AXAA e-Newsletter

Issue 2, 2012 **Australian X-ray Analytical Association**

President's Address

Dear AXAA Members and Readers,

Welcome to Winter,

I hope all of our members are warm and well. The AXAA National Council is gearing up activities in Victoria and NSW this year, and we are pleased to announce that Student Seminar days will be held in both of these States in September (NSW) and October (VIC). Both events this year have industry support, with the Victorian event being assisted by Bruker, and in NSW Panalytical representatives are helping to organize the event to be held at the University of Sydney (read on for further details about both events).

The next AXAA National Conference and Exhibition will be held in February 2014. The National Council have been busy obtaining venue quotes in several states and are close to reaching a decision – look out for the announcement in the next Newsletter.

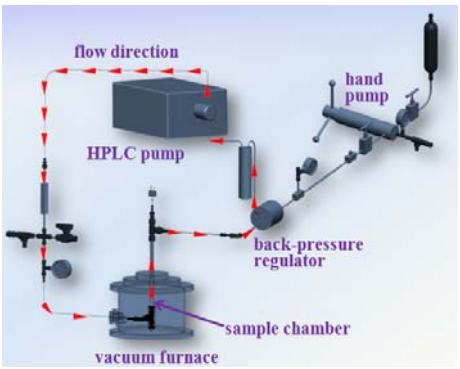
Finally, our website makeover is progress – thanks to our Newsletter Editor Nathan Webster for all his hard work.

Vanessa Peterson
National Council President

Matters for Scatterers

Hydrothermal crystallization reactions are responsible for mineral formation and alteration in nearly all hot aqueous environments, and are dominant reactions in many ore processing and hydrometallurgical processes and in hydrothermal materials synthesis for producing a wide range of technologically important materials. The knowledge of fundamental mechanisms and reaction kinetics of these reactions is of vital importance to a range of scientific disciples, but has been limited due to the lack of efficient tools to study these reactions by an *in situ* and time resolved approach. *In situ* neutron diffraction is a powerful tool offering the ability to observe gram scale samples and if we have hydrothermal cells suitable for neutron diffraction we can monitor the hydrothermal crystallization process *in situ*.

The University of Adelaide has recently developed a versatile hydrothermal flow-through cell which can independently and precisely control the temperature (up to 673 K), pressure (up to 46 MPa), flow rate (0.01–10 ml min^{-1}), and reaction-fluid volume (≥65 ml). Such versatility is realized by a design consisting of a room temperature and ambient-pressure external fluid supply module, a high-pressure reaction module which includes a high-temperature sample compartment enclosed in a vacuum furnace (provided by ANSTO), and a room-temperature and high-pressure backpressure regulation module for pressure control. The cell was successfully commissioned on the high-intensity powder diffractometer beamline, Wombat, at ANSTO.



Schematic drawing of the single-pass flow-through cell.

Hydrothermal cell on the Wombat diffractometer at ANSTO.

We have used the hydrothermal cell to investigate the effect of pressure on the hydrothermal pseudomorphic mineral replacement reaction from celestine ($SrSO_4$) to strontianite ($SrCO_3$) at a constant temperature of 473 K and flow rate of 5 ml min^{-1}. For the first time, we observe a nonlinear effect of the pressure on the mineral replacement rate, which first increases with increasing pressure from 14 to 20 MPa, and then decreases when pressure further increases to 24 MPa. This finding provides further evidence for our understanding of pseudomorphic mineral replacement reactions, and is consistent with the interface coupled dissolution-repreciptation mechanism (Journal of Applied Crystallography, 2012, 45, 166-173).

Dr Fang Xia
CSIRO Materials Science and Engineering
(formerly from the University of Adelaide)

Memories of Bruce Chappell

Newer AXAA members may not be aware of Bruce's considerable contribution to XRF science and the AXAA. In the early days, the AXAA conference was always held at the ANU, where Bruce was an integral part of the program and mentored many X-ray analysts. Later, he and Keith Norrish ran a series of X-ray Schools attended by many current X-ray analysts.

Bruce was born in 1936 in a small town near Armidale in NSW, showed great intelligence at school and eventually studied at UNE. His strengths were in physics and mathematics, and he initially wanted to be a physicist. The geologists at UNE had other ideas and took him on field trips in an attempt to lure him away. It worked. He eventually won the university medal (rumoured to be the only geologist ever to win) and followed on with post-graduate study.

When the Geology Department at ANU was being set up, Bruce was an early appointment. He lectured there until his 'retirement', after which he continued to provide valuable input to Macquarie University's GEMOC facility, the University of Wollongong and Geoscience Australia. He wrote many important papers on geology, geochemistry and XRF, and, with Keith wrote the chapter on XRF in Zussman's *Physical Methods in Determinative Mineralogy.* Bruce collaborated with many great geochemists. He has a very high citation index.

Bruce resisted being called a geochemist and insisted that he was a geologist. Along with the late Prof Allan White (La Trobe University/University of Melbourne) he was responsible for the classification of granites into I-, S- and A- types depending on their chemistry and genesis. Distinctions were made using geochemistry, hence he needed to analyse rocks. In 1963, he met Keith Norrish, who introduced him to XRF, and so began a whole new stream of research. Bruce particularly concentrated on trace element analysis and was renowned for his ability to reliably measure tricky elements at low concentrations. His strong background in physics must've helped.

Bruce was particularly proud of his successful XRF analysis of the early moon rocks. There was still some scepticism about XRF as a reliable analytical method, particularly for trace elements, but Bruce's results correlated well with Isotope Dilution data.

Bruce's lab was a great place to work and always a hive of activity. On the campaign to analyse all granites in Australia and the world, we worked our way through the Lachlan Fold Belt, western USA and Scotland.

Bruce in the field.

Bruce inspired great loyalty amongst his students and staff and his field trips were legendary. On one, needing to illustrate some points and not having any other means, he drew on the front of a white Kombi with indelible pen. The vehicle sported the fetching phase diagram for the rest of its life. In the days before explosives regulation, this was Bruce's sampling tool-of-choice, and many outcrops were obliterated in the hunt for large amounts of fresh sample.

He was a strong proponent of the 'garbage-in/garbage-out' principle and insisted on correct sampling and preparation.

A funny incident occurred when, before an exam, a student stood in the doorway of the lab to ask to what level of detail he should learn the periodic table – should he learn atomic numbers and weights? 'Yes', said Bruce. 'I bet you don't know them', said student. 'Try me'. Bruce flawlessly answered all that was thrown at him, even the most obscure elements. What the student didn't know was that there was a large periodic table poster on the wall adjacent to the door, visible to Bruce but not the student!

The last-minute panic before Bruce left on overseas trips was legendary. He would leave for the airport at the very last minute, just make the plane and then realise that he'd left something vital behind or that he hadn't turned off the lights in his house. Ross Freeman regularly came to the rescue. Once Bruce arrived at the airport, locked his car, only to realise he'd left passport and car keys in the car. He picked up a rock and smashed the window. Ross rescued broken car.

Bruce was a perfectionist and would spend inordinate amounts of time getting an analysis right. He was a great believer in ensuring that all the composite parts of an analysis were correct to eliminate errors. In the days before computers and end-window Rhodium X-ray tubes, everything was done manually and X-ray tubes with different anodes were changed to better analyse specific elements, so long runs of samples were done on one X-ray tube before changing to another. Sodium was analysed by Flame Photometry, borate fusions were done over an oxy-acetylene flame and the automatic sample changers for the XRFs were students. You couldn't buy prepared fusion mix, so we made our own - an irksome task. Regardless, Bruce would routinely analyse samples in triplicate. Data was collected on ticker-tape and run through a tape-reader. It took a long time to analyse a sample and a long time to compile the results.

When computers appeared, Bruce embraced the new technology and taught himself programming. Bruce was a very hands-on scientist and would happily weigh, press pellets and run instruments when required. We often joked about him being the highest paid technician in the lab. He expected a lot from his staff, but didn't ask of others what he wasn't prepared to do himself.

He had monumental fights with Philips over their XRFs and particularly their early software, which he thought could be improved. He always pushed instrumentation to the limit to achieve his analytical outcomes.

He had early HP computers, which often broke, causing Bruce great annoyance. On the umpteenth visit to HP to get one repaired, he asked a disinterested staff member where to leave it. Bruce took the reply - 'Just drop it there' literally and dropped it. He suddenly had their attention.

Bruce was particularly keen to analyse the Rare Earth Elements and in the 80s set up an Instrumental Neutron Activation Analysis lab. This was a huge task and had to be run 24/7 – decaying elements wouldn't wait for anyone. After sorting out this technique, he moved on to EDXRF. He acquired an instrument and became a great advocate for the method and so began an association with Spectro.

After Bruce's passing, Ed Stephens from St Andrews University wrote *'Granite is perhaps a good metaphor for Bruce. He was steadfast, brittle rather than malleable, and would be your rock of choice for any structure intended to last. He had a set of fundamental principles and would not be swayed by fashion, either in science or life in general'.*

I agree completely and deeply miss his conversation, humour and advice.

Liz Webber
Geoscience Australia

Upcoming Events and Conferences

1. National XRD Course

***X-ray Powder Diffraction Analytical Methods*, Curtin University, Perth**
Sat, 25 Aug – Tue, 28 Aug, 2012

Venue: Department of Imaging and Applied Physics, Curtin University, Bentley (Perth), Western Australia. [Client-specific version of the course can be presented at the customer's site].

Duration of Curtin Course: 4 days

Dates: 25-28 August, 2012

Course Presenters: Professor Brian O'Connor and Dr Robert Hart

Enquiries and further information:
B.O'Connor@curtin.edu.au

Cost: $2,585 including GST

THERE ARE STILL PLACES AVAILABLE

Overview: The course has been designed to give participants a theoretical and practical grounding in the principal characterisation methods which make use of x-ray powder diffractometry data. Approximately 60% of the course involves hands-on instruction. Participants personally collect diffractometry data sets and then process these, both manually and with PC computers, in exercises on various analytical methods, including Rietveld analysis.

Public domain software will be used, including *WINPLOTR* and *Rietica*. The course also includes overviews and demonstrations of the commercial software packages *X'Pert HighScore Plus* and *Diffracplus Topas*. While the course is relevant to the analysis of all classes of crystalline materials, attention is devoted mainly to materials relevant to the mining and mineral processing sector.

2. Internet XRF Course: Series 5, 2012

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 the 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: $2,530 including GST

Further information and enrolment:
brian_oconnor@iprimus.com.au
Tel: (08) 9291 7067

3. The Bragg Symposium: Celebrating One Hundred Years of X-ray Crystallography

The 11th of November 2012 marks the centenary of the founding of X-ray crystallography by Lawrence Bragg, a field in which he and his father, William, made pre-eminent contributions that were recognised by the joint award of the Nobel Prize for Physics in 1915. William was the Elder Professor of Physics at the University of Adelaide from 1886 to 1909 and Lawrence was born in Adelaide and received his schooling at St Peter's College in Adelaide and his first degree from the University of Adelaide. He remains the youngest Nobel Prize winner ever.

To celebrate this important anniversary, the *Bragg Centennial Symposium* will be held at the Elder Hall, University of Adelaide on Thursday, 6th of December 2012. The Symposium will explore the historical, social and broader scientific impacts of the Braggs' work. The Symposium will have a strong line-up of national and international speakers. Prior to the Bragg Symposium, a combined meeting of the Asian Crystallographic Association (AsCA) and the Society of Crystallographers in Australia and New Zealand will be held at the Adelaide Convention Centre from the 2nd to 5th of December. The 2012 AIP Congress in Sydney (9-13 December) directly follows the Bragg Symposium.

For further information:
http://sapmea.asn.au/conventions/crystal2012/bragg.html

Ph: (08) 8274 6048
Fax:(08) 8274 6000
crystal2012@sapmea.asn.au

John Carver
Chairperson

Date	Event	Location	Further Information
21st Sep 2012	AXAA NSW Student Seminar Day, "Scattering Matters"	University of Sydney	Flyer overleaf
17th Oct 2012	AXAA VIC Student Seminar Day, "Something to Bragg About"	CSIRO, Clayton	Flyer attached
28-31 Oct 2012	13th European Powder Diffraction Conference EPDIC 13	Grenoble	http://epdic13.grenoble.cnrs.fr/
1st and 2nd Dec 2012	10th TOPAS User Meeting	Intercontinental Hotel, Adelaide	martin.duriska@bruker.net.au
22-25 April 2013	Accuracy in Powder Diffraction IV	NIST	http://www.nist.gov/mml/apdiv_conference_2013.cfm

AXAA Contacts and Membership

Contact details for the members of the AXAA National Council, and details about how to become an AXAA Member, can be found on the AXAA website.

AXAA Website

http://www.axaa.org/

AXAA

Scattering Matters

Friday
21st September, 2012
2:00 pm – 5.00 pm

The University of Sydney

Information For Applicants

- Prizes for the best presentations include a **money award**
- Presenters are selected on the merit of their applications.
- Presentations will be 15 minutes in length.
- Eligibility: Undergraduate (3rd/4th year or Honours), Masters and PhD students, with results from any scattering technique(s) that have been applied to their research.

Applications must include:

1. Name and contact details.
2. Abstract of presentation (maximum 400 words).
3. Letter of support from supervisor.
4. A brief paragraph highlighting the diffraction/scattering technique(s) used and the relevance to your field of study.

Submission: Applications must arrive by email to vanessa.peterson@ansto.gov.au by 5pm 24th August, 2012.
Selected applicants will be notified by 7th September, 2012.

For more information contact: vanessa.peterson@ansto.gov.au

Wednesday 17th October, 2012
2:00 - 5:00 pm
G.K. Williams Room
CSIRO Process Science and Engineering
Bayview Ave, Clayton

"Something to Bragg About"

AXAA Victoria Student Seminar Day 2012

Information:

- For undergraduate, Honours, Masters and PhD students, with results from any X-ray or neutron diffraction or scattering technique(s) that have been applied to their research (any discipline).
- Student presentations 12 minutes in length, with 3 minutes for questions.
- 2 × Plenary seminars (15 mins) presented by career scientists.
- The **best presentations** in undergraduate and postgraduate categories will be awarded cash prizes.
- All are welcome to attend – students, supervisors, colleagues….Please RSVP to natasha.wright@csiro.au
- Refreshments will be provided after the seminars.
- Send applications by email to natasha.wright@csiro.au by 5 pm Wednesday 3rd October 2012.
- Applications must include you *name and contact details*, an *abstract* (maximum 400 words) and a *brief paragraph* highlighting the diffraction/scattering technique(s) used and relevance to your field of study.
- Successful applicants will be notified by Monday 8th October.
- For more information contact Natasha Wright or Nathan Webster (nathan.webster@csiro.au).

Society of Crystallographers in Australia and New Zealand
SCANZ

6 December 2012

Bragg Symposium: Celebrating 100 years of Crystallography

To be held at the Elder Hall, University of Adelaide, South Australia

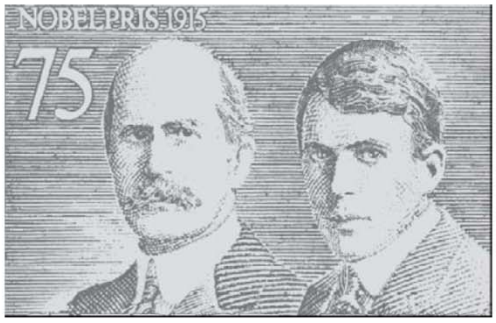
Featuring eminent lecturers discussing topics relating to the early days of crystallography, the institutions where the Braggs worked and the impact of their work including current exciting research activities in crystallography

Invited speakers include:

Prof Anthony Kelly (Univ. of Cambridge)
Prof Brian Matthews (Univ. of Oregon/Univ. of Adelaide)
A/Prof John Jenkins (author of "William and Lawrence Bragg, Father and Son: The Most Extraordinary Collaboration in Science")
Prof Thom Mason (Oak Ridge)
Prof Anthony Cheetham (Cambridge/Santa Barbara)
Prof Anders Liljas (Univ. of Lund)
Prof Colin Humphreys (U of Cambridge)
Prof John Spence (ASU/UC Berkeley)
Prof Wayne Hendrickson (Columbia U)
Mrs Patience Thomson (younger daughter of Sir Lawrence Bragg)

Further details on other speakers and activities will be available as updates at

www.sapmea.asn.au/crystal2012



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XFlash® 6 – The Next Generation of EDS Detectors

Bruker Nano Analytics introduces ***XFlash®6***, the next generation of silicon drift detectors (SDD) for energy dispersive X-ray spectrometry (EDS) on electron microscopes. With the availability of large detector sizes up to 100 mm², energy resolution down to 121 eV, and throughput up to incredible 600 kcps, the ***XFlash®6*** series sets new standards for speed and sensitivity in EDS analysis, and is exceptionally powerful for applications in nano technology and nano research.

With ***XFlash®6***, Bruker now offers the widest range of EDS detector sizes in the market, starting from 10 mm² up to an unequaled 100 mm², to ensure optimal conditions for any application at any type of electron microscope. Due to the innovative Slim-line design, all detectors of the ***XFlash®6*** family provide the largest possible solid angle per active area for maximum collection efficiency, which is especially important at low electron beam currents. Combined with the unique, most advanced hybrid pulse processing technology, ***XFlash® 6*** detectors can accept input count rates in excess of 1,500 kcps with throughput as high as 600 kcps, and maintain their best energy resolution over the widest range of count rates. With the new detector generation Bruker is pushing the limits of energy resolution again - the premium ***XFlash®6 |10*** achieves unmatched 121 eV at Mn-Ka and 38 eV at C-Ka, vital especially for efficient and accurate analysis at low energies.

The ***XFlash®6*** user can choose from six different detector models to find the optimal solution for any individual application, no matter whether using SEM, FIB-SEM, µ-probe or TEM. The 10 mm² ***XFlash®6 |10*** and the 30 mm² ***XFlash®6 |30*** detectors are well suited for the majority of tasks on SEM, including analysis at high beam current, low kV, and low vacuum. The 60 mm² ***XFlash®6 |60*** and the 100 mm² ***XFlash®6 |100*** are particularly useful in low X-ray yield situations, e.g. for low beam current / low kV operation as required for the investigation of nanostructures, or for the analysis of beam-sensitive biological samples. The two detector models for transmission electron microscopy, the 30 mm² ***XFlash®6T |30*** and the 60 mm² ***XFlash®6T |60***, are designed to cause minimal mechanical and electromagnetic interference on the TEM, and to guarantee the best take-off angle available.

*The **XFlash® 6** family of silicon drift detectors for EDS analysis on electron microscopes*

"Having pioneered the commercial use of SDD about fifteen years ago, we are proud to once again confirm our leadership in X-ray detector technology with today's launch of ***XFlash® 6***." states Thomas Schuelein, President of the Bruker Nano Analytics Division. "Our QUANTAX EDS system, with the new, versatile ***XFlash®6*** SDD series and the powerful ESPRIT analytical software suite enables superior analytical performance to the benefit of our clients from academia and industry. We expect to see many exciting results, as ***XFlash®6*** will not only make routine EDS analyses much more productive, but will also support solving even the most demanding analytical challenges at the forefront of science."

About Bruker Corporation

Bruker Corporation (NASDAQ: BRKR) is a leading provider of high-performance scientific instruments and solutions for molecular and materials research, as well as for industrial and applied analysis. For more information, please visit www.bruker.com For more information on ***XFlash® 6***, please visit www.bruker.com/quantax

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Bruker Acquires SkyScan Shares

Bruker has acquired all of the shares of SkyScan N.V., a scientific instruments company located near Antwerp, Belgium. SkyScan develops, manufactures and distributes worldwide advanced, high-resolution micro computed tomography (CT) systems for three-dimensional (3D) X-ray imaging. SkyScan's micro-CT instruments can be configured for numerous applications in materials research and in the life sciences, including *in vivo* preclinical animal imaging, *in vitro* bone and soft tissue imaging, 3D imaging of electronic components, synthetic materials, new devices such as micro-sensors and cardio-stimulators, geological samples, fuel cell components, ceramics, and more.

Following the acquisition, the company has been renamed Bruker *microCT N.V.*, and will continue to operate from the company-owned premises in Belgium under its previous management. The new Bruker *microCT* business will continue to produce all micro-CT instruments under the SkyScan brand, and will provide enhanced global support for the installed worldwide base of SkyScan.

With global Bruker distribution and customer service capabilities for materials research, preclinical imaging, and SEM accessories, the Bruker *microCT* business is now well positioned to drive the further profitable growth of the SkyScan 3D micro-CT imaging products. In addition, most successful SkyScan distributors will be able to continue to distribute SkyScan products in their countries or market segments. Bruker intends to invest in additional micro-CT applications and demo centers to further enhance customer collaborations and support worldwide.

Dr. Alexander Sasov, founder and CEO of the former SkyScan company, and now Managing Director and CEO of Bruker *microCT*, commented: "We have been looking for a strong and competent strategic partner to continue the rapid growth that SkyScan has enjoyed over many years. I am very glad that with the strong Bruker brand and the additional worldwide Bruker distribution channels and support network, we have found exactly what we were looking for to drive our micro-CT business to the next level of success."

Frank Laukien, PhD, President and CEO of Bruker Corporation, added: "We are excited to have Alexander Sasov and his capable SkyScan team join us, adding in-depth 3D X-ray micro-CT imaging experience to Bruker. We are impressed by the portfolio of high-performance SkyScan products which fit nicely into our global materials research and preclinical imaging distribution channels, and which complement our other X-ray analysis and preclinical MRI products. The Bruker *microCT* team shares our philosophy of innovation, quality and customer support excellence."

For further details on the SkyScan instruments, please contact Thomson Scientific Instruments Pty Ltd on (03) 9899-0095 or email: info@tsi.com.au