Australian X-ray Analytical Association
Newsletter Issue 1 2019

President's Address

Dear AXAA Members and Friends,

As announced in the preceding Newsletter, the AXAA-2020 Conference and Exhibition will be held from 27th April to 1st May 2020 at Bond University on the Gold Coast. AXAA-2020 will be an essential event for those in industry and academia who are working, researching and driving development in the fields of X-ray fluorescence, X-ray diffraction, and in synchrotron, neutron and other scattering techniques. Fields include, but are not limited to, mining and minerals processing, materials, energy, environment, biomaterials, cultural heritage, on-line analysis, technique development, and we are also planning a session celebrating the career and contributions of Keith Norrish. It's sure to be another fantastic event.

The AXAA-2020 website will go live shortly, with details of important dates and sponsorship opportunities. Details will also become available on the AXAA-2020 Facebook page and on Twitter (@axaa_org). The Conference Committee will meet in May to begin the design of an exciting and stimulating scientific, educational and social programme, and if you have any ideas about any of these aspects, please be in contact. Please also consider nominating yourself or someone else for the AXAA awards, which are to be presented at AXAA-2020.

Finally, I'd like to welcome a new member of the AXAA National Council, Dr Brianna Ganly. Brianna is a Research Scientist at CSIRO's laboratory at Lucas Heights in NSW, and would be known to many in the AXAA community having made a prize-winning presentation "Particle Size Effects in X-ray Fluorescence Analysis of Industrial Slurries" at AXAA-2017. Brianna is very passionate about X-rays, and has expertise in XRF and on-line analysis of industrial processes, in particular.

Nathan Webster
AXAA President

Natural speciation of silver in sulfide minerals by synchrotron X-ray microprobe analysis

Rong Fan (CSIRO, formerly University of South Australia)

Synchrotron X-ray microprobe analysis of a Ag-bearing ore was conducted at the Advanced Light Source (ALS) to characterise the speciation of Ag and its correlation with other elements, e.g., Cu, Pb and Zn, within the finely segregated heterogeneous ore matrix. These measurements, using synchrotron microprobe X-ray fluorescence mapping (μ-XFM), X-ray diffraction (μ-XRD) and X-ray absorption spectroscopy (μ-XAS), enable comprehensive characterisation of the Ag-bearing components, which are essential to design and/or modify floatation flowsheets for better recoveries of the Ag and other valuable metals (Fan et al. 2014).

The synchrotron microprobe measurements of the ore were conducted on beamline 10.3.2 of the ALS, Lawrence Berkeley National Laboratory, USA. (Marcus et al. 2004). Figure 1 shows a typical set-up for synchrotron microprobe experiments. The incident X-ray beam dimension was 5 × 5 μm. Characteristic elemental fluorescence emission intensities were measured with a Canberra seven-element Ge solid-state detector mounted at 90° to the incident beam and were normalized relative to the intensity of the incident beam. The chemical associations of Ag, As, Cu, Fe, K, Pb, S, Sr and Zn in the samples were imaged by scanning the sample stage using multiple beam energies. Figure 2 shows selected synchrotron μ-XFM elemental distribution maps of the sample. Fe is dominant over the region and the Fe rich minerals are mainly silicates, further confirmed by μ-XRD analyses.

The Ag rich grains (Spot 1 and Spot 2, red coloured particles in Figure 2c) are embedded in large silicate grains, up to 100 μm, and are significantly smaller in size. There is strong correlation between Ag and S which can be seen as purple coloured regions within the μ-XFM map (Figure 2e). No positive correlation among Ag, As and Pb was observed.

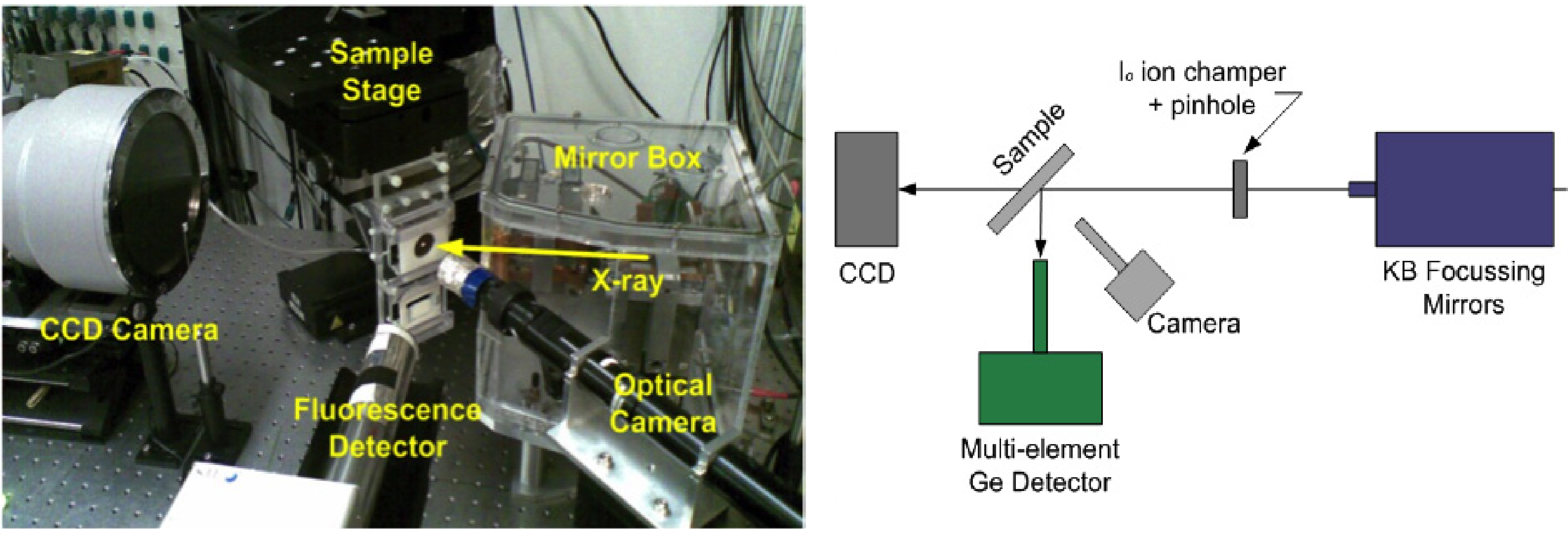
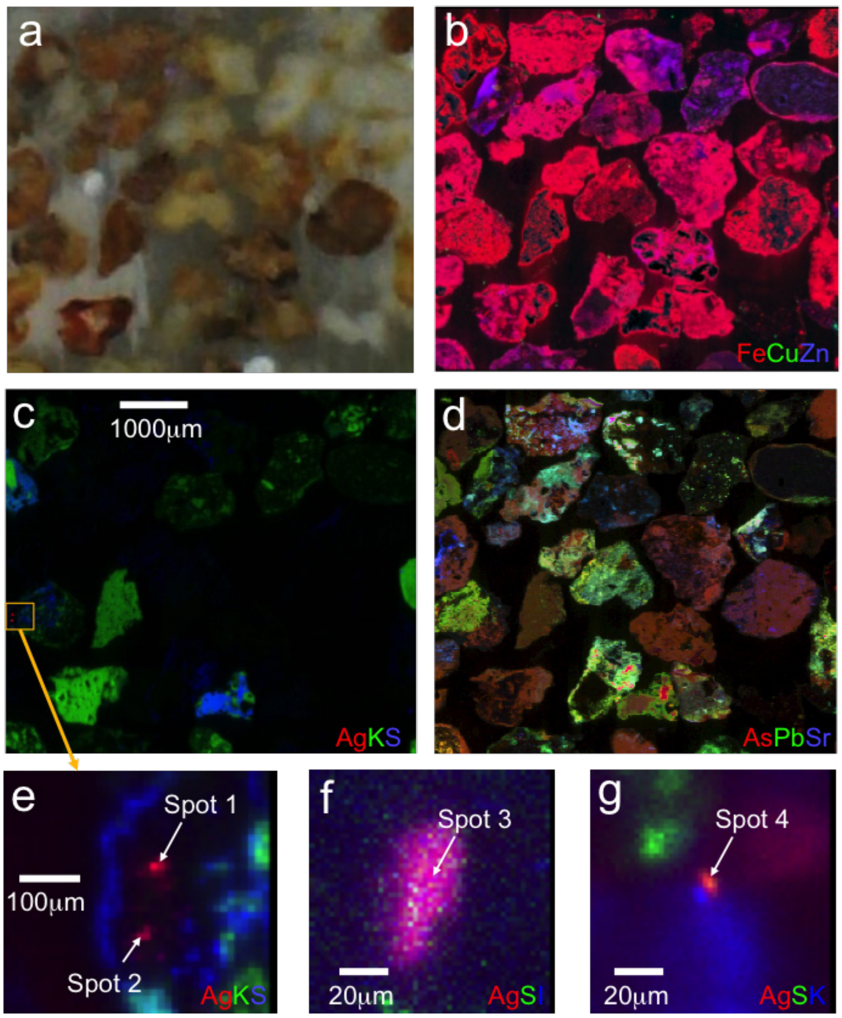


Figure 1 *View (a) and typical layout (b) of synchrotron X-ray microprobe experimental set-up. The view is from end-station ID-13C of the Advanced Photon Source. The diffraction detector (usually a CCD camera) is sitting behind the sample for transmission mode X-ray diffraction measurements. A fluorescence detector (high performance Ge or Si(Li)) is oriented at 90° to the incident micro-beam for µ-XRF and XAS measurement.*



Other µ-XFM maps were also obtained and two further Ag-containing grains, Spot 3 (Figure 2f) and Spot 4 (Figure 2g), were selected. In Spot 3, Ag is strongly correlated with iodine (I) rather than S. In contrast, Ag is found to be correlated with S and As in Spot 4.

µ-XRD results of Spot 4 (Figure 3) suggests the presence of quartz and pearceite ($(Cu(Ag,Cu)_6Ag_9As_2S_{11})$, a Ag-bearing sulphide. This is consistent with the µ-XRF results which shows the abundance of Ag and S in the grain. Moreover, it appears that, from similar µ-XANES spectrum, Ag in Spot 4 is more likely to be present in pearceite (Figure 4).

Figure 4 also shows the µ-XANES (X-ray absorption near edge spectroscopy, a subset of XAS) of the other selected Ag-containing grains as compared to pearceite, proustite (Ag_3AsS_3) and acanthite (Ag2S). µ-XANES spectra show clearly significant difference among different Ag-containing sulfide mineral groups. The similarity of µ-XANES data among acanthite, Spot 1 and Spot 2 suggests that Ag in Spot 1 and Spot 2 may be associated with a sulfide, possibly acanthite, rather than sulfosalts.

Figure 2 *Optical map of a selected region of the ore sample (a), and synchrotron µ-XFM maps (b-d) for the same region. Two locations (Spot 1 and 2) were chosen for µ-XAS analysis (e). Other µ-XFM maps were also obtained and two further Ag-containing grains, Spot 3 (f) and Spot 4 (g), were selected.*

Ag in spot 3 is probably associated with iodargyrite (AgI, silver iodide) given that both Ag and I but no S is present. This is consistent with its XANES spectra which shows different features compared to other Ag grains and sulfide standards and an adsorption edge at greater energy by approximately 1 eV.

Mineralogical investigations of the Ag in ores may provide important information towards the understanding of the behaviour of Ag and other metals during industry floatation processes. For example, it is noted that lime has little effect on floatation of acanthite but deleterious on floatation of pearceite, proustite and pyrargyrite (Ag_3SbS_3) (Gasparrini 1984). Starch has a positive effect in improving concentrate grade of the Ag in the floatation of acanthite but should not be used in the floatation of proustite. Lime and starch may therefore be considered for better recoveries of the Ag in the ore type of the sample.

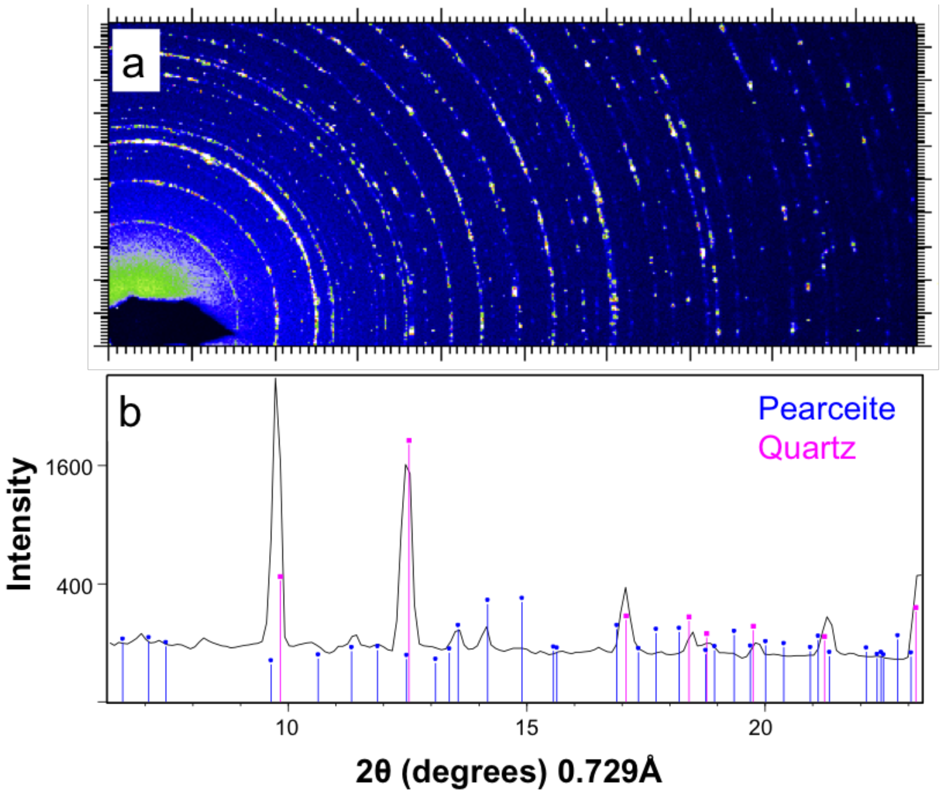


Figure 3 *XRD pattern from the location selected from the μ-XRF map (Figure 2g): (a) 2D μ-XRD image showing both continuous diffraction rings and discrete spot, and (b) XRD trace extracted from the CCD image using the Fit2d software (Hammersley 1999). Background removal and subsequent phase identification were performed with the HighScore Plus software with the PDF4 database.*

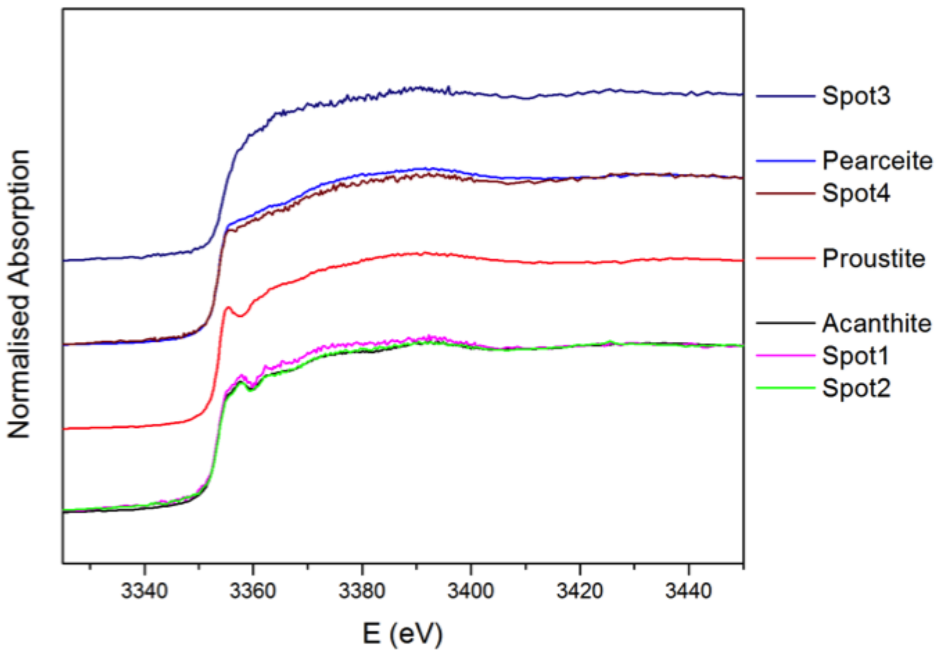


Figure 4 *Comparison of normalized XANES at the Ag L3-edge of selected locations from the μ-XFM maps of the ore and the spectra of Ag references: pearceite, proustite and acanthite.*

References

Fan, R. and A. R. Gerson (2014). Development of advanced methods for examination of Ag mineralogy and flotation losses. XXVII International Mineral Processing Congress, Santiago, Chile.

Gasparrini, C. (1984). "The mineralogy of silver and its significance in metal extraction." CIM Bulletin 77: 99-110.

Hammersley, A. (1999). FIT2D, version 10.31. Tech. Rep. Grenoble, France, ESRF.

Marcus, M. A., et al. (2004). "Beamline 10.3.2 at ALS: a hard X-ray microprobe for environmental and materials sciences." Journal of Synchrotron Radiation 11(3): 239-247.

CT in additive manufacturing

Cameron Chai (AXT Pty Ltd)

As Australia's manufacturing industry moves away from mass manufacturing and towards specialised small-scale and batch manufacturing, quality rather than price becomes our key differentiator. This highlights the need to implement testing procedures such as computed tomography (CT) to ensure consistent quality, especially in the area of critical components.

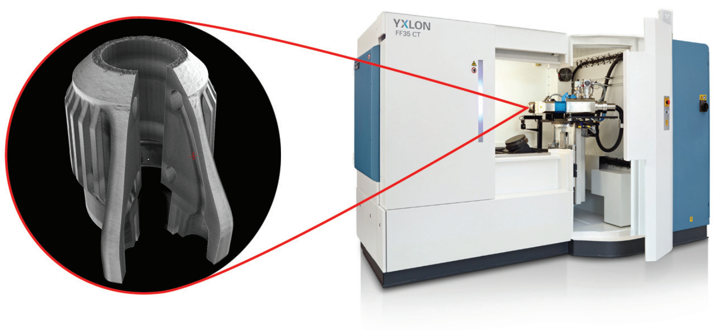
Many manufacturers are adopting 3D printing or additive manufacturing processes which suit their requirements. These may include reduced cost due and faster turnaround times with no need for tooling as well as opening up new design possibilities that were not possible using computer-controlled automated processes such as CNC machining and other conventional manufacturing processes. Minimal labour input also helps keep costs down and negates high labour cost to help Australian manufacturers remain competitive.

As in any manufacturing operation, quality control (QC) is an integral part of the operation. The major difference with small scale manufacturing is you can't test one in every thousand parts to get a representative picture of the entire production run. In many instances, only one or two components may be manufactured and with these components potentially being critical to a machine or structure, testing each and every component can be imperative.

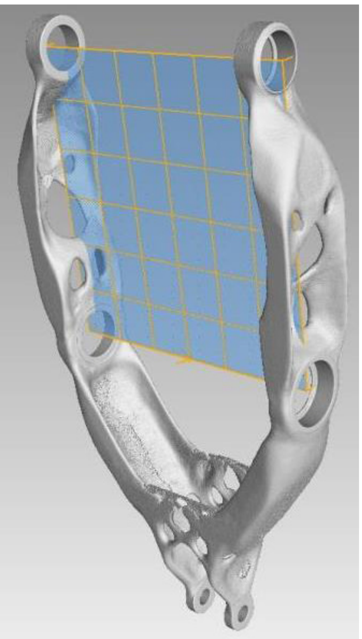
For simple components, traditional QC techniques such as optical microscopy and CMM (Co-Ordinate Measuring Machines) metrology might be sufficient, but can struggle to identify small manufacturing flaws. With more complex designs, these techniques can be totally inadequate, especially where internal or undercut features such as channels exist which are inaccessible by line-of-sight techniques. Non-destructive X-ray based techniques such as radiography and CT are ideal in these situations, in particular for metal components. They are analogous to medical techniques, just requiring higher power X-ray sources to penetrate the denser materials.

An aluminium component produced by additive manufacturing, analysed by CT to reveal areas of porosity.

While radiographs can produce 2D images, CT can see both surface and internal structures and has the ability to produce 3D images. In addition, CT can also create computer-generated reconstructions that can be assembled/disassembled and examined in detail to identify sub-micron defects and pinpoint them within the structure. Defects such as pores and voids in particular can be caused by non-optimal processing parameters, existing pores in raw materials or variations in raw materials e.g. particle size. Causes can even extend to feed powders and identification of unmelted particles in multi-material systems. With the ability to act as a high precision Non-Destructive Testing (NDT) technique, CT provides details and information that can help diagnose these issues, such that processing can be optimised and production problems dialled out in subsequent production runs. Post production, CT can be used to check dimensions against CAD drawings and to report back on any deviations.



A welding nozzel imaged using CT. This cutaway view shows the internal channels.



Helicopter bell crank produced by additive manufacturing where CT was used to check dimensional tolerances. The rough nature makes it difficult to achieve via CMM due to the rough surface

While CT is an established NDT technique used in industries that typically lead future development such as aerospace and motorsport, it is becoming more commonplace. As with the advanced materials that these industries work with, the price of CT systems will come down as the technology becomes more widely used and its application better understood, which will in turn accelerate its adoption. In terms of additive manufacturing, it is already being used by aircraft and medical implant manufacturers. With Australia needing to rely on quality rather than price to generate manufacturing exports, CT needs to be embraced either by the manufacturers themselves or by third parties providing CT as a testing service.

When questions are raised about the properties of 3D printed components, relating to their structural integrity, manufacturing tolerances, layer defects, residual stresses and material inclusions, CT stacks up as an indispensible QC tool.

Further XRF Training Success for OTML Staff in Papua New Guinea through the Internet XRF Course

Brian O'Connor

Four more staff from Ok Tedi Mining Limited (OTML) in Tabubil, which is situated the Western Province of Papua New Guinea (PNG), have completed Advanced Level certification for the Internet XRF Course directed by Dr Brian O'Connor of Perth. The Advanced Level XRF course is designed for people capable of developing new XRF methods as well as conducting day-to-day analyses. OTML staff have been trained in this way since 2007 following a joint initiative by Brian O'Connor and former OTML luminary Brian Evans. Almost 40 OTML trainee staff have completed the I-XRF course since then.

In addition to Brian O'Connor running the course via the internet, strong local mentoring support is now provided by Gloria Samiak, OTML Superintendent of Laboratory Services, who has succeeded Hillary Turnamur in that capacity. Gloria and Hillary had been course participants in the initial years.

Gaining a course completion award for the Advanced Level course requires participants to gain a high mark in the assignments set for all 11 modules of the course. Those gaining a very high mark overall are awarded Advanced Level certification 'With Distinction'.

The latest awards were presented at Tabubil in February, 2019. The OTML staff completing the course and their OTML mentor are shown in the photograph with their certificates:

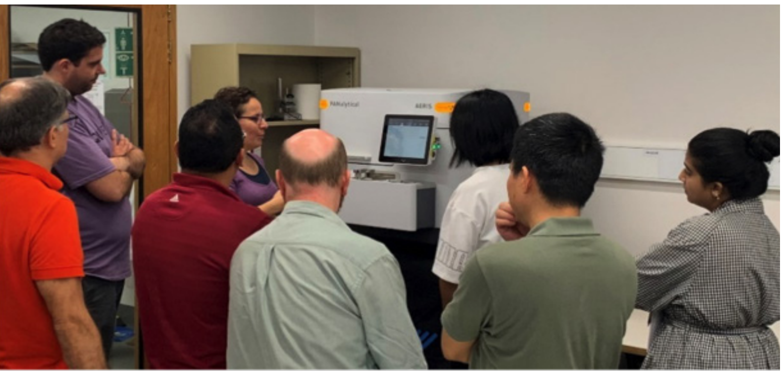
Vivienne Peter, Wesley Kiwingim, Gloria Samiak (Superintendent Laboratory Services), Yaki Meksie and Constance Vengiau. Wesley was honoured with a 'with distinction' certificate.

XRD hands-on learning workshop in University of Queensland by Malvern Panalytical

Samantha Singh (Malvern Panalytical)

X-ray diffraction has undoubtedly been integral towards materials science research. On March 27th, Malvern Panalytical gathered the XRD community at University of Queensland for a day of XRD hands on learning; particularly on how to get better data quality as well as make better analysis & interpretation. This workshop was led by their application specialist, Dr Olga Narygina, who is based in Brisbane. It was well attended by more than 30 researchers.

Dr Narygina, together with the researchers at University of Queensland, shared about different applications of materials research using XRD. From basic powder XRD for the characterisation of geological and synthetic materials, to advanced applications concerning in-situ and in-operando X-ray diffraction. A highlight of the session was the hands-on session on Malvern Panalytical's new compact XRD, Aeris. Researchers had the opportunity to operate the



diffractometer to collect data on their samples. "We wanted these workshops to be of value to researchers. That's why we focused a lot on learning how to not only collect good data but also how to properly analyse and interpret the data," shared Dr Narygina. She demonstrated how to perform advanced analysis of large data sets, including automatic Rietveld refinement, principle component analyses and clustering and partial least square regression method.

This XRD applications workshop organised in Brisbane, is part of Malvern Panalytical's series of workshops conducted across the Asia Pacific region. Look out for these workshops in Perth and Sydney in second half of 2019. Interested for Malvern Panalytical to conduct a session in your institution? Contact Samantha at Samantha.singh@malvernpanalytical.com

Upcoming XRF & XRD Workshops

XRF Courses (Malvern Panalytical)

XRF in the Workplace	• *Perth 13th - 17th May* • *Perth 9th - 13th September*
SuperQ Hands on	• *Perth 18th - 20h June* • *Perth 21st - 23rd August*

XRD Courses (Malvern Panalytical)

XRD in the Workplace	• *Auckland 2nd - 4th July* • *Sydney 21st - 24th October*
HighScore Hands on	• *Perth 24th - 26th July* • *Sydney 29th - 31st July*

Contact Samantha for further details and/or to register for any of the above courses. (samantha.singh@malvernpanalytical.com)

Upcoming Conferences

Denver X-ray Conference & International Congress on X-ray Optics and Microanalysis

Westin Lombard Yorktown Center, Lombard, Illinois, U.S.A. 5th - 9th August 2019

Student awards to support conference attendance are available via the Jerome B. Cohen Student Award, and the Robert L. Snyder Student Award.

The deadline to apply for both awards is June 3rd.

[Website: http://www.dxcicdd.com/](http://www.dxcicdd.com/)

AXAA Website and Contacts

Please visit our website, www.axaa.org, for further information, or follow us on Twitter @axaa_org.

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AXAA Membership

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

Candidates should provide their CV and a short statement about how they intend to contribute to the organisation. Please send these to the National Council Secretary Mark Styles.

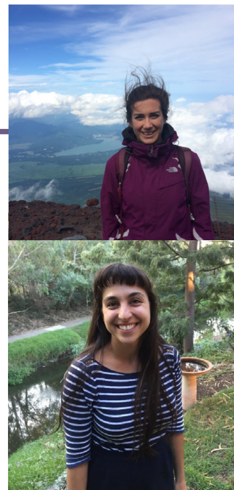
AXAA Resource Centre

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

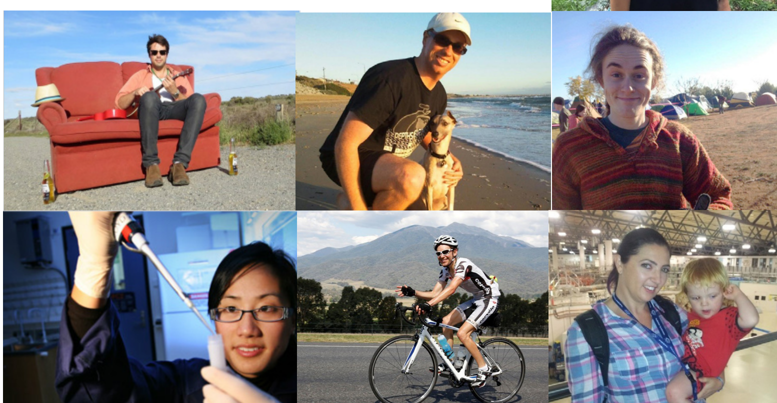
Next AXAA Newsletter

The next issue of the AXAA Newsletter will be distributed in August 2019. Please feel free to send contributions for the newsletter to Jessica Hamilton at *ausxray@gmail.com*. Any comments or feedback about the Newsletter are welcome.

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



We are seeking posts for our 'Day in the Life' series. If you'd like to contribute, or know someone who might be interested, please contact National Council Communications Editor Jessica Hamilton at ausxray@gmail.com.
W:www.axaa.org/a-day-in-the-life.html



Press Release :
3 times better performance with Next Gen Epsilon 1 XRF

Malvern Panalytical announces the new next generation of Epsilon 1 X-ray fluorescence (XRF) spectrometer. In terms of performance, expect 3 times the improvement in speed and sensitivity. This compact yet powerful instrument is a mainstay across industry, enabling fast and reproducible elemental analysis but now with greater flexibility and precision than ever before.

"The dramatic upgrade to the Epsilon 1's precision has been enabled by the integration of a high-power X-ray tube and a new high resolution Si-drift detector. The comprehensive upgrade has focused on harnessing all the power and sensitivity of a larger benchtop XRF instrument in a truly compact system – the new Epsilon 1 has a footprint of only 0.15 m^2 " says Hari Bhaskar, Malvern Panalytical application specialist.

Trace metals in pharmaceuticals, foods, soils and metal ores can now be quantified more quickly and accurately than ever before in an instrument of this size. The system's robust design prevents damage to its analytical heart by dust or oil, and ease of operation has also been improved, with the addition of a brighter more modern touchscreen.

For out-of-the-box simplicity and functionality without costly and tedious setup procedures, Epsilon 1 is available in a number of pre-calibrated versions which are dedicated to specific applications. The Epsilon 1 Lube Oil delivers ASTM 6481-compliant elemental analysis of unused lubricating oils; the Epsilon 1 Sulfur in Fuels quickly quantifies sulfur content in fuels according to ASTM D4294-10 and ISO 20847; the Epsilon 1 Academia enables rapid characterization of unidentified samples, using Omnian software for standardless analysis; and the integrated camera in the Epsilon 1 for Small Spot Analysis simplifies the investigation of very small objects, inclusions or inhomogeneities.

Press Play to see the newest features of the Epsilon 1 Next Gen

New Features

- LCD 10.4" touchscreen and 1024 x 768 Resolution
- Faster and 3 X more sensitive
- Higher-power X-ray tube, designed by Malvern Panalytical
- High Resolution Si drift detector
- 1.5 Mcps at 50% dead time
- Latest silicon drift technology
- Available in many pre-calibrated versions depending on application

Read More :

www.malvernpanalytical.com/Epsilon1

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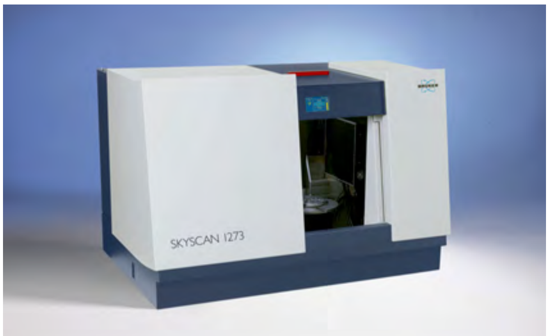
Bruker News Australia & New Zealand

Mar-19

Bruker Introduces New SKYSCAN™ 1273 Benchtop 3D X-ray Microscope



Bruker today announced the new SKYSCAN™ 1273 benchtop 3D X-ray microscope based on micro-computed tomography (Micro-CT) technology. The SKYSCAN 1273 sets a new standard for non-destructive testing (NDT) with benchtop instruments, providing a performance level previously only achieved by floor standing systems.

Samples with up to 500 mm length, 300 mm diameter, and a maximum weight of 20 kg can be investigated with powerful and precise positioning stages. The combination of a higher-energy X-ray source running at higher power (130 kV, 39 W) and a large format 6-megapixel flat-panel detector with ultimate sensitivity and speed provides excellent image quality in just a few seconds. The SKYSCAN 1273 produces 3D images of internal structures with high resolution based on a voxel size smaller than 3 µm.

The comprehensive software for straightforward data collection, advanced image analysis, and powerful visualization makes the SKYSCAN 1273 an easy-to-use 3D X-ray microscope. Micro-CT with helical scanning for distortion-free data acquisition and artifact-free reconstruction algorithms provides images without blurring, even for planar structures in all directions.

For high-speed requirements, InstaRecon®, one of the world's fastest CT reconstruction solutions, accelerates 3D imaging up to 100 times compared to traditional algorithms.

The SKYSCAN 1273 benchtop 3D X-ray microscope requires minimum lab space, is easy to use, and virtually maintenance-free to offer high system uptime with low cost of ownership.

Dr. Geert Vanhoyland, the Bruker AXS Product Line Manager for 3D X-ray Microscopy, commented: "The SKYSCAN 1273 is a turnkey solution for non-destructive 3D imaging, covering a variety of industrial and scientific applications. This includes defect detection for casting, machining, and additive manufacturing, inspection of complex electro-mechanical assemblies, pharmaceutical packaging, advanced medical tools, geological drill cores, and non-ambient microscopy."

Dr. Kjell Laperre, the Bruker BioSpin Micro-CT Market Product & Applications Manager, added: "The SKYSCAN 1273 significantly extends the capabilities in preclinical imaging. Its large sample compartment combined with the higher-energy, higher-power X-ray source allows ex vivo imaging of larger and higher density samples in forensic, orthopedic, paleontological, and zoology applications."

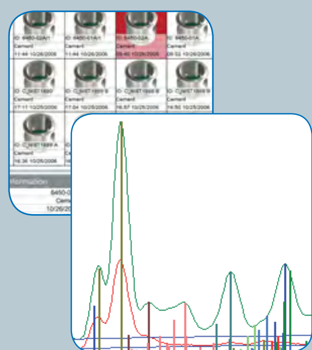
For more information about the SKYSCAN 1273, please visit **www.bruker.com/skyscan1273 .**

Upcoming Courses

Introduction to SPECTRA^plus V3/V4 : Learn the fundamentals of wavelength dispersive XRF and get an introduction to the external software package

Venue: Bruker Pty Ltd, 1/28A Albert St Preston VIC 3072
Dates: 14-17 October 2019

For further details, please scan the QR code (left) or visit: https://bit.ly/2TGR5Dp



AXT

PRESS RELEASE

Surface Science Capabilities of University of Sydney Nanoresearchers Taken to the Next Level

Sydney, Australia, March 20, 2019 - The University of Sydney has recently enhanced their surface science capabilities with the installation of a Thermo Scientific KAlpha+ X-ray Photoelectron Spectrometer (XPS). The system supplied by AXT Pty Ltd, Thermo Scientific's distributor was installed at Sydney Analytical, one of the university's Core Research Facilities.

The KAlpha+ is a compact XPS system that puts state-of-the-art measurement within the reach of more researchers through its ease-of-use. It is ideal for a multi-user facility, like Sydney Analytical, that caters for users with varying degrees of expertise and reduces pressure on facility managers.

The Thermo Scientific KAlpha+ XPS system installed at Sydney Analytical.

The KAlpha+ offers users of all levels improved spectroscopic performance, faster analysis times, improved elemental detection and the ability to acquire data at higher resolution. Furthermore, with the addition of the MAGCIS ion source, researchers will also be able to perform depth profiling experiments that will allow them to see how composition changes beneath the surface which will be of great value to those working with thin coatings and functionally graded materials.

According to Dr Michelle Wood, Senior Technical Officer in charge of the KAlpha+, "the XPS is quickly becoming one of our most actively used instruments, with various users commenting on both the quality of the data compared to other XPS instruments they have used as well as how user friendly the operation and software is." Sydney Analyical currently have around 20 users who already use the instrument constantly, with more operators expected to be trained shortly.

One area of research that has found XPS to be extremely useful is characterising the surface of biocompatible nanoparticles. Dr. Gurvinder Singh and Prof. Hala Zreiqat have been engineering the surface of nanoparticles aimed at diagnosing and treating cancers and other life-threatening diseases. XPS provides data to optimise the surface chemistry to increase the biocompatibility and functionality of the particles, as well as allowing researchers to understand biomolecular adsorption behaviour.

The areas of research that are utilising XPS are many and varied. Some examples include drug discovery, Metal-Organic Frameworks (MOFs), carbon and metal oxide nanoparticles for separation processes, catalysis, biomedical coatings and battery materials.

Prof. Peter Lay, Academic Director at Sydney Analytical said of the acquisition, "Sydney Analytical wanted a high-end instrument with multiple capabilities that was also user friendly in both data collection and analysis and the K alpha ticked all of these criteria. We are delighted that the K alpha has lived up to our expectations and has been embraced enthusiastically by our users." Sydney Analytical would like to acknowledge the University of Sydney Core Research Facilities program for providing funding to purchase of the KAlpha+.

XPS is just one of many technologies that AXT offer. They have a comprehensive portfolio of materials characterisation and analytical instruments to suit the needs of academic and industrial clients sourced from suppliers around the world. For more information, please visit www.axt.com.au.

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