



## ANBUG News – March 2018

### Save the date!

The AINSE/ANBUG Neutron Scattering Symposium (AANSS) will be held this year, 19-21 November at Lucas Heights ANSTO. After the success of the meeting in 2017, where we joined our colleagues in the synchrotron and accelerator communities in Melbourne, we will return to Sydney for a neutron specific symposium. The conference chair for this year's event is Gail Iles from RMIT. We hope to see many of you there.

### Schools

The 2018 AONSA neutron school will be held in Sydney 12-16 November. The school will mix hands-on practical experiments on the ACNS instruments with lectures from leading scientists, and will be a great opportunity for students and early career researchers.

There is also a powder diffraction data analysis workshop, 24-26 September at Sydney University. The powder diffraction teams from the Australian Synchrotron and the Australian Centre for Neutron Scattering are running the school, organised by Brendan Kennedy.

### Australia's Neutron Instrumentation Capability - *Tracy Rushmer*

In 2017, several OPAL instruments were most productive, in terms of number of refereed papers, in the world. This is quite an achievement. These include ECHIDNA (high-resolution powder diffractometer) and KOWARI (strain scanning). QUOKKA (SANS) has increased significantly since 2010 and now is top half in terms of publications and PLATYPUS (reflectometer) is in the top one-third. DINGO (the radiography/imaging station) is greatly increasing its activity and now we hoping to see the same with BILBY (the 2nd SANS), KOOKABURRA (USANS) and EMU (backscattering).

### 2017 AONSA Neutron School

In November 2017, five Australian PhD students received ANBUG/AINSE scholarships to attend the regional AONSA Neutron School in Japan. Ji Zhang from UNSW wrote us a short report.

*I received travel funding from ANBUG and AINSE to attend the 9<sup>th</sup> AONSA Neutron and the 2<sup>nd</sup> Muon school which was held in Japan from 16<sup>th</sup> to 20<sup>th</sup>, November. The school consisted of an intense series*

*of morning lectures, followed by facility tours in the afternoons. The contents of the lectures were very useful and easy to understand through the use of many figures and animations. Clearly the lecturers had put a lot of effort into their presentations. In the latter half of the school there were scheduled hands-on activities where the students could choose a beamline of interest to perform an experiment on. On the final day, the students then gave group presentations on the findings of their beamline experiments.*

*Having already performed a number of neutron scattering experiments at ANSTO I chose to partake in the hands-on activity on the S1 beamline to investigate muon spin rotation relaxation. Muon science was a completely new area for me, I found the sample environment to be similar to most of the neutron experiments I had previously performed, while the sample set-up was relatively simpler. I worked in a group with four other students to measure and analyse data and discuss our results with the help of the instrument scientist. We (students and instrument scientist) worked until 2:00 AM in the morning before the day of the presentation, which was well worth it in the end because our group won the best presentation award!*

*Overall, I found the AONSA neutron and muon school to be extremely useful. The lectures on introductory neutron science and neutron scattering techniques were informative and well presented. Through the school I was able to meet a diverse range of people, engage in research discussions and form friendships - not to mention the food was delicious! I would highly recommend other students to attend the AONSA neutron and muon school.*

## **ANBUG PhD student news**

Below we highlight the research projects of four ANBUG PhD students.

### **Structure-function properties of layered transition metal oxides**

**Jennifer Stansby, University of New South Wales.**



Batteries are ubiquitous in our modern, technology-dependent society. Due to concerns surrounding the cost and availability of Li based materials, there is significant interest in developing alternative technologies such as Na-ion batteries. At present, the commercialization of Na-ion batteries requires optimization of the positive electrode (cathode).

My research focusses on layered transition metal oxides,  $\text{Na}_x\text{MO}_2$  ( $\text{M} = \text{Ti}, \text{Mn}, \text{Fe}, \text{Co}, \text{Ni}$ ), which are a promising family of cathode materials due to their high capacity and rate capability. Through systematic substitutions on the Na site, we hope to improve the structural stability and energy density of these material. X-ray and neutron diffraction, electron microscopy and X-ray photoelectron spectroscopy are some of the techniques that will be used to characterise the materials to gain a greater insight into their structure-property-function relationships.

## Describing water within the Earth's interior

### Sarath Patabendigedara, Macquarie University

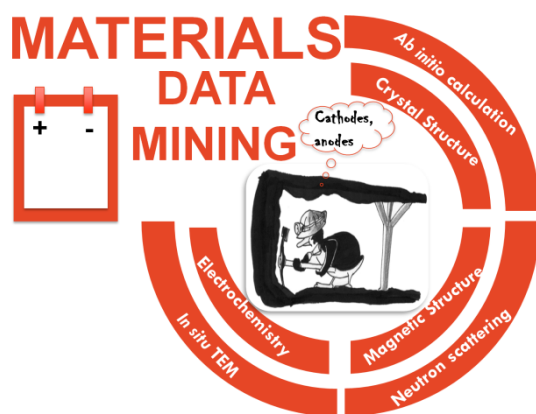
An understanding of water diffusion in mantle minerals is essential to describe the physical and chemical behavior of the Earth's interior. Water can be incorporated into mantle minerals as inclusions in the crystal structures or on the crystal surface in grain boundaries. A small amount of water lowers the viscosity of mantle minerals and rocks, reduces the melting temperature, modifies phase equilibria and petrologic processes and increases electrical conductivity. The presence of water in mantle rocks might be a critical factor for the initiation of plate tectonics. Quantification of the dynamics of water diffusion within mantle minerals is important to describe mantle behavior.

Direct observation of water is not possible inside deep earth. However, water content can be estimated by examining materials returned from the deep earth as mantle xenoliths or mantle-derived magmas. Although this has an inherent time delay of many tens of millions of years. Indirect real-time in situ observation is possible through geophysical means, using Magnetotelluric. The reported results from laboratory measurements on wet mantle minerals do not match with the field observations and needs more explanation to correct this discrepancy.

Our approach is to separately measure in-grain and grain-boundary water diffusion in a polycrystalline mineral matrix. We are using temperature dependent Quasi-Elastic Neutron Scattering (QENS) to measure the temperature dependent in-grain diffusion rates and Neutron computed tomography (NCT) to detect grain boundary diffusion rates in forsterite ( $Mg_2SiO_4$ ), a form of olivine, and hyrogarnet (katoite -  $Ca_3Al_2(OH)_{12}$ ). These data will be compared and contrasted with laboratory conductivity measurements to interpret magnetotelluric measurements.

## New Electrode Materials for Lithium & Sodium Ion Batteries

### Qingbo Xia, University of Sydney



With increasing energy demand, energy storage has become a growing global concern over the past decade. Electrochemical energy storage (EES) technologies based on batteries are beginning to show considerable promise as a result of many breakthroughs in the last few years due to their appealing features include high round-trip efficiency, flexible power, and energy characteristics to meet different grid functions, long cycle life, and low maintenance.

My PhD project focuses on the discovery, characterization and optimization of electrode and solid electrolyte materials in both lithium ion batteries and sodium ion batteries, in which the investigation of materials nuclear & magnetic structures and the dynamics of Li/Na ion is a key issue. Three techniques have been heavily utilizing to theoretically and experimentally characterize the new electrode candidates. Firstly, *ab initio* calculations, employed to identify and compare the energies of framework structures with hosting Li/Na from materials data mining, which give an improved understanding of how the experimentally determined structures arise and how they will evolve with mobile ion concentration under electrochemical cycling. Knowledge of the ground-state

magnetic structure also permits the accurate calculation of redox potentials, in conjunction with electrochemical measurements. Secondly, Neutron scattering investigations on new crystalline materials for light metal-ion batteries are performed. Neutron diffraction reveals the location and occupancies of Li/Na sites in the crystal lattice, and hence conduction pathways. In situ experiments explicitly reveal Li/Na ion mobility, as well as phase changes under operating conditions that undermine long-term stability. Inelastic and quasielastic neutron scattering probe the dynamics of the mobile ions and the supporting lattice. Besides, low-temperature neutron diffraction reveals the spin-ordered ground states of the transition metal counter-cations, which are not only fundamentally fascinating due to their complex super-super-exchange pathways, but also characteristic of their electrochemical states in batteries. Thirdly, *in-situ* TEM characterization is performed to study materials degrade on a larger scale over repeated cycling: nanocrystallisation, and changes in the roughness of the interfaces. The information of the materials failure collected by virtue of this technique will help to effectively design the accurate ways to optimize the materials.

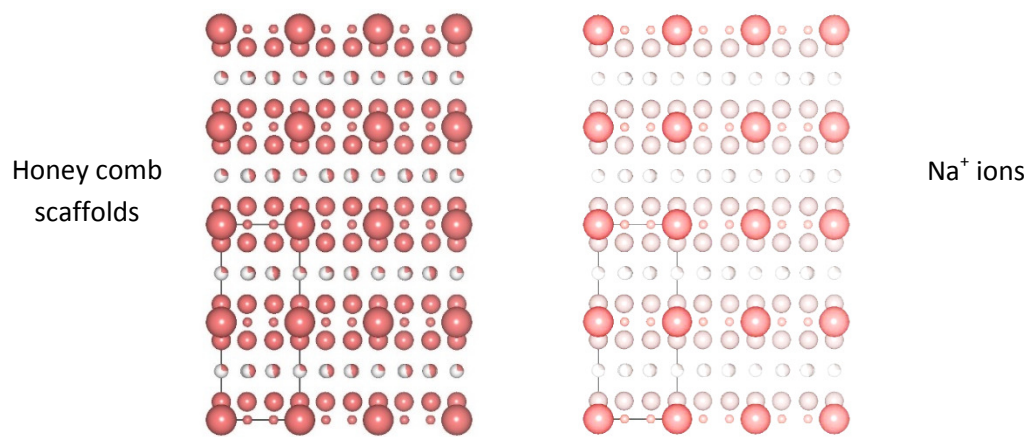
## **Cathode Materials with Layered Structure for High Performance Sodium Ion Batteries**

**Jiatu Liu, University of Sydney**

Sodium ion batteries are believed to be complementary to lithium ion battery in near future for a sustainable society relying on green energy. Sodium provides an economically pragmatic option for large scale energy storage. And the pursuit for higher capacity/energy electrode materials for sodium ion batteries never ceases.

Among a vast variety of candidate structures, layered materials show the greatest potential due to their stability during cycling and moderately high voltage. These layered materials are used in personal computers, cell phones and Tesla vehicles. They can be thought of as sheets of scaffolds stacking together between which mobile ions are found. These scaffolds are able to change their charge states, then incorporate or lose Li/Na ions during discharge or charge. Compared to the lithium counterparts, scaffolds that hold sodium ions appear to be more stable because the inter layer distance is larger and they distribute the sodium differently. That being said, the reason for the inevitable degradation of these scaffolds has eluded researchers ever since they gradually grow more interests in sodium ion battery.

Here at The University of Sydney, we are focusing on the synthesis and characterisation of layered structure materials containing inactive 5p elements which we believe will prove to be a class of high performance cathode materials with long cycle life and high energy output. These inactive heavy elements, together with active light elements such as nickel and cobalt can form interesting honey-comb structures within the scaffolds of the layered material, providing stability and high operating voltage. The large atomic number contrast between those in and out of the scaffolds, i.e. sodium ions, makes it difficult for X-ray diffraction to detect sodium in the presence of heavy atoms. Neutron diffraction, however makes that easier because the neutron scattering powers of both heavy and light atoms do not vary linearly with atomic number. By monitoring how the neutron diffraction patterns of the electrode materials vary at different states of charge and at different levels of cycling, we hope to understand possible degrading mechanisms of this class of materials and to demonstrate the advantage over other layered structure materials.



Layered structures “seen” by neutron diffraction (left) and x-ray diffraction (right). The brightness of each atom stands for the scattering power which equals to the diffraction intensity to some extent. The heavier the atom leads the higher X-ray scattering. For Neutron, there is no linear relation between atomic number and the scattering power, allowing more accurate information of light atoms to be detected.