



## 2009 Profile

ENABLING WORLD-CLASS RESEARCH

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The AMMRF is funded by



**An Australian Government Initiative**  
National Collaborative Research  
Infrastructure Strategy



## FROM THE MINISTER



Senator the Hon.  
Kim Carr

Having had the pleasure recently of opening the Cameca 1280 ion probe in Perth, I was struck by the knowledge and dedication of the AMMRF staff and their obvious passion for the high-end science and technology they provide. It gives me great confidence in the future of world-class science and innovation in Australia, the more so as I have come to realise just how essential such state-of-the-art microscopy and microanalysis are in our research and innovation priorities and to our nation's standing in the global research stakes. We can all be proud of the AMMRF – it is living proof of how collaboration enables us to build better research infrastructure and do better research.

### **Senator the Hon. Kim Carr**

Minister for Innovation, Industry, Science and Research



Australian  
Microscopy & Microanalysis  
Research Facility



The University of Sydney



THE UNIVERSITY  
OF QUEENSLAND



THE UNIVERSITY OF  
WESTERN AUSTRALIA  
*Achieving International Excellence*

THE UNIVERSITY OF  
NEW SOUTH WALES



 **ANU**  
THE AUSTRALIAN NATIONAL UNIVERSITY

SOUTH AUSTRALIAN REGIONAL FACILITY (SARF)



University of  
South Australia



FLINDERS  
UNIVERSITY  
ADELAIDE  
AUSTRALIA

[ammrf.org.au](http://ammrf.org.au)

## FROM THE CHAIR

I am very privileged to continue my close involvement with the AMMRF. As Chair of the Board, I find myself in a prime position to observe the success of this busy national collaborative facility and to assist in guiding its future.

You will read in the following pages of the 2009 AMMRF Profile about an impressive array of research and industry outcomes from the last twelve months, all of which will contribute towards developing Australia's future through the provision of high-technology solutions in fields that include the biosciences, materials, human health, sustainable resources and clean energy. The essential contributions of capital-intensive advanced microscopy and microanalysis to continuing developments across such a range of technologies should not be underestimated, especially as advances today so often rely on features at the nanometre and even the atomic scale.

Across the AMMRF, the quality of instrumentation and expertise now on offer is outstanding by any competitive measure. As a result, the national facility is providing leading-edge enabling solutions to many Australian researchers in academia, in other public-sector research facilities, and in industry. At the commercial end of this distribution, the 2009 Profile amply demonstrates the level to which the AMMRF has been supporting Australia's industrial research capacity, providing microscopy to manufacturing in ways that range from testing services to significant long-term joint research commitments. The AMMRF has also enabled new patents to be lodged and new companies to be formed.

The AMMRF has become an integrator for activities across many disciplines of science and industry, which require microscopy and microanalysis capabilities. These multiple collaborative interactions and the

AMMRF's national and international client base, enable ideas exchange and new technological developments to flourish. I strongly believe that this collaborative approach to the provision of research infrastructure, as is exemplified by the AMMRF's achievements outlined in this document, offers an excellent model for the support and development of capital- and instrument-intensive top-quality research within Australia and beyond. Furthermore, I am well aware that such an achievement would not have been possible without the NCRIS program and the mechanisms through which it has facilitated access to capability that otherwise would not have been available in Australia.

### **Dr Gregory R. Smith**

Chair of Board

## BOARD MEMBERS

### **Dr Gregory R. Smith**

Chair of Board

SciVenture Investments Pty Ltd



### **Prof. Caroline McMillen**

Deputy Vice Chancellor and

Vice President: Research and Innovation

The University of South Australia



### **Prof. Jill Trewella**

Deputy Vice-Chancellor, Research

The University of Sydney



### **Prof. Les Field**

Deputy Vice-Chancellor, Research  
The University of New South Wales



### **Prof. Simon Ringer**

AMMRF Executive Director & CEO  
The University of Sydney



### **Prof. Alan Lawson**

Pro-Vice-Chancellor,  
Research & Research Training  
The University of Queensland



### **Prof. Jim Williams**

Director, Research School of  
Physical Sciences & Engineering  
The Australian National University



We also thank Prof. Merlin Crossley from the University of Sydney and Prof. Richard Russell from the University of Adelaide for their participation on the board.

I am very pleased to bring you this 2009 AMMRF Profile as a showcase of our activities and achievements. As you will see, this last year has been a time of much activity and consolidation, and it is gratifying to follow just how successfully we have developed as an organisation. There can be no doubt that we are now a mature facility that has grown into the strong and dynamic AMMRF foreseen in our initial vision.

To reach this point not only was the business plan of the facility effectively implemented, but also key elements from our strategic planning days, including the installation and commissioning of the remaining two flagship instruments, have come to fruition. Other vitally important activities revolve around the development of our staff through secondments and the sharing of technical and scientific know-how between nodes. The ability to share, and draw from others' experiences is something that gives a strength and depth to our technical and scientific expertise and allows a certain operational consistency to be achieved across all nodes, which is so valuable in underpinning a collaborative facility like the AMMRF.

During this year, Prof. Tim White was appointed as director of the AMMRF node at the Australian National University. Tim's exciting plans for the future include a step increase in the research and teaching portfolio of the newly formed ANU Centre for Advanced Microscopy and to bring e-learning to microscopy through the creation of virtual microscopy labs. Tim has already inspired much activity in this area within the AMMRF and various staff are hard at work on the development of new online collaborative tools to assist with teaching and training.

The ultimate aim of all our activities is to provide the best possible service to AMMRF users in support of their research programs. Our user community is an extremely wide-ranging one and this is an exciting and rewarding part of our work. The research of our users spans broad fields of scientific endeavour from photonics to structural biology and from environmental science to metallurgy. Not only does research occur in the publicly funded sector, but a large and thriving research community exists in the biotechnology, manufacturing and mining



industries as well. These complement the academic sphere and feed directly into Australian innovation. There is increasing awareness that microscopy has a significant role to play in providing solutions for industry and the AMMRF is in a perfect position to provide these services, and indeed continues to do so on a daily basis. Five percent of all our hands-on users are from industry; in addition to those who use the AMMRF as a service facility. That amounts to over 130 industry-based clients – at least two or three calls from industry every week!

The importance of partnerships with industry cannot be underestimated. I was invited by DIISR to address the Australasian Industrial Research Group (AIRG) conference in Canberra recently where I was able to explain the AMMRF's microscopy solutions for industry to an audience of industry and government leaders. The aim of the meeting was to investigate the use of research infrastructure by industry. I was able to highlight the fact that we offer testing services and long-term research programs, either as contract research or through leveraged programs such as the ARC Linkage Projects scheme.

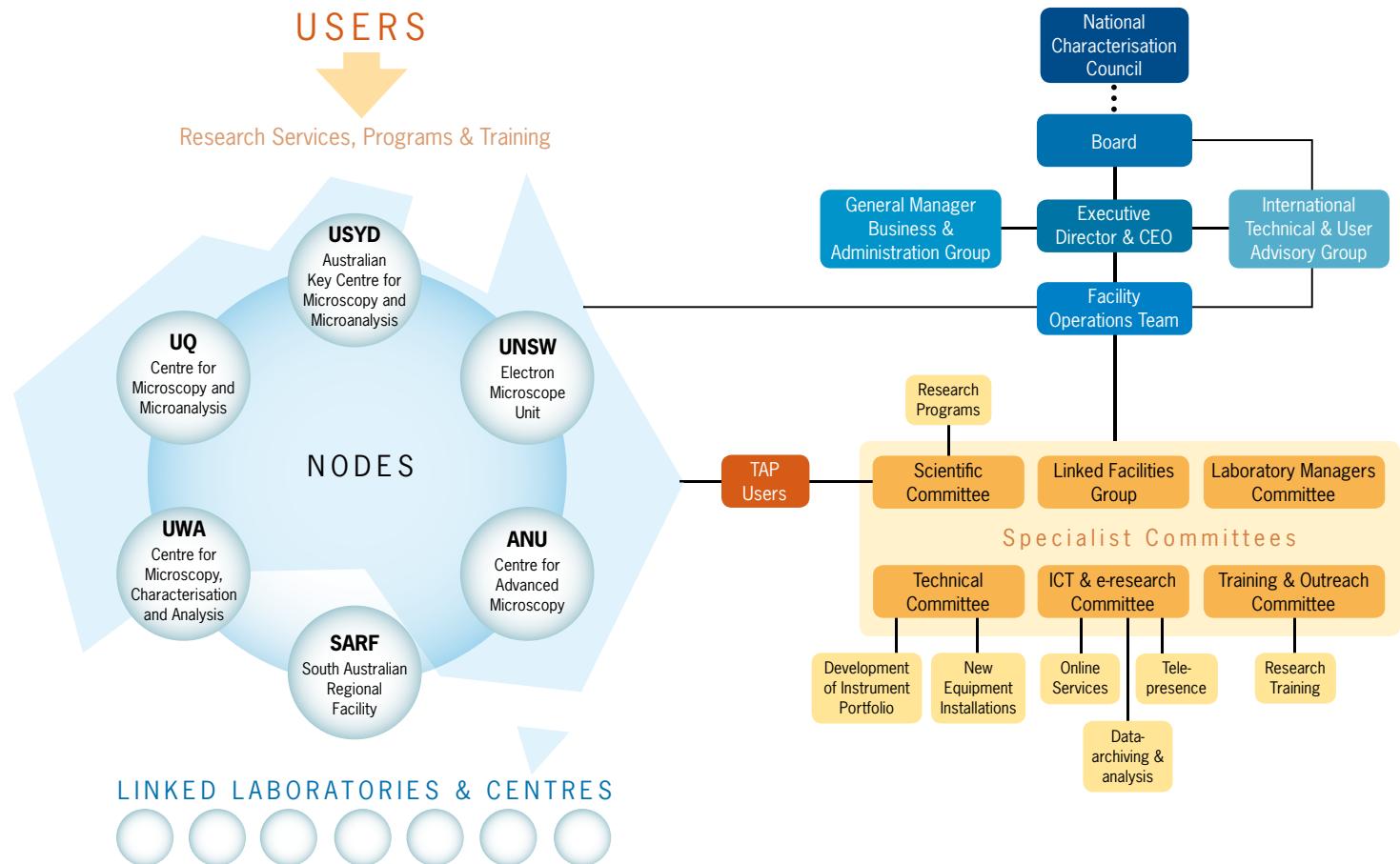
To share this aspect of our activities with you, snapshots within this Profile illustrate the diversity and strength of the AMMRF's research and industry portfolio. They highlight not only a range of publicly funded

research outcomes, but also examples of how microscopy is supporting manufacturing, and how we help to enable new commercialisation opportunities. Research and innovation outcomes enabled through the AMMRF benefit Australia in many ways: strengthening our position at the forefront of the world's research community and providing real-world solutions for improving healthcare, better communications and sustainability, to name but a few. From investigations into understanding the most basic properties of multi-ferroic materials and the biomineralisation in marine molluscs to studies into drug delivery and metallic coatings for extreme conditions, the projects span the range from basic to applied research.

Patents have been filed for several applications, for example the exploitation of Australian biodiversity for the creation of antibacterial medical devices, and a spin-off company launched to serve the worldwide oil industry.

The AMMRF is now well and truly operational and I urge researchers, students, engineers and technicians whether from academia or industry to contact the AMMRF to explore how we can extend your research horizons.

**Prof. Simon P. Ringer**  
Executive Director & CEO



## VISION

The AMMRF is Australia's peak research facility for the characterisation of materials through macro, meso, nano and atomic length scales by means of advanced microscopy and microanalysis.

## MISSION

The AMMRF is a user-focused, interdisciplinary organisation that employs microscopy and microanalysis to explore structure-function relationships of materials in the physical, chemical and biological sciences and their technologies. Accessible to all Australian researchers, the facility provides a quality user experience enabled through the provision of world-class research services, research training and research programs.

## NODES

As constituent of an unincorporated joint venture, the nodes retain their own identity but work together to provide a comprehensive and coordinated range of services to the research community through a collaborative grid of capability.

The nodes have a wide and overlapping range of expertise and experience, technically and through their own academic programs. As well as supporting publicly funded research, many of the nodes also offer services to industry, bringing microscopy solutions to the manufacturing and industrial sector.

The nodes, in conjunction with the Linked Labs, bring an impressive array of instrumentation and expertise to the Australian research community.

## LINKED LABORATORIES

**RMIT University: Microscopy and Microanalysis Facility**

Provides advanced electron microscopy facilities, including high resolution and environmental scanning electron microscopes (SEM), transmission electron microscopes (TEM), scanning auger nanoprobe, X-ray photoelectron spectroscopy, and dynamic light-scattering spectroscopy systems.

**CSIRO, Australian Animal Health Laboratory: Australian Biosecurity Microscopy Centre**

Offers a live-cell and cryo-TEM imaging facility within a PC3/PC4 bio-containment environment. This is a unique capability, enabling fundamental research with biological agents that need the highest level of containment.

**Macquarie University: Optical Microcharacterisation Facility**

Combines technologies in Raman microscopy, fluorescence excitation and lifetime spectroscopy, surface-enhanced Raman microscopy and near-field scanning microscopy.

**Queensland University of Technology: Analytical Electron Microscopy Facility**

Offers advanced SEM platforms, including a dual-beam focussed ion beam with mineral liberation analysis software, and an analytical environmental SEM complete with a cooling and heating stage.

**James Cook University: Advanced Analytical Centre**

Provides specialist microanalysis capabilities, including electron-probe microanalysis, low-vacuum chamber SEM, confocal laser scanning microscopy and an atomic force microscope fitted with a nano-indentor.

**Curtin University of Technology: John de Laeter Centre of Mass Spectrometry**

Houses single and multicollector sensitive high-resolution ion microprobes (SHRIMP) for quantitative isotopic and elemental analysis.

## LINKED CENTRE

**The University of Queensland: Australian Institute of Bioengineering and Nanotechnology (AIBN)**

The AIBN has become the first AMMRF Linked Centre. A dedicated microscopist advises and supports researchers on appropriate techniques and acts as a conduit to AMMRF capability.

## NODE DIRECTORS

**Prof. Simon P. Ringer**

The University of Sydney (AMMRF Headquarters)  
Australian Key Centre for Microscopy and Microanalysis

**Prof. John Drennan**

The University of Queensland  
Centre for Microscopy and Microanalysis

**Prof. David Sampson**

The University of Western Australia  
Centre for Microscopy, Characterisation and Analysis

**Prof. Paul Munroe**

The University of New South Wales  
Electron Microscope Unit

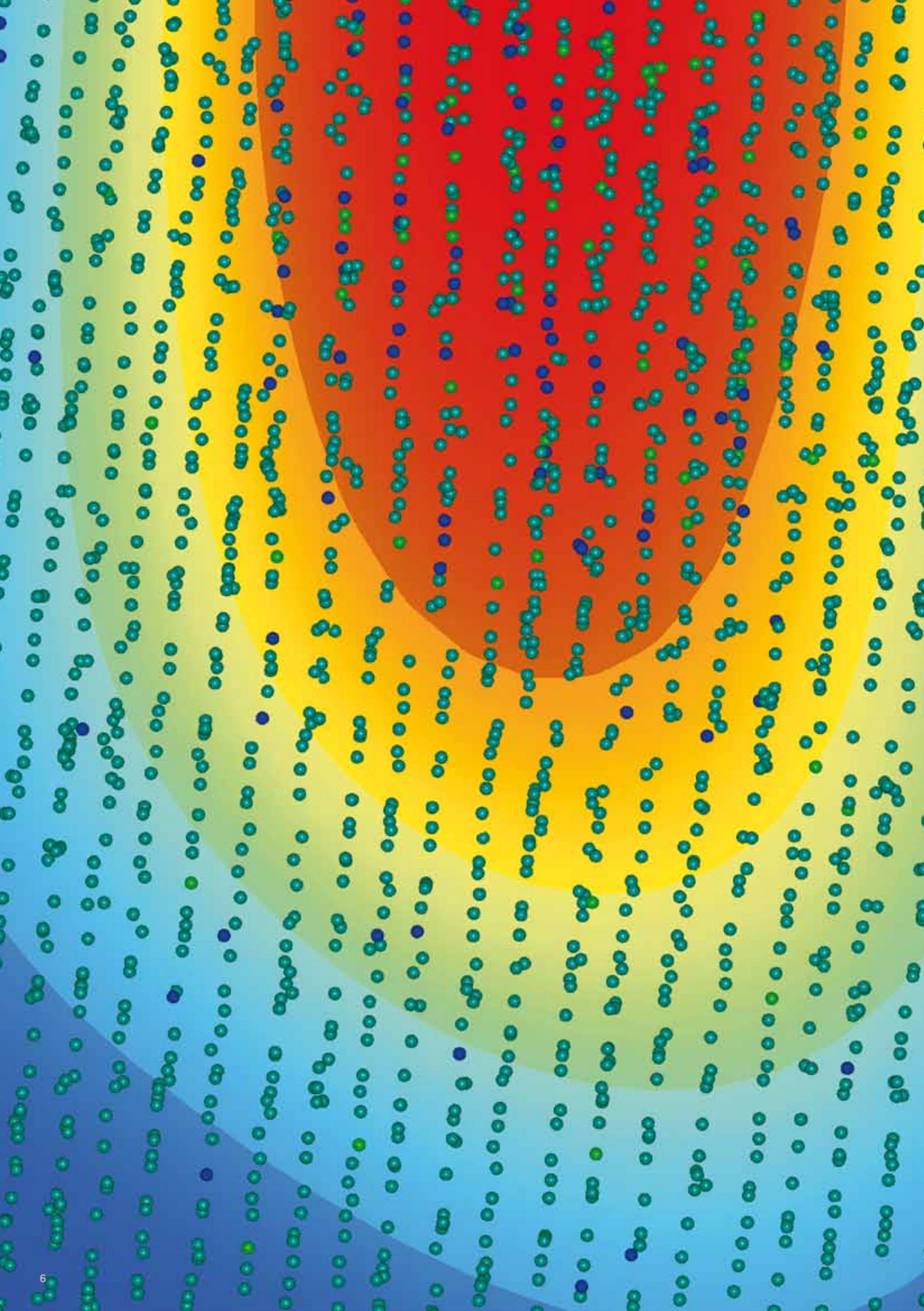
**Prof. Tim White**

The Australian National University  
Centre for Advanced Microscopy

**Prof. Hans Griesser**

South Australian Regional Facility (SARF)  
*Ian Wark Research Institute (University of South Australia)*  
*Adelaide Microscopy (The University of Adelaide)*  
*Flinders Microscopy (Flinders University)*

We also thank A/Prof. Tim Senden from the Australian National University for his period of directorship.



Your partner of choice for microscopy, microanalysis and image analysis at the atomic, molecular and cellular scale

The microscopy and microanalysis offered by the AMMRF covers those characterisation techniques that enable the visualisation and chemical analysis of matter using beams of electrons, photons, X-rays and ions. They cover a large size range, allowing single-atom resolution through the use of atom probe tomography to the micrometre resolution of centimetre-sized objects such as small organs or rock cores through the use of micro-CT.

A suite of flagship instruments is available through the AMMRF with individual instruments housed in most nodes around the country. These instruments provide highly specialised, state-of-the-art capability, often unique within Australia.

In addition to these top-flight multi-million-dollar flagships, a whole raft of high-demand instruments are available to enhance users' research. These include scanning and transmission electron microscopes with spectroscopy capabilities, confocal and light microscopes, Raman and scanning probe microscopes and XRD instruments. Additional and extremely important components of AMMRF capability are the expertise in specimen preparation and data analysis on hand for all users.

The techniques available on the instruments are wide-ranging and can be applied to an extremely broad range of research fields from the life sciences, such as cell biology, to materials science and mineralogy, polymer chemistry, engineering, nanotechnology, environmental sciences, and investigations of cultural artefacts. An introduction to these techniques is found on the following pages.

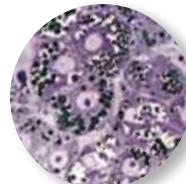
This diversity of instrumentation and research fields, along with the level of expertise, makes for a highly dynamic and vibrant facility. The staff are always happy to help users find the right technique to answer their research questions.

## Specimen Preparation



Biological Materials  
Cell Culturing and Molecular Preparation  
Thermomechanical Processing

## Light and Laser Optics



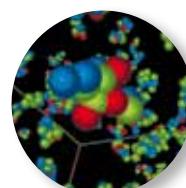
Confocal and Fluorescence Microscopy  
Optical Microscopy  
Flow Cytometry and Cell Sorting  
Live-cell Imaging  
Vibrational and Laser Spectroscopy  
Laser Microdissection



Scanning Electron Microscopy  
Analytical Spectroscopy  
In-situ Imaging and Testing  
Metrology



Transmission Electron Microscopy  
Analytical Spectroscopy  
Diffraction  
Phase and Z-contrast Imaging  
Cryo Techniques and Tomography



Advanced Ion Platforms  
Nanoscale Mass Spectroscopy  
Atom Probe Tomography  
Ion Milling and Machining  
Ion Implantation



Scanned Probe Techniques  
Atomic Force Microscopy  
Scanning Tunneling Microscopy  
Raman Spectroscopy



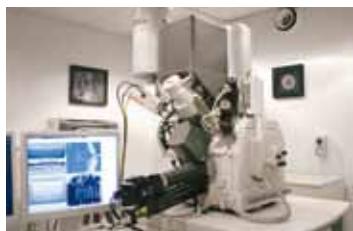
X-ray Technologies  
X-ray Diffraction  
X-ray Fluorescence  
X-ray Microtomography



Visualisation and Simulation  
Computed Spectroscopy  
Computed Diffraction  
Image Simulation, Analysis and Data Mining

◀ Atom probe image showing a small crystal as it begins to grow inside an aluminium alloy. The ordered planes of aluminium atoms (light blue spheres) deviate as they approach the growing crystal of zinc and magnesium atoms (dark blue and green spheres, respectively). Image by Peter Liddicoat @ The University of Sydney.

## FLAGSHIP INSTRUMENTS



### FEI NOVA NANOLAB 200 DUALBEAM FIB AND THE FEI HELIOS NANOLAB DUALBEAM FIB

#### **High-resolution scanning electron microscopes with focused ion beams (FIBs), energy dispersive X-ray spectroscopy and electron backscattered diffraction systems**

*Electron Microscope Unit; The University of New South Wales*

*Adelaide Microscopy; The University of Adelaide; South Australian Regional Facility (SARF)*

With dual high-resolution electron and ion columns, these advanced microscopes offer a key capability in sub-nanometre-resolution imaging, in high-precision cross-sectioning by ion milling, and in elemental and orientational analysis. They also make possible 3-D image reconstruction by slice-and-view, script-driven large-scale prototype patterning and preparation of thin-foil TEM samples of difficult materials.



### CAMECA IMS 1280 AND NANOSIMS 50 ION PROBES

#### **Ion probes for chemical and high-precision isotopic analysis and imaging to the nanoscale**

*Centre for Microscopy, Characterisation and Analysis; The University of Western Australia*

This secondary-ion mass spectrometry facility offers high-sensitivity and high-precision isotope-ratio analysis for a diverse array of materials.



### HIGH-THROUGHPUT CRYO-TEM FACILITY

#### **Unique high-throughput cryo-transmission electron microscopy (TEM) facility for structural analysis**

*Centre for Microscopy and Microanalysis; The University of Queensland*

This cryo-TEM facility has the latest technologies, including ultrahigh-resolution CCD cameras and specialist cryo-holders, creating a world-class platform for high-throughput structure determination.



### IMAGO LOCAL ELECTRODE ATOM PROBES

#### **Local electrode atom probes for atomic-level analysis of materials**

*Australian Key Centre for Microscopy and Microanalysis; The University of Sydney*

This world-leading facility provides comprehensive capabilities in atom probe tomography. Voltage-pulsed atom probe and pulsed-laser atom probe open up this powerful technique to a large variety of applications, from conductive to less-conductive materials.



### HIGH-RESOLUTION SEM MICROANALYSIS FACILITY

#### **Unique suite of field-emission scanning electron instruments for materials analysis**

*Electron Microscope Unit; The University of New South Wales*

This suite of field-emission scanning electron microscopes provides a high-throughput, high-precision facility able to structurally characterise materials and to detect and quantify elements at very high spatial resolution. High-resolution SEM imaging completes this comprehensive imaging and analysis platform.



### PHI TRIFT V NANOTOF TOF-SIMS

#### **Time-of-flight secondary ion mass spectrometer (ToF-SIMS) for surface analysis and depth profiling**

*Ian Wark Research Institute; University of South Australia; South Australian Regional Facility (SARF)*

The PHI TRIFT V nanoToF ToF-SIMS is able to conduct surface analysis at the nanometre level for the identification and mapping of elements and molecules. This instrument is unique in its ability to combine sensitivity, spatial resolution and chemical specificity with parallel detection of atomic and molecular species.

The AMMRF offers the means to answer diverse questions and can advise on the most appropriate techniques for your project. An illustrative summary of selected techniques is set out below.

## BIOSCIENCES

To determine the **structure or anatomy** of molecules, cells and tissues, a number of approaches are possible. For proteins and single particles, such as viruses, high-resolution cryo-electron tomography can generate 3-D structural reconstructions at the nanometre scale. At the level of microorganisms and eukaryotic cells, scanning and transmission electron microscopy are able to visualise the structure of exposed surfaces and internal structures, respectively. Hard tissues can be challenging to analyse, but the focused ion beam instruments allow thin sections to be cut for TEM. Elemental analysis can also be useful on hard tissues and is routinely done by using spectroscopy on an SEM or TEM. The ion probes offer isotopic

analysis at high sensitivity and resolution. At the tissue level, micro-CT (and the forthcoming nano-CT) offers additional possibilities to reveal complex internal structures, such as those seen in the inner ear, in 3-D without the need for destructive specimen preparation.

Of course light microscopy always remains an illuminating option. For studying the functional **interactions of proteins** with other macromolecules, a number of light and optical techniques are available. For example, the proximity of two molecules can be determined with FRET, protein movements can be tracked in live cells by using FRAP and surface molecules selectively visualised using TIRF.



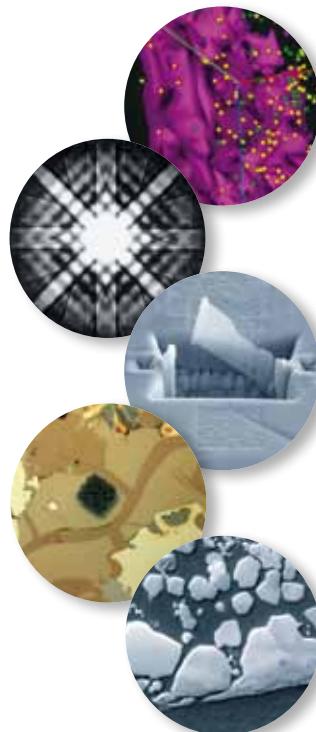
## PHYSICAL SCIENCES

For **visualising structure** at the atomic scale, atom probe tomography can identify and localise atoms in 3-D, while high-resolution TEM reveals crystalline lattices and their defect structures. At the intermediate length scales, SEM and TEM allow visualisation of grain boundaries, strain patterns and defects such as dislocation loops, while micro-CT enables the visualisation of internal structures without the need for destructive sample preparation. At the nanometre scale, atomic force microscopy and scanning tunneling microscopy allow imaging of structure, but also are able to measure dynamic electronic, mechanical or magnetic properties of samples.

Qualitative and quantitative information on **crystal structure** can be gathered by using X-ray or electron diffraction. Detailed insights into defect structures can be obtained with specialised electron diffrac-

tion techniques such as convergent-beam approaches. Electron backscattered diffraction on an SEM also provides information on crystal orientation and texture in polycrystalline materials.

**Elemental, isotopic and chemical composition** can be determined in diverse materials at high resolution and sensitivity with electron spectroscopy and ion probes, as can depth profiles and surface analyses. Energy- or wavelength-dispersive X-ray spectroscopy are available for routine elemental analysis and localisation at intermediate length scales on SEMs and TEMs. Rich chemical information, particularly about organic molecules and materials, can be obtained from Raman spectroscopy.





## SUPPORTING AUSTRALIA'S PRIORITY RESEARCH

Strategic relationships have been established between the AMMRF and various ARC Centres of Excellence including Functional Nanomaterials, Design in Light Metals, Quantum Computing, and Advanced Silicon Photovoltaics and Photonics. The AMMRF has worked closely with these centres to help them identify how microscopy and microanalysis can contribute to their research programs.

Take the ARC Centre of Excellence in Advanced Silicon Photovoltaics and Photonicse (CoE), for example. It is focused on advancing silicon photovoltaic research, and applying these advances to the related field of silicon photonics, for renewable energy solutions. The centre has been using the world-class facilities such as the dualbeam focused ion beam (FIB) flagship capability at the University of New South Wales (UNSW) node and high-resolution transmission electron microscope at the University of Sydney (USyd) node to characterise photovoltaic materials and devices.

The partnership between the AMMRF and the CoE has been strengthened with the recent award of an ARC Discovery Project grant that will see centre researchers collaborating directly on joint research projects with AMMRF research staff to harness the full strength of AMMRF capability.

This strengthened partnership will advance understanding of the structure-activity relationships in silicon-based photovoltaics by linking their measured photovoltaic properties and performance with observations of their nanostructure. The engineering expertise from the CoE combined with the microscopy skills of AMMRF researchers will provide a dynamic and synergistic research enterprise. In particular, the AMMRF flagship atom probe at USyd and the focused ion beam instrument at UNSW will generate data to inform the precise design of more efficient photovoltaics.

In a country where the sun shines so often, it is no surprise that solar energy is considered as a viable renewable energy alternative for Australia. The partnership between the AMMRF and the CoE will help this vision to become a reality.



The AMMRF supports researchers from major research centres and institutes every day, as highlighted by this sample of the centres:

- CSIRO
- DSTO
- ANSTO
- National Measurement Institute
- University of Wollongong
- Australian Institute of Bioengineering and Nanotechnology
- Centre for Exploration Targeting
- The Centre for Expertise in Photonics
- Centre for Strategic Nano-Fabrication
- The Australian Wine Research Institute
- Research Institute for Climate Change and Sustainability
- The Ian Wark Research Institute
- Australian Centre for Plant Functional Genomics

### Co-operative Research Centres

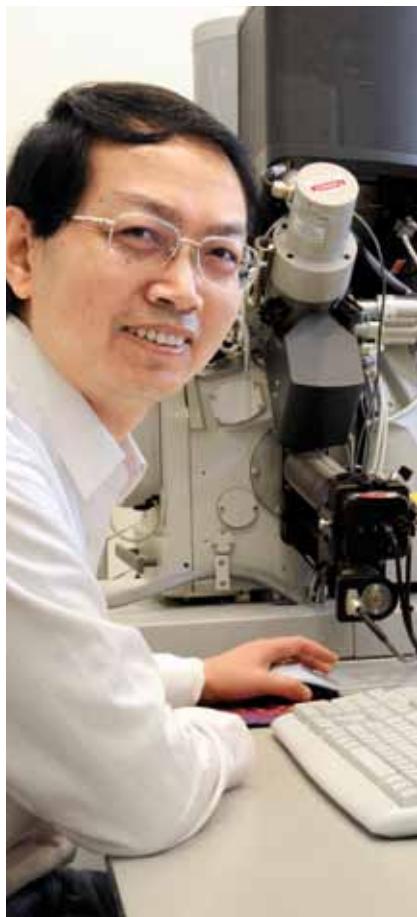
- CRC for Hearing
- CAST CRC (Alloy and Solidification Technology)
- CRC for Polymers
- Parker CRC for Integrated Hydrometallurgy Solutions
- CRC for Beef Genetic Technologies
- CRC for Greenhouse Gas Technologies
- Molecular Plant Breeding CRC
- CRC for Advanced Composite Structures

### ARC Centres of Excellence

- Advanced Silicon Photovoltaics and Photonics
- Functional Nanomaterials
- Quantum Computer Technology
- Ultrahigh Bandwith Devices for Optical Systems
- Design in Light Metals
- Integrative Legume Research
- Electromaterials Science
- Plant Energy Biology

### Health and medical research

- Institute for Molecular Bioscience
- Queensland Brain Institute
- Queensland Institute of Medical Research
- Western Australian Institute for Medical Research
- Telethon Institute for Child Health Research
- Lung Institute of Western Australia
- Special Research Centre for the Molecular Genetics of Development
- National Centre for Stem Cell Research
- The Hanson Institute
- Garvan Institute
- Centre for Vascular Research
- Victor Chang Cardiac Research Institute
- Prince of Wales Medical Research Institute
- Centenary Institute of Cancer Medicine and Cell Biology



## ACCESS IS THE CORNERSTONE OF THE AMMRF

The AMMRF is fundamentally a user-oriented facility, which aims to enable the best research from around the country, providing high-end instruments and expertise to all Australian researchers. Once a researcher has identified the location of the instrument they wish to access, they approach that node directly to discuss their project and to register as a new user. After a rapid evaluation of the merit and feasibility of the project they will get the technical

and scientific advice to help them make the most from their access. The expert AMMRF staff can help the new user to design their experiment more effectively, train them on the instruments and take them through collection and interpretation of data, publication and discussion of future directions.

For publicly funded researchers, only a nominal access charge is made to contribute towards instrument maintenance.

## TRAVEL AND ACCESS PROGRAM

The Travel and Access Program (TAP) continues to attract strong interest across the country. Through the provision of financial assistance to users wishing to travel to distant nodes or linked labs, the TAP enables the full access that is a key feature of the AMMRF's mission. TAP funding contributes to the costs of travel, accommodation and beamtime. This enabling funding lets

researchers do a pilot project in order to gather enough information to form the basis of a more substantial grant application. Applications for TAP funding are made through the AMMRF website and are assessed within three weeks. Applications come from the full range of publicly funded research institutions and all applications are judged on their merit.

### Examples of funded TAP projects

- Titanium laser welding for medical devices
- FIB tomography meets X-ray microtomography: a 3-D approach to aid enhanced oil recovery
- Why does stainless steel corrode?
- Switchable interfaces
- New approaches to understanding grain boundary chemistry
- Lithium niobate using standard Ti diffusion technique
- Amino-acid functionalised calisarenes as hydrogelators
- Identification of the mineral phases of chiton teeth
- Cubomedusae statoliths – an environmental archive?
- Modification of 3-D scaffolds via plasma polymerisation
- Constraining pressure in the solar nebula by analysis of primitive meteorites
- Climatic effect on violent CO<sub>2</sub> degassing in seismically active zones
- Investigations of complex nanodomains across a new lead-free morphotrophic phase boundary thin-film system
- Wool/hair keratin macrofibril structure
- Induction and identification of lysogenic in the fish pathogen, *Streptococcus iniae*
- Atom probe study of B, P & C in Incinel 718 superalloy
- Microstructure of cryogenically treated high-performance tool steels
- U-Th dating and mechanism of vein formation in contractional strike-slipsetting
- Chiral determination by electron diffraction
- The isolation of bacteriophage with the potential for biocontrol of luminescent bacteria in aquaculture hatcheries
- Material properties of Southern-ocean pteropods influenced by increasing ocean acidification
- Environmental exposure on properties of SiN<sub>x</sub>H<sub>y</sub> thin films for MEMS application
- Impact and causes of sponge disease in the Torres Strait and the Great Barrier Reef
- Improved oil recovery from Tensleep sandstone: studies of brine-rock interactions by micro-CT, AFM and contact angle
- Releasing the mandibular neuromuscular envelope with botulinum toxin type A (BTX-A): skeletal, dental, and muscular effects
- An atom probe study of high-strength spinodally-formed FeNiMnAl alloys
- Cryo-microtomy of frozen hydrated samples and immunogold labelling
- Micro-CT scanning of a fossilised dinosaur (*Iguanodon*) brain
- Distinguishing the formation of gunshot residue particles from environmental particles
- Grain misorientation in Al- and Mg-alloy high-pressure die castings
- Sn-Cu-Ni alloys for lead-free soldering
- H, C and Cl isotopes in lunar apatites
- Amine-functionalisation of polymer surfaces using plasma reactor for attachment of small molecules and peptides
- Microstructural and crystallographic analysis of nanostructured complex-oxide thin films
- Cryo-TEM investigation on the evolution of nanostructure of biomimetic protein *rec1-resilin*
- Intracontinental orogenesis and fluid flow in central Australia
- Mapping the intracellular distribution of gold(I) chemotherapeutics using nano secondary-ion mass spectrometry
- Microscopic origin of ferromagnetism of diluted magnetic semiconductors

## Outlines of four typical TAP projects

### **Alex Buddery from the University of Queensland visited the AMMRF at the University of New South Wales**

Titanium laser welding is used to prepare critical medical components such as heart valves and stents. The act of welding can influence the metal microstructure, giving rise to a weakening of the component. Focussed ion beam microscopy was used to prepare samples across welded joints with a view to building up a detailed microstructural map of different weld conditions.



### **Prof. Ian Baker from the Thayer School of Engineering, Dartmouth College, Hanover, NH, USA visited the AMMRF at the University of Sydney**

The control of precipitation in metal alloy systems provides the key to tailoring the mechanical properties of these materials. Increasingly it is observed that the scale of the precipitation process that can dramatically influence properties is as small as a few atoms across. Combining atom probe technologies with direct observation by electron microscopy at the University of Sydney, Prof. Baker has begun to explore the first stages of precipitation in important Fe-Ni-Mn-Al alloys.



### **Anne Sandstrom from the University of Queensland visited the AMMRF at University of South Australia**

Surface modification of materials used as scaffolds in medical implants is important in building the interface between the foreign implant and living tissue. As such, it can be key to the scaffold's successful incorporation into human patients. The facilities at the University of South Australia allowed Ms Sandstrom to analyse the surface modifications by using electron spectroscopy as part of her PhD research.



### **Dr David Wacey from the University of Western Australia visited the University of Adelaide**

The micro-CT at the AMMRF at the University of Adelaide was used to investigate the morphology of a living thrombolite from Lake Clifton in Western Australia. The thrombolites are a unique sub-class of stromatolite, notable for their clotted texture; in contrast, other stromatolites have a laminated texture. Little is known about the way thrombolites grow and mineralise and the micro-CT results are starting to shed new light on the formation mechanisms of these intriguing biological communities. Visualisation of the data was carried out by Derek Gerstmann at the University of Western Australia.



## HOW TO APPLY

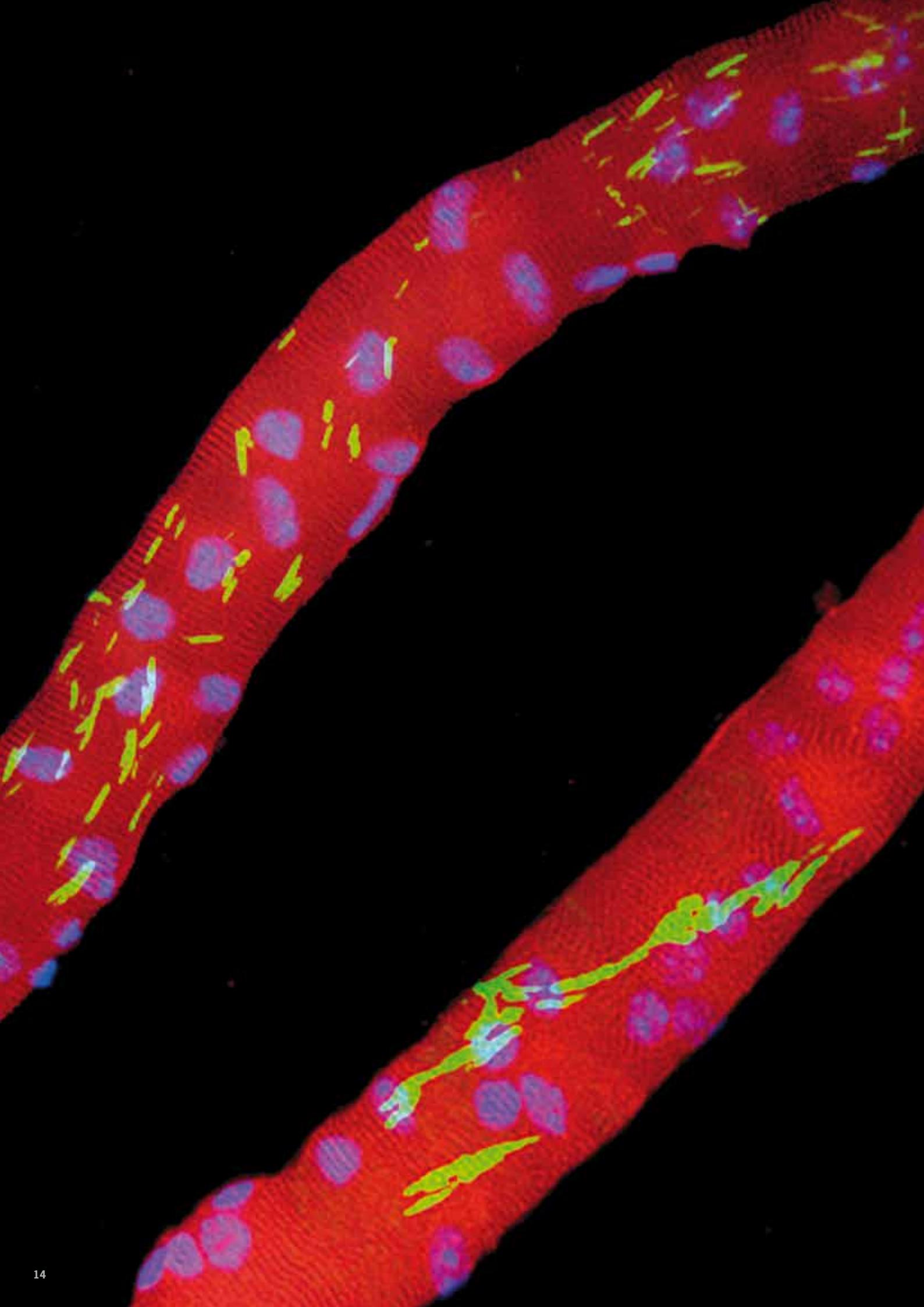
All Australian researchers are eligible to apply online to the TAP through the AMMRF website at

[ammrf.org.au](http://ammrf.org.au)

Applications are assessed rapidly by the Scientific Committee and funding decisions are based on the scientific merit and feasibility of the proposed research.

If you wish to use specific AMMRF infrastructure, please contact the relevant node to discuss the project and arrange access.

Contact details on page 37.



## THE AMMRF – ENABLING WORLD-CLASS RESEARCH

Research is at the heart of the day-to-day activities of the AMMRF. A satisfied stream of users passes through its doors to address and solve their research problems. The world-class outcomes that result help to place Australia at the forefront of many fields of research, with papers appearing in high-impact journals, and scientists being invited to present their work at international meetings and symposia.

The outcomes are also instrumental in researchers achieving considerable grant success, and numerous prestigious fellowships have been awarded to users. The AMMRF is providing access to technologies that were not previously available in Australia and so it is enabling new and high-quality research.

By its nature, research is serendipitous, so approaching experimental problems from

many angles is the best way to generate innovative, and often unexpected, outcomes. The AMMRF contributes significantly to providing effective experimental approaches and developing new and innovative techniques for future researchers to use.

## ADDRESSING THE AUSTRALIAN SCIENCE AGENDA



### Sustainability

- |                         |  |
|-------------------------|--|
| Agriculture             | » Developing lead-tolerant plants  |
| Australian Biodiversity | » <i>Eremophila</i> – patented antibacterial coatings                                    |
| Mining                  | » Usage by BHP Billiton saved many millions of dollars<br>» Mico-CT of oil-bearing rocks |

### Energy

- |                   |  |
|-------------------|--|
| Cleaner           | » Lighter alloys reduce emissions                                      |
| Alternative Fuels | » Biochar – generates energy, sequesters carbon and nourishes the soil |
| Renewable Energy  | » Photovoltaic CoE partnership – improving solar energy                |

### Climate Change

- |              |   |
|--------------|---|
| Ocean Health | » Monitoring coral-reef organisms – symbiotic algae |
|--------------|---|



### Frontier Technologies – Transforming Industries

- |                    |   |
|--------------------|---|
| Advanced Materials | » Bulk metallic glass and layered aluminium – new metals for new applications                                   |
| Nanotechnology     | » Electrical conductivity of DNA for nanoelectronics<br>» Carbon nanotubes for improved cell biology techniques |
| Manufacturing      | » BlueScope Steel linkage – more efficient production of better steel   |



### Healthy Population

- |                        |  |
|------------------------|--|
| Diagnosis & Treatments | » Magnetic drug delivery for difficult drugs<br>» Rescuing muscle-wasting diseases |
|------------------------|--|

### Safeguarding Australia

- |                      |  |
|----------------------|--|
| Defence Technologies | » High-temperature coatings for extreme applications |
|----------------------|--|

◀ Confocal microscopy image of irregular expression and location of actin (green) in isolated skeletal muscle fibres of transgenic mice.  
Image by Prof. Nigel Laing and Dr Gina Ravenscroft @ The University of Western Australia.

## THz LIGHT AT THE END OF THE FIBRE

**FLAGSHIP**

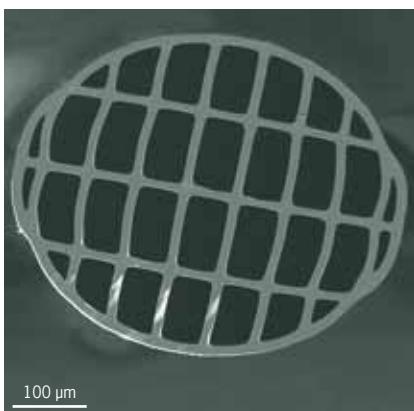
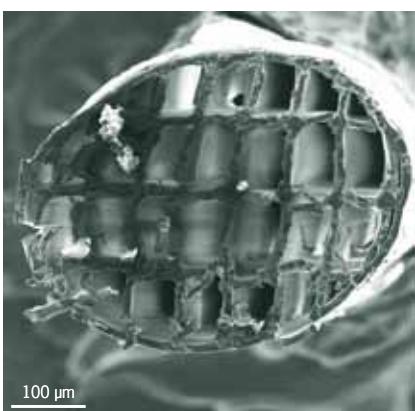
Far-infrared spectroscopy has many applications in the detection of biological and chemical substances. However, current spectrophotometers for biomedical applications can require inconveniently large samples. By using specially designed optical fibres that transmit far-infrared radiation, it should be possible to concentrate the light coming from a sample, so that much smaller samples can be used. The efficient transmission of far-infrared (terahertz, THz) radiation in optical fibres is still in its infancy and not easy to achieve.

Prof. Tanya Monro and her group at the University of Adelaide (UAdel) are working to develop such fibres. Transmission of the far-infrared light is based on the principle of total internal reflection, just like in conventional optical fibres. Efficiency is maximised by the presence of an array of air holes that are smaller than the wavelength of the infrared radiation being transmitted – the more porous the fibre, the more efficient the transmission.

To evaluate the integrity of their new fibres, and to accurately measure their porosity, the team has used the focused ion beam (FIB) instrument in the AMMRF at UAdel. Unlike with other techniques, the FIB can slice through the fibres without causing their delicate structures to collapse.

The most promising structure that the team has created exhibits an asymmetrical design of rectangular holes with a significantly higher porosity than the more

common design with a hexagonal array of circular holes. These asymmetrical fibres demonstrate low losses and low dispersion of the transmitted radiation and the maintenance of polarisation. They are therefore a promising solution and a good substitute for the free-space THz propagation systems in current instruments. They also open up new opportunities in THz biosensing, where the porous fibers could be used to sense ultra-small samples.



Rectangular THz fibre cut with a conventional blade (left) and with the FIB (right).

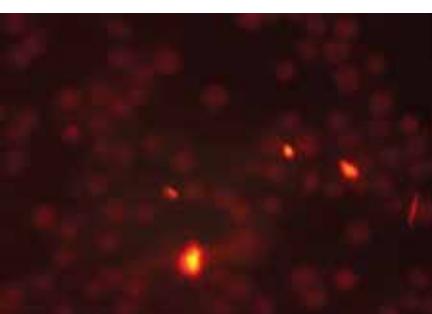
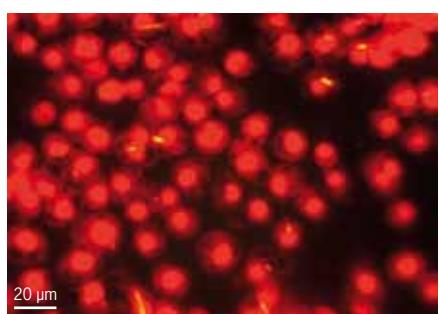
## PENETRATING LIFE'S LITTLE MYSTERIES

Carbon nanotubes have been heralded as potential components for advanced biomaterials, largely due to their structural, mechanical and electronic properties. The applications of carbon nanotubes in biosensing and drug delivery are being explored intensively, yet it is also important to understand how cells behave when in contact with carbon nanotubes. Prof. Joe Shapter, Prof. Nico Voelker and Dr Frances Harding at Flinders University have investigated cell attachment,

morphology and behaviour on vertically aligned carbon nanotubes attached to porous silicon. Immobilising nanotubes in this manner produces what resembles a nanoscale bed of nails. They were attached to the silicon in such a way as to make them able to cope with the extended exposure to water needed for cell culture. The team found that cells adhered well to the nanotube-decorated surfaces. In fact, the cells appeared to become impaled on the vertically aligned single-walled carbon nanotubes (SWCNTs). This was demonstrated by the use of fluorescent dyes in conjunction

with the confocal microscopes at Flinders Microscopy (part of the South Australian Regional Facility). The nanotubes appear to penetrate the outer cell membrane and even the nuclear envelope, allowing molecules to directly enter the nucleus as can be seen in the images. Despite the breaching of their membranes, the cells remained viable.

These intriguing results could have a number of applications including possible improvements to a technique called transfection. Widely used in cell biology research, it is a method where foreign DNA is delivered into cells under experimental conditions to test the function of genes. The team is currently planning proof-of-concept experiments to demonstrate the feasibility of this suggestion. In the longer term, incorporating surface-patterning techniques when laying down the initial surface modification for SWCNT attachment, could permit differently functionalised 'islands' of SWCNT to be precisely located on a surface. Such novel surfaces could form part of an array platform to screen DNA–cell or protein–cell interactions.



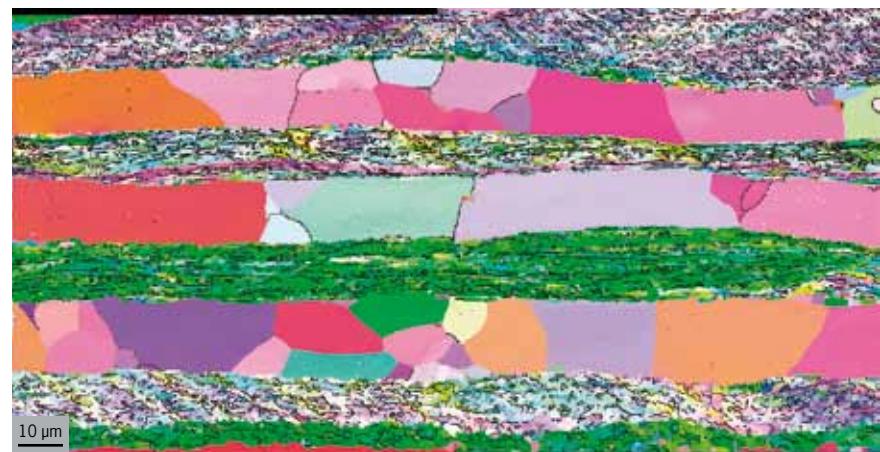
Permeabilisation of cells and their nuclei on a SWCNT array (left) and on plain porous silicon (right.) The orange dye will not normally enter the cell or the nucleus if their membranes are intact.

## LAYERS OF STRENGTH AND FLEXIBILITY

### FLAGSHIP

The mechanical properties of a bulk material depend on its microstructure, so materials engineers use the knowledge of the structure–property relationships to alter a material's performance. In this way, they can often transform a readily available, ordinary material into one with an excellent combination of properties. By exploiting this principle, Dr Quadir and Oday Buhamad from the University of New South Wales (UNSW) have engineered novel microstructures in aluminum, which is the most abundant metal in the Earth's crust. Their objective was to expand the uses of this low-cost and light-weight material in extreme engineering applications.

The researchers roll bonded 32 alternating layers of pure aluminum and aluminum containing 0.3% scandium into a 0.5 mm thick sheet. This generated a strong sheet of metal with nanoscale substructures in both types of layers. After a critically controlled heat treatment, the pure aluminum layers recrystallised to large grain sizes, but the aluminium-scandium (Al-Sc) layers retained their sub-micrometre structures. This was demonstrated by using focused ion beam



Crystal orientations in a longitudinal section of heat-treated roll bonded aluminium sheet.

milling and field-emission scanning electron microscopy in the AMMRF at UNSW. The lamellar-composite aluminium sheet, with a novel microstructure of alternating coarse-structured aluminum and extremely fine-structured Al-Sc layers, is shown in the image.

The composite sheet they generated has potential in various engineering applications. The nanostructured Al-Sc layers possess high strength and high wear resistance and the microstructured aluminum layers possess high ductility and formability. The combination of these two materials in a single sheet

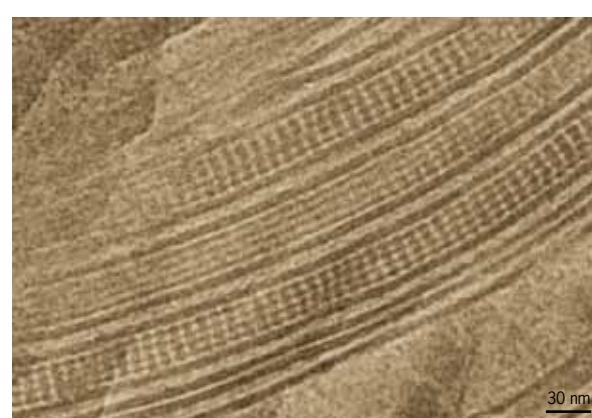
gives an excellent set of composite properties for industries such as automobile-body manufacturing, where high formability is vital for the complicated aerodynamic design of modern cars and high strength is essential to withstand the impact of shocks. The low weight and high-temperature stability of this material are also attractive for structural- and space-engineering applications.

## CATCHING THE LIGHT

### FLAGSHIP

Cyanobacteria are photosynthetic bacteria that, like plants, harvest light energy to survive. One species of cyanobacterium, *Acaryochloris marina* is especially intriguing, because it lives as a biofilm beneath sea squirts on the Great Barrier Reef. This unique and curious habitat places certain constraints on how these bacteria capture the light they need. Only light that has passed through the sea squirt is available and this has led to the evolution of a different type of chlorophyll (chlorophyll d) in these bacteria. So far this is the only organism in which this version of chlorophyll has been found. Organisms that use chlorophyll also rely on an additional complex of proteins called phycobiliproteins as minor pigments to capture additional wavelengths of visible light and transfer their energy on to the chlorophyll.

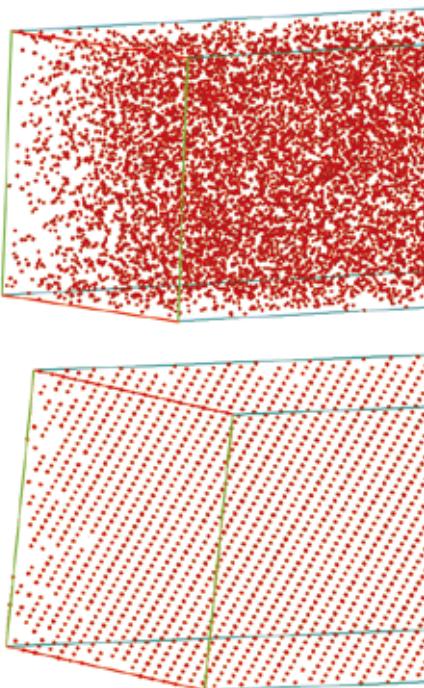
Dr Min Chen from the University of Sydney and Dr Tom Bibby, a visiting fellow from the



The near-crystalline stacks of the phycobiliprotein rods are visible filling the space between the photosynthetic membranes.

University of Southampton, have investigated the structure and supermolecular organisation of the phycobiliprotein complexes in *Acaryochloris marina* by using cryo-electron microscopy of vitreous sections at the AMMRF at the University of Queensland. The cells were frozen extremely quickly to form vitreous ice, preserving cellular complexes in close-to-natural conditions. In this way, Dr Chen found a novel and extremely simple rod

structure of the phycobiliprotein complexes. This is in strong contrast to the complicated and highly organised arrangement typically seen in other species of cyanobacteria. The energy transfer from these simplified proteins to the chlorophyll was shown to be very efficient and fast, a factor presumably necessary for the bacteria to survive in the low-light conditions of their unusual home.



The top panel shows a small sub-volume of an APT reconstruction of pure aluminium where there is no visual evidence of a crystalline structure. Below is the same volume after lattice rectification. The lattice structure has become readily apparent.

## CRYSTAL CORRECTION

### FLAGSHIP

Atom probe tomography (APT) provides highly accurate chemical identity and 3-D positional information at the atomic level. In practice, however, the spatial resolution of current atom probe instruments falls short of the ideal of characterising and exactly locating every atom within a sample. Atoms in the APT reconstruction are offset slightly from their true positions and further, due to instrumental limitations, a fraction of the ions striking the detector are not registered, meaning that some atoms bypass detection altogether. This results in structural and crystallographic characterisation of the specimen being incomplete.

Dr Michael Moody from the AMMRF at the University of Sydney is pioneering novel techniques for recovering missing structural information from APT datasets. The approach uses spatial distribution maps of atoms within the dataset, similar to radial distribution functions, to map out local atomic neighbour-

hoods and recover the limited structural information already existing in APT data across multiple crystallographic directions. Characterisation of the inherent crystal lattice in the reconstruction enables the researchers to pinpoint, in real space, each atom's most likely true lattice site. This technique then systematically re-places every atom in the data back onto the perfect lattice, while maintaining the integrity of the local atomic distributions, as depicted in the figure.

Restoration of the lattice in the APT reconstructions will enable the detailed investigations of lattice-rectified data, with unrivalled 3-D spatial resolution, taking characterisation of nanoscale features to new levels of precision. Furthermore, it will enable researchers for the first time to input atom probe data directly into atomic-scale simulations of mechanical properties. The insight gained through these techniques has exciting implications for our understanding of nanostructure and its relationship to material properties and performance.

## ALGAE – FULL OF SURPRISES

Coral-reef ecosystems are globally distributed between latitudes 30°N and 30°S, and form thousands of square kilometres of marine habitat that is vital for community livelihood, tourism, employment and coastal protection. Reef-building scleractinian corals (phylum Cnidaria, class Anthozoa) are the major component of these systems. They have the capacity to grow rapidly due to their symbiotic union with dinoflagellate algae (*zooxanthellae*). This association makes possible the formation of coral reefs in low-nutrient tropical seas.

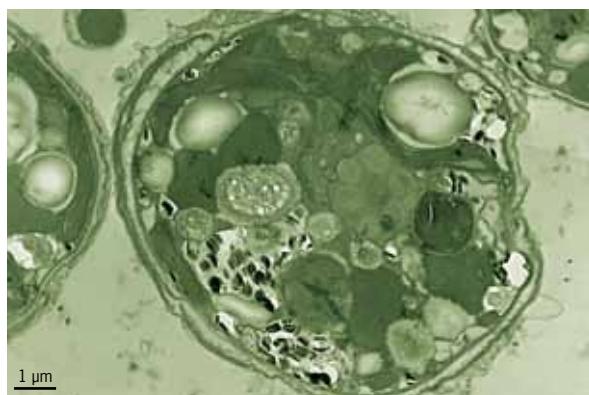
Global climatic change is now believed to have serious consequences for the survival of coral reefs, with the prediction of substantially increased coral death due to rising ocean temperatures and subsequent disruption of the symbiotic relationship between the corals and the algae (bleaching). Despite the apparent importance of this symbiosis, its physiological nature is complex, and little is understood at the cellular or molecular level.

Recent work led by A/Prof. Peta Clode in the AMMRF at the University of Western

Australia, in collaboration with others at UWA and Murdoch University, has resulted in the discovery of uric acid deposits in the symbiotic marine algae of cnidaria. The team investigated the nature of crystalline material prominent within marine algal symbionts of anemones by using a variety of microscopical and analytical methods, including transmission electron microscopy (TEM) imaging and diffraction, energy-filtered TEM, electron energy-loss spectroscopy and gas chromatography mass spectrometry. The deposits, which historically have been considered to be calcium oxalate, based on histochemistry data from the mid-twentieth

century, were shown by modern methods to be, actually, uric acid.

This work is the first report of uric-acid accumulation by symbiotic marine algae and begins to provide much-needed insight into the molecular physiology of cnidarian-algal symbioses. It reveals a new and previously unconsidered aspect of nitrogen metabolism in these systems. Work is continuing into the source and metabolism of these uric acid deposits, and how they are affected by climatic changes. This information could contribute to our understanding of how we can protect our valuable reefs.

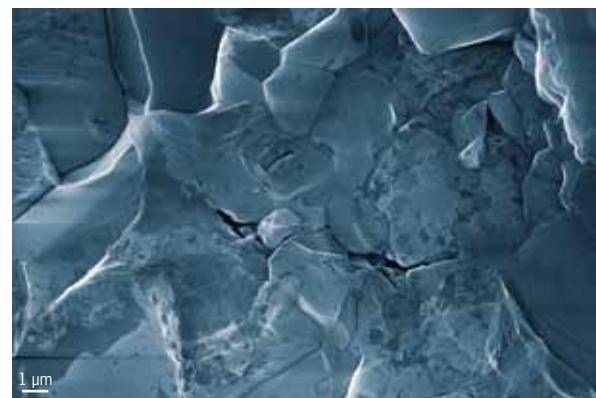


Algal cell showing the uric-acid crystals as small dark particles in the lower left of the cell.

## MICRO-PUMPS – MASSIVE MOVEMENTS

How can fluid move great distances through rocks deep in the earth's crust without some mechanism to generate very high pressures to force it along? Clearly fluids do move, but until now no good explanation had been found for how it happens. The geology community acknowledges that there is a clear interaction and feedback between fluid movement and the deformation of rocks in the mid-crust, and that this is a critical element for the initiation of earthquakes and subduction zones, and for the formation of mineral deposits.

Research by Dr Florian Fusseis with Prof. Klaus Regenauer-Lieb from the University of Western Australia, along with Dr Rob Hough from CSIRO Exploration & Mining, has found compelling evidence that demonstrates for the first time how fluids can migrate through rock in the middle of the Earth's crust where shearing movements deform the rocks. They focused on rock found near Alice Springs that had been deformed in the depths of the crust 320 million years ago. Data was col-



FESEM image of nanoscale-pore development in the rock sample.

lected from field-emission scanning electron microscopy at the AMMRF at the University of Western Australia and from high-resolution synchrotron X-ray tomography. The team could then visualise the nature of the pores and propose their model for fluid movements, which they called the 'granular fluid pump'. It describes how, when the rock is deformed, the strain opens up tiny pores between the grains within the rock and, as the deformation continues, fluid is slowly squeezed from one microscopic pore to the next. This effectively generates a small-scale pumping mechanism that can account for

large-scale fluid movement over considerable distances of the mid-crust without the need for high fluid pressures.

By explaining the mechanisms of fluid transfer in the middle crust, this work adds an important piece in the puzzle of fluid-rock interactions during the earthquake cycle and the migration of mineralised fluids from sources deep within the earth.

This work was published in *Nature* in June 2009.

## CONDUCTING DNA

Using individual molecules as electronic components is a challenging new research direction in science, and one that could have application in the fabrication of nanoelectronic devices and sensors. DNA is one of the molecules in which there is considerable interest for this purpose and understanding the nature of electrical transport along the double helix will be essential in exploring the possible use of DNA in molecular electronics and in the direct detection of DNA as a quantitative analytical tool. Furthermore, the techniques now available make it possible to use this fascinating material and its unique molecular recognition properties to design and create novel structures made from DNA. It is well known that directed assembly of a DNA double helix is controllable via the highly specific pairing of A-T and G-C bases along its length, the mechanism that gives DNA its ability to be accurately copied in biological systems.

Dr Wenrong Yang and colleagues from the AMMRF at the University of Sydney have

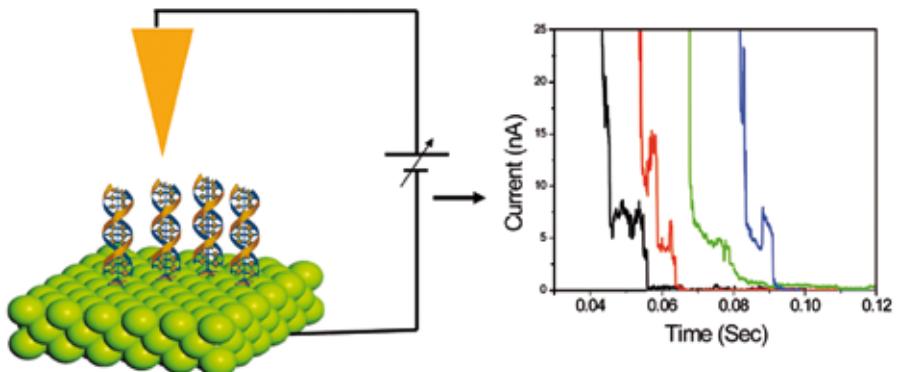


Illustration of the arrangement of single DNA molecules for STM alongside conductivity measurements of different sequences.

been examining the electrical conductivity of DNA molecules of differing base compositions. He has done this by using scanning tunneling microscopy (STM) of single DNA molecules strongly attached to a gold surface via thiol linkages. STM is being used widely as a tool in the rapid measurement of single-molecule conductance and the results Dr Yang has obtained show that the base composition of the DNA molecules substantially affects conductance. DNA chains made

up of G-C pairs are far more conductive than those made up of A-T pairs, and even a single A-T pair in the middle of a G-C chain markedly reduces the conductance. A wrongly matched base pair in the chain (such as A-G) reduces conductivity even more. This work supports the proposal that DNA can be used in the design of nano-electronic components. It also raises the possibility of the direct detection of mutations in DNA by measuring changes in molecular conductance.

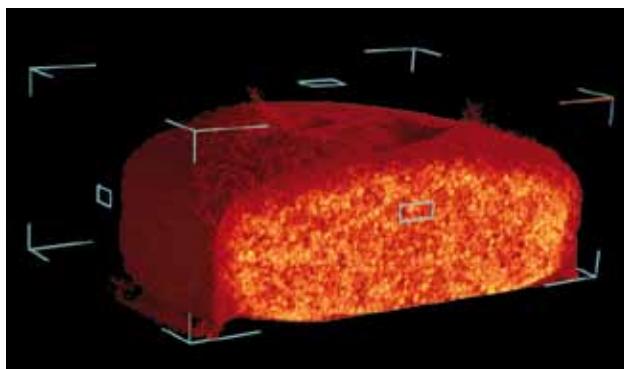
## KEEPING AN X-RAY EYE ON DRUG DELIVERY

Finding mechanisms to enhance the efficient delivery of drugs is a major area of research in pharmaceutical science. Tablets are often taken for granted, but the way they release their drug cargos inside the body is highly relevant to their therapeutic outcome.

The matrix-based tablet is the simplest form of controlled-release tablet available, with the matrix providing a barrier to the diffusion of the drug out of the tablet. For matrix-based materials, production and manufacturing variables can lead to variations in performance that could cause quality-control failure. The porosity of the matrix is therefore key to the effective regulation of drug delivery. Having a technique that could visualise the internal structure of such materials post-manufacture, and being able to focus on the dynamic property of porosity during dissolution and diffusion, would be highly advantageous.

Dr Paul Young and Dr Daniela Traini and colleagues from the University of Sydney

*3-D volume reconstruction of the matrix tablet taken 60 minutes into the dissolution process. The iron sulphate is shown in orange.*



have been working with the AMMRF at that university to evaluate drug release from tablets and consequently their performance as drug-delivery systems. Micro-CT was used as a non-destructive imaging technique to study the diffusion and dissolution process that occurs during drug release. Inert-matrix tablets were used containing ferrous sulphate as the active ingredient, distributed in a matrix of lactose with a protective enteric coating. The research team developed a system for dissolution tests that enabled periodic micro-CT analysis to be done on

the tablets during drug release over a period of 24 hours. The results revealed that the decreasing amounts of the iron sulphate within the tablet could be easily visualised by micro-CT.

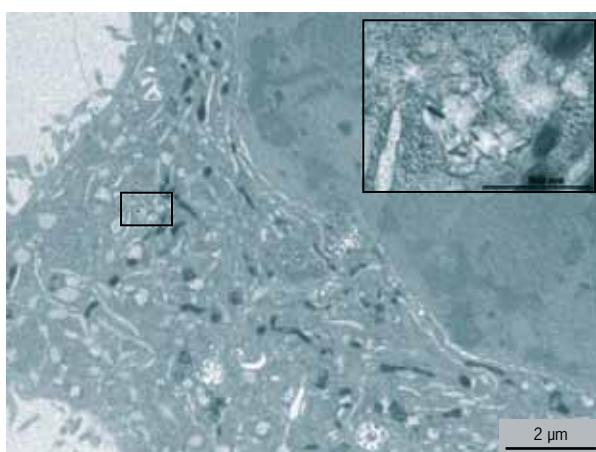
The team have shown micro-CT to be a sensitive technique with which to evaluate the rate of drug release from matrix-based tablets and potentially from other solid dosage systems, instead of relying on the classical theoretical or 'destructive' investigations.

## NANO-CLAYS DELIVERING BENEFITS

### FLAGSHIP

The potential of nanoparticles in industrial and medical products is enormous, but it is increasingly recognised that the interactions of nanoparticles with biological systems must be examined in detail, as they behave significantly differently from their corresponding bulk forms. This will provide a clearer understanding of the possible adverse consequences, particularly on health and the environment, of nanoparticle inclusion into products we will all use or become exposed to.

Layered double hydroxides (LDHs) are one such nanoscale material. They are layered, clay-based nanoparticles, traditionally used in industrial applications such as flame-retardants and polymer stabilisers. Positively charged clay layers are counterbalanced by exchangeable, negatively charged ions or molecules. Due to this characteristic, LDHs have been recognised as potential controlled-release nano-carriers, capable of delivering biomolecules such as drugs and genes to cells.

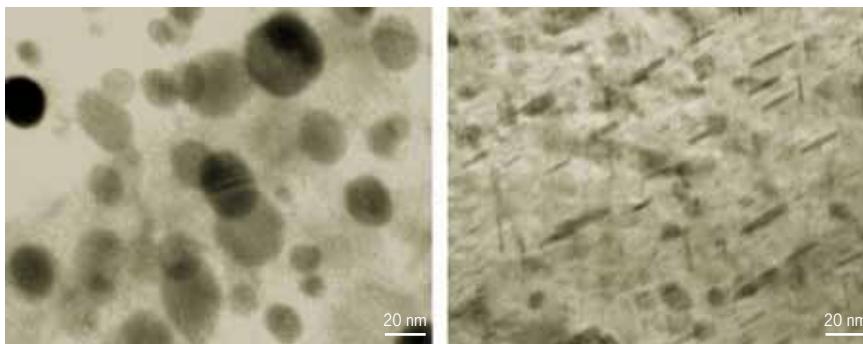


*TEM image of HeLa cells incubated with LDH particles. Inset: particles localised to vesicle cavities within cells.*

Dr Margaret Butler and colleagues at the University of Queensland (UQ) have begun to thoroughly investigate the biophysicochemical interactions of LDHs with human cells. By using a novel combination of methods, they synthesised LDH nanoparticles of a uniform and defined size that did not clump together (a common problem with nanoparticles). The particles were labelled with stable and highly fluorescent dye molecules in the interlayer cavity, which had negligible effect on the LDHs physical and chemical characteristics.

Transmission electron microscopy (TEM), carried out at the AMMRF at UQ, revealed the LDH particles had entered the cells and had become localised in discrete vesicles within them. As the first of a larger set of nanotoxicology experiments, this experiment has demonstrated the potential utility of these tailored LDH nanoparticles for delivering molecules into human cells. Further experiments will shed light on just how the nanoparticles affect different aspects of the cells' behaviour.

## A TALE OF TWO STRUCTURES



TEM micrographs of the aluminium alloy processed by SPD (left) and by conventional aging (right).

Altering the nanostructures of metals can give rise to improved mechanical properties ideal for a range of applications. Severe plastic deformation (SPD), a process where the metal is subjected to squeezing and shearing forces, is known to be effective in reducing grain size to produce ultrafine-grained and nano-crystalline materials. These materials offer unique properties superior to those of their coarser-grained parent materials. As a result, SPD has become a very hot research area. Most research activities on SPD have focused on exploring the grain-refinement effects. However, there is a lack of research on how SPD affects precipitation of elements in the alloy and the development of precipitate microstructure. Manipulation of precipitates is a potential alternative route to further enhancing properties.

Dr Gang Sha and colleagues at the

AMMRF at the University of Sydney have used transmission electron microscopy (TEM) to investigate two different light alloys to see how each responds to SPD with respect to strength and other properties. During SPD on a 7000-series aluminium alloy at 200°C, changes occurred in the rotation of precipitates, and in the atomic structure of the interfaces between the precipitates and the matrix. As a result, the precipitates adopted uniform growth, evolving into spherical particles, rather than the flat plate-like shapes obtained by conventional heat ('ageing') treatment. This change in the precipitate microstructure, reduced the strength of the SPD-processed alloy, contrary to initial expectations and hopes.

The same technique, when applied to a magnesium-rare earth alloy, produced a different outcome. Novel precipitate micro-

structures were also produced in this material when processed by SPD; however, they formed more slowly but more frequently, resulting in a microstructure that contained a high density of small cubic precipitates that remained stable at temperatures above 250°C. This produced a better strengthening effect with a hardness approximately 19% greater than that obtained by conventional ageing treatment. Thus SPD is an attractive mechanism for improving the strength of this alloy through the control of precipitate microstructure, making this a particularly suitable modified alloy for high temperature applications.

It is also evident that the behaviour of materials is not always as expected and that an experimental approach is essential to evaluate responses to processing.

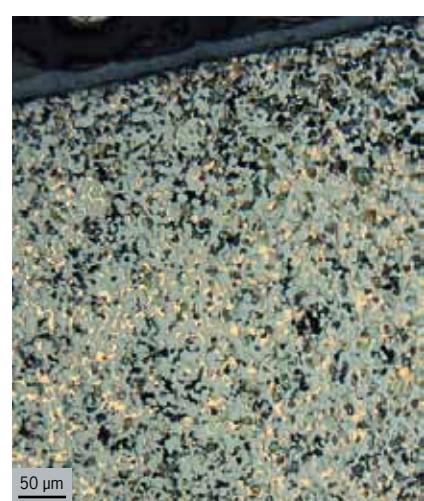
## EVAPORATIVE COOLING GETS HOT

The production and characterisation of materials for extreme environments, where temperatures can reach more than 3000°C, is quite a challenge, but one that is important for a number of uses, from high-temperature fuel cells to hypersonic vehicles. By making use of current knowledge of structure–property relationships and the effects of microstructure on macroscopic properties, these materials can be tailored to specific tasks. Dr Anna Lashtabeg from the University of Queensland (UQ) is investigating solutions to these challenges, and in particular the development of intelligent coating solutions. One approach is to produce replenishable ablative coatings. These involve graded layers of two different

materials, one of which evaporates out of the coating at the appropriate temperatures, providing a constant cooling effect. In this case the sacrificial component is copper dispersed in a tungsten matrix.

To investigate the microstructure of these coatings, Dr Lashtabeg has carried out scanning electron and optical microscopy at the AMMRF at UQ. The image shows the microstructure of a heat-treated copper-tungsten alloy in which the presence of pores indicates the evaporation of copper near the surface due to the extreme heat.

Successful development of these coatings will pave the way for hypersonic flight vehicles, new energy solutions and defence applications.



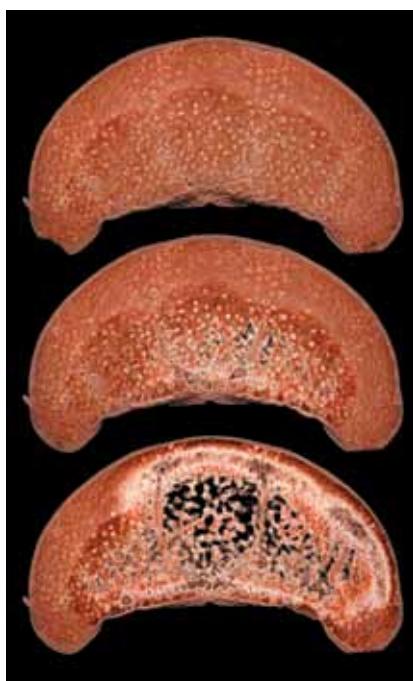
Optical micrograph of a copper-tungsten alloy after a 10 second heat treatment at more than 3000°C. The black spaces are the pores left behind after some of the copper (yellow) has evaporated.

## BEYOND THE BONES

The emergence of fish from the ancient seas, some 350 million years ago, was a defining period for all tetrapods (animals with four limbs). Limbs and the ability to breath air were not the only requirements to embark on a life on land, and other characteristics had to evolve to make the transition successful. Understanding how this occurred is vital to an in-depth knowledge of evolutionary processes.

Prof. Ken Campbell, Dr Dick Barwick and Dr Gavin Young from the Australian National University (ANU) have been examining some amazingly well-preserved, fossilised lungfish of the genus *Chirodipterus*, which swam in the ancient oceans during the Devonian period, around 380 million years ago. The fossils were found in rocks in the Kimberley region of northern Western Australia. Near-perfect preservation of the fine skeletal features in these fish fossils provided the researchers

with an excellent chance to evaluate the evolutionary stages of the nerves in the head and face along with their associated sensory systems. Micro-CT at ANU used X-rays to look inside the fossils without the need for destructive sample preparation. Successive slices were generated through the snout of the extinct lungfish, revealing hidden secrets buried deep within the bone. It was possible to trace two sets of channels that would have carried sensory nerves from the snout back into the brain of this transitional creature. One pair of these nerve tracts can be seen as a 'U' shape in the image and would have been part of the lateral-line system familiar in modern fishes as a dark line running down theirs flanks, but which also extends around the head and face, providing a way for the fish to sense movements through the water. These insights into the soft-tissue anatomy of ancient creatures, gleaned purely from fossilised bones, is a major step forward in the understanding of our evolutionary past.



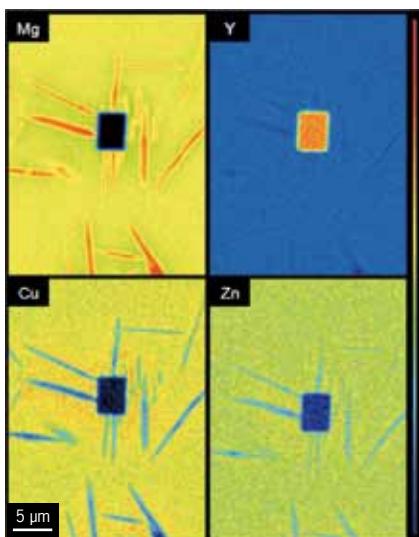
Micro-CT reconstruction revealing structures within the bone.

## METALLIC GLASS GAINING STRENGTH

## FLAGSHIP

Bulk metallic glass (BMG) is an exciting new type of metal. It does not have the usual crystalline lattice structure found in metals, but rather is amorphous, like glass. This highly unusual structure gives BMG great strength, but it is incredibly difficult to achieve. Molten metal must be cooled extremely rapidly to prevent the formation of crystals. More recently, however, the discovery that the introduction of crystalline components within the amorphous metal matrix can produce materials with still better mechanical properties has led to a new line of research. Although the importance of BMG composites is now widely recognised, little is known about the process by which the crystalline phases within them are formed and distributed.

Researchers at the University of New South Wales (UNSW), led by Dr Kevin Laws and Prof. Michael Ferry, are addressing these issues and have been studying the fabrication and characterisation of magnesium (Mg)-based BMG composites that retain their strength while maintaining a high degree of plasticity. These composites have outstand-



Intensity maps of Mg, Cu, Zn and Y peaks across cubic and flake crystalline features in amorphous BMG matrix. Colour gradient at right indicates scale of low (black/blue) to high (red) relative peak intensities for each element.

ing potential utility across a wide range of materials applications, for example protective scratch-resistant coatings for electronic components, and sports equipment such as golf clubs.

The team are investigating the distribution of elements in various crystalline features and in the amorphous matrix of certain Mg-based BMG composites, by using electron microprobe analysis (EPMA) in the AMMRF at UNSW. This research complements parallel

work on the 3-D reconstruction of crystalline features within the BMG matrix, which has been conducted with focused ion beam milling.

The EPMA investigations identified a variety of yttrium-rich, high-melting-point features that appear to serve as nucleation points for Mg-rich flakes, which are found throughout the amorphous matrix. EPMA mapping has also revealed an inversely proportional concentration gradient of Mg and Cu within the BMG matrix in the region of flake features. Further research into the relative concentrations of these elements within the crystalline features, and the compositional changes in the matrix surrounding them is ongoing, and will reveal more information on the processes responsible for the superior mechanical properties observed in such materials.

## CLEVER CERAMICS

Digital consumer electronics, computing power and data-storage capacities all rely on the ability to respond to applied electric and magnetic fields at fine scales. In general, the response to an applied electric field is independent of the response to an applied magnetic field. In a multi-ferroic material, however, these response properties are coupled. In principle, this will allow the further miniaturisation of more powerful devices that consume less power. Developing such materials is at the forefront of condensed-matter science. Multi-ferroic materials of this type have numerous potential applications including multiple-state memory elements, magnetic data storage and spintronic devices.

A team at the Australian National University (ANU) led by Prof. Ray Withers and Dr Yun Liu, working with colleagues at Monash University, has used a rapid liquid-phase sintering technique to produce a series of compositions in the  $\text{Bi}_{1-x}\text{Ca}_x\text{Fe}^{\text{III}}\text{O}_{3-x/2}$  system. These so-called BCFO compounds and related materials open up the possibility of electric-field control of magnetic properties and vice-versa, and so are of considerable



Electron-diffraction patterns collected from multi-ferroic BCFO compounds of different elemental compositions. They show subtle differences arising from the 4-D modulation of the basic crystal structure.

scientific interest; however, their structures and the origins of their properties are little understood. Consequently, the team has thoroughly characterised the structures of these BCFO compositions by means of X-ray and electron diffraction, and scanning and transmission electron microscopy, and then correlated these details with certain physical properties.

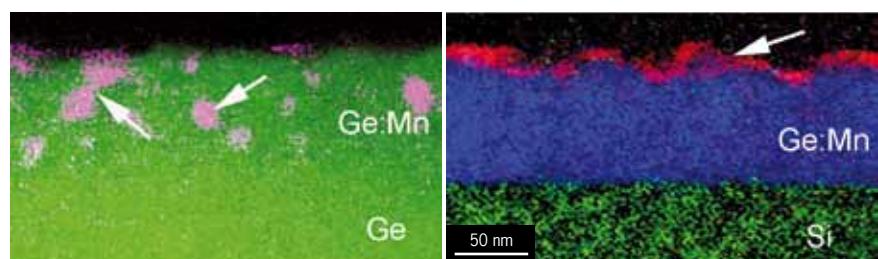
The electron diffraction patterns of the various structures were collected at the AMMRF at ANU and carry a wealth of crystal chemical information that can be related to their functionality. The team found that a complex, perovskite-related crystal structure of the ceramic,  $\text{Bi}_{1-x}\text{Ca}_x\text{Fe}^{\text{III}}\text{O}_{3-x/2}$  exists where  $x$  in the chemical formula can

be anywhere between 0.19 and 0.49. This extremely large range of compositional variation cannot be accommodated by the usual repeating crystal structure but needs to take up more unusual and defective structures. This means it cannot be described in three dimensions, but requires four- or higher-dimensional crystallography to describe its features. The diffraction patterns shown in the image above reveal the subtle differences in structure between crystals at different compositions. The conceptually difficult ideas surrounding the structure of these materials lie at the heart of BCFO's multi-ferroic abilities and this work is fundamental to their future application.

## BRINGING CHARGE AND SPIN TOGETHER

In conventional semiconductor and information technology, conventional integrated circuits made of semiconductors only use the charge of electrons. On the other hand, the mass storage of information in hard disks, magnetic tapes and magneto-optical discs uses only the spin of electrons in ferromagnetic materials. More sophisticated materials known as diluted magnetic semiconductors (DMS) harness both the charge and the spin of electrons in one material. Generally speaking, DMS are semiconductors such as silicon (Si) and germanium (Ge), in which a small fraction of the atoms have been replaced by magnetic atoms such as manganese (Mn). Mn-doped Ge is a key DMS due to its compatibility with current Si-based microelectronics.

Prof. Jin Zou's group from the University of Queensland (UQ), in collaboration with Prof.



Elemental maps of the Mn-doped Ge films grown on Ge (left) and on Si (right). The arrows indicate the Mn-rich regions.

Kang Wang and his team at the University of California, Los Angeles, grew Mn-doped Ge thin films on Ge or Si substrates and investigated their structural and chemical characteristics with advanced electron microscopy in the AMMRF at the UQ. They found different Mn behaviours in the Mn-doped Ge thin films depending on the substrates. When grown on Ge, the Mn forms particles distributed throughout the film; when grown under identical conditions on Si, Mn forms a layer on top of the film. These differences can be seen

in the images. The study also suggested that the substrates and the thickness of the films play a key role in the formation of the Mn-rich nanostructures, which, in turn, affect the magnetic properties of the thin films. The significance of these results is not yet clear, but they are forming the basis of further work to understand DMS properties in much greater detail and allow the development of the most efficient thin films.

## HEART PROTEINS TO THE RESCUE

Congenital muscle-wasting diseases are currently incurable and are therefore a major focus of research around the world. Muscles are largely made up of two proteins that work together, pulling past each other to make the muscles contract. One of these proteins, actin, comes in several different sub-types. Skeletal-muscle  $\alpha$ -actin acts in our skeletal muscles and cardiac-muscle  $\alpha$ -actin acts in the heart muscle. Mutations in skeletal-muscle  $\alpha$ -actin might therefore be expected to cause severe muscle disease, and this is indeed the case. Congenital myopathies are muscle diseases that tend to be present at birth. The most severely affected babies can be almost totally paralysed at birth. Interestingly, before birth we all express cardiac  $\alpha$ -actin in our skeletal muscles before the skeletal-muscle  $\alpha$ -actin gets switched on. On the basis of these observations, Prof. Nigel Laing and his team at the Western Australian Institute for Medical Research, along with collaborators from Oxford, Cincinnati and



TEM showing the normal ultrastructure of skeletal muscle containing cardiac  $\alpha$ -actin, instead of skeletal muscle  $\alpha$ -actin.

other centres around the world, thought that it might be possible to use cardiac actin to treat skeletal-muscle  $\alpha$ -actin diseases.

The first step to investigating this possibility was to show that cardiac actin can actually work properly in postnatal skeletal muscle by using an animal model. This is exactly what the team have now shown. Working with two different lines of transgenic mice, the team interbred them in such a way as to generate mice that had their skeletal-muscle actin completely replaced with cardiac actin. Normally, the mice with no skeletal actin die within just nine days of birth. Incredibly, the mice with the replacement cardiac actin

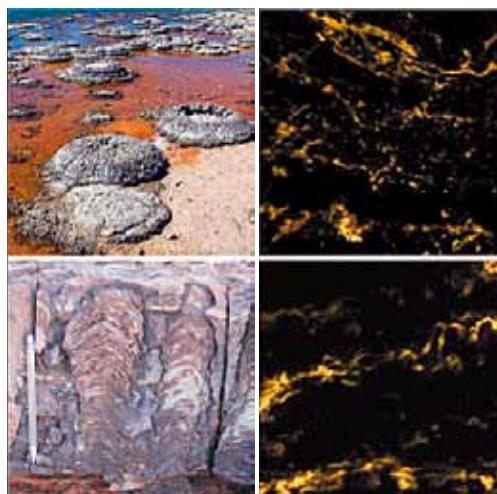
not only made it to adulthood, but survived healthily into old age. These ‘rescued’ mice also functioned just as well as normal mice in behavioural tests and transmission electron microscopy (TEM) of their skeletal muscle, done in the AMMRF at the University of Western Australia, shows apparently normal muscle. This exciting result proves that cardiac actin is definitely worth pursuing as a target for treating skeletal-muscle  $\alpha$ -actin diseases. The aim is to find a way to turn on the cardiac-actin gene in the skeletal muscle of patients, allowing it to take over from the damaged protein.

## LIFE ON MARS? TRACKING THE TRACES OF LIFE

## FLAGSHIP

Identifying signs of life on other planets has been a major scientific goal for a number of years and, as more missions are heading to Mars, increasingly diverse investigations can be undertaken. Essential to a meaningful interpretation of findings is the ability to differentiate between what is life and what isn’t. To do this, we need to turn to our attention to the origin and evolution of the earliest life here on Earth.

Stromatolites are rare bio-sedimentary structures found in several Western Australian lakes. They are thought to be analogous to some of the earliest forms of life on Earth. Modern stromatolites are formed by the interaction of biological, chemical and physical processes. However, it is very difficult to prove that biology had any role to play in ancient examples because obvious signs of life, in the form of microfossils, are rarely preserved. Hence, if stromatolites were to be found on Mars, how can we be sure that they are a signal of life?



Top row: modern stromatolite from Lake Thetis, Western Australia. NanoSIMS ion image of nitrogen picks out laminations within the stromatolite.  
Bottom row: 2.7-billion-year-old stromatolite from the Tumbiana Formation of Western Australia. Once again the NanoSIMS ion image of nitrogen correlates with laminations within the stromatolite.

Dr David Wacey and his colleagues at the University of Western Australia have been studying the biochemical signals associated with stromatolites of different ages to see if these provide a more reliable sign of life than the presence of microfossil remains. They have used the AMMRF flagship NanoSIMS instrument at the University of Western Australia to map biologically important elements within a modern stromatolite and within a 2.7-billion-year-old stromatolite from West-

ern Australia. Very similar patterns emerge from each of these stromatolites with co-occurrence of essential biological elements – carbon, nitrogen and sulphur – preserved along laminations in both stromatolites. This suggests that biochemical signals can be preserved even when microfossils have long since decayed.

These concepts are explored further in Dr Wacey’s book *Early Life on Earth: A Practical Guide*, available from Springer.com.

## UNRAVELLING ANCIENT TECHNOLOGY

In the early first millennium BCE, Gordion was the capital of ancient Phrygia in central Anatolia (modern Turkey). It was the seat of King Midas of the legendary golden touch, and where Alexander the Great would later cut the Gordian knot. Most importantly, Gordion lay on the main east-west trade routes that connected the Mediterranean with the Middle East.

Dr Wendy Reade from the University of Sydney and Prof. Janet Jones from Bucknell University, Pennsylvania, are interested in exploring the technological relationships between Gordion and the wider region through the physical and chemical study of ancient glass. Glass-making traditions of the first millennium BCE have been little investigated and are poorly understood.

Phrygia was one of the first civilisations in Anatolia known to have used glass as early as the ninth century BCE (Iron Age).



Fragment of a Gordion glass beaker.

After a gap of several hundred years, glass appears again in the Hellenistic period, from the fourth to second century BCE. Were the Phrygian master metalworkers also glass-makers? Was the later Hellenistic glass production a revival of an earlier industry, or the evidence of innovation? How did the Gordion glass compare with glasses from the Middle East, and with those of the Greek world to the west?

Glasses from Gordion during both periods have been analysed by electron probe microanalysis (EPMA) in the AMMRF at the University of New South Wales. They were typical of the earliest glasses, showing soda-lime-silica compositions with distinctive levels of alumina, iron, potash and magnesia to which modifiers such as colourants and opacifiers were added. The initial combination of EPMA results and physical archaeological evidence suggests that Gordion imported raw glass from the east and probably worked it locally. While it is unlikely that glass was made at Gordion itself, the production location may be revealed by future research.

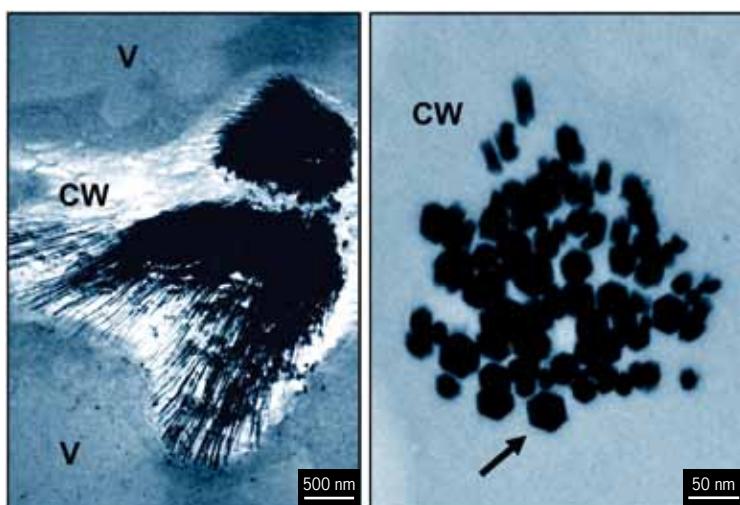
## COPING WITH CONTAMINATION

The cost of managing areas of contaminated land and groundwater and of dealing with contaminated waste in Australia is estimated to be \$5–8 billion each year. Lead (Pb) is among the worst 3% of pollutants based on risk to humans, risk to the environment and the extent of contamination in Australia. Although the identification of the types of Pb compounds within plants is crucial in understanding how they can tolerate Pb, little is known about its uptake, location,

or the chemical forms in which it is found within plants. Neither is it known how some plants manage to tolerate elevated Pb levels in their environment.

By using two grasses that differ in their tolerance to Pb, Dr Peter Kopittke from the University of Queensland has employed transmission electron microscopy (TEM) with energy-dispersive X-ray spectroscopy (EDS) to determine the distribution and types of Pb minerals found within the plants. In both grasses, Pb was initially present mainly in the cytoplasm of root cells before being taken

up into vacuoles, specialised liquid-filled sacs within plant cells) as the highly insoluble (and presumably non-toxic, chloropyromorphite ( $Pb_5(PO_4)_3Cl$ ). In the tolerant grass, Pb also accumulated within certain membranous structures, prior to being deposited in the cell walls as chloropyromorphite. By using TEM and EDS the researchers were able to identify that the nanoscale, lead-containing crystals had the distinctive hexagonal shape of chloropyromorphite as well as its telltale elemental profile. These findings suggest that the ability of this tolerant grass to tie up insoluble Pb in the cell wall is an additional and potentially important mechanism of Pb-tolerance not possessed by the Pb-sensitive grass. Other types of Pb-tolerance can occur if the plant prevents Pb from being taken up at all. This work could have important implications for distinguishing the two types of tolerant plants and therefore in preventing Pb from entering the food chain.

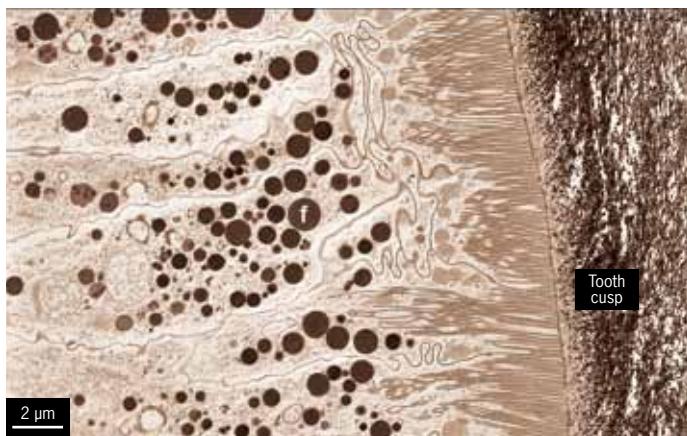


Transmission electron micrographs of transverse sections taken from the root of a Pb-tolerant grass after 14 days' growth in Pb-containing nutrient solutions. (V = vacuole, CW = cell wall).

## IRON-TOOTHED WONDER

Throughout history, researchers have looked to nature for the design of new devices, materials and medicines. The field of biomimetics specifically draws on the wonderfully diverse and complex range of structures present in nature, which have been honed to perfection over millions of years by the process of evolution. Such structures are often stronger, harder and stiffer than their synthetic analogues, and may possess unique magnetic, electronic or optical properties. Dr Jeremy Shaw and colleagues at the AMMRF at the University of Western Australia, Prof. David Macey from Murdoch University and Dr Lesley Brooker from the University of the Sunshine Coast have been studying biominerisation in chiton teeth, a unique model that may lead to the development of future materials and devices based on biomimetic principles.

Chitons are marine molluscs that use their iron-clad teeth to scrape algae from intertidal rocks. The design of the teeth is astoundingly complex, with magnetite (the hardest and most magnetic form of iron oxide) and calcium-based minerals deposited onto an organic scaffold within structurally discrete regions of the teeth. By studying the



Iron-loaded epithelial cells delivering elements to the teeth during early stages of the mineralisation process.

minerals and the organic microstructure of these steel-hard teeth, it is hoped that the resulting insights will advance our ability to develop tailor-made nanoscale materials.

Over the past year, significant advances have been made in our understanding of this novel biominerisation system. By using a combination of analytical transmission electron microscopy and scanning electron microscopy techniques the team has demonstrated that iron is transported into the teeth from the surrounding cellular tissue and from a reservoir at the base of the tooth. Dr Shaw's work in demonstrating

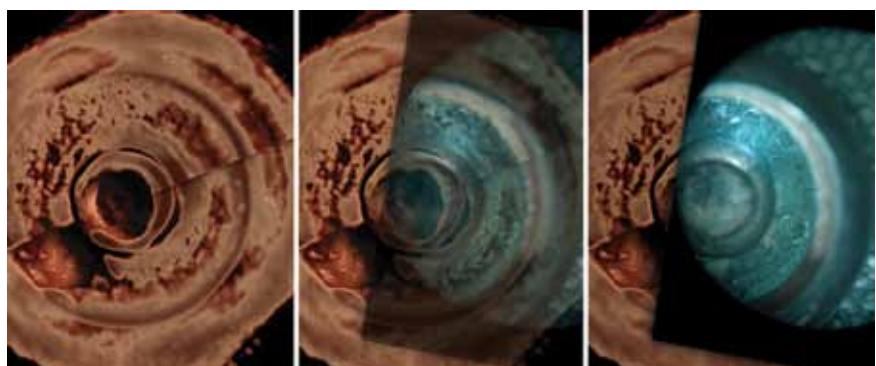
this double-front approach to tooth mineralisation resulted in him winning the inaugural Robert P. Apkarian Memorial Scholarship at America's premier microscopy conference.

 This research was featured in the Higher Education section of *The Australian* and the Channel 10 News.

## PRESS-STUD REVEALS ITS SECRETS

In 1941 the *HMAS Sydney* was sunk by a disguised German raider off the coast of Western Australia. There were no survivors from the tragedy and location of the wreck was not known. In 1942 when the body of an unknown sailor was washed ashore on Christmas Island in a Carley float, it was not known whether he could have been from the *HMAS Sydney*, partly due to the unknown location of the vessel. All that remained of his clothing were four heavily corroded press-studs.

When the wreck was discovered in 2008, a commission of inquiry was set up and some relics were analysed. As part of the investigations, the AMMRF at the Australian National University (ANU) carried out micro-CT analysis of the heavily corroded press-studs from the unknown Christmas Island sailor. The



Micro-CT image of the corroded press-stud, overlaid with an uncorroded stud from the same period, to highlight the writing that was still present on the corroded stud.

technique allowed the team to see below the corroded surface, revealing markings that indicated that the studs had been made by Carr Australia, a company that supplied the navy as well as many other clients. This, along with the prevailing winds and tides that could have led to the body being washed up

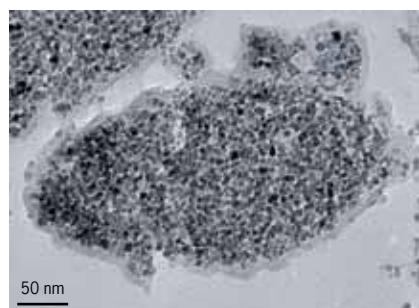
on Christmas Island, strongly suggested that the unknown sailor was a serviceman from the stricken ship. He has now been given a memorial service and laid to rest in the Geraldton War Cemetery, providing an important focus for many of the families of the sailors who were lost with the *HMAS Sydney*.

## MAGNETIC CAPSULES FOR TARGETED THERAPY

Targeted drug delivery is a focus of much research as it offers the advantage of safe and effective delivery of desirable dosages of drugs to specific sites in the body without adverse side effects. One strategy for targeting drug delivery is to use an external magnetic field to guide magnetically labeled drug carriers. For this to work, a drug or therapeutic molecule is bound to a magnetic material, and then the composite particles are introduced into the body, and concentrated in the target area by means of a magnetic field. The ability to concentrate drug carriers to desired cells or organs by an external magnetic field is an attractive option because magnetic forces act at relatively long range and magnetic fields do not adversely affect most biological tissues. The particles can be removed after treatment, thereby reducing toxic side effects.

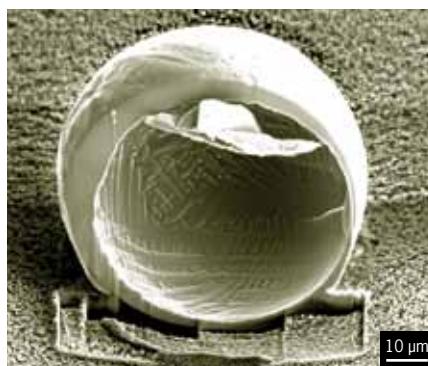
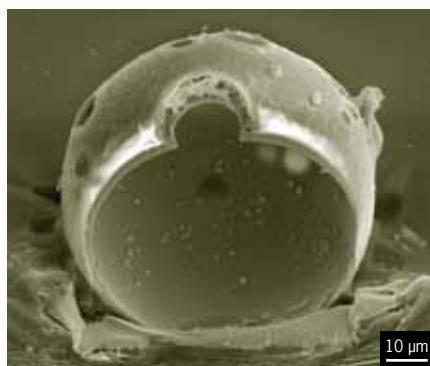
Dr Swaminathan Iyer and Prof. Colin Raston at the University of Western Australia (UWA) have developed a method to load a potentially therapeutic compound, curcumin, into porous magnetic silica capsules through a simple multi-step self-assembly approach. Curcumin is a polyphenolic yellow pigment found in turmeric, which is commonly used as a spice and food-colouring agent, but has also been used in traditional Indian and Chinese medicine. It has demonstrated applications in wound healing and has anti-cancer and anti-inflammatory properties. It is therefore very promising, but a major stumbling block to its effective use is its low solubility in water and poor bioavailability.

The encapsulation method developed by the UWA researchers creates particles with loads of magnetite ( $\text{Fe}_3\text{O}_4$ ) and high levels of surfactant-bound curcumin inside a porous-silica matrix. The high loading of  $\text{Fe}_3\text{O}_4$  nanoparticles inside the silica carriers was shown to be effective in making the



TEM cross-section of a capsule.

particles move when a magnetic field was applied. This is an interesting and novel approach for site-specific delivery of the stabilised curcumin. Concentrating it in desired cells or organs by use of an external magnetic field would increase its residence time in the vicinity of the target area. Furthermore, these particles show sustained release of curcumin under physiological pH. Their development is a first step towards effectively delivering curcumin to target areas for future treatment strategies.



Firearms versus fireworks – FIB cross-section of a GSR particle (left) and one from a sparkler (right).

## FIREARMS VS FIREWORKS

### FLAGSHIP

The detection and identification of gunshot residue (GSR) particles on suspects, victims and objects provide forensic evidence to assist police in their investigations and the results are ultimately tested by the judicial system. GSR particles are analysed in laboratories by scanning electron microscopy to provide morphological characterisation and by energy-dispersive X-ray spectroscopy to detect the constituent elements, coupled to an automatic particle-search-and-analysis system.

Originally, the composition of all GSR was consistent and unique; however, more

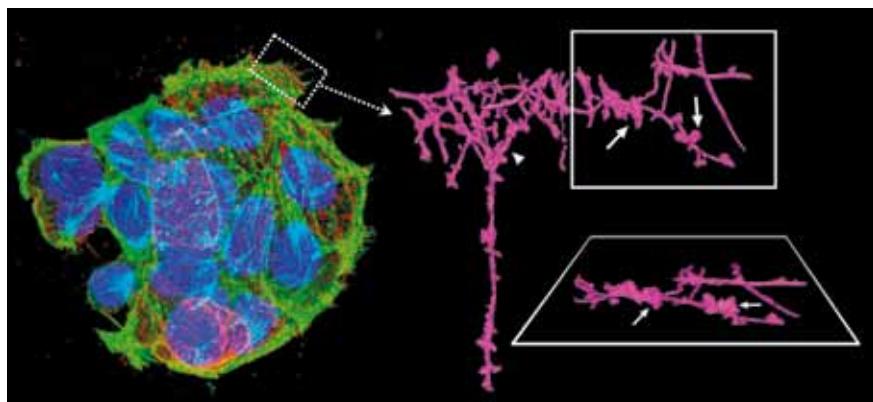
recently, a variety of new formulations have started being used, complicating forensics considerably. It is now far more likely that small particles similar to GSR could be present on suspects from other less-suspicious sources in the environment such as brake linings, fireworks and welding. These particles can appear similar to GSR when examined by the standard methods for detection of GSR described above. Such confusion could clearly have devastating results when someone's liberty depends on it. Ivan Sarvas from Forensic Science South Australia and colleagues from around the country and from the USA are investigating the internal structure and composition of a

variety of these particles. The team used the focused ion beam at the AMMRF at the University of Adelaide to slice the particles in half and compare GSR, sparkler particles and weld-slag particles. GSR is formed at extremely high temperatures and pressures and also cools extremely fast, which would be expected to leave telltale signs in the particles' internal structure. The results, seen in the picture, show an amorphous structure throughout the GSR particle, but a crystalline one in the sparkler particle. The weld-slag particle looked different again. This is entirely consistent with the different formation conditions – extreme temperatures and pressures with rapid cooling prevent crystallisation in GSR, but not in the sparkler where the less-extreme conditions allow crystals to form in the cooling particle. The detection of these differences is the first step to a clearer resolution of evidence involving GSR, and more wide-ranging work is continuing.

## ANCHORING THE RAFTS

Migration of cancer cells to new locations in the body, a process known as metastasis, is one of the main causes of death in many types of cancer. A number of cellular structures and processes contribute to this migration and understanding them is fundamental to stopping the migration from occurring. Kristina Jahn and A/Prof. Filip Braet from the AMMRF at the University of Sydney focus their work on dissecting the various structural and molecular aspects of membrane-mediated cancer metastasis in colorectal and oral cancer. This work relies heavily on microscopic examination of sub-cellular and molecular structures through a variety of correlative imaging techniques, such as multidimensional live-cell imaging, cell manipulation and analysis by electron tomography. In this way, novel structural information, over the nanometre to micrometre scales, can be gathered to underpin the development of new immunotherapeutic strategies.

The team has investigated the structure-function relationship between specialised membrane microdomains called rafts and the actin cytoskeleton of cancer cells. Their newly developed technique of 'whole-mount correlative light and electron microscopy' (CLEM) was applied to various cancer cell



Left: confocal laser scanning image of cancer cells stained for actin filaments (green), membrane rafts (red) and nuclei (blue). Dotted area shows filamentous extensions and the presence of membrane rafts at the leading edge of the cell. Right: transmission electron tomographic reconstruction of the filaments within such an extension.

lines that had been exposed to different experimental conditions known to alter membrane behaviour. By observing the changes, they have been able to demonstrate a close physical and functional relationship between actin-rich membrane protrusions and the specialised membrane rafts. Electron tomographic modelling was also carried out on the same CLEM samples, revealing the 3-D structure of the cytoskeletal actin filaments within these protrusions from the leading-edge of crawling cancer cells. Based on this combined imaging approach, the researchers demonstrated that these fine structures are all likely to be functionally

linked together and as such could regulate the cancer cells' fingertip dynamics. These leading-edge dynamics are crucial in the movement of cancer cells and therefore in the metastatic process.



Kristina Jahn won a 'University of Sydney, Postgraduate Research Prize for Outstanding Achievement' for this work.

## FINGERPRINTING ANCIENT CHINESE CERAMICS

Ceramics are the single most traded commodity from the ancient world and this is particularly true with Chinese ceramics, which have been exported around the world throughout history. It is important to identify origins of traded wares in order to reconstruct trade, cultural and technological interactions of ancient peoples. Ancient Chinese ceramics also enjoy great economic value and public interest as antiques, often auctioned for millions of dollars. However, modern fakes can be hard to distinguish by sight and lead to many controversies and lawsuits. The chemical fingerprints of ceramics originating from famous Chinese kilns would provide extremely valuable



White-pottery ritual drinking utensil, Chinese Xia dynasty (i.e., the Erlitou culture, c. 1900-1500 BCE).

criteria with which to source traded wares and authenticate antiques.

Dr Baoping Li from the University of Queensland (UQ), along with collaborators from La Trobe University, Oxford University, the Archaeology Institute of the Chinese

Academy of Science, the Museum of Oriental Ceramics in Japan and the Metropolitan Museum of Art in New York, has developed fingerprinting techniques to identify white pottery from China's first dynasty, the Xia dynasty, circa 1900–1500 BCE. This

white pottery is one of the most significant archaeological finds from Xia and is found around the capital at Erlitou and other sites in the surrounding areas of Henan province in central China. The white-pottery vessels appear to have been used as ritual drinking utensils and status symbols, and usually were only found in small numbers in elite burials at sites of major historical significance. Their paucity leads to the hypothesis that such wares were manufactured at only a few locations and then circulated among the elite

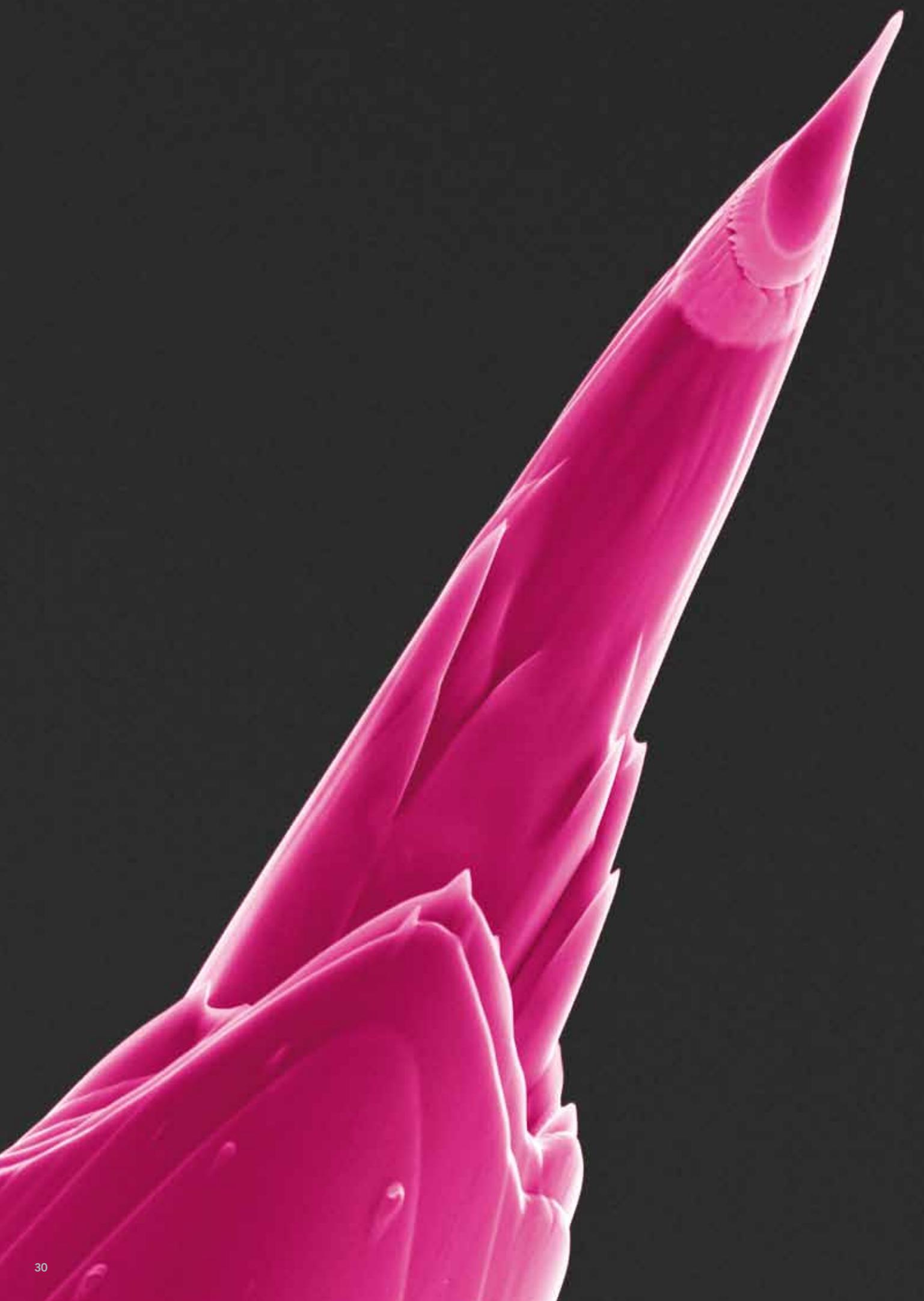
individuals in the region as prestige items. Recent excavations at the settlement site of Nanwa indicate that its whiteware finds were possibly produced locally, suggesting that Nanwa might have been a production centre. This also raises the interesting question of whether the whitewares found at Erlitou were produced at Nanwa. To answer this question, the team analysed and compared the elemental and strontium isotopic compositions of whiteware discovered at Erlitou and Nanwa by using inductively coupled plasma

mass spectrometry and thermal ionisation mass spectrometry at the AMMRF at UQ.

The analysis indicates that, while some of the whiteware found at Erlitou has identical elemental and strontium-isotopic compositions to the whiteware of Nanwa, much of it did not share these elemental and isotopic characteristics, indicating that there must have been additional production centres for white pottery during the Xia dynasty.

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## MICROSCOPY – SUPPORTING OUR INDUSTRY PARTNERS

The purpose of the AMMRF is to enable Australian research and innovation. Although this happens largely through its support for academic research, the AMMRF also plays an important role in industrial research and development. Industry access to the AMMRF can follow a number of paths, according to the needs of the individual business concerned. The major forms of industry interaction or access are:

### Contract R&D projects

These relationships occur where an industry partner will fund the costs of research, including beamtime fees, consumables and salaries for research staff or student scholarships.

### Leveraged R&D projects

This type of partnership includes ARC Linkage Projects and may involve multiple collaborative partners. ARC Linkage Projects provide an ideal way for industry partners to access the full range of academic and technical expertise that exists within the AMMRF. They provide long-term alliances to solve major research questions relevant to the industry partner and extend the research profile of the academics.

### Access by industry users to instruments and capability

Employees enter the AMMRF user experience in the same manner as other researchers. They are trained and provided with access privileges in-line with their level of competency. Instrument or beamtime fees are charged at commercial rates that are determined by individual nodes of the AMMRF.



### Testing and consultancy services

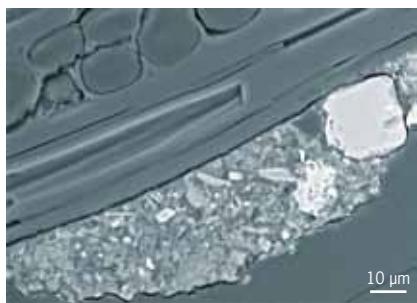
Testing and consultancy services are offered at commercial rates by the AMMRF. The variety of capability that the AMMRF has to offer is large and consequently the facility is used for testing services by a broad range of industry sectors.

### Training

AMMRF training courses and programs are open to industry users and researchers. Participation in these courses builds in-house competency for the companies or it can enable industry users to access instruments within the nodes themselves.

The variety of ways by which industry and business can interact with the AMMRF, combined with the broad range of microscopy and microanalysis capability available, ensures that the Facility is able to provide solutions to a broad range of industry sectors. Organisations that have accessed or engaged nodes of the AMMRF include those from the following areas:

- Mining and mineral resources
- Heavy manufacturing
- Construction
- Chemical
- Energy – oil and gas
- Transport
- Communications
- Nanotechnology
- Renewable energy – solar technologies
- Electronics
- Photonics
- Defence
- Semiconductors
- Biotechnology
- Biomedical, including surgical implants and prosthetics
- Pharmaceutical
- Art and museums
- Novel materials



*Synthetic terra preta soil from biochar.*

## CONTRACT RESEARCH

at the University of New South Wales for AnthroTerra

**Problem:** The rising level of carbon dioxide in the atmosphere is leading to global warming. There is a need to remove carbon from the atmosphere and sequester it in the long term.

**Solution:** Biochars come from pyrolysed biomass and can lock away carbon and improve soils and could contribute to this sequestration. By using microscopy at the University of New South Wales, researchers are characterising biochars that have been processed in different ways and from different feedstocks. This allows optimisation of the biochar structure to maximise the impact of these biochars in improving soil health and in carbon storage.

☞ [www.anthroterra.com.au](http://www.anthroterra.com.au)

☞ [www.venearth.com](http://www.venearth.com)



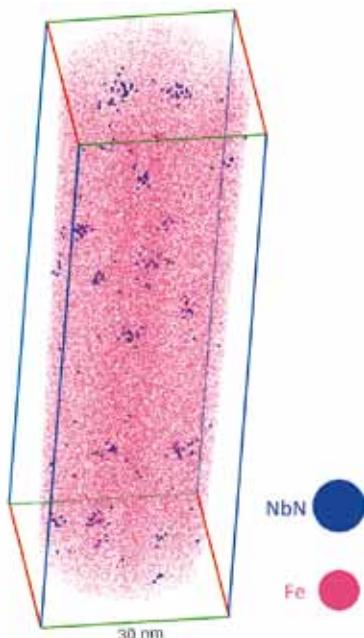
## INDUSTRY ACCESS

at the University of Adelaide by BHP Billiton

**Problem:** BHP Billiton needs to monitor efficiency of mineral recovery from Olympic Dam ore and determine how to optimise processing to extract the most gold, copper and uranium from the ore.

**Solution:** Through the AMMRF, BHP Billiton obtains regular access to the electron microprobe and scanning electron microscopes at the University of Adelaide. Their personnel analyse the ore and tailings samples from the mine. Through this microscopy, the company detected a processing flaw that, when corrected, saved BHP Billiton considerable sums of money (many millions of dollars) in the first year alone. These savings will accumulate to be very significant over the life of the process change.

☞ [www.bhpbilliton.com.au](http://www.bhpbilliton.com.au)



*Niobium-nitride clusters in 0.04%Nb as-received Castrip® steels.*

## LINKAGE PROJECT

at the University of Sydney with BlueScope Steel

**Problem:** BlueScope Steel has developed a new processing technology (Castrip®) to directly cast molten metal into strip steel. Although it is a much more efficient process than conventional casting and rolling, it creates new types of microstructures never seen before.

**Solution:** The AMMRF atom probe flagship instruments at the University of Sydney are revealing these microstructures in fine detail, allowing BlueScope Steel to further develop their Castrip® processes, especially their alloys, to create new and better products.

☞ [www.bluescopesteel.com.au](http://www.bluescopesteel.com.au)

## TESTING SERVICES

at the University of South Australia for Aus Systems

**Problem:** Aus Systems had developed a disposable rectal biopsy system that needed FDA approval to allow it to be used in the USA. The company needed to show that different sterilisation procedures didn't change the structure of the plastic components.



Rectal biopsy unit.

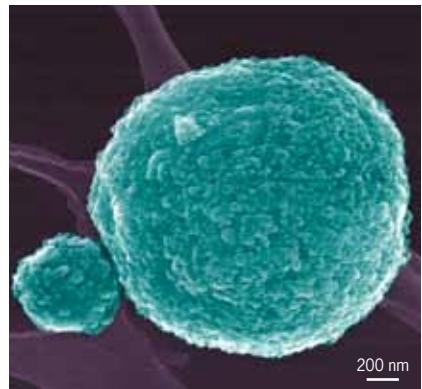
**Solution:** The AMMRF ToF-SIMS flagship instrument at University of South Australia was used to show that the structure of the plastic did not change. These results led directly to FDA approval. Aus Systems now export the devices to the USA from their manufacturing base in Adelaide.

[www.aussystems.com.au](http://www.aussystems.com.au)

## TESTING SERVICES

at the University of Queensland for the Very Small Particle Company

**Problem:** The Very Small Particle Company have developed a unique process to produce industrial-scale quantities of very accurately sized complex metal oxide nanoparticles. Monitoring of this process is critical to successful application.



Agglomeration of nanoparticles for lithium-iron-phosphate batteries – the material of choice for full electric vehicles.

**Solution:** The University of Queensland provides electron microscopy services to the Very Small Particle Company allowing them to effectively monitor the size and production phases of the nanoparticles as a crucial part of their ongoing quality control. They can then concentrate on producing quality particles from their Queensland base. This will result in efficient automotive catalysts, more-efficient and less-toxic industrial catalysts, and energy materials for use in rechargeable lithium-iron batteries suitable for electric vehicles.

[www.vspc.com](http://www.vspc.com)

## TESTING SERVICES

at the University of Adelaide for Origin Energy

**Problem:** Origin Energy has a pilot production facility in South Australia to develop and manufacture advanced solar cells using SLIVER Cell technology. SLIVER Cells are substantially thinner than most solar cells yet highly efficient, while using a lot less silicon. However, the details of the device structure at the nanoscale needed to be understood better.

**Solution:** Through the analysis of specific sites on production samples by using high-resolution scanning electron microscopy on the AMMRF flagship instrument at the University of Adelaide, several processes used in the manufacture of SLIVER Cells have been significantly finetuned. The engineers have used this knowledge to fast-track the development process, providing significant cost savings and helping to advance the development of SLIVER technology to the market phase.

[www.originenergy.com.au](http://www.originenergy.com.au)

The AMMRF has hosted industry trainees from a diverse range of organisations such as:

- Alcoa – aluminium producer
- Ammtech – metallurgy
- BHP Billiton – multinational mining
- Blue Circle Southern Cement – construction
- Cochlear – biomedical devices
- Fonterra – dairy industry
- National Measurement Institute – nanotechnology
- NSW Police Forensic Services – forensic science
- Origin Energy – sustainable energy
- Silverbrook Research – next-generation printing
- Sylvan Scientific – pharmaceuticals
- Ondek – biotechnology
- Pasipaley Pearls – jewellery
- Wriota – semiconductor devices

## PATENTS

Several patents have been lodged as a result of research enabled by the AMMRF and through technical developments taking place in the nodes. Three examples are highlighted here.

## Antimicrobial surfaces

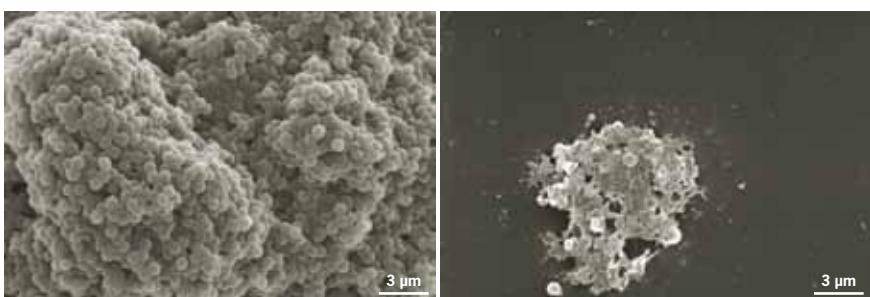
Enabled by the AMMRF at the University of South Australia

Prof. Hans Griesser and his collaborators at the University of South Australia have identified a suite of novel antibacterial serrulatane diterpenes, from the resin and leaves of many species of the Australian native plant *Eremophila*. They are using them to prevent bacterial growth on medical implants. The intention is that covalent binding of the diterpenes to the surface of implants will be able to provide permanent protection, stopping the coated implants from ever harbouring infection when inside the body.

- International patent application PCT/AU2009/000094



*Eremophila denticulata subsp. trisulcata (Chinnock)*, a taxon identified as having resin with antibacterial activity. The glossy and waxy appearance of the leaves is typical of resinous *Eremophila* species.



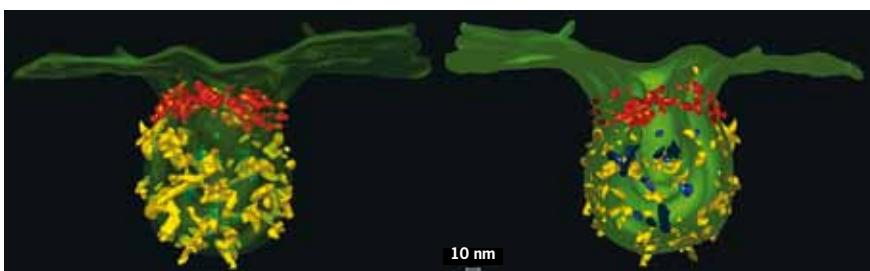
After 24 hours of growth: well-developed bacterial biofilms on a control surface (left), and very few adhering bacteria, with broken biofilm structures, on a serrulatane-coated surface (right).

## Delivery of molecules

Enabled by the AMMRF at the University of Queensland

Prof. Rob Parton, who works on proteins and processes at the cell surface, has developed a method for encapsulating drugs into tiny membrane-bound vesicles. Specific targeting signals are also engineered into the vesicles, directing them only to specific cells within the body, greatly increasing the drug's effectiveness and minimising adverse reactions.

- International patent application PCT/AU2008/001416



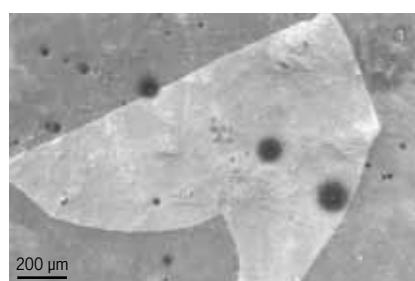
Two views taken from a tomographic reconstruction of a caveola. Green indicates the membrane surface. Left: view of cytoplasmic face of caveola, with cell surface to top. Right: rotated structure showing interior face of caveola.

Figure by Matthias Floetenmeyer, Charles Ferguson, James Riches, Brad Marsh and Rob Parton.

## Energy-filtering secondary electron detectors

Resulting directly from the AMMRF at the University of Western Australia

Investigation of contrast mechanisms in modern scanning electron microscopy (SEM) and scanning ion microscopy (SIM) has led AMMRF researcher, Prof. Brendan Griffin at the University of Western Australia, and his collaborators, to the recent discovery that secondary electrons are not all the same. The discovery has led to the design of a new range of energy-filtering secondary electron detectors that can be mounted on existing and future SEMs. It has the potential to improve the imaging performance of the workhorse SEM, and to lead to new SEM applications.



*Energy-filtering SEM. Top: conventional below-lens Everhart-Thornley secondary-electron image of a carbon-coated polished copper standard in resin. Bottom: in-lens, energy-filtering secondary-electron image of the same area under identical conditions. The dark areas are charging or surface contaminants, including small oval beam-contamination marks.*

- Provisional patent application  
2009902870

## SPIN-OFF COMPANY – DIGITAL CORE LAB

The micro-CT facility in the AMMRF at the Australian National University (ANU) is at the centre of a research consortium of ANU and University of New South Wales (UNSW) researchers and many of the world's largest oil and gas companies, including Shell, BP, Chevron and ExxonMobil. Their technology uses X-rays and clever data-processing software to analyse the oil-recovery potential of different rocks through the 3-D imaging of their oil-bearing pores. The research work has been so impressive that it has resulted in a company, Digital Core Lab, being formed to serve the energy industry through the provision of these new and highly effective technologies.



Micro-CT image of residual hydrocarbon saturation in an oil reservoir rock after flooding with water. The rock grains are transparent, showing the water, in yellow, and the residual oil in orange. Filed of view 1.5 mm. Image by Munish Kumar, ANU.

## COMMERCIALISATION OF IMAGES

The nature of the AMMRF means that researchers and staff produce attractive and informative images every day, with its nodes accumulating substantial image collections. The AMMRF is placing selected images into Science Photo Library where they can continue to benefit the AMMRF through the generation of an income stream from the sale of reproduction rights; the building of its reputation through the use of a credit line when the images are reproduced; and through the engagement of people with science as a result of the dissemination of striking scientific images.



SEM image of wattle pollen by Dr Ian Kaplin and Uli Eichhorn.

## USER SURVEY RESULTS

Each year the AMMRF asks its users for feedback on how they perceive its performance. The 2009 survey resulted in:

- Nearly 93% of users agreeing or strongly agreeing that they were satisfied with staff support.
- Over 93% of users agreeing or strongly agreeing that they were satisfied with the facilities.

The survey also provided much valuable feedback including the following comments.



*Outstanding commitment to improved techniques, made possible by dedicated staff. This allows cutting-edge science.*

*Great service with a big smile.*

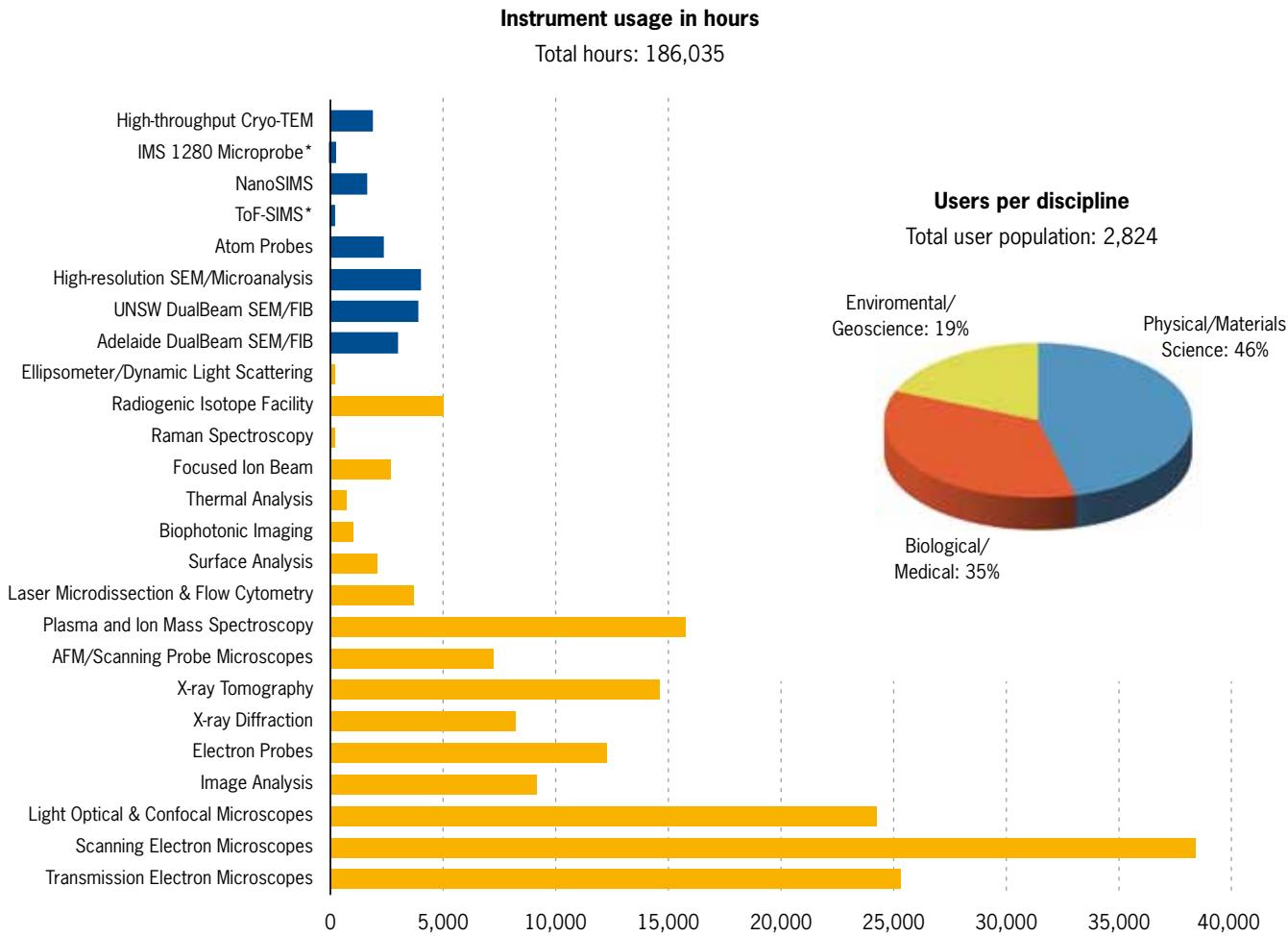
*I see the most valuable aspect as the staff – they have been excellent in helping me work through the challenges. The training is excellent.*

*Excellent facility – no complaints at all.*

*I am very new to this service, but I was made comfortable and confident of using sophisticated machines with ease.*



## INSTRUMENT USAGE



\* Commenced operation in Q4 2008–2009.

## CONTACTS

### Nodes

#### **The University of Sydney: Australian Key Centre for Microscopy and Microanalysis**

AMMRF Headquarters

T: 02 9351 2351, F: 02 9351 7682, E: info@ammrf.org.au

#### **The University of Queensland: Centre for Microscopy and Microanalysis**

T: 07 3346 3944, F: 07 3346 3993, E: cmm@uq.edu.au

#### **The University of Western Australia: Centre for Microscopy, Characterisation and Analysis**

T: 08 6488 2770, F: 08 6488 1087, E: admin@cmca.uwa.edu.au

#### **The University of New South Wales: Electron Microscope Unit**

T: 02 9385 4425, F: 02 9385 6400, E: emu.enquiries@unsw.edu.au

#### **The Australian National University: Centre for Advanced Microscopy**

T: 02 6125 5104, F: 02 6125 3218, E: tim.white@anu.edu.au

#### **South Australian Regional Facility (SARF)**

University of South Australia, Ian Wark Research Institute

T: 08 8302 3703, F: 08 8302 3683, E: hans.griesser@unisa.edu.au

The University of Adelaide, Adelaide Microscopy

T: 08 8303 5855, F: 08 8303 4356, E: microscopy@adelaide.edu.au

Flinders University, Flinders Microscopy

T: 08 8201 2005, F: 08 8201 2905, E: joe.shapter@flinders.edu.au

### Linked Laboratories

#### **James Cook University: Advanced Analytical Centre**

Dr Kevin Blake, T: 07 4781 4864, F: 07 4781 5550, E: kevin.blake@jcu.edu.au

#### **Queensland University of Technology: Analytical Electron Microscopy Facility**

Prof. John Bell, T: 07 3138 4298, F: 07 3138 1529, E: j.bell@qut.edu.au

#### **RMIT University: Microscopy and Microanalysis Facility**

Prof. Dougal McCulloch, T: 03 9925 3391, F: 03 9925 5290, E: dougal.mcculloch@rmit.edu.au

#### **CSIRO, Australian Animal Health Laboratory: Australian Biosecurity Microscopy Centre**

Dr Alex Hyatt, T: 03 5227 5419, F: 03 5227 5555, E: alex.hyatt@csiro.au

#### **Macquarie University: Optical Microcharacterisation Facility**

Prof. Ewa Goldys, T: 02 9850 8902, F: 02 9850 8115, E: goldys@ics.mq.edu.au

#### **Curtin University of Technology: John de Laeter Centre of Mass Spectrometry**

Dr Brent McInnes, T: 08 6436 8694, F: 08 9266 2377, E: brent.mcinnnes@csiro.au

### Linked Centre

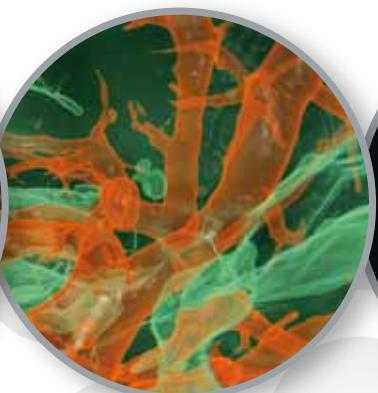
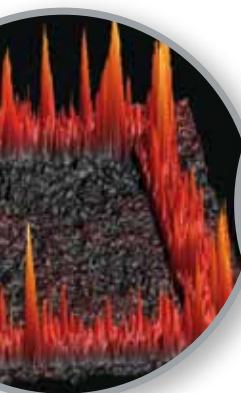
#### **The University of Queensland: Australian Institute of Bioengineering and Nanotechnology (AIBN)**

Dr Margaret Butler, T: 07 3346 3845, F: 07 334 63973, E: m.butler1@uq.edu.au





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