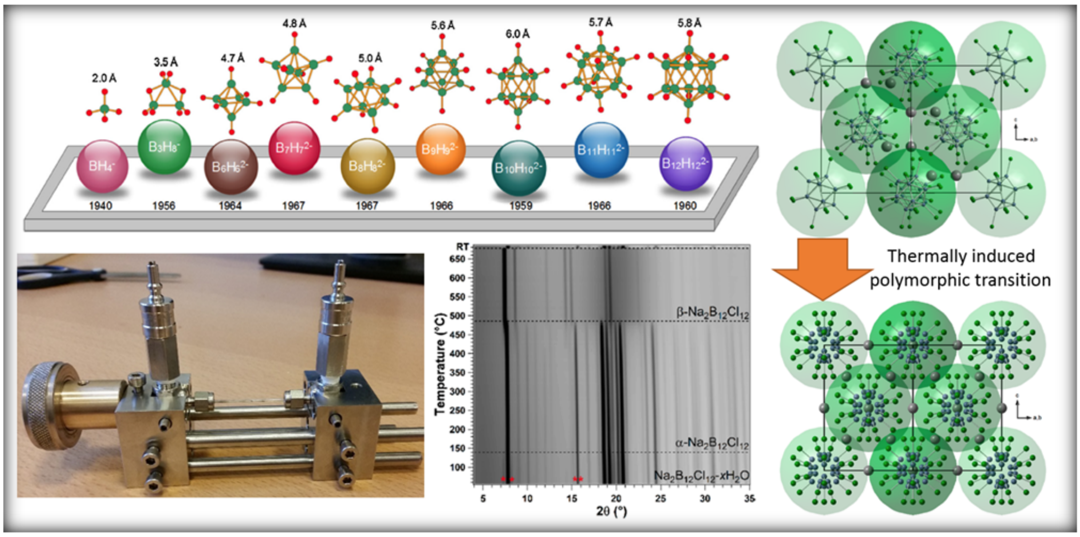
Curtin University

CURTIN X-RAY FACILITIES
WAVELENGTH

Quarterly Newsletter – May 2017 Edition



Feature image: *Tracking polymorphic transitions of metal boranes via in situ X-ray diffraction (article on page 3).*

IN THIS ISSUE

John de Laeter Centre

Welcome to the first edition of Wavelength – A quarterly newsletter dedicated to highlighting the latest news and research produced from Curtin's X-ray characterisation facilities, namely the X-ray photoelectron spectroscopy (XPS), X-ray diffraction (XRD) and small angle X-ray scattering (SAXS) facilities. As a special first edition feature, we have included an entire page that highlights the basic instrument capabilities of each facility.

News from the Facilities

General News:

- The deadline for the Australian Synchrotron call for proposals for round 3 is fast approaching (May 23). Please contact one of us (contact details on page 3) if you require assistance with applying for beamtime.
- A new seminar series (Materials Science @ Curtin) is being established to showcase the novel materials-related research being conducted at Curtin. Much of the research relates to data obtained using the Curtin X-ray Facilities. The first seminar is sponsored by the Australian X-ray Analytical Association (AXAA) and will be held from 12-1pm on Wednesday May 31 in the Shilbury lecture theatre (B204, R233). *Contact Will Rickard for further information.*

X-ray Photoelectron Spectroscopy (XPS) Facility:

- The XPS facility will be holding an XPS Software Analysis Training Day at Curtin – June 23: Learn how to use CasaXPS. *Contact Jean-Pierre Veder to book a spot.*
- Murdoch University is hosting a Vacuum Technology Short Course presented by the Vacuum Society of Australia – May 25. *Contact Jean-Pierre Veder for details.*

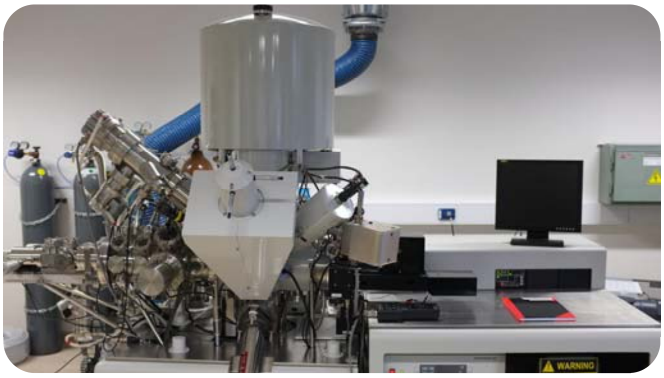
X-ray Diffraction (XRD) Facility:

- Submissions for the PANalytical Award 2017 are now open. If you have used X-rays for your research and analysis on any brand of X-ray instrument, you are invited to submit your paper for the chance to win a €5,000 cash prize (approx. $7,200). *Contact Veronica Avery for further information.*
- Will Rickard was recently elected as an AXAA committee member. He will be representing Curtin and the WA XRD community until 2020.

Small Angle X-ray Scattering (SAXS) Facility:

- The SAXS facility is up and running and actively seeking new users. If you have nano-sized features in your materials, be they hard or soft, SAXS may be able to help you out. Contact *Matthew Rowles* for further information.

X-ray Photoelectron Spectroscopy (XPS)



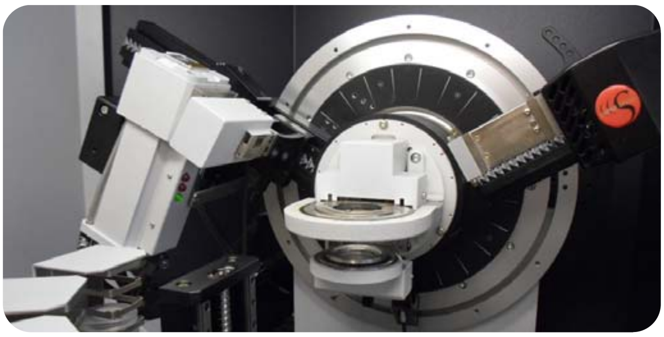
Kratos Axis Ultra DLD X-ray photoelectron spectrometer.

X-ray photoelectron spectroscopy (XPS) is a non-destructive, surface sensitive technique that is used to investigate the elemental and chemical composition of material surfaces. A sample is exposed to X-ray radiation under ultra-high vacuum conditions and the photoelectron spectrum is recorded using a hemispherical analyser situated above the sample.

In a typical experiment, a survey spectrum is first acquired to semi-quantitatively determine the elemental composition of the sample's surface. High-resolution spectra for the core levels of each element are then acquired as they reflect the chemical environments of those elements. For example, in a carbon-based sample, the high-resolution carbon spectra can be used to easily quantify the extent by which carbon is bonded to other carbon atoms, oxygen atoms, hydroxide groups, etc. Similarly, this same treatment can be applied to all other elements detected in a sample.

The XPS facility is also equipped to provide a range of other surface analysis techniques, including ultraviolet photoelectron spectroscopy (UPS), auger electron spectroscopy (AES), and ion scattering spectroscopy (ISS). These techniques combine to provide a detailed perspective of the elemental and chemical composition of the surface down to a depth of approximately 5 nm.

X-ray Diffraction (XRD)



Bruker AXS D8 Advance powder X-ray diffractometer.

XRD is an analytical technique that uses X-rays to investigate and quantify the crystalline nature of materials. This is achieved by measuring the diffraction of X-rays from the planes of atoms within the material of interest. The technique is primarily used to identify the crystalline content of materials, identify and quantify the crystalline phases present and also determine the spacing between lattice planes (d-spacing).

Curtin's XRD laboratory services the powder X-ray diffraction needs of our industry and research community. Samples can include minerals, metals and most other organic and inorganic materials. XRD data is collected using either the Bruker AXS D8 Advance (copper radiation source) or D8 Discover (cobalt radiation source) powder diffractometers which are fitted with a position sensitive (LynxEye) detector. This detector is 200 times faster than a conventional scintillator detector, resulting in faster data collection times. The XRD data collected can be analysed using the Diffrac EVA software for phase/mineral identification and TOPAS for phase/mineral quantification and crystallite size determination using the Rietveld method.

Small Angle X-ray Scattering (SAXS)

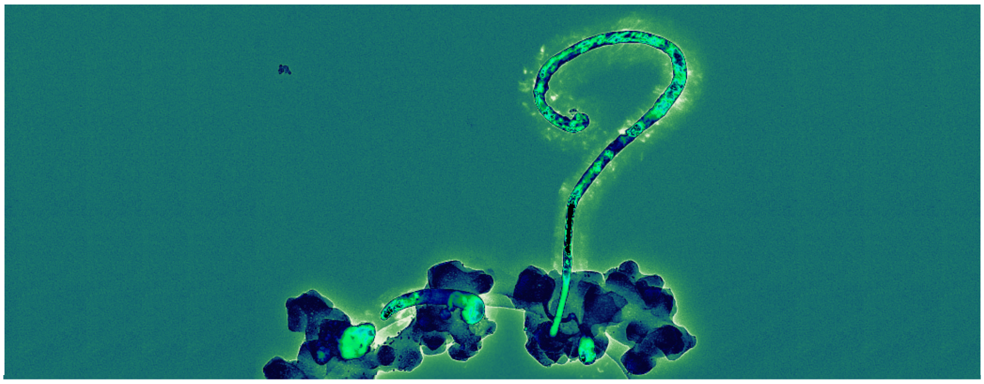
Bruker NanoStar small angle X-ray scattering instrument.

Small-angle scattering is an analytical technique for studying the structure of matter on the nano-scale. In the case of small angle scattering at Curtin, as the scattering angles are about 0.1 – 5° 2θ, the structures that are being probed are in the range of 6 – 600 Å. Small-angle scattering allows for the investigation of materials with structures on this length scale, such as nanoparticles, porosity, and protein structures.

The small-angle scattering instrument at Curtin is a Bruker NanoStar with a high-intensity gallium MetalJet X-ray source. This source, unique in Australia, allows for the very rapid collection of scattering data for high-throughput studies or *in situ* experimentation. The data collected is very good in its own right, and can also be used as justification for access to the Australian Synchrotron, where even higher X-ray intensities and energies are available.

SPOTLIGHT ON SCIENCE
RESEARCH TO BRAGG ABOUT

A feature page dedicated to highlighting some of the exciting research produced from the Facilities



Colourised Transmission Electron Microscopy (TEM) image of metallic Ag wire growth from $Ag_2B_{12}H_{12}$. [1]

Did You Know?

Thanks to a successful ARC LIEF bid, a new X-ray diffractometer is to be purchased for *in situ* studies of the type described in Dr. Mark Paskevicius's research.

Several manufacturers are being consulted, and it is expected to be ready by the end of 2017.

Not so Boring Boranes

Mark Paskevicius is investigating new metal boranes that could be used as solid-state ion conductors in next-generation batteries. Boranes typically consist of cage-like structures made from boron and hydrogen, which can be formed as anions in ionic salts. Remarkably, the borane anions typically undergo thermally induced disorder above a critical polymorphic phase transition temperature. Essentially the borane cages rotate and reorientate very rapidly. This reorientation of the anion is usually coupled by cation disorder, where a positive cation (such as Li^+ or Na^+) partially occupies crystallographic positions and can rapidly migrate, thus enabling fast solid-state ion conduction.

High resolution in-situ powder XRD data, collected during heating, was used to solve the crystal structures of metal boranes. It is particularly challenging to determine the crystal structures of the high temperature (highly disordered) polymorphs because they are metastable and transition back to the room temperature polymorph on cooling. They also exhibit rapid reorientational disorder of the anion that must be modelled as partially occupied borane cages, which are superimposed and rotated about their origin, along with partially occupied cation positions. Fortunately, the high temperature metal borane polymorphs tend to increase the crystal structure symmetry, often becoming cubic. Some metal boranes are unstable and can decompose thermally or under an electron beam (see figure above).

For further information, please refer to Mark's latest article:

[1] M. Paskevicius, B. Hansen, M. Jørgensen, B. Richter, T. Jensen, Multifunctionality of silver closo-boranes (2017), *Nature Commun.,* 8, 15136.

About the Researcher

Dr. Mark Paskevicius is a Senior Research Fellow in the Fuels and Energy Technology Institute at Curtin. He began this position in 2017 after a 3 year research fellowship at Aarhus University in Denmark. His research is focussed on renewable energy storage. He is dedicated to developing new materials for solid-state hydrogen storage to store energy for automotive, stationary and concentrated solar thermal applications. He is also leading research into new solid-state ion conductors for battery applications, focussing on boron-rich materials. Mark will be presenting his latest research at the Materials Science @ Curtin Seminar Series (12-1pm, May 31).

Recent Facility Publications

- Y. Cheng, S. Dou, J.-P. Veder, S. Wang, M. Saunders, S. P. Jiang, Efficient and Durable Bifunctional Oxygen Catalysts Based on NiFeO@MnO_x Core-Shell Structures for Rechargeable Zn-Air Batteries (2017) *ACS Appl. Mater. Interfaces*, 9, 8121-8133.
- H. Tian, S. Wang, C. Zhang, J.-P. Veder, J. Pan, M. Jaroniec, L. Wang, J. Liu Design and synthesis of porous $ZnTiO_3/TiO_2$ nanocages with heterojunctions for enhanced photocatalytic H_2 production (2017) *J. Mater. Chem. A*, Advance Article.
- J. Ke, J. Liu, H. Sun, H. Zhang, X. Duan, P. Liang, X. Li, M. O. Tade, S. Liu, S. Wang, Facile assembly of $Bi_2O_3/Bi_2S_3/MoS_2$ n-p heterojunction with layered n-Bi_2O_3 and p-MoS_2 for enhanced photocatalytic water oxidation and pollutant degradation (2017) *Appl. Catal. B: Environ.,* 200, 47-55.
- W. Tian, H. Zhang, H. Sun, M. O. Tade, S. Wang, Template-free synthesis of N-doped carbon with pillared-layered pores as bifunctional materials for supercapacitor and environmental applications (2017) *Carbon*, 118, 98-105.
- V. Sofianos, D. Sheppard, E. Ianni, T. Humphries, M. Rowles, S. Liu, C. Buckley, Novel synthesis of porous aluminium and its application in hydrogen storage (2017) *J. Alloys Compd.*, 702, 309-317.
- C. Su, X. Duan, J. Miao, Y. Zhong, W. Zhou, S. Wang, Z. Shao, Mixed Conducting Perovskite Materials as Superior Catalysts for Fast Aqueous-Phase Advanced Oxidation: A Mechanistic Study (2017) *ACS Catal.*, 7, 388-397.

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