



e-newsletter

Issue 3, 2011

Australian X-ray Analytical Association

President's Address

Dear AXAA Members and Readers,

Welcome to the October AXAA Newsletter. Firstly, my sincerest thanks to everyone for their support of me as one of the finalists for the Eureka Prizes People's Choice Award. I was very touched by the amount of effort that colleagues and friends went to for my bid. Unfortunately I didn't win, but enjoyed the process very much. It was a great opportunity to raise the profile of sustainable-energy research, and I got to speak to a wide variety of audiences about (mostly neutron) scattering techniques.

Spring is here! What better way to celebrate than by a new birth - a huge congratulations to AXAA member (and previous Vice President and Newsletter Editor) Dr. Catherine Kealley and her family on the birth of their new daughter.



Cat and Damien are pleased to announce the arrival of Sofia Marie. She was born at 2:40pm on Saturday 6th August. She weighs 3.22 kg (7 pounds 2 ounces) and is 51 cm long.

Also in this issue, we introduce a research highlight section into the Newsletter - the "Matters for Scatterers" section. Our first highlight comes from the University of Melbourne and the CSIRO. If you have something that you, or know someone who, would like to contribute to this section please send our Newsletter Editor Dr. Nathan Webster an email with the details.

Vanessa Peterson
National Council President

Eureka Prizes People's Choice Award

The Australian Museum Eureka Prizes celebrate the world-class achievements of Australia's top scientists, science educators and communicators. AXAA President Dr. Vanessa Peterson was nominated for the coveted 2011 People's Choice Award, for her work using neutron

scattering to find ways to store hydrogen for fuel cells and pave the way for a clean fuel future. Vanessa attended the gala awards dinner on 6th September in Sydney, attended by more than 900 of Australia's top scientists, decision-makers and celebrities. The award was won by Professor Alan Mackay-Sim for his research into spinal cord repair. However, Vanessa's nomination is testament to the high quality of her and her team's research, and helped to raise the profile of scattering techniques with a wide audience.



Dr. Vanessa Peterson at the 2011 Eureka Prizes Award Dinner.

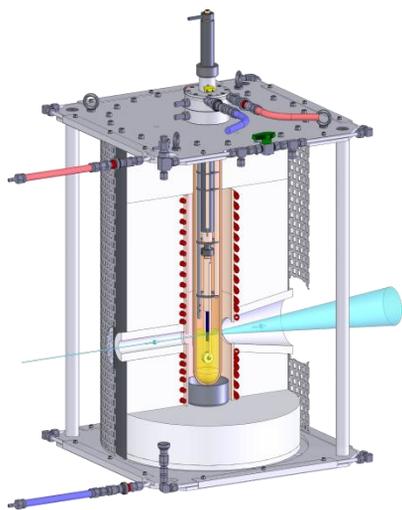
Matters for Scatterers

Molten salt electrolysis has for many years been an important process in the production of light metals such as aluminium, magnesium and more recently titanium. Despite the maturity of this technology, current electrowinning cell designs continue to rely on carbon-based anodes, which are rapidly corroded during electrolysis and require frequent replacement. In order to improve the efficiency of future electrowinning cells, an inert alternative to these carbon-based anodes is needed.

To date, it has proven difficult to identify an alternative material which exhibits both adequate corrosion resistance and sustainably high electrical conductivity, based on *ex situ* observations made prior to and following electrolysis. Information obtained in this manner is subject to experimental artefacts brought about by changes that can take place during cell shut-down and the preparation of samples for analysis.

In order to better characterise the performance of new anode materials, a novel *in situ* methodology has been developed which employs synchrotron-based (cont'd)

energy-dispersive X-ray diffraction (EDXRD). The advantages of this analysis technique include (i) high-energy X-rays capable of penetrating the walls of the cell, (ii) high spatial resolution which allows the anode to be selectively studied, and (iii) a very high intensity beam which allows reactions to be observed in real time.



Schematic diagram of the molten salt electrowinning cell used on the JEEP beamline at the Diamond Light Source.



Experimentalist loading an electrode stalk into the furnace.

To test this methodology, a specialised furnace and electrowinning cell were constructed, and an *in situ* experiment was performed on the Joint Engineering, Environmental and Processing (JEEP) beamline at the Diamond Light Source. The Fray-Farthing-Chen titanium electrowinning process was studied, using the Magnéli phase material Ebonex and a model inert anode. EDXRD patterns were collected every 60 seconds for 8 hours, during which time the Ebonex anode was gradually oxidised to the non-conductive rutile phase. The growth of this rutile surface layer was determined quantitatively using a novel, crystal-structure based analysis of the acquired energy-dispersive data. [*Journal of Synchrotron Radiation*, accepted for publication]

Mark Styles
The University of Melbourne/CSIRO

Powder Diffraction Workshop

Melbourne, 6-9 September 2011

The Powder Diffraction workshop, organised and run by the Australian Synchrotron Powder Diffraction beamline team (Kia Wallwork, Justin Kimpton, Qinfen Gu, Helen Maynard-Casely), covered experiment planning through to data analysis at both the Australian Synchrotron and OPAL facilities. The first two days consisted of lectures covering the basics of crystallography (James Hester, ANSTO), X-ray and neutron diffraction (Craig Brown, NIST; Michael James, ANSTO; Ian Madsen, CSIRO), proposal writing (Kia Wallwork, Australian Synchrotron), and data analysis including structure solution and refinement as well as phase identification and quantification. The final two days were tutorial based, allowing participants to gain experience with popular data analysis software such as CMPR (Kia Wallwork), GSAS (Craig Brown; Brendan Kennedy, University of Sydney), and TOPAS (Ian Madsen); and for those who had previously collected data at either the Synchrotron or Wombat/Echidna, this was a great opportunity to discuss analysis and troubleshoot problems with experts.

Thanks to everyone involved in organising and presenting the workshop – your enthusiasm and knowledge were appreciated by all the attendees – as well as the Australian Synchrotron, ANSTO, AINSE, and the ICDD for their support of this workshop.

Talitha Santini
The University of Western Australia

Science at Altitude: The 60th Annual Denver X-ray Conference

Does scientific performance increase at altitude? Should scientists train at altitude? I did not get an answer to these questions, but the foothills of the Rocky Mountains in Colorado Springs provided a scenic and high backdrop to the annual Denver X-ray conference. The first two days of the conference featured workshops encompassing various aspects of XRF and XRD, including Rietveld analysis, quantitative phase analysis, and high temperature experimentation to name a few. The following three days showcased examples of groundbreaking research and some tricky problems related to the workshops. There was a special session on energy storage and harvesting where I presented the work we have been doing with *in situ* neutron diffraction on Li-ion batteries, and scientists from Ford Motor Corporation, Advanced Photon Source, National Synchrotron Light Source and Sandia National Laboratories presented.

One of the highlights of the conference was a talk titled, 'edible nanostructures - the pleasures of chocolate' which illustrated how X-rays can be used to study chocolate!

The fat in chocolate is highly polymorphic and is a big influence on its properties, e.g. one polymorph melts at a temperature correlated to one's mouth. I also found out that something has to be 20 μm before we can taste it. Besides chocolate, a series of very interesting talks addressing a diverse range of problems were presented. I would recommend this conference for young researchers undertaking X-ray analysis.

Neeraj Sharma
The Bragg Institute, ANSTO



The township of Colorado Springs (in the distance) from Pikes Peak.

2011 IUCR Congress

It was a pleasure to escape the Melbourne winter for a short time and attend the XXII triennial Congress and General Assembly of the International Union of Crystallography, which was this year held in Madrid, Spain. As always the meeting provided a wonderful mix of scientific insight, culture and networking.

A significant Australian contingent was on-hand to talk up the latest developments in a wide variety of areas ranging from Nathan Cowieson's presentation on the application of Small-Angle Scattering (SAXS) to biology, to crystallography in industrial process control by Daniel Riley, and my own presentation relevant to cultural heritage applications of X-ray absorption spectroscopy and powder diffraction.



Dr. Kia Wallwork giving a presentation entitled *Investigations of Ancient Egyptian Faience using XAS and PD* (photo courtesy of Petr Bezdicka).

The IUCr Congress provided an opportunity to hear about the latest developments in diffraction data analysis, advances in experiment equipment to aid *in situ* experimentation and automation, and as always challenged us to expand our own research horizons. It was exciting to see that the trend that we see at the Australian Synchrotron to use powder diffraction as an integral tool for functional materials development is consistent with international trends. All this was complimented by various social opportunities including

the congress dinner, held in the grounds of the Botanical Gardens, which gave one and all an opportunity cement old collaborations and form new ones.

Kia Wallwork
Australian Synchrotron

Ed's note: Kia has moved into a new position at the Australian Synchrotron, and is now the Principal Beamline Development and Operations Advisor.

Membership Matters

We remind readers that AXAA-Inc membership is for the 3-year period starting from the last AXAA National Conference (existing membership will be re-approved in 2014). Membership is free. Candidates should provide their CV and a short sentence about what they intend to do in the organisation, and a nomination letter from an existing AXAA member (if possible). Please send these to National Council Secretary Natasha Wright. The council votes on membership at periods no longer than 6 months.

Job Announcement

Scientist – Powder Diffraction, Australian Synchrotron.

Closing date for applications: 28th October 2011.

<http://www.synchrotron.org.au/index.php/about-us/working-at-the-synchrotron/employment-opportunities/645-8-month-contract>

Upcoming Events

Internet XRF Course: Series 4, 2011

The course provides XRF analysts, particularly those new to the field, with on-site instruction in the practical principles of wavelength dispersive XRF. Features of course include -

- Start at any time, subject to the availability of places in the course.
- Self-paced instruction to accommodate the needs of busy people.
- Study materials transmitted as e-mail attachments in the form of 11 modules; with an assignment being set for each module.
- Feedback on the assignments provides excellent mentoring.

The course now has a substantial number of international participants, as well as Australians.

Course availability: Starting date by arrangement.

Course director: Dr Brian O'Connor

Course fee: \$2,420 including GST

Further information and enrolment:

brian_oconnor@iprimus.com.au Tel: (08) 9291 7067

Upcoming Events (cont'd)

7-25 November 2011

AXT and Rocklabs Equipment Expo
Unit 19 / 24 Baile Road, Canning Vale, WA
Further information: axtaw@axt.com.au

AXT plan to host an event for AXAA members during this time. Further details and information will be provided closer to the event.

27 November 2011

Australian Synchrotron Open Day
Australian Synchrotron, Clayton, VIC
<http://www.synchrotron.org.au>

5-7 December

17th AINSE Conference on Nuclear and Complementary
Techniques of Analysis
The Australian National University, Canberra
<http://ncta2011.anu.edu.au/>

8-9 December 2011

Australian Synchrotron User Meeting
Sebel Albert Park, Melbourne
<http://usermeeting.synchrotron.org.au>

AXAA Website and Contacts

<http://www.axaa.org>

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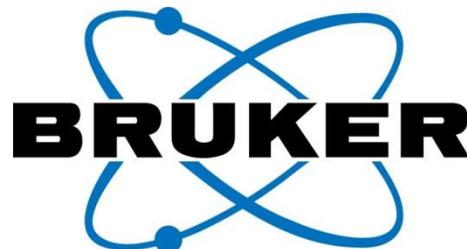
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Please email contributions to the final 2011 e-newsletter edition to Nathan Webster by Thursday 15th December. Any comments and feedback about the newsletter are welcome.

Advertising and Company News

Brand new high-resolution EBSD detector from Bruker

The e-Flash^{HR} is Bruker's high resolution, high sensitivity detector for electron backscatter diffraction analysis (EBSD) on electron microscopes. With close to 2 Megapixels resolution (1600 x 1200 pixels) in conjunction with high quality camera optics yielding minimum pattern distortion, the e-Flash^{HR} is the tool of choice to tackle the most demanding tasks in EBSD analysis, such as low beam current and low acceleration voltage applications, analysis of poorly conducting samples, nano-materials analysis, lattice strain investigation, phase identification and many more.



The e-Flash^{HR} complements Bruker's high speed EBSD detector version e-Flash¹⁰⁰⁰, further increasing the versatility of the Bruker high-performance CrystAlignTM EBSD system. Like its high speed counterpart, the e-Flash^{HR} detector provides vertical shifting of the fluorescent screen during electron microscope operation, allowing the analysis of both large and very small samples, at varying working distances and magnifications. The e-Flash^{HR} detector features a software-controlled, high precision motor drive with a travel length of 250 mm. Detector motion monitoring is integrated in a multi-level safety architecture which includes an LED screen position indicator, as well as visual and audio alarms. Should the phosphor screen accidentally come into contact with the sample or the stage, the detector automatically retracts. This protects the detector from damage and the user can easily replace the phosphor screen. (cont'd)

The eFlash^{HR} can be equipped with ARGUSTM, Bruker's unique foreshattered electron (FSE)/backscattered electron (BSE) imaging system. Consisting of 5 electron detectors arranged above and below the screen, ARGUSTM delivers real-time color FSE images with excellent grain orientation contrast, as well as BSE images for density contrast. The electron images provide very useful supplemental information about the sample under investigation.

The first two high-resolution EBSD detectors in Australia are currently installed at CSIRO in Western Australia.



Bruker eFlashHR

Diffraction Technology

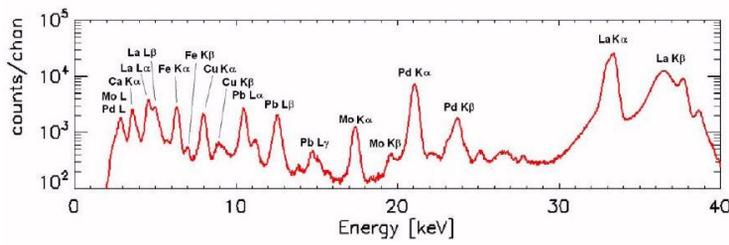
Instruments and accessories for X-ray analysis

AXO of Dresden, manufacturers of fine precision X-ray optics and Multilayers, introduce a new concept in standards - the

Thin Film XRF Reference Standard.

These have type Nos. RF4-100-S1749 and RF4-200-S1749 and were introduced at AXAA 2011. They consist of deposits of 7 elements in the ng/mm² range, to provide a wide elemental range with minimum overlaps and all of similar intensity. The elements are Pb, La, Pd, Mo, Cu, Fe, Ca, deposited on a Si Nitride substrate so Si is also present from the substrate.

Thin Film XRF Reference Samples
RF4-100-S1749 and RF4-200-S1749

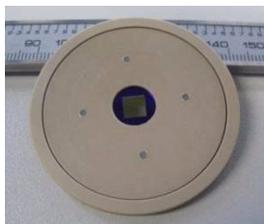


Energy spectrum of the 7-element reference sample RF4-200-S1749 measured at 40 keV excitation. The energy range from -2 keV to -40 keV is covered with peaks of comparable intensity.

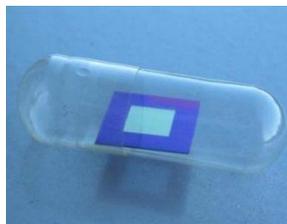
The standards are deposited on a SiN membrane 100nm or 200nm thick and each has only 1 – 3 atomic layers so they are "infinitely thin".

Deposited area is 5 x 5 mm and the Si frame is 10 x 10 mm

On PEEK holder



Bare membrane



**For more information and to obtain price and delivery Contact Rod Clapp (03 9787 3801).
Price Currently A\$1,500 F.I.S.
Delivery 4- 6 weeks.**



AXT & Rocklabs Equipment EXPO 2011

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- New RM2000 Mill
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- Optical Particle Sizer
- K2Prime XRF Fluxer
- Sample Press
- TESCAN SEM
- Bench Top EDXRF & WDXRF



AXT & Rocklabs Equipment EXPO

Date: 7 Nov to 25 Nov 2011

Time: 10 am to 4 pm

Venue: Unit 19 / 24 Baile Road
Canning Vale WA 6155

Contact: Julia (08) 9455 6111

Email: axtwa@axt.com.au

www.axt.com.au

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PANalytical expands with ultramodern X-ray tube factory in Eindhoven

14 September 2011, Almelo, The Netherlands, PANalytical, the global leading supplier of analytical X-ray instrumentation, services and software, has relocated its X-ray tube manufacturing to a new factory in Eindhoven, The Netherlands. PANalytical is the only supplier of analytical X-ray systems that also develops and produces its own X-ray tubes, which are an essential part of the instrumentation. The ultramodern Eindhoven facility has been built to further strengthen and exploit this technological advantage.

PANalytical, whose product range includes X-ray diffraction (XRD) systems and X-ray fluorescence (XRF) spectrometers as well as X-ray tubes, has outgrown its existing facilities in Eindhoven and is expanding into a showcase factory for the X-ray tubes division. The new factory which began production on 8 August 2011 features state-of-the-art technology to enhance the production of X-ray tubes and the quality and performance of its X-ray instruments and solutions.



The new PANalytical X-ray tube factory in Eindhoven

"We are excited to announce such a very important milestone in the evolution and visibility of PANalytical worldwide. This outstanding new building will enable us to consolidate our X-ray tube business and bring our employees together in a single, world-class, state-of-the-art factory that truly reflects the vision of our company. It will allow the X-ray tube division to optimize its operational effectiveness and efficiency as we execute our growth plans," said Peter van Velzen, CEO PANalytical.

"The Eindhoven area is the birthplace of several of the world's most brilliant high-tech companies, attracting engineers with a broad specialist knowledge in the field of X-ray physics, high voltage, vacuum, electron optics, materials science and process technology. By further strengthening its position as a technology leader and innovation facilitator, PANalytical leverages the dynamics of this region and ensures employment of highly educated personnel for the long term."

"PANalytical has great faith in the future and continues to invest in new tube technology and product development," continued van Velzen. "The three-storey building will break new ground in the field of X-ray tube manufacturing with its unique modern clean-room installations and efficient floor layout. The factory also includes light and spacious work areas that form a stimulating working environment, foster employee collaboration, and benefit health and well-being."

"Further, the new factory has been designed to contribute to sustainability and environmental care and complies with state-of-the-art building standards," said Maarten van An del, General Manager PANalytical Eindhoven. "We are proud to be moving to such an aesthetically pleasing property that contributes health and environmental benefits, not only to the employees who work here, but also to the surrounding community."



Empyrean wins prestigious 2011 R&D 100 Award

PANalytical's latest X-ray diffractometer selected by a renowned independent jury as one of the top high technology innovations of the year.

Almelo, The Netherlands, July 8 2011 – PANalytical, the world leading supplier of analytical instrumentation and software for X-ray diffraction (XRD) and X-ray fluorescence spectrometry (XRF), has announced that Empyrean, its newly designed X-ray diffractometer, has been recognized as a 2011 R&D 100 award winner. In their 49th year the R&D 100 awards are widely recognized as the 'Oscars of Innovation' identifying and celebrating the top high technology products of the year.

Awarded under the, 'winning technology' category, Empyrean has the unique ability to measure all sample types – from powders to thin films, from nanomaterials to solid objects – on a single instrument. Truly unique to Empyrean is the system's ability to see inside solid objects without having to cut them up. Using computed tomography (CT) analysis, it becomes possible to determine the area of interest for subsequent X-ray diffraction analysis, or check the presence of pores or inclusions inside the object.



Empyrean

"We are truly honored to receive this prestigious award," said Jan van Rijn, General Manager X-Ray Systems at PANalytical. "It is gratifying to be recognized for our commitment to developing new generation platforms, products and solutions that enable innovation within multiple industries, helping them to survive and thrive amidst today's challenges."

Empyrean is PANalytical's answer to the challenge of modern materials research. Coupled with a comprehensive software suite, Empyrean supports multiple users, unattended and remote operations, automatic data collection and analysis reporting. Today's research themes are nanomaterials, life sciences and renewable energy; tomorrow science may move in a different direction. The lifetime of PANalytical diffractometer stretches beyond the horizon of a single research program. For many scientists the ability to accommodate change is a unique 'must have' feature when deciding to invest in an XRD system. Time to value has become increasingly important for sustainable businesses today.

The 49th annual R&D 100 Award was open to a number of industry sectors, academia and government laboratories who despite the recent economic downturn have continued to innovate. Winners were chosen following an extensive review by an independent judging panel and the editors of R&D Magazine. The R&D 100 Awards have long been a benchmark of excellence for industry sectors as diverse as telecommunication, high-energy physics, software, manufacturing and biotechnology. The PANalytical award for its Empyrean product references cutting-edge technology in every aspect.

To find out how you can benefit from the Empyrean, contacting your local PANalytical representative, or visit www.panalytical.com