

Australian X-ray Analytical Association

Newsletter Issue 3-2021

President's Address

Dear AXAA members,

Thank you to all who were able to join us recently for the AXAA Annual General Meeting on 4th November. One of the items discussed was the idea of teaming up with another society to deliver our next National Conference. Preliminary talks with the Society of Crystallographers in Australia and New Zealand (SCANZ) indicate they are similarly excited by this possibility, and as a committee we have agreed to explore this further with our SCANZ counterparts over the coming months. I'm very excited about the prospect of an in-person event and re-connecting with everyone again!

Another initiative we are currently working on is compiling a database of X-ray laboratories across Australia. This information will be hosted on our website to help the community find techniques and capabilities to suit their needs. A second initiative is to collect information via a voluntary survey about various laboratory metrics e.g. modes of access, funding structure models, staffing levels etc. This information would be made available to laboratory managers who would like to know how their laboratory metrics compare to the national averages, and may provide a useful source of data for funding justifications for example. We intend to repeat the survey every few years to track long term trends across the nation. This idea is still at a conceptual stage, and it would be wonderful if you could please contact Valerie Mitchell (ausxray@gmail.com) to offer your suggestions and ideas about what information would best serve your laboratory. We are hoping to collect input and feedback from as many people as possible as we develop the survey.

This year we ran a series of online seminars with the most recent held on November 29th.



Dr. Kang Liang, UNSW

Dr. Kang Liang of the University of New South Wales detailed his innovative work incorporating proteins within metal organic frameworks (MOFs) and showcased how small angle scattering (SAXS) techniques are used to investigate pore sizes and structure of

these materials. One fascinating application of this was the self-assembly of MOFs within plant tissues, which can incorporate toxic metals within the plant. The MOFs then fluoresce to indicate the presence of metal, and concentration can even be determined via an app!

Dr. Debashish Mazumder and Dr. Jasmin Martino from ANSTO then described their industry-leading work in seafood provenance. While provenance is traditionally determined using isotopic and XRF fingerprinting



Dr. Debashish Mazumder, ANSTO

with lab based equipment, they have now shown that portable XRF instruments can be used to fingerprint the source of seafood with good accuracy. By proving their models within one of the most difficult industries (fish can range over large areas), they are now expanding this model for different types of seafood, as well as other industries like fruits and Australian bushfoods. This is an exciting example of how thorough characterisation work can lead to very tangible outcomes for the ecosystem and local businesses.

Summer is finally here and I hope for many of us that means holidays, reconnecting with family and friends, and a well-deserved break. Bring on 2022!

Jessica Hamilton

AXAA President



Determining the Long-Range Average and Short-Range Local Structure of Pyrochlore Oxides Using a Combination of X-Ray Diffraction and Spectroscopy

Bryce Mullens

The University of Sydney

Introduction

Pyrochlore oxides are an important class of materials of the form $A_2B_2O_7$, where A and B are cations. Due to their compositional and structural flexibility, pyrochlores have found numerous applications in catalysis, ferroelectrics, ferromagnetism, luminescence, solid-oxide fuel cells, and as host matrices for the immobilisation of actinide-rich nuclear wastes. An extensively studied class of pyrochlore oxides are the '2:2:7' oxides $(A_2B_2O_7)$ containing trivalent A-site rare-earth metal cations (such as Sc³⁺, Y³⁺, and trivalent landanoid species Ln³⁺), and tetravalent B-site Group IV transition metals (such as Ti⁴⁺, Zr⁴⁺, and Hf⁴⁺). However, the A₂O₃-BO₂ solid-solution series may be extended by adding excess A₂O₃, transitioning from a 2:2:7 pyrochlore to the 2:1:5 'stuffed' pyrochlore (A_2BO_5) . Considerably less is known about these

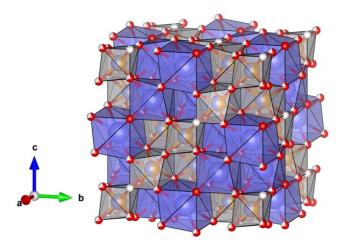


Figure 1: The $A_2(B_2.xAx)O_{7-x/2}$ 'stuffed' pyrochlore with two different metal cations being substituted across a single crystallographic site (denoted as different coloured spheres) and the oxygen ions (and their vacancies) denoted as red and white spheres respectively.

stuffed pyrochlores, and their structure is typically viewed as more disordered than the ideal 2:2:7 pyrochlore (Figure 1).

Pyrochlores have been proposed as solid electrolytes for solid-oxide fuel cells due to their high oxygen defect concentration and the mobility of the oxygen vacancies. Oxygen vacancies give a pathway for oxygen ions to move throughout the structure *via* low-energy interstitial sites. Previous studies have shown a complex relationship between the disorder present within pyrochlore structures and their maximum oxygen ionic conductivity. Debate continues as to whether creating additional oxygen vacancies within the pyrochlore structure (in the form of stuffed pyrochlores) increases or decreases their oxygen ionic conductivity.

This work considered the effect of longrange (average) and short-range (local) order in the series $Ln_2(Ti_{2-x}Ln_x)O_{7-x/2}$ (Ln = Tm, Yb). In particular, the importance of observing structural changes and disorder at different length scales has been previously demonstrated, with different studies on the same material sometimes reaching contradictory conclusions. Therefore, a combination of synchrotron X-ray and neutron powder diffraction was used to determine the average long-range structures, and X-ray absorption near-edge structure (XANES) spectroscopy was used to determine the evolution of the local structure. This was the first comprehensive study to relate the evolution of structural disorder with the oxygen ionic conductivity of stuffed pyrochlore oxides.

Methods Section

Samples with the composition $Ln_2(Ti_{2-x}Ln_x)O_{7-x/2}$ (x = 0.00-0.67) were prepared using a conventional solid-state synthesis route. The metal oxide reagents were dried at 900 °C for 16 hours to remove any adsorbed water and/or carbon dioxide. Stoichiometric amounts of each reagent, sufficient to prepare 8 g samples, were ground in a mortar and pestle with acetone and transferred to alumina crucibles. The samples were pre-annealed in air at



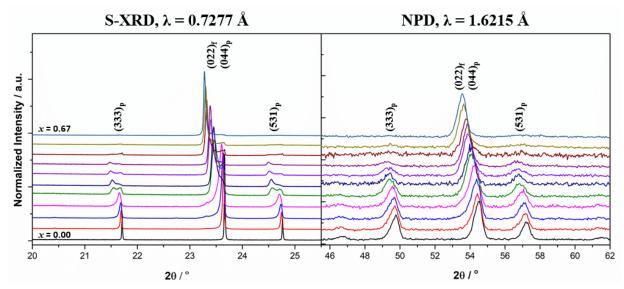


Figure 2: Plots of the pyrochlore superlattice reflections $(333)_p$ and $(531)_p$ along with the main $(044)_p/(022)_f$ peak from the synchrotron X-ray (left) and neutron powder (right) diffraction of the $Yb_2(Ti_2.xYbx)O_{7-x/2}$ series.

1000 °C for 24 hours before being re-ground and pressed into rods using a hydrostatic press at 200 bar for 10 mins. The rods were sintered in air at 1500 °C for 24 hours, before being cooled at 0.1 °C \min^{-1} to 1000 °C, then at 4 °C \min^{-1} to room temperature.

Synchrotron X-ray diffraction data was collected at room temperature on the X-ray powder diffraction beamline at the Australian Synchrotron. Neutron powder diffraction measurements were performed on the high-resolution powder diffractometer Echidna at the Open Pool Australian Lightwater (OPAL) reactor, operated by the Australian Nuclear Science and Technology Organisation (ANSTO). The Tm M_{5,4}-, Yb M_{5,4}-, Ti L_{3,2}-, and O Kedge XANES spectra were collected on the soft X-ray spectroscopy beamline at the Australian Synchrotron.

Results and Discussion

The pyrochlore structure displays ordering between the A^{3+} and B^{4+} cations, as well as anion/vacancy ordering. Such ordering gives rise to the appearance of additional 'superlattice' reflections in the diffraction patterns. As shown in Figure 2, the intensity of the superlattice peaks in the Ln_2 (Ti_{2-x} Ln_x)O_{7-x/2} series decreased in both the X-ray and neutron diffraction patterns with increasing

 Ln^{3+} content (increasing x). Upon substitution of further Ln^{3+} cations, an abrupt loss of ordering in the cation sublattice was observed from x=0.335. This was shown to be an evolution from the highly ordered pyrochlore ($A_2B_2O_7$) structure to the disordered defect-fluorite (A_2BO_5) structure.

The Ti $L_{3,2}$ -edge XANES spectra can be separated into three regions: pre-edge (peaks A and B), the L_3 -edge (peaks C and D) and the L_2 -edge (peaks E and F). As shown in Figure 3, there is a notable change in the line shape of peak D across each series, with the peak being split into a well-defined asymmetric doublet at the pyrochlore (x = 0.00)

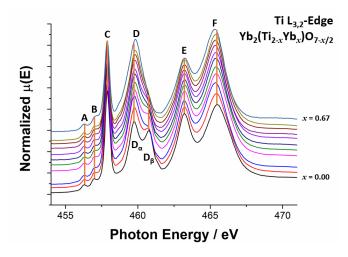


Figure 3: Normalised XANES spectra at the Ti $L_{3,2}$ -edge for $Yb_2(Ti_2.xYbx)O_{7-x/2}$. Each spectrum has been offset vertically to enhance visibility.



end. As the Ln^{3+} content increases, the doublet gradually merges, indicating a disordered coordination environment around the Ti^{4+} cations.

The energy difference between peaks C and D is directly related to the crystal field splitting (CFS) energy of the ${\rm Ti}^{4+}$ ion and provides further information on the coordination environment of the ${\rm Ti}^{4+}$ cations. As ${\it Ln}^{3+}$ content increases, the environment around ${\rm Ti}^{4+}$ evolves from a distorted ${\rm TiO_6}$ octahedra to the seven-coordinate scalenohedral present in the defect-fluorite structure. Therefore, despite the long-range average diffraction studies displaying a sudden onset of cation disorder, the short-range local spectroscopy studies display a gradual disordering across the series. This reflects the gradual evolution of disorder at different length scales throughout these materials.

Conclusions

Synchrotron X-ray and neutron diffraction data displayed a loss of pyrochlore-like ordering in the cations across the $Ln_2(Ti_{2-x}Ln_x)O_{7-x/2}$ series, with the disappearance of superlattice reflections with increasing Ln³⁺ content. In contrast to the sudden development of the structural transitions revealed by diffraction, XANES analysis demonstrated the gradual evolution of short-range local disordering throughout the entire composition range. This is most prominent in the Ti L_{3,2}-edge, with a gradual change in the geometry of the Ti⁴⁺ ions observed with increasing Ln^{3+} content. This is consistent with the Ti geometry changing from a distorted octahedra to a distorted scalenohedral. This work highlights the advantages of using both diffraction and spectroscopic techniques when quantifying disorder in metal oxide systems.

References

[1] Mullens, B.G.; Zhang, Z.; Avdeev, M.; Brand, H.E.A.; Cowie, B.C.C.; Saura-Múzquiz, M.; Kennedy, B.J.; Effect of Longand Short-Range Disorder on the Oxygen Ionic Conductivity of Tm₂(Ti_{2-x}Tm_x)O_{7-x/2} 'Stuffed' Pyrochlores. *Inorg. Chem.*, **2021**, *60* (7), 4517-4530.

[2] Mullens, B.G.; Zhang, Z.; Avdeev, M.; Brand, H.E.A.; Cowie, B.C.C.; D'Angelo, A.; Saura-Múzquiz, M.; Kennedy, B.J.; Average and Local Ordering of $Yb_2(Ti_{2-x}Yb_x)O_{7-x/2}$ 'Stuffed' Pyrochlores: The Development of a Robust Structural Model. *J. Solid State Chem.*, **2021**, *302*, 122412.

CHOS gas/fluid-induced reduction in ureilites

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¹ Monash University ² Curtin University ³ CSIRO Mineral Resources ⁴ University of Plymouth

Introduction

Ureilite meteorites contain regions of localized olivine reduction to Fe metal widely accepted to have formed by redox reactions involving oxidation of graphite, a process known as secondary smelting [1,2]. This process is highly pressure dependant and requires that the ureilite parent body (UPB) to have been catastrophically disrupted for both the large, equilibrated cores of olivine and pyroxenes to grow, and for other textures, such as the fine reduced rims and uninverted pigeonite in the same meteorite [3]. However, since the inception of this solid-state reaction mechanism, other pathways to olivine rim reduction have been largely ignored. Here, we have investigated the likelihood of solid/fluid reactions to produce these textures.

Methods

Seventeen ureilite samples were examined with optical microscopy, scanning electron microscopy (SEM) and electron probe microanalysis (EMPA) and mapping. Energy- and wavelength-dispersive spectral mapping was conducted using the JEOL 8500F and JOEL 8530F-CL Hyperprobes at CSIRO Microprobe Laboratory. X-ray and CL Spectra were extracted with the in-house software Chimage. Additional BSE and X-ray element mapping was conducted at Plymouth Electron Micros-



copy Centre using a JEOL 7001F FEGSEM equipped with an Oxford Instruments X-Max 80 mm² energy dispersive spectra (EDS) detector.

Chemical equilibrium modelling was conducted to evaluate the position of the C-CO, CH₄-C and olivine-pyroxene-iron (OPI) buffers across the ranges of olivine and pyroxene compositions observed in ureilites.

Results and Discussion

All coarse-grained ureilites in this study contained extensive smelted margins. However, contrary to previously described textures, we observed fine reaction front plume structures, smelting regions well removed from graphiteolivine boundaries and the presence of significant troilite in smelting margins (Fig. 1). Smelted regions also contained the fine porosity often seen in volume-reducing fluid-rock reactions [4], common in terrestrial hydrothermal systems. Element mapping also indicated the presence of Fedepletion around fine fractures deep within olivine cores, indicating that the fluid pressure within the fragments of UPB after disruption was high enough to fill all available space. In addition, CL imagery showed that regions of high C-content sometimes luminesced in diamond wavelengths. We propose that this fluid was comprised primarily of methane as an active phase with variable amounts of H2S (Fig. 2). Modelling of this fluid shows that smelting can occur at lower temperatures and higher pressures than solid-state reactions. Methane-based smelting is highly dependent on the water content of the fluid, with smelting being able to occur below 20% H₂O.

Methane-rich fluid infiltrating into small cracks offers a mechanism for other textural features within the carbon phases of the ureilites. Graphite and diamond often co-exist with hydrocarbons [5]. Current mechanisms for smelting require temperatures at which hydrocarbons would be largely removed from the system before smelting, and therefore require a source of H to generate hydro-

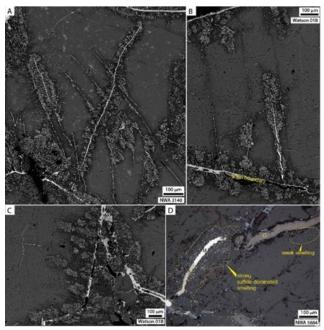


Figure 1. Smelting margins at olivine margins in ureilites displaying plumbous structure of reaction fronts.

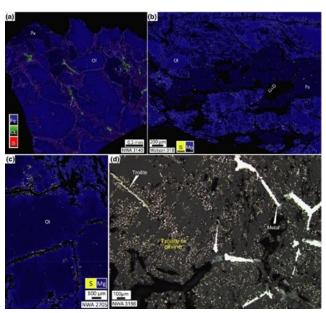


Figure 2. Smelted margins with anomalous sulphur. 2a displays CL from diamond and londsdaleite in smelted regions.

carbons at a later stage. Methane would be the most likely source of H in this system, as water content would inhibit smelting. Methane can be sourced through the differentiation process in forming planetesimals. H_2 can be sourced from silicate and metal-sulfide melt, and methane can be retained as a stable hydrocarbon at high P-T conditions. Should H_2 be the primary source of hy-



drogen within the system, reactions with other carbon phases can produce methane in a variety of simple reactions. H_2 -bearing fluids can deliver S for troilite formation through H_2 S-producing reactions.

Reactions that reduce Fe in olivine can also produce SiO₂ (Fig. 3). Elements maps in this study have shown that there is a significant increase in the relative concentration of Si in smelted margins. Some of this can be explained through conversion of olivine to pyroxene, however some silicate glass has been observed. This glass is likely remnant melt, and is mostly mafic, something not possible from smelting related SiO₂ generation.

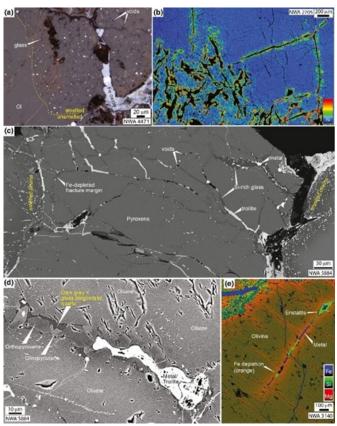


Figure 3. Silicate melt in ureilite smelted margins. Silica occurs in fine veins along fractures and grain boundaries where smelting-associated Fe-depletion has occurred.

Conclusions

The presence of fluid driven textures in ureilites indicates that the smelting reactions unique to ureilites are more complex than first thought. The presence of uninverted pigeonite requires a rapid cooling rate for all ureilites, howev-

er the presence of distinctive, fluid-based textures requires an alternative mechanism for generating smelted margins. In addition, the large range of polyaromatic hydrocarbons coexisting with diamond and graphite require the addition of H after these peak temperatures were reached. The presence of volatiles therefore allows for an additional mechanism for rapid cooling of the fragments of the UPB after disruption and indicate that volatile rich dwarf planets can retain significant volatile contents in their mantles despite considerable melt extraction unless disrupted by a catastrophic impact.

For the full article, please see:

Langendam, A.D., Tomkins, A.G., Evans, K.A., Wilson, N.C., MacRae, C.M., Stephen, N.R. and Torpy, A. (2021), CHOS gas/fluid-induced reduction in ureilites. Meteorit Planet Sci, 56: 2062-2082.

https://doi.org/10.1111/maps.13755

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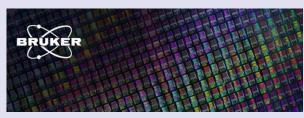
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Footnote: Andrew D Langendam is now at ANSTO Australian Synchrotron



AXAA Community News



Webinar-on-demand: **Introduction of Thin Film Structure Analysis**

Speaker: Dr. Wayne Lin, Business Development Manager, Thin Film Applications, Bruker AXS

Understanding thin film structures is one of the most important missions to modern material research. With advanced X-ray technologies, the details of the film structure can be revealed. This 60-min webinar will cover:

-Reciprocal Space Mapping (RSM)

What do we want to know about thin film structures?

-Grazing Incident Diffraction (GID)

The X-ray technologies for thin film structure determination

-X-Ray Reflectivity (XRR) -In-Plane Grazing Incident Diffraction (IP-GID)

-High-Resolution X-Ray Diffraction (HRXRD) -Thin film stress analysis

•How to make good X-ray measurements? Bruker's solution to thin film analysis

Register here to get the recording.





Live from the Lab: 2D X-Ray Diffraction - Watch it here.

2D XRD has become an important tool available on many X-ray diffractometers, but is it always the right tool for every job? We are going to go right to the source, one of the developers of modern 2D XRD theory, Bob He, to get his take. In addition, we will see how 2D is implemented on a typical multipurpose XRD solution.



I'm attending

26TH CONGRESS AND GENERAL ASSEMBLY OF THE INTERNATIONAL UNION OF CRYSTALLOGRAPHY

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As many will know, our colleagues at the Society of Crystallography of Australia and New Zealand (SCANZ) were successful in their bid to host the 26th International Union of Crystallography Congress in Melbourne. Preparations are well underway and all AXAA members are invited to register your expression of interest in the congress at www.iucr2023.org. Many of the AXAA community will have attended IUCr congresses before and know the scale of undertaking - the event is hoping to shine bright light on structural science in our region. The local organising committee of IUCr23 does include several AXAA members and will be calling for more involvement from the community in outreach activities as we get closer to congress. So, mark your diaries and let's get as many people as we can to Melbourne in 2023!



AXAA Community News



ACMM27 has been rescheduled for 30 January –3 February 2022, to be held at the University Club of Western Australia!

The 27th Australian Conference on Microscopy and Microanalysis brings together colleagues from life sciences and physical sciences who are working together towards new visions and goals in microscopy. Watch this space for more updates!

Registration for the biennial **Reynold's Cup** competition is now open! Test your skills at quantitative phase analysis of sedimentary rock compositions, and possibly win the coveted champion's trophy!

Hosted by the Clay Minerals Society and the German-Austrian-Swiss Clay Group, the competition is free to enter and open to all.

Find more information here





Applications for AINSE 2022 Honours Scholarships are NOW OPEN! Apply now to receive a A\$5,000 stipend if you are enrolled in an Honours program at an AINSE-member university from 1 January-2 March 2022 and undertaking research with ANSTO

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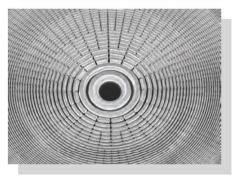


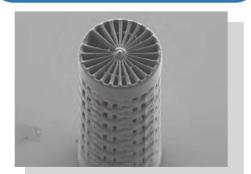
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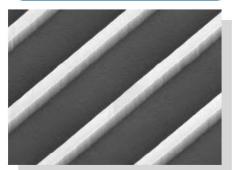
Zone Plates

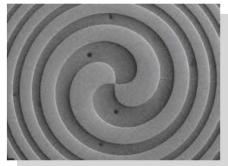
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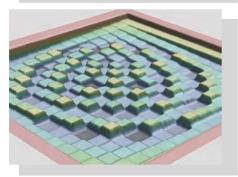
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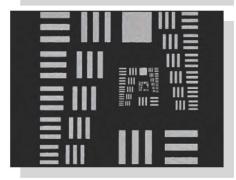












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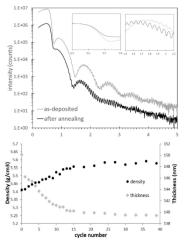
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Malvern Panalytical's "Ask an Expert" is back and we focus on thin film studies – namely PCRAM (Phase Change Random Access Memory). PCRAM is considered the next generation of memory storage given its simple device structure with a short write/erase time. Materials like germanium antimony telluride (Ge2Sb2Te5) are used in such memory storage devices and are considered as reference compounds because of their excellent properties with respect to crystallization speed, crystallization temperature and/or retention time. In phase change memory, Phase Change of a Material (PCM) from amorphous state to crystalline state is used for the set and the reset processes. The phase change is followed by a change in electrical resistivity of several orders of magnitude. This drastic electrical change is used to store bits of information. In spite of extensive studies, phase change mechanisms, and specifically volume shrinking behaviour during crystallization, are not fully understood. In this application note, expand your knowledge on

- How to access structure, strain or stress state and volume shrinking information.
- Using combined in situ X-ray diffraction (XRD) and X-ray reflectivity (XRR) experiments on PCM thin films

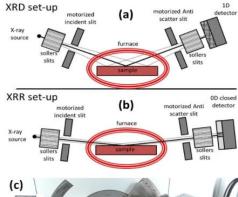
Results and implications



The comparison of the XRR curves reveals some significant density changes after isothermal annealing. A clear shift in the total reflection critical angle (wc) is observed at 2θ range [0.5°-0.8°]. The Kiessig thickness fringes also exhibit a period change between the initial and the final state after isothermal annealing. Zooms in 2θ range [1.5°-2.1°] as shown reveals a fringe's period reduction which is the result of a thickness reduction.

Observe the gradual evolution of the GST film during the isothermal annealing at 100°C. The main findings show that upon annealing, the crystallization induces a considerable tensile strain build up in the PCM film. Afterwards, a partial strain relaxation occurs and is related to grain growth and viscous flow. Concomitantly, volume shrinking is observed through densification and thickness reduction. The in-situ

combination of XRR and XRD techniques is powerful in understanding the mechanism during the amorphous to crystalline phase transition. This opens new pathways to engineer volume shrinking into PCM and then to help the design and improve the reliability of high performance PCRAM memory devices.





Incident beam optics for XRR Programmable divergence slit 1/32°, Soller slits 0.04 rad Programmable divergence slit 1/4°, Soller slits 0.04 rad Programmable divergence slit 1/4°, Soller slits 0.04 rad Anton Paar HTK 1200, secondary vacuum (turbo-molecular pump, -5 Pressure below 10 mbar) Programmable anti scatter slit 1/32°, OSO Soller slits 0.04 rad, PIXcel detector, receiving slit mode, 3 active pixels strips Programmable anti scatter slit open, Soller slits 0.04 rad, PIXcel detector, scanning mode		Source	Tube Ceramic Cu fine focus λ=1.5406 Å on a Malvern Panalytical Empyrean XRD
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Bruker AXS' X-ray Diffraction and X-ray Microscopy solutions for Battery Research



Tackling the global challenges of energy generation and storage require some of today's most complex materials developments. Read on here to find out how X-Ray Diffraction (XRD) sheds light on the workings of energy storage materials: bit.ly/BRKRbattery

The performance of any battery, whether in terms of its capacity, lifetime of energy density, is ultimately down to the intrinsic properties of its anode, cathode and electrolyte. X-ray powder diffraction is ideally suited to investigate the structure of novel battery materials, which are typically produced in powder form. For these active compounds, the lattice parameters and details of their crystal structure can be determined through Rietveld refinements, along with information of the crystallite size which is related to the reactivity of a compound.

In-situ XRD experiments are a convenient way to map the phase diagram of a battery with respect to its charge state, and to correlate the different phases with the measured voltage profile. For this, a specialized battery cell for in-situ XRD is used that allows to probe the cathode (or anode) without disassembling the cell. Compared to ex-situ measurement, this avoids relaxation of metastable phases as well as degradation and contamination of the active material. In-situ measurements also significantly increase the speed at which phase-diagrams can be established, as the same sample can be used to explore multiple charge states.

One important question in battery research concerns the reaction mechanism of the intercalation of Li-ions in the active compound. To study electrochemical processes "live", the battery cell is connected to a potentiostat, which drives the charge-discharge of the cell during the XRD experiment. Using in-operando measurements, researchers can follow the emergence of new phases at different charge states and determine whether a reaction takes places as an intercalation in a solid-solution (single-phase) or via multiple intermediated phases. In addition to a fundamental understanding of the cathode (or anode) material, in-operando XRD/ electrochemistry experiments can also rapidly identify critical voltage limits for phase-transitions with detrimental impact on the cell's capacity.

Watch our event-on-demand here to learn more.

Scan QR code or visit bit.ly/batteryday2021





AXAA Website and Contacts

Please visit our website, <u>www.axaa.org</u>, for further information, or follow us on Twitter @axaa org.

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AXAA Membership

All registered participants of the AXAA-2017 conference are automatically granted AXAA membership for 3 years. Alternatively, new memberships can be obtained free of charge, by making an application to the National Council.

Candidates should send the membership form from the <u>AXAA website</u>, and a short statement about how they intend to contribute to the organisation, to the National Council Secretary Anita D'Angelo.

AXAA Resource Centre

There are a range of resources available on the <u>AXAA website</u>, including video recordings of the two Public Lectures at AXAA-2017, tips for Rietveld Analysis, Clay Analysis, XRF tips, and more. We welcome further contributions to our Resource Centre.

Next AXAA Newsletter

The next issue of the AXAA Newsletter will be distributed in April 2022. Please feel free to send contributions for the newsletter to Valerie Mitchell at ausxray@gmail.com. Any comments or feedback about the Newsletter are welcome.

A Day in the Life of an X-ray / Neutron Scientist

We are seeking posts for our 'Day in the Life' series. If you'd like to contribute, or know someone who might be interested, please contact National Council

