured with the radiochromic EBT3 and XRQA2 films. Blood samples, irradiated skin and a variety of normal unirradiated tissues were collected for DNA damage analysis of double-strand breaks (DSBs) quantified as gamma-H2AX foci in tissue sections and oxidative clustered DNA lesions (OCDL) measured by constant field gel electrophoresis of genomic DNA treated with pyrimidine- and abasic site-specific enzymes. We also measured the systemic immune response (plasma cytokine concentrations) and the local immune response (in-situ quantification of immune cells). The 10 Gy 8x8 mm MRT irradiation experiment was repeated in immune-deficient mice; (i) NOD SCID gamma (NSG), (ii) CCL2/MCP1 knock-outs, and (iii) in C57BL/6 mice treated with anti-CSF1R ASF98 antibody which effectively depletes macrophages.

OCDLs elevated in a wide variety of unirradiated normal tissues. In out-of-field duodenum, a trend for elevated apoptotic cell death was observed under most irradiation conditions, however DSBs elevated only after exposure to lower doses (10 Gy peak dose, but not 40 Gy). These genotoxic events were accompanied by changes in concentrations of MDC, CCL2/MCP1, Eotaxin, IL-10, TIMP-1, VEGF, TGF -1 and TGF -2 plasma cytokines and by changes in frequencies of macrophages, neutrophils and T-lymphocytes in duodenum. Overall, systemic radiation responses were dose-independent (3). Strikingly, these effects and the abscopal innate and adaptive immune effector responses were completely or partially abrogated in the mice with various immune deficiencies (4), highlighting the role of the functional immune system in propagation of systemic genotoxic effects of localised irradiation.

These findings have implications for the planning of therapeutic and diagnostic radiation treatment to reduce the risk of radiation-related adverse systemic effects.

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Parallel Session 12 / 16

Broad CD8+ T cell receptor cross-recognition of distinct influenza A strains is facilitated by molecular mimicry in humans

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Newly-emerged and vaccine-mismatched influenza A viruses (IAVs) result in a rapid global spread of the virus due to minimal antibody-mediated immunity. In that case, established CD8⁺ T-cells can reduce disease severity. However, as mutations occur sporadically within immunogenic IAV-derived T-cell peptides, understanding of T-cell receptor (TCRab) cross-reactivity towards IAV variants is needed for a vaccine design. We investigated TCRab cross-strain recognition across IAV variants within two immunodominant human IAV-specific CD8⁺ T-cell epitopes, HLA-B37:01-restricted NP338-346 (B37-NP338) and HLA-A01:01-restricted NP44-52 (A1-NP44). We found high abundance of cross-reactive TCRab clonotypes recognizing distinct IAV variants. Structures of the wild-type and variant peptides presented revealed preserved conformation of the bound peptides. Structures of a cross-reactive TCR-HLA-B37-NP338 complex suggest that molecular mimicry underpins TCR cross-reactivity towards the mutated variants. Overall, cross-reactive CD8⁺ T-cell responses, underpinned by molecular mimicry, facilitate recognition of distinct IAV variants, thus CD8⁺ T-cell targeted vaccines could provide protection across different IAV strains.

Parallel Session 14 / 17

The Contribution of Brain Metal Homeostasis to Memory Loss and Dementia

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Dementia has increasing prevalence in western society and poses significant health and economical concerns. It is expected that 131.5 million people will be affected by 2050 (1) and the associated health care cost will be USD \$2 trillion in 2030 (2). In light of this, it is important to further characterise the chemical pathways leading to dementia onset and memory loss, which may help identify potential targets for therapeutic intervention. There is substantive evidence of increased free radical mediated oxidative stress during ageing, which may drive a switch from healthy brain function to dementia. Many studies have examined increased metal levels in the brain during ageing, as a potential driver of heightened oxidative stress, yet, localised metal deficiency may also contribute to the pathology. Fe, Cu and Zn are essential for healthy brain function, and metal deficiency during neurodevelopment is catastrophic.

Therefore, we are currently investigating the hypothesis that localised metal deficiency during ageing may contribute to memory impairment observed in dementia. To test this hypothesis we have begun characterisation of brain-metal levels in a mouse model of accelerated ageing (senescence accelerated mouse (SAM) model) using X-Ray Fluorescence Microscopy (see attached figure 1). Our results have revealed alterations to copper, zinc and iron concentration within the brain during ageing, in this model (3). Specifically, the accelerated ageing model is characterised by substantial Zn deficiency within the hippocampus – a key brain region for spatial learning and memory. We have complemented our XFM elemental analyses with Fourier Transform infrared Microscopy studies, which appear to highlight a correlation between biochemical alterations to lipids and metal homeostasis (3). We hope continued investigation of our hypothesis may provide further insights into disease and memory loss mechanisms, which in turn, could reveal strategies for prevention.

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UAC Breakfast Meeting (by Invitation only)

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In-situ synchrotron XRD characterisation of Cu₆Sn₅ lithium-ion battery anodes

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Metal-based anodes for Li-ion batteries, e.g. Si, Sn and Ge, have been widely studied for their superior storage capacities. However, they are known to suffer from poor cyclic performance due to large stresses induced during the lithiation and delithiation processes. Intermetallic compounds (IMC) combining these elements with an inert element are commonly used to reduce the stresses in these anodes. One such example, the IMC Cu₆Sn₅, has been proven to improve the cyclic performance of Sn. Sn-based anodes also have lithiation voltages around 0.4 V vs. Li/Li⁺, making them less prone to lithium dendrite growth and therefore intrinsically safer. This work proposes a two-steps method to grow the Cu₆Sn₅ IMC directly on the Cu current collector, thereby greatly simplifying the anode manufacturing process. The lithiation and delithiation mechanisms of the Cu₆Sn₅ produced via this method were characterised with in-situ synchrotron XRD and high-voltage transmission electron microscopy. The results highlighted the differences between reaction mechanisms during fast

lithiation/delithiation and those in slower reactions studied using conventional laboratory based XRD reported in the literature.

Parallel Session 5 / 21

Self-Assembly of Pluronic Polymers in Protic Ionic Liquids

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Development of alternate templating for mesoporous metals can contribute to various areas, including the development of novel battery electrodes and catalyst scaffolds. Due to their sol-gel properties and drastic rheology changes with temperature at high concentrations, Pluronic triblock copolymers in ionic liquids could potentially be used for templating applications. A high throughput study was conducted using Small Angle X-ray Scattering to understand the nanostructure of the self-assembly of Pluronic polymers as a function of ionic liquid solvophobicity. Representative Pluronic triblock copolymers with varying PEO block lengths, F127 (PEO106–PPO70–PEO106), P123 (PEO20–PPO70–PEO20) and L121 (PEO5–PPO70–PEO5) were investigated in the nitrate based protic ionic liquids of ethylammonium nitrate (EAN), ethanolammonium nitrate (EtAN) and propylammonium nitrate (PAN) using the SAXS/WAXS beamline at the Australian Synchrotron. Complex phase behaviour was observed in the polymer/ionic liquid compositions, with the solvophobicity of the ionic liquids having a noticeable effect on the formation of higher order phases.

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Characterisation of Cu-Ga intermetallics for future flexible electronic packaging

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There have been ongoing efforts to develop low temperature soldering processes to reduce energy consumption and facilitate the use of delicate or sensitive microchip components. Gallium (Ga) and Ga alloys have potential to be applied in microelectronic applications because of their lack of toxicity, low melting points and the ability to form stable high melting temperature solid solutions and intermetallic compounds (IMCs) with other metals such as Cu at low temperatures. In solder joints, IMCs play an important role in thermo-mechanical fatigue, and the directional thermal expansion behaviour of IMCs is a key thermophysical

property that is required to be measured for solder joint reliability. This work involves a microstructure analysis of the interfaces that result from reactions between liquid Ga-based alloys and Cu substrates at room temperature. Fundamental thermal expansion behaviour of Cu-Ga IMCs was characterised using Synchrotron XRD in conjunction with high voltage TEM. The results are used to discuss the potential use of Ga-based alloys in low temperature soldering applications. XRD experiments were performed at the Australian Synchrotron (Beamtime Reference No: AS161PD10430 and AS181PD13607).

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Higher Energy Capabilities Demonstrated at XFM

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Recent commissioning activities at the XFM beamline have demonstrated the ability to reach above 27 keV incident energy. Thus the K-edge fluorescence of elements such as silver and cadmium are now accessible. Some demonstrations of the high energy tests will be shown, such as Mo and Ag K-edge XANES. Motion control upgrades to the Kirkpatrick-Baez X-ray focusing optics will be required to achieve adequate flux for focus operations. Users are encouraged to speak to us about their experimental applications.

Parallel Session 8 / 24

Identifying optimal clinical scenarios for synchrotron microbeam radiation therapy

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Background: Synchrotron Microbeam Radiation Therapy (MRT) is a pre-clinical modality characterised by a periodically alternating peak-valley dose-distribution. Dosimetry studies using clinical datasets have not yet been conducted. Our aim was to identify optimal settings for a future Phase I trial from a range of clinical scenarios refractory to standard treatments.

Materials and methods: Seven clinical scenarios were chosen for MRT planning. A hybrid algorithm which combines Monte Carlo and convolution-based approaches was used for dose-calculation. The objective of MRT plans was to ensure the valley dose to organs at risk (OARs) was within the tolerance doses achieved in the corresponding clinical plans. We then assessed the corresponding peak doses and peak-to-valley dose ratio (PVDRs) at the tumour target volume.

Results: Tumours with small and shallow volumes could receive peak doses greater than 80 Gy in a single fraction with PVDRs greater than 10. These scenarios included recurrent glioblastoma, head and neck tumours, and select loco-regionally recurrent breast cancer sites. Treatment volume was a more important factor than treatment depth in determining the PVDR. The mean PVDR correlated strongly with the size of the target volume ($r = -0.70$, $p = 0.01$).

Conclusion: In the context of the current physical limitations of a horizontal beam-line, our findings suggest that intra-cranial and head and neck sites will be optimal scenarios for a future trial of MRT.

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Toxicity and gene regulation following synchrotron radiation therapy

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Background: Synchrotron microbeam radiation therapy (MRT) is a pre-clinical treatment that could be used for tumours refractory to conventional treatment, such as Diffuse Intrinsic Pontine Glioma. The aim of this work was to: 1) determine equivalent doses of MRT and broad-beam radiation therapy, and 2) discern the differential effects of each modality on the brainstem.

Method: In part one, C57BL/6 mice (8-10 weeks old, male and female) received whole brain irradiation using synchrotron broad-beam radiation therapy or MRT. A dose-escalation methodology was used with at least four dose-groups per modality and $n=5$ per group. Mice were monitored for signs of acute toxicity and were euthanised at the following endpoints: $>20\%$ weight loss, $>15\%$ weight loss plus signs of moribund behaviour, neurological symptoms such as fitting, ataxia or balance disorders. Median toxic doses were calculated for both irradiation modalities using PROBIT analysis. In part two, C57BL/6 mice were irradiated with the median toxic doses determined in part one and euthanised 4 or 48 hours post-irradiation ($n=6$ per group). RNA was extracted from the brainstem and sequenced at the Australian Genome Research Facility (Parkville, Victoria).

Results: The median toxic doses for synchrotron broad-beam and MRT were 13.1 (9.2-17.2) Gy and 268 (232-313) Gy, respectively. Mice in the 455 Gy MRT group experienced acute neurotoxicity within 2-4 hours of irradiation. Differences in gene expression are currently under analysis.

Conclusion: This is the first in vivo dose-equivalence data for MRT and broad-beam irradiation, allowing for a more robust comparison of the radiobiological properties of the two modalities, including gene regulation.

Parallel Session 6 / 26

Micro-Computed Tomography (MCT): A progress report

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The Micro-Computed Tomography (MCT) beamline is one of the first two beamlines to be constructed at the Australian Synchrotron as part of the BR-GHT program. This new beamline will be operational for user experiments by mid-2021. A report on the current status of the MCT project will be provided. MCT will complement the existing X-ray tomography capability provided by the Imaging & Medical Beamline (IMBL), targeting applications requiring higher (sub-micron) spatial resolution, with commensurately smaller field-of-view.

MCT is currently in the procurement phase, with particular emphasis being placed on the photon-delivery system (PDS). The key X-ray optical element in the PDS will be a double-multilayer monochromator (DMM). Detailed calculations related to the design of the DMM will be discussed, including important aspects which inform the possible choices for the multilayer stripes. The operational X-ray energy range (8 to 40 keV) and bandpass requirements for different imaging (including phase-contrast) modalities need to be considered. However, an appropriate choice of multilayer stripes also needs to be guided by practical issues such as: thermal and radiation-hardness properties of the materials involved; reducing surface roughness and thereby increasing reflectivity; reducing overall thickness to relieve possible stress and deformation; having grazing-incidence angles which are not so small that the mirror lengths and/or their separation become prohibitively large.

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User Portal used at the australian synchrotron

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Poster Only.

Overview of the current user portal.

Major changes that affect users that have happened in the last 12 months.

Major changes that affect users that are planned for the next 12 months.

Parallel Session 12 / 28

Cholesterol-Dependent Cytolysins: from Water-Soluble State to Membrane Pore

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Cholesterol-dependent cytolysins (CDCs) are a family of pore-forming toxins that punch holes in the outer membrane of eukaryotic cells. The CDCs exhibit a number of unique

features amongst pore-forming toxins including an absolute dependence on the presence of cholesterol-rich membranes for their activity and the formation of oligomeric transmembrane pores greater than 150 Å in diameter. The first crystal structure of a CDC was that of perfringolysin O [1] and most of our understanding of CDC function is based on studies of this toxin [2-4]. We have subsequently determined structures of other family members that have confirmed that the 3D fold first seen in PFO is shared by all family members [5-9]. We have determined a number of CDC structures which are providing valuable insights into the role of receptor binding, oligomerisation and prepore assembly [8,9]. The conversion from water-soluble monomer to pore is highly complex: it is essential that the pore does not form prematurely otherwise the target cell won't be successfully breached [10]. The crystal structures of the water-soluble states of these toxins, together with cryo-electron microscopy, small angle X-ray scattering data, fluorescence spectroscopy and molecular dynamics simulations have proved very useful for modelling their membrane pores.

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Parallel Session 15 / 29

Overview of in-situ synchrotron X-ray imaging and powder diffraction techniques for Pb-free micro-electronic interconnects

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The development of reliable advanced micro-electronic interconnects that are free of toxic materials such as lead depends on an in-depth understanding of the microstructures that exist in individual solder joints. This research combines the synchrotron techniques of in-situ imaging and powder diffraction (PD) to give an understanding of the development and stability of these microstructures. This presentation provides an overview these challenging techniques and provides key findings related to understanding the reliability of Pb-free solder joints. The following two advanced experimental approaches will be discussed, (1) Synchrotron X-ray imaging at SPring-8 synchrotron BL20B2 and BL20XU, (2) synchrotron PD at the Australian Synchrotron PD. This research has been conducted with support from the Australian Synchrotron (beamtime and international access grant IDs: AS101/PD/2249, AS112/PD/3712, AS113/PDFI/4113, AS/IA114/4743, AS121/PD/4524, AS122/PD/4903, AS123/PD/5327, AS/IA124/6235, AS131/PD/5784, AS142/PD/7943, AS/IA143/9218, AS/IA151/9538, AS161/PD/10430, AS/IA163/11874), SPring-8 synchrotron (beamtime IDs: 2009A1159, 2011B1048, 2012A1192, 2014B1620, 2015A1675, 2016B1319, 2017B1519) and an international cooperative research program between the University of Queensland and Kyushu University (Progress 100 project), Kyoto University, Imperial College London, UniMAP, Nihon Superior Co, with additional support from The Australian Research Council.

Parallel Session 15 / 31

Production of Light Metal Alloy Powders by Reduction of Metal

Oxides

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Light metal alloy components fabricated via powder metallurgy processes have significantly lower manufacturing costs compared to those formed by traditional methods. Traditionally, powders are made from bulk alloy ingots, but a number of recent reduction techniques allow high purity alloy powders to be generated directly from metal oxides.

The Powder Diffraction beamline at the Australian Synchrotron was used to understand the reduction mechanism of various metal oxides used in light metal alloy formulations.

By using in-situ synchrotron techniques, it was possible to observe these highly atmosphere sensitive reactions at high temperature, highlighting phases not predicted by thermodynamics. The high angular resolution available was essential to differentiate the peaks of the intermediate phases. When a mixture of oxides was studied, the reaction path and kinetics differed from those observed for single oxide reduction experiments.

These insights will allow better understanding of the parameters that influence the process to make industrial fabrication more efficient.

Parallel Session 6 / 32

Time resolved measurements of medical inhaler sprays at the Advanced Photon Source

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In the search for new and improved medical inhaler devices for the treatment of asthma and other pulmonary diseases, pharmaceutical device makers must understand how small changes in drug properties and the design of the delivery device can affect the properties of the micron-size droplets these devices produce. The scientific challenge underlying this effort is that dense liquid sprays present a very challenging measurement environment. The density of gas-liquid interfaces inside the spray scatters visible light so effectively that the spray becomes opaque. The time and length scales present in these sprays are typically on the order of microseconds and microns. As a result, the laser based techniques which the industry has relied on for decades are no longer able to deliver all the answers to the challenges they currently face. At the Advanced Photon Source at Argonne National Laboratory in Illinois USA, researchers from Monash University are working with beamline scientists from Argonne's Time Resolved Research group (Sectors 7-BM and 9-ID) to use synchrotron radiation to address these challenges. X-rays are not scattered as strongly as visible light by the droplet field, allowing us to probe inside the dense regions near and inside the inhaler nozzle where laser diagnostics cannot. This allows us to see in great detail for the first time the complex fluid mechanics that occur in these devices. A range of techniques have been applied. Time resolved X-ray radiography provides a quantifiable density distribution in the spray. Time resolved X-ray fluorescence spectroscopy allows us to track the drug concentration independent of any other chemicals in the formulation throughout the spray, as many common inhaled drugs contain

tracer elements such as bromine. Ultra-fast X-ray phase contrast imaging also reveals the qualitative structure of the liquid-gas interfaces in the device itself. Most recently, ultra small angle X-ray scattering has allowed us to make average composition measurements of the droplets in flight, in the dense region of the spray where the droplet size is ultimately determined. The insights gained through these measurements will enable the development of more physically robust models which can then be used in the development of new devices. The research also aims to address some major open questions about the physics of how droplets form in medical inhaler sprays. This work is supported by the Australian Research Council and Chiesi Limited through the DECRA and Linkage Project schemes. Travel to the APS for Monash Researchers was made possible through an International Synchrotron Access Program grant from ANSTO and the Australian Synchrotron.

Parallel Session 12 / 33

T cell recognition: few more tricks up their sleeves

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Our immune system comprises an army of diverse cell types designed to defend our body against infections. These immune cells specifically recognise invaders (pathogens) and, once activated, elicit a targeted attack to eliminate the infection. Within the army of immune cells are cytotoxic T cells equipped with receptors (TCRs). TCRs recognise a fragment of a pathogen (i.e., bits of peptides, lipids, or small molecules) that are presented by a host-specific major histocompatibility complex (MHC) molecule found on the surface of antigen-presenting cells (APCs). This interaction between a TCR and an antigen (i.e., a pathogen fragment) bound to an MHC molecule is critical, as it is the first event in the process of T cell activation that will, in turn, dictate the fate of an infection.

Over the last 20 years, the field of T cell immunology has greatly benefited from structural biology. The first structure of a TCR recognising an antigen bound by an MHC molecule was solved in 1996. Since then, numerous co-crystal structures of TCRs in complex with different MHC molecules bearing diverse antigens have been reported, providing us with a snapshot of this critical interaction. Studies linking structural and functional information about T cell recognition have also been highly informative. From the structures available, some common features of the molecular basis of antigen recognition by T cells have emerged. In particular, we have observed conserved docking modes and interactions across diverse TCRs and pathogens. However, new T cells have recently been discovered as a result of advances in isolating rare T cell populations and the development of targeted mass-spectrometry techniques to identify novel antigens. Surprisingly, these newly identified T cells do not follow the previous dogma, and have made us rethink the molecular basis of TCR recognition.

Using X-ray crystallography to determine the specific interaction between the TCR and the antigens-MHC complexes, my group's work has revealed some novel modes of antigen recognition by T cells. Those discoveries have opened up new avenues in the field of T cell immunology. Here I will present examples of those unusual TCR-antigen-MHC structures, and show that T cells still have a few more tricks up their sleeves in the fight against infection.

Parallel Session 12 / 34

Understanding CD4+ TCRs recognition of a single HIV epitope presented by multiple HLA class II molecules

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Human Immunodeficiency Virus (HIV) is a major health issue. With 2 million people newly infected per year, there is an urgent need to develop an HIV cure. Without treatment, HIV infection leads to the progressive disruption of the immune system leading to AIDS, and the occurrence of multiple opportunistic infections.

Surprisingly, a small fraction of HIV-infected individuals (< 0.5%) can spontaneously control HIV replication in the absence of antiretroviral therapy. These patients, named HIV controllers show signs of a particularly efficient cellular antiviral response, which control the virus.

The role of CD8+ T cells in HIV has been extensively studied; however the role of CD4+ T cells remains unclear, due to the elimination of those cells upon infection. In HIV controller individuals the count of CD4+ T cells stays high, as they are able to control the viral load. These individuals provide a unique opportunity to study the immune system in the context of HIV infection.

We recently identified a CD4+ T cell population specific from HIV individuals exhibiting a highly biased TCR repertoire recognizing an immunodominant capsid epitope (Gag293). Those T cells were of high affinity and polyfunctional, as well as able to recognise the Gag293 presented by diverse molecules called HLA.

Using structural and functional approaches, our work published in Science Immunology[1] revealed for the first time how a single TCR can recognise so many different HLA molecules, and the role of the HIV epitope in driving this recognition. We provided the first functional and structural basis of the role of CD4+ TCRs in HIV infection in the context of HIV controller individuals, offering new avenues to develop immunotherapeutic approaches.

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Structural insights into Cu binding with N-Truncated Amyloid- β peptides from X-ray Absorption Spectroscopy

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Alzheimer's disease (AD) is a progressive neurodegenerative disorder characterized by the presence of amyloid plaques composed mainly of amyloid- β peptides (A β). Soluble, diffusible A β oligomers of unknown structures may be involved in extensive redox chemical reactions possibly causing cellular toxicity. The first protein sequencing studies of the A β plaque core (APC) of AD patients identified NH₂-terminal heterogeneity; the majority (64%) of the APC-AD A β peptides begin with a F₄ residue.

Novel X-ray Absorption Spectroscopy (XAS) of A β under in situ electrochemical control (XAS-SEC) with propagation of uncertainty for hypothesis testing has allowed elucidation of the relationship between the truncated peptide structures and the redox properties of the

bound Cu^{II} bound. XAS of $Cu^{II}:A\beta_{1-16}$ and $Cu^{II}:A\beta_{4-y}$ ($y=9,12,16$) frozen solutions (10 K) and XAS-SEC at room temperature under potentiostatic control have been measured. In two experiments at the Australian Synchrotron XAS beamline, the excellent performance is demonstrated by the structural uncertainty of the final results.

The XAS of $Cu^{II}:A\beta_{4-y}$ ($y=9,12,16$) show the oscillations characteristic for Cu^{II} binding to the ATCUN site and prove that neither Y_{10} nor E_{11} are involved in Cu^{II} binding at pH of 7.4. The binding geometry is different from the Cu^{II} binding site of $A\beta_{1-16}$ which involves three histidine residues: $H_6H_{13}H_{14}$. XAS proves that $Cu^{II}:A\beta_{4-y}$ ($y=9,12,16$) peptides have ATCUN-type coordination with axial oxygen in pseudo-tetragonal pyramid geometry [1].

We show that the reduction potential, $E^0(Cu^{II/I})$, depends on the relative strength of Cu^{II}/Cu^I binding. The ATCUN motif is available only for the truncated $A\beta_{4-y}$ peptide giving both higher K_d and a more negative reduction potential, i.e. $E^0(Cu^{II/I}:A\beta_{1-16}) \gg E^0(Cu^{II/I}:A\beta_{4-y})$. There is some evidence that reduction of $Cu_{II}:A\beta_{4-16}$ may proceed from Cu_{II} bound (ATCUN) through a preorganization electron transfer (POET) mechanism to a linear $H_{13}H_{14}$ binding site (bis-His motif).

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Parallel Session 3 / 36

Low energy electron inelastic mean free path of Zinc from XAFS using XERT techniques at room temperature.

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The electron inelastic mean free path (IMFP) is the average distance travelled between successive inelastic collision for an electron moving with a particular energy in a given material [1]. IMFP and Electron Loss Functions (ELF) are used in electron diffraction and LEED and EELS techniques where low energy inelastic scattering is dominant in electron transport. Measurements and theoretical understanding of these are also critical for Monte Carlo detector and scattering transport codes and applications. However, experimental data usually only exists at high energies. Our research work develops a unique experimental technique at the XAS beamline for determining low energy electron inelastic mean free path of photoelectrons with high accuracy XAFS measurements.

There are significant discrepancies between theoretical and experimental IMFPs. Resolution of these discrepancies requires a series of high accuracy experimental XAFS measurements. Our group has developed the X-ray extended range technique (XERT) to obtain precise XAFS measurements. In this work, precise XAFS measurements of zinc metal at room temperature were collected at the Australian Synchrotron. We are able to diagnose and correct for most systematic errors such as dark current, thickness, scattering linearity etc. Our high accurate data sets permit the determination of beam-line independent, critical measurements of IMFPs.

Using these high quality data and with the technique developed to measure low energy based on the coupled plasmon models by Chantler and Bourke, room temperature low energy electron inelastic mean free paths of Zinc will be calculated. This will answer questions about the interaction of photoelectron with condensed systems. It will investigate the scaling of the theoretical curves which have often been said to follow a 'Universal Curve' despite different theory predicting different universal curves. It will also investigate the relevance of coupling

of plasmon resonances in the theory and experiment, and the possible influence of correlation as has been discussed by several authors recently.

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Phase-contrast computed tomography of full mastectomy samples with different types of breast cancer using Hamamatsu C10900D detector at IMBL

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Phase-contrast computed tomography (CT) using synchrotron sources has the potential to be developed into a medical imaging tool with superior characteristics compared to current mammography and breast imaging in general. We expect that propagation-based phase-contrast CT technique [1] will be applied for medical breast imaging in the near future. This technique will potentially reduce false negative and false positive cancer diagnoses due to overlapping regions of tissue in 2D mammography and remove patient pain and discomfort due to breast compression. The unique properties of synchrotron X-ray sources such as high coherence, energy tunability and high brightness are very useful for producing low-dose phase-contrast CT scans with short exposure times. The coherence of the synchrotron radiation enables phase-contrast imaging with superior sensitivity to soft tissues tumors. Moreover, the advanced step of phase retrieval in CT reconstruction, allows us to work with noisier images [1, 2], providing a potential for reducing the radiation dose delivered to the patients.

This study was conducted in hutch 3B, IMBL, Australian synchrotron, using a new Hamamatsu CMOS Flat Panel Sensor C10900D, with CsI scintillator deposited directly on 2D photodiode array, with 1216×1216 pixels with 100 μm size and maximum counts of 4000. The detector was used in “fine mode” with minimum exposure time of 59 ms and frame rate of 17 frames/s. This detector has high quantum efficiency and very low noise level, which is important for this type of study.

Certain essential parameters of phase-contrast CT technique relevant to breast cancer imaging [3, 4] were explored in this study, such as the optimal X-ray energy, effect of the propagation distance, minimal acceptable Mean Glandular Dose and key detector characteristics. Full and fresh mastectomy samples with different types and grades of breast cancer lesions, as well as cancer-free samples, were used in this study to approach the real condition of human breast imaging. A sufficiently large number of samples is required in order to verify the degree of superiority of this technique compared with the existing medical breast imaging techniques. This work will also help optimize protocols for a clinical trial in 2020.

Acknowledgements

This research was undertaken on the Imaging and Medical beamline at the Australian Synchrotron, part of ANSTO. The authors acknowledge funding from Project Grant 1138283 “Towards the clinical application of phase-contrast computed tomography in breast cancer imaging”, NHMRC, Australia.

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Parallel Session 10 / 39

Liquid Crystalline Structures in Digesting Milk-like Emulsions and Their Potential for Drug Delivery Studied Using Small and Wide Angle X-ray Scattering

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Milk is nature's lipid-based formulation for delivering fat-soluble nutrients to infants and remains a mainstay of the adult diet thereafter. Commercially produced lipid-based formulations are used in the oral delivery of fat-soluble drugs, in which the emulsified lipids help

to dissolve entrained drugs to promote absorption in the intestine and drug bioavailability. Critical to this absorption process is the digestion of the lipid carrier phase, in which apolar triglycerides are broken down into monoglycerides and fatty acids. Initial studies performed on the small and wide angle X-ray scattering (SAXS/WAXS) beamline of the Australian Synchrotron revealed that these amphiphilic milk fat digestion products spontaneously assemble into a progression of liquid crystalline structures over time during in vitro lipid digestion.[1] This presentation will elaborate on those initial studies, discussing the liquid crystalline structures formed in a variety of milks and milk-like emulsions and the influence of milk processing on the structures that form.[2] By utilising two camera lengths on the SAXS/WAXS beamline we have been able to correlate the extent of digestion of milk lipids with both the self-assembled liquid crystalline structures formed during digestion and the crystalline forms of drugs present in the digesting emulsion.[3,4] When combined, these data reveal the key role of the lipid digestion process in determining the fate of fat-soluble drugs co-administered with milk.

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Employing X-ray Fluorescence microscopy to quantify biometals of two distinct micro-aggregates in the Parkinson's disease brain

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Alterations in biometals and metalloproteins are increasingly associated with neuronal loss in multiple neurodegenerative diseases. In the Parkinson's disease brain, we have identified a marked reduction in Cu, and alterations in a metalloprotein superoxide dismutase 1 (SOD1), which normally binds Cu and Zn in a 1:1 ratio for antioxidant activity and structural stability. As a lack of Cu-binding is implicated in SOD1 misfolding and neurotoxicity in another disease Amyotrophic lateral sclerosis (ALS), we quantified Cu and Zn levels on these micro-aggregates to determine whether the Cu:Zn ratio is also altered in the Parkinson's brain. Through tagging SOD1 micro-aggregates and Lewy body aggregates with lanthanide metals in fresh frozen human post-mortem tissue, we obtained pilot data indicating that SOD1 aggregates contain a higher proportion of Zn than Cu. We aim to build on this data with further beamtime on the XFM beamline at the Australian Synchrotron later this year and obtain a full elemental fingerprint for both SOD1 and Lewy body aggregates. These elemental data, including Zn and Cu levels and their spatial stoichiometric ratios will provide crucial support for our hypothesis that aggregated SOD1 in the Parkinson's SN is Cu-deficient. In addition, although we have demonstrated that SOD1 aggregates are chemically and structurally distinct from Lewy bodies, a common pathological feature found throughout the PD brain, we aim

to perform Ptychography to identify distinguishing structural characteristics of these two distinct aggregates within the Parkinson's disease brain.

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Gas penetration in coals and cokes and its influence on coke reactivity

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The strength and reactivity of metallurgical coke determines its desirability for use in a blast furnace for iron-making. This project is examining the role of gas penetration on the reactivity of coke and coal specimens in order to improve prediction of coke properties.

Using sychrotron micro-CT at IMBL we have imaged the microstructure of coke and coal samples pressurised with high-contrast xenon gas to determine the gas penetration behaviour, including the behaviour of inerts within the coke (inerts are regions which did not soften and deform on transformation into coke) . By subtracting images before and after gas penetration, or using K-edge subtraction, the spatial distribution of gas within each sample can be determined.

We have carried out these imaging experiments before and after reaction of the coke samples with CO₂ at high temperature and are using 3D image analysis to examine the correspondence between regions of high gas penetration and regions of most mass-loss following reaction. The importance of gas penetration in coke reactivity determined from this analysis will be discussed together with the post-reaction changes in gas uptake behaviour of coke, particularly within inerts.

Plenary 1 / 42

Source II National Synchrotron Light –the First Three Years

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Abstract

National Synchrotron Light Source II (NSLS-II) is one of the newest 3 GeV storage-ring synchrotron facilities in the world. It is designed and built with a 792 m circumference, 500 mA operating current, and a horizontal emittance of ~0.6 nm-rad with optimized DBA lattice (<http://www.bnl.gov/ps/>). Since the start of its user operations in July 2015, NSLS-II has rapidly ramped up its science capabilities and user programs. As of August 2018, NSLS-II

operates at 400 mA top-off, and has 26 beamlines in operations and 3 other beamlines under construction. In fiscal year that ended September 30, 2017, more than 1000 distinct users conducted their experiments at NSLS-II. This number is projected to increase to above 1300 in 2018.

The vision for NSLS-II is to develop world-leading scientific capabilities and leverage them to enable and conduct a broad range of high-impact, discovery class science and technology programs to address the critical scientific grand challenges in energy security, advanced materials synthesis and manufacturing, the environment, and human health. Working with the scientific community, NSLS-II has identified three science priority areas that will drive the research and development activities in the near term at NSLS-II: quantum and complex materials, operando chemistry and structural science, and multiscale structures and functions. In this talk, I will present the current status of our facility and beamlines, and our plans and initiatives to further expand our technical capabilities and enhance our science programs at NSLS-II.

NSLS-II is a U.S. Department of Energy (DOE) Scientific User Facility operated for the DOE Office of Basic Energy Sciences by Brookhaven National Laboratory under Contract No. DE-SC0012704.

Parallel Session 5 / 43

Interfacial Structure of Tailorable Nanocarrier Emulsions: X-ray and Neutron Scattering Approaches.

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The oil/water interface is crucial to many industrial systems, for example emulsions (food, cosmetics, drug delivery and others), chemical extraction (both aqueous to organic and the subsequent back extraction). Tailorable nanocarrier emulsions (TNEs) are a novel class of oil-in-water emulsions stabilised by molecularly-engineered biosurfactants that permit single-pot stepwise surface modification with related polypeptides that may be chemically conjugated or genetically fused to biofunctional moieties. The interfacial properties of such materials particularly when one component is on the nanoscale have a profound influence on biodistribution and stability as well as the effectiveness of sophisticated surface-encoded properties such as active targeting to cell surface receptors.

The target droplet size for the TNE is in the 100 -200 nm range with low polydispersity. Molecular scale characterisation of the liquid/liquid interface in such systems is challenging. It is nonetheless of prime importance in a variety of physico-chemical-biological areas both fundamentally and practically. We have simplified the approach to this system by beginning with x-ray and neutron scattering methodologies to studying the structure and molecular conformation at planar oil/water interfaces.

I will discuss our current work on related to TNEs for drug delivery. The TNEs consist of an oil in water emulsion where the interface is stabilised by a rationally designed single alpha helix peptide (AM1). To the AM1 stabilised emulsion a related four-helix peptide (DAMP4) is added. The DAMP4 can be linked to a range of biologically functional elements including antibodies or protein resistant molecules. The arrangement of the AM1 and DAMP4 at the oil/water interface and competition between the two species are important questions, the answers to which help to guide the TNE design. Furthermore, the presentation, conformation

and orientation of the antibody into the aqueous phase impacts upon the TNE design and ultimately activity.

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A Gas tight in situ electrochemical cell for the simultaneous measurement of high quality XAS and electrochemical data for low electro-catalyst loadings .

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A significant limitation in the development of efficient catalysts for renewable fuel technologies is the lack of understanding of the mechanisms of their operation and degradation under working conditions. Herein, we present a spectro-electrochemical cell for in situ XAS studies that have been optimised to simultaneously collect high-quality XAS and electrochemical data, particularly for very low sample loadings relevant to the study of electro-catalysts. By flowing electrolyte in a loop the cell stops bubble formation and the development of headspace and enables solutions to be added and removed without removal from the beamline platform. The cell is demonstrated to achieve exceptionally good detection limits of 10 ng per cm² and is liquid and gastight. The cell was benchmarked using two heterogenite-like, cobalt oxide, water oxidation ($2\text{H}_2\text{O} \rightarrow 4\text{H}^+ + \text{O}_2 + 4\text{e}^-$) electro-catalysts and a nickel oxide electro-catalyst. The cell was used to quantify a series of beam photo-mediated effects including photo-reduction and parasitic photo-deposition which are described in detail.

Parallel Session 14 / 45

Examining spider silk properties with SAXS/WAXS for biomimetic applications

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With toughness greater than Kevlar®, spider dragline silk is nature's greatest performing fibre. Accordingly, there is immense interest in generating new synthetic fibres that mimic its mechanical performance. Biomimetics is a growing new field that looks to nature for inspiration to synthesize new high performance materials and processes. Nonetheless, there is currently little cross disciplinary engagement between biologists and engineers, meaning most biomimetic programs are making slow progress. I have expanded my spider silk research program, in which I have investigated the ecological and evolutionary basis for spider dragline silk property variability, probing the nanostructural basis for silk mechanical property variability using SAXS/WAXS at Australia Synchrotron and mechanical performance testing techniques. I am now working with engineers and designers to develop fibre spinning technologies to produce synthetics for incorporation into a range of new practical smart materials and adhesives.

Parallel Session 6 / 46

Phase contrast X-ray imaging of the lung at birth

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The transition to newborn life after birth represents one of life's greatest challenges, yet for most of us we pass through this phase of our lives with only a brief cry that is greeted with much relief and joy by our expectant parents. Nevertheless, even in developed countries like Australia, we are much more likely to die on the day we are born than on any other day of our lives, until we reach the grand old age of 110. This simply reflects the extent and complexity of the physiological changes that must occur for the newborn to survive after birth. At birth, the airways are liquid-filled and so the lungs must aerate before they can become the sole organ of gas exchange and the resistance to blood flow through lungs must dramatically decrease. The latter enables the lungs to accept 100% of right ventricular output and allows the two circulations (pulmonary and systemic) to separate. As a result, the pulmonary circulation can work at a much lower pressure than the systemic (15 vs 100 mmHg), which is vital for respiratory function after birth. Lung aeration is not only vital for establishing the onset of pulmonary gas exchange, but is also vital for triggering the decrease in pulmonary vascular resistance, which in turn triggers the circulatory changes required for survival after birth. However, until recently, our understanding of the mechanisms controlling airway liquid clearance (=lung aeration) were restricted to an osmotic process that is slow and cannot account for lung aeration in premature infants. This lack of understanding greatly limited the clinical options available to assist infants who are unable to aerate their lungs at birth and therefore require assistance.

The ability of phase contrast X-ray imaging to visualise air liquid interfaces has greatly advanced our understanding of lung aeration at birth, enabling us to identify the mechanisms involved and to investigate treatments that can be used to facilitate this process. Visualising lung aeration in real time allowed us to demonstrate that transpulmonary pressure gradients, however they are applied, regulates the rapid movement of water out of the airways and into the surrounding tissue from where it is cleared. Discovering this mechanism allowed us to devise strategies that can be used to facilitate this process, which have been adopted into clinical practice. In addition, when combined with angiography, we were able to define the spatial and temporal relationship between lung aeration and the increase in pulmonary blood flow after birth. As a result, we identified a major mechanism responsible for the increase in pulmonary blood flow that was previously undescribed. More recently, we have used phase contrast X-ray imaging to investigate laryngeal function in the newborn and a rapid computed tomography approach to track lung liquid as it leaves the airways after birth.

In summary, the unique properties of synchrotron generated X-rays have allowed us solve many of the questions surrounding how we make the transition to new born life. For the last 50 years or more we have either had no idea or mistakenly thought other mechanisms controlled these vital processes, which has greatly limited our capacity to intervene and assist infants at birth. However, with our new understanding we are making good progress in improving the outcomes for all newborn infants that require assistance at birth.

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Mapping of the trace elements Cr and V by Synchrotron X-ray fluorescence microanalysis in Al-Zn-Si alloy coatings on steel substrates

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Al-Zn-Si alloys are widely used in industry as coatings over steel substrates and display a multilayered microstructure (steel substrate/intermetallic layer/coating overlay) that offers protection from corrosion. Partitioning of trace level Cr and V into Fe-Al-Si based intermetallic particles has previously been noted in the galvanizing coating bath. The coating layer's corrosion performance may also be influenced by microalloying with Cr and V even at trace levels. In this experiment, the trace element distribution of Cr and V in the cross section of Al-Zn-Si alloy coatings on steel substrates has been mapped by using synchrotron X-ray fluorescence microanalysis. The concentration of each element was obtained by averaging through the sample thickness, 30 μm , for a beam size 0.4 $\mu\text{m} \times 0.4 \mu\text{m}$. It was found that Cr was distributed as particles in between the intermetallic alloy layer and the coating overlay, as well as on the top surface when the concentration is below 100 ppm. In contrast, V was shown to form a continuous thin band in the intermetallic layer in the three investigated coating layers even at concentrations less than 100 ppm.

Parallel Session 9 / 48

Understanding a million dollar reaction - the mineral replacement of chalcopyrite by chalcocite - insights from in-situ XRD and in-situ XAS techniques.

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Australia is host to world's 2nd largest copper deposits - yet it's only the 5th largest copper exporter in the world. The copper industry in Australia faces significant challenges in mineral process owing to the complicated paragenesis of these copper deposits. Chalcopyrite (CuFeS_2) is among the most common Cu minerals. The replacement of chalcopyrite to chalcocite (Cu_2S) in a copper-rich aqueous medium at mild hydrothermal conditions is an emerging method in mineral processing to upgrade the chalcopyrite-rich concentrates while rejecting deleterious elements. Despite its potential as a cost-effective method to upgrade copper ores, the underlying reaction mechanism and kinetics of the mineral replacement of chalcopyrite by chalcocite remains poorly understood.

In fluid-mediated mineral replacement reactions, a comprehensive understanding of the reactions involves the careful observation of changes in the mineral phase (phase transitions, mineralogical changes) and the fluid phase (concentrations and redox state of dissolved components) during the reaction. To probe the mineral replacement of chalcopyrite by chalcocite we have performed two sets of in-situ experiments at two different beamlines at the Australian Synchrotron.

At the PD beamline we have carried out a series of in-situ XRD experiments to follow the reaction at high temperatures (180 °C –240 °C). The results revealed the reaction pathway as well the metastable reaction species generated in the reaction. The major findings from the in-situ XRD experiments include, i) observation of in-situ replacement of chalcopyrite by digenite-high/covellite in all experiments, ii) observation of szomolnokite ($\text{FeSO}_4 \cdot \text{H}_2\text{O}$) and djurleite ($\text{Cu}_{1.96}\text{S}$) as the metastable species during the mineral replacement reaction.

Pivoted on the observation from the in-situ XRD experiments we have been successfully awarded beamtime to study the redox evolution of the fluid during the replacement of chalcopyrite to chalcocite using our in-house high pressure - high temperature 'mAESTRO' cell for in-situ XAS spectroscopy at the Australian synchrotron. Combining the results from the in-situ XRD experiments (nature and relative proportions of solids) and the in-situ XAS experiments (Fe and Cu concentrations and oxidation state in the fluid during the reaction) - we'll be able to construct a coherent understanding of the reaction mechanism governing the replacement of chalcopyrite by chalcocite.

Parallel Session 2 / 49

Investigating Sulfur Speciation in Biological Samples Using Medium Energy X-rays

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The medium energy x-ray range that spans ~2-5 keV covers the K-edge of important biological elements P and S, as well as important biological ions, Cl^- , K^+ , Ca^{2+} . This energy range fills the "gap" between energies typically accessed at soft x-ray or hard x-ray beamlines. The energy range is also referred to as the "tender" or "squishy" x-ray range, but regardless the name, this energy range is important to biology!

The wide range of oxidation states in which sulfur can exist facilitates many biological processes, and the thiol disulfide redox switch is a classic example. In addition, highly oxidised forms of sulfur, such as sulfonic acids (+4) and sulfates (+6) have important roles in biology. Studying the chemical form of sulfur, "sulfur speciation" in biological systems is difficult. Few methods exist for direct detection of sulfur, and due to its redox active nature, sample preparation and assay protocols frequently modify sulfur oxidation state. For these reasons, x-ray absorption spectroscopy is an ideal technique, to directly investigate sulfur speciation in biological samples. In this presentation I will describe my work at international facilities of the last 5 years, using medium energy x-rays to access the sulfur K-edge (~2470 eV), to study sulfur speciation in a range of biological samples (brain tissue, cartilage tissue, and muscle tissue).

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Structural characterization of nanostructures formed by native neuropeptides using small and wide angle x-ray scattering

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Despite the fact that amyloid formation is typically associated with protein misfolding diseases, lately growing set of evidences have shown that amyloid formation can also take place under physiological conditions where they perform native functions of the host¹. Furthermore, it has been recently proposed that most of the peptide and protein hormones are stored in secretory granules in the human brain as amyloid-like cross- β -sheet-rich structures, including neuropeptides such as somatostatin and endorphins. In this context, we studied the self-assembly of three different neuropeptides, somatostatin-14, substance P and luteinizing hormone-releasing hormone (LHRH) in conditions similar to secretory granules. Here we report the use of synchrotron small and wide angle x-ray scattering measurements (SAXS/WAXS) to characterize nanostructures formed by the self-assembly of these peptides in the presence and absence of heparin. We show that as a function of concentration somatostatin-14 forms oligomers, rod-like (nanofibrils) and flat 2D structures (ribbons). In contrast, with heparin, somatostatin-14 self-assembled into heterogeneous mixture of aggregates². In the case of substance P, SAXS data analysis indicates that both in the presence and absence of heparin substance P molecules associate into hollow nanotubes in 150 mM NaCl with a 2D hexagonal packing. To best of our knowledge, none of the natural neuropeptides has been reported to form nanotubes up to date. On the other hand, neuropeptide LHRH self-assembles into nanofibrils in 150 mM NaCl with a 2D hexagonal packing exhibiting a lattice parameter of 7.7 nm. SAXS profiles obtained for lower concentrations of LHRH revealed the presence of oligomers and rod-like (nanofibrils) structures in solution. Indeed, WAXS data for all three neuropeptides indicate the presence of cross- β structure which confirms amyloid nature of these self-assemblies. Our findings support the fact that most of the peptide hormones/neuropeptides are stored as amyloid-like structures in secretory granules. In addition, this study introduces new versatile templates for fabrication of nanomaterials.

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Parallel Session 10 / 51

Protective Frameworks for Biomolecule Applications

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Biomolecules such as enzymes, DNA and other proteins are increasingly being used in various biotechnology and industrial applications. However, they rely on their structural complexity for activity and specificity [1] making them vulnerable to environmental factors such as temperature, pH, and solvents. Encapsulation of these molecules, especially enzymes, improves their stability and allows them to retain their activity, therefore increasing their utilisation in a range of applications such as industrial catalysis and biopharmaceutical delivery.[2]

We have been developing an encapsulation process using Metal-Organic Frameworks (MOFs) with inspiration from natural biomineralisation processes whereby molecular architectures are fabricated by living organisms to provide exoskeletal “shields” and structural support.

Biomolecules of interest such as proteins and enzymes are used as agents to initiate the encapsulation process by introducing them into aqueous solutions containing the MOF precursors (organic ligands and metal cations). The MOF self-assembles around the biomolecules, forming a protective shield, analogous to the exoskeleton of the sea-urchin. The resulting bio-composites, are then simply separated via centrifugation and subsequent washing and can be released upon change in pH.[3]

Using Small Angle X-Ray Scattering (SAXS) at the Australian Synchrotron, we have been studying the formation mechanisms involved with the biomineralisation process. In this presentation we will discuss the investigation into crystallisation during the assembly, washing and releasing processes. Understanding the material behaviour and stability of the metal-organic frameworks is crucial for applying these materials in industrial applications.

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Parallel Session 14 / 53

Development of Spectroscopic Protocols to Study the Relationship between Epicuticular Surface Chemistry and Flora during Stress and Ripening

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Expanding human populations require increased land use and increased efficiency of land use, which makes it important to mitigate the effects of environmental stress on native flora and crops. The wax coating on the surface of plant leaves (epicuticular waxes) holds important physiological functions to protect plants against environmental stress, for example, minimising water loss, UV protection, protection from disease, as well as acting as an anti-feedent. Studying the composition and distribution of epicuticular waxes on the surface of plant leaves can provide valuable insight into plant fitness and the presence of environmental stressors. Current methods to study plant waxes require extraction of the wax from the leaf surface. This approach reveals substantial insight into chemical composition of plant waxes; but, destroys valuable information relating to the spatial distribution of waxes on the leaf surface. The development of analytical methods that can directly image epicuticular waxes across the surface of plant leaves is therefore, sought after to complement existing bulk analyses. I will present initial work on the development, adaption and validation of direct spectroscopic imaging methods, specifically Fourier transform infrared (FTIR) spectroscopy, to enable investigation of the epicuticular wax distributions on plant leaves. This methodology may provide deeper understanding of how wax composition and distribution changes in response to altered plant physiology during environmental stress. Such information may be used to help monitor health and fitness of native flora populations, or assess fruit ripening processes.

Parallel Session 4 / 54

Industry Engagement at the Australian Synchrotron: Lessons learned

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It is important that Australia's landmark research infrastructure, including the Australian Synchrotron, is well-recognised, understood and, where relevant, utilised by industry for the economic and social benefit of Australia and all Australians. A dedicated Industry Engagement team aims to support Australian Industry to utilise synchrotron technology to problem-solve and innovate for the ultimate benefit of the community.

Working with industry clients, however, presents different challenges to working with the academic community and requires a different approach. In most instances, you will need to work with clients to help them articulate the problem they are trying to solve which is very different to presenting your own research and hoping it is of interest to a client.

This presentation will:

- Highlight key differences in approach to commercial vs academic engagement
- Explore ways to measure your efforts and track the progress you are making towards a successful project
- Examine how to manage client expectations and avoid getting off target
- Discuss how to raise awareness and understanding of technical capabilities to potential commercial clients
- Share ways to inspire businesses to explore applications and capitalise on the opportunities arising from our world class infrastructure
- Showcase existing case studies, demonstrating successful connections between science and industry

Parallel Session 5 / 55

Lyotropic liquid crystal phase behavior of various amphiphiles in ternary protic ionic liquid containing solvents

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Amphiphiles exhibiting micellar and higher order lyotropic mesophases in aqueous solvent environments have been extensively used in a broad range of applications such as detergents, sol-gel processes, biocatalysis, microencapsulation, nano-material synthesis and drug delivery [1, 2]. In recent years the use of non-aqueous solvents, or their binary/ternary mixtures, in self-assembly processes has received increasing attention due to their advantageous features, such as enhanced protection against hydrolysis of bio-active materials and hence, improved long-term stability of these compounds [3]. In particular, protic ionic liquids (PILs) are the largest and most tailorable class of non-aqueous solvents possessing the ability to support amphiphile self-assembly.

In this work, lyotropic liquid crystal phase (LLCP) behavior of the various amphiphiles including cetyltrimethylammonium bromide (CTAB), sodium dodecyl sulphate (SDS) and monopalmitolein (MP) as investigated in the ternary solvent system of water, ethylamine and nitric acid, where the stoichiometric acid-base composition corresponds to the well characterised PIL of ethylammonium nitrate (EAN). A total of 26 unique solvent environments

were used, covering the pH and ionicity ranges of 0-13.5 and 0-11 M, respectively. The effect of amphiphile concentration and temperature on the formation of LLCs was also determined. The LLCs in these solvent environments were studied using differential scanning calorimetry, cross polarized optical microscopy and small and wide-angle X-ray scattering. Neat water and EAN were used as reference environments for comparison. Phase diagrams were separately constructed for amphiphile concentrations of 50 wt% and 70 wt% between 25 °C and 75 °C, as it is given in Fig. 1 for CTAB representatively. LLCs were identified as micellar, hexagonal and cubic phases and were present from 35 °C in some of the solvent composition. Thermal stability and diversity of phases were found to be greater and broader in solvent compositions with excess ethylamine present. In acid-rich solvent combinations, some structural changes were observed due to the dramatic change in solubility of amphiphiles and its effect on the phase behavior was also examined.

Parallel Session 3 / 56

Element-specific contributions to the electronic structures of inorganic cesium lead halide perovskites revealed by resonant X-ray photoelectron spectroscopy

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Inorganic cesium lead halide perovskites (such as CsPbI₃) constitute an emerging class of high performance semiconducting materials, delivering promising device performances in solar cells and light emitting devices. Despite the rapid development of device optimisation, many fundamental issues remain open. One of the questions attracting huge attention is: do the constitute elements contribute equally to the electronic structures of perovskites? To address this, synchrotron-based resonant photoelectron spectroscopy (resPES) was employed to resolve the low binding energy region from -5 eV to 100 eV (valence band and shallow core levels) of a model inorganic perovskite material -CsPbI₃, which delivers high air stability and satisfactory solar cell performance. Taking advantage of the energy tunability provided by synchrotron, the incident X-ray energy was tuned around the absorption edges of the component elements, including Cs+, Pb2+, I-, and Br-. With the high-resolution photoelectron spectroscopy at the low binding energy region from -5 eV to 100 eV, it was found that the photoemission cross-sections at the shallow core levels of the constitute elements and valence band region follow different trends when the incident X-ray energy was tuned toward the absorption edges of different elements. Intriguingly, additional photoemission bands emerged around the valence band region when Cs+ was selectively excited, while the X-ray excitations around other constitute elements do not show such extra photoelectron bands. With the aid of resPES, direct experimental evidence was obtained to support that different constitute elements contribute, in a dissimilar way, to the electronic structure of inorganic cesium lead halide perovskites. This exciting work is believed to be able to experimentally verify or disapprove the proposed electronic structure models of high performance perovskite materials.

Parallel Session 11 / 57

Demonstrating an electric field-tuned Topological Phase Transition in ultra-thin film Na₃Bi using ARPES and STM

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The electric field induced quantum phase transition from topological to conventional insulator has been proposed as the basis of a topological field effect transistor. In such a device an electric field can switch ‘on’ the ballistic flow of charge and spin along dissipationless edges of the two-dimensional (2D) quantum spin Hall insulator [1], and when ‘off’ is a conventional insulator with no conductive channels. Here we demonstrate that few-layer Na₃Bi, normally a 3D Topological Dirac semimetal in its bulk [2][3], is a viable platform for realising such a topological transistor at room temperature.

Using scanning tunnelling microscopy (STM)/spectroscopy (STS), supported by complementary angle-resolved photoelectron spectroscopy (ARPES), we observe that mono- and bilayer Na₃Bi behave as effectively 2D topological insulators with bulk bandgaps >400meV.

Further, we demonstrate that upon the application of an external electric field [4] with an STM tip, a topological phase transition to trivial insulator with conventional gap greater than 100meV can be reversibly induced. The large bandgaps in both the conventional and quantum spin Hall phases suggest that Na₃Bi is suitable for room temperature topological transistor operation.

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Parallel Session 13 / 58

Periodic Density Functional Theory for the Prediction of Terahertz Spectra

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Accurate simulation of low-frequency fundamental and lattice modes, as obtained from terahertz and far-infrared spectroscopy of crystalline molecular solids, has long proved difficult. Long-range intermolecular interactions in the solid-state are notoriously difficult to model, resulting in deviation from experimental frequencies obtained from thin films and aerosols. In this talk, we present a series of calculations using the periodic DFT code CRYSTAL17 now installed on the parallel ASCI environment. These studies have yielded highly accurate simulated far-IR spectra (frequencies and intensities) for comparison to molecular crystal spectra measured at the THz and Far-IR beamline. The results have allowed the assignment of lattice vibrations pertaining to astrophysical ice and forensic samples, many for the first

time. Potential for the coupled theory-to-experiment method applied to molecular organic framework (MOF) and pharmaceutical systems will be discussed.

Parallel Session 3 / 59

Strategies for morphological control in organic photovoltaic devices

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While inorganic solar cells enjoy a level of success, high manufacturing costs and bulky modules limit their broad applicability. In contrast, organic photovoltaic (OPV) devices employ flexible thin-films of organic small molecules or polymers as the photoactive layer. OPV could provide a low-cost alternative to traditional PV, and a system of solution processing coupled with roll-to-roll printing of lightweight, flexible solar cells is envisioned.

One complication of organic photovoltaic technology is the heavy dependence on the active layer morphology obtained by the component donor and acceptor materials. The optimal morphology will maximize the donor/acceptor interface to insure free charge generation but must also conduct charges efficiently, which requires pure and crystalline percolation pathways oriented relative to the electrodes. These qualities are often at odds with each other, and their interplay and the resultant photovoltaic performance are of great importance to the OPV field.

Here we discuss two routes we have taken towards understanding and controlling morphology in OPV thin films. In the first system, the modulation of bulky substituents in nematic liquid-crystalline small molecules was investigated as a means of controlling their orientation, phase separation, and crystallinity in films. In these novel p-type materials, it was demonstrated through depth-dependent grazing-incidence wide-angle X-ray scattering (GIWAXS) studies that the degree of crystallinity and the distribution of crystallite orientations throughout the films is determined by solubility and can have a large effect on device performance. In the second system, we have employed a bottom-up approach to morphological control by covalently linking the donor and acceptor materials to form amphiphilic block copolymers. Using GIWAXS in conjunction with resonant soft X-ray scattering, these block copolymers were shown to spontaneously self-assemble into well-defined and crystalline domains in thin films. The results of both systems demonstrate the value of careful molecular design for the morphological control of organic photovoltaic active layers.

Parallel Session 10 / 60

Stiffness of modified collagen fibril structures manipulated by moisture content

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Flexibility and strength are both desirable characteristics in skin derived collagen materials, like leather. Dehydration of skin during production of leather transforms tissue into a stiffer material. The hydration state is a key parameter in leather production controlling the material strength and flexibility. The structural basis for flexibility in leather was investigated and the moisture content varied. Mechanical properties of collagen are known to change with moisture content. Leather produced by tanning under strain increases the leather strength through increased fibril alignment but also reduces flexibility. Small angle X-ray scattering was used to determine collagen structures and three point bend tests to measure flexibility. Results show how the interplay between moisture content and fibril alignment can be used to optimize properties in leather.

Parallel Session 10 / 61

Using SAXS/WAXS to determine the influence of collagen structure on material properties within acellular dermal matrix materials.

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Collagen tissues such as skin and pericardium are remarkably strong and malleable. These physical characteristics, along with the biocompatibility benefits of being a natural product, make collagen tissues an excellent source material for making surgical patches and implants. Our studies have involved investigating the natural collagen structure within acellular dermal matrix (ADM) materials derived from skin of various species to see how collagen structure can affect material properties and how collagen is able to react to strain. Synchrotron based small angle X-ray scattering was the main investigative technique. We were able to show that collagen fibril alignment in the planar direction results in a stronger ADM material, and when under tension, collagen fibrils realign in the direction of the force before stretching themselves. This behaviour was demonstrated by an initial increase in orientation index upon strain, followed by an increase in D-spacing and decrease in intermolecular spacing and fibril diameter at greater strains.

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Using bacterial biosensor proteins to evaluate SAXS-based screening

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The entire arsenal of biophysical tools are currently being employed as means discover potential pharmacological leads. In this context, small-angle X-ray scattering (SAXS) offers useful complementary information to initial high-throughput assays based on thermodynamics, protein function, and other modes. However, it has hitherto been under-utilised as a screening tool, and we believe one factor is a lack of an appropriate benchmark that can be tested across different beamlines and benchtop sources, and used to formulate expectations for prospective users on aspects of material consumption, throughput and precision.

We will report the state of progress in evaluating bacterial periplasmic binding proteins as a candidate for benchmarking. A set of amino-acid uptake proteins HisBP, DEBP, GlnBP, and LAOBP was chosen to cover affinities ranges between nM to mM, and screened at ESRF Grenoble at concentrations between 0.5-4 mg/ml. The twelve-point SAXS-titrations are used to predict the selectivity profile of respective proteins, and compared against values derived from ITC and NMR.

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X-ray Computed Laminography on the IMBL

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For parallel beam x-ray computed tomography (CT) such as performed on the IMBL a cylinder is the ideal sample geometry. This shape minimises the mean change in x-ray attenuation as the sample is rotated during projection image collection, and makes best use of the detector field of view.

However, producing a cylindrical sample is not always possible or desirable. Non-ideal sample geometries with high aspect ratios such as plates or disks, are difficult to image using standard CT protocols. However, they can be imaged by using a slightly different technique. The sample rotation axis is deliberately tilted with respect to the plane of the beam so x-ray paths through the sample are never parallel with the plate surface during the rotation. This technique is called Computed Laminography (CL) and is currently being deployed on IMBL. Here we show a summary of instrumentation and software development for IMBL CL, and preliminary results from some aerospace material imaging.

Parallel Session 15 / 64

In situ small-angle x-ray scattering measurements of ion track etching in polymers

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When a highly energetic heavy ion passes through a target material, the damaged region left in its wake often exhibits preferential chemical etching over the undamaged material. This etch-anisotropy can be used to create very high aspect ratio channels (pores) of up to tens of microns in length, with pore diameters as small as several nanometres. Membranes formed by this method are ideal for many advanced applications including ultra-filtration, bio- and medical sensing, nano-fluidics, and nano-electronic devices. The shape of the etched pores can be cylindrical, conical or double conical, depending on the etching conditions. One major advantage of the technique is the ability to generate arrays of pores that are highly parallel with extremely narrow size distributions.

The aims of this research are to develop a detailed understanding of the track etching process and the etching kinetics in polymers by performing in situ small angle x-ray scattering (SAXS) measurements during the etching process. The SAXS measurements were carried out at the Australian Synchrotron in Melbourne, Australia. Investigating the influence of etching parameters and pore areal density on nano-pore formation enables the controlled fabrication of nano-pore membranes with size and shape-specific pores. For our experiments we used 12 μm thick foils of PET and 20 and 30 μm thick polycarbonate (PC) foils, irradiated with 2 GeV ^{197}Au -ions at the GSI UNILAC in Darmstadt, Germany. The irradiated material was subsequently etched in diluted sodium-hydroxide (NaOH) at several concentrations and temperatures. The etching was conducted in a custom-built sample environment while performing the SAXS measurements in transmission mode to determine the track etch rate as a function of etch time. These in situ scattering images were analysed using a batch fit method to determine the pore size as a function of etching time. An example of a transmission SAXS scattering image of cylindrical pores in PC is shown in Fig. 1. The results of the study indicate that the track etching behaviour is strongly influenced by temperature and concentration of the etchant, whereas the pore areal density only has a small effect on the etch rate. This allows the calculation of activation energies for radial etching of PET and PC depending on their pore areal densities. The etch rates for PC are largely linear, however PET seems to have two etch rates indicating a damaged halo.

Parallel Session 10 / 65

Insights into flash-nanoprecipitated drug solubilisation during in vitro digestion in milk using the SAXS/WAXS beamline

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Strategies to administer drugs in a low cost and an effective manner are priorities to global health and many efforts have been made to re-formulate drugs that are highly lipophilic and

have high variations in their oral bioavailability. One example of such drug is clofazimine, which has been recently identified as a potential new drug to treat cryptosporidiosis, the second leading cause of diarrhea in infants.¹ Design of formulations for fast acting treatment of cryptosporidiosis with superior oral bioavailability to the commercially available clofazimine (Lamprene®) is therefore necessary and recent studies have demonstrated that flash-nanoprecipitated clofazimine nanoparticles could provide faster kinetics of drug release compared to Lamprene®.^{2,3} We herein investigate the solubilisation behaviours of clofazimine nanoparticles, taking into account intestinal digestion to more closely mimic the in vivo setting by tracking the evolution of diffraction peaks from crystalline drugs using the SAXS/WAXS beamline.⁴ The effects of fat in milk and infant formula on the solubilisation of clofazimine were also studied to better understand potential food effects in paediatric populations. Our results confirmed that clofazimine exhibits a fat-dependent solubilisation and that the solubilisation of drug from flash-nanoprecipitated clofazimine nanoparticles is faster than Lamprene®, which is highly desirable for treating *Cryptosporidium* infections in the small intestine.

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****Exploration of Multistep Kinetics of Dehydroxylation of Antigorite through In-situ Synchrotron PXRD****

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Mineral carbonation represents one of the most promising techniques for safe and permanent sequestration of CO₂ on a global scale, at a rate of Gigatonnes/year. The abundance and geographical distribution of serpentine minerals distinguish them as a favourable raw material for mineral carbonation. Although carbonation of serpentinite rocks (Mg-silicates) appears thermodynamically favourable, slow dissolution kinetics hinders the commercialisation of this process for large-scale CO₂ sequestration ^[1]. A practical procedure for improving the chemical reactivity of these mineral resources involves dehydroxylation via heat treatment. Dehydroxylation eliminates the structurally-bounded hydroxyl group, resulting in partial amorphisation of the structure and higher reactivity of the mineral towards subsequent carbonation ^[2]. However, this increase in reactivity is achieved at the expense of an additional energy cost ^[3]. Therefore, understanding the mineralogical changes and estimation of kinetic parameters for dehydroxylation of serpentine minerals remain crucial for providing an energy efficient solution for large-scale implementation of mineral carbonation. This study employed in-situ synchrotron

powder X-ray diffraction (S-PXRD), prograde heating of antigorite (one of the polymorphs of serpentine minerals) samples at nominal rates of 2, 4 and 6 °C min⁻¹ at temperatures between 25–1000 °C for detailed observation of the mineralogical changes and applied the isoconversional formalism to estimate the kinetic parameters governing the heat treatment. The result shows that antigorite remains stable up to 573 °C; dehydroxylation above this temperature converts it into either amorphous phases or disordered form. On further heating, these resulting phases transform to forsterite (700 °C), with enstatite formation (837 °C) occurring only after complete dissociation of antigorite. The isoconversional kinetic modelling revealed the variation of activation energy (E_a) from 110 to 210 kJ.mol⁻¹ with reaction progress (α), elucidating the multi step features of the dehydroxylation of antigorite. Based on the variation of E_a , dehydroxylation of antigorite can be divided into four stages; i) condensation of adjacent hydroxyl group to form water or liberation of H⁺/OH⁻ ions (E_a decreased from 110 to 95 kJ.mol⁻¹ for α 0.25), ii) formation of disordered or amorphous phase hindering the bulk diffusion of water/ ions (E_a increased from 95 to 170 kJ.mol⁻¹ for α 0.25–0.5), iii) formation of newly formed boundaries e.g. forsterite and talc-like phase supports diffusion (constant E_a at around 170 kJ.mol⁻¹ for α 0.5–0.8), and iv) difficulty in diffusion due to break down of final dehydroxylation (E_a increased from 170 to 210 kJ.mol⁻¹ for α > 0.8). This work explores the potential of in-situ S-PXRD to determine the underlying heterogeneous kinetics, and to resolve the formation of individual mineral phases during dehydroxylation of serpentine minerals.

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Examination of the microstructure of the plastic layer using synchrotron micro CT

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Coke is formed in industrial coke ovens when a coking coal undergoes a plastic transformation during pyrolysis. During heating some components of the coal (vitrinite macerals) melt to form a viscous zone called the plastic layer. The plastic layer is a bubbling foam like material consisting of the molten vitrinite, solid coal components (inertinite macerals) and gases generated by the decomposition of the coal. Further temperature increases cause the plastic layer to solidify through cross-linking reactions locking in the structure of the final coke. The plastic layer moves from the heating walls to the centre of the coke oven over a period of about 20 hours. Physical properties of coke are linked to its microstructure which is largely determined by the behaviour of the plastic layer. The internal dynamics of the plastic layer are poorly understood and clearly better understanding will lead to a greater ability

to predicting the coking behaviour of coking coals in a coke oven. It has been known that the plastic behaviour is associated with properties of coking coals. Therefore, it is critical to understand a correlation between coal properties and plastic behaviour during the coking process. In this study, a micro-CT at the Imaging and Medical Beamline (IMBL) of the Australian Synchrotron was used to investigate a variety of morphological characteristics of both the plastic layer samples formed under practical heating conditions using a 4kg coke oven (University of Newcastle). The micro CT images showed regions consisting of coke with fine and compacted pores and fissures, visible plastic layers exhibiting high porosity and loose coal as discrete coal particles. The image analysis of the cross section CT images allowed the observation of changes in porosity of the plastic layers. A selection of six Australian coals for the micro CT imaging covered a wide range of vitrinite reflectance and maceral concentrates. The examination of these coals allowed the investigation of the effect of the coal properties on different physical changes of the plastic layers. As the direct imaging analysis allowed research to directly measure the physical structure of the plastic layer without damaging, the samples were subjected to further analyses which were chemically informative on the same samples. The combination of the analytical techniques provided a fundamental understanding of the underlying mechanism of the plastic layer.

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Chlorine-induced orientation transformation for systematic grain orientation control in hybrid perovskite thin films

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Hybrid organic-inorganic perovskites with appealing optical and electronic properties have attracted significant interest for photovoltaic applications. Conventional perovskite film fabrication methods had successfully prepared films with uniform morphology, good crystallinity and large grains with low grain boundaries and trap densities, leading to a remarkable power conversion efficiency over 23.3% in perovskite solar cells. However, little attention has been paid to understanding microstructure evolution and control of crystal orientation (texture) in perovskite thin films. This is an important topic since microstructure is intuitively responsible for electronic and photovoltaic properties. Controlling the microstructural arrangement of perovskite thin films offers a strategy to tailor these properties toward their single crystal counterpart for high device efficiency and stability. Here, the manipulation of microstructure orientation within methylammonium lead triiodide (MAPbI₃) perovskite polycrystalline films via methylammonium chloride (MACl) post-treatment is studied. It is found that the crystal planes stacking along tetragonal <110> orientation can be improved by post-treating the randomly oriented MAPbI₃ films with increasing MACl concentration. With two-dimensional synchrotron-based grazing incidence wide-angle X-ray scattering analysis, an in-depth understanding of orientation transformation mechanism was provided. We demonstrated that the transformation of orientationally disordered polycrystalline films to highly orientated films with the tetragonal <110> direction oriented perpendicular to the substrate was templated by a chlorine-containing intermediate phase. The resulting highly oriented perovskite film exhibits micron-scale grain size with vertical grain boundaries, as well as low charge carrier recombination and efficient charge carrier transport. As a result, an improvement in solar cell device efficiency and stability was achieved. Our findings highlight the importance of microstructure and crystal orientation control for further understanding perovskite thin film properties and optimizing perovskite solar cell performance improvement.

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High throughput synthesis and characterization of Protic Ionic Liquids (PILs)

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Due to the range of cation-anion combinations that exist for Protic Ionic Liquids (PILs) synthesis, more efficient methods are needed. The ability to quickly categorize and determine protein stability is what is most desired. Using an automated high-throughput approach, 48 brønsted acid-base combinations were used to prepare and dry a list of PILs. The brønsted acid-base combinations consisted primarily of alkyl amines paired with alkyl carboxylic acids. Automation was used in the form of a Chemspeed robotic synthesis platform. Its implementation was used to increase workload and make the high-throughput approach more viable.[1] Visual screens were developed and used to determine which acid-base pairs led to PIL candidates, as well as their surface tension, viscosity and melting points. The water contents obtained using Karl Fischer coulometric titration indicated noteworthy differences in the drying rate between the different PILs. The liquid nanostructure of the PILs were determined using synchrotron small and wide angle X-ray scattering (SAXS/WAXS) at the Australian Synchrotron. The results showed that both the alkyl chains on the anion and cation contributed to the overall correlation distance. Maximum correlation distances were obtained when there was an alkyl chain present on only one ion. Additional work is underway to extend the library of PILs made in this manner to a broader range of cations and anions. These have been selected as ions which are relevant for ongoing protein stability studies and amphiphile self-assembly. The automated synthesis of solvent libraries will be used for screening protein stability, including using the SAXS/WAXS beamline for protein structural information. This will be used for understanding protein-solvent molecular interactions, and in designing solvents for biological molecules.

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Galectins Targeted Therapy Using Structure-Based Inhibitor Design

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Galectins are a family of -galactoside-specific lectin proteins with a highly conserved carbohydrate recognition domain (CRD). Galectins have been distributed in a varied range of animal species; 15 mammalian galectins have been identified, 12 of which are in human. Galectins are found both in the intracellular and extracellular environments. The imperative roles of galectins as mediators of cell-cell and cell-matrix interactions in tumor cell metastasis, angiogenesis, immune and inflammatory response makes them promising candidates for cancer therapy and drug targeting. This study investigates galectin-8 that belongs to the “tandem repeat”, which can simultaneously bind different carbohydrates through two CRDs in the protein structure. Interestingly, design of specific inhibitors aids to probe the

potential and specificity of each CRD in glycan binding and regulate their activity. For this purpose, the recombinant proteins of two CRDs: galectin-8 N-terminal CRD (galectin-8N) and galectin-8 C-terminal CRD (galectin-8C) were produced in active forms using heterologous expression system and biochemical process. In the following, the analytical binding assays for the generated proteins revealed a specific potential binding for galectin-8N to 2-O-Methyl- β -D-N-acetylneuraminic acid (Neu5Ac 2Me), as the sialic acid derivative, compared with galectin-8C. To achieve the three-dimensional binding conformation structure, galectin-8N in complex with Neu5Ac 2Me, X-ray crystallographic studies have been recruited which enables us to investigate how the protein interacts with the ligand, where it is bound on the protein, and what orientation it adopts, towards design potential inhibitors from sialic acid-based derivative molecules.

Parallel Session 11 / 71

ARPES - A Toolbox for Surface Discoveries

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Angle Resolved PhotoEmission Spectroscopy (ARPES) is a widely used technique for the investigation of the electronic structure of materials and can also be used to study many-body interactions such as electron-phonon couplings. The characteristic that separates ARPES from other surface science techniques is that it enables the direct visualisation of the electronic structure.

In this talk, I will introduce the technique and provide examples of the type of measurements facilitated by ARPES. The focus of these examples will be on 2D materials.

A short overview of research on other light sources, as well as the emerging possibilities on the newly installed ARPES beamline at the Australian Synchrotron will be discussed.

Parallel Session 4 / 72

Working with industry - from the scientist's perspective

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Scientists live in an environment of other scientists, technical journals and complex equipment and often esoteric scientific interests. The economy, which generates wealth for the country, is run by industry people that are less interested in the curiosity of scientific discovery and more interested in solving practical problems and creating new products and processes. Governments are understandably interested in the huge investment in science leading to economic benefits in timescales that are measured in years not multiple decades. Therefore there is increasing pressure for scientists to address issues that may lead to higher value industrial outputs. One obvious way to facilitate this is to get scientists talking to industry and creating a connection between research and industry. Some grant applications require this. There are both advantages and risks in working with industrial partners. This talk will give a personal account of many years of working with industrial partners with some good experiences and some not so good experiences with observations of how to make it a rewarding scientific exercise and to avoid some of the potential pitfalls.

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ASCI - Remote Analysis for users

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The Australian Synchrotron Computing Infrastructure (ASCI) offers users a service to analyse their data from their home institute without having to copy their experimental data or install analysis software locally.

Australian Synchrotron users can start and connect to their own desktop environment with a web-browser, find their analysis applications, and access their experiment data.

This service is currently available for users of the IMBL, XFM, PD, IRM and THz beam-lines.

Parallel Session 11 / 74

Understanding the effect of thionation on naphthalene diimide using first-principles predictions of near-edge x-ray absorption fine structure spectra

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Conjugated organic materials, such as semiconducting polymers and small molecules, have shown great potential for electronic applications, boasting favorable properties such as being mechanically flexible and having low processing temperatures. Additionally, the organic components comprising semiconducting polymers and small molecules are interchangeable, resulting in a high degree of synthetic control over the electronic properties of conjugated semiconductors. For example, it has recently been shown that an increasing degree of thionation –the replacement of oxygen atoms with sulfur atoms –results in a systematic shift in the NEXAFS spectra of naphthalene diimide (NDI)-based molecules. While such changes are systematic, it is difficult to directly connect the changes to the measured C 1s to π^* manifold to the electronic properties of the molecule since the NEXAFS spectrum obtained contains transitions from carbon atoms in different environments and hence different core levels. In this study, we have used a first-principles approach, the eXcited Core Hole (XCH) model, in order to resolve individual atomic contributions to the NEXAFS spectrum of a series of NDI molecules with increasing thionation. The simulations yield an excellent correspondence with the experimentally measured NEXAFS spectra. Furthermore the calculations of both the x-ray absorption spectra, as well as the neutral and excited molecular orbital density distributions, indicate that the sulfur substitution decreases the core level shift of the molecule, lowering the energy required to excite an electron from a core level. The simulations also reveal how changes in the symmetry of the molecule with thionation affect the resulting molecular orbitals and hence electronic transitions. The successful application of computational methods to explain the rich fine structure observed experimentally thus enables direct connection between measurement and the underlying molecular transitions. Such insights are important for underpinning the application of NEXAFS spectroscopy and related soft x-ray techniques in characterizing the microstructure of organic semiconductor films, which in turn are being used to optimize the performance of organic electronic devices.

Parallel Session 9 / 75

Shining a light on Jarosite alteration and stability using synchrotron microdiffraction and imaging techniques.

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Jarosites and related minerals are of great importance to a range of mineral processing and research applications. They are used in the removal of iron species from smelting processes; they occur in metal bioleaching systems, and in the desulphurisation of coal; they are present in acid mine drainage environments.

There has been a recent resurgence in interest in jarosite and associated minerals since their detection on Mars by the MER rover Opportunity. In this context, the presence of jarosite has been recognised as a likely indicator of liquid water at the surface of Mars in the past and it is hoped that their study will provide insight into the environmental history of Mars.

Acid sulfate soils cover large areas of the Australian coastline and are likely to be a major constituent of the Martian environment. The oxidation of acid sulfate soils, coupled with potential release of heavy metals and acidic groundwaters, can have serious consequences for fragile ecosystems. Understanding these sediments will provide insight into the biogeochemical processes that affect the lifetimes of transient mineral species on Earth, and may be used to better understand soil acidification, contaminant mobility at sites affected by acid and metalliferous drainage, and even constrain past weathering and putative biosignatures on Mars.

Knowledge of the behaviour of jarosite minerals under the actual conditions that they are found in is crucial to understanding their potential environmental impacts on both Earth and Mars. To this end, we are engaged in a program to study the formation, stability and alteration of jarosite minerals using a complementary suite of in situ synchrotron and neutron techniques.

In this contribution we discuss the results of parallel neutron and X-ray imaging at OPAL and the Australian Synchrotron, combined with synchrotron microdiffraction to map the mineralogy and structural relationships within naturally occurring jarosite nodule handspecimens formed from hydrothermal alteration.

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X-ray Absorption Spectroscopy Study of Multinary Metal Chalcogenides for Electrochemical Energy Storage

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Multinary metal chalcogenides are promising anode candidates for high-performance rechargeable batteries that are urgently needed for the booming electric vehicles and portable devices, due to the synergistic effects of the electrochemically active multi-components. To understand the nature of their excellent performances, we used ex-situ X-ray absorption spectroscopy (XAS) to detect the variation of local coordination environment around Sb element

in Sb₂Se₂Te, and Sn/In element in SnIn₄S₈, when they were applied as anodes for lithium-ion batteries (LIBs) or sodium-ion batteries (SIBs). It demonstrates the metal chalcogenides possess a lithiation/sodiation mechanism that involves an original conversion reaction with obvious valence changes, followed by an alloying reaction. The local environment relating to the reversibility of these electrodes attributed to the outstanding electrochemical performances was also identified by XAS. We concluded the difference of the spectra of pristine and fully charged electrode (Sb₂Se₂Te in LIBs or SnIn₄S₈ in SIBs) comes from the incomplete lithium/sodiation extract and/or formation of distinct Sb/Sn/In environments. But when SnIn₄S₈ electrode was applied in LIBs, the spectrum of fully charged electrode was almost identical to that of the pristine electrode, demonstrating the excellent reversible lithium storage of the electrode.

Parallel Session 8 / 77

Preparations for the first veterinary trials of synchrotron radiation therapy

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The aim of our research is to undertake the first large animal trial of Microbeam Radiation Therapy (MRT) at the Australian Synchrotron to treat spontaneous cancers. The Imaging and Medical Beamline (IMBL) at the Australian Synchrotron is one of only four facilities in the world expressly designed to enable the clinical application of synchrotron radiotherapy for cancer patients. There are new robotic positioning systems recently installed at the IMBL now which are capable of positioning large animals and humans in the synchrotron beam for radiotherapy purposes. We will use this new capability and our existing medical physics research program to treat canine tumours with synchrotron radiotherapy. This has never been attempted before and we anticipate this proof-of-concept work will directly lead to clinical trials in humans for certain unresponsive, or recurring cancers where conventional radiotherapy has failed.

Synchrotron MRT is an experimental form of radiotherapy that is fundamentally different to conventional radiotherapy (CRT) [1, 2]. There is emerging evidence that synchrotron MRT is more effective in destroying tumours than CRT and has fewer side effects than CRT [3-6]. There is therefore potential for MRT to significantly improve outcomes for cancer patients. The long-term aim of our project is to translate MRT to a clinical reality. At present, MRT can only be performed using a synchrotron, which is not achievable at hospitals. As a pre cursor to clinical trials, we propose veterinary trials of synchrotron radiation with real, spontaneous cancers in dogs in close collaboration with our veterinary colleagues.

Our aim in 2019 is to plan and treat approximately 9 live dogs with spontaneous tumours. Our preferred, initial target will be skin tumours or bone tumours in the legs of the dog. These tumours are easy to locate and position in the synchrotron beam. For this pilot study, we will select small tumours that are located at shallow depths in order to maximise the dose coverage. We will do a simple dose escalation ('3+3 study') whereby we irradiate 3 dogs with a low dose (e.g. an integrated dose of 8 Gray in a single fraction) followed by an approximately 20% increase in the dose to 10 Gy for the next 3 dogs. We will increase to 12 Gy for the next 3 dogs if the acute radiation toxicity is minimal.

We will consider the project a success if we can safely and verifiably deliver a low dose (palliative dose) of synchrotron radiation to live (sedated) dogs using the robotic couch at the IMBL. If this pilot study is successful we will have made major steps towards initiating a new radiotherapy paradigm with synchrotron radiation; the significance of such an outcome for cancer patients everywhere cannot be overstated. There are some brain and lung cancer patients for example who have failed current treatments and have few if any treatment options

available to them. Synchrotron MRT may offer hope to these patients. Our group has over 10 years' experience of Synchrotron MRT experiments using mouse models of healthy and malignant tissue. We now want to progress from our mouse model work (with artificial tumours) to larger animals with real, spontaneous tumours (e.g. pet dogs). Such work will be an invaluable proof of concept before we attempt MRT in human cancer patients.

In recent years, we have made tremendous progress towards realising our goals, most notably in the medical physics area. We have commissioned a computerised Treatment Planning System [7], completed a protocol for measuring the absorbed dose from the synchrotron radiation [8, 9], and developed sophisticated image-guided radiotherapy protocols [10]. Our work is at the stage now where we can plan and treat a dog on the robotic positioning system on the IMBL. In the radiobiology field, we have systematically characterised the toxicity of MRT for a range of treatment sites which is crucial for choosing safe doses for our proposed veterinary trials [11]. In recent work (April & July 2018) we planned and irradiated a dead lamb and dog with conventional (uniform) and microbeam radiation fields to test the physical components of the beamline.

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Parallel Session 1 / 78

Can you trust your data? Sample integrity and radiation exposure

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With increased resolution and extra dimensional imaging, an increased radiation dose is delivered to the specimen. After some point, the integrity of the specimen will be compromised, and therefore so will the data. Here we present a method for assessing the radiation induced changes in the specimen, taking into account variations in noise and resolution between measurements. We apply this method to X-ray fluorescence microscopy of the nematode, *Caenorhabditis elegans*, providing data that sets elemental specific limits for an acceptable radiation dose.

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Status of the Fast NEXAFS user system for the Soft X-ray beam-line

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The soft X-ray beamline is commissioning the latest system designed to allow users to take specifically NEXAFS data. The pressure requirements and the training levels required to be able to use the system have been significantly reduced, in comparison to the standard UHV user system. In future it will be renamed the high throughput NEXAFS system, some results are presented as to its current operational condition. Users will be scheduled to run on this system from February 2019.

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Dosimetry of Image Guided Microbeam Radiation Therapy

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Synchrotron Microbeam Radiation Therapy is a promising new pre-clinical treatment modality for otherwise inoperable cancers. However, the absence of effective quality assurance procedures presents a barrier for clinical implementation. In this study, some of these challenges are addressed through performing image-guided Microbeam Radiation Therapy on rats and small-animal phantoms using tools currently available at the Australian Synchrotron Imaging and Medical Beamline. High resolution computed tomography is used for treatment planning and co-registered to planar x-ray images enabling treatment accuracy to within ± 0.3 cm. Dosimetry performed during the imaging process verifies radiation exposure to be appropriately low with a surface dose due to the pCT imaging and planar x-ray processes of 8.63 ± 0.24 cGy and 2.8 ± 0.4 mGy respectively.

Parallel Session 8 / 81

High resolution fibre-optic dosimetry: towards a MRT quality assurance device

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Synchrotron microbeam radiation therapy (MRT) is a novel external beam therapy under investigation for its application in the treatment of brain tumours. Key characteristics of these x-ray microbeams is their high flux, high spatial fractionation and large dose rates. For clinical quality assurance, the dose rate in the microbeams (peaks) and between them (valleys) must be measured accurately. We present the progress we have made improving a scintillator fibre optic dosimeter design to be applied to MRT quality assurance. From a one-dimensional spatial resolution initially of 100 μ m, we have now achieved microbeam measurements with a 10 μ m dosimeter probe. Challenges with these devices include low sensitivity due to the small scintillator volume optically coupled to the fibre optic. The high dose rate of MRT partly overcomes this issue but remains a challenging area with smaller probe sensitive volumes. There is also a 20-30% over-response at low depths when compared to dose readings with ionisation chamber, consistent across all resolution probes tested. The works presented have demonstrated the incremental improvements in the scintillator fibre optic dosimeter and the achievability of its application in MRT. The probe has many desirable qualities, such as water-equivalence, ease of manufacture and relative inexpensiveness compared to other dosimetry devices. We anticipate that this work can lead to a commercial QA dosimetry device in the future.

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X-Tream dosimetry of synchrotron radiation with the PTW microdiamond

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In this study, we demonstrate that the X-Tream dosimetry system developed by the Centre for Medical Radiation Physics can be used with both commercial and non-commercial dosimeters. A custom adaptor was constructed to connect the PTW microdiamond to the X-Tream DAQ. This allowed for the measurements of continuous depth dose curves, broad beam and microbeam profiles. The X-Tream system enables real time measurement of the instantaneous dose rate of synchrotron generated radiation with the PTW microdiamond. Finally, measurements undertaken in this study demonstrate that the X-Tream system is able to mitigate the alignment issue that has previously hampered the uptake of the PTW microdiamond in clinical QA for small field dosimetry.

Parallel Session 1 / 83

Chemical Crystallography at the Australian Synchrotron MX Beamlines: an update

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The recent deployment of a Dectris 16M Eiger detector on MX2 has changed the 'standard' data collection protocol for CX work to a new shutterless 360° oscillation yielding 3600 frames in 36 seconds. This step change in sample and data throughput has led to challenges in user workflow and highlights that the biggest dead time during beamtime arises during manual sample handling with the need to search and secure the endstation (robotic mounting does allow sample changes to occur in less than 40 seconds).

The dynamic range of the Eiger is substantially greater than a CCD detector, however 'overloaded' pixels can occur. These 'overflows' are not immediately obvious in the frames, but can have a significant effect during count rate correction of the Eiger output. New tools are being developed to better quantify data quality prior to structure solution. Other software tools are being developed to aid in data processing.

Future upgrades are underway to further improve MX1 with goniometer modifications and a Dectris 9M Eiger detector scheduled for early 2019. Given the dramatic increase in experimental throughput, what additional opportunities can be embraced by the Australian Synchrotron's chemical crystallographic community? A review of current developments and discussion of future directions will be presented.

Parallel Session 14 / 84

Electronic structure of phenolic anti-oxidant trihydroxybenzoic acid using combined XPS and NMR spectroscopy

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Gallic acid (GA, 3,4,5-trihydroxybenzoic acid) and derivatives have been found in a number of phytomedicines with diverse biological and pharmacological activities, including radical scavenging, interfering with the cell signalling pathways and apoptosis of cancer cells. GA exhibits both antioxidant as well as prooxidant characteristics [1]. The chemical properties of phenolic hydroxyls are significantly different from those of the aliphatic-OH in glucose. As result, it is important to understand the properties of phenolic acids such as GA at molecular level. The present study uses theory and experiment, which combines state of the art XPS and NMR techniques to reveal detail of gallic acid. Our accurate quantum mechanical calculations connect the measured XPS and NMR spectra to the structure of GA, revealing the nearly-equivalent atoms in GA which are unable to differentiate by XPS nor NMR. Certain structural correlation between the XPS and NMR signals indicates that the para-position, C(5) as indicated, to the carboxyl group -COOH of GA, contains interesting information.

In addition, the present quantum mechanical calculation is able to decompose the valence orbital contributions to the binding energy spectrum based on their orbital symmetry.

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Small-angle X-Ray scattering structures of human APOBEC3 bound to single-stranded DNA

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The APOBEC3 (A3) protein subfamily of seven proteins (A3A-H, excluding E) are cytidine deaminases that suppress retroviruses and other pathogens by inducing hypermutation through deamination of cytidine residues on single-stranded DNA [1]. At the same time some APOBEC family members, particularly A3B and A3G, are utilised by viruses and cancer cells to increase the rates of mutagenesis, escape adaptive immune responses, and become drug resistant [2, 3]. Inhibition of A3 proteins may therefore be used to augment existing anticancer and retroviral therapies [4]. Previous structures have shed some light on single A3 catalytic domains bound to single-stranded nucleic acid substrate [5]; however, the structures of two-domain A3-ssDNA complexes remains unclear. We have used small-angle x-ray scattering (SAXS) to elucidate the structural changes upon binding of single-stranded DNA to A3 proteins. We obtained envelope models of the catalytic domain of A3B-ssDNA inhibitor complex, which exhibited slight changes in the radius of gyration (R_g) upon complex formation. Additionally, our putatively dimeric form of A3B catalytic domain provided SAXS envelopes and structural models consistent with dimeric form. However, upon binding of ssDNA the dimer interface disassembles into a monomeric A3B-ssDNA complex. Using data obtained with the SEC capacity of Australian Synchrotron, we have established SAXS structural models for a two-domain A3G-ssDNA complex, which revealed structural changes upon complex formation. This constitute the first full-length A3G-DNA model and will provide a platform for further structural studies and development of A3G inhibitors.

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Using structural biology of methanogenic archaeal enzymes to enhance our understanding of methane formation

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Methane is a potent greenhouse gas, and is a significant factor contributing to climate change. The rumen archaeal community is typically dominated by hydrogen-utilising methanogens from the order Methanobacteriales which possess cell walls composed of pseudomurein, a peptidoglycan analogue of bacterial murein. Rumen methanogenic archaea also possess a number of other unusual traits such as isoprenoid-based lipids linked to glycerol-1-phosphate through ether bonds, and a unique energy metabolism (methanogenesis) that requires six specialised cofactors. To gain insight into the fundamental biology of rumen methanogens we have determined crystal structures of key enzymes with archaeal-specific traits. Over 300 enzymes were targeted for structure determination, which produced in excess of one hundred purified soluble enzymes for crystallographic screening. More than 50 different enzymes produced crystals and 30 protein structures have been solved thus far. We exemplify the protein structural data with examples from the methanogenesis pathway, and from biosynthetic steps involved in lipid, cofactor F420 and cell wall formation.

Parallel Session 15 / 87

Synchrotron based techniques for studying energy storage materials

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Synchrotron radiation with high flux and high resolution gives us information that would not be possible from laboratory X-ray instruments. Synchrotron X-rays are available as an extremely intense beam that allow fast scattering or diffraction studies of energy storage materials. At synchrotron sources various techniques at different beamlines offer structural and chemical information on different time and length scales. For example, in-situ X-ray powder diffraction (XRPD) uses the high intensity and resolution of synchrotron radiation for fast studies of phase transitions and detailed structure solution of novel compounds, while X-ray absorption spectroscopy (XAS) uses the energy tunability properties of synchrotron radiation to provide inter atomic distances, bonding valence, and oxidation states of the samples. This

presentation will describe some case studies of the energy storage materials undertaken at the PD and XAS beamlines and demonstrate the method to the user community.

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Exposure to Synchrotron Terahertz (THz) Radiation Induces Cell Membrane Permeability in Pheochromocytoma (PC12) Cells

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Novel applications requiring the use of non-ionising THz radiation have rapidly emerged in recent years due to the progress in semiconductor and laser technologies. The terahertz (THz) band lies between microwave and infrared rays in wavelength, and consists of non-ionizing radiation with a frequency from 0.1 to 10 THz (where 1 THz = 10¹² Hz): wavelength (= 30-3000 μ m); wavenumber (k = 3.3-334 cm⁻¹); period (period sign = 0.1-10 picoseconds); temperature (T = 4.8-478 K); photon energy (E = 0.4-41 milli-electron volts). It is of utmost importance to explore and understand the effects arising from the interaction of electromagnetic fields with living entities in the micro and nano scales.

The aim of this study was to investigate the effects that synchrotron THz radiation have on pheochromocytoma (PC12) neuron-like cells. PC12 cell lines are derived from a pheochromocytoma of the rat adrenal medulla and the cell line has been employed to generate understanding of the neurodegenerative diseases including spinal cord injuries and brain diseases, such as Parkinson's disease.

The results obtained in this study revealed that PC12 cells were permeable after exposure to synchrotron THz radiation, as confirmed by internalisation of silica nanospheres, while remaining viable and metabolically active. The metabolic response of PC12 cells exposed to synchrotron THz radiation was evaluated using 3-(4,5-dimethylthiazol-2-yl)-5-(3-carboxymethoxyphenyl)-2-(4-sulfophenyl)-2H-tetrazolium (MTS) and bicinchoninic acid protein (BCA) assays. PC12 cell permeability was assessed using hydrophilic silica nanospheres visualised using confocal laser scanning microscopy (CLSM) and transmission electron microscopy (TEM). The cell morphology was studied using scanning electron microscopy (SEM).

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The influence of dragonfly *Austrothemis nigrescens* and damselfly *Calopteryx haemorrhoidalis* wing nanostructures on *Candida albicans* ATCC 10231 colonisation

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Colonisation of surfaces by fungi is a significant problem, especially in the healthcare sector, where antifungal drug resistance is becoming more prevalent. The majority of these infections are associated with biofilms on a medical device such as catheters, pacemakers and bone screws, making them especially difficult to treat and prevent. *Candida albicans* is the most common pathogen found in hospital-acquired fungal infections and causes significant morbidity and mortality, especially in immunocompromised patients. It is also an excellent model organism for investigating potential antifungal properties of a surface. Nanostructured surfaces, based off natural templates such as the arrays of nanopillars found on some insect wings, are a promising new development shown to greatly reduce bacterial adhesion and mechanically rupture bacterial cells that manage to attach. This research investigates the effect of the naturally nanostructured wing membranes of the dragonfly *Austrothemis nigrescens* and the damselfly *Calopteryx haemorrhoidalis* on the adhesion of the pathogenic, multidrug-resistant strain *C. albicans* ATCC 10231. The epicuticular layers of the insect wings were investigated using high-resolution scanning electron microscopy and Australian Synchrotron-sourced Fourier transform infrared (S-FTIR) microspectroscopy. The nanopillar arrays present on the nanostructured epicuticular surfaces were found to significantly reduce the attachment of *C. albicans* in comparison to the control following 1, 3, 5 and 7 days of incubation.

Parallel Session 1 / 90

X-ray Optics Upgrade Program for the AS Powder Diffraction Beamline

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The Powder Diffraction (PD) beamline at the Australian Synchrotron has operated its User program for 11 years with over 1200 proposals submitted and more than 650 experiments completed. These experiments and the collaborations with the beamline team have assisted the User Community in producing over 600 publications across a broad range of scientific fields. As the PD beamline ages, several end-of-life issues have arisen requiring replacement of key internal optical components. A major upgrade program has been undertaken by ANSTO to address the end of life issues at the PD beamline and improve its performance by modifying the design to match the properties of the source, optics and endstation to enable enhanced performance for the future. This presentation will describe the features of the optical upgrade program to be undertaken at the PD beamline and outline the future benefits to the User Community.

Evidence of Anatase Intergrowths Formed During Slow Cooling of Reduced Ilmenite

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Unusual and distinct features were observed in the powder X-ray diffraction (PXRD) pattern of a slow cooled reduced ilmenite (RI), which were not observed for a rapid-cooled RI. The PXRD pattern of the slow-cooled RI showed the M_{3O_5} peak at 20.6° (002) 2θ was not apparent, and the peaks at 37.9° (203), and 47.9° (204) and 48.4° (402) 2θ had significantly decreased in intensity. Using transmission electron microscopy (TEM), selected area electron diffraction (SAED) and pair distribution function (PDF) analysis, we attribute these features to M_{3O_5} -anatase intergrowth formation, which causes a loss in long-range order along the M_{3O_5} c-axis. These results demonstrate the importance of cooling rate during the formation of these industrially relevant RI materials.

Parallel Session 2 / 92

Correlative synchrotron infrared spectroscopy and super-resolution fluorescence microscopy for the detection of cellular DNA damage

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Single molecule localization microscopy (SMLM) and Synchrotron Fourier transform infrared (S-FTIR) spectroscopy are two techniques capable of elucidating unique and valuable detail and are especially suited for interrogation of biological samples. SMLM provides images of the structures and distributions of targeted biomolecules at spatial resolutions up to an order of magnitude better than the diffraction limit, whereas S-FTIR spectroscopy objectively measures the holistic biochemistry of an entire sample thereby revealing any variations in overall composition.

Previously, we have correlated these two techniques to probe the biochemical changes that common cell fixation and labelling methods cause, characterizing crosslinking and dehydration-induced losses of biomolecular composition and perturbation to cellular ultrastructure. More recently, we have expanded these studies to investigate the effects of drugs that cause DNA replicative and transcriptional stress.

In this study we acquired S-FTIR spectra of single druged live cells alongside SMLM super resolution images of these cells after fixation, visualizing the DNA damage sites and their associated repair proteins. The complementary nature of these techniques allowed us to detect subtle changes to the cellular metabolism as well as the chromatin structure. Remarkably, we were even able to differentiate undamaged cells from those treated with low drug dosages that cause damage usually undetectable by conventional methods. These studies strikingly demonstrate the potential sensitivity of these combined techniques for the correlated detection of biochemical changes while also highlighting the types of structural and compositional changes that could only be revealed by one of the two techniques.

Parallel Session 11 / 93

Calcium and Magnesium Intercalation of Graphene on Silicon Carbide

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Graphene has transformed experimental two-dimensional (2D) physics and has proven itself an indispensable testing-bed for improving our understanding of condensed-matter physics. Recent theoretical and experimental results from literature have suggested that graphene, highly doped with alkaline earths (through intercalation and/or surface decoration), can superconduct at relatively high temperatures, and is a potential platform for electronically mediated superconductivity. Furthermore, highly electron-doped graphene may be useful as a transparent conducting electrode with low workfunction for applications such as photovoltaics.

This work will present recent results from X-ray photoemission spectroscopy (XPS) - conducted at the Soft X-ray (SXR) beamline at the Australian Synchrotron - and scanning tunnelling microscopy (STM)- conducted in the Fuhrer Laboratory at Monash University - which elucidate the structure of calcium and magnesium intercalated graphene on silicon carbide.

We study both epitaxial monolayer graphene (EMLG) and quasi-free standing (hydrogen intercalated) bilayer graphene (QFSBLG) on 6H-SiC(0001) substrates. The former consists of monolayer graphene on a carbon interface layer, which is partially covalently back-bonded to the silicon face on SiC - often termed 'zero layer graphene' or 'the buffer layer'. The latter is formed by hydrogen treatment of the EMLG whereby the hydrogen is able to bond to the silicon on the SiC surface, releasing the interface layer and forming another layer of graphene.

Our XPS and STM data suggests that calcium and magnesium are able to intercalate underneath the graphene and bond with the silicon on the surface of the SiC -forming calcium/magnesium intercalated quasi-free standing bilayer graphene (Ca-QFSBLG/Mg-QFSBLG). Furthermore, the calcium may also intercalate between the graphene layers, which could result in highly n-doped graphene. Secondary electron cut-off (SECO) measurements show the change in workfunction for both intercalated materials. Surprisingly, the Ca-QFSBLG is stable to brief air exposures, indicating it may be useful as a transparent conducting electrode with low workfunction.

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Biomechanics of Collagen Fibrils During the Compression of Bovine Meniscal Cartilage

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Collagen is one of nature's building blocks and type I collagen is a major component of the extracellular matrix of meniscal cartilage in the knee. The hierarchical structure of collagen imparts strength and elasticity, which are both functionally and aesthetically important, thus collagen plays a crucial role in the biomechanical properties of the knee joint. Studying bovine meniscal cartilage provides useful insights into the human meniscus because bovine knees have similar mechanical properties to those of human knees. Here we have conducted simultaneous compression experiments and small angle X-ray scattering measurements to study the complex nanostructure of collagen fibrils in meniscal cartilage and their biomechanical response to compression. Collagen fibril orientation, D-spacing, compression distance and force were measured. Upon compression the collagen fibrils initially become more highly oriented perpendicular to the direction of compression. Following the reorientation of the fibrils they then begin to take up the stress along their length with a 1.5% elongation with compression up to 1.4 MPa represented by an increase in D-spacing from 65.0 to 66.0 nm. Therefore, the elasticity of the collagen fibrils in tension along their length when the meniscus is compressed contributes to the elastic response of the meniscus, rather than dissipation. Dissipation of energy in cartilage under compression results in water being exuded from the tissue. This research advances our fundamental understanding of the structural mechanics of collagen in meniscal cartilage and exemplifies how nature utilizes collagen for both its elasticity and strength. By understanding collagen's hierarchical structure and its biomechanical response to compression we gain insight into how the elasticity of collagen necessitates the arrangement seen in nature with fibrils aligned perpendicular to the direction of compression.

Parallel Session 15 / 95

In-situ X-ray Diffraction Studies on Age Hardening of Mg alloys

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Precipitates influence the relative hardening of slip and twin in magnesium alloys. The current work employs in-situ synchrotron X-ray diffraction to investigate the role of precipitate shape (e.g. plates and spherical precipitates) on the strengthening of Mg alloys. The critical resolved shear stresses (CRSS's) of dislocation slip and deformation twinning and the subsequent changes following aging are evaluated during uniaxial compression deformation. By application of a high-angular resolution diffraction experiment, the effect of precipitates on the age hardening is investigated based on the lattice strain response of the precipitate and bulk Mg matrix phase.

In this study, wrought Mg AZ91 and Mg-Sn-Zn-Na (MSZN) alloys were aged at 200°C for ~12 h to produce basal plate and near spherical precipitates. In-situ compression tests were performed in the twin dominated strain paths –compression along the rolling direction in case of rolled AZ91 alloy and compression along the extrusion direction in case of extruded MSZN alloy. Based on the in-situ measurement of the lattice strain evolution with load, the CRSS for basal slip was determined. It was observed that the strengthening of basal slip is low (~5 MPa) in case of basal plate precipitates in agreement with literature, and the spherical precipitates strengthened the basal slip by ~15 MPa. The CRSS for deformation twinning was calculated from the drop in the intensity of parent grains. In the case of basal plate and spherical precipitates, the twins are hardened in the range of 30 –35 MPa, despite the differences in the precipitate morphology and their elastic lattice strain changes in response to the applied deformation. By application of line profile analysis methods, the apparent area weighted twin size and dislocation density during twin onset was determined.

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Advanced Diffraction and Scattering Beamlines: High-energy diffraction and imaging for the materials, chemistry, engineering and earth science communities

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The Advanced Diffraction and Scattering (ADS) beamlines have been approved for inclusion in the BRIGHT program for new beamlines at the Australian Synchrotron. The ADS beamlines are designated as beamlines 4 and 5 in the BRIGHT program build order that will be planned, built and commissioned over a five-year period; with the first users expected mid-2023. The ADS beamlines will complement the existing Powder Diffraction beamline and enable the materials, chemistry, engineering and earth sciences communities to access high-energy synchrotron X-rays for diffraction and imaging experiments. ADS will employ a high-field, super-conducting multi-pole wiggler source to produce high energy and high intensity X-rays for two endstations operating simultaneously. The main endstation, located externally of the main building, will permit monochromatic (30 keV –150 keV) and pink beam modes for diffraction and imaging experiments, and will be large enough to accommodate complex sample stages and variable temperature and pressure environments. This will be particularly useful for geoscience community whose high-pressure multi-anvil experiments are a feature at many synchrotrons around the world. A support laboratory will be provided to allow offline experiment set-up and testing. The side-station, located within the main building, will have three fixed monochromatic energies (~45 keV, ~74 keV and ~86 keV) allowing a variety of high-throughput diffraction experiments to be performed, such as in-situ pair distribution function measurements. This presentation will describe the ADS beamline, its capabilities and potential scientific applications.

Parallel Session 13 / 97

Four-angle polarisation-resolved transmission FTIR mapping for materials orientation analysis

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Molecular orientation in polymeric and composite materials can play a significant role in overall mechanical performance. Infrared absorption by specific functional groups occurs preferentially when the electric vector of the probing beam is aligned with the dipole oscillation corresponding to the absorbing frequency, and can therefore be used to gain information on the molecular orientation of selected molecules. The team at the Tokyo Institute of Technology has developed a method whereby the dipole orientation angle, θ , for each pixel of a hyperspectral image can be determined from equation 1, where A_1, A_2, A_3, A_4 are absorbances at the four polarisation azimuths separated by $\pi/4$. The dipole orientation angle and strength are then plotted as “vectors” over each pixel within the spectral map, enabling the visualisation of molecular orientation. We have applied this method in the analysis of molecular re-orientation in silk fibres and in the study of the effects of additives to poly-lactic acid (PLA) composite materials. Figure 1 shows an example of the orientation vector map of spherulites formed in a PLA solvent cast film, showing the absorption strength (colour) and dipole orientation (vector line) for the C=O absorption at 1759 cm^{-1} (map = $150 \times 150\text{ }\mu\text{m}$).

Parallel Session 13 / 98

High-Resolution Macro ATR-FTIR Chemical Imaging Capability at Australian Synchrotron IR Beamline and Its Applications in Food Science

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This work presents advances in high-resolution chemical imaging capability at Australian Synchrotron Infrared (IR) beamline, achieved through the use of an in-house developed synchrotron macro ATR-FTIR microspectroscopic device (1). The device was developed by modifying the cantilever arm of a standard macro-ATR unit to accept germanium (Ge) ATR elements with different contact facet sizes (i.e. 1 mm, 250 μm and 100 μm in diameter). Coupling synchrotron-IR beam to the Ge ATR element ($n = 4$) used in this device, has the effect of not only reducing the beam focus size (improving the lateral resolution) by a factor of 4, but also reducing the mapping step size by 4 times relative to the stage step motion. As a result, the macro ATR-FTIR measurement at Australian Synchrotron IR Beamline can be performed at minimum beam size of 1.9 μm using a 20x objective, and at minimum mapping step size of 250 nm, allowing high-resolution chemical imaging analysis. It can also be coupled to a temperature control unit, allowing temperature-dependent study, as well as measurements that require a fixed temperature such as analysis of dairy products at 4 $^{\circ}\text{C}$ similar to the usual storage condition in a household fridge.

The development of the macro ATR-FTIR device has so far led to successful analysis of samples from a diverse range of research disciplinary. Key applications in food science to be presented include a range of dairy products (e.g. cheese and yoghurt), microencapsulated oil (2), plants and vegetables.

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Parallel Session 11 / 99

Substrate dependent ultrafast charge transfer dynamics in self-assembled monolayers with intramolecular orbital coupling –a combined spectroscopic and computational study

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Abstract: Metal surfaces coated with self-assembled monolayers (SAMs) are often used in large area based molecular junctions. However, characterizing these junctions still eludes chemists and physicists alike because of the complexity associated with the orbitals involved in charge transport and their behaviour under varying temperature. The metal-molecule interaction in such a system helps to understand the mechanism of charge transport, whether it is the Landauer (coherent transport) model or the Marcus model (incoherent transport) or an "in-between" regime, and thus, is crucial to the field of molecular electronics [1]. Recently, we reported that SAMs terminated with ferrocenyl (Fc) units and attached to a conjugated diphenylacetylene (DPA) backbone via an alkyl bridge showed diode behaviour in that "in-between" regime. Tuning intramolecular orbital coupling between Fc and DPA resulted in the diode being in either the inverted Marcus regime or the direct Marcus regime [2]. While the number of alkyl units separating the conjugated units is one variable to change the metal molecule coupling, it can also be varied by changing the surface dipole at the metal-sulphur interface. Here we used synchrotron-based core-hole clock measurements to study the ultrafast charge transfer dynamics (in the order of a few fs) of the series shown in Figure 1 (FcCn(DPA)CS SAMs on M where M = Ag, Au, Pt; n = 0-3). In addition to the spectroscopic characterization, we also attempt to understand the quantum chemical picture of the metal-molecule interface by performing density functional theory calculations. The coupling of Fc is in the order Ag > Au > Pt.

Keywords: charge transfer dynamics, core-hole clock spectroscopy, self-assembled monolayers, molecular electronics, density functional theory

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Merging Colourful Images and Nanometer Resolution –Combining X-ray Fluorescence and Coherent Imaging at the XFM Beamline

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The investigation of innovative materials or biological and chemical processes requires deep insight into their morphology and dynamics. Equally important is the knowledge of the chemical distribution in terms of elements and oxidation states. For these purposes, high-resolution X-ray microscopy is an important tool in nanoscience to analyse individual features of various kinds of specimens such as biological samples or heterogeneous catalysts.

A great strength of the XFM beamline is the fast and highly sensitive X-ray fluorescence mapping of trace elements and versatility in all kinds of samples at a spatial resolution (beam size) of $\sim 1 \mu\text{m}$.

We will present our ongoing work to implement high-resolution coherent imaging at the XFM beamline, bringing together hard X-ray ptychography, spectroscopy and X-ray fluorescence imaging to reveal the morphology at nanometer resolution in addition to the chemical specification of various samples in one measurement.

In recent years, the scanning coherent imaging technique ‘ptychography’ has been established in the synchrotron community providing high spatial resolution and also element sensitivity. In ptychography, the sample is scanned through a coherent beam, recording a (far-field) diffraction pattern at each scan point. Due to appropriate overlap between adjacent scan points, numerical phase retrieval algorithms allow for the reconstruction of the complex transmission function of the object, which in turn gives access to the absorption and the phase shift of the sample. As a so-called lensless imaging technique, ptychography reaches a spatial resolution beyond the diffraction limit of focusing optics. In the hard X-ray regime, spatial resolutions down to the 10 nm range have been reached for strongly-scattering as well as for weakly scattering objects. Figure 1 shows a high-resolution reconstruction of a weakly-scattering catalyst model sample to illustrate the capabilities of ptychographic imaging – individual nanoparticles down to a size of approximately 15 nm can be seen in the phase reconstruction (red circles).

The combination of ptychography with resonant scattering or with X-ray fluorescence enables access to both the morphology and the chemistry of the sample.

Parallel Session 5 / 101

Fluctuation powder diffraction of the lipidic cubic phase: a 3D view of lattice disorder

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Fluctuation scattering methods employ the statistical analysis of large scattering datasets to obtain a more accurate structural characterization of a material. Fluctuation methods were extensively developed for electron microscopy and are also topical in coherent x-ray imaging. We have developed novel fluctuation analysis methods for powder diffraction data to exploit extra structural information in spotty or textured powder rings. Generalizing 1D pair-distribution analysis, we extract 3D real-space distributions of three- and four-atom statistics from the fluctuation powder data. We have made a proof-of-principle demonstration with lipidic cubic phase collected at the SAXS beamline at the Australian Synchrotron. We have studied the lattice disorder induced by cholesterol doping, protein uptake and lysozyme crystallization. Data was collected with the Lipidico viscous-flow syringe injector that was used at the SAXS beamline for the first time.

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Highly Potent and Selective Plasmin Inhibitors Based on the Sunflower Trypsin Inhibitor-1 Scaffold Attenuate Fibrinolysis in Plasma

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Plasminogen (Plg) is the zymogen of serine protease plasmin (Plm) which plays a key role in fibrin dissolution, tissue remodeling and wound healing. Therefore, it is an attractive therapeutic target for a range of different medical conditions, e.g. traumatic bleeding, thrombosis and cancer. Plasmin inhibition is crucial to overcome undesirable plasmin activity, predominantly excessing blood loss. Currently, clinically used Plm inhibitors, also known as anti-fibrinolytic agents, lack either specificity or efficacy. Therefore, the search for a specific and efficacious Plm inhibitor is an ongoing research effort in the field.

Sunflower trypsin inhibitor-1 (SFTI-1) is a 14-amino-acid cyclic peptide that is well known for its exceptional stability and potency for trypsin ($K_i=100$ pM). It is therefore a very promising drug scaffold for developing specific inhibitors of serine proteases. Here, we determined the molecular interactions between SFTI-1 variants and Plm, with the aim to accelerate the process of structure-based drug design. We report the high-resolution x-ray crystal structures of catalytic domain of Plm in complex with SFTI-1 variants and reveal the importance of the P2 and P2' sites for the potency of the inhibitor. This work forms the bases for future development of specific Plm inhibitors that might be of great values in the management of bleeding, also cell metastasis, angiogenesis and cell proliferation.

Parallel Session 1 / 103

Update on X-ray Fluorescence Microscopy at the Australian Synchrotron.

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X-ray fluorescence microscopy (XFM) can be used for elemental and chemical microanalysis across length scales ranging from millimeter to nanometer. XFM is ideally suited to quantitatively map trace elements within whole and sectioned plant, biological specimens such as tissue sections, environmental and soil samples. The high elemental sensitivity of X-ray fluorescence microprobes coupled with deep penetration of hard X-rays enables measurement of an incredibly diverse range of samples in situ and under environmental conditions with a minimum of preparation.

Event mode X-ray fluorescence detection methods pioneered by the Maia detector system at the Australian Synchrotron XFM beamline enable high definition imaging which can approach megapixel per minute rates. The ability to rapidly acquire 2D images enables higher-dimensional studies such as fluorescence tomography, X-ray absorption near edge structure (XANES) imaging, and XANES tomography in realistic times.

Full spectral XANES imaging takes advantage of fast XFM and results in X-ray absorption near edge structure spectra from X-ray fluorescence at each pixel in the image. The speed and efficiency is gained by employing on-the-fly raster scanning and large solid angle, often multi-element, detectors with high count rate capabilities. The efficiency and speed ensures the lowest possible dose alongside high throughput.

In addition we are implementing high-resolution coherent imaging combining hard X-ray ptychography correlated with X-ray fluorescence imaging to reveal ultrastructure at high resolution.

Parallel Session 14 / 104

Ironing out the links between iron, mitochondria and disease.

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X-linked sideroblastic anemia with ataxia (XLSA/A2), is an incurable heterogeneous nonprogressive neurodevelopmental disorder. Mutations in ABCb7, a gene encoding a mitochondrial transport protein involved in biogenesis of iron sulfur clusters (ISC), underlie the disease. ISC are import co-factors, intimately involved in the chemical reactions that power the cell, repair DNA and create new proteins.

XLSA/A2 sees all of these processes disrupted to some extent but the chain of cause-and-effect is difficult to unravel. Despite the importance of ISCs biology, the tools available to study these species in situ are limited. To understand how mutations in ABCb7 disrupt ISC metabolism and injure the cell we need tractable biochemical models that retain elements of iron-sulfur biology salient to man. Fortunately, *Caenorhabditis elegans* is ideally suited to this task. By deploying X-ray micro-imaging and micro-analysis we have analyzed the chemistry of iron within intact, fully hydrated *C. elegans*. These data highlighted accumulation of inappropriate iron-sulfur species within the mitochondria upon disruption to abtm-1 (the nematode ortholog of ABCb7). This deleterious process drives dysfunction, accelerates age-related loss of mitochondrial ISC synthetic capacity and fosters mitochondrial dysfunction. The cycle of mitochondrial iron accumulation seen in XLSA/A2 recapitulates aspects of

dysfunction observed across a range of diseases, including Fredrich's ataxia. The gatekeeper role played by ABCB7's is unique but poorly characterized.

Coupling versatility of *C. elegans* models with micro-analytical techniques afford unprecedented opportunities to study ill-defined aspects of mitochondrial iron biology. The relevance of these findings and applicability of the approach to other diseases will be discussed.

Parallel Session 6 / 105

Towards clinical imaging and radiotherapy of human patients: An overview of IMBL techniques and programmes

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The Imaging and Medical beamline provide facilities for imaging, including phase contrast and in vivo movies, computed tomography (CT), including physio-triggered CT, radiotherapy, and with

- samples from 1mm³ to 1m³ and weight up to 100kg
- beam width up to 50cm
- energies from 20keV to 120keV (monochromatic) and 350keV (pink)

It is a unique research tool applicable to many fields, from engineering to life sciences, paleontology to new materials, food science, cultural heritage, volcanology, minerals, industrial processes, and of course in vivo research with translational applications, clinical programmes, veterinary research and other work, especially the study of physiological processes.

IMBL leads the field of large animal imaging by combining robotic positioning with an extensive support infrastructure and an experienced veterinarian on the staff. As many programmes are well established and bringing in new users, we are now concentrating on two new programmes: clinical imaging of human patients and canine radiotherapy.

For clinical imaging we have installed a 'patient positioning system' to be used from 2020 for phase contrast breast CT with human patients. This newly funded programme includes the addition of a beam expander to deliver a 10cm high, 20cm wide beam to our satellite building bunker at 140m. Combined with a high efficiency, large area detector, this new feature will enable new applications of imaging and CT in many fields besides our clinical programmes.

The canine programme will validate the full treatment process - treatment planning, dosimetry and image guided dose delivery - using a broad beam and patients supplied by veterinarians. After validation this process will be reliably established to move to micro-beam radiotherapy treatments to quantify the response of spontaneous tumors to micro-beams and gather long term toxicology data. This work is a critical and necessary step towards validation with human patients.

This presentation will include descriptions of the techniques available on the IMBL, examples of the most exciting work done by users across the fields listed above and an overview of our clinical imaging and canine RT programmes.

Figure caption: Imaging bunker at 140m with the large sample positioning robot, the standard CT stage (middle) and the patient positioning robot.

Australian Synchrotron IMBL facilities for in vivo research

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The Imaging and Medical Beamline (IMBL) has extensive facilities for holding and preparing animals, and for performing imaging, computed tomography (CT) and radiotherapy experiments with them. To date mice, rats, rabbits, sheep and pigs have been held and prepared, but the capability to hold and prepare other species can be requested.

Animal care

IMBL staff includes a veterinary surgeon who provide excellent support to our users with ethics applications, anesthetics, surgery, animal care practices as well as specialized support for imaging and radiotherapy.

Technical - Radiotherapy

- High-throughput dynamic micro-beam radiation therapy (DynMRT) setup for rodents
- Image guidance and dose delivery with conformal masks
- DynMRT includes a validated Patient Safety System (PaSS), an important step in our program towards human trials.

Technical - Imaging

- Convenient and modular positioning stages for mice and rats
- Robotic positioning system for large animals (up to 100kg)
- Software and hardware dose reduction triggering of shutters and detectors
- Ex vivo CT during imaging experiments upon request
- In vivo CT using physiological triggering

Technical - General

- Radiotherapy and imaging setups include laser beam guidance and live monitoring
- Large range of specialized surgery, anesthetic, ventilation and monitoring equipment
- All the drugs required for preparation and experiments
- Two surgeries and three laboratories are available for our users.

X-ray movie and detector data

- 15ms images at 30fps
- Field of view from 1cm² to 100cm²
- Detectors covering a wide range of resolution and efficiency (high efficiency = low dose but lower resolution) with pixel sizes of 10 to 200µm
- In vivo feature resolution of 30µm

Plenary 2 / 107

Elucidating structural transformations of electrodes while they are being used: The wonderful world of in situ synchrotron X-ray diffraction

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Electrodes account for a significant proportion of battery function, where atomic-scale perturbations or changes in the crystal structure during an electrochemical process permit the reversible insertion/extraction of charge carriers. A method to both understand battery function and improve their performance is to probe the crystal structure evolution in operando or in situ, i.e., while an electrochemical process is occurring inside a battery. In my group we heavily utilize the Powder Diffraction Beamline to track the evolution of the lattice parameters and/or charge carriers, e.g. sodium and potassium, in electrode materials used in rechargeable alkali-ion, primary lithium-metal, Li-S and solar batteries.

In undertaking in situ and in operando experiments there are a number of critical factors that need to be considered, for example optimised cell design to marry electrochemical performance with sufficient diffraction signal. Once the practicalities of such experiments are achieved, the parameter space that can be explored and correlated allows for unprecedented insight into function. Electrochemical parameters such as applied current rates, potential cut-offs and long term cycling can be correlated to chemical parameters such as composition and particle size distribution. Using this information we can design next generation electrode materials, optimising electrochemical performance parameters at a crystallographic level.

In addition to diffraction, my group is expanding our footprint with in situ analytical techniques, including in operando neutron imaging, in operando X-ray absorption spectroscopy and in situ solid-state NMR allowing us to probe non-crystalline components in devices. The combination of these techniques provides more (and multi-scale) insight into the mechanism of device operation and the interactions at play.

This talk will provide a flavor of the work being undertaken in my group, emphasizing the highlights and our future directions.

Parallel Session 1 / 108

Detector Development for Photon Science at PSI

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The detector group of the Swiss Light Source (SLS) at the Paul Scherrer Institut (PSI) in Switzerland has many years of experience in the development of single photon counting systems like Pilatus, Mythen and Eiger. Due to the startup of the european XFEL and SwissFEL the focus of the SLS detector group moved from photon counting to charge integrating detector systems. The charge integrating systems overcome many limitations of single photon counting systems like count rate, energy range or pixel size. In the talk I will present the charge integrating hybrid pixel detectors Jungfrau and Mönch and first results using them. Mythen3, a new single photon counting microstrip system, will also be presented and compared to Mythen 2.

Parallel Session 3 / 109

Elastic Flexibility in Molecular Crystals

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Molecular crystals are generally considered to be brittle and inelastic but they can in fact display remarkable elastic flexibility. For example, acicular crystals of bis(acetylacetonato)copper(II) can be tied, reversibly, in an overhand knot.

We have investigated the dynamic crystalline supramolecular chemistry that gives rise to this elasticity. In so doing we have determined the mechanism of elastic flexing of a single crystal, for the first time and with atomic resolution, using single crystal microcrystallography (1) (MX2 Beamline, Australian Synchrotron).

The relationships between supramolecular chemistry, crystal packing, crystal morphology, elasticity and the mechanism of elastic contortion of [Cu(acac)₂] crystals will be presented along with a discussion of the significance of this work in the context of current and widely-held perceptions of crystalline materials.

(1) Worthy A, Grosjean A, Pfrunder MC, Xu Y, Yan C, Edwards G, Clegg JK, McMurtrie JC, Nature Chem., 2018, 10, 65-69.

Parallel Session 13 / 110

A Novel Soft Contact Piezo-Controlled Liquid Cell for Probing Polymer Films under Confinement using Synchrotron FTIR Microspectroscopy

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Soft polymer films, such as polyelectrolyte multilayers (PEMs), are useful coatings in materials science. The properties of PEMs often rely on the degree of hydration, and therefore the study of these films in a hydrated state is critical to allow links to be drawn between their characteristics and performance in a particular application. In this work, we detail the development of a novel soft contact cell for studying hydrated PEMs using FTIR microspectroscopy. FTIR spectroscopy can interrogate the nature of the polymer film and the hydration water contained therein. In addition to reporting spectra obtained for hydrated films confined at the solid-solid interface, we also report traditional ATR FTIR spectra of the multilayers. The spectra (microspectroscopy and ATR FTIR) reveal that the PEM film build-up proceeds as expected based on the layer-by-layer assembly methodology, with increasing signals from the polymer FTIR peaks with increasing bilayer number. In addition, the spectra obtained using the soft contact cell indicate that the PEM film hydration water has an environment/degree of hydrogen bonding that is affected by the chemistry of the multilayer polymers, based on differences in the spectra obtained for the hydration water within the film compared to that of bulk electrolyte.

Parallel Session 2 / 111

It's a living breathing moving thing: advances in functional X-ray imaging of respiratory system health for Cystic Fibrosis.

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The absence of non-invasive measurements of detailed lung and airway function has greatly limited respiratory system health and disease management in medicine. Recent developments in synchrotron phase-contrast imaging in live animal models has enabled significant advances in measurement of physiological stability and change airways and lungs. Beginning at SPring-8, and paralleled later at the IMBL, our team has progressively advanced the animal care and outcome-measurement technologies to enable us to ask and answer novel and detailed questions about the physiology of the respiratory system, related to the genetic disease of cystic fibrosis (CF). Using examples from free-breathing or ventilated animals - from mice to pigs - new techniques, key findings, and future directions will be presented that reveal a steady advance in respiratory system functional imaging capabilities.

Parallel Session 9 / 112

Illuminating the mineralogy and geochemistry of mineral processing byproducts with coupled synchrotron XRD and XANES

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Increasing global demand for mineral and energy resources, coupled with declining ore grades, are creating growing global stockpiles of by-products including waste rock (~ 56 GT/yr) and tailings (~7 GT/yr). These are unusual geological materials, at extremes of pH and salinity, typically exhibiting elevated concentrations of elements and minerals present at otherwise low concentrations throughout Earth's crust, bearing process-derived minerals that are unstable under Earth surface temperatures and pressures, and in the case of tailings, with very fine particle sizes. Understanding and predicting the weathering behaviour of these materials, and opportunities for reprocessing or reuse, hinges on accurate mineralogical identification and quantification, which can be challenging in such complex, multi-phase mixtures often bearing novel minerals. Using synchrotron XRD and XANES, our work with bauxite residue (alumina refining tailings) has solved structures for novel process minerals in the sodalite and cancrinite groups, and identified mineralogical hosts and speciation of trace elements including As, Cr, and V. Incorporation of anions into sodalites increases unit cell size in the order carbonate

Parallel Session 5 / 113

Ten years of asking "why not?" at the SAXS/WAXS beamline

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We have been using the SAXS/WAXS beamline extensively since it began operation to ask questions that we have not been previously able to address around lipids, lipid self-assembly, their behaviour under a wide range of stimuli, what that means for drugs where present, and what happens when we combine lipids with other materials at interfaces both spatially and temporally. In the process we have continually pushed the capabilities of the beamline, invented new ways of looking at systems, and had a lot of fun in the process. In this presentation, I will highlight a number of aspects of our research on the beamline, that demonstrate its versatility and have led to some important breakthroughs in drug delivery and other fields.

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Machine learning for volumetric data analysis Meeting the Synchrotron challenge

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Keywords: (Micro-CT, Machine Learning, Bread, Automatic Analysis, Microstructure)

Over the past few years, analysing materials using Synchrotron micro-CT has had profound results, with the capability of capturing more complex structures at a higher resolution within a very short period of time. This makes it possible to study changes in material microstructure over time.

Tomography experiments at IMBL at the Australian Synchrotron were used to study the behaviour of different bread dough formulations during rising and baking. However, this poses a burden when it comes to analysing the very large set of data generated. The current pipeline to extract useful information from raw volumetric images involves semi-automated methods that require large amount of user-interaction. This work focuses on developing machine learning methods using a Convolutional Neural Network (CNN) architecture to facilitate automated analysis of sequences of volumetric datasets. The outcome of this work would alleviate the time-consuming aspects of the analysis workflow and would pave the way to perform 4D characterization experiments with a smaller time steps and larger datasets.

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Fabrication of OSAKA MIRROR for Synchrotron Applications

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We have been fabricating OSAKA MIRROR using EEM, nano-fabrication, and RADSI and MSI, nano-measurements, developed by Osaka University [1]. We have delivered more than 600 Super Precision Mirrors to synchrotron radiation facilities worldwide.

We will give an overview of our production technologies. Then we will report some focusing results using our OSAKA MIRRORS installed at SPring-8 and SACLA [2][3].

In addition, we will show fabrications of challenging mirrors which are developed in collaboration with Osaka University and SPring-8.

[1] K. Yamauchi et al., Rev. Sci. Instrum. 73 (2002) 4028.

[2] H. Ohashi et al., J. Phys.: Conf. 425 (2013) 052018.

[3] H. Yumoto et al., Nature Photon. 7 (2013) 43.

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Single Crystal to Single crystal Phase Transitions Metal Co-ordination Compounds.

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There is widespread interest in stimulus/response materials which a change in environment can be reported through property changes in a material. Phase transitions in crystalline compounds occur in response to environmental conditions, so are often examined. Single crystal X-ray diffraction structure determination of crystals before and after a phase transition often yield key insights into the mechanism of change and potentially allow for the design of new materials with favourable properties.

A series of phase transitions in metal co-ordination compounds will be examined for both discrete complexes and co-ordination polymers.

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Energy Drift behaviour of the XAS DCM

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The high power load produced by wiggler sources poses an engineering challenge when designing double crystal monochromators that exhibit high energy stability and reproducibility. The first crystal is subject to significant power load that must be dissipated with minimal geometric change of any mechanical assemblies that could lead to variations or drifts of the DCM Bragg angle (energy). At the XAS beamline, the power load incident on the first crystal can reach as high as 500 watts. The DCM is cooled with liquid nitrogen, effective at protecting the crystals and assemblies from permanent damage under beam load. However, the Bragg axis and crystal cage mechanical assemblies experience a temperature gradient between the cryogenic crystal temperatures and the ambient conditions external to the monochromator.

This gradient is perturbed when the power (heat) load on the first crystal changes. Subsequent thermal equilibration of the mechanical assemblies results in geometric changes that manifests as energy drift from the time of opening the front-end shutter and/or changing the white beam slits. The weekly cycle of user beam and maintenance/machine studies (no beam) means the thermal gradient is perturbed at least weekly.

A new high-performance double crystal monochromator (IDT Ltd.) was installed and commissioned on the XAS beamline in July 2017, and has been employed for user operations since cycle 2017/2. This contribution presents characterisation of the energy drift of the DCM as-delivered, characterisation of the thermal structure of the crystal cage assembly, describes solutions implemented to mitigate the observed energy drift and presents data quantifying the efficacy of those solutions.

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Structural and Magnetic Transitions in Osmium Scheelites

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Magnetic oxides containing 5d transition metals are currently attracting considerable attention as a consequence of their interesting and sometime unique magnetic behaviors observed when compared to the analogues. The differences in magnetic behaviors can be attributed to the diffuse nature of the 5d orbitals, giving rise to itinerant rather than localized electron behavior couple with strong spin orbit coupling effects. Spin orbit coupling often results in a decrease of observed magnetic moment, as is often observed in Re, Os and Ir containing compounds. The majority of studies to date have focused on systems where the transition metal is in an octahedral environment as occurs in the perovskite, in which octahedra of metal cations are linked via corner sharing oxygens. Hybridization results in strongly correlated systems, where superexchange mechanisms play a significant role in the observed magnetic ground state. In contrast in the ABO₄ scheelite structure the smaller B cation is tetrahedrally. The ionic nature of the larger A cation drives an ionic close packed structure of A cations and BO₄ anions. The isolated nature of the BO₄ tetrahedra precludes oxygen mediated superexchange interactions.

In this study the 5d1, S = ½ magnetic oxide KOsO₄ was successfully synthesized. Using a combination of synchrotron X-ray and neutron diffraction it has been demonstrated that KOsO₄ is isostructural to KRuO₄, and the observed changes in magnetic properties between these are attributed to the changed electron structure of the 4d1 versus 5d1 electrons, in which more diffuse nature of the 5d orbital gives rise to increased spin orbit coupling effects, and electron delocalization about the OsO₄- tetrahedra, resulting in an exceptionally small ordered magnetic moment. The effect of replacing K with a larger cation will also be discussed.

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Maia Mapper: High Definition XRF Imaging of Geological Samples at Intermediate Spatial Scales

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Maia Mapper is a laboratory XRF mapping system for efficient elemental imaging of drill core sections serving minerals research and industrial applications. It targets intermediate spatial scales, with imaging of up to ~ 80 M pixels over a 500×150 mm sample area, as part of the analytical workflow of the Advanced Resource Characterization Facility [1], which spans spatial scales from ore deposit to atomic scales. It brings together (i) the Maia detector and imaging system [2], with its capabilities for high efficiency detection (1.3 sr solid-angle), event-mode operation, millisecond pixel transit times in fly-scan mode and real-time spectral deconvolution and imaging [3], (ii) the high brightness MetalJet D2 liquid metal micro-focus X-ray source from Excillum [4] with high-In I2 alloy anode and 200 W power at 70 kV into an effective 20 μ m source size, and (iii) an efficient XOS polycapillary lens [5] with a flux gain $>20,000$ at 19-24 keV into a ~ 32 μ m focus, all integrated with stage raster scanning for automated imaging and analysis of drill-core sections.

Users select scan regions on a computer screen from a tiled optical image of the entire sample area. The list of scans is then executed in sequence with display of deconvoluted element component images accumulated in real-time in the Maia FPGA using a Dynamic Analysis (DA) method transform matrix constructed using the GeoPIXE software [3]. Further off-line refinement and re-processing (at rates of ~ 100 k pixels per second) and exploration of the large image data-sets are done using GeoPIXE [6].

[1] <http://www.sief.org.au/FundingActivities/ARCF.html>

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[3] Ryan et al., X-Ray Optics and Microanalysis, Journal of Physics: Conference Series 499 (2014) 012002.

[4] <http://www.excillum.com/>[5] <https://www.xos.com/focusing-optics>

[6] Ryan et al., J. Instrumentation 13 (2018) C03020.

Parallel Session 7 / 121

XAS and MEX –Reflection and Perspective

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Mark Ridgway was a key advocate for XAS techniques at the Australian Synchrotron. He was and will remain an integral part of its family.

Looking into the near future of X-ray absorption spectroscopy in Australasia, we wish to connect with Mark's legacy and highlight trends and developments around the MEX and XAS beamlines.

Parallel Session 7 / 122

In memory of Professor Mark Ridgway: a champion for Australian synchrotron science

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This presentation will give an overview of Mark's distinguished scientific career and the leadership he provided for Australian synchrotron science. It will present some examples of the work Mark has done at the ANBF and the AS that has defined the state of the art in the field and inspired many young researchers.

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Recent Progress on the Toroidal Angle Resolving Electron Energy Spectrometer

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Angle Resolved Photoelectron Spectroscopy (ARPES) is the “complete” photoemission experiment. It simultaneously measures a photoelectron's kinetic energy, emission angle and sometimes spin, relative to the crystallographic axes, constructing a direct image of the electronic bandstructure. This makes ARPES the most powerful contemporary technique for determining the electronic structure of novel materials. ARPES has been instrumental in the discovery and understanding of new electronic phases of matter. For example, important aspects of the electronic structure of high-Tc superconductors, such as the pseudogap were discovered using ARPES, as was the experimental discovery of three dimensional topological insulators Bi_{1-x}Sb_x and Bi₂(Se,Te)₃. Over the years, a dramatic improvement in the energy and momentum resolution possible with ARPES has occurred as a result of advances in photoelectron analysers and 2D detectors, allowing a range of new physics to be probed.

Despite the popularity of ARPES overseas, within Australia it has until now remained as a niche technique due to a small (albeit dedicated) user community. However, the continually growing local interest in studying novel materials with exotic electronic properties has led to the demand for our own synchrotron-based ARPES instrument. The ARPES detector, a toroidal geometry analyser, is now installed at the Soft X-ray beamline with commissioning completed and first experiments conducted. An overview of the latest developments on the instrument is presented. A substantial upgrade to the system's capabilities has been completed via the recent installation of (i) an intense microwave-based, monochromated, helium discharge VUV source and (ii) a scanning tunnelling microscope (Fermi SPM, Scienta Omicron GmbH).

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The THz/Far-IR beamline: Techniques and Applications

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Synchrotron infrared radiation offers a signal-to-noise advantage over conventional thermal sources, and the magnitude of this advantage varies to a great degree upon the spectral range, sample size and the spectral resolution required by the experiment. The THz/Far-IR beamline at the Australian Synchrotron is coupled to a Bruker IFS125 Fourier transform spectrometer equipped to cover the spectral range between 10 and 5000 cm⁻¹. There is a suite of instruments at the beamline to accommodate the requirements of the diverse User community. For gas-phase applications, we have room-temperature and cryogenic cells with variable path-length optics capable of achieving up to 35 m in path-length; we also have a furnace that can be used to study short-lived radicals by pyrolysis. Condensed-phase Users can perform transmission studies using a couple of cryostats which can be coupled to our custom liquid-cells equipped with diamond windows. They can also study their samples in reflection at grazing or near-normal incidence, and have access to a furnace. Finally, there is also a new ATR system which has been adapted to the IFS125 spectrometer, and a new cryostat for reflection at grazing angle or for matrix-isolation type studies. In addition, Users have access to a 25W cw CO₂ laser and a 10 Hz 480 mJ Nd:YAG laser for photolysis studies. In this paper, some applications undertaken at the beamline as well as the capabilities of the THz/Far-IR beamline will be presented

Plenary 3 / 125

DIY meets Bespoke in Synchrotron Radiation Science: an Australian perspective

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Dudley Creagh
Emeritus Professor, University of Canberra

Radiation science using dedicated synchrotron storage rings has existed for only 38 years. And in that time the advances in both technology and techniques have been astonishing. The time when researchers took their own laboratory equipment to SR beamlines is long gone. Researchers now have a wide range of beamlines and techniques from which to choose to satisfy their experimental needs: each beamline possesses a variety of environmental specimen stages; dedicated beamline scientists are on hand to give advice; funding for travel and accommodation is provided. This lecture reflects on how it is that Australian scientists are blessed with the facilities they currently enjoy.

Parallel Session 2 / 126

Exploring the Spatial Distribution of Chemical Species within Latent Fingermarks using Infrared Microscopy

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The successful recovery of latent fingermarks is a valuable tool in crime scene investigations to establish connections between suspects, objects and locations. The chemical composition of fingermark residue is a complex mixture of aqueous (eccrine fraction) and lipid (sebaceous fraction) secretions with contaminants such as food and cosmetic residues. By visualising the relative abundance and distribution of chemical components within fingermark residue we can provide explanations for the variation in reproducibility of latent fingermark detection with existing methods, and to identify new strategies to increase detection capabilities.

Infrared (IR) spectroscopy has been used to study fingermarks in situ to investigate aging, donor variation and chemical changes in fingermark residue over time. Despite the relative improvement of spatial resolution obtained with FTIR compared to other methods (e.g. imaging mass spectrometry), the spatial resolution is still hampered by the long wavelengths of light used relative to optical microscopy, as well as the limitation of substrates when working with transmission FTIR. One alternative is to use attenuated total reflectance FTIR (ATR-FTIR), which improves spatial resolution and enables measurement of fingermarks deposited on infrared-opaque substrates (such as glass). [1] The best possible spatial resolution is achieved with the technical capabilities of the synchrotron, the increased signal to noise ratio provides increased sensitivity and spatial resolution for the complex analysis of fingermark residue. [2]

In this investigation, we have used infrared microscopy to probe the spatial distribution of the sebaceous and eccrine chemical components within latent fingermarks. Whilst conventional FTIR spectroscopy fitted with a focal plane array detector allowed imaging across a large area of the sample, synchrotron sourced ATR-FTIR was used for the complex analysis of fingermark residue at sub-micron pixel resolution. By imaging individual droplets across a fingermark ridge we have been able to prove that fingermark droplets have a varying chemical composition of hydrophilic and hydrophobic components, closely resembling an emulsion (see attached Figure 1).[3] These results advance our current understanding of fingermark composition, providing information which will assist in future research into fingermark residue and its interaction with fingermark detection methods.

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2. Fritz P, van Bronswijk W, Lepkova K, Lewis SW, Lim KF, Martin DE, et al. Infrared microscopy studies of the chemical composition of latent fingermark residues. *Microchemical Journal*. 2013;111:40-6. 10.1016/j.microc.2012.08.005
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Revealing the Elemental Distribution within Latent Fingermarks using Synchrotron Sourced X-ray Fluorescence Microscopy

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Fingermarks are an important form of crime-scene trace evidence however, their usefulness may be hampered by variation in response or lack of robustness in detection methods. Understanding the chemical composition and distribution within fingermarks may provide explanation for the variation in latent fingermark detection with existing methods and identify new strategies to increase detection capabilities.

Previous research has focused on the organic components of fingermark residue, leaving the elemental distribution less well understood. This research has successfully demonstrated the capabilities of synchrotron sourced XFM for imaging the distribution of inorganic components within fingermark residue (see attached Figure 1), including the trace metals (Fe, Cu, Zn) and ions (Cl⁻, K⁺, Ca²⁺). The results indicate the potential contamination of fingermark residue with some metals detected which can only have originated from exogenous sources such as contact with metal alloys or cosmetics (Ni, Ti, Bi).

A better knowledge of elemental distribution within latent fingermarks is important to increase understanding of the effects ions and transition metals may have on the underlying chemistry of current methods of detection, as well as supporting the development of novel techniques. It is understood that fingermark chemistry is highly influenced by the environmental conditions, such as water exposure which contributes to the removal, particularly of labile, hydrophilic eccrine secretions. This study explores the ion and trace metal concentration and distribution within fingermarks prior to and post water immersion, demonstrating how the endogenous metallic deposits (Cu, Zn, Cl⁻) are removed with water exposure, whilst some exogenous material remains (Ti, Fe, Zn).

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High Energy Diffraction on the IMBL

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Diffraction using high energy X-rays (30 –110 keV) is a useful tool for phase and strain mapping through moderately large or small dense objects. It has been used within both mechanical and aeronautical engineering. In the future, Australian Synchrotron users will be able to perform high energy diffraction at a new purpose built beamline, but in the interim we have been developing a basic high energy diffraction capability on the existing IMBL beamline. We present two sets of examples of high diffraction from IMBL. A depth profile of phase from an oxidised ultra-high temperature ceramic, and the depth profile of strain from an aluminium strip during in-situ 4 point loading. The simple experimental layout is also presented.

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Oxidant or Catalyst for Oxidation? A Study of How Structure and Disorder Changes Selectivity for Direct vs. Catalytic Oxidation Mediated by Manganese (III,IV) Oxides

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Structural type and disorder have become important questions in catalyst design with the most active catalysts often noted to be “disordered” or “amorphous”. To quantify the effects of disorder and structural type systematically, a test set of manganese (III,IV) oxides was developed and their reactivity as oxidants and catalysts tested against three substrates; methylene blue, hydrogen peroxide and water. We find that disorder destabilises the materials thermodynamically making them stronger chemical oxidants but not necessarily better catalysts. For the disproportionation of H₂O₂ and the oxidative decomposition of methylene blue- MnO_x mediated direct oxidation competes with catalytically-mediated oxidation, making the most disordered materials the worst catalysts. Whereas, for water oxidation the most disordered materials and the strongest chemical oxidants are also the best catalysts. Even though the manganese (III,IV) oxide materials were able to oxidize both methylene blue and peroxides directly, the same materials were able to act as catalysts for the oxidation of methylene blue in the presence of peroxides. This implies that effects of electron transfer timescales are important and strongly affected by structural type and disorder. This is discussed in the context of catalyst design.

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Technology and Production Advances in the Australian Composites Sector and the Role of Collaborative Partnerships

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N/A

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Industry Session Q&A

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N/A

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End of session

Upgrades to the Soft X-ray beamline

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This poster describes the upgrades that has been made to the Soft X-ray beamline during the last year (monochromator and mirror 4 upgrade) as well as a new upgrade (1D delayline detector) which will take place during the 2019 January shutdown.

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Michael Fuller Talk

Parallel Session 7 / 135

Mark Ridgway Talk: Chris Glover

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Mark Ridgway Talk: The Early Days, ANBF and ASRP

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Multi-Modal Spectroscopic Approach to Studying Chemical Alterations During Biological Health and Disease

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Spectroscopy encompasses a range of valuable techniques that can provide opportunities for detecting molecular and elemental markers of biological processes in health and disease. The measurements are direct and stain free which reduces the risk of artifacts that can occur during chemical fixation and staining of biological samples. In addition, direct spectroscopic methods have the ability to measure chemical species that cannot be detected by traditional histochemical microscopy methods. Coupling spectroscopic techniques with the brightness of a synchrotron light source enables spectro-microscopy modalities and the opportunity to image chemical markers at cellular and sub-cellular resolutions that bench top instruments cannot provide. Previous studies have taken a multi-modal approach, incorporating techniques such as X-Ray Fluorescence Microscopy (XFM), Fourier Transform Infrared Microscopy (FTIR), synchrotron ATR-FTIR and confocal Raman micro-spectroscopy to reveal both elemental and molecular alterations at a cellular and sub-cellular level in mouse and rat brain tissue (1, 2). The studies demonstrated that, when coupled, these techniques can reveal alterations to metal and lipid distribution between different brain regions and also allow for chemical characterisation at a sub-cellular level (1, 2). The ability to determine both molecular and elemental information provides greater biological context, which will be capitalised on in future studies of disease mechanisms.

1) Summers KL, Fimognari N, Hollings A, Kiernan M, Lam V, Tidy RJ, et al. A Multi-modal Spectroscopic Imaging Method To Characterize the Metal and Macromolecular Content of Proteinaceous Aggregates ("Amyloid Plaques"). *Biochemistry*. 2017;56(32):4107. DOI: 10.1021/acs.biochem.7b00262

2) Fimognari N, Hollings A, Lam V, Tidy RJ, Kewish CM, Albrecht MA, et al. Bio-Spectroscopic Imaging Provides Evidence of Hippocampal Zn Deficiency and Decreased Lipid Unsaturation in an Accelerated Ageing Mouse Model. *ACS Chemical Neuroscience*. 2018. DOI:10.1021/acschemneuro.8b00193.

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

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Magnetite (Fe₃O₄), the oldest known magnet, is still a hotly debated material in scientific research, due to its complex magnetic, electronic and transport properties. One of the most interesting physical phenomena associated with Fe₃O₄ is the occurrence of a metal-insulator transition at ~120 K (TV), the so-called Verwey transition, which is associated to a charge ordering below TV, accompanied with a structural transition from the cubic phase to the monoclinic phase. However, due to the twinning of crystal domain, the detailed crystallographic structure is not fully solved yet and different charge ordered and bond-dimerized ground states have been proposed. In order to overcome this problem, we have investigated Cu-doped Fe₃O₄ and have determined the stability range of the Verwey phase in the phase diagram of Fe_{1-x}Cu_xFe₂O₄.

Using neutron diffraction and high resolution X-ray synchrotron diffraction we have investigated both the crystallographic and magnetic structure of Cu-doped Fe₃O₄ (Cu_xFe_{3-x}O₄ with $x = 0$ to $x = 0.95$) in order to elucidate the effect of doping on the Verwey transition. Data obtained from both complementary diffraction techniques indicate that the Verwey transition temperature and the magnetic structure, in particular the magnetic moment remains unchanged up to highest doping levels of 75% Cu-substitution. The large stability range of the Verwey phase is a surprising result and did require a systematic investigation. The analysis of our high resolution X-ray synchrotron diffraction data in combination with the neutron diffraction data did allow us to extract detailed information on the precise doping mechanism, for example if the Cu-ions are placed on tetrahedral or octahedral sites in the spinel structure. The obtained diffraction data provide therefore valuable information on the charge order transition, i.e. the Verwey transition.

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If a volcano erupts underwater, and no one is around to see it...

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Submarine volcanism is a dominant feature on Earth, yet most volcanism in the marine realm lacks observation and remains poorly understood. The 2012 Havre eruption in the SW Pacific was a once in a 100-year event that went unobserved and eruption style is debated. Current theoretical models suggest that the eruption was effusive (lava producing; Manga et al., 2018), however, pink pumice produced by the eruption suggests high temperature oxidation of iron based on subaerial analogues and would therefore require an explosive eruption (Tait et al., 1998; Morizumi et al., 2009). Here we map both the elemental content and the iron oxidation state in white, pink, and dual coloured Havre pumice to test eruption models and the ocean depths in which highly explosive volcanic eruptions are possible.

References

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Morizumi, M., et al., (2009). *Bulletin of Volcanology*, 71(1), 1-13.

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Session finishes

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Synthesis of Chemicals from Biomass Gasification

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Biomass as a low cost, renewable substitute for the fossil fuels has received tremendous attention over the past few decades. Moreover, biomass as a feedstock itself contributes to nearly 8% of the total energy mix in Australia. Conversion of biomass through thermochemical means, particularly gasification, is attractive due to high throughput, fast reaction rates, and low separation costs. The gasification product, syngas, a mixture of carbon monoxide (CO), carbon dioxide (CO₂) and hydrogen (H₂), is either combusted for energy or converted into numerous products using unique catalysts particularly into important industrial intermediates such as methanol which can further be converted into fuels such as dimethyl ether (DME) and chemicals such as dimethyl carbonate (DMC). In this project, both aspects, the gasification of biomass and the subsequent conversion of syngas into these chemicals is addressed with special emphasis on the CO₂ utilization opportunities.

In this study, Pine bark has been evaluated as a feedstock for gasification. The experiments have been carried out on pulverized biomass particles in the temperature range of 1000-1200°C in a purpose-built entrained flow gasifier that mimics industrial gasification conditions. We have made use of CO₂ as a gasifying agent in place of the conventionally used steam which is expensive. Through this process, near complete conversion of the carbon in the biomass was achieved at a relatively lower temperature of 1200°C in a time frame of <10 seconds!

In the next phase of the project, the syngas (CO, H₂ and CO₂) produced from the biomass gasification was converted into chemicals and fuels using a catalyst. The catalyst is the core of this process and its composition is to be optimized such that the operational conditions are viable in a large industrial setting. In order to achieve the objective of CO₂ utilization, several catalysts have been designed, synthesized and tested under industrially relevant conditions. Among these, copper supported on cerium oxide were found to be the most efficient and stable catalysts for the conversion of CO₂ into methanol. Further changes to the catalyst composition enable the co-synthesis of methanol, DME and DMC. This research contributes to the scientific and the technical community by laying the groundwork for the commercial development of the synthesis of chemicals DMC and DME directly from syngas. Currently, process modelling and life-cycle analysis is being undertaken to compare the environmental impact such as carbon footprint and toxicity of the novel process against the traditional routes.

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Role of X-ray absorption spectra in deducing the mechanism of conductance switching in E2-Lftn based biomolecular tunnel junctions

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In large area based molecular electronics, a field of widening research interest, molecules are sandwiched between metal electrodes in the form of a self-assembled monolayer (SAM) on a metal. These SAMs can be further modified to study interesting charge transfer (CT) phenomena across such junctions. We recently reported that electrode-SAM-biomolecule-electrode junctions of the form AuTS-linker/ferritin//GaOx/EGaIn (where TS stands for template stripped; EGaIn is eutectic alloy of Ga and In and acts as the top electrode; GaOx is Ga-oxide layer; “//” stands for van der Waals' contact; “/” stands for covalent contact and the linker is a modified self-assembled monolayer (SAM) of 6-mercaptopentanoic acid on the AuTS surface) show long range coherent tunnelling behaviour, meaning that the phase of the tunnelling wave is conserved across the junction (7 nm) (Adv. Mater. 2016, 28, 1824).

Currently we are also investigating conductance switching in E2-LFtn (modified ferritin with a size of ~13 to 19 nm) as a function of Fe-loading. A cartoon of the monolayer used in such a study is shown in the figure below. According to X-ray absorption spectra (XAS) also shown in the figure below, as the temperature changes from room temperature (RT 1) to 120 K and back to room temperature (RT 2), the relative ratio Fe²⁺/Fe³⁺ undergoes a reversible phase change (J. Electron Spectros. Relat. Phenomena 2015, 199, 19). We correlate the associated phase changes with temperature dependent charge characterization experiments.

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Using beamlines to look inside complex Additive Manufactured structures

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Conflux Technology has a mission to pioneer thermal technology. We are a passionate team of Additive Manufacturing scientists, engineers and commercialisation experts. Our goal is to facilitate the rollout of AM through its application in addressing thermal management challenges. The primary technical challenge we face is “managing the meltpool” in order to reduce the variability in the microstructures of the geometries we produce. This requires high resolution imaging of very complex structures that can not be inspected through traditional means. Collaboration with the Australian Synchrotron makes this analysis possible and influences the development direction of Conflux specifically and will impact AM more broadly.

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Novel Insights in the Seeded Growth of Gold Nanorods –a SAXS Study.

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The applications of gold nanorods reach over a broad range in biology, plasmonics and sensing. Recent progress in understanding of the growth mechanism at different stages, including the symmetry breaking step, renewed the interest in wet-chemical nanoparticle growth processes. Despite several studies of the crystallisation dynamics of ascorbic acid-catalysed nanorod syntheses, the growth kinetics of gold nanorods in a hydroquinone-based synthesis are not well-understood. A more detailed insight into the growth of nanocrystals can be provided by in-situ observations. Time-dependent optical properties of growing gold nanorods can be analysed using absorption spectroscopy, whereas in-situ investigations of structural evolution are more challenging, making the use of strong X-ray scattering sources such as the synchrotron necessary. This provides sufficient time resolution to detect single steps of nanoparticle growth to study the growth kinetics.

We studied the growth process of hydroquinone-based seeded-growth of gold nanorods in-situ. The synthesis in aqueous CTAB solution showed a novel double-sigmoidal growth, which can be linked to the reaction speed of single gold crystal facets. We find this optimised synthesis route as highly reproducible, resulting in monodisperse gold nanorods without significant side

products. From the spectral and scattering data, we can explain for the first time the growth mechanism of the hydroquinone-based synthesis from cuboctahedral seeds to an ellipsoidal state to the final spherical capped cylinder. Furthermore, the influence of changing the gold acid and hydroquinone precursor concentrations in the initial solution was investigated, giving further insights into the nanoparticle growth mechanism. The high quality of the synthesis and X-ray scattering experiments made it possible to actually determine the size of the CTAB layer around the growing nanorod during the synthesis.

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Indium Oxide Electron Transport Layer for Preferential Molecular Packing and Efficient Charge Extraction in Organic Photovoltaics

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Organic solar cells based on a bulk-heterojunction (BHJ) structure are promising candidates for next-generation solar cells. The electron transport layer (ETL) plays an important role in determining the power conversion efficiency of OSCs. A high mobility In₂O₃ synthesized by a solution processed combustion reaction is successfully used as a universal ETL. A thin layer of PEIE is deposited on the surface of In₂O₃ film to tune the work function for the more efficient charge extraction. The device based on PEIE modified crystalline In₂O₃ outperforms its counterpart, PEIE modified ZnO, in both fullerene and non-fullerene systems. The effects of ETL on the morphology of active layer are investigated by using grazing incident wide angle X-ray scattering technique. The crystalline In₂O₃ ETL exhibits highly aligned nanocrystallites, which induces the crystallization of polymer into preferential molecular pack. The favorable morphology of polymer facilitates the charge transport across the active layer.

Reference

[1] W. Huang et al., Nano Lett., 18, 5805 (2018).

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A synchrotron and a nano-fab lab met in a bar

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It is rare for a synchrotron and a fabrication facility are located so near each other. But we have this at Clayton. This talk will look at what ANFF Vic Node and the Australian Synchrotron are trying to do together as they aim to set up an x-ray lithography capability. This will enable extreme ultraviolet high-resolution lithography (EUVL) that is usually limited to industrial high-volume manufacturing and accessible by few research groups outside Australia due to cost. By combining Australian Synchrotron and ANFF capabilities, a novel EUV will be created.

This will be useful as nanolithography has enormous potential. Not only will it be the driving-force behind manufacturing the next generation devices, it will also enable scale reduction in the fields of nanotechnology with applications in areas including future electronics, microbiology, biomaterials and surfaces and do this at scale. To realise this potential, routine and cost-efficient large-area nanopatterning and manufacture at length-scales below 50 nm must be achieved. EUVL is the leading candidate to meet this challenge, and will likely offer the next generation of lithography capable of high volume manufacturing at the sub 10 nm length scale. This talk will describe the vision and identify the unresolved technical challenges that limit EUVL resolution.