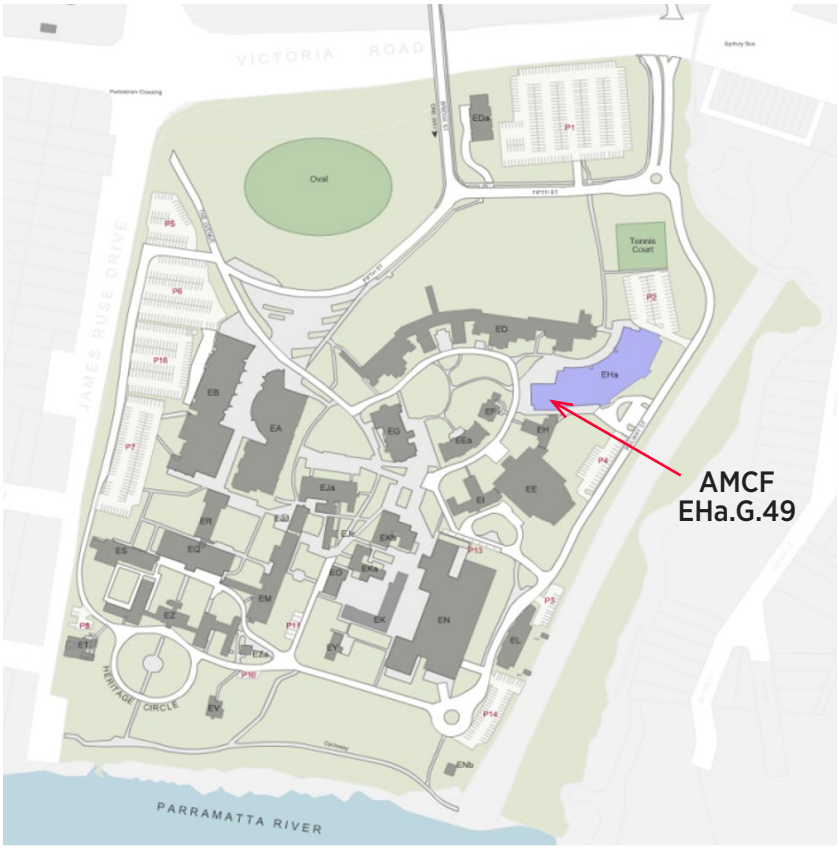
WESTERN SYDNEY
UNIVERSITY

AMCF

Advanced Materials Characterisation Facility

Newsletter: July 2023



Address

Western Sydney University,
Advanced Materials Characterisation Facility (AMCF),
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Building EHa G.49,
Victoria Road, Rydalmere NSW 2116

Front Cover Image:

A colourised SEM image of a wood cross-section, showing starch granules (pink), and inner vessel walls where water is transported (blue) - *Imaging: Dr Sebastian Pfautsch, Colourisation: Dr Laurel George.*

Back Cover Image:

Micro-CT (top) and XRM (bottom) images of a Eucalyptus robusta leaf, showing the location of different leaf structures. The XRM shows calcium in green, silicon in red and oxygen as blue – *Research project: Dr Ben Moore and PhD student Karen L. M. Catunda, Imaging: Dr Laurel George*

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11th Edition Newsletter, July 2023

Editors: Laurel George, Richard Wuhrer, and Daniel Fanna.

The AMCF is a part of:
Research Infrastructure
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FROM THE FACILITY RESEARCH MANAGER

This year began with many users trying to catch up on their incomplete analysis, mostly due to previous research delays caused by COVID. The rapid increase in the number of HDR's requiring access and training in our facility is great to see.

As you read this newsletter, you will see why the last six months have passed so quickly. It has been busy! Many users have operated a variety of instrumentation. A large number of courses, workshops and masterclasses have been offered to students. The presence of international visitors and tour groups, instrument upgrades and new software upgrades have occupied staff time and expertise. Indeed, the AMCF in conjunction with Netzsch just completed a workshop on "*Thermal Analysis Using Differential Scanning Calorimetry (DSC)*". We thank Netzsch for leaving a DSC in our facility for a couple months to provide the opportunity for our researchers to develop skills and understanding in this area.

In the early part of the year, we also had a couple of SEM's placed in the AMCF for demonstrations, and these are proving to be very popular with our users.

Congratulations are extended to Dr Laurel George and Dr Daniel Fanna for their successful election to become the NSW representative for the Australian Microbeam Analysis Society (AMAS) and the NSW representative for the Australian X-ray Association (AXAA) respectively. This a great honour and shows the high regard they are building for their respective research experience.

The Pro Vice-Chancellor International, Professor Linda Taylor, came to visit the AMCF recently. This provided a wonderful opportunity for her to talk with many of the HDR researchers about their research and to meet AMCF staff.

The latter half of this year will continue to be busy as we plan to run various introductory workshops on the use of our instruments and run advanced masterclasses on specialised instrumentation. Currently, we are running smaller group training. As always, we are happy to show anyone our facility. Do not hesitate to contact us for a walk through our labs.

Dr Richard Wuhrer
Facility Research Manager, AMCF
July 2023

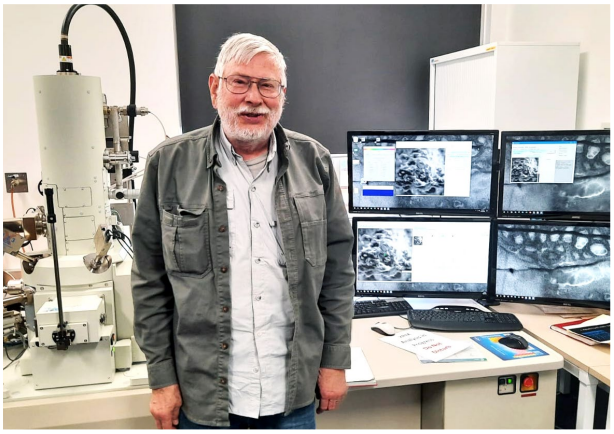
RECENT ACTIVITIES

AMCF Visitors (Attack of the Presidents)

The AMCF has welcomed many visitors this year. We have been pleased to connect with international friends and collaborators that are now allowed to come "down under" after covid travel restrictions. Guests have included many well-known microanalysts with prolific publications, who have travelled to the AMCF after attending the 27th Australian Conference on Microscopy and Microanalysis (ACMM27) in Perth, Western Australia. International guests have included:

- Prof. Nestor Zaluzec (*Argonne National Labs, Illinois, USA.*), past president of the Microscopy Society of America (MSA).
- Prof. Raynald Gauvin (*McGill University, Montreal Quebec, Canada*), former president of the International Union of the Microbeam Analysis Societies (IUMAS), Microanalysis Society (MAS) and the Microscopical Society of Canada (SMC).
- Dr Mike Matthews (*Atomic Weapons Establishment, UK*), president of the International Union of the Microbeam Analysis Societies (IUMAS) and past president of the European Microbeam Analysis Society (EMAS).
- Patrick Camus (*EDAX, USA*). President of the Microanalysis Society (MAS).
- Dr John Fournelle (*Senior Scientist, University of Wisconsin*).

These visits are of great importance to the facility, as it gives our community an opportunity to ask technical questions and stay updated with what is happening internationally. Our students and researchers also get the chance to hear talks from the best in the world.

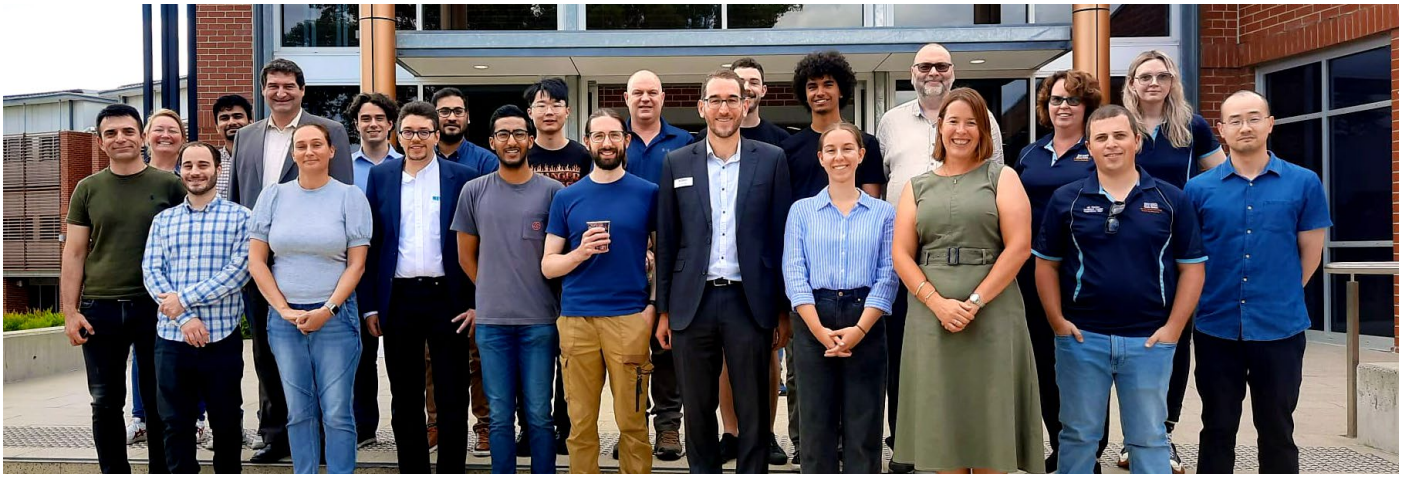


Top: Dr Mike Matthews, Prof. Raynald Gauvin, Dr Ric Wuhrer (former AMAS president) and Ken Moran (Moran Scientific). Bottom Left: Dr John Fournelle. Bottom Right: Pat Camus, Mark Blackford (former AMAS treasurer) and Prof. Nestor Zaluzec.

Netzsch DSC Workshop

Earlier this year, the AMCF hosted a workshop on thermal analysis using differential scanning calorimetry (DSC) run by Netzsch. Presenters included Andrew Gillen, a Materials Engineer from Netzsch Australia, and Chemist Dr Markus Meyer from Netzsch Germany. Attendees included WSU students and staff, as well as many visitors from industry. Participants learned that DSC is one of the most commonly used types of thermal analysis and allows users to monitor their samples' thermal events, such as melting points, glass transitions, thermal decomposition and phase transitions. Andrew and Markus provided us with a bounty of useful information, including many tips and tricks on sample preparation, correct methodology, experimental setups and results analysis. Dr Laurel George from the AMCF was also invited to give a brief talk on the importance of gas atmosphere choice from thermal experiments, with some examples of samples run in different atmospheres here at the AMCF.

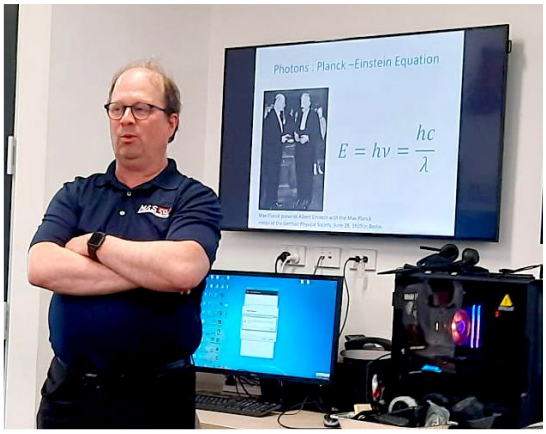
Netzsch was kind enough to bring along a brand new Netzsch 300 Caliris DSC to demonstrate some sample runs. The AMCF was lucky enough to be allowed to keep this instrument for a few months to test it out. Special thanks to Patrick Blessing and Dr Jiunn Jieh from Netzsch for setting this up for us and letting us test it out.



Top: Presenters and participants at the Netzsch DSC Workshop. Bottom Left: Dr Markus Meyer demonstrating the new Netzsch 300 Caliris DSC. Bottom right: Dr Markus Meyer presenting to workshop participants.

Prof. Raynald Gauvin

While visiting, we were fortunate enough to be treated to a talk from Prof Raynald Gauvin from McGill University in Canada, titled "Chemical analysis of lithium of battery materials with high spatial resolution using EDS and EELS in the electron microscope". The talk explored the difficulties of analysing and imaging lithium samples, as well as STEM results for beam energies of 0.1 - 30keV. These STEM results are of particular interest, as the AMCF has instruments capable of this type of analysis.



Prof. Gauvin in action

Tour groups from India

In June, the AMCF was visited by two tour groups from India, both organised by Associate Dean of Global Engagement, A/Prof Kamali Kannangara. The first group consisted of 24 Indian exchange students from Sher-e-Kashmir University of Agricultural Science and Technology (SKUAST-Kashmir), accompanied by Dr Y.S. Parmar from Parmar University of Horticulture and Forestry. Students were offered hands-on experience on our advanced instrumentation and looked at samples of interest in the agricultural sciences. We had some great responses from students and organisers:

> *"These students have not seen such instruments before and were amazed of their capability! It was an eye-opener for them to consider pursuing HDR studies. Thanks again for your continued support."*

The AMCF was also visited by a tour group of Senior Education Counsellors from India. Included in the group were school Chief Academic Officers, Managing Directors and CEOs. Together they had a combined total of over 185 years experience in the education sector. As part of their 2-day tour of Parramatta and Hawkesbury campuses, they stopped by for a short talk and tour of the AMCF. Just enough time to have a quick look at some spider fangs and fly eyes on our electron microscopes and chat with staff about the benefits of studying at WSU.

The Senior Education Counsellor tour group from India with A/Prof Kamali Kananngara (far left) at the AMCF.

The exchange student tour group from India at the AMCF, having fun on our instrumentation.

Technique Developments

The AMCF has participated in a number of conferences and seminars this year, to ensure we are up to date with the latest techniques and technology for the advanced instrumentation we house. We have showcased the innovative upgrades and technique developments we have been implementing in the facility, to help our researchers turn data into knowledge. The following are some of the developments we have been involved with:

Correlative micro-CT, SEM and XRM:

In February, Laurel presented a poster at the 27th Australian Conference on Microscopy and Microanalysis (ACMM27) in Perth, Western Australia. Titled "*Correlative Micro-CT, SEM and XRM for Locating Calcium Oxalate in Eucalyptus Leaves*", Laurel collaborated with Dr Ben Moore and Karen Catunda from the Hawkesbury Institute for the Environment (HIE) and Ken Moran from Moran Scientific. The presentation explained how the same area of a sample can be examined using three different complimentary techniques, to better analyse and determine a sample's structure.

In-situ XRD analysis of cements:

Daniel has been busy setting up our XRDs to run timed *in-situ* experiments. This is of particular interest for our researchers from Engineering (Prof. Zhong Tao), as these experiments allow us to watch concrete cure and follow the phase changes occurring over time. Not only has he developed this technique, but with the help of some of our AMCF friends, special sample holders specifically designed for these experiments using CAD have been 3D printed on our new 3D resin printer here in the AMCF.

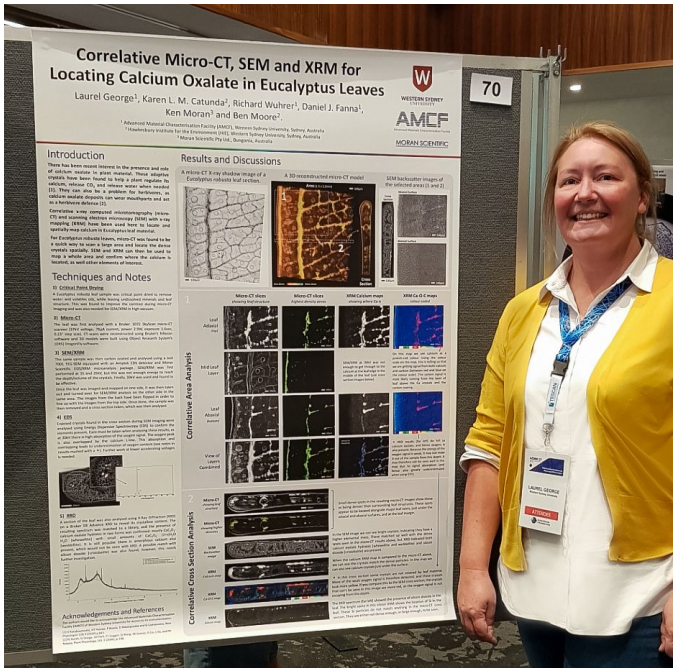
Upcoming Microscopy and Microanalysis (M&M) Conference:

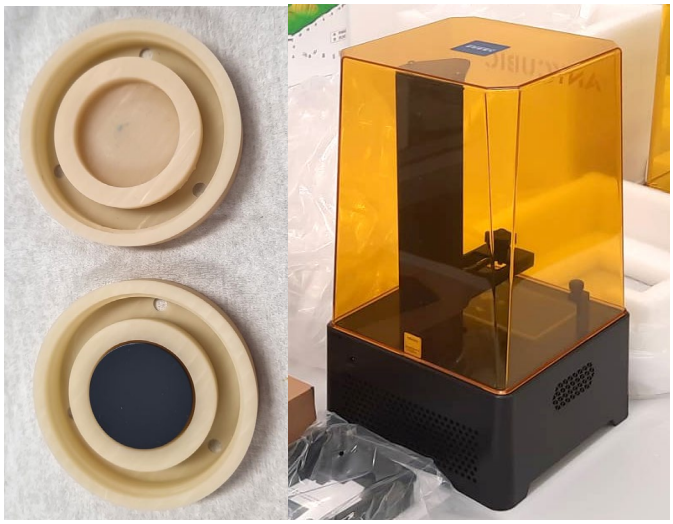
Ric has recently returned from M&M, held in Minneapolis, Minnesota, USA. He gave three presentations on behalf of the AMCF and our researchers. These include:

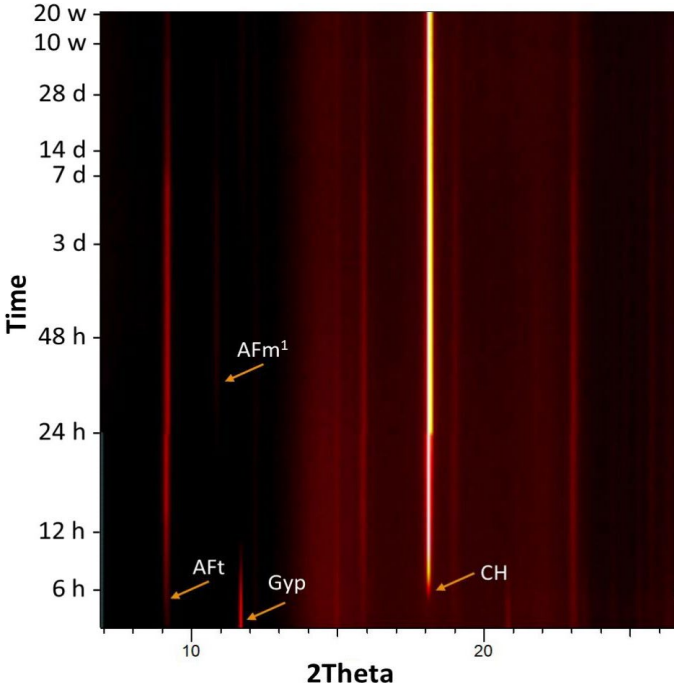
Oral presentation: "*WDS-SD: Next Generation of Wavelength Dispersive Spectrometers (WDS) with a Silicon Drift Detector (SDD) – What Can it Do, Where are We Now and Where is it Going?*", Richard Wuhrer, Ken Moran and Michael Matthews.

Poster: "*Long-term In-situ X-ray Diffraction Studies on Ordinary Portland Cement Hydration with Correlative X-Ray Mapping*", Daniel J. Fanna, Qingtao Huang, Laurel George, Zhong Tao, Ken Moran and Richard Wuhrer.

Invited Talk: "*The Use of Correlative Micro-CT and XRM to Locate and Identify Dense Structures in Plant Material*", Laurel George, Karen L. M. Catunda, Richard Wuhrer, Daniel J. Fanna, Ken Moran and Ben D. Moore.





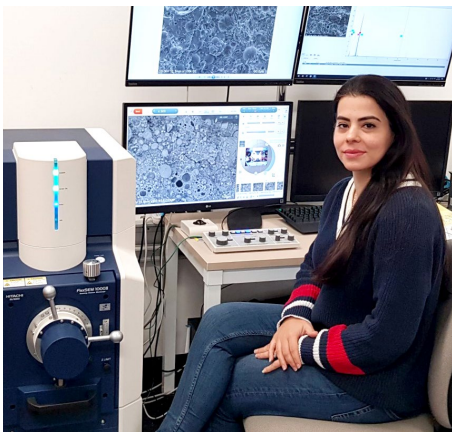


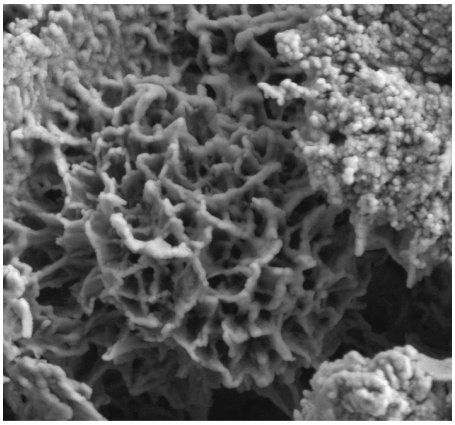
Top to Bottom: Laurel presenting at ACMM27 in Perth, Daniel's 3D printed XRD holders and the new resin 3D printer in the AMCF, and a 2D *in-situ* XRD spectrum of cement hydration over time showing the rise and fall of different peaks (represented by the bright lines) and hence phases present as cement hydration occurs.

RESEARCHERS AT THE AMCF

Aida Rahmani

School of Engineering, Design and Built Environment





RESEARCH

Aida Rahmani is a PhD student with the Centre for Infrastructure Engineering. Her research studies are focused on the development of durable and water-resistant concretes made using glass-based waste materials. Use of waste glass in concrete has attracted a lot of attention recently, as it has promising strength and mechanical properties. However, it does not have sufficient durability in harsh environments. Aida is working to improve the durability of glass-based concrete.

IMPACT

According to statistics, Australia produced 1,160,000 tonne of glass waste in 2018–19. Of this, only 59% was effectively recycled and the remainder was dumped in landfills. By using this glass-waste product, Aida hopes to reuse this waste while also turning it into a valuable concrete product.

SUPERVISORS

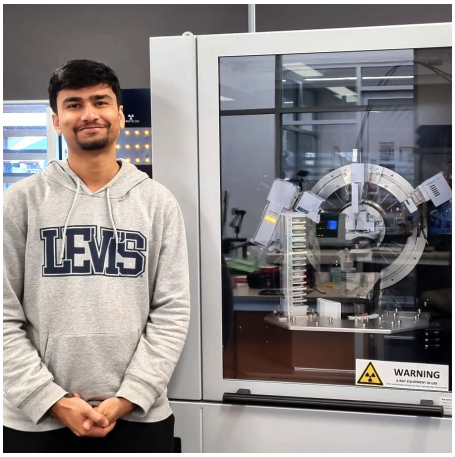
Professor Zhong Tao, Dr Utsab Katwal

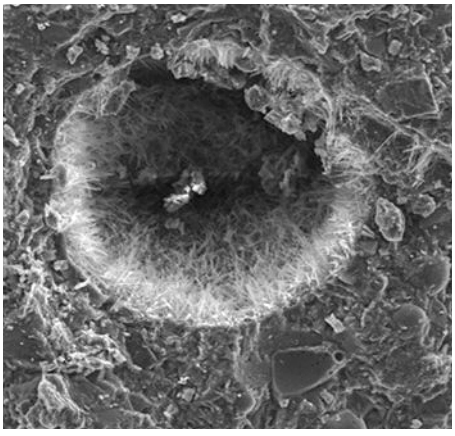
INSTRUMENTS

Aida has been carrying out **SEM/EDS** and **XRD** tests to evaluate the microstructural properties of glass-based concrete when exposed to different corrosive environment.

Dr Sanket Rawat

School of Engineering, Design and Built Environment





RESEARCH

Dr Sanket Rawat is a postdoctoral fellow working with Prof Sarah Zhang on an ARC Discovery Project investigating the fire performance of magnesia-based fibre-reinforced cementitious composites. Sanket has been researching the impact of elevated temperatures on both calcium and magnesia-based binder materials. In particular, his main focus has been on the effective utilisation of industrial by-products in infrastructural sectors requiring high fire resistance. As part of this research he is currently investigating how post-fire curing methods could play a substantial role in the restoration process.

IMPACT

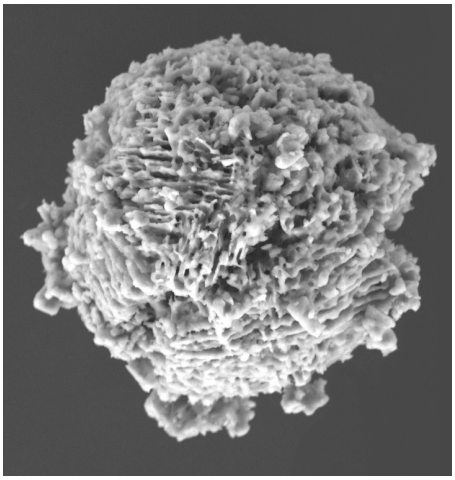
Considering the major role of the construction sector in CO_2 emissions, it is important to find viable alternatives to reduce the usage of traditional cement. A better understanding of these alternative sources (such as magnesia based by-products) and the subsequent use of captured CO_2 in these alternative materials, could help us progress towards a sustainable future.

INSTRUMENTS

Sanket has been using AMCF's **XRD** and **SEM/EDS** to compare the microstructural changes at elevated temperatures (picture left shows initiation of rehydration on 7-day post-fire curing) and **TGA/DSC** to check the thermal stability of the binder system.

Dr Chandra Malladi

School of Medicine



RESEARCH

Dr Chandra Malladi is a Sr. Scientific Officer associated with the School of Medicine. Chandra's research fields include cell signaling, protein biochemistry, protein-protein interactions, proteomics and lipidomics. Recently he has been researching pharmaceutical drug carriers and protein delivery to target-specific tissue using nanoparticles (NPs) in collaboration with an international group of scientists. Nanoparticles are defined as particles with a size of 1 to 100nm. Although various NPs are used for diagnostic and therapeutic purposes, studies suggest these can be toxic. Chandra is focusing on developing non-toxic, charge-free bismuth trioxide (Bi_2O_3) NPs for drug-protein delivery to target cells.

IMPACT

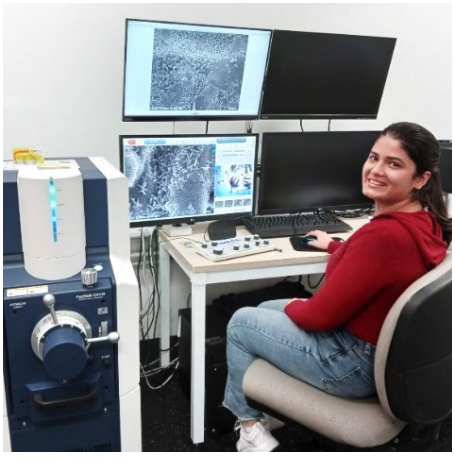
A new generation of NPs could help deliver therapeutic drugs and proteins with reduced toxicity compared to other solid NPs.

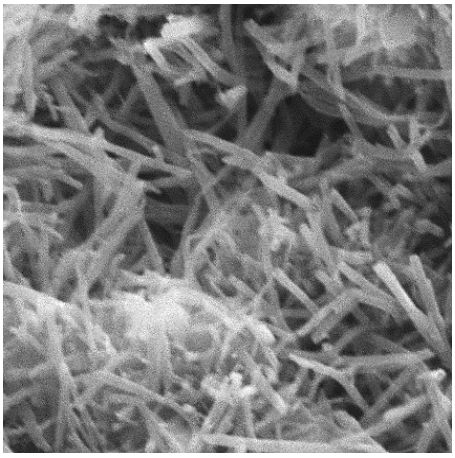
INSTRUMENTS

Chandra has been using a suite of instruments at the AMCF to fully characterise his Bi_2O_3 NPs, including:
SEM: To image samples and see surface structure
EDS: To check the elemental composition
XRD: To check the phases present
Raman/UV-Vis spectroscopy: To confirm the structure
ASAP 2020: For BET surface area analysis

Anu Middha

Hawkesbury Institute for the Environment (HIE)





RESEARCH

Anu Middha is a PhD student with the Hawkesbury Institute for the Environment (HIE), and has been characterising leaf cuticles in Australian native grasses (Kangaroo grass and *Sorghum*). The cuticle is the outermost layer of all the aerial organs of a plant. Anu is specifically interested in studying leaf cuticle structure, biochemistry, physiology, and genetics. She aims to find out how cuticles in these grasses respond to abiotic stresses, such as heat and drought.

IMPACT

Being the outermost layer of the leaf, the leaf cuticle acts as a barrier against excessive loss of water from leaf tissues, thereby preventing plants from desiccation. By understanding the physiological and molecular basis of this, we can learn how to develop more stress resilient plants by making their cuticles more resistant towards environmental stress.

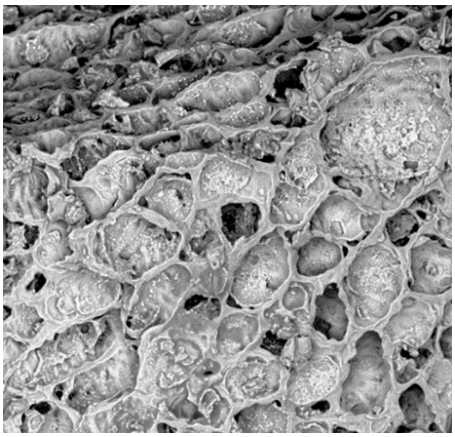
SUPERVISORS

Prof. Ian J Wright, Prof. Brendan Choat, Dr. Vinod Jacob.

INSTRUMENTS

Anu has been using the AMCF's **Critical Point Dryer** and **Gold Plasma Coater** for sample preparation and **SEM** to look for epicuticular wax deposits on the grass leaf surfaces (tubular shaped cuticle wax on a kangaroo grass leaf is shown left).

Dale Kennedy
School of Science (Food Science)



RESEARCH

Dale Kennedy is a MSc (Food Science) student identifying the changes in structure and chemical composition of spent coffee grounds during different brewing processes. These spent grounds contain many biologically beneficial components. By understanding the differences between raw and differently brewed grounds (e.g. hot vs. cold brews), Dale hopes to determine if wastes from particular methods could be suitable for recycling into other products.

IMPACT

Spent coffee grounds are a large waste product generated from the coffee industry. By understanding the composition, we can find ways to best recycle it into other food, garden and agricultural products, helping to reduce food waste.

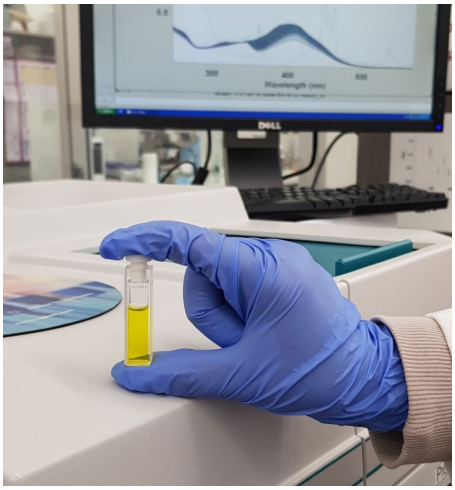
SUPERVISORS

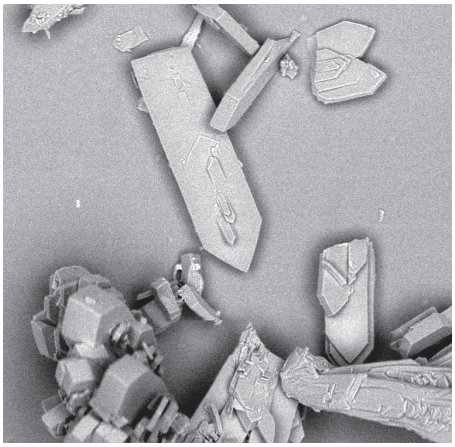
Dr Sunil Panchal and A/Prof Kamali Kannangara

INSTRUMENTS

Dale has been using: **SEM/EDS** for elemental analysis and imaging surface morphology (such as the porous structure of a coffee ground shown left) and **FTIR and TGA/DSC** fo observe composition changes.

Judith Stuparu
School of Science





RESEARCH

Masters student Judith Stuparu is researching and designing chemical indicators that could be used to detect metals such as cobalt, copper and mercury. These tiny detectors are made up of ligands, which are molecules that have a functional group that can bind to other atoms. The ligands that Judith is designing and testing will change colour when bound to a specific metal, making them useful as indicators.

IMPACT

Judith's indicators could have many substantial uses, particularly as heavy metal detectors. They could be used in biological and environmental settings, such as testing water and soil samples for contamination and toxicity. They could also be a valuable detectors in biomedical applications, such as Alzeimer's research and testing, and instrumentation development, such as use in conjunction with HPLC testing.

SUPERVISORS

Dr Feng Li, A/Prof Andrew Shaliker

INSTRUMENTS

Judith has been making us of the AMCF'S:

SEM/EDS: For imaging and elemental analysis of ligand crystals

UV-Vis: To analyse colour changes when ligands bind to metals.

FTIR: To analyse the binding sites of the ligands.

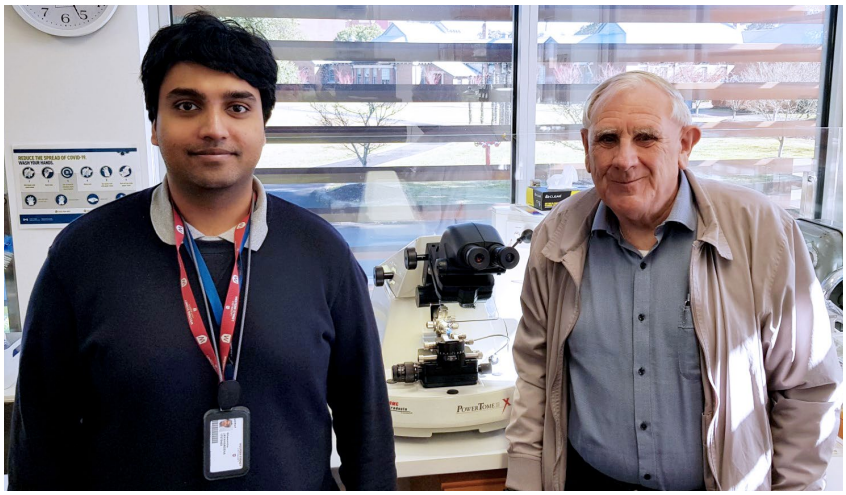
INSTRUMENT NEWS

Ultramicrotome

The AMCF has acquired a PowerTome XL Ultramicrotome and a glass knifemaker. These instruments are essential for the preparation of thin sections for STEM and TEM analysis, especially for biological samples and soft materials.

After setting up, training was provided by the incredibly experienced Trevor Hinwood, who is RMC product manager at HinSci and an expert in histotechnology. Trevor has been closely associated with Light Microscopy Australia (LMA) for many years. Thanks is extended to Sue Lindsay from Macquarie university for her help and advice on sample preparation in the lead up to ultramicrotoming. Both Trevor and Sue have provided us with many tips, tricks and supplies to get us up and running, and slicing samples.

Our test samples for training were provided by PhD student Bhawantha Jayawardena, who kindly brought along some cells set in LR White resin to test out on the ultramicrotome. After some attempts to cut some 50nm thick sections (divide a millimeter by 20,000, and that's how thin these slices are!), we have started imaging them on our Zeiss Merlin FEG-SEM in scanning transmission electron microscopy (STEM) mode with some success. We have found that preparing samples in resin and ultramicrotomy are both challenging, and can take days to get good results. There is much practice to be done in the near future, but the results are looking promising.

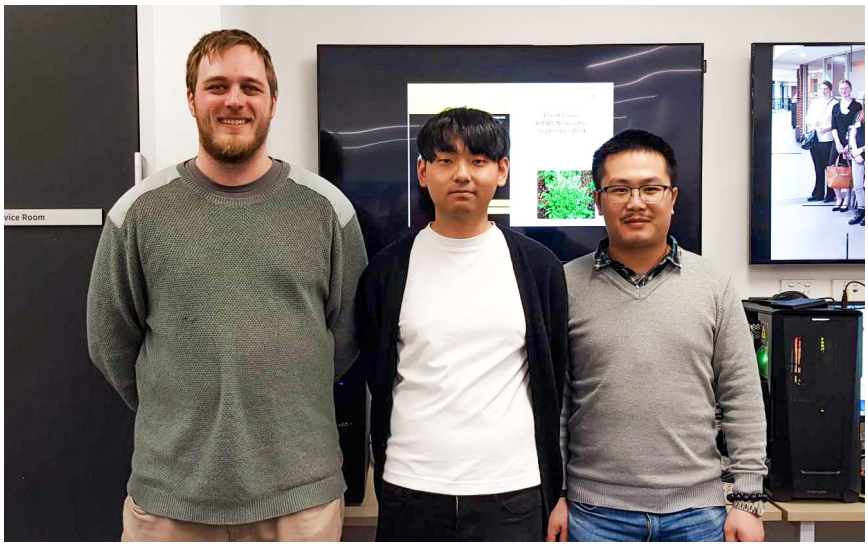


Top to bottom: Bhawantha and Trevor ready for training, the PowerTome XL Ultramicrotome and a close up of a sample being cut with a glass knife.

STAFF NEWS

Interns around

Those that have been using the AMCF over the past 6 months may have met full time intern **Matthew Wallis**. Matthew has just completed his Master Science, and will be embarking on a PhD with Science at WSU later this year. Unlucky for us he will no longer be with us full time, but will be staying on with us as a casual intern one day per week. Casual interns **Hyunsung Min** and **Edward Huang** will be back in the lab to help us out for the rest of this year.



Matt, Hyun and Edward ready for action.

Staff Represent!

Congratulations to **Daniel** and **Laurel,** on their successful nominations as state representatives for their member associations, and also ex-AMCF staff member and great mate **Tim Murphy** on his nomination as an industry representative. All will be serving on their respective executive committees:

Australian X-ray Analytical Association (AXAA) - Dr Daniel Fanna, NSW representative.

Australian Microanalysis Society (AMAS) - Dr Laurel George, NSW representative.

Australian Microanalysis Society (AMAS) - Dr Timothy Murphy, Industry representative.

We look forward to seeing WSU more involved with these analytical societies. Keep an eye out for members events. If you'd like to join either of these associations, please contact Laurel and Daniel.

Daniel, Laurel and an old photo of Tim in his graduation gown.

RECENT PUBLICATIONS

1. Wallis, M.J., Min, H., Lindoy, L.F. and Li, F., (2023), "Investigating the Conformations of a Family of [M2L3]4+ Helicates Using Single Crystal X-ray Diffraction", *Molecules*, 28, pg1404.

2. Malone, M., Radzieta, M., Schwarzer, S., Walker, A., Bradley, J., and Jensen, S.O., (2022), "In vivo observations of biofilm adhering to a dialkylcarbamoyl chloride-coated mesh dressing when applied to diabetes-related foot ulcers: A proof of concept study", *International Wound Journal*. pg1–11.

3. George, L., Catunda, K.L.M., Wuhrer, R., Fanna, D.J., Moran, K. and Moore, B., (2023), "Correlative Micro-CT, SEM and XRM for Locating Calcium Oxalate in Eucalyptus Leaves", *ACMM27 Proceedings*, (Abstract and Poster presentation).

4. Pan, Z., Tao, Z., Cao, Y.F., George, L. and Wuhrer, R., (2023), "High-temperature performance of alkali-activated binders of fly ash and calcium aluminate", *Ceramics International*.

5. Sigdel, L.D., Lu, M., Al-qarawi, A., Leo, C.J., Liyanapathirana, S. and Hu, P., (2023)," Application of engineered compressible inclusions to mitigating soil-structure interaction issues in integral bridge abutments", *Journal of Rock Mechanics and Geotechnical Engineering*, doi.org/10.1016/j.jrmge.2022.12.033.

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UPCOMING EVENTS

26TH CONGRESS AND GENERAL ASSEMBLY OF THE INTERNATIONAL UNION OF CRYSTALLOGRAPHY

22-29 August 2023
Melbourne Convention and Exhibition Centre
www.iucr2023.org

DIFFRAC.EVA Workshop

Western Sydney University, August 2023

BRUKER

Limited seats are available for our 1-day DIFFRAC.EVA workshop, jointly organized with Western Sydney University.

Workshop contents:

In this workshop, participants will be introduced to DIFFRAC.EVA and how it can be used to analyse diffraction data. This workshop is ideal for beginner to intermediate level users. Participants will be guided through examples covering various functionalities and analyses in DIFFRAC.EVA, including:

- Working with multiple diffraction data format
- Phase identification (search/match)
- Search/match with database and chemical filters
- PDF, COD, and user-defined databases
- Semi-quantitative analysis with SQUALL and RIR methods
- Peak fitting tool
- Crystallite size analysis
- Degree of crystallinity analysis
- Plotting tools (PIP, VIP, Waterfall display)
- Working with 2D data
- Report generation and templates
- Exporting a TOPAS project file

Presenter:
Dr. Manoel Manuputty, Bruker AXS

Date & Time
17 August 2023 (Thurs)
8:45 AM - 5:00 PM

Venue
Building EHa, Ground Floor, Room 17,
Parramatta South Campus, Western Sydney University

Registration Details
$150 per person ($100 for students), by 10 August.
This event is complimentary for WSU attendees.

Coffee/tea breaks provided, lunch is not included.

Please email AMCF staff for details on how to register

Seminar: Powder XRD and Non-Ambient Chambers by Anton Paar Australia

Presenters:

Dr. Shamim Shahrokhi
Anton Paar Australia & New Zealand

Dr. Andrew Jones
Anton Paar Austria

When: Wednesday 16th of August, 9-11am

Where: EHa.1.30, Parramatta South Campus, WSU

This Seminar will include talks on:

- Welcome & introduction to Anton Paar
- Intro to X-Ray Diffraction (XRD), and the latest advances in XRD instrumentation
- Non-ambient XRD: Theory, applications, tips, tricks and common challenges
- Intro to Small Angle X-ray Scattering (SAXS) instrumentation

Please email AMCF staff for details on how to register

Save the Dates!

Queensland University of Technology

AMAS AUSTRALIA

The University Of Queensland AUSTRALIA

AMAS XVI:

the 16th biennial symposium of the Australian Microbeam Analysis Society

Dates: 5th – 9th February 2024

Location: Brisbane, QLD

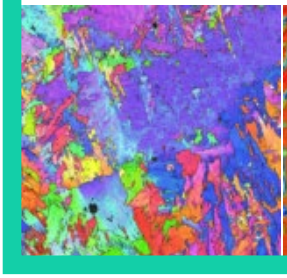
Main Venue: Queensland University of Technology

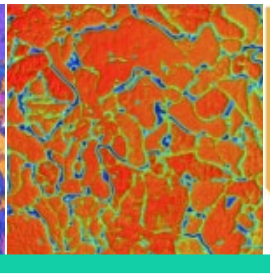
Workshops Venue: University of Queensland

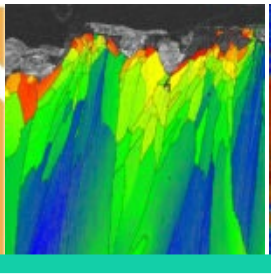
Symposium Co-Chairs: Henrietta Cathey & Rob Jones, QUT

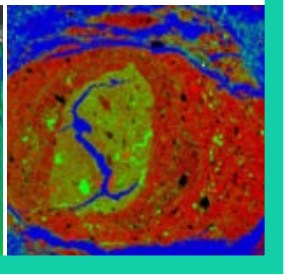
For up-to-date information, check the AMAS website: microscopy.org.au/amas/

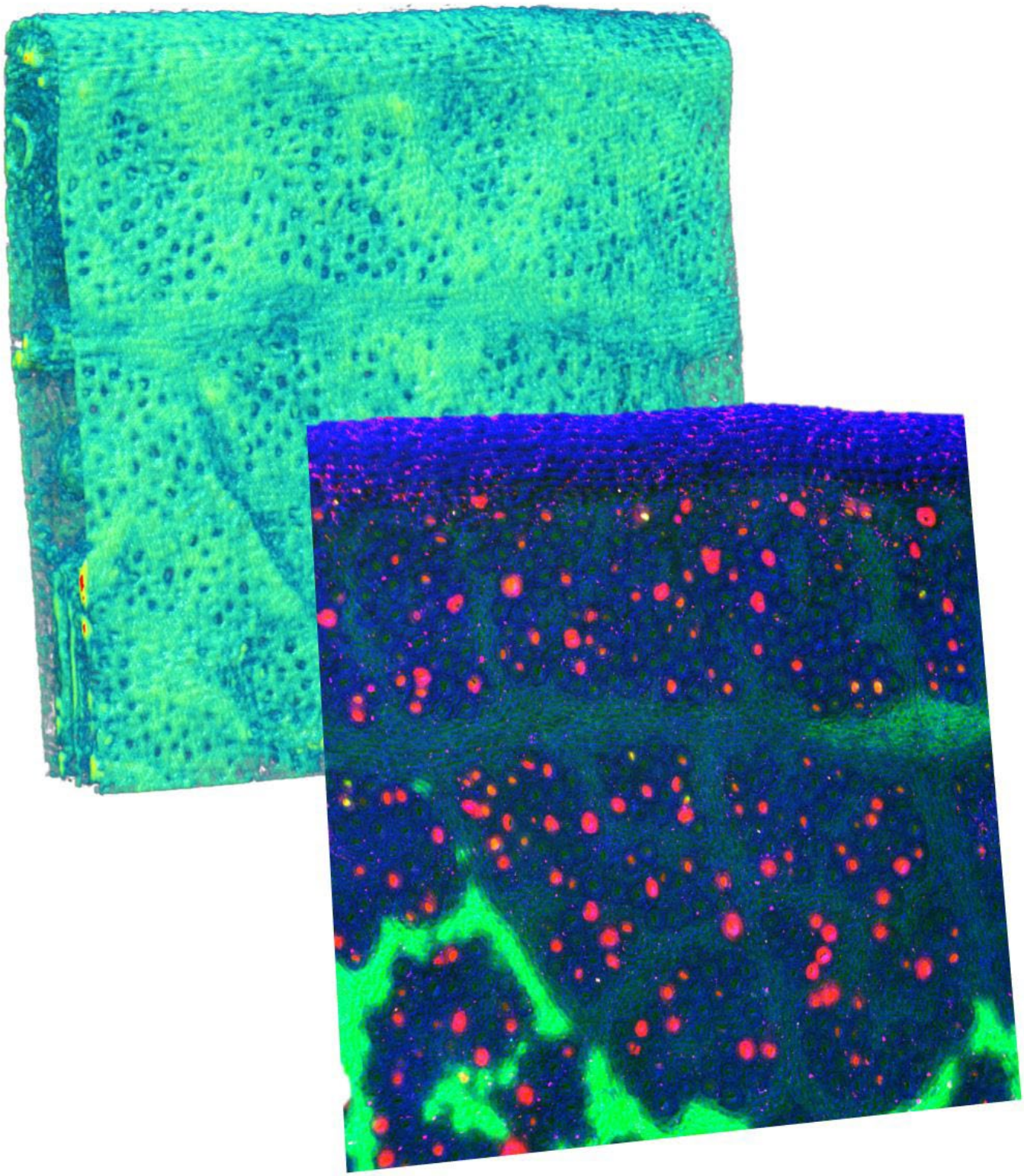
The committee is interested to hear from potential sponsors for the symposium & associated events – please contact the organisers for more information.







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