AXAA e-newsletter

Issue 2, 2015 **Australian X-ray Analytical Association**

President's Address

Dear AXAA Members and Friends,

In May the National Council visited several potential venues in Melbourne for the AXAA 2017 National Schools, Conference and Exhibition. We have narrowed the field down to two and are waiting on final details and negotiations, but we are close to making a decision and will announce our AXAA-2017 venue shortly.

In July, AXAA was the Poster Session Sponsor at the Australia Oceania Conference on Neutron Scattering (AOCNS 2015) held in Sydney. In continuing efforts to extend our outreach we are a Bronze Sponsor for the 2015 Australian Synchrotron User Meeting which is being held in conjunction the Asia-Oceania Forum for Synchrotron Radiation Research (AOFSRR 2015). The joint meeting, which is being held at the National Centre for Synchrotron Science in Melbourne, has received nearly 200 abstracts and is an excellent opportunity to further increase the visibility of AXAA, and outside of Australia.

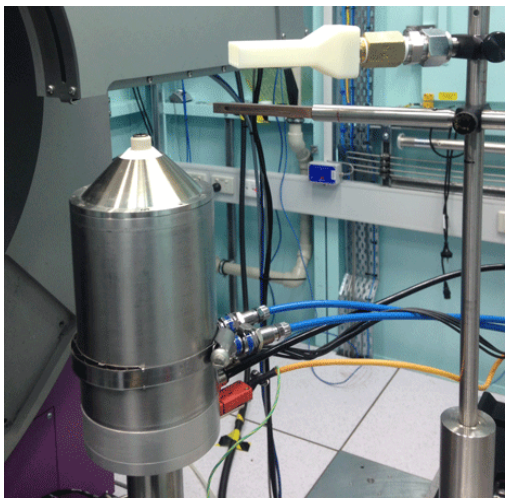
Finally, September and October will see AXAA Student Seminar Days being held across Australia. These events are an excellent opportunity for students to present their work in a friendly, collegial atmosphere and meet others in the X-ray and neutron scattering community. Prizes will be awarded to the best presentations. Stay tuned for details about these events in your state.

Nathan Webster
AXAA President

Matters for Scatterers: The Heat Is On at the Powder Diffraction Beamline

The high beam intensity and rapid data collection capability of the Australian Synchrotron's Powder Diffraction (PD) beamline offers research teams in Australia and New Zealand the opportunity to perform *in situ* experiments on a wide range of samples in a number of different configurations and geometries. High angular resolution powder diffraction measurements in Debye-Scherrer (DS) geometry can be collected while varying parameters such as temperature, pressure and gas atmosphere *in situ* to observe structure changes in samples for many different research applications.

In DS configuration, high temperatures up to 1000°C can be achieved by heating samples in rotating quartz glass capillaries using a hot-air blower. In a typical beamtime round, over half of the experiments performed at the PD beamline requested variable temperature, and most of these exploit the high temperature capabilities offered by the hot-air blower. The original Cyberstar was acquired and commissioned in 2007 at the beginning of the user operations at the PD beamline. However, after more than seven years of extensive use it has been retired.



The new FMB-Oxford hot-air blower available on the PD beamline at the Australian Synchrotron, capable of heating samples up to 1000°C with a significantly wider hot zone than the original Cyberstar device.

A new, upgraded FMB-Oxford hot-air blower was acquired earlier this year and has been successfully in use at the PD beamline over the past several months. When comparing the new and original units it is clear that the new one is essentially the 'big brother' with the same temperature range (ambient to 1000°C) and excellent temperature stability (±1°C). The new unit has a larger 8 mm diameter nozzle compared to the original hot-air blower (5 mm) meaning that that standard horizontal slit size can be used, thus allowing data to be collected more rapidly. This is will be a significant improvement for experimental efficiency.

For enquiries regarding future experiments at the powder diffraction beamline, please contact the PD team at PD@synchrotron.org.au.

Justin Kimpton
Principal Beamline Scientist, PD Beamline

Energy and Spatially Resolving X-ray Detectors Shed New Light on Materials Analysis

X-ray detectors with both energy-resolution and spatial resolution open up a range of new possibilities for materials analysis. The ability to identify the elemental components within a bulk sample through energy-resolved detection of the X-ray fluorescence is a staple technique of most synchrotrons and forms an essential analysis tool in most X-ray labs. The determination of crystallographic orientation in polycrystalline samples is also a key part of materials analysis and modelling.

Historically these two measurements have either required two separate detectors or two separate measurements. The Maia Detector System [1] in use at the X-ray Fluorescence Microscopy (XFM) beamline at the Australian Synchrotron is one of a small number of X-ray detectors which possess both energy-resolving and spatially resolving capabilities. Whilst the array of 384 detector pixels in the Maia detector allow for only limited spatial resolution, we demonstrate in a proof-of-principle experiment that it is still sufficient to uniquely determine crystallographic orientation in a polycrystalline sample. Figure 1 shows the results of using the Maia detector in back-reflection geometry for orientation analysis in a polycrystalline nickel foil.

A two dimensional map was obtained by scanning a 2 x 2 µm probe across the sample to characterise a total area of 300 x 60 µm. At each point a full energy-resolved spectrum was obtained yielding both fluorescence data (used for elemental analysis) and reflection data (used for orientation analysis).

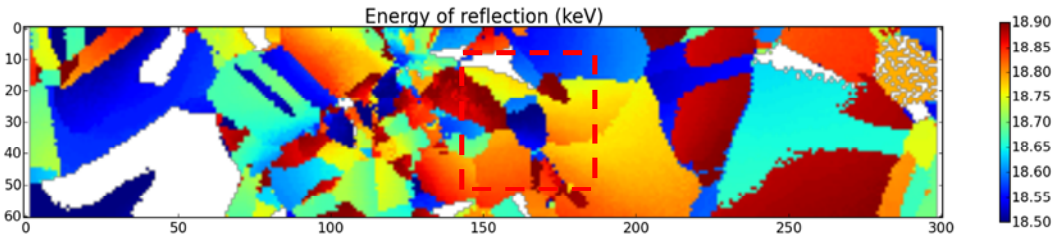


Figure 1. Two-dimensional map of polycrystalline nickel foil showing the energies of the most intense reflections for each grain. White areas indicate that no reflections were observed within the incident energy window. The corresponding orientation map for a sub-region of the foil is shown in Figure 2.

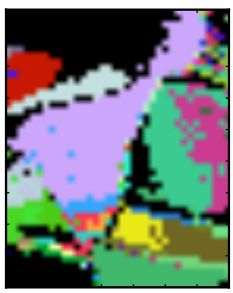


Figure 2. Orientation map obtained via analysis of data shown in dashed box in Figure 1.

The analysis of the Maia data consisted of two parts: fitting of the fluorescence peak data to determine the elemental composition of the sample, and locating the diffraction peaks corresponding to each incident X-ray energy for orientation determination. The fluorescence analysis (not shown here) is part of the standard measurements conducted at XFM, whilst the analysis of the backscatter data leading to an orientation map (Figure 2) is unique to our experiment. The aim is to eventually develop simultaneous elemental and orientation mapping as a standard technique at the Australian Synchrotron paving the way to a new class of materials science measurements. In the wider context this experiment highlights just one potential application that will be enabled by the next generation of energy and spatially resolving X-ray detectors [2].

Brian Abbey[1], Henry Kirkwood[1], Martin de Jonge[2], and Chris G. Ryan[2]
[1]ARC Centre of Excellence for Advanced Molecular Imaging, La Trobe Institute for Molecular Science, La Trobe University
[2]XFM Beamline, Australian Synchrotron

Part of this research was undertaken on the XFM beamline at the Australian Synchrotron, Victoria, Australia

[1] Siddons DP *et al.*, *J. Phys.: Conf. Ser.*, 2014;**499**(1);012001.
[2] Kirkwood HJ *et al.*, *In preparation*, 2015.

SAXS Investigation of New Ultra-high Strength Steels

Steels represent 95% of all engineering alloys used today. They dominate applications such as construction, transportation, power generation and oil and gas transmission. As a class of engineering alloys, steels are capable of exhibiting a large range of different properties and this suitability, for many different types of applications, is part of the reason they are the most important engineering alloy. The other reason is steels offer these properties at a very low price. The importance of steel as a modern engineering alloy means that it is subject to intense research and development activities – for the development of new and better steel grades, as well as improvements to existing grades. As an example of the level of research and development taking place, none of the steels currently used in new automobiles even existed 10 years ago.

A particularly interesting class of steel is the 'maraging' steels. These steels are the strongest grades, with strengths approaching 2000 MPa. This is twice as strong as titanium alloys and five times as strong as aerospace aluminium alloys. These remarkable strengths are obtained by the formation of a nanoscale distribution of particles within the steel that form through a nucleation and growth process during thermal processing. A very large number of particles form (~10^{24} m^{-3}) and these are largely responsible for the properties of these ultra-strong steels. The catch is these 'Maraging' steels are very expensive because they contain large additions of

expensive elements such as nickel and cobalt. The cost has significantly limited the use of maraging steels in common engineering applications.

As part of a project funded by ArcelorMittal (the worlds largest steel company), PhD student Ms Wenwen Sun and her advisor A/Prof Christopher Hutchinson, have developed a new class of cheap, ultra-high strength steels. The design of this new steel class used modern computational alloy design approaches (coupling computational thermodynamics, optimization algorithms such as Genetic Algorithms, and diffusional nucleation and growth theory) to identify new compositions capable of strengths approaching 2000 MPa without the use of large nickel and cobalt additions.

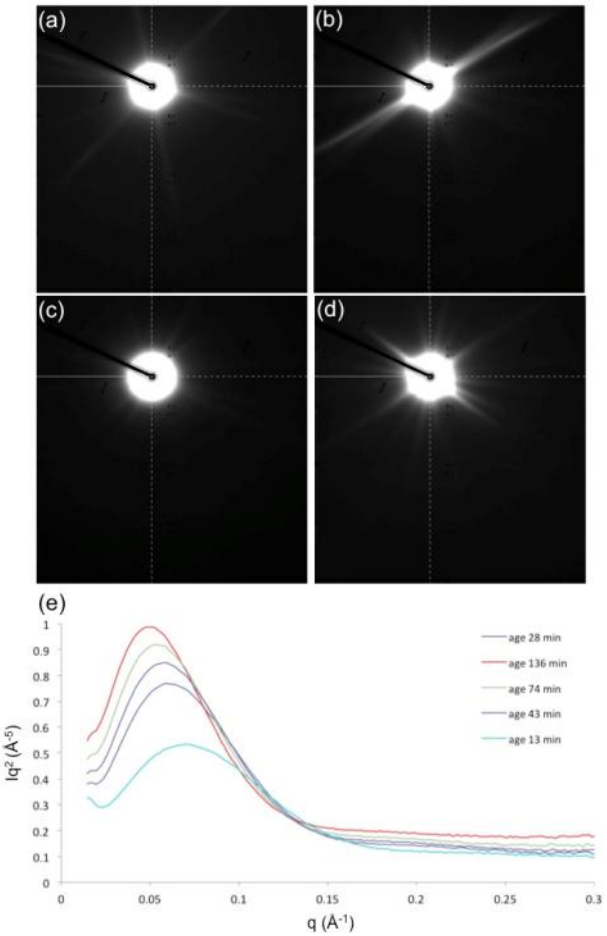


Figure 1: Example raw SAXS patterns of (a) Fe-0.06C-2.14Si-1.12Ti-3.9Mn-2.78Ni-0.95Cr (wt.%), (b) Fe-0.04C-2.16Si-1.09Ti-3.84Mn-2.8Ni (wt.%), (c) Fe-0.05C-2.32Si-1.59Ti-3.81Mn-2.78Ni (wt.%), and (d) Fe-0.03C-1.92Si-0.76Ti-1.46Mn-2.95Ni (wt.%), which have been solution treated at 1200°C for 20 min followed by ageing at 500°C for 80 h. (e) Example Kratky representation showing the evolution of the peak position and area under the curve of Fe-0.04C-2.16Si-1.09Ti-3.84Mn-2.8Ni (wt.%) as *in situ* ageing proceeds at 550°C.

These alloys exploit the nucleation and growth of nanoscale precipitates and a full understanding of the strengths experimentally observed required a characterisation of this particle size distribution. The team performed *in situ* small angle X-ray scattering experiments at the Australian Synchrotron to characterize the evolution of the nucleation and growth of the particles during thermal processing (Figure 1). Coupling these SAXS experiments with 3D Atom Probe Tomography allowed both quantitative particle volume fractions and sizes to be extracted from the SAXS data which are required for rationalizing the observed strengths and identifying avenues for further increasing the strength. An example of the evolution of the average particle size, volume fraction, number density and strength for one of the newly developed steels is shown in Figure 2. The particles are a few nm in size and the new steels reach 1700 MPa in strength without the use of expensive alloying elements. This work is part of an ongoing collaboration between ArcelorMittal and A/Prof Hutchinson's group at Monash University.

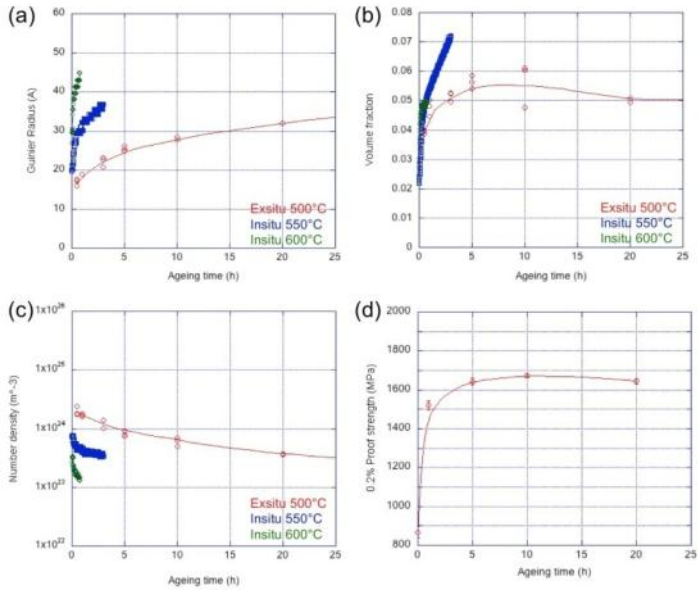


Figure 2: Evolutions of (a) precipitate radius, (b) volume fraction, (c) number density as a function of ageing time at 500°C, 550°C and 600°C of Fe-0.04C-2.16Si-1.09Ti-3.84Mn-2.8Ni (wt.%), (d) age hardening behaviour at 500°C measured by compression tests.

Wenwen Sun and Christopher Hutchinson
Department of Materials Science and Engineering,
Monash University

Part of this research was undertaken on the SAXS/WAXS beamline at the Australian Synchrotron, Victoria, Australia

The Importance of Incorporating TGA in the Analysis of Fused Beads by XRF

Thermogravimetric analysis is used to determine the moisture content as well as the loss or gain on ignition when heating the samples to an elevated temperature (typically 1000°C). For illustrative purposes, typical iron ore samples have been used.

ISO 9516-1 2003: Determination of various elements by X-Ray Fluorescence spectrometry - Section 8.3 (c) states: "Any calculation program shall use a calculation algorithm similar to that of the program given in Annex H.

It should be based on concentrations rather than intensities; this applies to both the alpha and the overlap corrections. The program shall be capable of using loss-corrected alphas, and in particular it shall accept and use the alpha (i,i) factor for each element. If catch weights are used in weighing, it shall be able to correct for the differing flux/sample ratios. Loss eliminated alphas are used and the correction for catch weights involves multiplying all results for a sample by a constant factor after all other corrections have been made. If the nominal flux and sample masses are F and S and the actual masses are f and s, then the factor is S.f/F.s"

To satisfy the above requirement of "loss corrected" alphas, the mass measurement of sample and flux needs to be corrected for moisture loss and ignition loss. This is normally achieved as described below.

Most TGA's operate on a similar basis. Samples are weighed for the XRF fusion process and an aliquot of typically 1 g is weighed at the same time for TGA analysis. For the purpose of this experiment the IMP laboratory TGA was used. The TGA has the ability to accept 23 samples and one calibration standard. Crucibles are weighed in a carousel, after which the samples are added and the mass of each sample is measured automatically. The TGA then heats the sample to the predetermined firing and soaking temperatures.

Figure 1: Test TGA system.

For example 105°C is typically used to determine the hygroscopic moisture content of the sample. Once the set temperature is reached and after the required soaking time has passed, samples are weighed with an internal balance inside the TGA. The TGA chamber can be purged with dry nitrogen in order to speed up the drying process as well as ensure an inert atmosphere is maintained to prevent oxidation at the lower temperatures. The mass loss obtained for each sample measured at temperature is compared to the original sample mass at room temperature and the difference in mass is recorded as a percentage moisture. Note that the TGA can soak at 105°C for a fixed time before measuring the mass, alternatively samples can be continuously weighed until constant mass is obtained for all samples. Importantly from the moisture content, the dry sample mass can be calculated in the aliquot used for the fusion sample. This correction is very important and as will be demonstrated later, failure to correct for moisture content can significantly bias the reported analytical results.

Total loss or gain of ignition is generally measured at 1000°C. Once the moisture content is determined, the TGA will heat up at the specified heating rate to the next measuring temperature, for example 1000°C. After the required soak time, the mass of each sample is measured in situ at the set temperature in the instrument. The difference in mass between the mass obtained at 105°C (dry weight of the sample) represents the loss or gain of ignition. Note that heating to constant mass is also possible instead of using a fixed soak time.

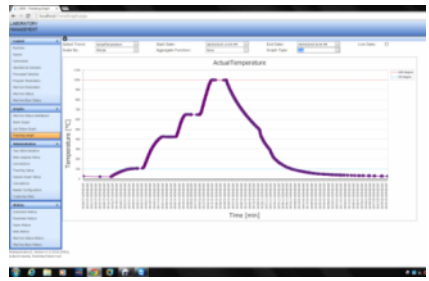


Figure 2: Example of typical temperature profile during test.

In order to demonstrate the effect of re-hydration on moisture determination, two sets of milled samples were weighed for fusion and TGA analysis. 0.7g and 1g was weighed out respectively. One set of samples were pre-dried for 12 hours at 105°C prior to analysis and a second set was analysed without pre-drying (these were dried prior to pulverising). All samples were tested in duplicate. Pre-dried samples were removed from the drying oven and allowed cool down in the ambient conditions of the laboratory environment so that they could be handled. Total time outside the oven prior to TGA weighing was around 40 minutes.

Moisture [%] 105 Pre dried	Moisture [%] 105	LOI 1000 [%]
0.0105	0.0304	-3.9015
0.0020	0.0110	-3.8990
0.0597	0.0824	-0.9295
0.0122	0.0386	-0.9266
0.1944	0.3221	0.8185
0.1944	0.3395	0.8200
0.2889	0.5764	4.7595
0.3077	0.5991	4.7695
0.1973	0.6120	6.7870
0.2102	0.6176	6.7941
0.3294	1.4653	8.0582
0.3363	1.5143	8.0753
0.3121	1.5429	9.0387
0.3284	1.5630	9.0544
0.3272	1.5168	9.7673
0.3136	1.4928	9.7880
0.5519	1.7898	18.1089
0.5323	1.7978	18.1496

As is evident from the data presented here, the inherent hygroscopic moisture in these samples ranged from 0.01% to 1.8%. If the pre-dried samples were fused and analysed on the XRF and the spectroscopist assumed that the samples received from sample preparation were dry, the dry mass would be overstated by up to 1.8%. This would result in the analytical results being understated by up to 1.8%. Please keep in mind that some samples can have significantly higher hygroscopic moisture contents when compared to these samples, highlighting the need to determine the dry mass of samples.

Samples that were pre-dried reabsorbed moisture in the time it took them to cool and weigh them inside the TGA. A similar re-absorption of moisture would occur for the samples weighed for XRF analysis. The error in determining the dry mass of sample was as high as 0.55% highlighting the importance of weighing samples

immediately after drying and/or retaining these in a desiccator. In this example the reabsorption of hygroscopic moisture can bias analytical XRF results by up to 0.55%. This value could vary significantly depending on the time the sample is exposed to the laboratory ambient conditions – especially affected by the sample type and relative humidity and time.

The LOI / GOI ranged from – 3.970 % to + 18.149 % in the samples analysed. Such a range of LOI values will have a significant effect on the analytes being measured on the XRF. For example if a 10 times dilution factor (0.7g sample + 6.3g flux to produce a 7g bead) was used for the sample with an LOI of 18.15% (assuming a dry sample) the analytical results would be overstated by 1.81%. (0.7g *(100-18.15)/100 +6.3 = 6.873g, dilution = 6.873/0.7=9.819, relative error (10-9.819)/10*100=1.81%). In the case where there was a gain on ignition of -3.97% the Fe content for example would be understated by 0.39%. . (0.7g *(100-18.15)/100 +6.3 = 6.873g, dilution = 6.873/0.7=9.819, relative error (10-9.819)/10*100=1.81%)

Conclusion:

It is imperative to determine both the hygroscopic moisture content and LOI/GOI in samples used for XRF fused bead analysis. Best practice would be to weigh the respective aliquots for fusion and TGA at the same time and then using the TGA values to correct the XRF results. An uncorrected moisture of 0.5% will result in a financial loss of 0.5%. Assume $0.60 per dry unit iron ore, the financial loss will be of 0.5 times 0.6 which is $0.3 per ton or $300,000 per million tons for the shipper.

Boyne Hohenstein
IMP Group

Bruker XRF Training School in New Zealand

After many years of running a successful XRF training school in Australia, Bruker travelled across the ditch in May and held the first XRF school in Wellington, New Zealand.

Attracting Bruker XRF users from both the North and South Islands, the 5-day intensive course presented by Mr. Elvy Grigolato covered XRF theory and best practice for sample preparation, including a special fusion presentation by XRF Scientific and in-depth training in the Spectraplus XRF software.

The attendees came from a diverse range of industrial and academic areas including geological, cement, and aluminum industries. Cheryl Kemp from New Zealand Aluminium Smelters commented that she “found this course to be worthwhile and feel it has given me more confidence in the day to day running (operation and minor maintenance) of the XRF. Also the confidence to complete a full calibration if required. We also had the opportunity to get a look at some of the new features in the next version of the software. There are several features that will improve how we can use the software.” Cheryl also found valuable “being given a practical demonstration on the use of the polishing lathe will allow us to use ours with confidence and prolong the life of our platinum ware.”

Attendees enjoyed the sights in Wellington, including memorable visits to the Te Papa Museum, Anzac centenary exhibition and Weta Workshop.

We will be running another XRF course in Melbourne from October 26th to 30th. If you are interested in attending please contact Neil Hughes via email on neil.hughes@bruker.com or by phone on (03) 9474 7000.

Chris Kelaart
Bruker Pty Ltd

Thin-Film XRD Seminar Report

In conjunction with the installation of the new Rigaku SmartLab X-ray diffractometer at the Centre for Microscopy and Microanalysis at The University of Queensland, AXT brought out Rigaku’s thin film expert Shintaro Kobayashi to give a series of seminars in Melbourne (Swinburne University), Sydney (University of New South Wales) and culminating in Brisbane (The University of Queensland) with live demonstrations on the newly commissioned system.

The thin films and coatings that are probed range in thickness from fractions of a nanometer i.e. monolayers to several microns. They are used to modify the surface of a material to make it more suited to specific applications. Whether the requirement is electronic, wear resistant, biocompatible or purely aesthetic, coatings can make the surface of a material behave differently to the bulk or substrate. The development and refining of these thin films requires techniques such as X-ray diffraction (XRD) to be able to characterise them.

While XRD is a common technique for characterising bulk powders and solids, the analysis of thin films is far more demanding. This is largely due to the fact that in many cases, the films are very thin. Thus, using conventional XRD, penetration of the incident beam may go beyond the coating and signals generated by the coating may be drowned out by those from the substrate. Furthermore, thin films can be highly oriented due to the way in which they have been deposited or grown, adding further complexity to the analysis.

Mr Kobayashi, who works in the Rigaku Applications Lab in Tokyo has been working on thin-film XRD development since 2007, assisting customers with their analytical requirements and providing training in the area. His presentation was divided up into three sections:

1. Introduction to XRD, the Rigaku SmartLab, its capabilities and applications of XRD
2. Thin-film analysis using XRD
3. Measuring thin films using a multi-dimensional pixel detector

The SmartLab installed at The University of Queensland has been specifically configured for thin-film analysis. In particular it features an in-plane arm which allows the detector to pivot in the same plane as the sample. Kobayashi explained how this is of particular significance to thin films with a highly-oriented crystal structure which could potentially remain undetected using more conventional hardware. The in-plane arm also enables depth profiling which would be of interest to those working on Functionally Graded Materials (FGM), multi-layer coatings, coatings that have a diffusion layer or other similar systems where composition varies as a function of depth.

Course participants receiving a live demonstration of the capabilities of the new 9 kW Rigaku SmartLab.

He also explained how a diffractometer suited to thin-film analysis offers a range of analytical modes such as rocking curve analysis, X-ray reflectivity (XRR) and grazing-incidence XRD (GIXRD). These modes can provide valuable information about phase composition, preferred orientation, thickness, density and roughness, all of which are relevant to fields such as solar cells, optics, electronics, computing etc.

In the final section of his presentation, Kobayashi-san examined how different types of detectors (OD, 1D and 2-D) can be used for the various thin-film measurement modes and the benefits of each type of detector. As the prime example, he spoke about the HyPix-3000 next-generation Hybrid Pixel Array Detector (HPAD) capable of multidimensional detection, and its ability to seamlessly switch between each mode. The various detection modes were highlighted with a series of case studies which demonstrated the detectors' performance in the various scenarios.

Attendances at all seminars were excellent, with outstanding levels of participation in Brisbane, no doubt stimulated by the new SmartLab acquisition and the live demonstrations provided after the seminar. While the presentation contained a high degree of technical detail, Kobayashi-san kept the audience entertained with his human goniometer impersonations.

While the presentation and technical material was very relevant to the audience, one of the most pleasing things was the questions and audience interaction. Throughout the presentation, attendees, in particular younger students posed some complex questions to the presenter about their own research and how this type of analysis could benefit them. This was particularly encouraging as they showed a good understanding of the technology, demonstrating we have a strong crop of younger researchers coming through.

Feedback on the seminars has also been very positive, with attendees commenting on what they had learnt and the opportunity to speak directly to someone with applications expertise about their specific requirements. Their sentiments were probably best summed up by Alanna Fernandes from Bluglass who said, "It was great to hear Mr Kobayashi speak and to have an opportunity to talk to him and to receive feedback on our analysis procedures. It was definitely most helpful and informative, and also very interesting to hear about the new features of their system".

AXT would like to thank Materials Australia and the Australian Ceramic Society as well all three venues for helping to put these seminars together. We also hope to bring more international experts to Australia in the near future to help educate and raise awareness of technologies that can benefit Australian researchers.

Cameron Chai
AXT

Upcoming Events

9th Asia Oceania Forum for Synchrotron Radiation Research, and Australian Synchrotron Users Meeting

25-27 November 2015
National Centre for Synchrotron Science, Melbourne

The Australian Synchrotron is proud to host the 9th Asia Oceania Forum for Synchrotron Radiation Research (AOFSRR 2015), in conjunction with User Meeting 2015. The two meetings will share a joint program across three days, on 25-27 November, at the Australian Synchrotron's award winning National Centre for Synchrotron Science in Melbourne.

Early bird registration closes 30 September 2015. Submissions for oral presentations have now closed, but posters are still being accepted.

For more information:
Email: aofsrr2015@synchrotron.org.au
Website: http://aofsrr2015.synchrotron.org.au

AXAA is a proud sponsor of this event

Bruker XRF School

October 26 - 30
Bruker Pty Ltd Office, Melbourne

This course is aimed at intermediate Bruker XRF users who wish to extend their knowledge of XRF theory and practice with best practice on Spectra plus software use and calibrations. The course will also include a ½ day workshop presented by Danny Verbeeten on fusion sample preparation. For more information please contact Neil Hughes: neil.hughes@bruker.com (03) 9474-7000

X-ray Materials Analysis Internet Courses – Wavelength Dispersive XRF and Powder XRD

Mode of Instruction for XRF and XRD Courses

These internet-delivered courses provide XRF and XRD analysts, particularly those new to X-ray analysis, with on-site and/or at-home instruction on the underlying principles and principal analytical methods. Features of the two courses -

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

The courses have a substantial cohort of international and local participants, and are being used by companies as vehicles for in-house XRF and XRD training.

Courses Director: Dr Brian O'Connor

Internet XRF Course: Series 8, 2015

The Internet XRF Course comprises modules on - XRF Overview; X-ray Excitation of the Specimen; X-ray Dispersion and Detection; XRF Data Measurement; Data Analysis Basics; Methods of Quantitative Analysis; Absorption-Enhancement Corrections; Specimen Preparation; Major Component Analysis Using Fusion Buttons; Trace Element Analysis Using Powders; and Analysis of Sub-Milligram Environmental Samples.

Course fee: $2,850 including GST

Internet XRD Course: Series 3, 2015

The internet XRD Course comprises modules on - XRD Overview; Essential XRD Fundamentals; XRD Measurement Strategies (I); XRD Measurement Strategies (II); Search/Match Identification Analysis (I); Search/Match Identification Analysis (II); Case Studies in Search/Match identification Analysis; Phase Composition Analysis Using Line Intensities; and Introduction to Advanced Methods (indexing, Rietveld phase analysis, structure solution, etc.)

Course fee: $2,850 including GST

Further Information and Enrolment Procedure:

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

AXAA Website and Contacts

Please visit our website, www.axaa.org, for further information.

NATIONAL COUNCIL PRESIDENT:
Nathan Webster
CSIRO Minerals Resources Flagship, Box 10, Clayton South, VIC 3169
Telephone: (03) 9545 8635
e-mail: nathan.webster@csiro.au

NATIONAL COUNCIL VICE PRESIDENT:
Vanessa Peterson
Bragg Institute, ANSTO, Locked Bag 2001, Kirrawee DC NSW 2232
Telephone: (02) 9717 9401
e-mail: vanessa.peterson@ansto.gov.au

NATIONAL COUNCIL SECRETARY
Natasha Wright
CSIRO Manufacturing Flagship, Normanby Rd, Clayton, VIC 3168
Telephone: (03) 9545 2041
e-mail: natasha.wright@csiro.au

NATIONAL COUNCIL TREASURER
Gordon Thorogood
Institute of Materials and Engineering, ANSTO, Locked Bag 2001, Kirrawee DC NSW 2232
Telephone: (02) 9717 3183
e-mail: gordon.thorogood@ansto.gov.au

NATIONAL COUNCIL NEWSLETTER EDITOR
Mark Styles
CSIRO Manufacturing Flagship, Private Bag 10, Clayton South, VIC 3169
Telephone: (03) 9545 8179
e-mail: mark.styles@csiro.au

Please email contributions for Issue 3 of the 2015 AXAA Newsletter to Mark Styles by Friday the 4th of December. Any comments or feedback about the Newsletter are welcome.

AXAA Membership

All registered participants of the AXAA-2014 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 provide their CV and a short statement about how they intend to contribute to the organisation. Please send these to the National Council Secretary Natasha Wright (see AXAA contacts) if you would like to apply.

Trial the latest Bruker equipment at our new Applications Facility in Melbourne

Bruker Pty Ltd has established a dedicated Applications Facility in the Melbourne head office for the demonstration and development of XRF and XRD applications. Customers and prospective users of Bruker equipment in Australia, New Zealand and across the APAC region are invited to come and trial the instruments and ask questions of our scientists. Customers will have the opportunity to thoroughly examine instruments without interruption or the pressure of quickly returning the equipment to service, as can happen at a customer site.

The Facility features an S8 Tiger 4 kW XRF spectrometer with application packages for the analysis of geological majors and traces, cements, oil and polymers. The lab also has a Bruker S2 Ranger EDXRF benchtop instrument calibrated for cements and geological materials. The XRF instruments are supported by a dedicated applications scientist, Mr. Elvy Grigolato, who is on hand to conduct demonstrations and training. As part of the APAC region, the Australian office is involved in a wide range of projects which provides experience in topics not routinely requested in Australia. These include analysis of food and pharmaceuticals, polymers, RoHS samples, metals, multi-layer analysis (electronics, metal coatings to solar panels) and waste material recycling.

Used for training Bruker engineers in the latest software and service tools, the Facility is backed by a full sample preparation laboratory with equipment for pressed pellet, fused bead preparation and loss on ignition determinations.

Bruker has partnered with XRF Scientific to develop fused bead equipment including the recently released xrFuse2 fully automated cold-to-cold fusion instrument. This machine has full ceramics for contamination-free beads and industry-leading safety features. Along with a range of flux formulations, there is also equipment on hand for regular platinum labware maintenance.

The Applications Facility also features a self-contained D2 Phaser benchtop XRD instrument for rapid powder analysis. It uses a standard XRD tube operated at 300W and a Lynx-Eye 1-D Silicon strip detector. The instrument is supported by our XRD technical specialist, Dr. Martin Duriska, who can provide demonstrations, customer training and application support.

For further details, or to arrange a visit, please contact us on sales.anz@bruker.com or (03) 9474 7000.

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Melbourne Office:
1/28A Albert St Preston VIC 3072
PO Box 8432, Northland Centre VIC 3072
Ph: (03) 9474-7000
Fax: (03) 9474-7070

Sydney Office:
Unit 7, 163-167 McEvoy St, Alexandria NSW 2015
PO Box 202, Alexandria NSW 1435
Ph: (02) 9550-6422
Fax: (02) 9550-3687

Wellington Office:
Unit 6, 5 Bouverie St Lower Hutt 5012, NZ
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NZ Company #: 4042738

Bruker Launches OPTIMUS™ TKD - A Unique Solution for Transmission Kikuchi Diffraction in the Scanning Electron Microscope

BERLIN, GERMANY – 9 July 2015 – Bruker introduced today the new **OPTIMUS™ TKD** detector head for Transmission Kikuchi Diffraction (TKD) in the scanning electron microscope (SEM). This innovative new product features a horizontal phosphor screen that can be placed directly beneath electron-transparent samples. **OPTIMUS™ TKD** can be used interchangeably with the standard detector head of all Bruker *e*Flash EBSD detectors, giving easy access to both EBSD and TKD using the same detector.

The **OPTIMUS™ TKD** detector head provides optimum geometric conditions, resulting in two major advantages compared to TKD using standard EBSD detectors that use only a vertical screen. Firstly, the signal is acquired where it is strongest and secondly the produced patterns display the lowest possible distortion.

The OPTIMUS™ TKD detector head beneath an electron-transparent sample

Thanks to the gain in signal provided by **OPTIMUS™ TKD,** users can either acquire data faster using the same SEM probe current as before or obtain improved lateral spatial resolution by using lower probe currents. Alternatively, the SEM acceleration voltage can be reduced which improves the analysis of very thin samples, as low energy electrons are more likely to be diffracted on the grain lattices. The second advantage, minimal pattern distortion, leads to further improvement of both band detection and indexing accuracy.

OPTIMUS™ TKD can also be used to acquire Selected Area Electron Diffraction (SAED) patterns very similar to those seen in a traditional transmission electron microscope, but at a fraction of the cost and effort.

The ARGUS™ direct electron detection system integrated into **OPTIMUS™ TKD** is benefitting from the same ideal geometric conditions. Dark field and bright field images acquired with ARGUS™ in transmission mode show nanometer scale microstructural features as well as details like single dislocations or dislocation walls in deformed materials.

The **OTPIMUS™ TKD** detector head is designed to be compatible with all Bruker *e*Flash EBSD detectors. Existing detectors can be easily upgraded by trained users in less than 20 minutes. Depending on the measurement task at hand, users can switch between TKD and EBSD analysis. The new **OPTIMUS™ TKD** detector head works perfectly in combination with the Bruker TKD sample holder as well as with Bruker's XFlash® EDS detectors.

For further information, please contact Jens Bergmann, jens.bergmann@bruker.com or 0438-014-771

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1/28A Albert St Preston VIC 3072
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PANalytical contributes to Save the Children foundation

PANalytical, one of the world's leading suppliers of analytical instrumentation and software presented a 'cheque' representing € 8,000 to Ms. Wendy Flik from the Save the Children foundation. This substantial donation was generated by the most recent PANalytical Global Customer Satisfaction Survey.

As PANalytical puts the customer at the center of its business, customer satisfaction is a fundamental business driver. Customer satisfaction surveys are therefore a key instrument in measuring our performance and identifying improvement areas.

Traditionally PANalytical has donated € 5 to a chosen charity for every completed survey. The number of completed surveys 2014 has by far surpassed previous years and PANalytical's CEO Peter van Velzen was pleased to present a 'cheque' for the amount of € 8,000 to the international charity Save the Children.

Ms. Wendy Flik passionately stated that it is Save the Children's mission to achieve immediate and lasting change in the lives of children. Quality education for boys and girls is the key to a better future. Peter van Velzen explained that: "PANalytical has high ethical standards and takes its social responsibility very seriously. Supporting the education of children worldwide is part of PANalytical's contribution to create a better world".

PANalytical's CEO Peter van Velzen presents the cheque to Ms. Wendy Flik from Save the Children

About Save the Children
Save the Children saves the lives, dreams and futures of children by providing medical care and good-quality education and by creating better living conditions. Save the Children is the largest independent children's rights organization in the world. Every child aged 0-18 whose childhood is threatened, as a result of poverty or because they have been mistreated, abused or forced to flee, has the attention of Save the Children.

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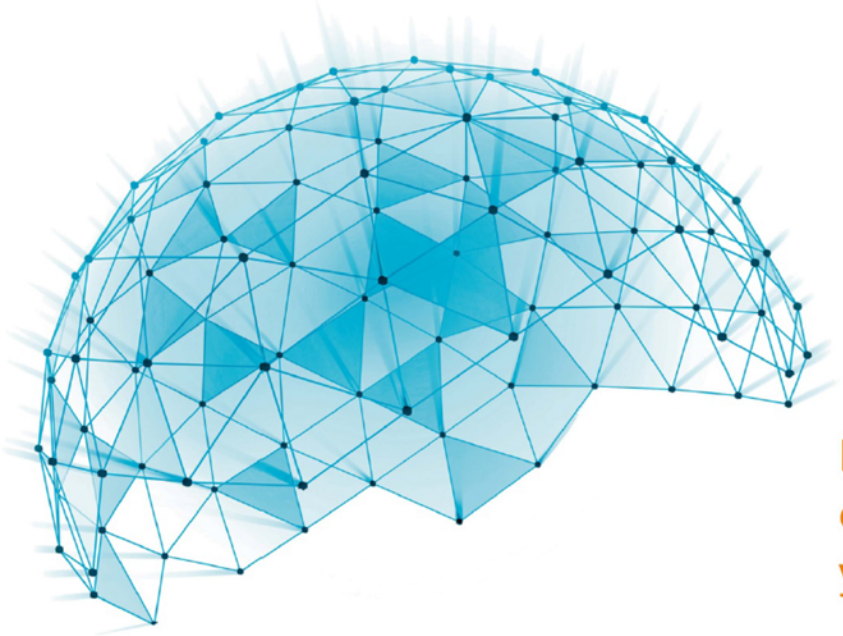

Promoting exceptional young scientists

For groundbreaking research using X-ray techniques

The PANalytical Award

PANalytical, the world's leading supplier of analytical instrumentation and software for X-ray diffraction and X-ray fluorescence spectrometry, continues to reward young scientists.

The PANalytical Award endorses an exceptional journal article dealing with the use of laboratory X-ray sources as the primary analytical technique.

The winning scientist receives a € 5 000 payment.

Only first authors who have never held a professorship are eligible for the award. An independent committee will select the winning entry.

No restrictions are placed on the manufacturer of the X-ray equipment that was used.

Closing date for applications 1 December 2015

For more information:
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- Obtain target precision faster compared to traditional WDXRF
- Obtain the lowest LLD across the periodic table faster through SumXcore technology

See the unexpected

- Track unexpected elements that can affect process element analysis without increasing measurement time
- Collect the entire spectrum for each sample without compromising accuracy

Increased confidence in quality

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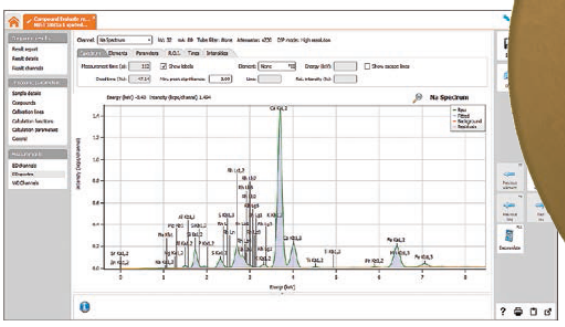
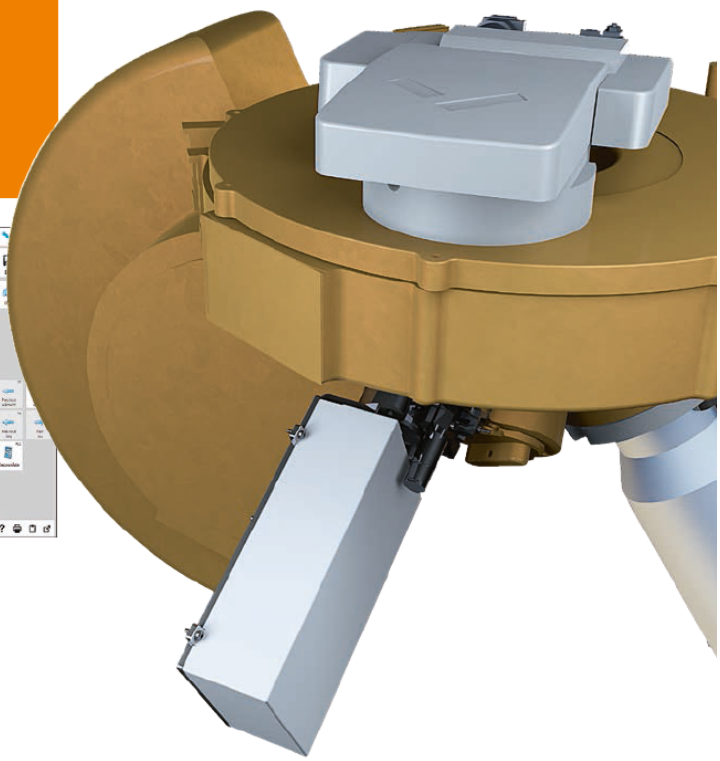
- Robust backup analysis

Key specifications

- **Elemental range of Na - Am**
- **Concentration range of ppm - 100 wt%**
- **Customized SDD detector for high X-ray flux environment**
- **Variable signal attenuation for optimum performance flexibility**
- **High count rate capability of up to 1 Mcps**



Spectrum acquisition



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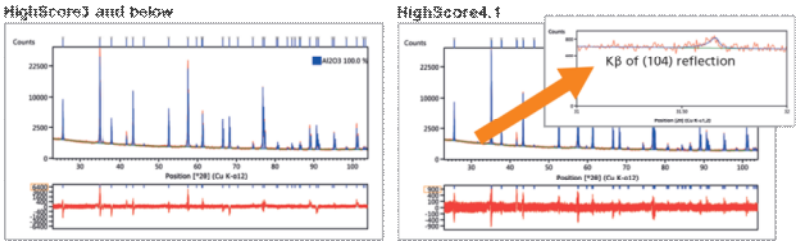

Significant improvements noted between experiment and model using the new HighScore 4.1 (right diagram) as seen from the difference plots beneath the diffractograms.

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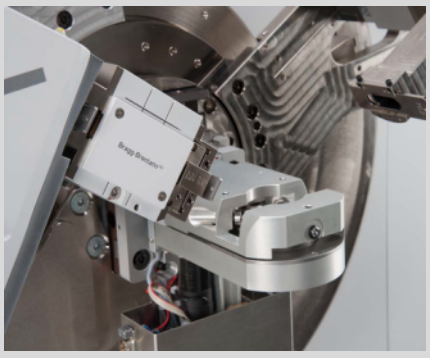

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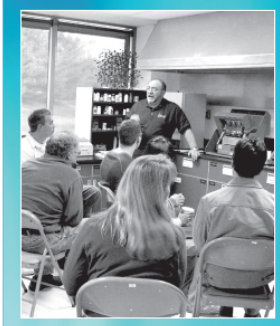
LET OUR TEAM OF EXPERTS HELP YOU TAKE YOUR SKILLS TO THE NEXT LEVEL!

Rietveld Refinement & Indexing Workshop:
Basic Workshop – 28-30 September 2015 / *Advanced Workshop – 1-2 October 2015

Powder Pattern Indexing and Rietveld structural refinement techniques are complementary and are often used to completely describe the structure of a material. Successful indexing of a powder pattern is considered strong evidence for phase purity. Indexing is considered a prelude to determining the crystal structure, and permits phase identification by lattice matching techniques. This workshop introduces the theory and formalisms of various indexing methods and structural refinement techniques. One unique aspect of this workshop is the extensive use of computer laboratory problem solving and exercises that teach method development in a hands-on environment.

Take the three-day basic workshop, the two-day advanced workshop or both together for a full week of hands-on training.



NEW! Polymer Diffraction 13- 15 October 2015

This three-day workshop is designed to provide more in-depth understanding of Polymer Diffraction. Learn the basics of XRD, polymer structure, morphology and diffraction data collection, polymer crystallinity, diffraction peak shape analysis, orientation analysis, small-angle scattering, special methods and advanced techniques.

Practical X-ray Fluorescence: 25 – 29 April 2016

From theory to hands-on exercises, this course offers techniques and skills to improve lab performance. Discover the latest in cutting-edge instruments such as TXRF, hand-held devices, energy dispersive and wavelength dispersive spectrometers through live demonstrations.

The XRF course covers the basics of X-ray spectra; instrumentation design; methods of qualitative and quantitative analysis; specimen preparation and applications for both wavelength and energy dispersive spectrometry. Emphasizing quantitative methods; use of automated X-ray spectrometers; review of mathematical matrix correction procedures and new developments in XRF.

Fundamentals of X-ray Powder Diffraction 16 – 20 May 2016

For the novice with some XRD knowledge or for the experienced with an interest in the theory behind XRD, this clinic offers a strong base for increased lab performance.

The clinic covers instrumentation, specimen preparation, data acquisition and qualitative phase analysis. Hands-on use of personal computers for demonstration of the latest software; data mining with the PDF. The powder diffractometer: optical arrangement, factors affecting instrumental profile width, choice and function of divergence slit, calibration and alignment, detectors, X-ray optics.

Advanced Methods in X-ray Powder Diffraction 23 – 27 May 2016

For the experienced XRD scientist, this session offers enhanced analysis skills through intense problem solving, as well as an introduction to the Rietveld Method. Emphasizing computer-based methods of data collection and interpretation, both for qualitative and quantitative phase analysis.

The advanced clinic covers factors affecting d-spacings of crystals: unit cell, crystal structure, and solid solutions, as well as factors affecting diffraction-line intensities: relative and absolute intensities; structure-sensitive properties (atomic scattering and structure factors), polarization effects, and multiplicity; specimen-sensitive effects (orientation, particle size), measurement-sensitive effects (use of peak heights and peak areas), and choice of scanning conditions.

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Powder Diffraction

An International Journal of Materials Characterization

Published quarterly (four issues and one topical supplement) by Cambridge University Press

Powder Diffraction (PDJ) focuses on materials characterization employing X-ray powder diffraction and related techniques. With feature articles covering a wide range of applications, from mineral analysis to epitactic growth of thin films to advances in application software and hardware, this journal offers a wide range of practical applications.

Proceedings of the Australian X-ray Analytical Association (AXAA) Conference held February 9-13, 2014

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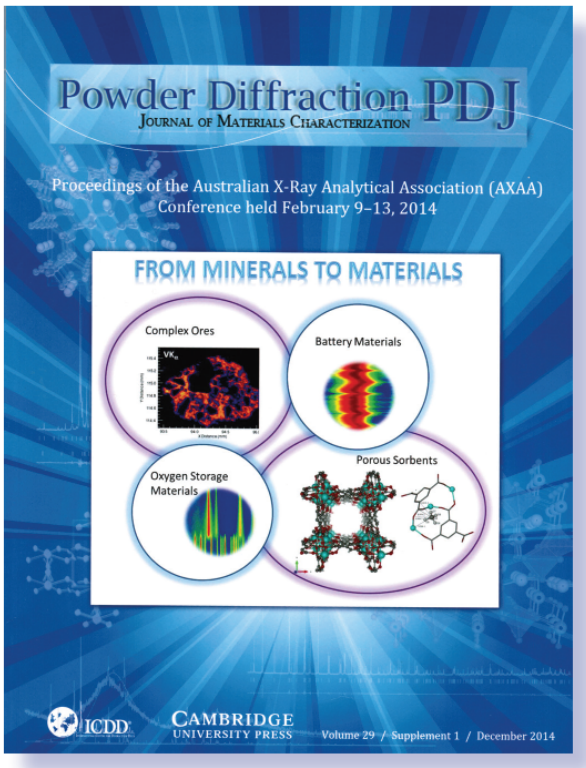

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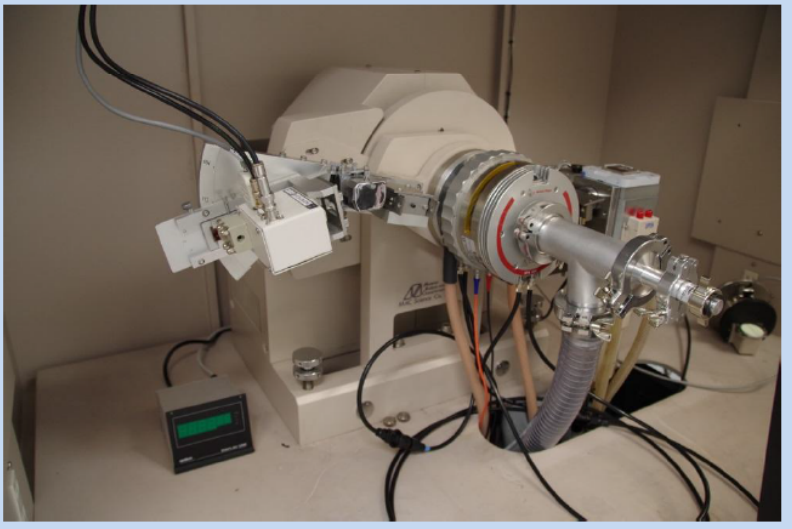
Diffraction Technology

Instruments and accessories for X-ray analysis

How to give an older instrument a new lease of life

The MAC Science diffractometer dating from about 1997 was unuseable due to outdated operating system and software (UNIX based). The generator, goniometer, shielding etc were all OK. The user also wanted a high temperature facility so a Paar HTK 16N was fitted together with vacuum and gauges. All automation was discarded and replaced with a current model GBC 122E controller. It is now another working XRD in the Laboratory at a fraction of the price of a new instrument.

The goniometer was updated with new stepping motors, new home and new limit switches. A GBC Xe proportional detector was fitted to the monochromator. The auto slits were replaced with fixed slits The tubeshield was updated with new warning lamps and a re-wired shutter assembly to conform to current safety requirements. Likewise the shielding cabinets and warning lamps were re-wired. A spinning stage was also fitted with a new motor and controls.



This is the sort of work we can do. If you have an instrument in the same predicament, please don't hesitate to contact me…
Rod Clapp , Diffraction Technology

Diffraction Technology e-mail diffraction@bigpond.com phone 03 9787 3801 Web www.diffraction.com.au

PRESS RELEASE

Australia's Highest Power X-Ray Diffractometer Installed at the University of Queensland

Sydney, Australia, July 14, 2015 – AXT have recently completed the installation and commissioning of Australia's highest powered X-ray diffractometer (XRD) at the Centre for Microscopy and Microanalysis (CMM) at The University of Queensland. The Rigaku SmartLab features a 9kW rotating anode X-ray source and the system has been configured for thin film analysis with Rigaku's unique in-plane hardware.

This instrument is truly world-class and will ensure that Australian research is able to keep pace with other leading research institutions around the world. The X-ray flux is second only to a synchrotron. It will enable high-sensitivity measurements to be achieved, making detection of trace phases and ultra-thin films possible, while at the same time increasing throughput and speeding up research.

The unique in-plane arm allows detailed studies to be carried out using data collected perpendicular to the sample surface. This ability is of great relevance to thin film analysis, as thin films and coatings are often highly oriented due to the manner in which they are deposited or grown. This opens the door to perfect pole figure determination which would not otherwise be possible as well as sophisticated orientation studies that would be extremely time-consuming using a more conventional goniometer. Coupled with the SmartLab's hardware that maintains the sample in the horizontal plane and high X-ray flux, this makes the ideal combination for thin film analysis.

The SmartLab is also the perfect partner for high-end research. Once installed it requires little maintenance and can be readily re-configured to perform a range of differing measurements. Its user-friendly guidance software holds the operators hand and walks them through any hardware changes. Smart components that the software can identify ensure configuration changes are made correctly. Smart cabling and Rigaku's patented auto align functionality are additional features that make the system suitable for use by non-experts, maximising measurement time and minimising the time required for training.

The Guidance software also helps optimise experimental parameters. It will help The University of Queensland researchers perform a range of thin film measurements such as:

- Out-of-plane diffraction
- High-resolution XRD
- In-plane XRD
- X-Ray Reflection (XRR)
- Small Angle X-Ray Scattering (SAXS)
- Pole figures
- Rocking curves

While the diffractometer has been designed for thin film diffraction measurements, it can also be easily configured to analyse bulk materials such as powders and solids and has the ability to carry out spatially-resolved (micro-) diffraction and in-situ temperature studies. Additional flexibility is possible with the addition of the battery cell attachment, giving UQ researchers access to a state-of-the-art instrument. There are also a host of other attachments available that can be added at a later date that provide even further levels of versatility. In combination with the SmartLab's high flux, this system provides perhaps the most futureproof solution available on the market today.

Dr. Kevin Jack, Acting Director of the CMM said of the installation of the SmartLab, "we are very excited about this new acquisition. It makes an excellent addition to the analytical capabilities of the centre and will be extremely beneficial to our researchers who are developing materials for a range of applications such as solar cells, electronic devices, membranes, fuel cells, semiconductors and biomaterials. We have a number of users queuing up to use it already."

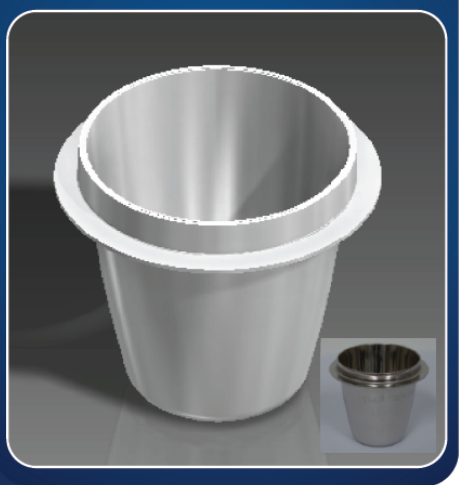
The system commissioning coincided with thin film XRD seminars presented by Rigaku's thin film expert Shintaro Kobayashi. Successful seminars were held in Melbourne at Swinburne University and Sydney at the University of New South Wales, before the main event at the University of Queensland that included live demonstrations of the SmartLab's functionality and capabilities.

Rigaku is represented in Australia and New Zealand by AXT who provide sales, service and technical assistance with product lines including ray diffraction, X-Ray Fluorescence (XRF), radiographic non-destructive testing (NDT), thermal analysis and computed tomography (CT), as well as the Rigaku Oxford Diffraction of single crystal diffractometers. For more details please visit www.axt.com.au or call (02) 9450 1359.

IMP Introduces Zirconia Stabilised Platinum Ware

Traditional platinum ware consisting of platinum alloyed with gold or rhodium has been used in XRF fusion techniques for years. With the advent of high throughput, fully automated fusion machines, these conventional alloys have been found to be inadequate. The introduction of zirconia (ZrO_2) stabilized Pt 95% Au 5% platinum ware has allowed it to meet the demands of these high throughput automated applications.

Introducing finely dispersed grains of ZrO_2 on the grain boundaries dramatically reduces grain growth and creep in the alloy. This equates to no structural changes during recrystallization.



The ZrO_2 stabilised platinum ware results in:

- Dramatic increase in chemical resistance
- Improved mechanical strength at temperature
- Two fold increase in tensile and yield strength
- Significant increase in creep resistance
- 20% increase in maximum working temperature range
- Significant reduction in tendency to warp or deform
- More than a 3X longer working life

All of this equates to significant savings with regard to expenditure on platinum ware over the lifetime of a laboratory. IMP is able to supply a full range of zirconia stabilised platinum ware for any sort of analytical requirement. Please get in touch with us to discuss your specific platinum needs.

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Challenging Conventional Sample Processing!

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HP-DT2 Automated Flux Weighing Machine

The HP-DT2 automated flux weighing machine is capable of weighing out one portion of flux every two minutes to an accuracy of ~2 mg. The system has a built-in load cell capable of measuring to 0.1mg. If the tolerance is increased, then the throughput dramatically improves to several vials per minute.

Trays of 30 vials are placed in the weighing system and it processes the entire tray automatically. The system can accept up to three trays of vials at a time.

The operator simply places a tray into the machine and presses the start button. All other processes are fully automated.

The system can be used to add fixed amounts of flux to the vials which is the standard mode of operation, or optionally the catch weight of the sample can be added to the vial prior to placing the vials in the trays.

The unit can then make up to the required dilution ratio by adding the correct amount of flux.

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