AXAA e-newsletter

Issue 3, 2012 **Australian X-ray Analytical Association**

President's Address

Dear AXAA Members and Friends,

The weather has warmed up and so have our AXAA activities. Since our last newsletters we have had student seminars in both NSW and Victoria. Both events were well attended and showcased the work of some of our very talented student members. Read further on in the Newsletter for reports on both. Thanks to PANalytical for supporting the NSW event.

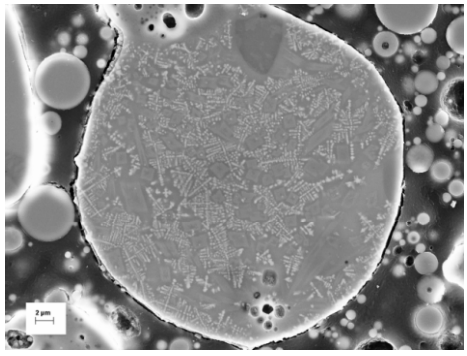
The student seminar days mark the start of increasing AXAA events in the lead-up to the 2014 National Conference and Workshops. National Council have finished site inspections and deliberations, and are pleased to announce that the event will take place in Perth! If you have interest in holding a pre or post workshop as a satellite to the main event, now is the time to contact us. Similarly, if members want to see something specific in the program we also greatly appreciate this information. Hope you enjoy our action-packed Newsletter!

Vanessa Peterson
National Council President

Matters for Scatterers

Fly ash is a by-product from coal fired power stations and is produced in large volumes in Australia (>10 MT/yr). It is used as a supplementary cementitious material for conventional concrete as well as other applications such as road stabilisation. However, there remains a gap between utilisation and material produced and currently the remainder is disposed of in landfill or tailings dams. A possible use for fly ash is as an aluminosilicate source for producing geopolymer binding materials. Geopolymers can be used for a wide range of applications from concretes to fire resistant barriers.

The challenge limiting the utilisation of fly ash in geopolymers is the varying reactivity of the ash which is influenced by the chemical and mineral composition. Researchers in the Centre for Materials Research at Curtin University have used lab based radiation and synchrotron radiation to quantitatively determine the crystalline and amorphous composition of various fly ashes. The composition of the amorphous phase is determined by subtracting the chemical contribution of the crystalline phases from the chemical composition of the bulk as determined by XRF. Fly ashes are a challenging material for x-ray analysis due to the high number of phases (>10 phases in some cases) with wide ranging x-ray absorption coefficients. A single particle can contain more than 5 phases (see SEM micrograph).



SEM image of a fly ash particle containing numerous phases.

In-situ high temperature diffraction experiments have also been performed on geopolymers to assess their phase stability when exposed to a simulated fire. It was found that some phase changes occurred in the non-geopolymer (unreacted source material) phases but they were to thermally stable phases which actually improved the performance of the material.

Photograph of a HT-XRD experiment performed at the Australian Synchrotron.

Dr William Rickard
w.rickard@curtin.edu.au
Centre for Materials Research, Curtin University

AXAA Student Seminar Days

NSW – "Scattering Matters"

In September 2012, the AXAA 2012 NSW Student Seminar day was held at Sydney University. Around 30 people attended this meeting to hear from our young researchers' work using X-ray, neutron, and other scattering techniques of analysis. There were seven speakers from three different Universities. The speakers and topics included:

1. Tom Whittle (USyd) - "Synchrotron X-ray Powder Diffraction Investigations of Relaxor Ferroelectric Materials with Tungsten Bronze Structures".

2. Emily Reynolds (USyd) - "Zirconium Pyrochlores".

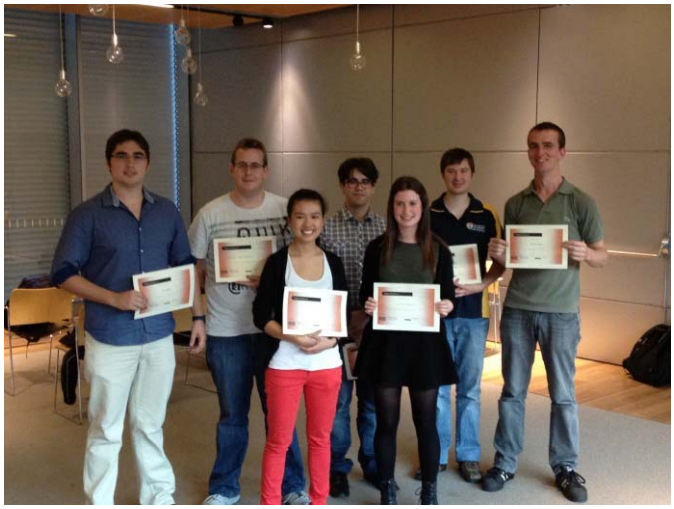
3. Joel Bertinshaw (UNSW) - "Multiferroic $BiFeO_3$ and $La_{0.66}Sr_{0.33}MnO_3$ Materials".

4. William Brant (USyd) - "Investigating Structure-Property Relationships in Cathode Materials via Combined *Ex-situ* and *In-situ* Diffraction Techniques".

5. Denissa Murphy (USyd) - "Structure and Property Investigations of Spinel Type $LiMnTiO_4$".

6. Tim Murphy (UWS) - "Dispersion of Bismuth in the Environment: a Chemical and Mineralogical Understanding".

7. Thomas Godfrey (USyd) - "Structural Investigation of Prussian Blue Type Compounds as Cathode Materials for Rechargeable Batteries".



Speakers at the NSW Student Seminar Day (L-R): Tim Murphy, Tom Whittle, Denissa Murphy, Joel Bertinshaw, Emily Reynolds, William Brant and Thomas Godfrey.

After the student's presentation, the audience joined the speakers for a drink and a few nibbles. I would like to thank the speakers for their fantastic and informative presentations and for making the meeting such a great success. I would also like to thank Vanessa Peterson for organizing this excellent meeting. On the night, the prizes went to:

First Place: William Brant (USyd)
Second Place: Joel Bertinshaw (UNSW)
Third Place: Tom Whittle (USyd)

Congratulations to the winners. The quality of your research work and your knowledge really showed in your presentations. I would have hated to have been one of the judges on the day, as all talks were of high quality. I would like to suggest that AXAA members and even other societies in future keep an eye out for this student seminar day. You would really learn a lot from the presenters and see the future quality students coming from different Universities.

On a final and very important note, thank you to PANalytical for sponsoring this meeting and putting up the prize money for the best presentations. Everyone that attended really appreciates the support you give the students, Universities and the AXAA Association.

Dr Richard Wuhrer
Advanced Materials Characterisation Facility (AMCF)
University of Western Sydney

VIC – "Something to Bragg About"

On 21st November 2012, the AXAA 2012 VIC Student Seminar day was held at CSIRO Process Science and Engineering in Clayton. The event was attended by about 20 people from across Melbourne and Geelong, and was kicked off by a plenary presentation by Dr Stacey Borg from CSIRO on the topic of "*In-Situ* X-ray Absorption Spectroscopy at Elevated Temperature and Pressure: Take it Away, mAESTRO". Stacey's thoughts and experience regarding "*in-situ* experiments at extreme conditions are the most challenging, but also the most rewarding" rang true with many in the audience and were wise words for the younger participants. This was followed by the first student presentation from Sitarama Raju Kada, a 4th year PhD student from Deakin University, who spoke about, "Application of Laboratory

Speakers at the VIC Student Seminar Day (L-R): Dr Stacey Borg, Renata Lippi and Sitarama Raju Kada.

Based XRD for High-Throughput Processing of Light Metal Alloys." The final student presentation was from Renata Lippi, a 5th year Bachelor of Engineering student from University of Sao Paolo and currently undertaking a 1 yr industrial traineeship at CSIRO, who presented on the topic of "Characterization of Ruthenium Nanoparticulate Formation in Silica Mesoporous by *In-Situ* X-ray Powder Diffraction." The quality of the presentations was outstanding, and each of the students came away with prize money for their presentations, Raju for "Best presentation by a postgraduate researcher", and Renata for "Best presentation by an undergraduate researcher". The organisers are looking for more student presenters in 2013 for what is always an enjoyable event which helps establish new links within the AXAA community, and maintain and strengthen existing ones.

Nathan Webster and Natasha Wright

Brian Evans, XRF Guru, Retires from Ok Tedi Mining Ltd in Papua New Guinea

Brian retired in September 2012 after a distinguished career as a chemist in the mining sector In Australia and PNG. He is widely recognised as one of the leading XRF practitioners in the Asia Pacific region.

He was born and bred in the Western Australian mining city Kalgoorlie and appropriately went on to spend his entire career in the mining industry. HIs appreciation of the importance of the fundamentals underpinning analytical chemistry goes back to his studies in the Chemistry Department at the University of Western Australia in the period 1966-1970. He acknowledges the influence of the exceptionally strong department developed under the leadership of Professor Noel Bayliss and recalls especially the staff members Brian Johnston, Jim Parker, Dieter Wiege and Brian Figgis.

Brian Evans also speaks warmly of the scientists who have "given his work focus and informed the way in which he has practiced chemistry" - notably Charles Rann, Ted Pilkington, Keith Norrish and Brian O'Connor. Brian O'Connor remembers with much gratitude the influence that Brian Evans has had on the current Internet XRF Course, having been one of the first enrollees in the XRF Correspondence Course developed at the WA Institute of Technology during the early 1980s. The recent refinements of the I-XRF course has been influenced substantially by Brian in bring a practical focus to the course.

Brian Evans retires from OTML in Papua New Guinea. Left to right – Hillary Turnamur, Gabriel Saulep, Gloria Samiak, Brian Evans and Doris Turbarat.

In the final seven years of his career, Brian served as a development chemist with OTML in Papua New Guinea. This involved working at the Ok Tedi copper and gold mine in the remote Western Province and near the border with West Papua. He sees this experience as the highlight of his career, through being able to make a difference, especially in staff development. In particular, he has trained a group of PNG female chemists as competent x-ray spectroscopists. This group has been equipped with the necessary technical knowledge that underpins x-ray spectroscopy as well as the skills needed to maintain the on-site XRFs. Importantly they are now a resource for the nation and not just Ok Tedi. Their particular skills will become much more important as new mines are developed in PNG.

Brian will enjoy his retirement living in both Thailand (Chumphon) and Australia (Cairns). We wish him well.

Brian O'Connor
AXAA Life Member
7 November 2012

Vale Ross Freeman

Ross Freeman has been involved in the XRF and XRD field since the 1970's and passed away in July this year.

Ross began his geological career in 1956 at the DSIR in New Zealand (their equivalent of CSIRO). He told many wonderful tales of fieldwork in wild places, and at the start of the Rugby season had him fitter than the other players. An opportunity arose to move to Australia in 1968 and he and Margaret took it up. His early working life in Australia saw him working at Robertson Research in Sydney and then Planet in southern NSW.

Margaret and Ross Freeman.

In 1972, Bruce Chappell convinced Ross to join him at the ANU in the XRF laboratory. Ross worked with both Bruce and Keith Norrish to establish the early XRF schools run at the ANU which led to the formation of the first branch of the AXAA. The AXAA schools and conference were always held at the ANU in the early days.

Ross joined Ian Browne and Rod Clapp at Sietronics in the early 80's initially working in the laboratory at Sietronics running XRD samples and assisting with both XRD and XRF sales. Ross was also very proficient with sample preparation and developed a close relationship with Ian Devereux at Rocklabs and was responsible for the introduction of many of their sample preparation products to the Australian market.

He had a hand in the development of the Sietronics built instrumentation and worked tirelessly to keep the company going through the good times and the bad. For many years, Sietronics Christmas parties and other significant events were held at Ross's home which has always had a welcoming atmosphere.

Ross was key to developing Sietronics representation of Siemens (later Bruker AXS) XRF and XRD instruments in Australia and traveled extensively throughout Australia and New Zealand in support of the agency. The older hands at Bruker in Germany were very saddened to hear of Ross's passing as they all remember his drive and enthusiasm for their products.

One of the very few times I saw Ross lose his cool was when we were moving an XRD unit into the office in Belconnen. Ross had carefully measured the door width and checked against the specs of the instrument however when the XRD got to the doorway, it was a few centimeters too wide. It was only then that Ross saw that his cheap tape measure didn't start a 0 cm!

Ross was a very ardent supporter of the Sietronics/CSIRO development of the Siroquant package and keenly watched developments and improvements in the software and worked with the late John Taylor to bring the package to market.

We have lost a true gentleman and mentor for many of the younger generation.

Chris Kelaart (Bruker)
Liz Webber (Geoscience Australia)

Upcoming Events and Conferences

1. National XRD Course

***X-ray Powder Diffraction Analytical Methods*, Curtin University, Perth**
Sat, 31 Aug – Tue, 3 Sep, 2013

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: 31 August – 3 September, 2013

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

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

Cost: $2,695 including GST

Availability of places strictly limited.

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 6, 2013

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 –

- 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: Series 6 of the course will commence 1 January 2013.

Only 6 places remain.

Course director: Dr Brian O'Connor
Course fee: $2,695 including GST

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

Membership Matters

AXAA-Inc membership is for the 3-year period starting from the 2011 AXAA National Conference (existing membership will be re-approved in 2014). Membership is free. Candidates should provide a brief CV and a short statement of intention about what they intend to do in the organisation. Please send these to National Council Secretary Natasha Wright. The council votes on membership applications at least once every 6 months.

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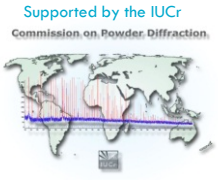
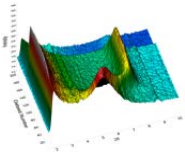
http://www.axaa.org/

APD-IV
Accuracy in Powder Diffraction

NIST National Institute of Standards and Technology U.S. Department of Commerce

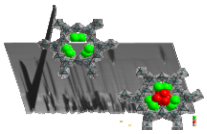
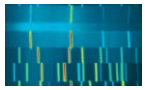
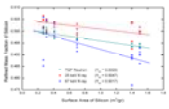
- **We are pleased to announce the location and dates for our next *Accuracy in Powder Diffraction* meeting.**
- **The meeting will comprise :**
  - A review of the state-of-the-art in:
    - Powder diffraction metrology and methodology
    - Instrumentation, and
    - Data analysis methods.
  - A program of invited presentations
  - A contributed poster session
  - An exhibition area for leading instrument manufacturers and providers






- **Where**
  - NIST - Gaithersburg, MD, USA
- **When**
  - 22nd – 25th April, 2013
- **Target audience**
  - Industry, Government, Academia

- **Planned sessions for APD-IV**
  - Standards
  - Instrumentation
  - Data Collection and Analysis
  - Structure Determination and Refinement from Powder Data
  - In situ Studies
  - Quantitative Phase Analysis
  - Proteins & Pharmaceuticals
  - Mineralogical Applications
  - Pair Distribution Studies and Total Pattern Analysis
  - Stress / Strain
  - Where to from here?

- **Conference Co-chairs**
  - James Cline, NIST, USA
  - Jeffrey Post, Smithsonian Institution, USA
- **Technical Organizing Chair**
  - Ian Madsen, CSIRO, Australia
- **How do I register ?**
  - Through the conference website www.nist.gov/mml/apdiv_conference_2013.cfm

Non U.S. citizens please note: All foreign national visitors MUST register at least 2 weeks prior to the meeting. Failure to do so will result in significant delays (up to 24 hours) in entering the facility.

Advertising and Company News

Diffraction Technology

Do you want a simple low-cost, no-frills X-ray Powder Diffractometer for routine materials characterisation?

This is what the MMA and now the eMMA is designed for.

It is lightweight, bench-top mounted, and can be moved or transported without losing alignment. The unique Harmonic Gearbox goniometer and the tube-shield are attached to the cabinets, so the whole instrument moves as one.

It has a radius of 250mm, so there is adequate resolution for separating closely spaced mineral peaks.

It can be fitted with a 10-sample loader, which is unobtrusive so can be left in place permanently if desired.

AND – it can be driven from your Laptop via the Ethernet port either directly or via a network by software which is integrated with ICDD ® databases for rapid qualitative identification.

For more information: www.diffraction.com.au
Or contact Rod Clapp at diffraction@bigpond.com

PANalytical celebrates its 10th birthday

PANalytical, the leading global supplier of analytical X-ray instrumentation and software, is today celebrating its 10th year in the industry. PANalytical arose from the acquisition of Philips Analytical by Spectris a decade ago, allowing the company to combine 'The curiosity of youth with a century of experience'. Since its inception PANalytical has continued to be a trusted and reliable partner focussing on customer satisfaction.

PANalytical's product range includes X-ray diffraction (XRD) systems and X-ray fluorescence (XRF) spectrometers as well as X-ray tubes, produced in its own state-of-the art facility in Eindhoven. In conjunction with increased investment in R&D and applications laboratories, PANalytical pushes the boundaries and with the aim of achieving customer satisfaction time after time.

"We are excited to reach this milestone in the evolution of PANalytical worldwide," said Peter van Velzen, CEO of PANalytical. "We have always strived to use technology to innovate and add value to our work. Through continuous collaboration with customers and research groups we ensure that our products are at the forefront of innovation. Together with everyone at PANalytical I am looking forward to the next decade of serving our global clients".

For further information, visit: www.panalytical.com or contact your local PANalytical representative.

PIXcel^{3D} 2x2 – Super sharp vision and a large field of view

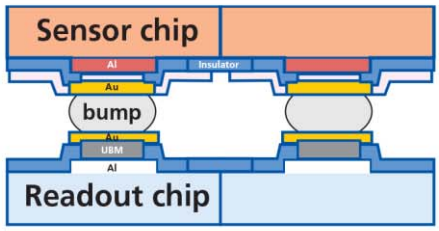
Scanning 2D micrograph from one of the brown layers on the stalagmite

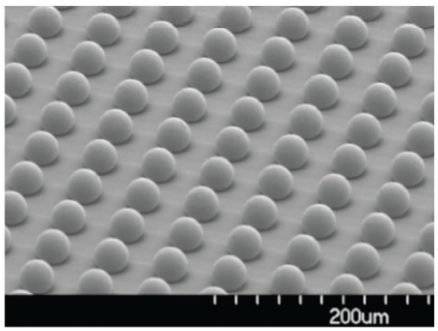
PANalytical introduces the PIXcel^{3D} 2x2 detector with solid-state hybrid technology, the big brother of PANalytical's ground breaking PIXcel^{3D}. Not only does it have an unmatched resolving power and the same high dynamic range, but the PIXcel^{3D} 2x2 also has an extraordinarily large field of view up to 28.8° 2θ in static mode. The field of view can be expanded further by the PIXcel^{3D} 2x2's unique 2D scanning capabilities.

Perfectly suited to 2D area scanning and 3D tomography detection, the PIXcel^{3D} 2x2 also offers point (0D) and fast line (1D) scanning. Like the PIXcel^{3D}, in elegant light-weight casing the PIXcel^{3D} 2x2 is a highly versatile, state-of-the-art piece of technology where the novel 'PreFIX-on-PreFIX' design enables rapid exchange of anti-scatter slits.

As a partner of the Medipix2 consortium consisting of 16 leading research institutes including the European Institute for Fundamental Particle Physics CERN in Geneva, Switzerland, PANalytical has exclusive access to their revolutionary technology for analytical X-ray applications. The new detector consists of two main parts, four readout chips with in total 512 x 512 (262144) independent detectors with 55 µm spacing; and a sensor chip. The chips are designed so they can be placed close to one another with a spacing of only 220 µm and covered by one large common sensor chip. The charge created in the sensor chip is transported to the nearest readout circuitry, causing only a minor loss of resolution at the readout chips borders.

Schematic cross-section of the sandwich construction of sensor chip and readout chip (upper image); electron micrograph of the readout chip with solder bumps before the sensor chip is placed on top (lower image).

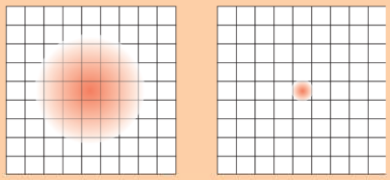




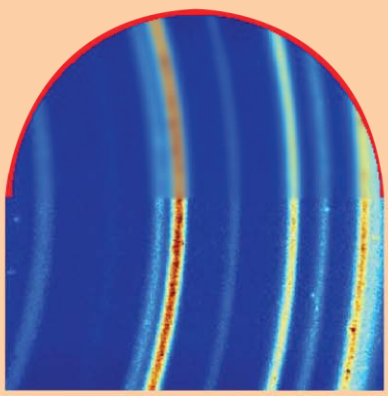
The PIXcel^{3D} 2x2's unrivalled resolving power is characterized by its pixel size, point spread function and sensor thickness. The pixels measure only 55 µm x 55 µm and the point spread function is less than a pixel unlike with other detector technologies. On the PIXcel^{3D} 2x2 one photon triggers only one pixel resulting in unparalleled image sharpness. Additionally the sensor thickness is only 300 µm, allowing to bring the detector very close to the sample without parallax effects.

What is point spread function (PSF)?

PSF is a function which, in the case of an X-ray detector, describes the influence of one incoming photon on neighboring pixels. PSF can have profound effect on the achievable resolution of a detector, as it imparts a blurring effect when this value is high, such as is the case for detectors containing gas for amplification. For example if a detector's PSF is 250 microns and the detector pixel size is 70 microns, one incoming photon will influence more than 16 neighboring pixels.



PSF of 250 micron *PSF of 55 micron*



Top: 2D XRD image with simulated 250 micron point spread function (PSF)

Bottom: PIXcel^{3D} 2x2 with no blurring PSF effect

Combining measurement methods: The benefit of fast analytical instruments.

Jana Berlin#, Andi Käppel, Birgit K. Hansen, Daniel Goran,
René Chemnitzer, Roald Tagle, Stephan Boehm and Ulrich Waldschläger

Bruker Nano GmbH, Schwarzschildstrasse 12, 12489 Berlin, Germany

Innovation with Integrity

Every analytical technique has advantages and disadvantages, especially when it comes to spatial resolution, detection limits, number of elements that can be analyzed (at the same time), acquisition times and sample preparation. Many analysts know that they need to combine the results obtained with different methods in order to completely understand the chemistry and internal structure of a sample - let it be a ceramic, metallic alloy, semiconductor or a rock sample.

Fig. 1. A sample of the Gujba meteorite.

Figure 1 and 4 show photographs of samples from the Gujba meteorite. This meteorite contains different types of objects with a wide range of sizes and structures (larger rounded metal particles, silicate spherules and fine-grained materials). It lends itself ideally to demonstrate how combining different methods can reduce precious analysis time while getting the fundamental information needed to learn about the history recorded in this rock.

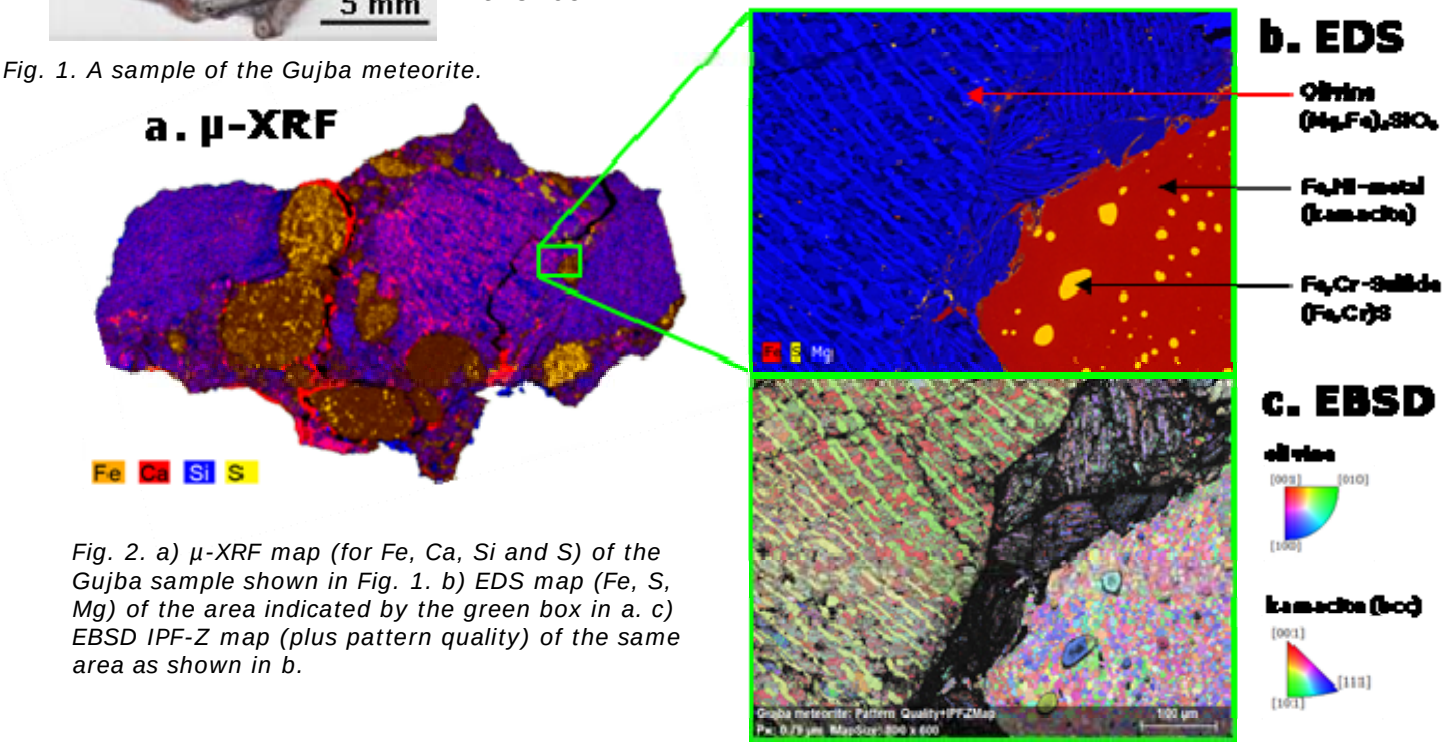


Fig. 2. a) µ-XRF map (for Fe, Ca, Si and S) of the Gujba sample shown in Fig. 1. b) EDS map (Fe, S, Mg) of the area indicated by the green box in a. c) EBSD IPF-Z map (plus pattern quality) of the same area as shown in b.

Figure 2a shows a mixed element map (Fe, Ca, Si and S) obtained with an M4 Tornado µ-XRF spectrometer (at 50 kV, 200 µA, ~150 kcps input count rate). The total acquisition time was 77 minutes (image resolution: 1067x680 pixel, 5 ms dwell time per pixel). This map quickly provides an overview and is helpful in selecting areas of interest on a smaller lateral scale with additional techniques.

The integration of EBSD into the EDS software allows the simultaneous acquisition of EDS and EBSD datasets. Fig. 2b and 2c show the results of a combined EDS/EBSD measurement performed at 20 kV with an XFlash® 5030 EDS detector and an e⁻Flash^{1000} EBSD detector at a speed of ~70 points per second (image resolution: 800x600 pixel). Note that the metal particle (lower right) consists of many small grains (average diameter: ~10 µm) and that the olivine bars (left) show a change of orientation.

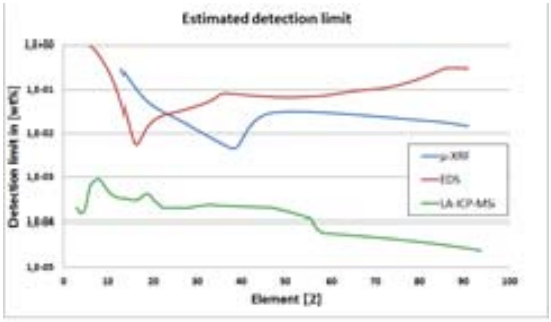


Fig. 3. Diagram with generalized curves for typical detection limits of µ-XRF, EDS & LA-ICP-MS.

All data (µ-XRF, EDS and EBSD) can be saved as "hyperspectral databases" (spectra/crystallographic data behind each pixel of the map), allowing offline-data mining and, at the same time, reducing valuable instrument time.

A notable technique to complement the acquired dataset with high precision quantitative analyses (and even isotopic information) is LA-ICP-MS. Figure 3 shows typical detection limits for LA-ICP-MS in comparison with µ-XRF and EDS. LA-ICP-MS provides a high sensitivity even for light elements like Lithium.

***Taking it a step further:* 3D chemical analysis for sample sizes in the µm to cm range!**

Routinely, X-ray computed tomography (CT) has been used to display structural information in 3D. To obtain 3D chemical and structural information, a sample has to be sectioned in order to avoid absorption effects. High speed µ-XRF and/or EDS element mapping combined with serial sectioning allows producing datasets that only need to be exported into a software for 3D reconstruction, such as Amira®.

Figure 4 shows the Gujba meteorite sample that was "polished away" for 3D chemical analysis (with µ-XRF and EDS) during the course of this study. In Table 1, analytical details are listed for the results shown in Figure 5.

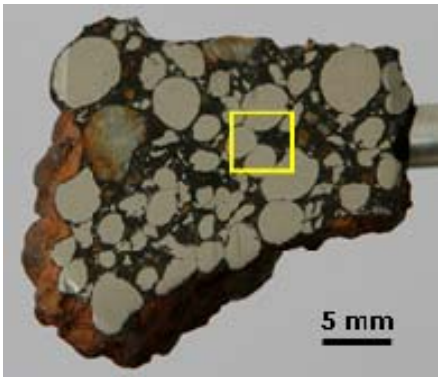


Fig. 4. Gujba sample that was used for 3D chemical analysis. Yellow box indicates the location of 3D-µXRF Volume 2.

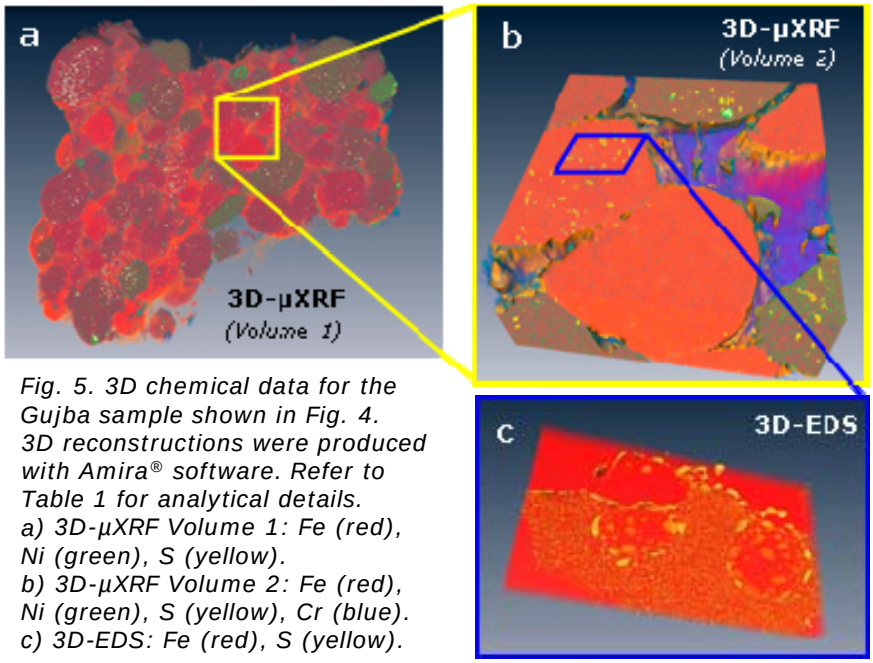


Fig. 5. 3D chemical data for the Gujba sample shown in Fig. 4. 3D reconstructions were produced with Amira® software. Refer to Table 1 for analytical details.
a) 3D-µXRF Volume 1: Fe (red), Ni (green), S (yellow).
b) 3D-µXRF Volume 2: Fe (red), Ni (green), S (yellow), Cr (blue).
c) 3D-EDS: Fe (red), S (yellow).

Table 1. Analytical details for the 3D datasets.

	µ-XRF (Volume 1)	µ-XRF (Volume 2)	EDS
Volume	~2200 mm³	41.5 mm³	0.6 mm³
Layers	36	38	21
Sectioning depth	148 µm	48 µm	4 µm
Total depth	5.18 mm	1.78 mm	80 µm
Voxel size	32 x 32 x 148 µm	6 x 6 x 48 µm	1.6 x 1.6 x 4 µm
Instrument	M4 Tornado	M4 Tornado	XFlash® 6|60
Accelerating voltage	30 kV	30 kV	15 kV
Beam current	500 µA	600 µA	5 nA
Input count rate	~150 kcps on Fe	~150 kcps on Fe	~130 kcps on Fe
Acquisition time per layer	30 min	60 min	90 min
Grinding/polishing per layer	~20 min	~15 min	~15 min
Total time spent	**~ 60 h**	**~ 50 h**	**~ 40 h**

The 3D-µXRF data (Fig. 5a,b) show that the metal particles in Gujba contain different amounts of Ni (green) and a variable abundance of sulfide grains (yellow). The better spatial resolution of the 3D-EDS data (Fig. 5c, here only a part of the complete EDS dataset is shown) allows to visualize bowl-shaped structures for some of the sulfides found within the metal. These structures were resolved here for the very first time in 3D.

Recent progress in the automation of serial sectioning [e.g., 1,2] combined with advances in silicon drift detector (SDD) technology [e.g., 3,4] for EDS and µ-XRF appears to create a lot of potential for this type of 3D chemical analysis in the near future. One has to keep in mind that although the sample is polished away, all the data can be kept in hyperspectral databases for later re-examination ("sample digitalization"). The total times (refer to Table 1) spent on obtaining the 3D chemical data (including sample preparation, acquisition times and time spent on the 3D reconstruction) are comparable to the typical time spent for serial sectioning with the Focused Ion Beam (FIB) technique – although the sample sizes covered are quite different (<100 µm for FIB versus sample sizes up to several mm and even cm here). For 3D animations of the data shown in Figure 5 and more information about (3D)-µXRF, (3D)-EDS, EBSD, LA-ICP-MS and micro-CT/SEM visit http://www.microscopy-analysis.com/brukerwebinars to watch the recording of an online-webinar (1 hour) about the topic of this article.

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