

The seeds of food security
Most commendable paper
Carbon's got it covered



Calibrating cantilevers for AFM
4th Strategic Planning Workshop
Characterisation Virtual Laboratory

RESEARCH

UQ Zinc oxide has been industrially produced as a white pigment for painting since the 1840s. Artists were attracted to zinc oxide based paints as they were less prone to discolouration than traditional whites. Manufacturers valued its lower toxicity and the beneficial properties it conferred to other paints. Zinc oxide peaked in popularity in the first half of the twentieth century and is found in many paintings from this period through to the present day. Unfortunately its presence has recently been linked to serious deterioration resulting from reaction of zinc oxide with fatty acids from the oil-based paints. In some paintings the resulting zinc carboxylate soaps aggregate to the point where they cause the paint to crack and flake or even erupt as lumps through the painting surface. Art conservators now want to know how these zinc stearate compounds form, why they accumulate, what conditions promote formation, and the all-important question, can the formation be prevented or reversed?

Gillian Osmond is an art conservator at the Queensland Art Gallery and is currently doing a PhD at the Australian Institute for Bioengineering and Nanotechnology at the University of Queensland with Prof. John Drennan and Prof. Michael Monteiro. Through an ARC Industry Linkage Project, *The Twentieth*



TEM image (inverted and colour-enhanced) of zinc oxide artists' pigment

Century in Paint, she is investigating the mechanism of zinc stearate formation and its distribution in affected paint samples. She is using a combination of characterisation techniques in the AMMRF at the University of Queensland and at the Australian Synchrotron, including

electron microscopy, X-ray analysis and Fourier transform infrared microspectroscopy.

The challenge is significant given the complexity of paint formulations and the many variables influencing drying and aging processes. Gillian has had access

to reference paints from the Smithsonian's Museum Conservation Institute enabling her to consider the influence of specific pigment, oil and paint additive combinations on the formation and behavior of zinc stearate in naturally aged paint films. She found that zinc stearate forms rapidly in the paint samples. She also observed that the problem was significantly worse in paint where aluminium stearate is present as it significantly influences the amount of stearic acid available to react with the zinc. Once formed in sufficient concentrations, the zinc stearate moves through the paint film, gradually aggregating into clumps. This aggregation has very important consequences for the stability of art works. A particularly high profile case involves Vincent van Gogh's, "Falling Leaves (Les Alyscamps)".

Continuing to improve our understanding of the formation and behavior of zinc stearates will enable art conservators to identify susceptible paintings and prescribe protocols for minimising associated deterioration and will have significant impact on art conservation.

Gillian's findings have recently been published in *Applied Spectroscopy* and are part of a collaboration with researchers from JAAP Enterprise for MOLART Advice in Amsterdam, the AMMRF at the University of Queensland and the Australian Synchrotron. ■

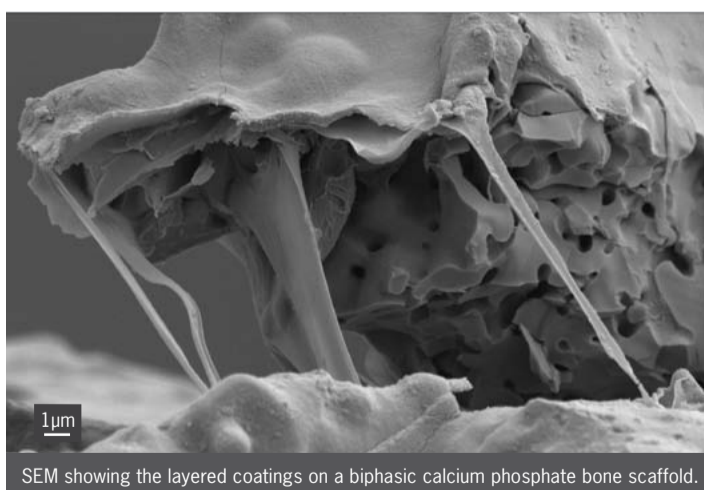
Silk and polyester could help repair bones

USyd Bone substitute materials are very widely used in orthopaedics, cranio-facial surgery and dental applications to repair damage caused to bones by injury or disease. If only a small area of bone needs to be replaced it can often be taken from elsewhere on the patient, but where large areas have to be reconstructed artificial bone scaffolds have to be used. These provide a framework into which the patient's own bone can gradually grow, resorbing the scaffold material as it goes. Around 2.2 million bone grafts are done annually and the bone scaffold market is worth more than \$7 billion in the US alone.

Certain ceramic materials are widely used for bone scaffolds but when used alone, they are quite brittle, especially when pore size is

optimal for encouraging new bone growth. This leads to limitations in their use for repairing leg bones and in other load-bearing applications. A/Prof. Hala Zreiqat and her team from the University of Sydney are investigating a number of new scaffold materials. In recently

published work, silk, and a type of biodegradable polyester, poly (ϵ -caprolactone) (PCL), are being combined to strengthen ceramic



SEM showing the layered coatings on a biphasic calcium phosphate bone scaffold.

bone-scaffold materials made of biphasic calcium phosphate (BCP).

The team successfully addressed the

brittleness of the ceramic scaffold by coating it with PCL. They then further improved its bioactivity (since PCL is inert) with a silk coating. This familiar natural polymer happens to bind particularly well to the PCL. The strength of the combination material under compression is approximately seven times that of the ceramic alone. The surface layer of silk is also responsible for improved growth of bone cells on the scaffold. This composite material appears to be a significant step forward in bone tissue engineering and regeneration applications, particularly at vitally important load-bearing sites.

This work has been enabled through scanning electron microscopy in the AMMRF at the University of Sydney. ■

RESEARCH

The seeds of food security

SARF Salty soils are an increasing problem in agriculture. To ensure future food security it is vital that we improve agricultural yield from these unpromising soils. Wheat for bread is somewhat tolerant to salty conditions but durum wheat, used in the production of pasta and other foods, is far less so. A collaborative team, mainly from CSIRO Plant Industry and the Waite Institute at the University of Adelaide, has identified a gene from an ancestral relative of modern wheat and bred this gene into durum wheat. Microscopy in the AMMRF at the University of Adelaide by

postgraduate student Bo Xu, showed that the protein encoded by this gene sits in the cell membrane of the roots, reducing the amount of salt reaching the leaves. The knock-on effect is that the plants carrying the gene have a 25% increase in grain yield on salty soils when compared to normal durum wheat.

The work was published recently in *Nature Biotechnology* and offers great promise for improved food yields under challenging environmental conditions. It also serves to highlight the value of biodiversity as a versatile toolbox for managing our response to climate change. ■



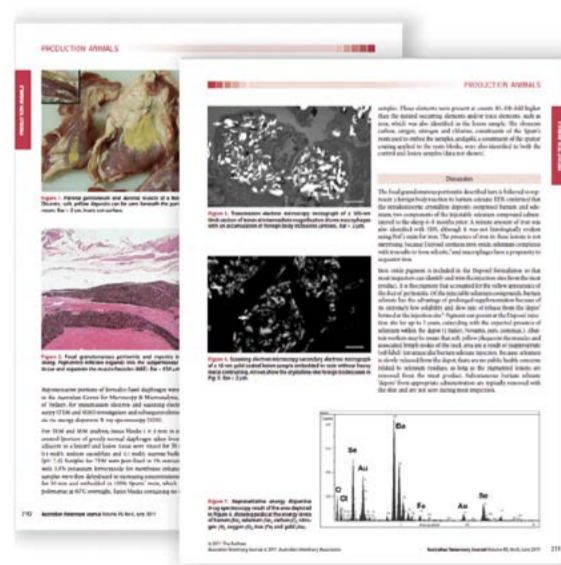
Most Commendable Paper

USyD A/Prof. Filip Braet, Deputy Director of the AMMRF at the University of Sydney with colleagues from the Faculty of Veterinary Science and the Australian Quarantine & Inspection Service, has been awarded the Most Commendable Paper prize by the Australian and New Zealand

College of Veterinary Scientists. Their paper identifies the source of an unusual outbreak of granulomatous peritonitis in a large flock of merinos in the Northern Tablelands of NSW. Yellow lesions were found on the meat of these sheep when they were slaughtered, confounding meat inspectors and pathologists alike. However, one vet got suspicious of injectable selenium supplements that had been given to the flock. Microscopy in the AMMRF at the University of Sydney was able to characterise the lesions, finding high levels of selenium in the granuloma cells, confirming the supplements as the cause of the problem. Although the lesions can be removed with no threat to human health, they can cause the meat to be downgraded causing significant financial loss to the producer.

In pastures where selenium is in short supply it is often given as a sub-cutaneous injectable supplement to the sheep. By injecting it under the skin behind the ear, areas of muscle are normally avoided. However it appears that in this case contractors wrongly injected the selenium into the body cavity where it caused the unsightly lesions on the surrounding muscle, only becoming obvious when the animals were slaughtered. Instead of increased profitability through improved flock health, inappropriate administration of the supplement has led to financial loss through downgraded carcasses.

Jeffrey Henriquez did a lot of the microscopy work on this project while still a masters student. He has now started a PhD with A/Prof. Filip Braet at the University of Sydney with this award already under his belt. ■



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EXECUTIVE DIRECTOR'S COLUMN

At the end of May, the AMMRF team gathered on the Sunshine Coast for a strategic planning and outlooks meeting. It was the first such meeting since 2009 when we gathered in Fremantle. Our 2012 meeting was extremely positive with strong buy-in and participation from across the Facility's nodes, Linked Laboratories and Linked Centres. One of the key issues that we discussed most enthusiastically, was the successful integration of our online training resource, MyScope (www.ammrf.org.au/myscope/) to many user-support activities. This online

remaining modules and work with our users to ensure that the tools are well integrated into the user-experience.

Another particularly positive aspect of the meeting was our planning around future microscopy technologies based on the developments in the field, and the things that our users are telling us that they wish to achieve in their research. This is important information as it allows us to be primed and ready to seize funding opportunities when they arise.

The AMMRF leadership, and particularly our board, is also working in earnest to define



resource was developed with funding from the Australian Learning and Teaching Council to enhance the training of our current and potential future users. The applications of this wonderful project were far more diverse than originally anticipated, and approaches to integrating it further into our practice are continually arising.

The MyScope module for transmission electron microscopy will soon be live and we are very proud of where the initiative is heading. The ongoing efforts of Dr Bronwen Cribb and her band of coordinators in making this project a success, is a credit to them, and to all of us. Our next steps are to complete the

potential mechanisms that could enable the AMMRF to continue to operate out to a longer time-horizon. This is exciting news for our users and staff because, of course, it is one thing to build up a facility such as ours, but quite another to ensure that it can sustain itself and flourish in the long-term. There are now at least a couple of scenarios that we are planning for and there is strong commitment to keep the team together and to continue to "go forth and magnify!"

Prof. Simon P. Ringer
AMMRF Executive Director and CEO

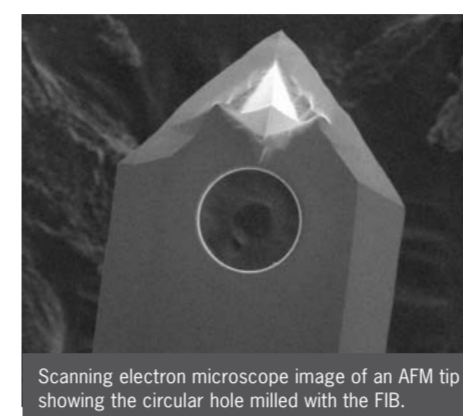
Carbon's got it covered

USyD A team from the University of Sydney, Harbin Institute of Technology and Peking University in China has carried out work in the AMMRF at the University of Sydney to show just how much force it takes to pull a grafted carbon nanotube (CNT) off a carbon fibre. This configuration, where carbon nanotubes are grafted onto carbon fibres, is typical of that used to strengthen composite materials. The force measurements were done inside a scanning electron microscope by using an atomic force microscope tip connected to a micromanipulator. The tip was glued to the end of each CNT in turn and then pulled until the CNT detached from the fibre. This study has shown that the strength of the graft is much less than the strength of the CNT itself, leaving much

scope for improved grafting methods to increase the strength of these materials. The work has been published in the journal *Carbon* with an image from the study gracing the cover. ■



RESEARCH



Scanning electron microscope image of an AFM tip showing the circular hole milled with the FIB.

Calibrating cantilevers for AFM

SARF Precise force measurements can be made using atomic force microscopy (AFM) but to do this it is essential to know accurately the stiffness of AFM cantilevers. This ensures the repeatability of measurements and, if the cantilever is being used as a sensor, it allows accurate determination of added mass. AMMRF staff Dr Chris Gibson (Flinders University) and Len

Green (University of Adelaide) have worked closely to develop a new calibration technique for accurately determining the stiffness of AFM cantilevers.

The technique involves measuring the change in resonant frequency of the cantilever after a small amount of cantilever mass has been removed. The focused ion beam (FIB) was used to remove a precise amount of material

from specific locations on the cantilever. The calibration technique was compared to other methods and is one of the most accurate yet developed. Significant improvements were also made to the applicability and accuracy of two already established methods. This work was recently published in *Nanotechnology* and was picked up by *Nature Methods* as a Research Highlight. ■

COMMUNITY



The definitive Atom Probe Microscopy

A new textbook published by Springer has hit the shelves. Written by four AMMRF atom probe experts, Prof. Simon Ringer and A/Prof. Julie Cairney (above), Drs Baptiste Gault and Michael Moody, it gives practical descriptions of the latest atom probe analysis techniques and their application to materials science and

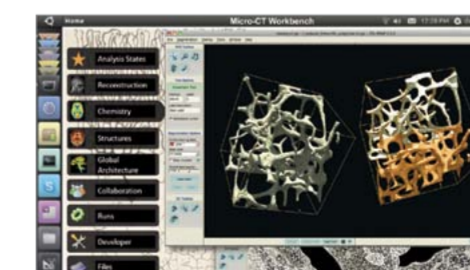
engineering. Two of the authors have now moved on to academic roles at McMaster University in Canada and the University of Oxford underlining the AMMRF's expertise in this field and the importance of the flagship atom probe instruments, not only to Australia, but to the international research community. ■

The Characterisation Virtual Laboratory (CVL)

The CVL will be a cloud-based eResearch environment for analysis and visualisation of multi-modal and multi-scale data. It is being driven largely by the fields of neuroimaging, structural biology and energy materials. By integrating existing analysis and visualisation tools with high-performance computing and data storage facilities, it will facilitate research collaborations.

Development of the CVL kicks off this month with the AMMRF developing specialised 'workbenches' in the areas of atom probe tomography and X-ray microtomography. These virtual workbenches will allow users to analyse, visualise and manage their data.

The ANU and Sydney nodes of the AMMRF have partnered with Monash University and several other institutions to develop the Characterisation Virtual Laboratory (CVL) as part of the National eResearch Collaboration Tools & Resources (NeCTAR) project. NeCTAR is part of the Super Science initiative and financed by the Education Investment Fund (EIF). ■



International spintronics conference (WUN-SPIN 2012) hosted in Sydney

The 4th World Universities Network International Conference on Spintronics was held in July at the University of Sydney. 140 researchers from more than 20 countries came and presented their work, which will heavily impact the future of ICT. Plenary speakers Prof. Hideo Ohno from

TECHNIQUE

What is Transmission Kikuchi Diffraction?

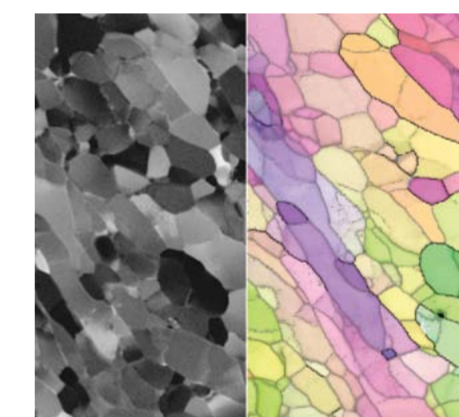
Electron backscattered diffraction (EBSD) has, for many years, been the technique of choice for the rapid characterisation of microstructure and microtexture in the scanning electron microscope (SEM). However, the ongoing push into the truly nanoscale world has left EBSD struggling: it has become harder to resolve

managed to improve on the original so that it is now possible to characterise exceptionally challenging samples at high speed with sub-10nm spatial resolution. The sample is kept horizontal at a short working distance in the SEM, and the diffraction pattern is projected from the lower surface of the electron transparent region and collected using a standard EBSD detector. The short acquisition times (typically 10–25ms), enable orientation mapping of over 100 μm² with measurement spacing in the 2–20nm range.

These advances will be published in the September 2012 edition of *Ultramicroscopy*, and have resulted in a more accurate name for the technique, namely transmission Kikuchi diffraction in the SEM (SEM-TKD).

Early applications of SEM-TKD have focused on aluminium alloys deformed using high pressure torsion (HPT), work done in conjunction with researchers at the University of Sydney's School of Aerospace, Mechanical and Mechatronic Engineering. This work has been accepted for publication in *Materials Science and Engineering A* (Tugcu et al., 2012). Further work has looked at the analysis of samples that have been deformed at room temperature: the extreme density of dislocations coupled with the ultrafine grain size renders conventional EBSD analyses almost impossible, yet SEM-TKD has enabled detailed characterisation of these samples at a scale previously only achieved using TEM.

Additional benefits of the technique are the ability to measure simultaneously the chemistry, using EDS, and the high-quality dark-field imaging that is possible using forescatter detectors mounted on the EBSD detector. This is sure to establish itself as a commonplace characterisation technique for all researchers involved with nanomaterials. ■



Oriented dark field technique of a deformed Al-Sc alloy collected using a conventional EBSD forescatter detector (left) and SEM-TKD orientation map (right). The field of view is approximately 5 μm across and in the SEM-TKD image, the step size is 20nm. Sample and data courtesy of Katja Eder.

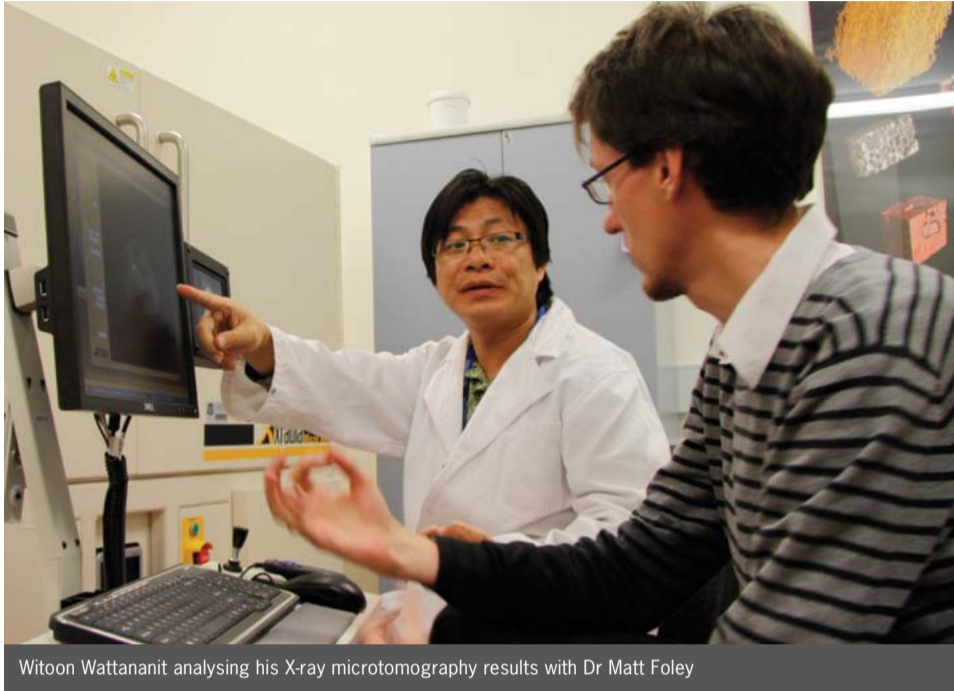
features of interest as grain sizes dip into the realms of a few hundred or even a few tens of nanometres. Work published earlier this year by researchers at the National Institute of Standards and Technology in the US showed how a conventional EBSD system in the SEM could be used to measure diffraction patterns from electron-transparent TEM samples, with resolutions down below the ten-nanometre level. They called this transmission-EBSD.

This approach has been successfully taken up by technical staff and researchers in the AMMRF at the University of Sydney and is now being routinely used to characterise a wide range of nanomaterials. Dr Pat Trimby has been optimising the technique and has

progress of this burgeoning technology and some exciting new international collaborations were forged that depend on AMMRF infrastructure. WUN-SPIN 2012, hosted by Prof. Simon Ringer and Dr Rongkun Zheng, was sponsored by the AMMRF. ■

COMMUNITY

Thai visitors take home new-found expertise



Witoon Wattananit analysing his X-ray microtomography results with Dr Matt Foley

The AMMRF welcomed two Thai scientists, Witoon Wattananit, from Silpakorn University, on a two-week visit to the University of Sydney node and Man Tuiprae from Chiangmai University on a nine-month visit to the University of New South Wales node. Witoon came to get an inside view of the management and operation of a busy core characterisation facility from a user's perspective. While Witoon was in Sydney, with assistance of AMMRF staff, he undertook a project to examine tiny fresh water crustaceans by using transmission electron microscopy and X-ray microtomography. He collected good images and data that he will analyse further back home. The real impact of

his visit though, is the new found knowledge and experience that he has taken back home and will implement at Silpakorn to manage users and operate their characterisation facility.

Man Tuiprae, a materials scientist, came to analyse hard, wear-resistant coatings. Aside from the acquisition of experimental data, Man was also able to learn a range of new experimental methods, such as focused ion beam microscopy and transmission electron microscopy, not readily available in his homeland. Man not only gained considerable microscopy experience but also enjoyed his extended stay in Australia. ■

STAFF NEWS

UWA **Mr Francesc Tinto**, the new Technical Operations Manager, will look after instruments and facilities at Centre for Microscopy, Characterisation & Analysis (CMCA).

Ms Alysia Buckley has joined the CMCA team to provide support to optical, confocal and flow cytometry areas.

Dr Laure Martin has joined CMCA's team through the ARC Centre of Excellence in Core to Crust Fluid Systems. She will be working closely with the AMMRF's flagship ion probe team with a particular focus on the IMS 1280.

Dr Rong Liu will be providing support to researchers making use of the AMMRF flagship ion probe facilities at the CMCA.

USyd **Lisa Abraham** will be responsible for operational activities of the the Australian Centre for Microscopy & Microanalysis (ACMM) in her new role as Administration Manager.

Paul Xu is the ACMM's new light and optical specialist and has expertise in the application of fluorescence in both the life and physical sciences.

Bec Potter will be the first port of call for all ACMM users in her new role as Administrative Assistant.

Curtin **Dr Cristina Talavera** has joined the SHRIMP facility, John de Laeter Centre for Mass Spectrometry.



The AMMRF is funded by



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Government of South Australia



Irish visitor at UQ

Dr Ian Reid is the Research Manager for the Nano Imaging and Material Analysis Centre at University College Dublin. He is setting up a cryo-electron microscopy lab in his Centre and wanted to learn more about the technique and what is required to support it. He found out about the expertise in the AMMRF at the University of Queensland and won a Short Term Travel Fellowship from Science Foundation Ireland specifically to visit our facility. During the four weeks he spent in the UQ labs, and with help of the knowledgeable staff, he learnt the secrets of good specimen preparation, microscope operation, data analysis and lab operation. He was most impressed with the AMMRF facilities and appreciative of the help he received. ■



Self-portraits in the SEM



Self-portraits are a familiar form of artistic expression but one Sydney artist, known only as SWPA (pictured above), has been approaching the idea somewhat differently. He has created images of parts and products of his own body in the scanning electron microscope at the AMMRF at the University of Sydney. An exhibition of the works was recently on display at the Verge Gallery at the University, introducing a new audience to the world of microscopy.

Of his use of SEM the artist said, "the exciting thing about microscopy is the unexpected nature of discovery. By doing research, one can get an idea of the surface topography, however once on the microscope everything changes; worlds are discovered within worlds. Shifting your field of view slightly or changing magnification can present a vastly different landscape, scenes that no level of pre-visualisation could reveal or predict." ■

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Editorial enquiries:

Prof. Simon P. Ringer
Executive Director & CEO
Ph: 02 9351 2351

Email: simon.ringer@ammrf.org.au

Dr Jenny Whiting
Marketing & Business Development Manager
Ph: 02 9114 0566

Email: jenny.whiting@ammrf.org.au

ammrf.org.au