

A LIGHT FOR SCIENCE

# ESRF **news**

Number 75 March 2017

## **Palaeontology**

Seeing into the past



**EBS beamlines  
reviewed**

**Reports from  
the User Meeting**

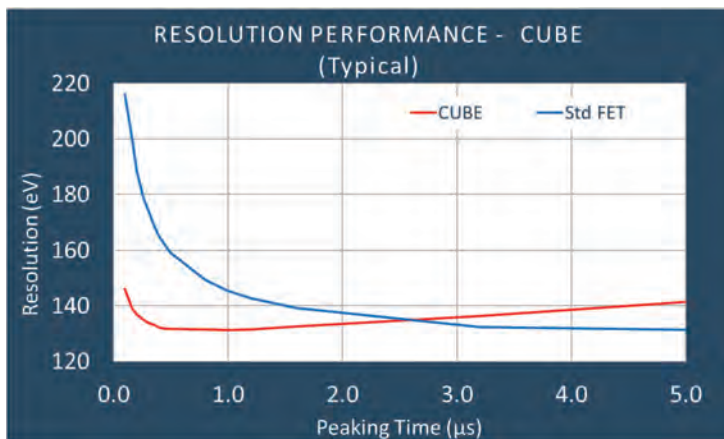


**Exciting news.....  
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## SDD detectors for beam-line applications

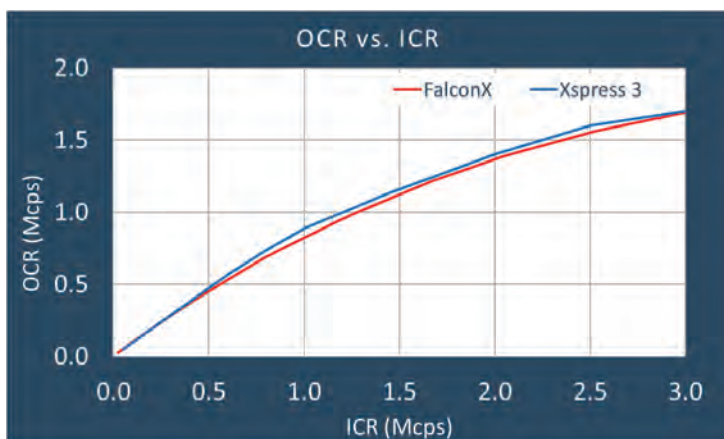
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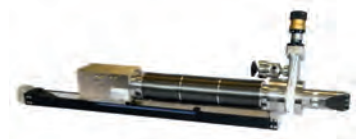


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Example of single sensor design



Example of multi-sensor design with UHV compatibility

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- Resolution from 126eV
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- High count rate to >4Mcps
- Windows: Thin Polymer / Beryllium / Silicon Nitride / Windowless
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- Gate valve and bellows available for UHV compatibility

### Examples of Customised Designs



4 Sensor 'Beam Through' Detector  
Circular Focused Array SDD



4 Sensor Vertical  
Focused Array SDD



7 Sensor Circular  
Focused Array SDD



Windowless SDD  
design for UHV



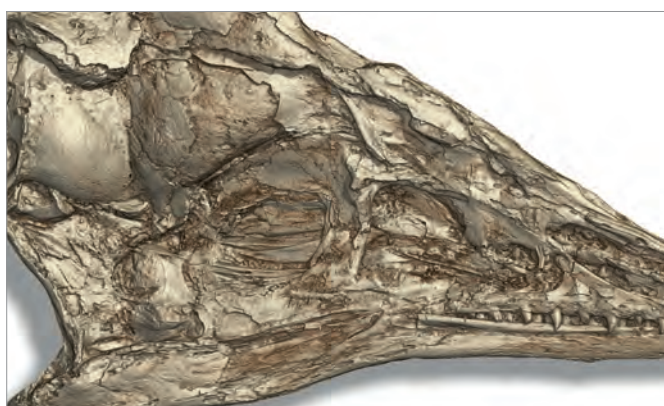
Modular SDD  
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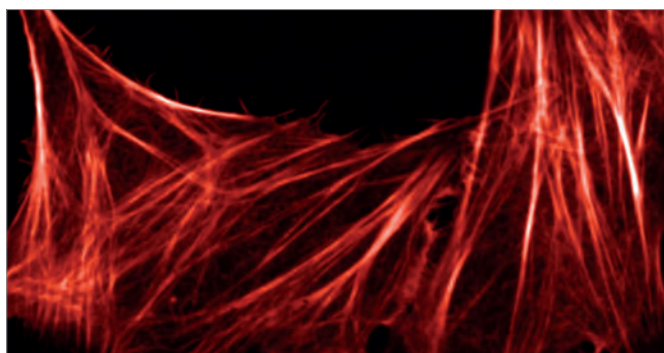
# The European Synchrotron



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Palaeontology: new light on the science of old, p15.



The brightest red fluorescent protein, p30.



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Microtomographic scan  
of an *Australopithecus*  
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# Picking up the pace

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Last year saw new records set in the ESRF's operational performance. The number of submitted and accepted proposals was greater than ever before; so too was the number of experimental sessions and user visits. Yet amazingly, all this has taken place during a demanding period in which activities for the Extremely Brilliant Source (EBS) upgrade are in full swing. It reflects the continued interest of our users to exploit ESRF beamlines, and it underlines the ESRF's commitment to deliver the best possible user service – right until the last day of operation before the long shutdown, starting in 2019.

The main part of the upgrade, the EBS storage ring project, is now advancing rapidly, with the first components from commercial suppliers having arrived on site (p8). The next steps include the serial production of the new storage ring components; the launching of the assembly phase of the girders in September this year; and detailed planning for the installation and commissioning of the new storage ring, and of the public and CRG beamlines, for the 2019–20 period.

**“The EBS storage ring project is now advancing rapidly”**

The EBS programme includes funding for the construction of four new beamlines and an ambitious instrumentation development programme, mainly in the areas of photon detection and data handling. Last year, we started to reflect on the new public beamline portfolio. Out of 48 expressions of interest from our user community, eight candidate projects were identified in full consultation with our Science Advisory Committee (SAC). Conceptual Design Reports (CDRs) for these exciting new projects were discussed in a workshop with our user community in December (p11), and then critically evaluated at an extraordinary meeting of the SAC last month. The CDRs will continue to be refined during the spring until the most attractive scenario for the future experimental programme is identified and submitted to the ESRF Council in June for discussion and validation. That will lead to the scheduling of the EBS beamline construction between 2020 and 2022.

In identifying the new projects, the aim is to maximise the profit to ESRF users of the tremendously increased brilliance of the EBS source, along with its associated increase in photon flux and beam coherence. One field that could well profit is palaeontology. The focus of this issue (pp15–25), palaeontology has seen rapid development at the ESRF thanks to our high-quality, high-energy beams – not to mention a team of dedicated scientists and an ever-expanding palaeontological user community. Much progress has been made since *ESRFnews* put palaeontology in the spotlight six years ago, and even more will be enabled by taking advantage of the future EBS source.

**Harald Reichert and Jean Susini**, *ESRF directors of research*



The SESAME synchrotron.

## SESAME project is underway

An ESRF-coordinated EU project to help the best use of the SESAME synchrotron in Jordan was instigated on 1 January. Known as OPEN SESAME, the €2 m project will help to train staff, run workshops, provide fellowships, and improve public outreach.

SESAME is the Middle East's first synchrotron, and aims to provide a world-class research facility for the region that fosters international scientific co-operation. A joint undertaking of Bahrain, Cyprus, Egypt, Iran, Israel, Jordan, Pakistan, the Palestinian Authority and Turkey, the light source will undertake its first experiments this summer with an operating energy of 2.5 GeV. Also in January, the light source celebrated the circulation of its first electron beams.

OPEN SESAME is co-ordinated by the ESRF with a consortium of nine other European facilities, many having a long history of support to SESAME, such as the particle-physics lab CERN, the French light source SOLEIL and the German lab DESY. Over three years, there will be staff exchanges, training schools, a short-term fellowship programme and an industrial workshop, as well as a proactive communications strategy. "This is a partnership of light sources and other facilities to help nurture the exploitation of this amazing new facility in the Middle East," says Edward Mitchell, the ESRF co-ordinator.

"SESAME is very grateful to the European Commission for the support it is providing through the very valuable OPEN SESAME project," says Chris Llewellyn Smith, president of the SESAME Council. "It will allow SESAME to benefit from the expertise of the European light sources through staff exchanges and other programmes, and indeed it is already raising awareness of the potential for the synchrotron among its members."

# Poland renews ESRF membership



Left to right: Roman Puzniak, Krystyna Jabłoriska and Francesco Sette.

Poland renewed its "scientific associate" status with the ESRF in December, ensuring that its scientists have continued access to the synchrotron. The country, which signed its original contract with the ESRF in 2004, has a well-established light-source community, and last year it began commissioning its first national synchrotron, SOLARIS, in Krakow.

"The signing of this agreement testifies the continued support and engagement of Poland to the ESRF programme," says Roman Puzniak, director of

the Institute of Physics of the Polish Academy of Sciences and one of the renewed contract's signatories. "The extension of our collaboration is crucial in the framework of the ongoing ESRF-EBS project, and also to our national programme on the new synchrotron source, SOLARIS."

Krystyna Jabłoriska, the Polish observer to the ESRF Council, points out that the renewal gives Poland access to a high-energy synchrotron. "Experiments that cannot be done at SOLARIS, or in other European countries, can

now be performed at the ESRF," she says.

Francesco Sette, the director general of the ESRF, believes that scientists from Poland and those from the other 20 ESRF partner countries will "greatly benefit" from the ESRF's new capabilities. "The Polish scientific community has made essential contributions to the excellence of the ESRF, and to the establishment of a powerful European Research Area," he says. "I look forward to many more years of fruitful collaboration."

## River could fund own cleaning

An ESRF study of pollution in the Tinto river in southern Spain has uncovered traces of valuable rare earth metals that could help to fund the river's decontamination.

Deriving its name from "red wine" in Spanish, the Tinto river is a red, acidic body of water that is highly polluted thanks to the presence of more than 100 pre-Roman mines that line its banks. Together with a neighbouring river, the Odiel, the Tinto is believed to be the source of up to 40% of the oceans' zinc and 10% of the oceans' copper. In 2011, a team including the Universidad of Huelva, which is close to the Tinto, and the Institute of Environmental Assessment and Water Research (IDAEA) in Barcelona attempted



Some of the Tinto's pollution is valuable.

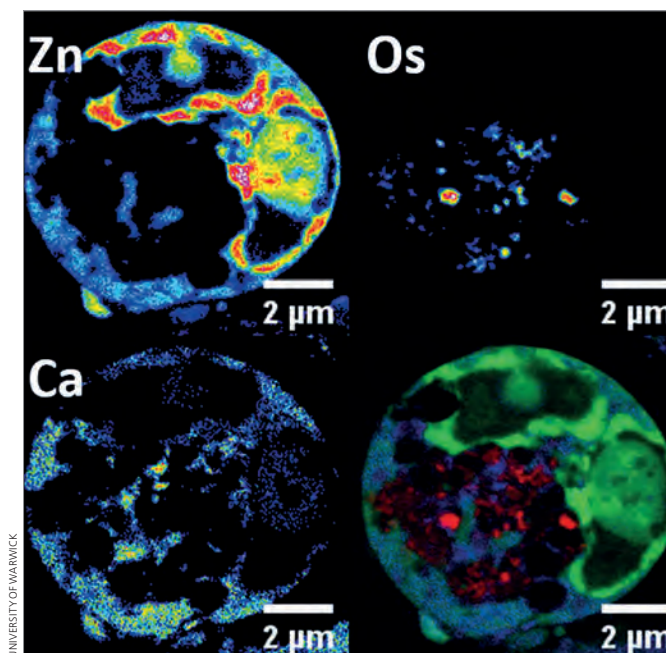
to build pools in the two rivers in which they could reduce the acidity by dissolving limestone. But when they analysed the precipitates at the ESRF beamlines ID21 and BM25 using X-ray micro-fluorescence and X-ray absorption spectroscopy, they were surprised to discover high concentrations of rare earth metals associated with the structures of the aluminium minerals that carried the pollutants.

The scientists believe that the metals, which are routinely used in consumer electronics, represent a "game changer" for the river's decontamination. "We could potentially exploit them to fund at least the purifying process, so it wouldn't cost money and the population in the neighbouring area could use the water," says Jose Miguel Nieto of the Universidad of Huelva.

# Osmium could be cancer drug

Researchers at the University of Warwick, together with ESRF beamline scientists, have demonstrated the effectiveness of a new metal in killing ovarian cancer cells. Osmium is relatively inexpensive, is not associated with cancer resistance and, the researchers say, has promise for treating other types of cancer, such as colon.

More than half of all cancer chemotherapy treatments currently use platinum compounds. Despite its past success, some ovarian cancers are now resistant to platinum, and the metal can bring harmful side-effects such as renal failure, neurotoxicity and vomiting. Having previously shown that osmium has the right properties to be an effective replacement, Peter Sadler and colleagues at the University of Warwick in the UK have now used the new nano-imaging beamline ID16A to analyse the effects of Organo-Osmium FY26 – a compound they discovered – in actual ovarian cancer cells. By detecting the emission of fluorescent light, Sadler and colleagues were able to observe the osmium compound positioning itself in the cells' mitochondria, and destroying their functions from within. Moreover, the scientists showed that the compound was more selective between healthy cells and cancer cells than the platinum-based compound cisplatin (*Chem. Eur. J.* doi:10.1002/chem.201605911).



**Nano X-ray fluorescence of ovarian cancer cells depicts the location of osmium, as well as zinc and calcium, the mobilisation of which indicates cell death.**

Sadler's group worked with Peter Cloetens of ID16A and others at the ESRF. "The advanced nano-focussed X-ray beam at the ESRF has not only allowed us to locate the site of action of our novel Organo-Osmium FY26 candidate drug in cancer cells at unprecedented resolution, but also to study the movement of natural metals such as zinc and calcium in cells," says Sadler. "Such studies open up totally new approaches to drug discovery and treatment".

• Meanwhile, researchers at University College London in the UK, Ludwig-Maximilians University in Germany and scientists at the ESRF biomedical beamline ID17 have developed a new type of phase-contrast imaging that could deliver breast cancer screening with one-tenth of the dose of conventional mammography. Though developed at the ESRF, the technique is compatible with lab X-ray sources – boding well for clinical use (*Phys. Med. Biol.* 61 8750).

## Zebrafish could help gout

The ESRF has enabled researchers in Italy to identify a genetic mutation in our human ancestry that could be key to our inability to degrade uric acid – a characteristic that makes us susceptible to gout. Normally the enzyme urate oxidase (Uox) helps catalyse the degradation of uric acid, but Riccardo Percudani and others at the University of Parma identified a particular mutation of it in the hominoid line (humans, and other great and lesser apes). As a result, Giuseppe Zanotti of the University of Padova took a similar but more stable Uox from a zebrafish and studied tiny crystals of it with the ESRF's microfocus beamline ID23-2. The scientists found that when this Uox was affected by the mutation, its effectiveness was stymied – providing, says Percudani, "an insight into the loss of the urate degradation pathway in humans" (*Sci. Rep.* 6 38302). The scientists believe that the administration of the non-mutated zebrafish enzyme could help humans who suffer from juvenile gout.

## Flu mechanism uncovered

A French team of scientists have used the ESRF to uncover how flu polymerase interacts with human polymerase – a key step in the spread of the virus, and one that could help in the development of anti-viral drugs.

Stephen Cusack of the European Molecular Biology Laboratory in Grenoble and colleagues studied the binding of the two polymerases using X-ray diffraction at the ESRF beamlines ID29 and ID23-1. They found that when they manually disrupted the binding, the flu polymerase was unable to use lengths of the human RNA, which could be used for the creation of viral proteins. The result suggested that the binding was crucial for the spread of the virus when it enters a human host (*Nature* 541 117). "We've uncovered the details of a mechanism that's common to all influenza strains, so we believe this could be a good target for developing new flu drugs," says Cusack.

## Pantaleo Raimondi wins accelerator prize

The European Physical Society Accelerator Group (EPS-AG) has awarded the ESRF's Pantaleo Raimondi the Gersch Budker Prize for his pioneering work in accelerator science. Raimondi, the director of the ESRF's accelerator and source division, won the prize for his invention of the "Hybrid Multi Bend Achromat" (HMBA) lattice, which has become the design basis of most future "fourth generation" synchrotron sources.

Awarded once every 2–3 years, the Gersch Budker Prize recognises a recent significant, original contribution to the

accelerator field. Raimondi's HMBA lattice has allowed the ESRF to reduce its emittance by a factor of 30, while still keeping a large enough dynamic aperture. According to the EPS-AG, the design "shows Raimondi's ability to foster new ideas, his deep understanding of accelerator physics and [his] mastering of technological aspects." As underlined by the EPS-AG, the HMBA lattice has inspired the design of other big light-source facilities around the world, such as the Advanced Photon Source upgrade at Argonne National Laboratory



and the Advanced Light Source Upgrade at Berkeley Lab in the US; SPring-8 at the Japan Synchrotron Radiation Research Institute; and the Shanghai Synchrotron Radiation Facility and the Beijing Synchrotron Radiation Facility in China.

STEF CANDE



Cleaning in the booster spares users delays

# Electron-bunch cleaning boosted

The ESRF celebrated the end of last year with the launch of booster-based electron-bunch cleaning – a time-saving measure that will prove particularly beneficial to the EBS’s mode of operation.

Electron-bunch cleaning is necessary to remove unwanted, “parasitic” electrons that are accidentally injected into parts of the storage ring that are supposed to be kept electron-

free. Normally it is performed in the storage ring itself, with a signal exciting the electrons to their resonant frequency until they escape their orbit and collide with metal jaws on either side of the vacuum chamber. But this process takes some 40 seconds, during which users cannot take data. Over a typical week in “top-up” operation – the filling mode of choice for the EBS – this represents nearly five lost hours.

Over the last few years, a team of diagnostic engineers has adapted and developed this cleaning system so that it takes place in the booster instead. The principle is the same, but the cleaning takes just a few milliseconds and means that a pre-cleaned beam can be injected directly into the storage ring, avoiding any disturbance to users. “Performing cleaning in the booster is more challenging

than in the storage ring because the booster characteristics fluctuate from cycle to cycle,” says Benoit Roche, a member of the diagnostic team.

In December, the new system was used for the first time in operation with 16-bunch filling – one of several modes of filling the storage ring. The team are now preparing to test the cleaning in the other filling modes.

STEF CANDE



## New magnets delivered

The first batches of final-prototype, or “pre-series”, quadrupole and sextupole magnets were delivered at the end of November. The magnets – which are responsible for focusing and controlling aberrations in the electron beam, respectively – were followed by the remainder of components for the main series of dipole magnets, which steer the electrons. These components included over

13,000 high-performance permanent magnets, 640 magnet modules and 128 dipole supports. Meanwhile, two more girders arrived for the creation of a mock-up cell (see story, right).

Deliveries will continue this year with the arrival of supports to fix the quadrupoles, beam-position monitors and vacuum chambers to the girders, as well as pumps, controllers, gas analysers, gauges, and a new radio-frequency cavity.

Taking magnetic readings on a quadrupole.

## Mock cell begins

Preparations are underway in the ESRF’s Chartreuse Hall to install four newly delivered girders with pre-series magnets and vacuum chambers as part of a mock-up construction of an EBS standard lattice cell. Recommended by the Machine Advisory Committee at its last meeting, the exercise will ensure that there is no interference between assembled parts due to design or machining errors. As this issue went to press, the girders were anchored on the concrete floor, ready for the installation of magnet supports and magnets.



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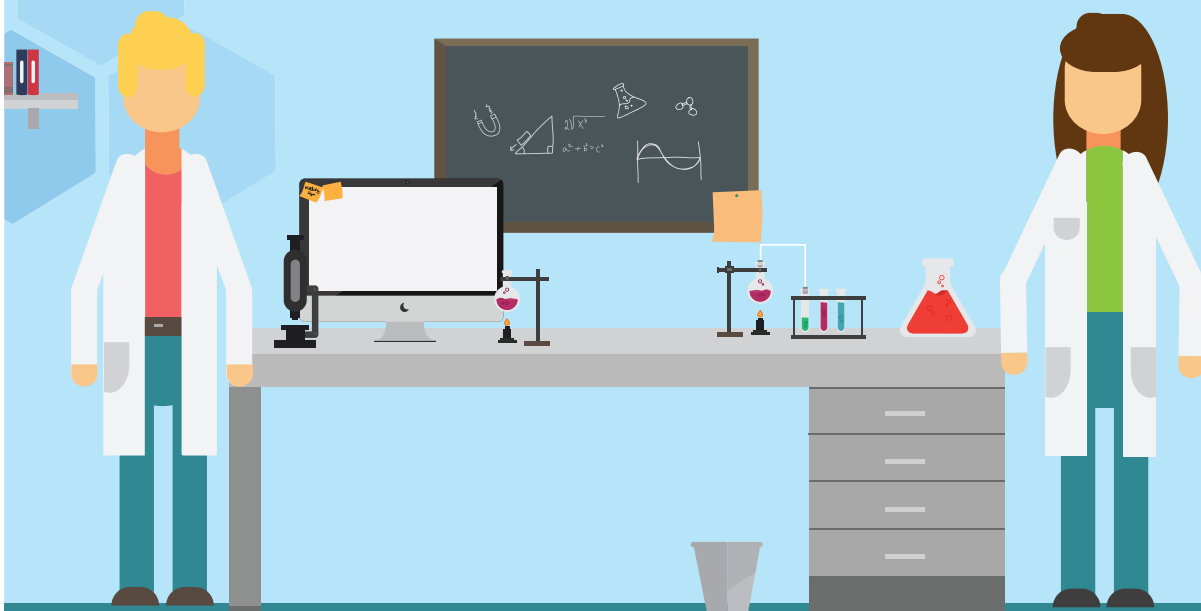
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## News from the User Office

The review by the Beam Time Allocation Panels in October last year saw 482 proposals accepted for the 2017/1 scheduling period –42% of the 1154 proposals that were submitted before the September deadline. For the current round, long-term project proposals submitted for the 15 January deadline, and standard and MX BAG proposals submitted for the 1 March deadline will be reviewed in the meetings on 27 and 28 April this year.

The Beam Time Allocation Panels would like to remind proposers of the importance of submitting experiment reports for all beam time allocations previously used. Proposal forms should also include more than one proposer; if this is not the case, the proposer should include

a comment on who will carry out the work in the technique section of the experiment methods template. The need for synchrotron radiation and for the specific beamline(s) requested should be clearly explained, and any reference in the proposal to previous experiments should always be accompanied by the proposal number of that experiment. Resubmitted proposals should be clearly marked as such, and it is mandatory to clearly indicate what aspects of the proposal have been modified or improved. Reviewers advise that proposers omitting these details or resubmitting an unchanged proposal are unlikely to be awarded beam time.

*Joanne McCarthy, Head of the User Office*

## News from the User Organisation Committee

The User Organisation would like to thank all the users who participated in the 2017 User Meeting (see p12) and contributed to the pleasant and dynamic atmosphere. More users got involved than ever before – as was reflected in the number of attendees to the different sessions of the three-day event, and the number of posters submitted. We would like to acknowledge the ESRF's efficient organisation and thank the Communication Group for their support in advertising the meeting, as well as promoting it through videos, articles and interviews.

Attendees have been sent a link to an online questionnaire, which will help us evaluate the event and improve future

meetings. We kindly invite them to complete it, and propose ideas for keynote speakers and topics for next year's user-dedicated microsymbosia.

The User Meeting took place at a busy time for the ESRF, especially with the new Extremely Brilliant Source (EBS) upgrade in full swing. Finally, therefore, we would like to thank the user community for having been so active over the past year, by submitting numerous expressions of interest to identify potential new experimental programmes and beamlines for the EBS project, as well as a record number of proposals for both the March and September deadlines.

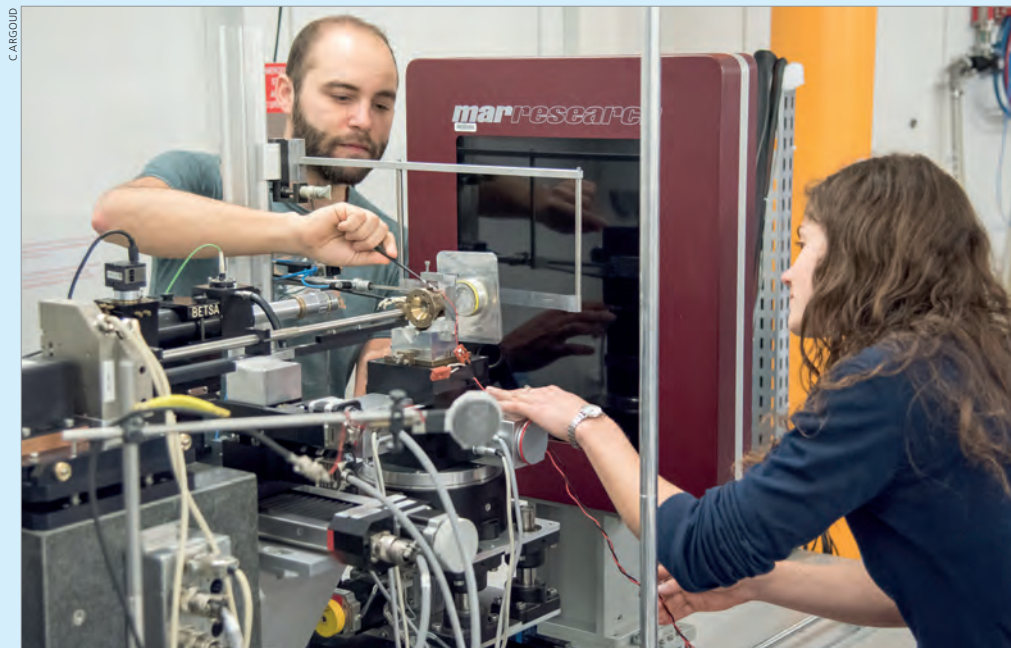
*Paola Coan, chair of the UOC*

## News from the beamlines

- **ID01** and the ESRF Data Analysis Unit have made available a data analysis software package with a graphical user interface for its quick Mapping diffraction microscopy (KMAP). This greatly enhances the independence of users in their data treatment. For the smallest beams, new zone plates now allow a beam size down to 60 nm in both dimensions for incoherent illumination and about 60 nm × 140 nm for coherent beams.

- On **ID15A**, a new cryogenically cooled Laue-Laue monochromator was installed during the winter shutdown. A new setup for combined X-ray microscopy and X-ray diffraction has been installed in the **EH3** experimental station and **ID15A** has received a new DECTRIS Pilatus 3X 2M CdTe detector that is especially optimised for X-ray diffraction measurements in the range 30–100 keV.

- New hardware was installed at the microtomography beamline **ID19**: pco.edge 4.2 cameras (financed by the Long Term Project ES-295 by Renard *et al.*) are now available and, thanks to the increased quantum efficiency of the CMOS sensor, the image acquisition can be carried out with substantially lower dose. To improve the image quality at



Baptiste Journaux (left) and Ines Collings on the new ID15B.

high photon energies, a set of linear refractive lenses made of aluminium has been installed in the first optics hutch to condense the beam vertically.

- X-ray absorption spectroscopy is being used more and more as a 2D imaging technique at the **ID21** beamline: full-field XANES in transmission mode and XANES mapping in fluorescence mode. The so-called Spectrocrunch library has been recently

developed to allow systematic processing of data from both techniques, as well as introducing feedback from data processing in the experiment. In particular, XANES mapping in fluorescence mode can be used to acquire XANES spectra on micrometric regions of interest.

- Crispy, a modern graphical user interface for core-level spectroscopy simulations, is now available. Developed using open-source software, it

provides a set of tools to help the users of spectroscopy beamlines to perform ligand-field multiplet simulations of experimental measurements, including X-ray absorption (XAS) and resonant inelastic X-ray scattering (RIXS). It relies on the Quanta software developed by Maurits Haverkort at Heidelberg University. More information can be found on the official webpage [www.esrf.eu/computing/scientific/crispy](http://www.esrf.eu/computing/scientific/crispy).

# Users debate future of the EBS

December's EBS Workshop saw the presentation of possible new EBS beamlines.

Nearly 300 participants attended a workshop at the ESRF on 8 and 9 December to discuss possible new beamlines that could form part of the synchrotron's Extremely Brilliant Source (EBS) upgrade. The workshop paves the way for the final selection of four new beamlines later this year.

The EBS is a €150 m upgrade to the ESRF that will see the synchrotron's X-ray brilliance and coherence boosted by two orders of magnitude. As part of the upgrade the existing beamlines will be prepared for the EBS, and four new beamlines will be constructed.

In May last year, the ESRF Science Advisory Committee whittled down 48 expressions of interest from the user community to eight possible new beamlines, and shortlisted other major refurbishment projects to existing beamlines. Conceptual design reports (CDRs) for those eight new beamlines were then drafted by expert groups (see "The new beamlines on offer", below).

On the first day of the EBS Workshop, the CDRs were presented in parallel sessions to the respective scientific communities and further refined. On the second day, an open plenary session saw all CDRs presented and discussed. "The aim of the workshop is to identify the most impactful and scientifically promising projects that we can realise on the fantastic new source we are planning to install and start operating in 2020," said Harald Reichert, ESRF director of research, during the workshop.

The Science Advisory Committee will



New beamlines were the centre of discussions at the EBS Workshop.

analyse the eight CDRs in early spring, prior to ranking the projects. A final decision on the four successful beamline proposals, in the context of a consolidated experimental

floor plan, is expected to be made by the ESRF Council in June.

"The workshop is full of tremendous ideas, creativity, and passion," said Andrew Harrison, the CEO of Diamond Light Source in Didcot, UK, and the moderator of the workshop. "There's a huge turnout from those people who already use synchrotron radiation in their science. But the interesting question is how this is going to evolve, because these new beamlines will open up to new areas of science and reach into new communities. I think that in the future, once the idea has become more concrete, we will see an even wider community engaging with the ESRF."

Kirstin Colvin

**"It was full of tremendous ideas, creativity and passion."**

## The new beamlines on offer

**CDR1** is dedicated to X-ray photon correlation spectroscopy and coherent X-ray diffraction imaging for dynamics studies and advanced imaging of complex systems. Applications include sub-millisecond ordering during phase separations, nucleation in complex environments and *in vivo* imaging of bacteria in micro-fluidic devices.

**CDR2** is dedicated to hard X-ray diffraction microscopy for the tomographic study of bulk properties in three dimensions in millimetre-sized samples. Applications include the characterisation of complex, multiscale phenomena *in situ*.

**CDR3** introduces a high throughput, large

field phase-contrast tomography beamline for materials research, materials engineering and large biological samples on multiple scales. Automation and high throughput will also feature.

**CDR4** exploits the EBS's extremely high brilliance for a surface-science beamline, in particular for the investigation of chemical and physical processes at solid and liquid interfaces. Applications include catalysis, electrochemistry, biomimetic membranes and polymers.

**CDR5** exploits the EBS's much higher photon flux density and higher coherence in a high-flux, nano X-ray diffraction beamline, for science at extreme conditions.

**CDR6** extends the applications of the recently approved high power laser facility

in a facility for dynamic compression studies, with single-shot and fast time-resolved X-ray diffraction and imaging.

**CDR7** proposes the conversion of one branch of the energy-dispersive X-ray absorption spectroscopy beamline ID24 to a scanning extended X-ray absorption spectroscopy beamline, for time-resolved and extreme-conditions studies. Applications include highly dilute systems in extreme conditions and high time resolution.

**CDR8** pushes the delivery of the 'Henderson Limit' in terms of X-ray dose into the microsecond range using sub-micron focal spots, for room-temperature and time-resolved serial protein crystallography on micro- and nano-crystals.



Right: Magnus Rønning describes how the ESRF's specialist facilities allow the testing of catalysts in real conditions. Left: Users exchange ideas during a coffee break.

# A catalyst for discussion

This year's ESRF User Meeting brimmed with science, discussion and ideas.

Catalysts are integral to almost every industrial process: nearly 90% of products involve a catalytic reaction. That is the reason for the huge volume of research devoted to catalysis, says chemical engineer Magnus Rønning of the Norwegian University of Science and Technology in Trondheim – but amazingly, catalysts are not always tested under realistic conditions. “People doing catalysis are coming from materials design, from physics, from chemistry,” Rønning explained in a keynote lecture during the plenary session of this year's ESRF User Meeting, which ran from 6–8 February. “They do not always have the necessary training in chemical engineering.”

The failure to replicate realistic conditions often arises because of the need to position a certain piece of instrumentation next to a catalytic cell. Doing this might, for example, require an experimenter to leave the cell open, rather than closed as it should be – but this could well mean that the gas is flowing over the catalytic bed, rather than through it. Clearly, the risk is that the results do not reflect what the performance of a catalyst would be in practice. “You might make the wrong interpretation,” said Rønning.

The problem is potentially worse at synchrotrons, where bulky instrumentation can be even more intrusive on space, and might even require samples to have certain

## “The User Meeting is a key event in the ESRF calendar.”

properties, such as being thin and transparent to X-rays. But this is an area where the ESRF, with help from Rønning and his colleagues, has been pioneering. Nearly a decade ago, they helped transform the Swiss–Norwegian collaborating research group beamlines at the ESRF, BM01 and BM31, to accommodate facilities for realistic catalytic conditions, in particular high pressures. The overhaul was not easy, and to cope with concentrated hazardous gases at high pressure the researchers had to work closely with the beamline scientists and the ESRF's safety group. In the end, however, the results have spoken for themselves – and, of course, the fact that synchrotrons around the world have begun to copy the idea. “It's been a great success,” said Rønning.

### Back to reality

One of the first industrial processes Rønning and colleagues explored at BM01 and BM31 was the Fischer–Tropsch process, a set of reactions that converts hydrogen and carbon monoxide – so-called synthesis gas, or syngas – into liquid hydrocarbons and water. Hydrogen and carbon monoxide are both reducing molecules, but as the reaction proceeds and more water is created, the conditions become more oxidising. That is bad news for the catalyst, commonly cobalt, which is unstable in oxidising conditions and turns to non-catalytic cobalt oxide. For that reason, says Rønning, their work on the Fischer–Tropsch process at the ESRF has always attempted to maintain a realistic level of conversion – for if it is unrealistically low, the catalyst will resist deactivating for longer than it would in industry.

More recently, Rønning's group have been studying alternative fuel-cell catalysts. Platinum is the common catalyst today, but it is one of a number of precious metals on a list of critical raw materials given by the EU, and the search is on to find effective replacements. As with the Fischer–Tropsch process, says Rønning, the trick is to study the alternative catalysts at normal operating conditions – and fortunately at the ESRF, they can measure the reaction kinetics while simultaneously performing X-ray measurements. There is

## Young Scientist Award goes to Amélie Juhin

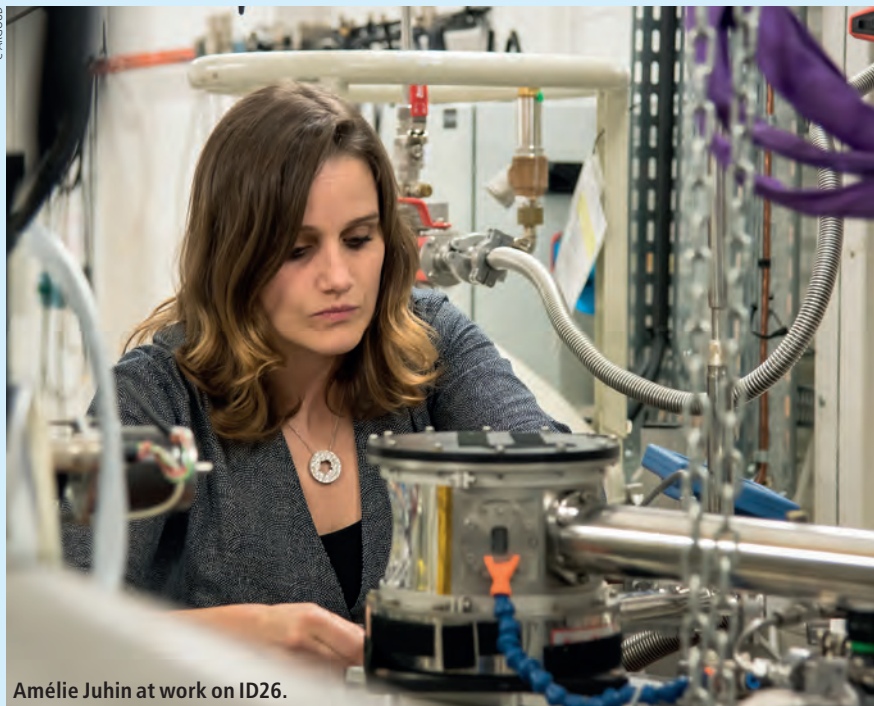
The 2017 Young Scientist Award has been presented to Amélie Juhin, a regular user of the ESRF's ID12 and ID26 beamlines, for her experimental and theoretical studies of resonant X-ray scattering and X-ray dichroism. The award, which was announced during this year's User Meeting on 7 February, is presented annually to an ESRF user to recognise outstanding work carried out at the ESRF.

"Amélie Juhin has matured into an independent scientist whose contributions are marked by a deep and thorough understanding of the physics and mathematics behind the interactions of X-rays with different substances, including minerals and magnetic materials," said Andrei Petoukhov, the chair of the prize jury.

### Stimulating science

Juhin began her studies at the École Normale Supérieure in Cachan, where she qualified as an associate professor in the physical sciences. In 2004, she left for Cambodia to teach physics and French – but her mind kept returning to research. Eventually she relented and undertook a masters in materials science and nano objects at the Pierre and Marie Curie University in Paris, followed by a PhD, during which she was sent to the ESRF to carry out some experiments – first on the French collaborating research group beamline, FAME, and then on ID26. "It became obvious then that I wanted to be a scientist," she recalls. "I chose X-ray spectroscopy because it is a very diverse field of study – I can do different experiments and calculations. It's very stimulating."

A post-doctoral fellowship at the University of Utrecht in the Netherlands allowed Juhin to strengthen her collaboration with ID26 scientists Pieter Glatzel and Mauro Rovezzi, and develop a novel "photon in, photon out" magnetic spectroscopy, known as RIXS-MCD.



Amélie Juhin at work on ID26.

The technique has since opened up new paths of investigation in magnetism and other disciplines, such as the earth sciences, and has led to a number of original results.

Now working as a CNRS researcher at the Institute of Mineralogy, Physics of Materials and Cosmo-Chemistry in Paris, Juhin focuses her research on the electronic and magnetic properties of nanoparticles and molecular magnets. She explores both the experimental and theoretical aspects of soft and hard X-ray spectroscopies with a particular focus on natural and magnetic dichroism. She is no stranger to prizes, having won the Best Poster Award at the 2010 ESRF User Meeting, as well as the Farrel Lytle Young Scientist Award from the International X-ray Absorption

Society in 2015 and, last year, a Bronze Medal from the CNRS. "I'm very honoured and pleased to be awarded the Young Scientist Award 2017," she says.

With the ESRF–EBS project, she has set her sights on new projects and the novel opportunities that will be made available with the greatly improved X-ray source. "I'm interested in materials whose magnetic properties can be switched through the application of pressure, and in textured magnetic liquids," she explains. "With the EBS upgrade, the gain in resolution and brilliance opens up very nice prospects for measuring in even better conditions than today."

*Kirstin Colvin*

an additional benefit to working like this, he adds: the results can more or less be directly applied to industry. "It's easier to persuade them of the research's relevance," he said.

Rønning's talk followed the enlightening keynote lecture by biophysicist Massimo Reconditi of the University of Florence in Italy, who described his group's spectacular work using X-ray diffraction to study the molecular mechanism of heart regulation. The day also saw a progress update on the ESRF's upgrade, the Extremely Brilliant Source, the 2017 Young Scientist Award (see above) and the poster session.

### There at last

The winner of the Best Poster Prize was Aram Bugaev, a third-year PhD student at the Southern Federal University in Rostov-on-Don, south Russia. Bugaev, 25, presented his research of combined X-ray diffraction

and absorption spectroscopy at BM01 on palladium hydride nanoparticles, revealing a structure in the core that is different to the shell – unusual, yet confirming theoretical predictions. "I'm especially pleased that the title of the work and the poster are the same as the title of the initial proposal submitted for my PhD two years ago," Bugaev said. "I've finally managed to do what was planned!"

Set up by the User Organisation Committee, the annual User Meeting is a key event in the ESRF calendar, giving users, staff and management the opportunity to share results and shape the future of their science. This year's meeting – the 27th since they began – featured a variety of other well attended events. On the first day, tutorials brought attendees up to speed in areas ranging from volume image analysis of tomographic data and coherent diffraction

imaging reconstruction, to scientific communication and social media. The third day saw a selection of user-dedicated microsymposia on advances in resonant inelastic X-ray scattering, quantitative coherent X-ray diffraction imaging and *operando* on structural studies of materials.

In his report, Francesco Sette, the ESRF director-general, pointed out that experimental activity marked 2016 as a year with new records in operational performance, with more submitted and accepted proposals, and more experimental sessions and user visits. The continuing trend of great science is one that should continue, given the ESRF's plan to operate as usual until the EBS shutdown at the end of next year. "We are committed to providing full operation at the highest standards right up to the end of 2018," he said.

*Jon Cartwright*

# The future of the past

Thanks to the ESRF, palaeontology has reached a new stage of evolution.



PIAFOREAU/ESRF

At the turn of this millennium, seeing the internal structure of a precious fossil was a challenge: a researcher could either slice it up, or place it in a conventional computerised-tomography scanner. The first option allows data collection at the expense of partial or even total destruction of the fossil, while the second offers only low quality and resolution.

That all changed in 2000 when Paul Tafforeau came to the ESRF. Encouraged by José Baruchel, who was then head of the ESRF imaging group, the young PhD student hoped that synchrotron microtomography would give him a detailed, non-destructive insight into modern and fossil primate teeth. He was right. The scans at the beamline ID19 were so successful that Tafforeau returned to the ESRF again and again for his studies of the origin of monkeys, apes, and then humans (p19) – originally using absorption-contrast microtomography, but within a few years turning to propagation phase-contrast microtomography, an approach that is up to 1000 times more sensitive. After his PhD, Tafforeau began to image large fossils at ID17, including Toumaï, the oldest known hominid skull.

The ESRF recognised its trailblazing role in palaeontology in 2006 by funding its first palaeontology PhD student, Vincent Fernandez, to work on tiny Cretaceous eggs, and then, in 2007, by giving Tafforeau a beamline scientist position at ID19. Thus began a steady diversification of work, including the study of “mammalian reptiles” in a fruitful collaboration with South Africa from 2008 (p16); the origin of land pioneers from 2009 (p21); the branching

**Above: Microtomography reveals the 4 cm-long skull of the “Eichstätt” specimen of *Archaeopteryx*, the earliest known bird, which together with modern crocodiles gives an insight into the branching of archosaurs into dinosaurs, including modern birds. The ESRF–EBS Upgrade brings the possibility of scanning entire skeletons of *Archaeopteryx* instead of only details. Below: Paul Tafforeau adjusts a crocodile skull on the ID19 beamline.**

of archosaurs into dinosaurs and birds via studies of *Archaeopteryx* fossils (see image) and modern crocodiles from 2011 (p25); and the first work on fossil plants from 2012 (p23). The scans of crocodiles, some of which are Egyptian mummies, are now leading the ESRF into yet more uncharted waters: synchrotron microtomographic imaging for Egyptology and archaeology, which Tafforeau calls “the next big thing”.

The ESRF Upgrade, the Extremely Brilliant Source, brings the potential for a new

project, CDR3, which combines a renewed ID19 and a new beamline to scan fossils five times bigger and ten times heavier. Though still to be decided upon, says Tafforeau, these beamlines would open up almost limitless prospects. “What is missing now is the ability to access small details in really big fossils. Today we can image the arm of a *Tyrannosaurus rex* – with the CDR3 project, we could scan it entirely in pieces, including its huge skull.”

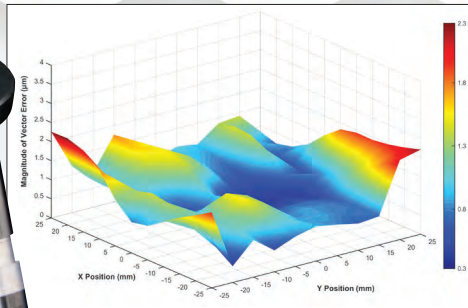
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# The secret of sur

At the end of the Palaeozoic era, ninety percent of all species were wiped out – yet the ancestors of mammals survived. An ESRF collaboration with South Africa is helping to explain how.

It is the most dramatic mass extinction the world has ever seen. About 252 million years ago, probably due to a spate of volcanic eruptions in Siberia, the global climate suddenly shifted, eradicating nearly 70% of all terrestrial life and almost all marine life. Before the extinction, a group of animals called therapsids dominated the land; afterwards, spurred by so many ecological niches left vacant, dinosaurs came to rule. The event marks the boundary not only between the Permian and Triassic periods, but also between the greater Palaeozoic and Mesozoic eras.

In fact, therapsids were not completely wiped out. Out of the six lineages existing in the Permian, three survived into the Triassic – and importantly one of these, the cynodonts, ultimately evolved into mammals. Indeed, therapsids are often referred to as mammal-like reptiles, and over the long course of history their anatomy became more and more mammalian: their posture switched from semi-sprawling to erect, for instance, and their once-uniform teeth split into incisors, canines and post-canines. When it comes specifically to the Permo–Triassic mass extinction, however, we are left asking: what was so special about those that survived?

## South Africa marks the spot

South Africa is a treasure trove for fossils. An area outside Johannesburg has so many notable hominin remains that it has become a UNESCO World Heritage Site known as the Cradle of Humankind. Less famously, though still tremendously important, is the Beaufort Group, a deep swathe of sediments covering a huge portion of the country that were deposited for 25 million years across the Permo–Triassic boundary (see map). It is the world's longest terrestrial record of

this key event, and an ideal place to study how therapsids were affected by the mass extinction. Realising that the ESRF's unique instrumentation had a lot to offer this region, in 2008, collaborations were initiated between the staff of ID19 and the scientists of this region – first to study the hominin remains (see p19), and then to study therapsids and other animals.

One of the first ESRF–South Africa collaborations in this area was our scan using the ESRF's ID17 beamline, published in 2013, of the cast of a fossilised burrow that had been flooded and then lithified. To our surprise, the burrow contained a cynodont therapsid, *Thrinaxodon*, buried together with a *Broomistega*, a completely different type of animal resembling a big salamander, from an extinct group of amphibians (see image, right). This was an odd association, as both animals were comparable in size and had similar diets, and in normal circumstances the *Broomistega* – a semi-aquatic creature, and therefore probably the intruder – ought to have been chased away.

Aside from uncovering the puzzle, the tomography offered a unique opportunity to imagine a possible scenario. For instance, the images revealed that the *Broomistega* had several broken ribs, which may have forced the amphibian to seek shelter in locations it would not normally go. We think the *Thrinaxodon* did not chase the intruder away because it was aestivating – in a deep torpor – to ride out the hot weather. When the flood waters suddenly came, neither animal was able to react quickly enough to escape.

The aestivation hypothesis remains to be fully tested. If it were indeed a characteristic of cynodont therapsids, it didn't help this particular creature but it could have played a major role in the ultimate survival of the

lineage in general, enabling its members to withstand the increasingly extreme climate at that time, which included prolonged arid seasons (*PLoS ONE* **8** e64978).

*Thrinaxodon* was very common, and hundreds of skulls of different ages have been discovered. That has allowed the ESRF–South Africa collaboration to better understand how its biology changed over a typical lifespan, in areas such as teeth replacement. We already know that, while reptiles and other animals replace teeth continually throughout their lives, mammals like us are limited to one or two cycles of tooth replacement; this means that we are toothless at birth, which is important for suckling. In 2013, a wide-ranging study of *Thrinaxodon* skulls scanned at the ID19 and ID17 beamlines, and in the lab, showed that the species' tooth replacement was also limited – albeit to seven cycles rather than one or two – and that, towards adulthood, its teeth developed complex cusps, like mammalian molars (*J. Vert. Paleontol.* **33** 1408). A separate analysis of the data two years later revealed a growth over a *Thrinaxodon*'s life of cranial muscles better suited to biting food than grinding it, like mammalian carnivores (*Anat. Rec.* **298** 1440).

## The mystery deepens

Synchrotron and laboratory-based tomographic studies like these do not directly help to solve the puzzle of how certain therapsids survived the mass extinction, though they do open up new avenues for exploration. One particularly promising avenue is the study of fossilised skull cavities, which once enclosed soft tissues such as the brain, veins and nerves. The shapes of these cavities can tell us how an animal was receiving sensory information.



## Dinosaur squeezes into ID17

The ESRF has not only been scanning therapsids around the time of the mass extinction. In July last year, a team of scientists from the University of the Witwatersrand, supported by the Department of Science and Technology and the National Research Foundation in South Africa, pushed the ID17 beamline to its full potential with a study of a complete skeleton of *Heterodontosaurus*

(pictured left), the largest dinosaur ever completely scanned in a synchrotron. A metre-tall biped of the Early Triassic, *Heterodontosaurus* is of the same lineage that produced such mighty beasts as *Triceratops* and *Stegosaurus*, yet the ESRF scan showed that the skeleton had stomach ribs – a feature never seen before in this group of dinosaur. It was yet another example of the surprises that so often comes when combining palaeontology with synchrotron tomography. *JC*





# vival

Last year, scans at ID17 and elsewhere of fossilised therapsid skulls at various stages of evolution revealed canals that would have given nerves freedom to move, increasing facial sensitivity. Along with other features, these canals led to the conclusion that some therapsids were covered in fur – but only those living some 10 million years after the Permo–Triassic boundary (*Sci. Rep.* **6** 25604). Fur is a crucial characteristic of mammals to regulate body temperature, so it is interesting to see that it did not participate in helping these therapsids survive the mass extinction.

This result typifies how X-ray studies of fossils are like digging for them in the first place: you never know what you're going to find. That was true of a recent dinosaur scan (see "Dinosaur squeezes into ID17", below), and it was also true of one of the therapsids scanned for the previous study, *Choerosaurus dejageri*, which lived in the Late Permian period and whose lineage survived for a few million years after the mass extinction. Since the specimen was dug up almost a century ago, no-one had been sure why part of its snout was covered by paired bony studs, or bosses. Palaeontologists had believed the bosses were used for fighting, like the antlers of deer, but the tomography revealed that they were in fact innervated with numerous canals reaching up to the surface of the bone, meaning they would have been too sensitive for combat (*PLoS ONE* **11** e0161457). *Au contraire*, we suspect the bosses were used during the more romantic activity of courtship, and could even – if supplied with veins – have allowed the animal to blush.

Vincent Fernandez, ESRF



**Right: Unwelcome visitor.** A tomographic scan of a burrow reveals *Broomistega* (white), an amphibian, sharing a burrow belonging to a *Thrinaxodon* (brown), a therapsid.

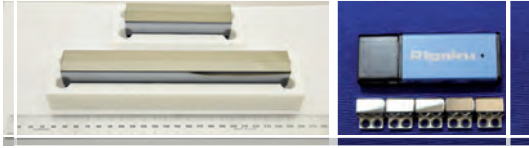
**Left: The Beaufort Group of fossil sediments (orange) lies within the greater Karoo Supergroup (red), which occupies some 800,000 km<sup>2</sup> of South Africa.**



V.FERNANDEZ/ESRF

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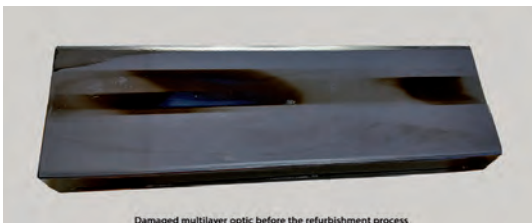


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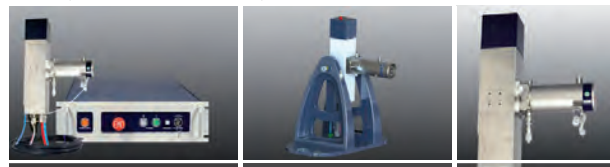
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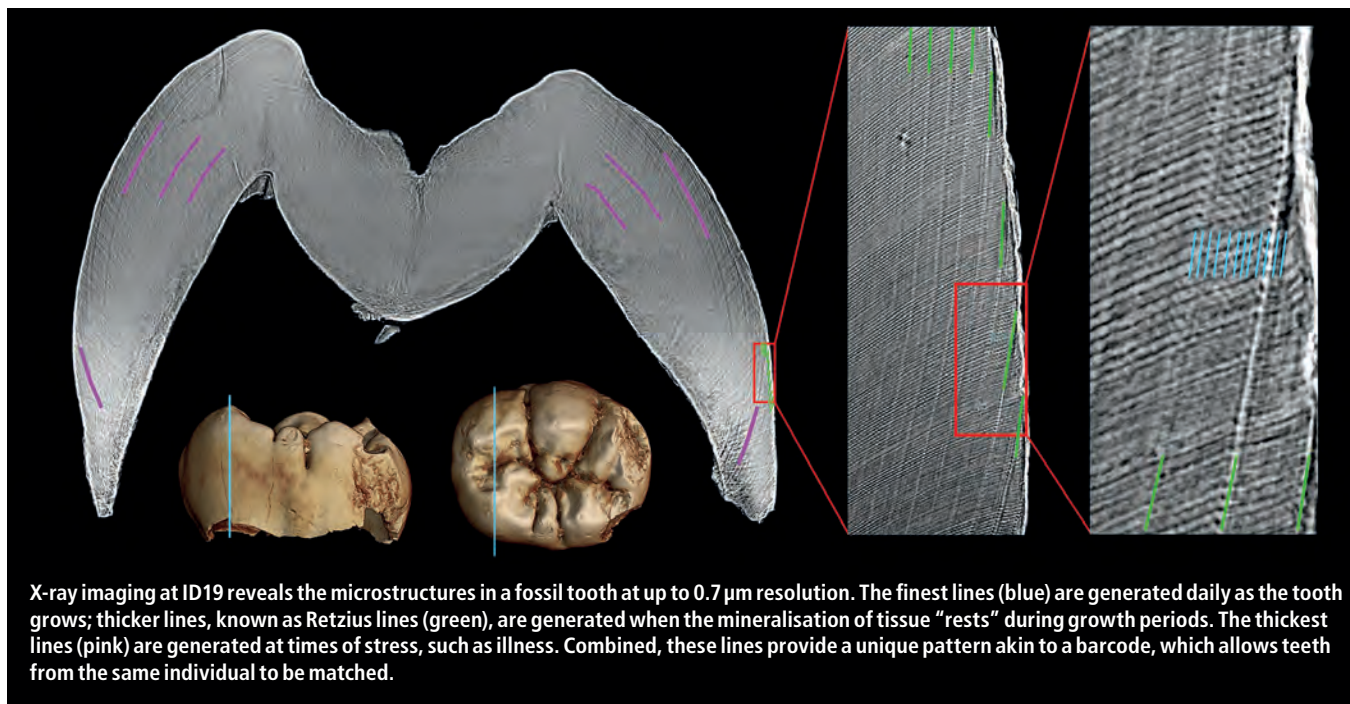
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# Teeth record ancestors' lives

Microstructures reveal whether early hominins grew up fast like apes, or slow like humans.



The Taung Child, the fossilised skull of a hominin – a human ancestor or a close relative – who lived some three million years ago, has intrigued anthropologists since its discovery in 1924. Its teeth look similar to a human’s, yet its primitive skull structure and small brain bear more relation to a young ape. These contradictory traits have led to the question: did our early ancestors grow slowly like us, or did they grow faster and with a shorter childhood like apes?

This question is important for understanding human evolution, because rates of growth are linked to other key physiological aspects, such as brain size and complexity. Fortunately, it turns out that the teeth of fossilised specimens can shed light on the puzzle, because their microstructure preserves records of birth, daily growth, developmental stress and death, allowing precise determination of age at death in juveniles. Observing dental microstructures often involves physically cutting up a tooth – a practice that has severely limited the samples that can be studied – but work at the ESRF’s ID19 beamline has shown that very similar information can be obtained from a synchrotron non-destructively (see figure above).

In 2010, initial studies showed that the evolutionary “cousins” of modern humans, the Neanderthals, developed faster on average (*PNAS* doi:10.1073/pnas.1010906107). Recently at ID19 we have gone a step further, by conducting the largest ever study of dental development in early hominins, using more than 20 fossils of four

ancient African groups: *Australopithecus anamensis*, *Australopithecus africanus* (of which the Taung Child is an example), *Paranthropus robustus*, and an early unknown species of *Homo*.

The first surprise of this study was a sizeable revision of some of the fossils’ ages at death: one young fossil hominin did not live to three years four months as previously reported, but more than one year longer. Another surprise was greater developmental variation than previously estimated. The hominin *P. robustus*, for example, exhibited particularly short molar crown formation times, late tooth initiation ages and, possibly, late molar eruption ages. In the case of *A. africanus*, even individuals from the same species exhibited markedly different growth rates. Finally, while the timing of molar crown formation in most fossils was more like chimpanzees than humans, certain *A. anamensis* and *A. africanus* individuals

exhibited more rapid development than either living group. (*PLoS ONE* doi:10.1371/journal.pone.0118118).

Following the rise of synchrotron studies on fossils, a debate arose as to whether intense X-rays could damage any ancient DNA preserved in some of the younger samples (see “The aDNA debate”, below). The resolution of that debate is good news for X-ray studies, as the more we look, the more we find that our earliest ancestors and their relatives prohibit simplistic “human-like” or “ape-like” characterisations – a fact that underscores the uniquely slow development of *Homo sapiens*. Having a long childhood appears to have allowed modern humans to grow bigger, more complex brains, which helps to explain why we are still here, while other hominins have not been as fortunate.

Tanya M Smith, Griffith University, Australia, and Paul Tafforeau, ESRF

## The aDNA debate

Fossils less than one million years old sometimes hide traces of ancient DNA (aDNA), which encodes valuable information about evolutionary relationships. While it is well known that X-rays also provide valuable information on fossils, recent concerns have emerged regarding their potentially harmful effect on aDNA. For synchrotrons – which deliver far more powerful X-rays than laboratory scanners – these concerns were even stronger.

Last year, a multidisciplinary team showed that while high X-ray doses can affect aDNA retrieval, the threshold for damage is thousands of times greater than for DNA in biological conditions: fossils do not contain water, which X-rays break up into harmful free radicals. Low-dose imaging approaches for ID19 developed by the team ensure that synchrotron and conventional X-ray imaging can safely be performed on recent fossils without fear of damaging aDNA (*Sci Rep.* doi:10.1038/srep32969). PT

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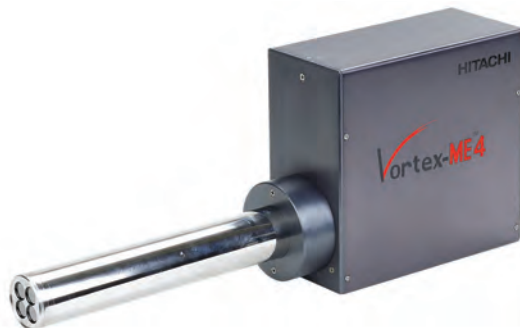
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# Seeking the land pioneers

An X-ray study of bone rings uncovers a twist in the search for the first land dwellers.

It is one of the most important events in the story of evolution: the transition from water to land. Fins became limbs, gills disappeared, and sucking turned to biting. Palaeontologists believe the switch occurred gradually in the Devonian period some 400 million years ago – but quite how this evolutionary transition played out, no-one has been sure.

*Acanthostega*, a metre-long vertebrate that resembled a giant salamander, is one of the most well studied animals of the water–land transition. It had legs – but they were feeble, and it still retained a lot of other fish-like characteristics, such as a large tail fin, and lateral lines on the skull, which fish use to sense movement in water. These and other features have led palaeontologists to believe that *Acanthostega* was purely aquatic, but there is always the possibility that, like modern amphibians, it started out life in the water and then crawled onto land only once it fully metamorphosed.

It is possibilities of this last type that, for want of data, palaeontologists have not been able to assess properly, says evolutionary biologist Sophie Sanchez at Uppsala University in Sweden. “There has been no serious attempt to investigate the life histories of *Acanthostega* – how long they lived or whether they had an aquatic juvenile stage, for example.”

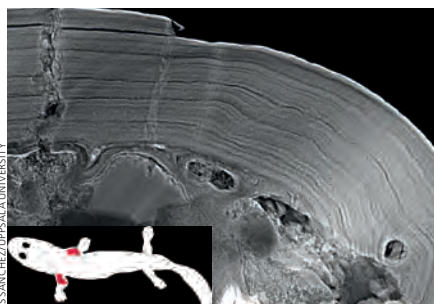
## Mass death

That is now changing, thanks to a new study by Sanchez and others of a “mass death” deposit of *Acanthostega* skeletons, which was discovered in 1987 in Greenland by Jennifer Clack of the University of Cambridge in the UK. Researchers believe that all these specimens died together when the small stream in which they were living dried out. Also, researchers had assumed the specimens were all adults.

Sanchez brought the fossils to the ESRF where, with help from Paul Tafforeau of the ID19 beamline, she scanned sections of the upper-arms, the humeri, using propagation phase-contrast synchrotron microtomography. The technique allowed them to see the bones’ annual growth rings – which, like tree rings, reveal age and growth rate – as well as the bones’ microstructure (see image above). “The fossils were dense like rock, so we needed powerful X-rays to go through them,” says Sanchez.

With additional help from Per Ahlberg

**Right: An *Acanthostega* fossil scanned at the ESRF**  
**Below: X-ray scans of the upper-arm bones (red, inset) of an *Acanthostega* fossil reveal growth lines, from which age and growth rate can be calculated.**



at Uppsala and Clack for analysis and interpretation, Sanchez and Tafforeau realised from the consistent spacing of the growth rings that the *Acanthostega* specimens must have still been growing, and therefore that they must all still have been juveniles, not adults, at their time of death, at eleven years or older. Moreover, the microstructures revealed that the bones were cartilaginous until late in the animals’ development, supporting the idea that they were still aquatic and unable to support themselves on land (*Nature* doi:10.1038/nature19354).

In one sense, Sanchez and colleagues’ study plunges the water-to-land transition into yet greater uncertainty, as palaeontologists do not now know what adult *Acanthostega* look like. “As we don’t have any adult *Acanthostega* at our disposal, we cannot say if they were aquatic or terrestrial,” explain Sanchez. “Our study

**“The dense fossils needed powerful X-rays to go through them.”**

therefore shows that we need to find adult [specimens] before being actually able to build up evolutionary theories on the tetrapod move to land.”

Given the late ossification of cartilage into bone in the juvenile specimens, Ahlberg speculates that *Acanthostega* may not represent transitional creatures at all, but tetrapods that were, evolutionarily speaking, settling into a fully aquatic lifestyle, like many modern salamanders. But whatever the truth on that subject, he adds, the study is pioneering itself, by investigating the life histories of the earliest tetrapods. “The popular literature in particular is full of pictures of Devonian tetrapods in landscapes, which somehow manage to give the impression that we know a fair amount about the actual lives of these creatures. That is very far from the truth.”

Jon Cartwright



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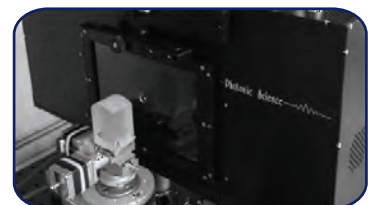
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Western France in the Cretaceous? According to palaeobotanists, the region could have borne similarities to the Isle of Pines in New Caledonia (pictured).



JEREMY REDD/SHUTTERSTOCK

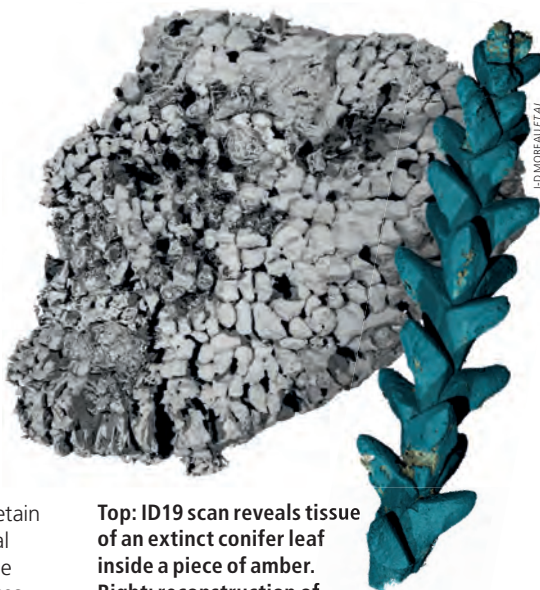
# Amber clues to ancient climate

From cloudy resin, a 100 million year-old plant emerges intact – and helps describe the Cretaceous climate.

The Cretaceous period, 65–145 million years ago, is perhaps most famous for seeing the end of the dinosaur age in a major extinction event. But with a relatively warm climate, the Cretaceous is also well known for its flora – dominated by ferns and conifers at first, and then progressively colonised by diversifying flowering plants. For palaeobotanists, however, questions persist. How did the diversity and the ecology of forests evolve? And what can their features tell us about the ancient environment?

Amber could be one of the best places to find answers. Unlike fossils found directly in sediments, which are more like imprints, fossils inside amber retain many of their delicate, three-dimensional features. In the case of plants, not just the external cuticle but also most of the tissues and cells are preserved. Generally these inner structures are inaccessible using standard techniques, such as light microscopy, and those that have been observed have required the specimens to be cut up beforehand. Worse, for cloudy ambers, which result from impurities in the resin, there has been no way to know if a sample even contains anything inside. But now we have demonstrated that the microstructures of ancient plants entombed in amber can be extracted non-destructively with synchrotron X-rays, at unprecedented resolution. Indeed, our studies of amber fossils open up a new window for the study of the entire histology of cretaceous plants.

The amber we study was dug up from



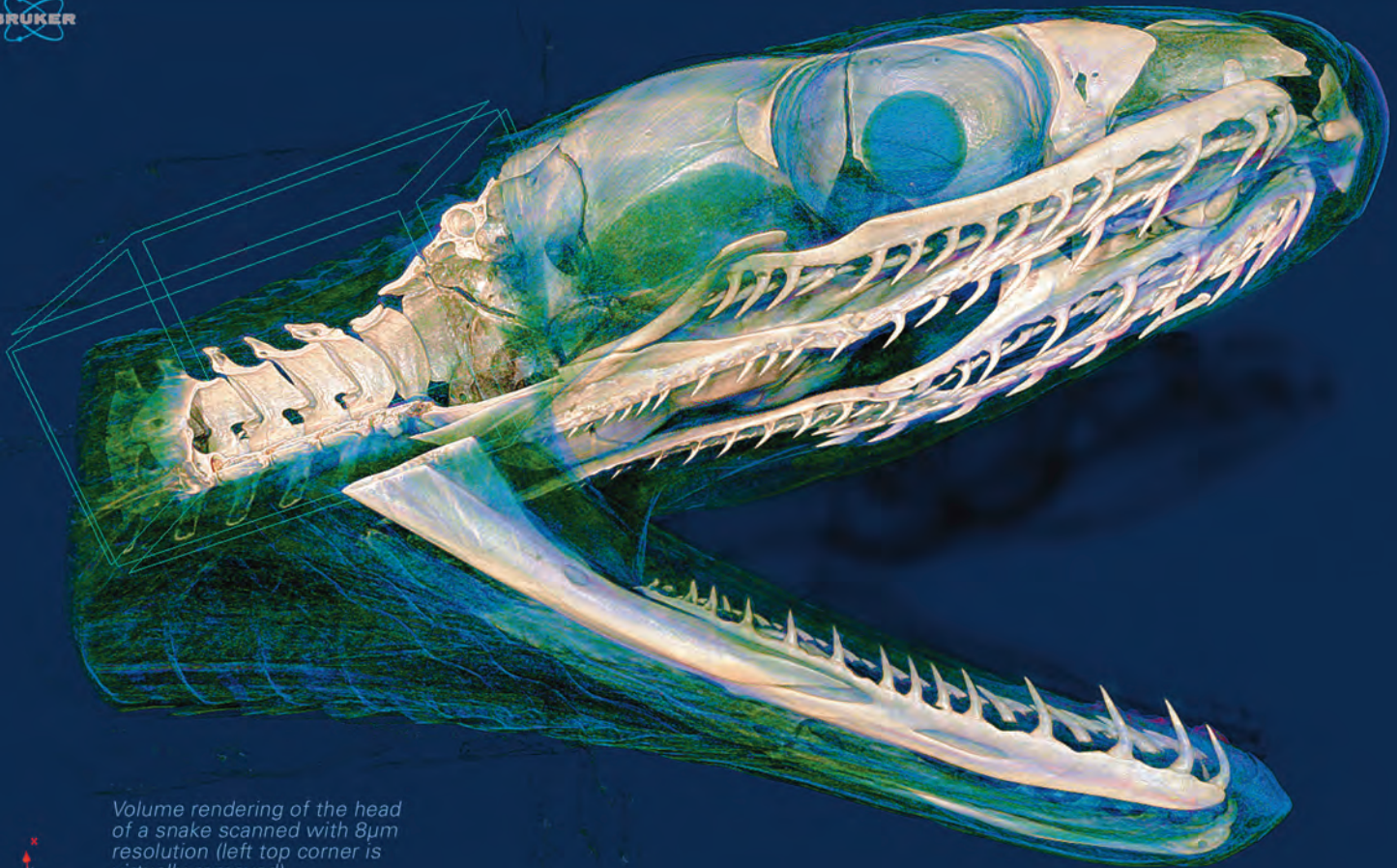
**Top: ID19 scan reveals tissue of an extinct conifer leaf inside a piece of amber. Right: reconstruction of the conifer twig.**

the end of the last century onwards, in sedimentary beds in the Charentes region of western France. The presence of charcoal beds, woods and other abundant plant remains clearly showed that this region was home to a Cretaceous forest. Previous studies have also uncovered amber containing dinosaur feathers, insects and mammalian hairs, while remains of crustaceans, algae and other aquatic life have suggested that the forest lay on the shoreline – like the Isle of Pines in New Caledonia (see image top). But in 2005, palaeontologist Malvina Lak of the University of Rennes 1 in France,

unearthed what was to become a particularly impressive specimen. Some 100 million years old, the 3 cm-long amber piece disguised its contents with a milky orange appearance, yet at the ESRF beamline ID19 we discovered that it withheld Charentes' first remains of a Cretaceous plant with full 3D preservation down to the cellular level – a branch of an extinct conifer, *Glenrosa carentonensis* (see image, left).

The microtomography revealed that *Glenrosa carentonensis* had small, thick leaves. Its respiratory cells were nestled in deep cavities – unknown among living conifers, and probably a way of controlling water loss, particularly in fast-changing conditions. In addition, it contained a protective outer layer of cells, which might have decreased the penetration of the sun's rays. All this suggested that *Glenrosa carentonensis* was adapted to withstand intense sunlight, hot and dry weather, and coastal environments exposed to salty winds and recurrent storms (*Ann. Bot.* **119** 117–28).

Studies like this demonstrate that the gross morphology and tissues of a plant fossilised in amber help to estimate ecological and climatic conditions which occurred in the past. More generally, they help us to reconstruct the Cretaceous forest ecosystems that were crucial for the evolution of the plant kingdom. In the future, we hope to find flowers or other reproductive structures in the Charentese amber, to gain new insights into the diversification of flowering plants, too. *Jean-David Moreau, University of Bourgogne Franche-Comté, France*



Volume rendering of the head of a snake scanned with  $8\mu\text{m}$  resolution (left top corner is virtually removed)

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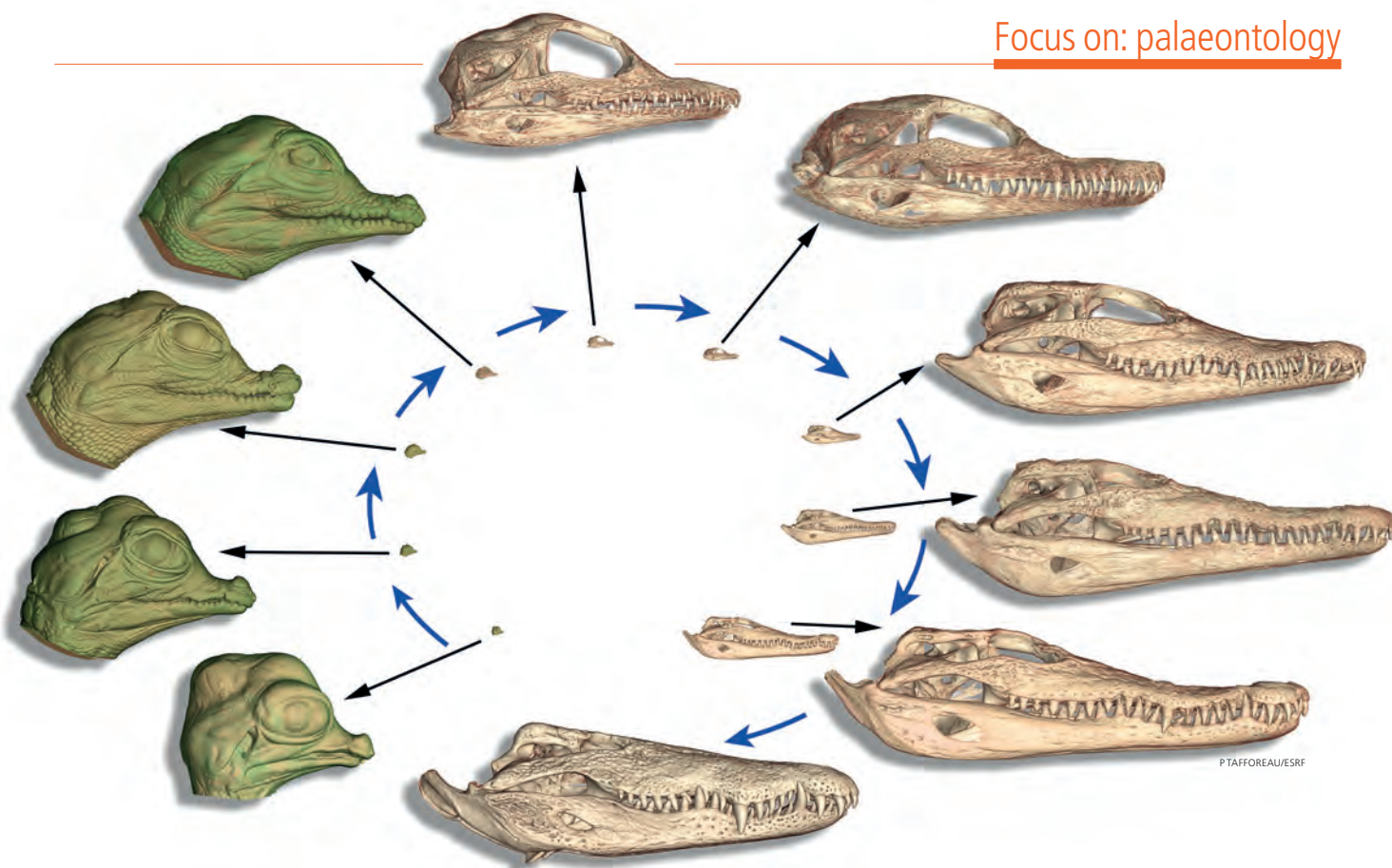


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# Like bird, like dinosaur?

Birds could shed light on the lives of extinct dinosaurs – if compared to crocodiles first.

Surprising though it may sound, palaeontologists do not only study fossils. To learn about important aspects of extinct animals that are generally not preserved in fossilised remains – soft tissues and behaviour, for example – they also turn to living representatives of the same groups. Birds, for example, are living representatives of dinosaurs, a group whose other members went extinct at the end of the Cretaceous period, 65 million years ago. By studying birds, we can explain biological particularities of the extinct dinosaurs – under the assumption that their anatomy at least partially preserves their deep family history.

In fact, birds descended from a specific group of meat-eating dinosaurs, and have since enjoyed a long and independent evolutionary history. That means their ancestors' features may have changed or disappeared altogether over time, while those that do remain may or may not relate to dinosaurs outside of that original group. But dinosaurs themselves fall within a larger group, the archosaurs, whose other branches include the extinct pterosaurs and all modern and extinct crocodylians – that is, crocodiles, alligators and so on. If we spot a feature that is common to both crocodiles and birds, we can assume that this feature is likely to have its roots back in the archosaurs – and therefore

## The growth pattern of the crocodile

The image above (clockwise, from bottom-left) shows the growth of the crocodile *Crocodylus niloticus* from 31 days, when still inside an egg, up to 20 years as a large adult male. At the bottom, the oldest and largest skull is about 64 cm long: the outer circle shows the other skulls scaled up to this size, while the inner circle shows their relative sizes.

The four first stages are embryos extracted from eggs at 31, 41, 62 and 72 days respectively; the following skulls are 93 days (a hatching), 3 months, 1.5 years, 3 years, 4 years and 20 years. While most of the samples are from La Ferme aux Crocodiles de Pierrelatte, the 3 and 4 year specimens are 2000 year-old Egyptian mummies from the Musée des Confluences in Lyon, France. The series illustrates how the shape of the embryos – which are superficially bird-like – drastically changes in their growth to adulthood, and could bring valuable insights into the origins of dinosaurs and their closest living relatives, birds.

must have been common to all dinosaurs, not just those from which birds descended.

This is the motivation behind a team of palaeontologists now working at the ID19 beamline, who are acquiring and studying tomographic data from crocodiles sourced from a crocodile farm, La Ferme aux Crocodiles de Pierrelatte, and from museum collections – the latter even including a few Egyptian crocodile mummies (see “The growth pattern of the crocodile”, left). The chosen crocodiles are at different growth stages, which will help to monitor the morphological changes that occur during the development from embryo to adult. Shifts in developmental timing represent important evolutionary opportunities; for example, humans develop more slowly than their ancestors, a fact that is betrayed by adult human skulls, which look more like those of baby than adult chimpanzees. In comparing the crocodiles' internal morphology and associated biological functions with the fossils of extinct dinosaurs and birds, including the earliest fossil bird, *Archaeopteryx*, the team hopes to gain valuable insights into the lifestyles of extinct dinosaurs, including the biological changes that occurred during the evolutionary transition from “non-avian” dinosaurs to birds.

Dennis Voeten, Palacký University, the Czech Republic

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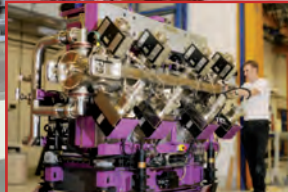
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# From chisels to X-rays

**Bruce Rubidge**, one of South Africa's leading palaeontologists, extolls the benefits of the ESRF for fossil exploration.

Bruce Rubidge was just five years old when he found an interest in palaeontology. On his family's sheep farm near the old town of Graaff-Reinet, South Africa, he would pedal his tricycle two miles to his grandfather's house, to discuss his grandfather's huge collection of local fossils. "It was uphill on the way back, so my parents used to fetch me," he recalls.

The fossil collection had a serendipitous origin. South Africa is home to the Karoo Supergroup, a sequence of rock layers some 12 km deep and 800 000 km<sup>2</sup> in area, deposited over 120 million years beginning in the Late Carboniferous period. Today the Karoo is widely recognised as one of the world's most important sedimentary basins because of its wealth of fossils, yet in the first half of last century it was still largely untapped. In 1934, Rubidge's grandfather, Sidney Rubidge, had come upon a fossil skull on the farm, and he sought advice from the then-leading palaeontologist in South Africa, Robert Broom, who informed him it was a new species. The two men began an informal partnership, with Sidney collecting the fossils and Broom describing them. By the end of his life, Sidney had amassed hundreds of fossils, including 118 new species. "Because of its wide diversity of species of vertebrates from around 250 million years ago, it is a very important collection," says Rubidge. "And from a global perspective too, as at that time the continents were all joined, and these animals had global distribution."

As a boy, Rubidge knew that he wanted to pursue his grandfather's hobby as his sole vocation. He read geology and biology at Stellenbosch University, and while doing a field project discovered the fossilised skull of a therapsid. The dominant group of fossil animals in the Karoo, therapsids are of great importance as the ancestors of modern mammals; the specimen



Rubidge uncovered happened to be a new species and the oldest therapsid ever unearthed in South Africa. "That intrigued me," he says, "and that's what really started my career." His first job was as a palaeontologist at the National Museum of Bloemfontein. A year later, he was head of department.

## No place like home

Tracing the origin of therapsids has led Rubidge around the world, to places such as Russia, China and Brazil where similar forms have been discovered. But it is in the Karoo, "the most wonderful place", where most of his research has focused. One of his group's milestones was their claimed confirmation in 2015 of a major extinction event 260 million years ago – the result of three decades of fieldwork, noting the sudden disappearance of fauna at certain levels in the rock record, and the emergence of new ones (*Proc. R. Soc. B* 20150834). They believe that the event, which coincided with the

onset of volcanism in what is now Emeishahn in China, saw the loss of 74–80% of land animals.

Rubidge spent 10 years at Bloemfontein before landing the biggest palaeontology job in the country: the director of the Bernard Price Institute (later the Evolutionary Studies Institute) at the University of the Witwatersrand in Johannesburg, where he remains to this day. It was here, in 2008, that the ESRF palaeontologist Paul Tafforeau gave a talk on the merits of synchrotron light to the field. Rubidge was impressed by the possibilities, and their co-operation helped form the basis for what was to become a highly productive ESRF–South Africa partnership. Though lab-based computed tomography scanners had been used in palaeontology for years, the resolution provided by the ESRF was breathtaking. Fossils inside rock that would have previously taken years or even decades to chisel out could be seen in all their glory, in a matter of days. "We can

now explore areas that we have never been able to see before," says Rubidge. "We can get inside skulls, we can look at the brain cases; we can even look at the paths of nerves and blood vessels."

Now, says Rubidge, palaeontologists have begun using synchrotrons to explore all sorts of fossils – even fossilised dung, to study prehistoric diets. But he still recalls one of the first ESRF scans with which he was involved: that of a fossilised burrow, which revealed the presence of two unusual bedmates, a therapsid and an amphibian that lived 250 million years ago (see p16). "When I saw the beautiful detail that Vincent Fernandez [now an ESRF palaeontologist, but then a postdoc in South Africa] got out of that burrow, it was absolutely amazing," he says. "You know, if you had prepared the fossil in the usual way, by removing the outside, you wouldn't have had the burrow cast left anymore – you wouldn't have had that evidence. It revolutionises our science." *Jon Cartwright*

## Bruce Rubidge in brief

**Born:** 1956, South Africa.

**Education:** BSc biology and geology (1978), BSc *Hons cum laude* palaeontology (1979), MSc *cum laude* (1983) Stellenbosch University; PhD geology/palaeontology (1988), University of Port Elizabeth.

**Career:** Palaeontologist (1980), Head of Palaeontology (1981), National Museum of Bloemfontein; Director, Bernard Price Institute for Palaeontological Research, University of the Witwatersrand (1990); Interim Director, Evolutionary Studies Institute, University of the Witwatersrand (2013–2017); Director, DST-NRF Centre of Excellence in Palaeosciences, University of the Witwatersrand (2013–)

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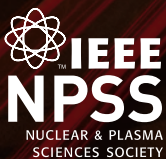
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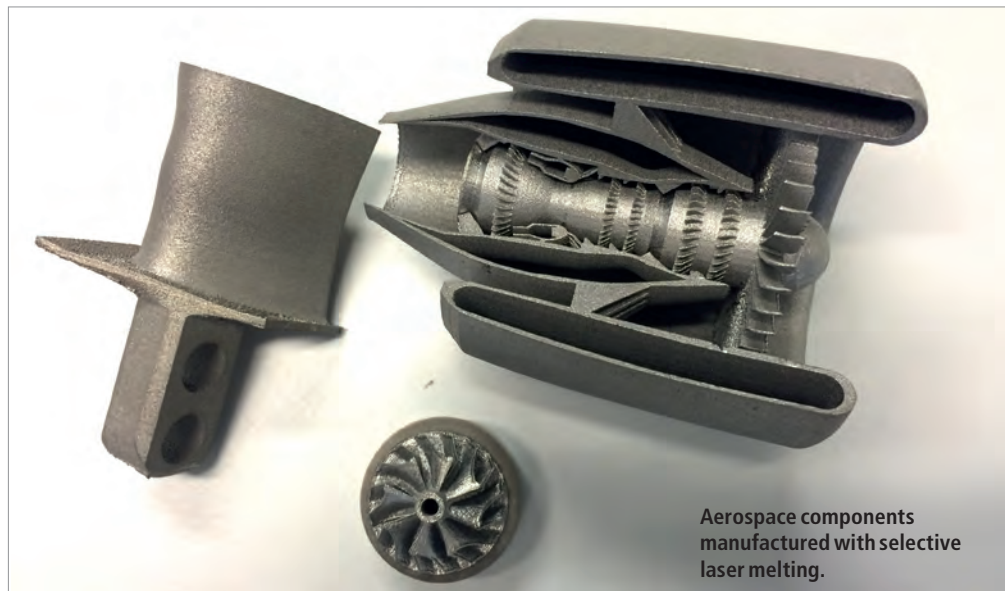


# Learning to print alloys

The ESRF is helping to improve the future manufacture of aerospace components.

Strong and stable, titanium and nickel alloys are increasingly used in the aeronautics and aerospace industries to meet the latest performance and environmental targets. They are traditionally cast or wrought into components, but manufacturers would like to switch to selective laser melting (SLM), a type of 3D printing. SLM can produce even lighter, more intricate components with more efficient use of materials, but currently leaves titanium alloys too brittle for aerospace.

Beginning with PhD work by Pere Barriobero-Vila at the Vienna University of Technology (TU Wien) in Austria, materials scientists at TU Wien as well as the German Aerospace Centre (DLR) have been working at the ESRF to understand the changes in microstructure that occur during SLM. Based on forged materials and target properties supplied by the Austrian company Böhler Schmiedetechnik, as well as SLM equipment and target properties supplied by the European aerospace joint venture Airbus Safran Launchers, the ultimate aim is to create titanium and nickel alloys via SLM that



Aerospace components manufactured with selective laser melting.

outperform their traditionally manufactured counterparts.

## Incomparable

"The ESRF is a unique X-ray source," says team member Guillermo Requena of the DLR. "It provides the temporal resolution we need to study phase transformation kinetics, and the spatial resolution necessary to determine the 3D

microstructures, like no other synchrotron source."

The performance of alloys comes down to their microstructure. Wrought or cast titanium and nickel alloys used in aerospace have microstructural arrangements that are engineered to provide the necessary mechanical properties – high fatigue resistance, for example – whereas SLM of, say, titanium alloys tends to produce microstructures that result in brittle behaviour. This is due to the high solidification and cooling rates in SLM, in which a laser quickly and selectively melts a bed of powdered alloy according to a 3D computer design. Engineers have ideas how to improve SLM-made alloys – for example, by warming the powdered bed, to reduce cooling rates – but they are lacking basic knowledge of how the microstructural phases react during thermal and thermomechanical treatments.

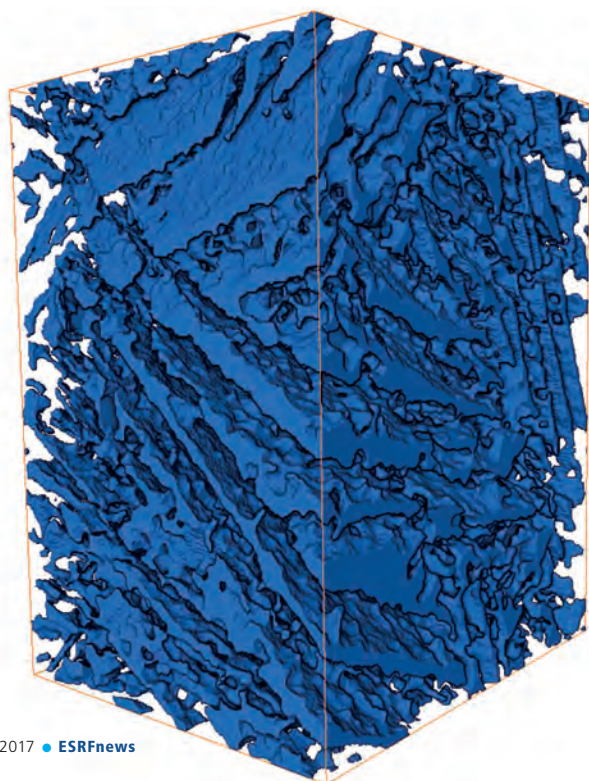
**Left: X-ray tomography performed at ID16A of a titanium alloy reveals the connectivity of the hexagonal titanium lattice (blue) with that in a cubic titanium lattice (transparent). The degree of connectivity affects the mechanical behaviour of the alloy.**

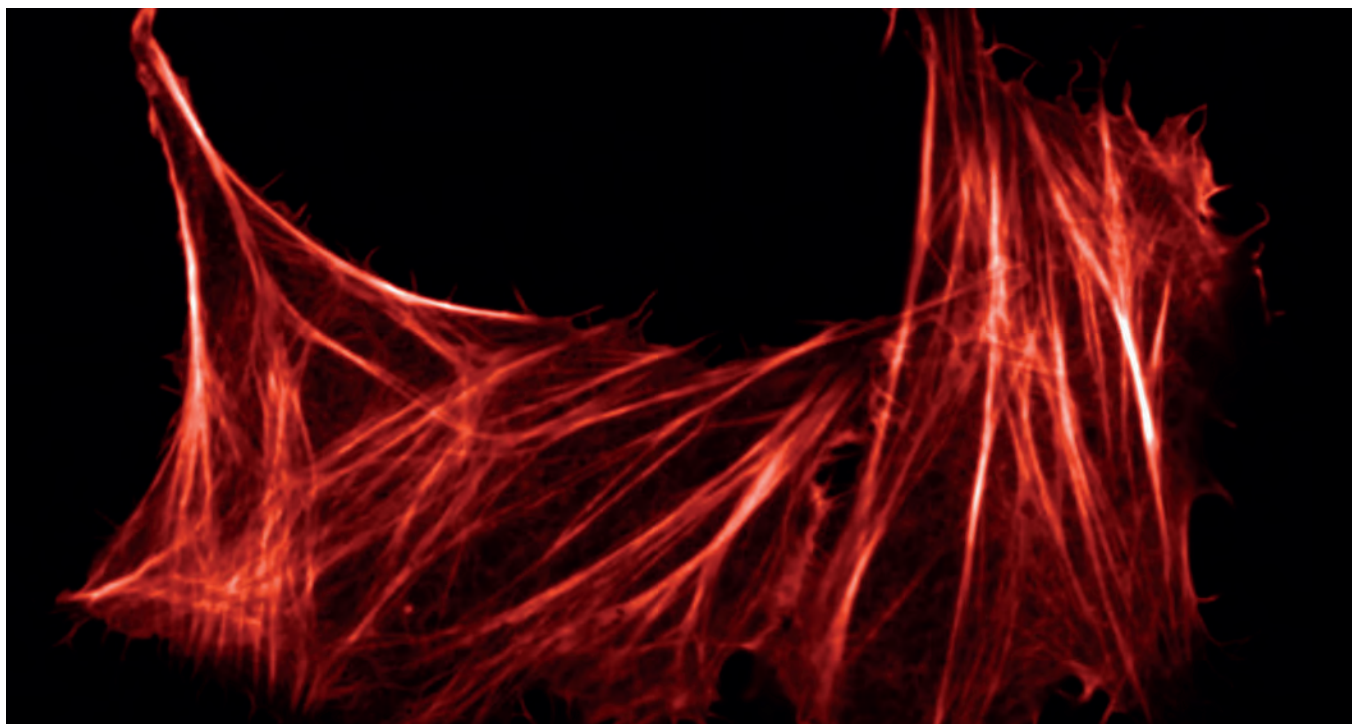
## A new phase

To gain this knowledge, since 2014 Requena and colleagues have performed high-energy synchrotron X-ray diffraction experiments at the ID15 beamline while heat-treating samples *in situ*, to determine the phase transformation's kinetics. They have also performed high-energy magnified synchrotron tomography at the ID16A beamline to identify morphological features of the microstructures, and their relationships to the mechanical properties.

Requena believes that the data will help him and his colleagues to develop new theoretical models to predict the alloy microstructures – and properties – that will result from different treatments during SLM. With any luck, they will hit on processing conditions that will allow all the manufacturing benefits of SLM, while retaining or even surpassing the properties found in traditional, metallurgical components. "We have to find the way to re-engineer these alloys and design new ones so they can achieve the necessary properties," Requena says.

Jon Cartwright





LINDSAY HAARBOSCH/UNIVERSITY OF AMSTERDAM

**Shining bright:** The brightest ever red fluorescent protein glows through the actin filaments of a mammalian cell, as observed through a fluorescence microscope. The protein, mScarlet, derives from proteins found in the 1990s in corals that for a long time proved hard to optimise. Scientists at the University of Amsterdam in the Netherlands, the Institute of Structural Biology (CEA/CNRS/University Grenoble-Alpes) and the ESRF have done so by identifying passages that repeated in the genetic codes of various different coral proteins; they joined these passages together into a new strand of DNA, which they then introduced into a bacterium to generate the mScarlet. Work at the ESRF ID29 beamline determined mScarlet's detailed arrangement, and the key to its brightness – a flat, rigid chromophore, which is responsible for the red light emission. The protein can be used to track cellular processes (*Nature Methods* **14** 53).

## In the corridors



### New website launched

The ESRF is the proud owner of a new website. With a modern design and revised navigation, the new [esrf.eu](http://esrf.eu) aims to increase the visibility of the ESRF and to provide easier access to ESRF web content and services. It also channels information through different entry points depending on the type of user, though the homepage still displays the latest news, scientific spotlights and events. Much care and work has been put into the new design, switchover and debugging phase, but there may be one or two errors. Users are advised to report any they come across to [communication@esrf.fr](mailto:communication@esrf.fr).

### Industry newsletter circulated



The ESRF's Business Development Office (BDO), helped by the Communication Group, is launching an industry e-newsletter that includes the latest news and case studies, as well as events and conferences where the BDO will be present. The newsletter aims to maintain client relationships and inspire new ideas for research and innovation. The first subscribers will be those on the BDO's contact list, although anyone can choose to subscribe (or unsubscribe) by contacting [communication@esrf.fr](mailto:communication@esrf.fr).

- Meanwhile, users are invited to follow a new showcase page on the social-networking site LinkedIn, "ESRF for

Industry", which provides a way for further outreach and to promote industry's success stories.

### Happy birthday, ILL!

The ILL, neighbour to the ESRF, celebrated its 50th birthday on 19 January by welcoming Thierry Mandon, the Minister of State for Higher Education and Research in France, Nikolaus Meyer-Landrut, the German Ambassador to France, and Edward Llewellyn, the British Ambassador to France. The original aim of the facility was to create an intense, continuous source of neutrons devoted exclusively to fundamental research in the civil realm. Founded in 1967, it produced its first neutron beams in 1971 and steadily grew in membership. In half a



century, it has generated 21,000 publications and one Nobel prize.

### Beetle battle

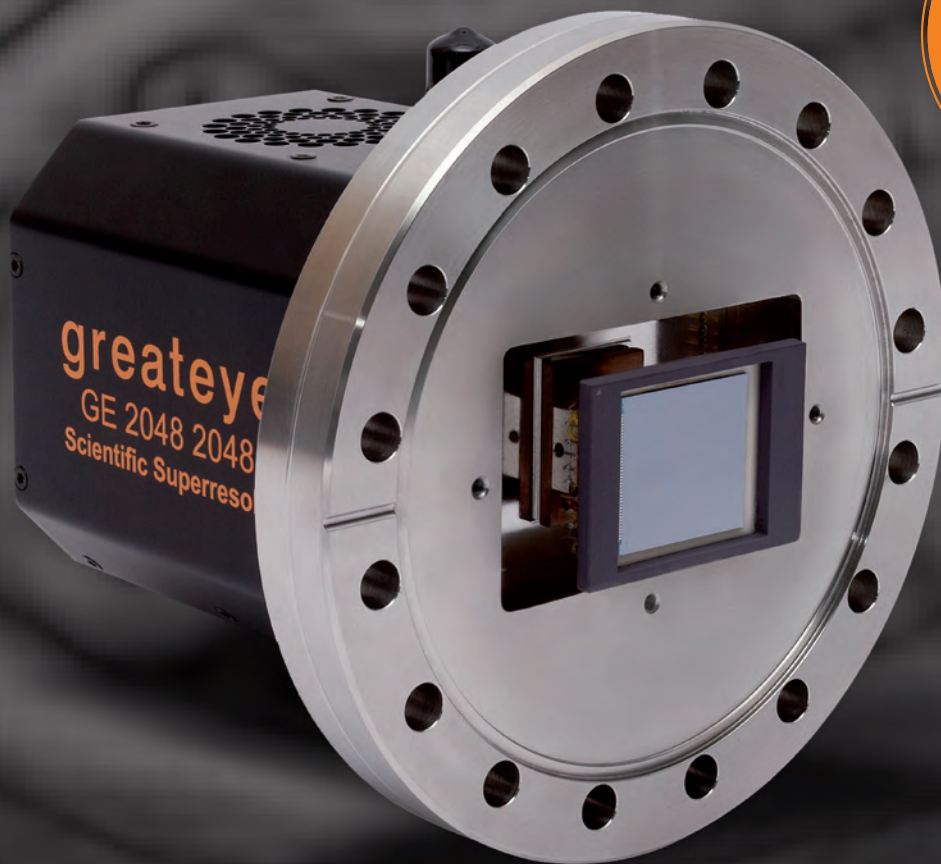


The Australian Synchrotron is gathering dust – not because it is now 10 years old, but because of an infestation of tiny beetles. According to the *Herald Sun* of Melbourne, "flea-sized bugs" were first noticed on the light source's roof a few years ago, but a pest controller was unable to tackle them because of the danger of fumigation to personnel and equipment. Since then, the beetles have been steadily nibbling through the 2500 or so compressed-straw roof tiles and generating piles of dust. A spokesperson denied that the bug problem was affecting operations; nonetheless, they are covering the tiles with polyester panels.

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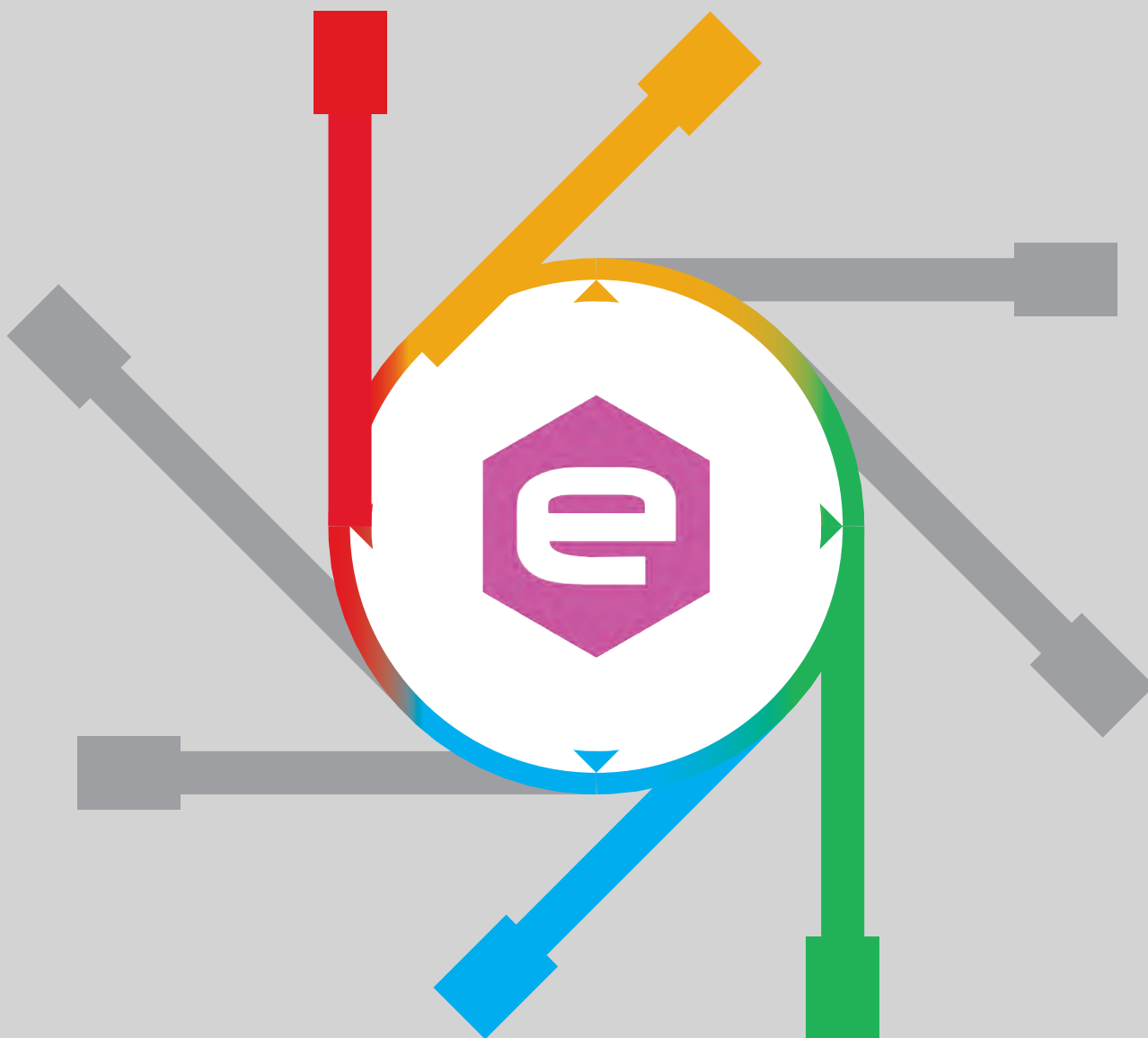
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