



- Hydrogen from methane – and no CO₂
- Dinosaurs give up their secrets
- ITUAG gives good advice in Brazil
- A microscopist's adventures in Mongolia

RESEARCH

Ancient starch a hit in *Science*

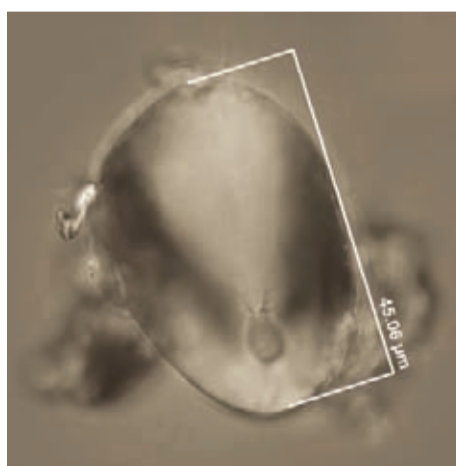


The Ivane Valley, at an altitude of 2000 metres in the South-Eastern highlands of Papua New Guinea.

AMMRF @ USYD

Archaeologists have found new evidence for some of the earliest known settlements in Papua New Guinea. The sites were occupied during the last ice age when New Guinea and Australia were one landmass, called Sahul. The colonisation of Sahul was a key milestone in human history. The study sites are located 2000 metres up in the mountains of the Owen Stanley Range and are dated from around 44–49,000 years ago.

The findings were published in the 1 October issue of the highly prestigious journal *Science*. The team included Dr Judith Field from the AMMRF at University of Sydney, who was responsible for the microscopic analysis of starch grains in stone-tool residues. She said, "The degree of preservation of starch residue on the surface of the stone tools exceeds anything else we have studied. These residues are direct evidence that point to starchy plants being processed." These were found to be largely pandanus nuts and yams. Evidence comes from campsites buried by volcanic ash, where people made stone tools, hunted small animals, gathered the nuts of the local pandanus trees and processed yams.



A yam starch-grain from one of the stone tools.

Yams only grow at altitudes below those of the Ivane Valley, so the discovery of yam starches on stone tools coupled with the presence of stone from outside the Ivane Valley shows that people were moving and living between several altitudinal zones. The cold conditions would have made life uncomfortable at that altitude and the Ivane Valley may have been at the very edge of routinely habitable territory. It is no surprise that occupation of the area ceased during the coldest periods of the last ice age. ■

Cracking methane and growing nano-onions

AMMRF @ UWA

Ongoing research at The University of Western Australia (UWA) has been developing novel technology for the cracking of methane (natural gas) into hydrogen and graphitic carbon. The research collaboration, led by Professor Hui Tong Chua of the UWA Centre for Energy, makes extensive use of electron microscopy facilities at the AMMRF at UWA to understand the cracking process and to characterise the resulting nanomaterials.

Hydrogen is a valuable industrial chemical, at present, mainly used for the preparation of ammonia for fertiliser and other applications. However, in future, it is likely to be a critical component of a reduced-emission energy market. Current methods of generating hydrogen are energy inefficient and directly or indirectly create significant amounts of carbon dioxide emissions. The current research generates hydrogen with no carbon dioxide emissions.

In addition to the clean production of hydrogen, the reaction produces significant quantities of graphitic carbon nanomaterials, predominantly in the form of nano-onions. Consisting of sequentially self-encapsulating carbon shells, the carbon nano-onions have potential application and value in a range of areas, in particular as high-quality electrodes for use in electrochemical processes and batteries. A tonne of the carbon material has already been requested for trials by a Chinese battery manufacturing company, although the process has to be scaled up considerably first.

The research has had strong support from the private sector throughout the project. Initially supported by Wesfarmers and the US Technology development group XLTG Inc. through an ARC Linkage Project, the research has formed the basis of a new spin-out company, Hazer Pty Ltd, which has successfully raised capital from seed investors and is now funding further development work at the university. Patents are in place to cover the cracking process and purification of the carbon nanomaterials. The company name is an acronym of Hydrogen And Zero Emission Research.

The researchers rely on the high-resolution scanning and transmission electron microscopy (SEM and TEM) facilities at the UWA Centre



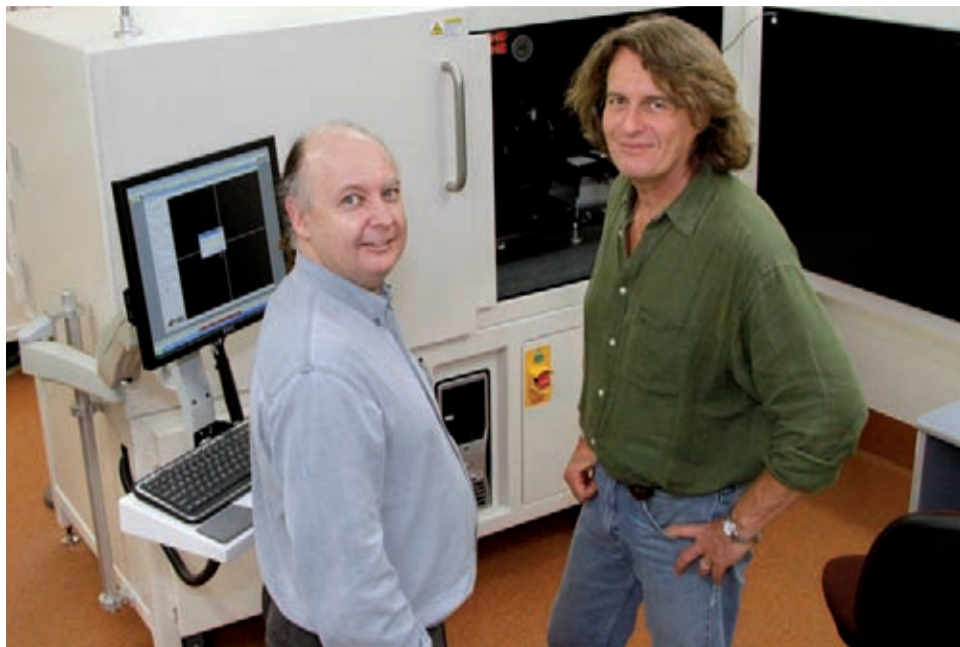
for Microscopy, Characterisation and Analysis (CMCA) to investigate the structural and chemical properties of the carbon and the catalyst materials used in the cracking process. CMCA Deputy Director, Prof. Martin Saunders (above), is a core partner in the research team, providing expertise in advanced TEM techniques to correlate the properties of the catalyst with those of the resulting carbon nanomaterials. He has co-supervised several students involved in the project, including Rahi Varsani who recently received an award from the Australian Microscopy and Microanalysis Society for his research on the use of electron tomography to investigate the 3-D morphology of the nanomaterials.

The capital raised by the formation of the spin-out company is being invested into the ongoing research at UWA to further develop and scale-up the methane cracking process. Microscopy will continue to play a crucial role in this future development. ■



TEM image of carbon nano-onions with encapsulated catalyst particles.

Searching for hidden secrets of the sauropods



AMMRF @ USYD

Recently, world authority on dinosaur eggs, Dr Gerald Grellet-Tinner from the Field Museum in Chicago, visited the Australian Centre for Microscopy & Microanalysis (ACMM) at the University of Sydney. Dr Grellet-Tinner has largely pioneered the application of scanning electron microscopy (SEM) to the characterisation of fossilised dinosaur eggshells. He sought to extend this approach by looking at the microstructure of

these eggshells in 3-D, using X-ray microtomography. It was a busy two weeks as Dr Grellet-Tinner worked with staff to collect SEM images of a variety of fossilised eggshells, and generate the first 3-D data by X-ray microtomography. Dr Grellet-Tinner and one of his PhD students recently published the background to this study in *Nature Communications*, where they reported that certain dinosaurs regularly returned to geothermal fields to shape nests and deposit eggs to be incubated by the ambient heat.

Sauropods are a group of extremely large herbivorous dinosaurs that seem to have reproduced in globally distributed, but very particular and localised, nesting sites. The overall rarity and nature of these rookeries, combined with the overwhelming abundance of egg clutches in the sites that have been found, led Dr Grellet-Tinner to propose that sauropod dinosaurs were colonial nesters and migrated to the same selected sites to lay their eggs. This raises a new question about the origin of these behaviors. Part of the answer is locked in the eggshell structures themselves, as they reflect evolutionary adaptations to particular nesting environments by preserving information in their carbonate microstructures and their minute inclusions. At the ACMM, Dr Grellet-Tinner aimed to discover new eggshell features that could act as keys to understanding these adaptive processes and the particular nesting behaviours of the sauropod dinosaurs. The analysis of the data that was collected will take many months, but already it is clear that the micro-CT data can reveal, as never before, the internal pore-structure connectivity of the eggshell fossils and that SEM-based EBSD mapping can reveal a wealth of information on the structure and formation of the eggs. When all the results are analysed, we can all expect a fascinating story to emerge. ■

COMMUNITY



Australia – second place

In September, a team from the two AMMRF nodes in Sydney, representing the Australian Microscopy and Microanalysis Society, made the long trek to the International Microscopy Congress (IMC) in Rio de Janeiro in a bid to host the IMC-18 in Sydney in 2014.

Unfortunately, it was not meant to be. Sadly, when the final votes were cast on 23 September, 'Team Australia' lost to Prague who managed to secure 34 votes; Sydney came second with 14 votes, followed by Beijing (7) and Istanbul (4). It turned out that the large number of European voters were very united and felt that the congress, after previously being held in South Africa, Japan and now Brazil, should go back to Europe in 2014. C'est la vie. So we'll see you in Prague in 2014, instead!

GOVERNANCE

International Advisory Group meets in Rio de Janeiro

Members of the AMMRF International Technical and User Advisory Group (ITUAG) met during the 17th International Microscopy Congress in Rio de Janeiro, Brazil. This was the third meeting of ITUAG members and built on successful meetings held previously in Sydney (2008) and Richmond, Virginia, USA (2009). Participants were from the UK, France, Switzerland and the USA. Australian attendees included the AMMRF Executive Director, Prof. Simon Ringer, Chairman of the AMMRF Board, Dr Greg Smith, and the AMMRF General Manager, Dr Miles Apperley.

Prof. Ringer gave a presentation summarising the AMMRF's current status and recent achievements. A wide-ranging discussion followed, covering issues including future Australian government processes for ongoing investment in research infrastructure investment, world-class training and future scope for the AMMRF to link with European Union-funded programs such as ESTEEM and Euro-Biolmaging. The ITUAG members were impressed with the facility's performance to date and are committed to continue their support in the future. ■



From left: Dr Miles Apperley, Prof. Paul Munroe, A/Prof. Guy Cox (all AMMRF), Prof. Simon Watkins (University of Pittsburgh), Dr Tom Kelly (CAMECA Instruments), Prof. Simon Ringer (AMMRF), Prof. Roger Wepf (Swiss Federal Institute of Technology), Dr Greg Smith (AMMRF), Prof. Marin van Heel (Imperial College), and Dr Richard Leapman (National Institute of Biomedical Imaging and Bioengineering, USA).

EXECUTIVE DIRECTOR'S COLUMN

The recent metrics collection for the AMMRF made this a busy time for many of our staff, collecting a mass of data for inclusion in the various reports required as part of our funding agreement. Particularly important information comes from the annual user survey. We had a record response from our user community this year, and the results were uniform across the nodes. They confirm that the most important asset of the AMMRF is the staff, with strong ratings recorded across the board for staff support. Their knowledge, expertise and attitude are routinely tested when we consider that the AMMRF operates one of the largest (if not the largest) scientific user facilities in Australia. So, not only would I like to thank all of our users for their valuable feedback, I wish to thank and congratulate our staff, and especially our technical staff, who have a key frontline role in making the user experience work so well for so many researchers.

Next year, we are looking forward to continued successful partnerships with our colleagues working in the e-Research space to develop better online collaboration tools that can facilitate new and more efficient interactions between users and facility management. Coping with the demands for research training on advanced instruments for this community is also a major undertaking. To better tackle this issue, our team is already well down the track on an initiative funded by the Australian Learning and Teaching Council (ALTC), and led by our colleagues at the University of Queensland. It will provide an online environment within which users can achieve a basic competence in the operation of certain instrument types prior to coming along to their first training session on the microscope column. When operational, this will be a boon to the technical and academic staff involved in user training.

Accompanying this issue of the News is our 2010 Profile, focused around the theme of 'making an impact'. Not only is this evident through the research outcomes described in its pages but also through the steady increase in instrument usage. We are now serving over 3000 users and delivering in excess of 200,000 hours of beamtime a year.

Please have a good read of the 2010 Profile – I believe that it represents the state-of-the-art in what a national microscopy and microanalysis research facility can achieve. ■

Best wishes for the festive season,
Simon Ringer
Executive Director & CEO



TEACHING

A microscopist in Mongolia

AMMRF @ UQ

Graeme Auchterlonie from the Centre for Microscopy and Microanalysis (CMM) at the University of Queensland (UQ) recently took his long-service leave, but rather than driving around Australia, he combined it into a six-month trip to China and Japan. He first spent two weeks at the Central South University in Hunan Province, where he taught scanning electron microscopy and a little transmission electron microscopy.

The next several months were spent in Baotou, Inner Mongolia, where he was a guest of Prof. Ruiping Ren at the Inner Mongolia University for Science and Technology. On the doorstep of the Gobi Desert, Graeme (below) taught high-end microscopy to very appreciative microscopists and students. Graeme was able to pass on all his tips and tricks, learned over many years, to make the operation of their in-

struments easier. He was also able to reassure the microscopists that they were actually doing a very good job with the instruments they have.

The microscopy community was keen to show its gratitude, so, when not in front of a microscope or an audience, Graeme was welcomed into the local culture, attending many Mongolian feasts that were liberally lubricated by *bijo*, the local firewater. These were considerably more enjoyable than the sand storms that blew in off the Gobi Desert every few days.

Leaving the desert behind, he then went to Tsukuba, Japan, on an Australian Academy of Science Clean Energy Grant for two months to research thin-film anodes on electrolytes for solid-oxide fuel cells.

Now safely back at UQ, Graeme will continue to build on the connections with his newfound friends in Chinese microscopy and Japanese research. ■



OUTREACH

Inspiring the the young – Sleek Geeks and beyond



Dr Karl, one of the two Sleek Geeks, being filmed for Catalyst, with the primary- and secondary-school Sleek Geeks Science Prize winners touring the ACMM at the University of Sydney.

AMMRF @ USYD

Microscopy is one of those things that has great power to inspire people in the wonders of science. The stunning visual imagery that it generates induces an immediate 'wow factor' that can be used by science communicators at all levels of engagement. At the time of writing, images from the Australian Centre for Microscopy & Microanalysis (ACMM) at the University of Sydney are going to air in the second series of *Sleek Geeks* to embellish the set and provide mystery objects to be identified.

The ACMM also played host to the primary- and secondary-school Sleek Geeks Science Prize winners, showing their videos and giving

them a tour of the centre with the Sleek Geeks themselves, all the while being filmed by the ABC Catalyst film crew. The footage of the tour is available online on the Catalyst website as a featured 'video extra'.

In addition to this high-profile outreach, many of the nodes and Linked Labs of the AMMRF regularly interact with young people, at open days, science events and school visits, providing hands-on microscopy experiences that allow students to engage with science in memorable ways that remain with them for years to come. The popularity of these events puts microscopists in a unique position to excite future generations, helping them to see the possibilities that science can offer. ■

COMMERCIAL

TechVouchers available

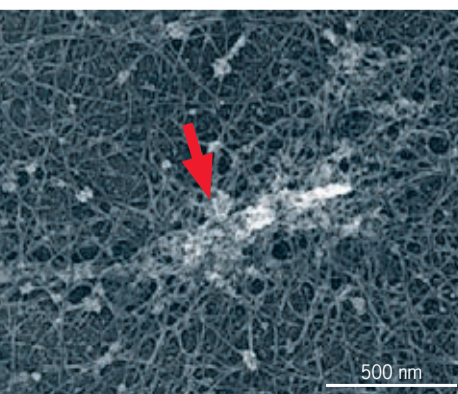
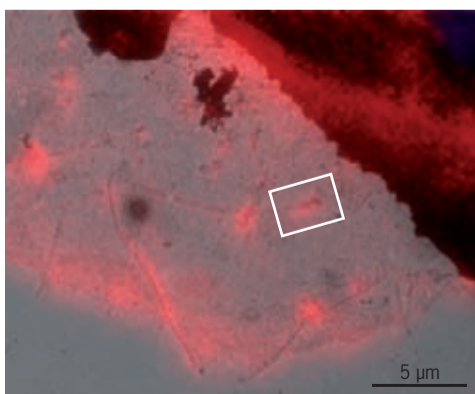
The TechVoucher scheme has been established by the NSW Department of Industry and Investment to encourage engagement between small and medium enterprises (SMEs) and public-sector research organisations (PSRO). Vouchers of up to \$15,000 are available to SMEs in NSW to enable testing services, research and development, or consultation. A complimentary part of the scheme is the funding of a position, called a Connector, to sit within the PSRO to facilitate these engagements and to promote the services available to industry clients.

The Victorian State Government, through their Small Technologies Industry Uptake Program (STIUP), is providing support for innovative Victorian businesses to incorporate small technologies into their processes in a bid to increase competitiveness. Vouchers are available for fee-based services through to prototype development projects. These schemes offer opportunities for increasing engagement between industry and researchers that may be of interest to our readers. ■

TECHNOLOGY

What is correlative microscopy?

The combined use of light or laser microscopy and electron microscopy (EM) has become increasingly important to our understanding of the structure and function of cells and tissues at the molecular level. Combining two or more different microscopy techniques, usually with different spatial and temporal resolution, is referred to as correlative microscopy (CM).



Left: merged confocal image, stained for membrane rafts (red), and low-resolution TEM image of a cultured cell. Right: high-magnification TEM image of actin-rich structures in association with one of the rafts identified by the confocal microscopy. The TEM image has been colour-inverted to aid visualisation. Images by Kristina Jahn.

It allows researchers to gain novel structure–function information that provides a greater degree of confidence about the structures of interest, as observations from one method can be compared to those from the other(s). The more popular combinations to correlate are light or fluorescence with either scanning or transmission electron microscopy.

The main issues that need to be considered in CM are positioning, sample preparation and labeling techniques. Special sample devices are used to allow the relocation of the structures of interest in different instruments and length scales. Dedicated cover slips, culture dishes, EM-supports and fiducial markers are readily available from commercial suppliers to facilitate alignment. Some vendors offer total solutions by using sample-transfer stages across different microscopy platforms for relocation purposes.

Sample preparation depends on the combination of techniques being used, and is crucial to success. Minimising any structural changes that occur during fixation or sectioning will help in the correlation process. Labeling techniques are available to allow visualisation of features in different techniques. For example, quantum dots are both luminescent and electron-dense and come in a variety of shapes, enabling different components to be labeled at the same time. For more see ammrf.org.au/techniquefinder. ■

STAFF NEWS

The University of Queensland

Dr Garry Morgan joined the Centre for Microscopy and Microanalysis as a support engineer, specialising in electron tomography. After graduating from the University of Colorado with a BA in molecular and cellular biology, he worked as an electron-microscopy technician for several years. Moving to Queensland in 2003, he joined the group of Dr Brad Marsh (IMB), whose research focus has been defining beta-cell structure and function (ie, diabetes) primarily by using electron tomography. Adding to his prior experience, the six years working in the Marsh Group has given him expertise in biological sample preparation for EM, as well as in acquisition and processing of tomographic data. ■

The University of New South Wales

Dr Deming Zhu has recently joined the Electron Microscope Unit as a senior technical officer to provide scanning electron microscopy (SEM) support to users. Deming has a PhD in materials science from Monash University and three years' experience working in the University of Newcastle, where he helped clients access SEM and transmission electron microscopy. He also collaborated with the School of Engineering, undertaking nanotechnology research. ■

The University of Sydney

Have you ever wondered who is behind the high-quality publications produced by the AMMRF? This person is **Uli Eichhorn**, our Communications & Design Officer, who, after six and half years, is leaving the AMMRF to accompany her husband to Brisbane. Uli joined the NANO-MNRF (the predecessor to the AMMRF) in 2004 and quickly started creating professional and high-quality designs and layouts. With the formation of the AMMRF in 2007, she set about developing the facility's brand and corporate look and feel, which is now a highly-recognisable and professional image that reflects greatly on our organisation. She was also involved in the organisation of special events like the AMMRF Strategic Planning Workshops and various conferences. Uli will be greatly missed by all her friends and colleagues. ■



IN BRIEF

The **Electron Microscope Unit at the University of New South Wales (UNSW)** has recently installed an ultra-high-resolution FEI Nova NanoSEM 230 to add to its flagship suite of analytical scanning electron microscopes. It features retractable in-lens SE and BSE detectors, signal-filtering capabilities and a Bruker silicon drift EDS detector. The ability of this microscope to work over a wide range of complementary variables, such as voltage, chamber vacuum and beam current will enable researchers to optimise imaging conditions for most materials irrespective of their size or conductivity.

In addition, a highly versatile Tecnai G2 200 keV transmission electron microscope has been installed to facilitate research

in materials science, nanotechnology and semiconductor fields. This also includes a Bruker silicon drift EDS detector. ■

The **Centre for Microscopy, Characterisation and Analysis at the University of Western Australia** has recently commissioned a new, state-of-the-art JEOL 8530F field-emission electron microprobe. The instrument performs high-resolution, in-situ chemical analyses on a diverse range of materials, but is particularly well suited to the analysis of rocks and minerals. It also provides essential precursor characterisation of samples destined for the IMS 1280 and NanoSIMS 50 flagship ion probes. ■

AWARDS

Congratulations to two young materials scientists



Chris Killmore (Product Design Manager, BlueScope Steel) with Sachin Shrestha holding his award.

The 7th Pacific Rim International Conference on Advanced Materials and Processing (PRICM) held in Cairns earlier this year, gave our young materials scientists a great opportunity to present their work, and AMMRF microscopy was at the heart of two of the awards presented at the meeting by the organisers.

PhD student **Sachin Shrestha**, from the Australian Centre for Microscopy & Microanalysis (ACMM) at the University of Sydney, jointly won the best poster award for his work on the high-temperature ageing response of CASTRIP® steels, part of a Linkage Project with BlueScope Steel and supervised by Prof. Simon Ringer and Dr Julie Cairney. Sachin's multi-faceted approach combined atom probe tomography, TEM and optical metallography, along with micro-hardness testing of his samples. Sachin won the award over 300 others and received an engraved crystal plaque and a cash reward of \$200.

The best lecture presentation out of the 850 at the conference was judged to be by **Dr Ilana Timokhina** from Deakin University whose work on nanoscale analysis of nano-bainite formed in advanced high-strength steels caught the judges' imagination. She too used atom probe tomography at the ACMM to reveal the structural features in her steel samples. ■

OUT OF THE FRAME

Play the name game

In pulling together techniques for the technique finder, it has come to our attention that our illustrious Executive Director & CEO, Simon Ringer, has a technique acronym to match his initials – Surface Plasmon Resonance (SPR). We don't think this is at all fair and, as we are all so fond of acronyms, we would like to create some interesting techniques so our other node directors don't feel left out.

So, while you are all digesting your Christmas dinners and trying to fill those empty holiday moments, please have a go at the name game. Send your suggestions to Jenny Whiting (details see box at right) and we will publish the best, most humorous creations in a future issue. Here is your starting material:



- JD John Drennan
- HJG Hans Griesser
- PRM Paul Munroe
- SPR Simon Ringer
- DDS David Sampson
- JGS Joe Shapter
- JGLT John Terlet
- TJW Tim White

Let your imagination run wild!



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