

# Materials Monthly

Making materials matter

January 2002

Measuring

## Hot rock

A new furnace is providing insights on what lies deep below

Scientists at the ANU Research School of Earth Sciences (RSES) are unraveling some of this planet's deepest mysteries thanks to the development of a novel high-temperature furnace designed for use in synchrotron studies. The furnace provides researchers unsurpassed control of not only temperature but also the oxygen environment allowing them to mimic conditions deep inside the Earth.

Much of what is known about the evolution of our planet's interior is based on what we can learn from lavas brought up to the surface by volcanos. An understanding of how elements distribute themselves between the molten lava and the hot, solid rock that is left behind provides valuable information on the composition and evolution of the Earth. So, the aim is to take a small sample of lava, heat it up until it melts, and then study characteristics of various elements. Of particular importance in controlling how an element behaves is its charge or oxidation state.

At RSES, Andrew Berry and Hugh O'Neill are interested in the oxidation states of metal cations in lavas such as basalt. The oxidation states of metal cations are best determined with high intensity X-rays produced by synchrotrons, but until now there was no way of controlling the oxygen environment around the molten basalt while the X-ray measurements were being made. So, they needed to create their own furnace especially for this purpose.

Michael Shelley (Senior Technical Officer at RSES) came to the rescue. He designed and constructed a high temperature spectroscopy furnace that can oper-

ate up to 1500°C. The water-cooled furnace is essentially a Pt/Rh wire heater wound on to an alumina tube. The windows are made of aluminised kapton plastic which allows excellent transmission of X-rays but almost no radiative heat loss. Samples of basalt lava are suspended in the furnace on loops of wire.

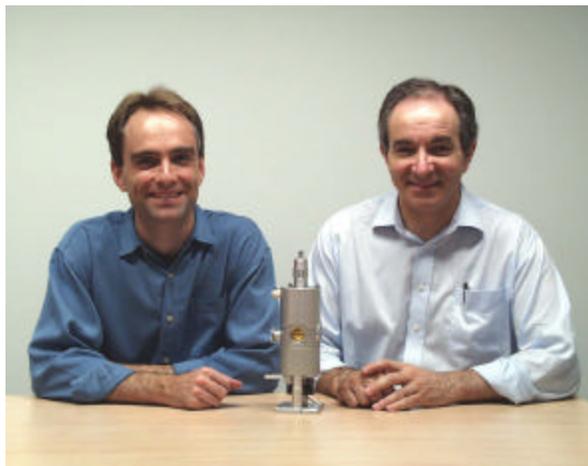
The furnace is compact (standing only some 30 cm high and 7.5 cm in diameter) and simple to use. It was designed so that it could be quickly and easily hooked up to a synchrotron beamline. Synchrotron radiation then passes through the windows of the furnace allowing the oxidation states of cations in the sample to be identified.

The furnace is now used routinely at the Australian Nuclear Beamline Facility, Photon Factory, Tsukuba Japan (with support from the Australian Synchrotron Research Program).

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Andrew Berry (left) and Michael Shelley with their new furnace.



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## Rock Tech

The ANU **Department of Geology**, Faculty of Science, owns and operates a range of analytical and experimental equipment for work in the field of geology. In general, the equipment is operated and maintained by a small, multi-tasking team of technical staff. Students who require a facility more than occasionally are encouraged to become trained in its use. The extent to which this is appropriate depends on the technique, complexity of sample preparation and data analysis, and the experience of the student.

### Thin Section Laboratory

➤ **Rock crushing and milling facilities:** (hydraulic splitter and mills). In order to get a reliable bulk average chemical analysis of a rock, it's important to be able to mill a representative sample down to a homogeneous powder.

➤ **Rock sawing, polishing and thin section making:** Much identification of phases and analysis of textures in rocks is done under the optical microscope, using either thin sections (usually 30  $\mu\text{m}$  thick) in transmitted light, or polished slices mounted in epoxy blocks and reflected light for opaque material such as ore samples.

➤ **Optical microscopes:** Petrographic microscopes are equipped with polarizers in the light path before and after the sample. Change in specimen colour (with polarization direction of the light - pleochroism) and the interference colours between crossed polars (caused by birefringence in the sample) are important identification techniques.

### Micropaleontology & Limnology

➤ State of the art **micropaleontological laboratory** and micro-fossil collections

➤ **Low-temperature room** for growing micro-organisms in marine waters for geochemical analysis.

➤ Standard range of equipment for **limnological investigations** of inland waters.

### Coral Reef & Marine Geochemistry

➤ **18Hp hydraulic drill** for shallow (5-15m) drilling of soft rocks, used for taking core samples from coral reefs.

➤ Portable Mtrom auto-titrator for analysing carbonate chemistry of marine waters .

### X-ray Laboratory / Electron Microscopy

➤ **2 X-ray powder diffractometers** ( $\theta$ - $\theta$  with Co tube,  $\theta$ - $2\theta$  with Cu tube) . The X-ray diffraction patterns from a flat powder sample contain distinctive 'fingerprint' spectra for each mineral that may be used to identify minerals, quantitatively analyse mixtures of phases, or refine the crystallographic unit cell and structure of a



X-Ray Diffraction Lab

single mineral.

➤ **Software** for quantitative analysis of multiphase powders, structure refinement, etc.

➤ **Sample preparation facilities** (WC ring mill, kiln and drying oven, ultrasonic bath, centrifuge, etc)

➤ **Ion-beam thinner** for transmission electron microscope.

➤ **Transmission electron microscope** (JEOL 200CX), providing high-magnification images and electron diffraction patterns from very thin edges of crushed-grain samples or specimens that have been milled in the ion-beam thinner.

### Analogue Deformation Laboratory

➤ **Deformation stage** for studying textural changes as a function of strain, strain rate and temperature in softer, lower-melting analogues of rocks.

➤ High-quality optical microscopy with digital video capture.

### Fluid Inclusion Laboratory

➤ **Heating-cooling stage** for measuring freezing and homogenization temperatures of fluid inclusions (bubbles of liquid + gas + salts) trapped in mineral crystals. This can allow determination of the density and composition of the fluids in which the crystal grew.

### High-Pressure Laboratory

➤ **Two Piston-cylinder uniaxial presses** . In these, very small samples can be compressed to pressures of over 2 GPa (20,000 atmospheres) and temperatures of 1500°C, allowing syntheses and reactions to be performed at conditions approximating those of the deep crust.

➤ **Cold-seal bombs** . For larger-volume experiments at high pressure and temperature incorporating fluids.

➤ **1-atmosphere furnaces**  
➤ **Sample preparation facilities**

### Geochemical Laboratory

➤ **XRF pressed-pellet and fused-disk making facilities**. X-ray fluorescence spectroscopy requires a few grams of sample, either a pressed pellet of fine, homogeneous rock powder, or a glass disk made by dissolving the rock powder in molten lithium borate.

➤ **XRF spectrometer** for major and trace elements in whole rocks. XRF cannot be used for very light elements (below Na in atomic number, usually) but can quantitatively determine heavier elements down to ppm levels.

➤ **ICP-Mass Spectrometer with Laser Ablation Unit**. A UV laser is focused onto a small spot (tens – hundreds of  $\mu\text{m}$ ) in a flat sample such as a thin section of rock. The atomized spot is then sucked into the argon plasma torch of the Mass Spectrometer, which allows chemical analysis (including light elements and others that are difficult to measure by other techniques) down to parts per billion levels.

➤ **ICP-Atomic Emission Spectrometer** for simultaneous analysis of multiple trace elements in liquid samples down to parts per billion.

➤ **Volatile Analyser** for water and carbon content determination, and quantitative analysis of volatile-rich minerals.



The Piston-cylinder apparatus in the High-Pressure Laboratory.

For more information  
Andy.Christy@geology.anu.edu.au

# Positions vacant

## Australia

### Postdoc Fellow/complex materials (closes 14/2/02)

RSPHySE, Applied Maths, ANU  
<http://www.anu.edu.au/hr/jobs/academic.html#736/01>

### Postdoc Fellow/Research Fellow/theory of photonic crystals (closes 8/2/02), Dept of Physics, ANU

<http://www.anu.edu.au/hr/jobs/academic.html#715>

### Fellow/Snr Fellow/2 positions: Organic Chemistry, Physical Chemistry (closes 1/3/02)

RSC, ANU  
More info: <http://www.anu.edu.au/hr/jobs/academic.html#699>

### Lectureships in Chemistry (closes 7/2/02)

Sydney Uni, <http://bull.ucc.usyd.edu.au/personnel/FMPro?-db=personnel.fm&-format=jobdetail.html&Ref=A02/002310&-find=>

### Postdoc Fellow/Astrophysical plasmas, (closes 1/02/02)

School of Geosciences, Sydney Uni  
<http://bull.ucc.usyd.edu.au/personnel/FMPro?-db=personnel.fm&-format=jobdetail.html&Ref=A50/002260&-find=>

### Sesqui Lecturer in Nanoscience, (closes 14/02/02)

Sydney Uni  
<http://bull.ucc.usyd.edu.au/personnel/FMPro?-db=personnel.fm&-format=jobdetail.html&Ref=A02/002312&-find=>

## Overseas

### Postdoc positions: glass scientist, ceramist, solid state chemist, electrochemist (closes 15/1/02)

Environmental Tech Institute of Singapore  
[http://www.mrs.org/career\\_services/classified/ads/eti.html](http://www.mrs.org/career_services/classified/ads/eti.html)

### Academic Post in Applications of Ion Beam Analysis (closes 15/3/02)

University of Surrey, UK  
<http://jobs.ac.uk/jobfiles/KC311.html>

### Lectureship in Polymer Science. (closes 28/2/02)

Uni of Manchester, UK  
<http://jobs.ac.uk/jobfiles/NB906.html>

### Postdoc/Processing of nanotubes (closes 5/2/02)

Cambridge Uni, UK  
<http://jobs.ac.uk/jobfiles/BD472.html>

### Postdoc research Fellowship/Urban meteorology and air quality (closes 15/2/02)

Uni of Leeds, UK  
<http://jobs.ac.uk/jobfiles/YF928.html>

### Postdoc/Nanoscale materials

(closes 18/3/02), Dartmouth College, UK  
<http://jobs.ac.uk/cgi-bin/advsearch2.cgi>

### Research fellowships/Protein Crystallography (closes 1/3/02), Laval University Medical Center,

Canada; <http://www.iucr.ac.uk/cww-top/job.anc1180.html>

## For the Diary

- |  |                   |
|--|-------------------|
| ◀▶ Australian Society for Biomaterials<br>12th Annual Conference, ANU, RSPHySE<br>see <a href="http://www.biomaterials.org.au/ASBConf.html">http://www.biomaterials.org.au/ASBConf.html</a>  | 19-21 March, 2002 |
| ◀▶ 15th Biennial Congress of the AIP incorporating<br>▶ the Australian Conference on Optical Fibre Technology &<br>▶ the Australian Optical Society conference<br>Sydney Convention Centre, Darling Harbour, see <a href="http://www.aip.org.au/Congress2002/">http://www.aip.org.au/Congress2002/</a> | 8-11 July, 2002   |
| ◀▶ HPSC 2002<br>International Conference on High Performance Structures and Composites, Seville, Spain<br>see <a href="http://www.wessex.ac.uk/conferences/2002/hps02/">http://www.wessex.ac.uk/conferences/2002/hps02/</a>  | 11-13 March, 2002 |
| ◀▶ CIMTEC 2002<br>▶ 10th International Ceramics Congress &<br>▶ 3rd Forum on New Materials<br>Florence, Italy; see <a href="http://www.dinamica.it/cimtec/">http://www.dinamica.it/cimtec/</a>   | 14-19 July, 2002  |
| ◀▶ WCPT 4<br>4th World Conference on Particle Technology, Convention/Exhibition Centre, Sydney<br>see <a href="http://www.wcpt4.com/index.htm">http://www.wcpt4.com/index.htm</a>  | 21-25 July, 2002  |
| ◀▶ ICNDST-8<br>8th International Conference on New Diamond Science and Technology, Uni of Melbourne, Melbourne<br>see <a href="http://www.conferences.unimelb.edu.au/icndst-8/">http://www.conferences.unimelb.edu.au/icndst-8/</a>  | 21-26 July, 2002  |

## Welcoming CSEM



Professor John Baird (follow the arrow) welcomed CSEM to the fold at the Faculty of Engineering and Information Technology (FEIT) Christmas drinks held in the final days of last year.

As you all know, CSEM's new Director is Dr Zbigniew Stachurski from the Department of Engineering. That means CSEM's new host Faculty is FEIT.

Thanks to all those CSEM members who managed to get along to the drinks and rub shoulders with our engineering colleagues. A fun time was had by all.

CSEM's office will be moving to the Department of Engineering in the near future.

# Hot rock

*(Continued from page 1)*

Using the furnace the researchers have demonstrated that in Mid Ocean Ridge Basalt, the most common magma on the Earth's surface, around half of the chromium exists as chromium(II) even though this oxidation state has never been previously identified in terrestrial material. This is because on cooling chromium(II) undergoes an electron exchange reaction with iron(III) to form chromium(III) and iron(II). These results are now being used to explain the behaviour of chromium in natural rocks.

Experiments can also be undertaken on crystalline samples and researchers from the University of Sydney have already expressed interest in using the furnace.

Results so far from the furnace suggest it will be a valuable tool that may play an important role in boosting our understanding of how the Earth was formed.

For further information, please contact Andrew Berry (Andrew.Berry@anu.edu.au).

## A FORUM ON SURFACE PREPARATION

Are you involved in surface preparation? Is there some topic in this area that you'd like discussed? Is there something you're doing in the area of surface preparation that other people could learn from?

A group of materials researchers at ANU with a particular interest in surface preparation techniques (for a wide range of materials ranging from optical fibres and semi-conductors through to thin sections of geological materials) are interested in running a forum to share ideas and methods.

CSEM has offered to help with the event. At this point we're brainstorming possible formats and themes for the forum, and we'd be interested in your feedback.

Please send your ideas to David Llewellyn at RSBS (davidl@rsbs.anu.edu.au) and later in the year CSEM will let you know what's happening.



◀◀ The furnace with its lid off and under test  
▼▼



## Furnace Specs

The oxygen fugacity environment inside the furnace is determined by a variable CO/CO<sub>2</sub> atmosphere using mass-flow controllers. The estimated accuracy of sample temperature is  $\pm 10^\circ\text{C}$  while the thermal stability is better than  $\pm 1^\circ\text{C}$ . Gas mixes equivalent to logfO<sub>2</sub>'s over a range from -3 to -13 at 1400°C can be used. .

## MM webspotting: What's in a name

### ◆ Molecules with silly names

<http://www.bris.ac.uk/Depts/Chemistry/MOTM/silly/sillymols.htm>

### ◆ Molecule of the month

<http://www.bris.ac.uk/Depts/Chemistry/MOTM/motm.htm>

### ◆ Named things in chemistry and physics

<http://www.chem.yorku.ca/NAMED/>

### ◆ Domain names for sale relating to chemicals and raw materials

<http://www.hotwebnames.com/chemicals.html>

### Wanted: Communicators

Would you like to be more involved in communication activities in your school or department? We'd like to know if any members would be interested in CSEM running a skills workshop on writing for the web, a newsletter or the general public. Contact the editor for details.

# Introducing Facets

IUMRS

### A new international materials newsletter

The International Union of Materials Research Societies has launched a new quarterly publication, IUMRS *Facets*.

Complimentary copies of the first issue (Jan 2002) are now available through the IUMRS website (<http://www.iumrs.org/>). Subscription costs \$US20.

CSEM has spare copies for those wishing to view it.

# CSEM

ANU Centre for Science & Engineering of Materials

#### Faculties

Department of Chemistry  
Department of Engineering  
Department of Forestry  
Department of Geology  
Department of Physics

#### Institute of Advanced Studies

Research School of Biological Sciences  
Research School of Chemistry  
Research School of Earth Sciences  
John Curtin School of Medical Research  
Research School of Physical Sciences & Engineering

#### Institute of the Arts

Materials Workshops

# Materials Monthly

Volume III, Issue 1

January 2002

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*Materials Monthly* is CSEM's monthly newsletter. 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.

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