CSEM's

Materials Monthly

September 2003 — Making materials matter

HARE-brained success

Pushing plasma to produce new materials

Creating and modifying thin films using plasma is common in many manufacturing processes. Plasmas are used in the production of anti-reflective coatings on sunglasses, TV screens and windows; the manufacture of silicon chips used in computers; and the creation of waveguides used for optical-fibre communications.

▲▲ Prof Rod Boswell and Dr Christine Charles with one of SP3's latest ventures — a reactor for building electrodes for use in hydrogen fuel cells.

However, the potential of using plasma processing to produce new materials is far from fully realised. Indeed, ongoing research by scientists at the Space Plasma and Plasma Processing group (RSPhysSE) is revealing there's a world of new materials being opened through a greater understanding of how plasma processing works.

"Plasma processing lies at the interface between plasma physics and solid state physics," says Prof Rod Boswell, leader of the Space Plasma and Plasma Processing group more colloquially referred to as SP3. "We're still trying to understand the basic science of plasmas, how they form and how they can be applied. It's exciting stuff because we're really pushing into areas about which little is known."

Introducing HARE

At the heart of much of the group's success has been their development of a plasma assisted reactive evaporation device known as the Helicon Activated Reactive Evaporation (HARE) system. It combines an evaporation source (an electron-beam evaporator) with a high-density plasma source (Helicon plasma source) so that the evaporant material is transported through the plasma source.

SP3 has demonstrated that plasma enhancement of a simple electron-beam vapour deposition system has a number of advantages. It allows for greater control of the properties of the deposited film. The passage of the evaporant material through the plasma assists with its bonding to the target substrate and facilitates the growth of a more uniform film. In addition to this, HARE allows for virtually any material to be evaporated allowing for the fabrication of a wide range of new exotic thin films. It has the ability to deposit oxides, nitrides and carbides, especially of silicon, and has applications in microelectronic and optoelectronic device manufacture and the production of hard and corrosion resistant coatings.

It can also implant ions into silicon wafers. By using a specially designed pulsed high-voltage

(Continued on page 2)

▲▲ "SP3 has the capacity to move its research in different directions as new opportunities present themselves," says Prof Boswell

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HARE-brained success

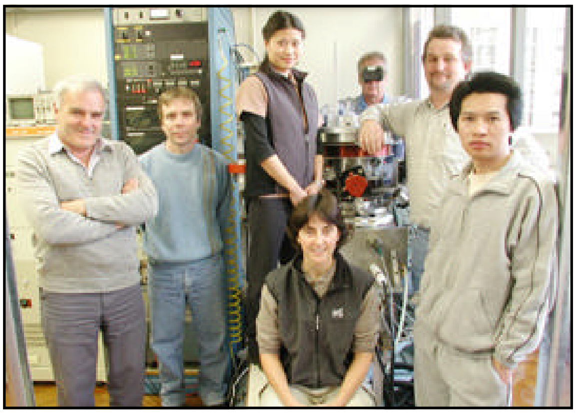
(Continued from page 1)
power supply, ions from the plasma can be attracted and implanted into silicon substrates. The group has demonstrated that germanium ions can be implanted into crystalline silicon wafers at high doses. This novel system provides a very cost-effective way of implanting large areas very quickly without using expensive ion accelerators.

Problems with hydrogen

One important breakthrough in recent years has been the use of the HARE system to produce germanium-doped silicon oxide films that aren't contaminated with hydrogen atoms. The presence of hydrogen in these thin coatings causes big problems because it absorbs certain wavelengths of light that limit their capacity to be used in optical devices used in information systems (see box below).

Unfortunately, keeping the thin film free of hydrogen is quite a challenge. Hydrogen is present as a trace contaminant on both the silicon substrate and the 'pure' silicon and germanium evaporant material. It gets incorporated into the thin film during and after the deposition process unless special steps are taken.

To get rid of it, the researchers first bake the silicon substrate and the plasma system with an argon plasma to clean away any contaminants on its surface. After 20 minutes of this, small pieces of commercially available pure silicon and germanium are first melted and then evaporated. The melting process is done within an oxygen and argon plasma for another 20 minutes to eliminate any traces of hydrogen



◀◀ The team working on HARE and hydrogen-free waveguides.

in the raw silicon and germanium. By increasing the helicon plasma power and the substrate bias during the deposition process a high-density film is produced that will not absorb any hydrogen after the process is complete.

The result is a material that is well suited for use as gratings or planar waveguide devices. Most importantly, the technique can be geared up for commercial processing.

New frontiers in plasma processing

The work on HARE and plasma deposition of waveguides is continuing and has formed the basis of several commercial patents and processes. SP3 is continuing to study how the plasma deposition process can be modified to produce different effects.

"And it's not just the plasma itself that interests us," says Dr Christine Charles, who has been leading the work on HARE. "It's their interactions with materials and the materials themselves that are a large part of our focus. Currently we're doing a lot of work on the stresses that can be built into the thin layers we're depositing. This involves studies on both extrinsic stresses that result between different layers with different properties, and the intrinsic stresses that can be produced by doping their crystal structures with different ions. Different stresses yield different properties."

Dr Charles believes their approach is yielding some important insights on the generation of plasmas.

"Most commercial applications of plasma processing treat the plasma source as a bit of a black box," she says. "You plug it in and switch it on and work with whatever plasma is produced. Our equipment and collective expertise allows us to pull the black box apart, and explore the plasma in all its guises. It's a feature of the SP3 group that we perform basic science while simultaneously solving real world problems. Our ability to produce hydrogen-free waveguides is an example of this."

SP3 is currently involved in several exciting projects including the development of plasma based focused ion beam and the construction of a high density plasma sputter system that will modify porous carbon membranes to contain small platinum aggregates. These films could serve as electrodes in hydrogen fuel cells, another industry that looks set to take off in the next decade. The latter research is a collaborative programme with Dr Pascal Brault of the University of Orleans in France.

"We're a small group but we have a depth of experience and flexibility to take our research on plasma processing in any direction as new opportunities arise," says Prof Boswell of the SP3 lab. "This is important in an area that is opening up new areas of potential all the time. I wouldn't like to predict what we'll be working on in five years time but I'm confident it'll be both interesting and relevant to Australia's technological future."

More information: Rod.Boswell@anu.edu.au / http://sp3.anu.edu.au/

Unplugging the information bottleneck

The information revolution is straining our telecommunications infrastructure. The more we get connected, the faster our computers run and the greater our demand for data.

Encoding information into beams of light and firing them down optical fibres has worked well, but demand is quickly outstripping capacity. How can we transmit more data using current systems?

To send more information, you can encode it on different wavelengths of light, but optic fibres absorb different wavelengths at different rates. The presence of hydrogen atoms in the fibres, which are made of silicon dioxide, causes light at 1400 nanometres to be absorbed rapidly.

Materials scientists have found a way to produce optical fibres free of hydrogen atoms. While these hydrogen-free optical fibres can transmit much more information, the optical devices that transmit and receive the multi-wavelength signals, known as planar array wavelength gratings, suffer the hydrogen problem.

Planar array waveguide gratings are made on silicon wafers using plasma deposition that builds them layer upon layer. The unit is less than one millimetre thick and a few centimetres across. Hydrogen atoms become trapped during the deposition phase.

Materials scientists in the Space Plasma and Plasma Processing group led by Prof Rod Boswell are pioneering new ways of producing hydrogen-free planar waveguides using the HARE system (see main story). The technique is still being refined, however its success promises to unplug a major bottleneck on the information highway and give Australia a competitive edge in a booming industry.

Materials Technology

Introducing the FIB SEM

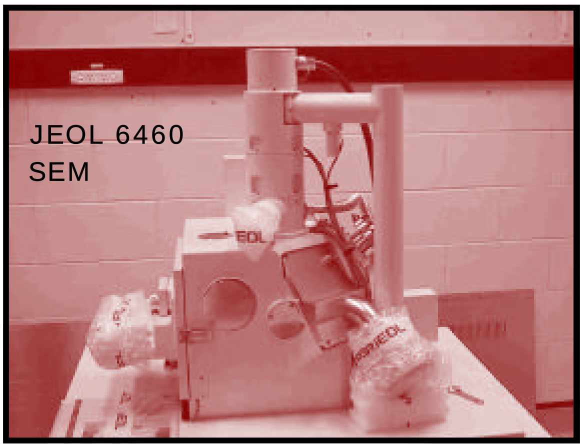
ANU's latest tool for the nanotechnology frontier

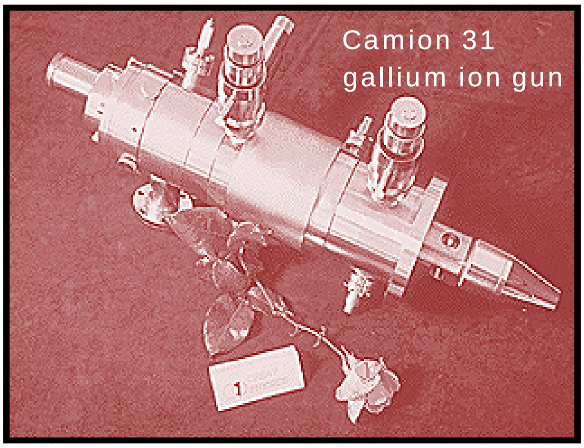
What do you get when you attach a state-of-the-art focused ion beam (FIB) to the latest in scanning electron microscopes (SEM)? A nanoscale Swiss Army knife! Well, not exactly but it's the same idea – a multi-purpose, highly flexible tool for manipulating and working materials at the scale of nanometres. And that's just what's been unpacked at the ANU Electron Microscopy Unit.

▶ **deposition:** The instrument uses a precursor gas injection system that allows for very precise deposition of conductive or non-conductive materials (in the ANU instrument, tungsten). In conjunction with the ablation function this can be used in the repair or construction of micro or nanoscale structures.

Greater than the sum of its parts

That's the FIB, what about the SEM? Combining the devices enables all the operations just described to be monitored with the SEM in real time and at high resolution. There's no need to transfer the sample to other electron microscopes to view what you've produced. By adding a cold stage you increase the flexibility of the set up again. It will allow you to work with beam-sensitive materials including organic polymers and biological samples. There are few set ups like this in the world.





It's come a long way

It's taken a lot time and effort to acquire but hopes are high that the new FIB/SEM will keep ANU at the leading edge of nanotechology for many years to come. Funded by the ANU, an ARC Linkage Infrastructure grant, and UNSW, the FIB/SEM has been championed by Sally Stowe (EMU), Tim Senden (Applied Maths) and Paul Munroe (UNSW). It's taken a year to get here, and in that time the device has travelled from Japan, where the scanning electron microscope was manufactured, to France, so the focused ion beam could be attached, and finally to Australia where a cold stage is being added at ANU. The configuration is an Orsay Physics "Camion 31" gallium ion gun on a JEOL 6460 SEM, with 3-gas injection.

So, what's all the fuss about? The FIB produces a fine beam of 30 KV gallium ions that can be scanned across a target sample with a resolution of less than 10 nm. There are three modes of operation, each with a myriad of applications.

▶ **imaging:** ions and secondary electrons emitted when the beam scans the sample may be used to form images with information on shape, atomic composition and crystal orientation. The ion beam is not significantly affected by magnetic fields so it's an excellent technique for imaging magnetic materials.

▶ **cutting:** the beam ablates the target in a very controllable way, either programmed or freehand. It can be used to progressively reveal 3D structure, to carve out nanostructures, and to prepare specimens for observation in other microscopes faster, more precisely and with much less mechanical stress than other methods.

What can it do?

And what can the new FIB SEM do? There's not enough space here to list potential applications, but here are some of the projects currently being worked on that will benefit from the new device:

▶ sample preparation: the FIB SEM offers multiple advantages over existing techniques for sectioning materials for use in the transmission electron microscope. This will especially benefit the preparation and study of materials including semi-conductors, opto-electronic devices, ceramics, polymers, biomaterial composites and crystalline aggregate minerals.

▶ microfabrication: FIBs are used by industry to improve the yield of expensive large multilayer wafers by burrowing down to a fault, adding or removing material and reconstructing the top layers. The FIB can also selectively implant ions; fabricate high temperature superconductor-metal junctions; fabricate and tune quantum wells; and construct and modify scanning tips for atomic force microscopes.

▶ 3D reconstruction: FIB allows ablation of a structure so that progressive slices can be imaged (by the SEM).

▶ preparation of highly polished surfaces: Stress free FIB polishing will open up new fields of imaging that have never been available in the past, especially with soft or inhomogenous material.

More information: Sally Stowe, ANU EMU (STOWE@rsbs.anu.edu.au)

Opportunities

Professional & personal

Professional Way provides you with a personalised, free email listing every Wednesday evening of upcoming professional development activities in Canberra. The email you receive only lists events in the areas you choose. It isn't a generic newsletter — it's personalised to your interests. The event listing includes

- conferences & seminars
- lectures & talks
- workshops & training courses
- exhibitions & trade shows
- new product launches
- calls for papers.

To receive the e-newsletter (it's FREE), go to **http://www.professionalway.com.au/index.html**

ProfessionalWay

Your profession, your events...by email™

Building with straw

CSIRO has just released a guide for the growing band of enthusiasts who are building homes with straw bales. The author, Dr Murray Hollis, says, "The guide details the basics of straw bales, their manufacture and properties, their preparation and use in building, and details one of the many techniques used".

More info:
http://www.csiro.au/index.asp?type=mediaRelease&id=Prstrawbales

Latest in synthetic metals

ICSM 04 Wollongong, Australia

In June/July 2004, a thousand scientists from Australia and around the globe (including several Nobel Laureates) will spend 5 days at ICSM 2004 at the Uni of Wollongong, Australia, discussing the latest developments in Synthetic Metals with world leaders in the field.

Synthetic Metals such as conducting polymers and carbon nanotubes are finding application in areas as diverse as artificial muscles, electronic noses, plastic solar cells, light emitting diodes, corrosion inhibition, biological and chemical sensors, electronic textiles and nerve cell communications.

The conference theme is: *"SYNTHETIC METALS: The role and impact of nanoscience and nanotechnologies".* Sub-themes will include: molecular electronics, inherently conductive polymers, organic magnets, organic spintronics, advanced coatings, nanotubes and nanostructures, biofunctional materials, materials for advanced display technologies, energy storage materials, transport phenomena in single molecules, electronic fibres, and organic conductors & superconductors

ICSM 2004 is the first time this important conference will be staged in the southern hemisphere and as such is an excellent opportunity for Australian researchers to attend this prestigious international event.

ICSM2004 is preceded by the ICSM2004 Satellite Research Symposium: "New Materials for Energy Conversion and Storage", Queenstown, New Zealand. 23 -25 June, 2004.

More information: http://icsm2004.uow.edu.au

Diary: conferences and seminars

◀◀✧▶▶ **New Materials and Complexity** — **3-7 November**
incorporating the Australian fundamentals of soft matter workshop
Canberra and Kiola (NSW), http://wwwrsphysse.anu.edu.au/newmaterials

◀◀✧▶▶ **2nd International Symposium on Ultrafine Grained Structures** — **11-13 November**
Geelong, Victoria, http://www.mateng.asn.au/ISUGS/

◀◀✧▶▶ **ACOLS 03** — **1-4 December**
Australasian Conference on Optics, Lasers and Spectroscopy 2003.
Uni of Melbourne, Melbourne http://www.swin.edu.au/bioscieleceng/soll/ACOLS03/

◀◀✧▶▶ **Photons@work** — **27 Jan—5 Feb 2004**
Australian Synchrotron Summer School, ANU, http://wwwrsphysse.anu.edu.au/sync.school/

◀◀✧▶▶ **Aust Conference on Microscopy and Microanalysis 18** — **2-6 Feb 2004**
Geelong, http://www.deakin.edu.au/events/acmm18/

◀◀✧▶▶ **17th Australian Geological Convention** — **8-13 Feb 2004**
Hobart, http://www.17thagc.gsa.org.au/

◀◀✧▶▶ **ICSM 2004** — **28 June—2 July 2004**
International Conference on the Science and Technology of Synthetic Metals,
Uni of Wollongong, http://icsm2004.uow.edu.au/

◀◀✧▶▶ **ACCM-4** — **6-9 July 2004**
Fourth Asian-Australasian Conference on Composite Materials
Sydney, http://www.camt.usyd.edu.au/accm4/

Materials Grab Bag

Wood odyssey

What do you do if you're visiting Australia and you've got a few spare days? If you're Phil Evans, CSEM's former director and wood fanatic, you race off into the Tasmanian wilderness in search of a rare tree in order to characterise its wood.

In July, Phil travelled from Canada to Canberra to conduct a wood science course at the ANU School of Resources, Environment and Society. A former colleague, Dr Roger Heady, asked Phil if he'd be interested in joining him on a whiplash trip to Tasmania to take wood samples from a little known conifer called *Diselma archeri*. It's closely related to *Callitris* (native cypress) which Roger has spent many years studying. *D archeri* is only found in Tasmania where it occurs at high altitudes. Its wood anatomy has only been partially described.

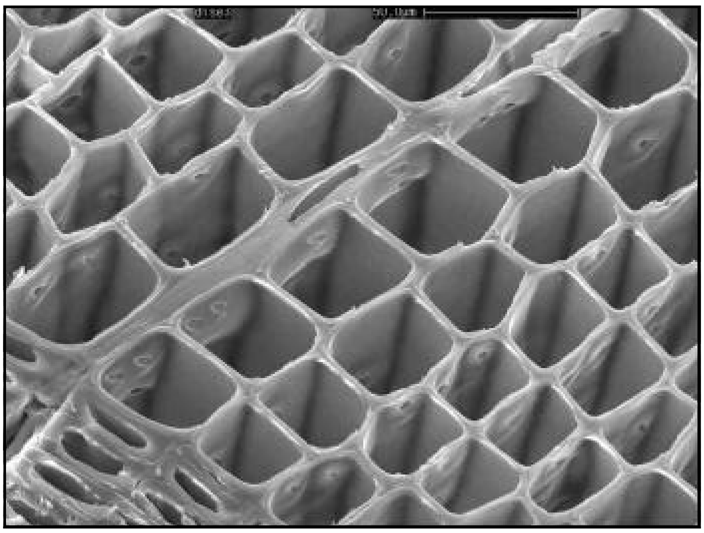
Naturally, Phil jumped at the opportunity. So, on Saturday, 19 July, Roger and Phil flew into Hobart. From there it was a rapid drive to Queenstown on the west coast of Tasmania. Next day it was up an old bush track to Mount Dundas, some 20 km north of Queenstown, and then an uphill march for two and a half hours along a track marked by bits of red tape tied to the trees. It was wet and cold, but the weather remained bright and sunny. No fog thank goodness.

Specimens of *Diselma archeri* were growing near the peak in a sodden, moss-covered rainforest. Five millimetre cores of the wood were taken so as to cause only minimal damage to the tree sampled.

The walk back took 3 hours (and included wading through a freezing creek). Then it was a dash back to Hobart where Roger headed for Canberra while Phil jetted on to Canada. You'd have to be keen.

Roger is now undertaking the first electron microscope studies of the wood. The results will be published soon.

More information: Roger.Heady@anu.edu.au



▲▲ A transverse section through the wood of *Diselma archeri*. The cells (tracheids) are thin-walled. The bordered pits, which interconnect tracheids, can be seen inside the tracheids. The narrow cells in the lower left of the image are cells of latewood – formed in winter when growth is slow. (Photo by R Heady.)

Nine new centres

Nine new ARC Centres of Excellence have been selected for funding totalling $47.1 million over the next five years. Two of these are based at the ANU. Three centres (including one of the ANU centres) will be working in the area of materials science and engineering. The centres are:

- the **ARC Centre for Complex Dynamic Systems and Control** at the University of Newcastle;
- the **ARC Centre for Functional Nanomaterials** at the University of Queensland;
- the **ARC Centre for Nanostructured Electromaterials** at the University of Wollongong;
- the **ARC Centre for Perceptive and Intelligent Machines** at Monash University;
- the **ARC Centre for Structural and Functional Microbial Genomics** at Monash University;
- the **ARC Centre for Complex Systems** at the University of Queensland;
- the **ARC Centre for Genome-Phenome Bioinformatics** at the University of Queensland;
- the **ARC Centre for the Kangaroo Genome** at the Australian National University; and
- the **ARC Centre for Solar Energy Systems** at the Australian National University.

More information: www.arc.gov.au/grant_programs/arc_centres/default.htm

Composite durability & the aerospace industry

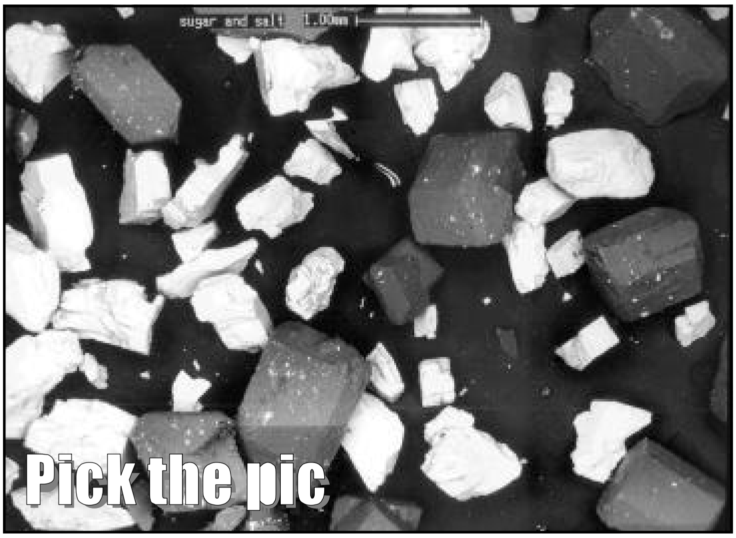
At our most recent CSEM seminar, Dr Jonathon Hodgkin from CSIRO Molecular Science spoke on various CSIRO studies on the long term durability of commercial aerospace composites. He compared standard techniques of determining durability using ageing exposures under highly accelerated conditions with cheaper chemical testing of the structural changes in the matrix resins of the composites. He's pictured above (on the left) talking to engineering students at the after-seminar drinks.

the backpage

MM webspotting

Plasma processing

- **Perspectives on plasma**
  http://www.plasmas.org/photo.htm
- **Electron interactions with plasma processing gases**
  http://www.eeel.nist.gov/811/refdata/
- **Thermal plasma processing (INEEL)**
  http://www.inel.gov/env-energyscience/materials/thermalplasma.shtml
- **Los Alamos Plasma Processing Research Facility**
  http://www.lanl.gov/orgs/ibdnew/usrfac/userfac39.html
- **Lab for Plasma Processing of Materials (Maryland)**
  http://www.ireap.umd.edu/ppm/laboratory.html
- **Internet sites for plasma physicists**
  http://www.ipr.res.in/~saroj/Plasite1.htm
- **Plasma basics**
  http://www.ncpst.ie/plasma



▲▲ The image above shows sugar grains mixed with salt grains. But which is which?

With a basic light microscope you'd be hard pressed to pick them apart but in this image, produced using backscattered electrons, the differences are crystal clear - indeed, almost black and white.

Backscattered electron images can yield instant compositional information about a specimen because atoms low in the periodic table reflect fewer electrons of an electron beam than elements higher in the periodic table.

Therefore, sugar composed of atoms of carbon and oxygen would appear darker than salt composed of atoms of sodium and chlorine (elements that are higher up in the periodic table). Thus, in this image, the sugar grains are dull grey while the salt grains are bright.

Dr Roger Heady captured this image using one of the scanning electron microscopes at the ANU EMU.

For another image taken by Roger, see page 5.

Elemental humour from RSC

(courtesy of the RSC News letter)

What do you do with a dead chemist? Ba

What does a doctor do with a sick chemist? He

What did the cowboy chemist do with his horse? Rh

What do you do if you can't swim? Zn

What element doesn't belong to you? None of your Bi

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www.anu.edu.au/CSEM