Pioneering work by researchers at the ANU Laser Physics Centre is demonstrating how the University’s new Focused Ion Beam can be a powerful tool for chiseling out nano-photonic structures.

When the ANU Electron Microscopy Unit purchased its new Focused Ion Beam (or FIB) last year*, it knew it was getting a fabulous tool for the preparation of samples for transmission electron microscopy. The powerful beam of focused gallium ions could quickly and easily whittle samples down into thin sections of 100 nm or less that were difficult to produce using other techniques. (* See the September 2003 issue of Materials Monthly.)

Whilst it was known that focused ion beams could also be used to cut smooth nano-scale patterns in materials such as silicon, it was never thought that the ANU FIB would achieve a lot in this area because it wasn’t set up for this use. Indeed, there are several FIB facilities around Australia and none of them are used for nano-patterning which is a specialised task requiring specialised software and skills.

The ability to create patterns on the nanometer scale is, however, increasingly relevant to technologies such as photonics – one of the major topics of research in the Research School of Physical Sciences and Engineering (RSPSE).

“Given the rapid evolution of photonics and the strong drive to miniaturise optical processing devices down to the nano-scale, we realized there were powerful reasons to try and harness the FIB to cut out nano-photonic structures.”

**FIBs for photonics**

One of those reasons is that the FIB produces a precision beam of gallium ions that can be scanned across a target sample with a resolution of around 10 nanometres. If given the right instructions, it can cut a target material more precisely and smoothly and with much less mechanical stress than other methods.

Another reason is that it can cut a pattern or structure directly into the material of interest under computer control. The more traditional technique is a multi step process in which an electron beam is first used to print the desired patterns into photo resist. This pattern is then transferred to the working medium (eg silicon) using dry etching in a machine such as a reactive ion etcher.

There are many steps in this process and if you get some parameter wrong, such as the resist thickness or the parameters in the etching step, then the process is ruined but you might not know this until the very end. By comparison, the FIB lets you see instantly what you did wrong and allows you

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**Pushing a FIB**

(Continued from page 1)

...to try again right away. It’s simpler, more versatile and faster than the conventional approach and what’s more can be applied to almost any material.

“It’s the nanotechnologists’ ‘chisel’,” says Professor Luther-Davies. “However, as with any tool, it’s only as good as your ability to control it. In the case of the FIB we weren’t sure if we could use it at all given its initial configuration and the software available to drive it. But it was worth trying so we’ve let one of our young researchers see if he could tame the beam sufficiently to make some nanophotonic structures.”

**Taming the beam**

Darren Freeman is the young researcher they’ve let loose. He’s a doctoral student with a background in electronic engineering. In the short time he’s been ‘playing’ with the FIB he’s amazed everyone with the degree to which it can be used to cut patterns and shapes. The work he’s been doing is part of a project at ANU being done in conjunction with the ARC Centre for Ultrahigh bandwidth Devices for Optical Systems (CUDOS, see box).

“I began by using the available software that came with the FIB to cut a lattice of holes into a silicon wafer,” says Darren. “The holes were approximately 500 nm apart. Because it took me several minutes to calculate where each hole should go in relation to each other, their relative position was pretty rough because the sample tended to move in the time it took to figure out the position of the next hole. This is just one of many problems we need to deal with when cutting patterns that need to be precise at the nanoscale. Also, the holes weren’t particularly neat and didn’t have straight sides.”

It rapidly became clear that the control available with the standard machine was painfully inadequate for nanophotonic patterning so Darren set out to develop new hardware and software capable of rapidly cutting arbitrary patterns into a sample with very high precision. Writing text was a good test and soon a nano-DARREN and a nano-CUDOS had been produced.

“The nominal width of the characters is 0.5 μm and the height is 1 μm,” Darren explains. “The DARREN pattern was written on the smallest aperture but some very bizarre proximity effects were present.” Darren used his nano-DARREN as a door label for a while since he didn’t have a proper one. With letters one thousandth of a millimetre high it’s debatable whether anyone noticed it!

**The real thing**

The next step was to see if the FIB could actually produce devices needed for nano-photronics. The starting point was to make Bragg gratings in a silicon waveguide and this was followed by a series of patterns made in chalcogenide glasses including nano-tapers and a set of sub-micron holes forming a planar ‘photonic crystal’. The gratings are sets of shallow ‘scratches’ that are only around 100 nm deep and extend over a length of 300 μm. These scratches or slots are placed along a waveguide separated by a distance of 222 nm. There are some 1365 slots aligned next to each other to form the grating.

“The purpose of a Bragg grating is to reflect a very specific wavelength of light and transmit the rest,” says Darren.

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“We do this by using a periodic array of reflectors along a waveguide. If the light reflected at each point constructively interferes at the output we see a strong reflection. The structure is quite demanding to produce since any errors in the position of the lines can ruin the optical response. Tests of the device turned up some unexpected things, in fact things that had never been seen before - but that’s a standard part of life when you’re in research!”

The latest patterns are far more impressive and are now starting to provide a real boost to photonics research at the ANU. Photonic crystals are one of the hottest research topics in optics and are made up of arrays of nano-rods or nano-holes patterned into thin film of material like silicon. The pattern has special optical properties and the structures, in fact, mimic similar patterns found in nature. The colour in the wings of many butterflies, for example, is created by nanopatterns rather than pigments (chemicals), which is what we use to create colour.

**Working at the nanoscale**

Photonic crystals really challenge the capabilities of the FIB because the structure has to be regular over a rather large area.

It takes quite a long time to ‘chisel’ these patterns and because the FIB is currently not located in a suitable ‘clean environment’ (see box on the clean room), it and the samples it works on are subject to a range of mechanical and thermal vibrations. These vibrations are imperceptible at the scale of our ‘macro’ world, but have the potential to bury any pattern you’re trying to create at a nanoscale.

“The ‘noise’ can drown out the ‘signal’,“ comments Darren. “Patterns and drawing that can be created quickly may not be affected, but some photonic crystals that might take hours or even days to draw simply can’t be formed.”

So Darren has become a ‘night owl’ working late into the night when everyone else has gone home and the environment becomes as ‘quiet’ as possible. The results have been worth it, and with more night work ahead of him Darren is confident some really exciting devices will soon emerge.

So, while Darren is demonstrating that the FIB has the capacity to create some amazing structures, there are still some formidable challenges to overcome. But then no-one ever said that working at the nano-scale is easy.

More info: Darren Freeman <daz111@rsphysse.anu.edu.au>

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**CUDOS and the ‘great leap forward’**

The Centre for Ultrahigh bandwidth Devices for Optical Systems (CUDOS) commenced operation in January 2003. It’s vision is to develop the experimental and theoretical expertise to design and build linear and nonlinear all-optical signal processing devices, and to miniaturise these, leading to a ‘photonic chip’ believed to be the building blocks for the next generation of optical systems. The miniaturisation is a technical advance that is the photonic equivalent of the great leap forward that occurred in electronics when bulky, inefficient thermo-ionic valves and tubes were replaced by small, cheap, mass-produced integrated circuits and computer chips.

Such miniaturisation will be achieved using a range of novel optics, including photonic crystals, microphotonic structures, microstructured optical fibers, nonlinear photonic materials and will rely on advanced fabrication techniques, new material systems and possibly entirely new principles.

CUDOS is a research collaboration that combines and builds on the established expertise of researchers at the Australian National University, Sydney University, Macquarie University, Swinburne University, the University of Technology, Sydney, and CSIRO. It also builds on research links with photonics research groups at other Australian universities including the Optical Fibre Technology Centre and the Australian Photonics CRC. and with international partners such as Bell Labs and OFS Laboratories in the USA, and CNRS in France.

More info: http://www.cudos.org.au

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**Darren maps Australia at a scale where 1km = 1 nm.**

**Another of Darren’s early test patterns carved into silicon.**

**In this test Darren replaced the holes with spirals with spacings of 1.60 µm. This pattern may become useful in fabricating chiral metamaterials, in which there is a preferred direction of rotation.**
Dung story

Researchers from the Department of Engineering have been burning cow dung to assist East Timorese villagers produce a better quality ceramic for use in water filters.

The researchers in question are Tony Flynn and David Goggin, and the work has been part sponsored and carried out through CSEM. In 2003 they were asked to characterise the clays and ceramics being used by the local women at Manatuto, an East Timorese village located north east of Dili (see April, 2003). The villagers had successfully produced ceramic water holders using the local clays but needed to develop a method for creating a local ceramic that might serve as a water filter to remove silt from local water supplies.

“Our research looked at the possibility of the East Timorese using their own ceramics as filters,” says Tony Flynn. “Unfortunately, our results found that the local clays, by themselves, are too fine. They make good water containers but poor filters. The resulting ceramic contains little void space and quickly blocks up.”

“The challenge then was to see if there was some locally available material that we could add to the clay mix that might make it suitable as a water filter. We’ve tested a number of additives and it looks like we may have found several additives that work well.”

(This aspect of the research was discussed in the February 2004 issue of Materials Monthly.)

“However, the challenge is not just coming up with the right mix for use in a western-style kiln,” says Tony. “We also need to devise a relatively full proof method of firing the ceramics using their traditional techniques. The have access to a kiln but it’s frequently out of action. The traditional East Timorese method, on the other hand, is to heat clay in fires fuelled with dried cow dung. This produces a wide range of firing temperatures, and often produces inconsistent ceramics. To produce ceramic water filters we’ll need to refine the way they build and use their fires to make sure they produce the appropriate range of firing temperatures.”

And how do establish the right kind of fire to build? You go out and collect dry cow dung, build a variety of fires made from different sized lumps of dung and measure the temperatures produced inside. It’s a hands on job.

Towards this end, Tony, David and Rob Gresham (also from the Department of Engineering) visited a friend’s farm near Gundaroo earlier this year to do some testing. They collected dried dung and set up four fires built from different sized lumps of dung (full cow pats and smaller broken down lumps). They then lit the fires and monitored the temperature using a thermocouple placed in a hollow stainless steel tube held some 15 cm above the ground.

“The best results came from the fire using lumps of dung that hadn’t been broken up at all,” Tony said. “In other words, in lumps as they were found. In these fires we found that the internal heat climbed up to some 700°C in ten minutes. A maximum of around 960°C was achieved, and we could sustain temperatures of 850°C for around 20 minutes.

Whether this is sufficient to fire the cylindrical forms that the East Timorese would be producing is the next thing to test,” Tony explained. “My guess is it might not be enough. I think we may have to add some medium sized sticks to attain a slightly higher and more sustained firing temperature. However our first go at the dung fires have demonstrated what’s the best lump size to use and given us valuable data on just hot these fires can be.”

More info: Tony.Flynn@anu.edu.au

It’s a tough job but someone has to do it. (From the right) Rob, Tony and David monitor the burning dung.

The finished product
**Grab bag**

**Chicago, here I come**

Last month the Science Minister, Peter McGauran announced the inaugural intern scholarships from the *Australian Synchrotron Research Program*. Bernt Johannessen, a first year PhD student in the Department of Electronic Materials Engineering, RSPSE, was one of two lucky interns. Bernt explains what the intern scholarships are all about and why they are important to both his research and to Australia.

I’m attempting to structurally characterise copper nanocrystals. To do this successfully I need access to a synchrotron but, at the moment, Australia doesn’t have one. This intern scholarship will allow me to travel to Chicago to familiarise myself with one of the most advanced synchrotrons in the world.

Copper nanocrystals are made by literally pounding a thin film of silica glass (quartz) with copper ions. The result is trillions upon trillions of copper atoms buried inside the silica film. This copper-rich film is then heated to several hundred degrees allowing the copper atoms to precipitate into tiny clumps of copper atoms. This is what we call ‘nanocrystals’ (so named because they are only around 10 nm in diameter) surrounded by a matrix of silica. Tiny crystals of copper atoms like this have completely different properties to bulk copper, and these materials may form important components in opto-electronic devices. However, we’re attempting to do more than just create nanocrystals. By further bombarding the samples with even higher energy ions we are attempting to introduce disorder into the nanocrystals. In so doing we further modify their properties.

Having created and modified the copper nanocrystals, the next task is to describe their local atomic environment (to test how successful we were at introducing atomic disorder). This is best done by studying them using intense X-rays such as produced by synchrotrons. The actual technique is known as EXAFS (Extended X-ray Absorption Fine Structure). When an energetic X-ray photon enters the nanocrystal and interacts with an atom, it is absorbed and in turn a photoelectron is emitted. This electron is emitted radially outwards as a wave, and may or may not experience some form of backscattering from the immediate atomic environment. Such backscattering events can be measured (via subtle variations in the X-ray absorption coefficient) for energies above the absorption energy. These variations enable us to say something about the short-range order of the nanocrystal, including bond-length and coordination number. By comparing these results with those of a bulk copper crystal, we can determine their size-dependent atomic structure.

These experiments can only really be done using a high flux high energy photon beam, obtainable from a synchrotron source. The Advanced Photon Source (APS) in Chicago is rated among the top three synchrotron facilities in the world and the largest in the US. For the first time this year, the Australian Synchrotron Research Program (ASRP), which has strong ties to the APS, has offered two internships to Australian PhD students, each worth $20,000. These internships span three months working with on-site scientists learning how to operate and utilise a synchrotron.

I’ll spend my internship working with Dr David Cookson at the APS from the beginning of September. My primary goal is to learn in more detail how a synchrotron works and also learn a new experimental technique (Small – and Wide Angle X-ray Scattering, SAXS/WAXS). This experience will not only benefit my project, but I will also be the first person to gain hands-on practice with this technique in my group, which I hope will also be a future benefit to my colleagues.

Currently there is no synchrotron in Australia, however there is currently a community of more than 150 research groups utilising synchrotron radiation in their research. These are all going overseas to perform their experiments (e.g. to facilities like the APS or similar institutions in Japan and Taiwan, all accessible through the ASRP). To accommodate an ever-increasing demand in Australia, a state-of-the-art synchrotron is being built in Melbourne ready for operation in 2007. Estimates suggest that by the opening of the Australian synchrotron more than 1200 members of the scientific community in Australia will be accessing the synchrotron. The ASRP internships are meant to encourage, educate and prepare the young local scientists for a new future with a synchrotron in our own backyard.

More info: Bernt Johannessen, bej109@rsphysse.anu.edu.au

**Chapman SEM Courses**

The Steve Chapman (Protrain) SEM courses are happening again in September, this time in conjunction with Anaspec Australia.

**Course 1**

A Basic Introduction to Scanning Electron Microscopy (3 days)
Monday 13th to Wednesday 15th September 2004
The course is based firmly upon the practical side of the instrument, with theory introduced as and when it is required. Cost $300.00 plus GST

**Course 2**

Advanced Techniques in Scanning Electron Microscopy (2 days)
Thursday 16th to Friday 17th September 2004
For scientists who need an introduction to high resolution SEM and energy dispersive x-ray analysis or who have some experience but need to brush up their skills. This is not a course for the SEM novice, however the Basic SEM course should prepare the less experienced. Students are invited to bring along one of their own problem specimens for investigation. Cost $200.00 plus GST

**Or both Courses (5 days)**
Monday 13th September until Friday 17th September 2004.
Cost $450.00 plus GST

More info: Ruth Grinan
Anaspec <ruth@anaspec.co.za>
or
Dr Sally Stowe
ANU EMU <sally.stowe@anu.edu.au>
Rice ceramics

Pictured is a thin section of a ceramic mix being tested by David Goggins as part of his research on porous ceramic filters for East Timorese villagers (see story on page 4). The ladder-like structures are actually openings created by organic matter in the ceramic mix (in this case rice husks). These burn away when the ceramic is being fired giving the ceramic a degree of porosity that also makes them an effective filter for removing silt from water.

More info: David Goggin <goggs79@ozemail.com.au>

Eureka!

Guess what? *Materials Monthly* is a finalist in this year's Australia Museum Eureka Prizes.

The prestigious Australian Museum Eureka Prizes raises the profile of science and engineering in the community by acknowledging and rewarding outstanding achievements in Australian science, engineering and science communication. Science rewarded through the Eureka Prizes covers a broad range of research, innovation, engineering, training and other science-related activities highlighted by individual prizes. Begun in 1990, the Eureka Prizes have grown into Australia’s premier and most comprehensive national science awards.

We entered *Materials Monthly* into the Engineers Australia Eureka Prize for Engineering Journalism category and on the 15 July it was announced that we were a finalist in that section. The winner will be announced at an award dinner in Sydney on 10 August.

More info: http://www.amonline.net.au/eureka/

Exploring materials with ANU

**a Future Materials Industry & Research Workshop**

If you have any interest in materials science and engineering make sure you set aside **Tuesday, 24 August**, to attend Future Materials first research and industry workshop. A galaxy of University’s top materials researchers will present brief rundowns on what they’re doing and what they can offer in the way of materials science and engineering. Come along if you want to know more about the various materials technologies available on campus, and make sure any of your business contacts know about this fabulous opportunity. Lunch will be provided.

The workshop is free but you will need to register. It is being run in the Huxley Lecture Theatre (Leonard Huxley Building 56). **For a full program or to register, please email act@future.org.au by 17 August 2004.**

And while we’re on the topic of Future Materials, check out their new monthly newsletter at http://future.org.au/

**Contacting CSEM**

**Director:** Dr Zbigniew Stachurski / Ph: (02) 6125 5681 / Email: zbigniew.stachurski@anu.edu.au

**Communications:** David Salt / Phone: (02) 6125 3525 / Email: david.salt@anu.edu.au

**Administration:** Tiina Hatherall / Phone: (02) 6125 3525 / Email: tiina.hatherall@anu.edu.au

Fax: (02) 6125 0506, Postal: Department of Engineering (Bld #32), Australian National University ACT 0200

Location: Room E212, Department of Engineering (Bld #32), cnr of North Road and University Ave, ANU

*Materials Monthly* comes out each month. We welcome your feedback and contributions. Please send them to David Salt, Editor, *Materials Monthly*, care of CSEM.

Please let us know if you wish to be added to our electronic or postal mailing lists.

Electronic copies of *Materials Monthly*, useful links and additional information about CSEM can be found at our website: www.anu.edu.au/CSEM