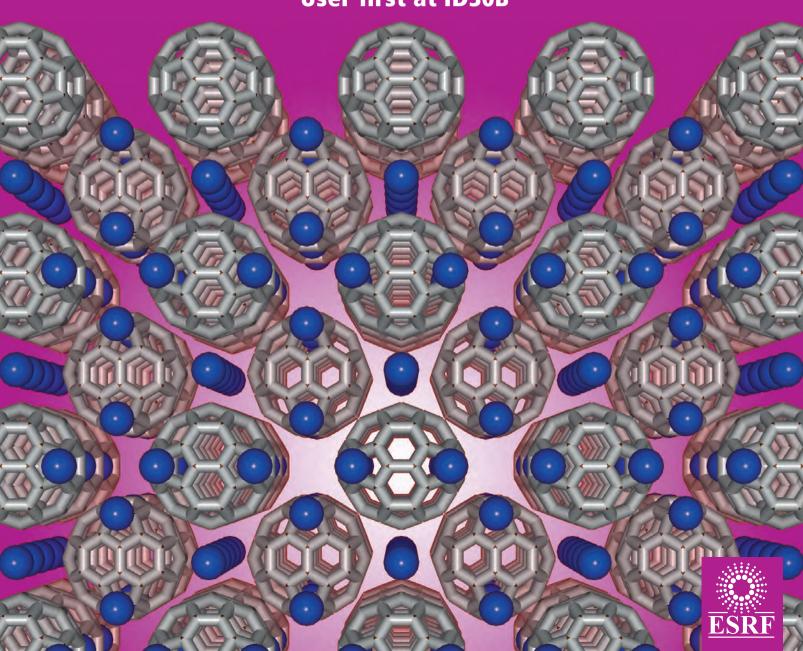
THE EUROPEAN SYNCHROTRON



Number 73 July 2016

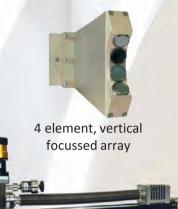
Matter at extremes

Magnets reach atomic scale User first at ID30B



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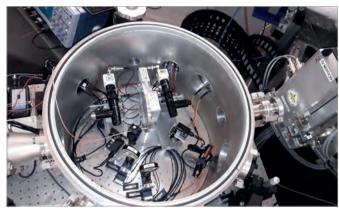
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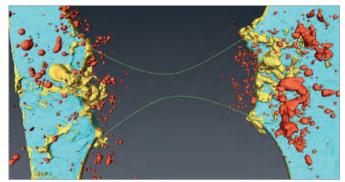
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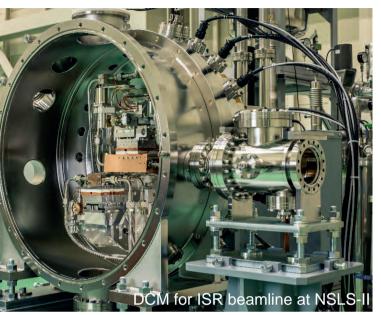
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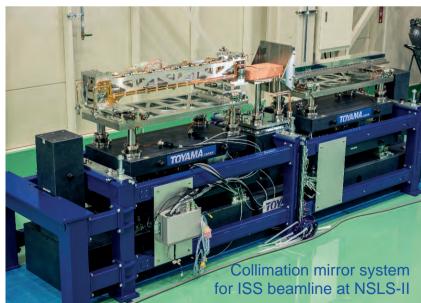


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A landmark for European science

In 1988, 11 European countries joined forces to build the brightest third-generation light source in the world. For more than 20 years, the development and success of the ESRF has relied on the strong support of its now 21 partner countries. Together we are driven by a common quest for scientific excellence, the commitment to enable research at the forefront, and the desire to show that science provides benefits to wider society.

On 10 March, the European Strategy Forum on Research Infrastructures (ESFRI) presented the 2016 ESFRI roadmap update, which contains 21 projects and 29 "landmarks" singled out for their scientific excellence, pan-European relevance, socio-economic impact and innovation. In most cases, landmarks are ESFRI projects that have appeared in previous roadmaps and that have either reached implementation or are in an advanced construction stage. The ESRF Upgrade Programme Phase I, which was successfully completed last year, is one such landmark. It has appeared on the ESFRI roadmap since its inception in 2006, underlying the ESRF's key contribution to European science and innovation.

The 2016 roadmap contains eight new entries from a total of 50 projects and landmarks. We are proud that the ESRF–EBS project is one of them. By delivering a conceptually new and first-of-a-kind storage ring, the ESRF–EBS project will provide scientists with unprecedented X-ray tools to better understand the infinite ways atoms combine, give origin to materials and make life possible.

"The inclusion of ESRF–EBS in the ESFRI roadmap reinforces our founding principle"

The inclusion of the ESRF in the 2016 ESFRI roadmap also reinforces the ESRF's founding principle of supporting and enabling scientific excellence, as established in 1988 with the signing of the ESRF convention. Today, the growing complexity of science and technology, combined with global challenges in areas such as health, environment, energy and food security, require even greater international co-operation to share costs and investments, in addition to increased collaboration between different research disciplines. The sustainability of our world, based on growth rooted on the principles of peace, democracy and mutual respect, can only flourish and advance if it goes hand-in-hand with cutting-edge research. Large international research infrastructures such as the ESRF have a leading role to play in demonstrating collaboration for the benefit of science and society.

This issue, dedicated to matter at extremes, offers a snapshot of the scientific breakthroughs that have been achieved in this field thanks to the ESRF Upgrade Programme. Users are carrying out experiments across many fields ranging from Earth and planetary sciences to fundamental physics, chemistry and materials research, and are now extending such studies into the biophysics and biochemistry arena – including questions concerning life's biological function under extreme conditions. In the frame of the ESRF–EBS project, the ESRF will maintain the spirit of exploration by developing new scientific projects to further expand its high-pressure capabilities and reach new extreme states of matter.

Francesco Sette, ESRF director-general

In brief



Coherent imaging puts the heat into solar cells

A team of ESRF users from France, Italy and the Netherlands has shown that phonons can be confined in sub-micron structures, which could help enhance the output of photonic and photovoltaic (PV) applications. Using a pioneering technique at ESRF beamline ID10 called coherent diffraction imaging (CXDI), the researchers investigated the photoluminescence of large silicon nanoparticles and observed an enormous photoluminescence emission band in the infrared upon increasing laser irradiance (Scientific Reports 6 25664).

"Phonon management is now increasingly recognised as an important ingredient for boosting the efficiency of PV cells," explains team member Giovanni Mannino of the National Research Council (CNR), Italy. "Typically, more than 50% of energy absorbed by a solar cell is converted into heat, but we show that this heat can be used to formulate a new PV strategy: rather than avoiding heat generation one can accept it as unavoidable and then try to make use of it."

CXDI is a powerful technique for imaging micrometre-sized objects at very high resolution in 3D and ID10 is currently the only facility where such imaging is possible, he adds. "Having structural information in 3D is essential for determining bulk properties like the porosity, and we can also look inside the morphology of our samples non-invasively."

Hercules goes from strength to strength

The 26th Hercules training school (Higher European Research Course for Users of Large Experimental Systems) took place on the EPN campus from 29 March to 29 April, giving young researchers hands-on experience of some of the world's most advanced light sources. Taking place every year since 1991, the Hercules school brings together around 80 scientists from all over the world and exposes them to the techniques and scientific possibilities available at neutron and synchrotron-radiation sources. Competition is fierce, with only half of applicants obtaining a place on the course. This year. Hercules included

students from Russia, South Africa and Taiwan – some of whom are already studying for their PhDs at a synchrotron facility. The five-week event saw students divide their time between the ESRF, Institut Laue-Langevin and Institut de Biologie Structurale, as well as spending one week at either: Soleil and



On 22 April Taiwanese students attending the Hercules school received the visit of Ming-Zhong Zhang, Ambassador of Taiwan in France.

Laboratoire Léon Brillouin in Paris-Saclay; DESY and the European XFEL in Hamburg; Elettra and Fermi in Trieste, Italy; or PSI in Villigen, Switzerland. "It has been very exciting," said attendee Alexandra Mannig from ETH Zûrich in Switzerland. "The practicals give you a different view of the facility compared to the lectures, although both are necessary."

Hercules is co-organised by the Université Grenoble Alpes and Grenoble Institute of Technology, in partnership with the CNRS, CEA and relevant large-scale research infrastructures. A visit from the Ambassador of Taiwan in France, Ming-Zhong Zhang, this year provided an opportunity to present the school in presence of the president of COMUE Grenoble, Patrick Lévy, the president of the University Grenoble-Alpes, Lise Dumasy, and the Administrator General of Grenoble INP, Brigitte Plateau (see photo).

Car industry seeks out benefits of X-rays

In partnership with CEA-Tech, the technology research unit of the French Atomic Energy and Alternative Energy Commission, the ESRF welcomed a delegation from Nissan and Renault on 27 May. The visit provided the ESRF with an opportunity to highlight its unique expertise in highenergy synchrotron X-ray studies of batteries under operating conditions, and the new research perspectives offered by ESRF–EBS.



Machine tops up

Top-up operation began at the ESRF during the last week of April, providing users with better beam stability, lower vertical emittance and a near constant beam current.

The new operation mode ensures that the storage ring is refilled with electrons every 20 minutes, resulting in a much higher integrated current. This translates to more photons for users and a better beam stability due to smaller current variations, in addition to a low vertical emittance in all filling modes. "This is really good for us," explains Sakura Pascarelli, head of the Matter at Extremes group. "Apart from the periodic injection instability, we take full advantage of having almost constant X-ray power on the optics."

A new ramped injection power supply (RIPS) system for the booster has also been commissioned. This will further improve top-up operation by enabling cleaning to be carried out in the booster, which reduces disturbance to the beam during and after injection. "We're looking forward to detailed feedback from users and we expect to implement future refinements over time," said Pantaleo Raimondi, director of the Accelerator and Source Division.

New EU nano platform

A new distributed platform for nanoscience, which is available to all ESRF users, was launched on 12 April. Funded by the European Union, NFFA (Nanoscience Foundries and Fine Analysis) offers scientists co-ordinated access to dispersed European nanoscience laboratories for state-of-the-art synthesis, nanofabrication and nano-characterisation. Potential users can now check a box on ESRF experiment proposal forms to signal their interest in accessing NFFA, which does not affect the ESRF proposal itself (www.nffa.eu).

Study gets to the heart of fossils

An international team led by researchers in Brazil has discovered a fossilised heart in a 119 million-year-old fish, providing clues about the evolution of the heart in vertebrates. Until now, no cardiac structure in vertebrates has been observed in the fossil record because the heart is made from soft muscle tissue and normally decays much faster than bone.

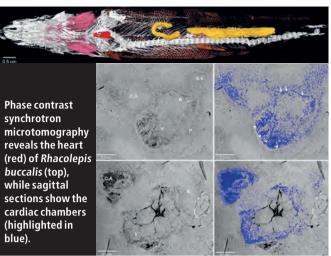
First author Lara Maldanis of the University of Campinas and the team used X-ray microtomography at ESRF beamlines ID17 and ID19 to test approximately 60 fossils from Rhacolepis buccalis, an extinct ray-finned fish that lived during the Cretaceous in a geographical region that is now Brazil. X-ray microtomography allows fossils to be imaged in thin sections, which were then processed to render the heart slice by slice and to digitally restore features of the organ. "The combination of the ESRF's large beam, high energy and high coherence made it the perfect tool," explains Vincent Fernández of the ESRF.

Two of the samples revealed fossilised hearts, allowing

Metallic ink revealed in ancient scrolls

Metals were used in ink several centuries earlier than previously thought, according to new results obtained at the ESRF. An international team of researchers used a combination of synchrotron techniques to resolve the chemical composition of ink in two fragments of Herculaneum papyrus that were carbonised during the eruption of Mount Vesuvius in 79 AD. Discovered more than 250 years ago, the papyri contain unique philosophical texts and constitute the only complete library dating from antiquity.

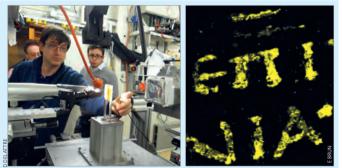
Last year, researchers used X-ray phase contrast tomography at ESRF beamline ID17 to decipher words and reconstitute an almost complete Greek alphabet from inside the badly damaged and rolled papyrus scrolls (*Nat. Commun.* **6** 5895).



observations of the heart's architecture – in particular a conical extension of the ventricle that helps regulate blood outflow via valves called the conus arteriosus. While most living ray-finned fish have one valve row in the conus, the ESRF data revealed that the heart of *Rhacolepis* contains five. The research therefore suggests that the fossil represents a transitional morphology between a primitive multivalvar, conal condition and the

monovalvar structures found in modern ray-finned fish, which is the largest group of vertebrates alive today with nearly 30,000 species (eLife 2016; 5:e14698).

"This work will stimulate researchers all over the world to place their best-preserved fossils under synchrotron light to find further cardiac fossils, so we can rapidly fill up the gaps in their fascinating evolutionary history," said José Xavier-Neto of the Brazilian Biosciences National Laboratory in Campinas.



A papyrus fragment being set up for a diffraction study (left), and X-ray fluoresence image showing the level of lead contained in the ink.

This latest study saw members of the team return to the ESRF. where they combined multiscale X-ray fluorescence (XRF) microimaging at ID21, X-ray diffraction at ID11 and X-ray absorption near edge structure on BM26 to analyse the ink composition. In particular, XRF data revealed that the ink contained high levels of lead, which could not be attributed to lead from water contamination or from contact with bronze containers (DOI:10.1073/pnas.1519958113 and DOI:10.1038/srep20763).

Until now it was assumed that metal was only introduced to carbon-based ink from the 4th century AD, but the findings put this date back to the first century. "For nearly 2000 years, we thought we knew everything, or almost everything, about the composition of antique ink used to write on papyrus," says papyrologist Daniel Delattre of the CNRS-IRHT-Institut de Recherche et d'Histoire des Textes. "The highly specialised studies carried out at the ESRF show us that we must be wary of our ideas."

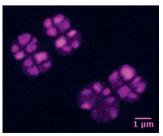
Extreme bacteria viewed under the nanoscope

Researchers have used the advanced nano-imaging capabilities of the ESRF to investigate why certain bacteria can withstand very high doses of ionizing, ultraviolet and gamma radiation. Deinococcus radiodurans is an extremophile bacterium that presents the highest resistance to extreme conditions of any known organism. One hypothesis is that the organism contains high amounts of manganese, which may act as a scavenger of reactive oxygen species and therefore protect macromolecules from damage. But there are still numerous guestions to address, such as how the manganese concentration is regulated and stored.

Célia Romão of the Instituto de Tecnologia Química e Biológica at Universidade Nova de Lisboa and co-workers used X-ray fluorescence imaging at nanometre resolution at ESRF beamline ID16A-NI to look at the cellular distribution of manganese on *Deinococcus radiodurans* cells and to study how they respond to damaging environments.

"The use of the nano-imaging beamline ID16A-NI has been crucial to study the sub-cellular complexity of metal homeostasis at nano-resolution," says Romão.

The results showed that manganese is localised in specific regions and co-localises with other metals, such as phosphate. However, under specific conditions, the researchers observed a difference in metal distribution. "The high spatial resolution obtained at the nanoscale is a big step forward in our understanding of the molecular mechanisms underlying the extreme resistance of this organism," Romão told *ESRFnews*.



X-ray fluorescence microscopy shows the presence of manganese.

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Pantaleo Raimondi (left), MAC vice-chair Hans Braun of the Swiss XFEL, MAC chair Richard Walker of Diamond Light Source and ESRF director-general Francesco Sette at the third MAC meeting.

MAC notes impressive EBS progress

The third Machine Advisory Committee (MAC) meeting took place at the ESRF on 21 and 22 April, at which the ESRF–EBS team presented progress made during the past six months to the panel of international accelerator experts. ESRF–EBS, the revolutionary new storage ring due to be operational at the ESRF from 2020, is now well into the procurement phase and the MAC exists to advise the project team.

Around 90% of the design work for the new machine has been completed, with only the one-of-a-kind "special" vacuum chambers and magnets remaining, and all of the critical prototypes and tests have been successful. Concerning procurement, approximately 60% of the planned calls for tender for equipment have been launched, with all of the magnet contracts and three of the vacuum chamber contracts having already been signed on time and on budget.

The panel was impressed with the progress of the project. In particular, the MAC commended the Insertion Device Group for the detailed work that has been carried out, which allows the ESRF to offer a range of different X-ray sources (namely the lattice dipoles or additional short bending magnets, two-pole wigglers or three-pole wigglers) to ESRF and CRG bendingmagnet beamlines.

Feedback from MAC members included a suggestion to test as much as possible the crucial tasks to be carried out during the installation phase. This could be performed by placing four fully assembled girders in a mock-up tunnel and executing all the steps foreseen such as cabling, piping, vacuum connections and alignment. "All the recommendations were in line with what has been studied by the project team and will greatly contribute to the advancement of the project," said ESRF-EBS project leader and director of the Accelerator and Source Division. Pantaleo Raimondi. The next MAC meeting will be held on 22 and 23 September 2016.

Users express interest in new beamlines

Following the ESRF's call for expressions of interest for its Extremely Brilliant Source (EBS) in October, 48 proposals were received before the deadline on 11 March. The call mobilised scientists from across Europe and from all areas of research represented at the ESRF, demonstrating the keen interest of the user community for the ESRF–EBS project.

The major technological challenge of ESRF–EBS, which is due to be delivered in 2020, is the construction of a new low-emittance storage ring, followed by the construction of four new beamlines and the refurbishment of several others. To ensure that the user community is actively involved in shaping the science case of the future X-ray source, the ESRF invited ideas for new science to be performed on upgraded beamlines, endstations and instruments.

The proposals were either submitted by individuals or, in many cases, by large groups of scientists working in specific areas such as spectroscopy, science at extreme conditions and structural biology. ESRF management presented a summary of the proposals at the meeting of the Science Advisory Committee on 26 and 27 May. Plans are now being drawn up to host a dedicated user workshop in December to discuss potential ESRF-EBS beamlines, before final approval is sought from Council next year.

EBS seminars get under way

The first of a series of events to raise the profile of the ESRF–EBS project among other facilities, and to exchange expertise, was held at the ESRF on 2 June.

Pedro Fernandes Tavares, who became machine director at MAX IV in Sweden in February, delivered a seminar about the MAX IV project. This novel storage ring, which was inaugurated on 21 June, is the first of a new class of light sources based on the multibend achromat lattice (MBA).

This machine aims to achieve an ultralow horizontal emittance to

increase the X-ray brightness and has inspired many other facility upgrades including ESRF–EBS.

Tavares, who has experience in all aspects of the design, construction, installation and commissioning of MBA-based storage rings, stressed the importance of communication when delivering large projects such as ESRF–EBS. "You have to understand the limitations, identify bottlenecks in due time and try to get help from other labs," he said.

The ESRF–EBS seminars, which will take place once per



Pedro Tavares delivers the first of a series of talks to raise the profile of ESRF–EBS and encourage collaboration.

month and are open to all ESRF staff and users, are designed to encourage collaboration between facilities and to share experience in the construction of low-emittance storage rings.

News from the User Office

An impressive 1249 proposals were submitted at the last proposal submission deadline on 1 March, requesting a total of nearly 18,000 shifts of beam time. This is an all-time record for the ESRF, bringing the total number of proposals received for beamtime in 2016 to 2384. On 27-29 April, 117 external scientists met at the ESRF to evaluate the new proposals and provide recommendations for beam time allocation, and we thank them for their contributions to this very important task. In the end, 507 proposals were allocated beam time (40% success rate) totalling nearly 7300 shifts.

News from the beamlines

 Structural dynamics beamline ID09 has become dedicated to time-resolved experiments following the transfer of the high-pressure part of the beamline to ID15B in January. ID09 is designed for pumpprobe experiments in chemical and biological systems, allowing users to measure structural changes induced by short laser pulses. The beamline has setups for Laue diffraction, small- and wide-angle scattering and X-ray emission spectroscopy (XES). The time resolution can be pushed to the single-pulse limit of 100 ps in most experiments and, with the increase in beam time available, more complex experiments can now be undertaken. For example, combining wideangle scattering with XES allows unique information to be obtained about changes in the average distances between pairs of atoms, whereas XES probes atom-specific changes in the spin or oxidation state of transition metals. In February, a new large area CCD detector was installed, a Rayonix 170 HS, which significantly improves the signal-to-noise ratio and Q range in many experiments. Please contact ID09 scientist Michael Wulff (wulff@esrf.fr) for more information.

• ID11 has commissioned a new end-station optimised for sub-micron (0.15–0.6 µm) beams at high energies (30–

The next deadline for standard proposal submission is 10 September for beam time during the period March to July 2017. Potential proposers are invited to check the status of open ESRF beamlines for the 10 September deadline (www.esrf. fr/UsersAndScience/UserGuide/ Applying/beamline-status).

The ESRF User Meeting 2017 will be held on the EPN Science Campus from 6–8 February 2017 inclusive. We very much look forward to welcoming users on site for this important event which, in addition to the plenary session, will include a range of tutorials on topics relevant to users and user-dedicated microsymposia covering three major scientific themes.

News from the User Organisation Committee



The User Organisation would like to thank all participants of the User Meeting

2016 who completed our feedback survey, which acknowledged the high quality of the speakers and the organisational work carried out by ESRF staff. We are now preparing the programme for the User Meeting 2017 and invite users to suggest tutorial topics. The meeting will see changes to the eligibility conditions for the Young Scientist Award, a call for which will be opened in the summer: from 2017, nominees can be of any nationality with 2–7 years of experience after their PhD and must be younger than 37. We also invite users to continue to help shape the experimental programmes for ESRF-EBS by participating in dedicated workshops during 2016 (see p9). Finally, we would like to remind all users that they are welcome to contact the User Organisation directly at any time via e-mail. Representatives of each user scientific community can be found at www.esrf.eu/ UsersAndScience/users org. Paola Coan, chair of the UOC

A new press at ID19 is available to the user community through standard proposals (top right: a sample scanned using the press).

60 keV), greatly enhancing its capabilities to exploit small focal spot sizes.

• Work on upgrade beamline **ID15** continues with the

assembly and testing of beamline components. Commissioning of the beamline will start in September, with first user experiments expected in November. • A new *in situ* microtomography press called Hades (image, left) has been installed at beamline **ID19** as part of a Long Term Project undertaken by the University of Grenoble, allowing time-lapse microtomography to be performed at pressures up to 100 MPa and temperatures up to 525 K.

• An Eiger 4M detector has been installed and successfully commissioned at MASSIF-3 (**ID30A-3**), which has been in user operation since December 2014. Besides the detector's reduced pixel size of 75 µm², individual data sets can now be collected at frame rates of up to 750 images per second. This allows high-quality data to be collected in favourable cases in the sub-second time range.

 Users at the structural biology beamline ID30B can now exploit the full potential of its FlexHCD sample changer robot by manipulating samples mounted both in SC3-like SPINE baskets (i.e. base-up, with vials, 10 samples per basket) and in vial-less Unipucks (i.e base-down, no vials, 16 samples per Unipuck) during the same experimental session. Additionally, in situ data collection from crystallisation plates is currently being commissioned and will be made generally available to users from September 2016.

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Feature

Atomic magnets come into view

X-ray magnetic circular dichroism at beamline ID32 has helped prove the existence of single-atom magnets, which could find applications in storage or logic devices.

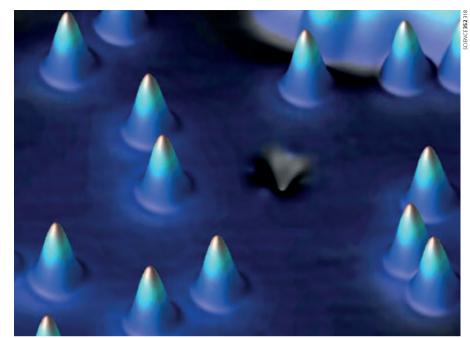
Steady miniaturisation during the past few decades has meant that magnets can now be found in a vast range of technologies. Modern rare-earth permanent magnets, which have 10 times more usable magnetic energy than ferrite-based magnets, have led to miniaturised electric motors for electromechanical actuators in industry, cars and consumer electronics. Likewise, the miniaturisation of magnetic bits makes it possible to store terabytes of information in magnetic hard drives.

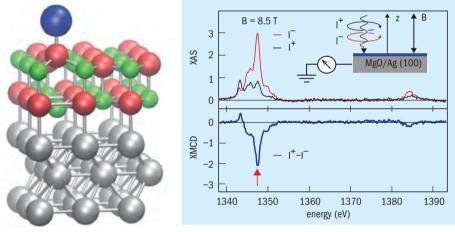
But the miniaturisation of magnets, like that of transistors, cannot go on indefinitely. Eventually, thermal fluctuations overcome the anisotropy barrier that keeps the magnetic axis stable, leading to the loss of permanent magnetisation. Scientists have therefore been working for a long time to reduce the size of permanent magnets to the smallest possible limit.

A recent study led by the present authors based on experiments at the ESRF and the Swiss Light Source (SLS) has taken us closer than ever towards this goal. We have been able to show that single holmium (Ho) atoms deposited on a nonmagnetic magnesium oxide (MgO) substrate display high coercivity (the magnetic field required to reverse the magnetisation) and magnetic remanence (the magnetisation that "remains" in the absence of an external field), on timescales lasting up to an hour. Such a long-lived magnetic state enables individual atomic spins to be manipulated using microwave fields and even DC electric currents, which is key for the development of memory devices based on single-atom magnet arrays or logic devices based on ensembles of a few atoms (Science 352 318).

Unprecedented stability

We used ESRF beamline ID32 to probe holmium electrons with element-specific sensitivity, also providing information on the system's magnetic spin and orbital moments. Specifically, X-ray magnetic circular dichroism (XMCD) measurements at the M_{4,5} absorption edges of Ho showed that the Ho atoms possess a high magnetic moment in a magnetic field of 8.5T and at a temperature of 6 K. Surprisingly, when we measured the magnetisation as a





STM image of an ensemble of holmium atoms deposited on a MgO thin film (top). XAS and XMCD measurements taken at the ESRF (above right) led to a magnetisation curve showing hysteresis.

function of applied field we observed hysteretic behaviour that reveals Ho atoms display magnetic remanence – just like a permanent magnet. Additional measurements carried out at the SLS showed that the hysteresis persists up to 30 K and correlates with the thickness of the MgO film. The magnetic relaxation time was found to be of the order of 1500s or possibly larger, being limited by the X-ray photon flux. Such magnetic stability is unheard of for such tiny structures. For comparison, the magnetic relaxation time in the most stable single molecule magnets is of the order of 1 ms at the same temperature.

The key to magnetic stability in single atoms lies in the choice of the rare earth element and its supporting environment. Ho provides an electronic ground state that is immune to quantum tunnelling and first order spin reversal processes, which tend to kill magnetisation, while MgO is a stiff insulator. It therefore provides isolation from both electrons and thermal vibrations, which are also responsible for inducing spin-flips and destroying magnetic remanence in small structures.

This work is the culmination of an effort that begun 15 years ago at the ESRF, where the upgrade of beamlines ID12 and ID08 allowed researchers to probe the magnetic ground state of monodispersed single atom arrays on surfaces. The planar geometry and ease of fabrication of the Ho/MgO system offers the possibility of manipulating atomic spins in a controlled way. By optimising the substrate and experimental techniques we can look forward to a new generation of nanomagnets for use in spin logic and few-atom spintronic devices.

Pietro Gambardella and Harald Brune, ETH Zurich and EPF Lausanne, Switzerland.

Feature

Mother's milk blocks infection

ESRF users have shown that components of human breast milk act as natural decoys to block norovirus from binding to antigens and causing infection.

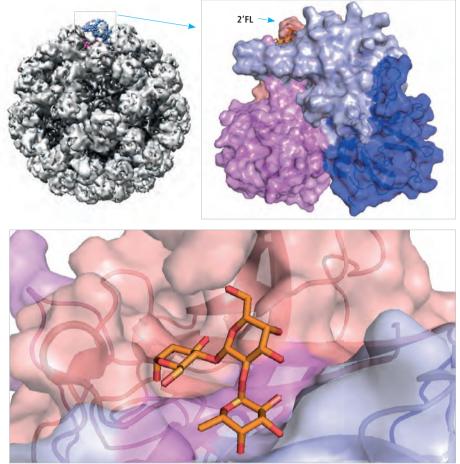
Breast milk has long been associated with health benefits in babies, but a new ESRF study suggests that the structural secrets of human milk could also prevent the spread of norovirus in the wider population. This highly contagious stomach bug is responsible for one in five cases of gastroenteritis worldwide, according to the Center for Disease Control and Prevention in the US. Occurring in many genetically distinct forms, noroviruses are transmitted via fecal matter and many infections result from the consumption of contaminated foods. But there are still no suitable antivirals or vaccines commercially available to prevent infection.

A team of researchers from Germany has used the ESRF's macromolecular crystallography facilities, including the highly automated upgrade beamline ID30B, to show that components of breast milk may block norovirus from attaching to cellular ligands that are necessary for an infection. This is the first structural study showing the binding interaction at the atomic level, raising the prospect of new antiviral drugs to treat norovirus (Journal of Virology 90 4843).

Decoy tactics

Human noroviruses bind to so-called histoblood group antigens (HBGAs), which are found in saliva as well as the tissue that lines blood vessels and internal organs. At least nine different HBGA types are known to interact with human noroviruses. It is also known, however, that the molecular building blocks of the third most abundant component of human breast milk – human milk oligosaccharides (HMOs) - are structurally similar to those of HBGAs, and that HMOs can bind with HBGAs.

Since HMOs resist degradation in the gut and are mostly excreted intact, it is thought that they act as decoys for norovirus and other pathogens. Yet the precise binding sites through which the blocking takes place have remained a mystery. Using beamlines ID23-1, ID30A-1 and ID30B at the ESRF, researchers from Heidelberg University, the German Cancer Research Center and biotech firm Jenneweln Biotechnologie analysed the



Top: a cryo-EM image of the norovirus particle (grey) overlaid with the complex X-ray crystal structure of the P dimer (coloured according to its different subdomains) and the human milk oligosaccharide 2'FL. Bottom: a close-up of the 2'FL (orange sticks) bound to the P dimer.

ability of human norovirus to bind to two oligosaccharides from human breast milk: 2'-fucosyllactose (2'FL) and 3-fucosyllactose (3FL). X-ray diffraction data revealed the norovirus "spike" protein in complex with the two HMOs, showing that both 2'FL and 3FL are capable of blocking the binding of certain norovirus types to HBGA samples.

"The ESRF's structural biology beamlines were crucial in obtaining this result," explains Grant Hansman of the German Cancer

throughput sample changer robot called FlexHCD that speeds up experiments. "A highly intense, stable beam coupled with the possibility to screen many samples is crucial for many experiments in Structural Biology" says ID30B Beamline Responsible Christoph Müller-Dieckmann. Developed in collaboration with the European Molecular

Research Center, who says that the study raises the prospect of using 2'FL and 3FL as norovirus antivirals. Currently 2'FL is already used as a safe food supplement for infant formula, but the potential for norovirus antiviral therapy has not yet been explored. "Clinical trials with 2'FL and 3FL are expected in the very near future," says Hansman. "It probably needs two to three years before it can be tested on human volunteers." Montserrat Capellas and Matthew Chalmers

Biology Laboratory (EMBL), the FlexHCD robot accepts samples in either EMBL/ ESRF pucks or Unipucks and can even manipulate crystallisation trays for in situ screening and data collection. The new BLISS beamline control software (an ESRF in-house development, designed to optimise beamline motor control) is also implemented on ID30B.

User first at ID30B

This study represents the first publication based on a user experiment at the ESRF's new state-of-the-art macromolecular crystallography beamline ID30B. This variable-focus, tuneable-energy beamline features a new generation of high

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ESRF thrives under pressure

Pioneered in the mid-1990s, X-ray studies of matter under extreme conditions are driving breakthroughs in fields ranging from the Earth sciences to fundamental physics, chemistry and materials science.

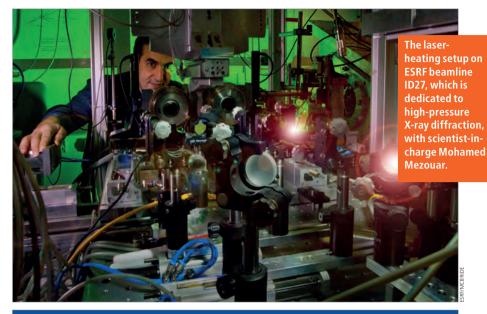
There is currently great interest in studying the properties of matter at extreme pressures and temperatures. Firstly, the conditions that we call "ambient" are not at all ordinary: most condensed matter is found inside planets at millions and billions of atmospheres and thousands of Kelvin. The ability to reach these conditions in the lab therefore improves our understanding of our own planet and others in the universe (see p19), some of which may host life. Secondly, extreme-matter research is important for synthesising materials with improved properties (p22).

There are two major challenges in the extreme-matter arena. The first is how to bring matter to such high pressures and temperatures: this can be done statically by placing the sample between the tips of two diamonds and pushing them together (p16), or dynamically by impacting the sample with a high-power laser shock (p21) or high-energy projectile (p25). The second major challenge is to be able to probe a sample's properties in situ, which is where synchrotron X-rays come into play. Since the dimensions of a sample compressed to megabar pressures and heated to thousands of degrees in a diamond anvil cell are of the order of a few microns, high-energy X-rays are needed to penetrate the cell and a highly brilliant beam is necessary to probe the microscopic samples.

Pioneering beamlines

The first structural studies by single crystal X-ray diffraction (XRD) at and above megabar pressures were performed at ESRF beamline ID09A in the mid-1990s. The success of ID09 for experiments in the megabar regime led to the construction of a second beamline (ID30) in 1996 that was fully dedicated to highpressure research. Operational until 2005, ID30 was optimised for accurate structure determinations using polychromatic powder and single-crystal XRD and saw pioneering work in laser heating coupled with angle dispersive XRD.

Following demand from users, ID30 was progressively converted to a monochromatic beamline, but full exploitation of



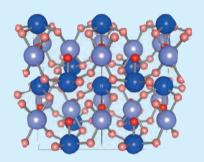
Xenon joins forces with oxygen at depth

Under extreme pressures, even traditionally inert noble gases such as xenon can become reactive. Recently, a team used micro-focused X-ray diffraction and X-ray absorption at ESRF beamlines ID27 and BM23, together with *ab initio* models, to synthesise two new xenon oxides – Xe_2O_5 (pictured) and Xe_3O_2 – under high pressure. The results demonstrate that xenon reacts with oxygen at conditions relevant to the Earth's interior and therefore could help resolve geology's so-called "missing xenon" paradox (DOI: 10.1038/nchem.2528).

monochromatic high-resolution XRD became possible only with the completion of ID27 in 2006. Thanks to the increase in flux, reduction in beam size and the development of double-sided laser heating, ID27 has become a reference beamline worldwide for micro-diffraction at extreme pressures and temperatures.

Meanwhile, in order to compete with large-volume research programmes in the US and Japan, a dedicated 2000-tonne large-volume press was installed on ID06 to provide new opportunities for ESRF users (see p22). In parallel, strong high-pressure programmes got under way on many other beamlines involving a much wider range of X-ray techniques, including inelastic X-ray scattering on beamline ID16 and pioneering studies of magnetism using X-ray magnetic circular dichroism at ID24.

The ESRF also pioneered single-bunch acquisition in order to probe laser-shocked matter in the warm dense regime, and more recently, extended X-ray absorption fine structure at megabar pressures on BM23 (see panel above).



Towards ESRF–EBS

The availability of these and many other techniques has enabled ESRF users to make many scientific breakthroughs across several fields, with recent studies addressing biological function under extreme conditions. The new ESRF storage ring, ESRF–EBS, will provide significantly higher photon flux density and higher coherence, with important implications for extreme-matter studies.

Among the specific EBS projects proposed by the Matter at Extremes group, one is a new high-pressure XRD and imaging beamline that allows time-resolved experiments down to 100 ps timescales and high-resolution nano- and micro-XRD. Another is a proposal for a dynamic compression sector, based on a high-power laser to produce matter at pressure and temperature conditions beyond the static limit of the diamond anvil cell. If approved, these facilities would have a profound impact on extreme-conditions science and guarantee the ESRF's leadership in this field. Sakura Pascarelli, head of the Matter at Extremes group.

Static compression ente



Press turns up the volume

Along with other major synchrotrons in Japan and the US, the ESRF now offers users the ability to study macroscopic samples under extreme pressures and temperatures. The large volume press at beamline ID06, which has been operational since 2012, is a 38-tonne device that is able to exert a force equivalent to 2000 tonnes on samples measuring 2–250 mm³. The press is designed to be able to handle a wide range of samples, and its non-compact nature makes it compatible with many techniques and environments – including heating to beyond 2500 K under controlled atmospheres.

"About 25% of our clients are chemists investigating synthesis and the eventual recovery of new materials, for which the setup allows you to collect diffraction data continuously at a rate of 10 Hz and therefore rapidly measure phase transitions, reactions and other phenomena," says ID06 scientist Wilson Crichton.

Not only can the instrument compress a sample to obtain thermodynamic and structural information, but it can also change its shape. "This allows users to work out the mechanics of processes such as subduction inside the Earth," says Crichton. "It is the only device currently operating in Europe for such experiments."

The pressures obtained depend on the size of the samples, with values of 20 GPa routinely achieved. More adventurous users are welcome to extend these limits, says Crichton. At these conditions, the press operates with two-stage compression and offers a range of anvils. "The higher flux and smaller beams of ESRF–EBS will open the door to faster kinetic studies, and allow users to couple different techniques." *Matthew Chalmers*

High-pressure investigations of condensed matter gained a solid scientific footing in the first half of the 20th century. In work that would win him the 1946 Nobel Prize for Physics, Percy Bridgman introduced an apparatus and experimental technique that allowed researchers to achieve pressures of the order of 10 GPa in a reliable manner. The device comprised an anvil made of hard metals with small flat areas that were pressed one against the other. Demonstrations of the drastic effect that pressure has on phase relations, electrical and thermal conductivity, compressibility and rheological properties gave birth to modern high-pressure physics.

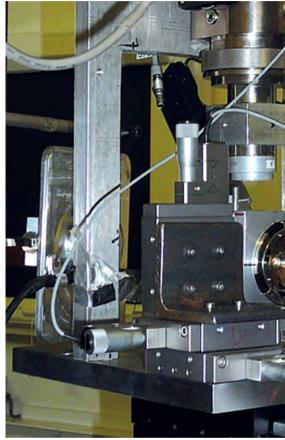
In static compression experiments, the sample is confined in a specially designed vessel. The maximum pressure is limited by the design of the high-pressure apparatus and the strength of the materials used. In order to make in situ observations at variable pressure and temperature, however, the sample must somehow be probed through the vessel walls. To this end, in 1959, researchers introduced a Bridgman-type apparatus that used single-crystal diamonds as anvils. By the early 1970s, the diamond anvil cell (DAC) technique had demonstrated broad opportunities for high-pressure researchers dealing with Mössbauer, infrared and Raman spectroscopy, in addition to resistivity measurements, X-ray diffraction and inelastic scattering.

Two major types of cells were introduced: opposite plate and piston cylinder. The principles of opposite plate DACs were developed by Leo Merrill and William Bassett in 1974 to keep the overall size small and to allow optical access to the sample chamber. However, this design did not provide the possibility of generating extremely high pressures. Two years later, Dave Mao and Peter Bell reported the long piston-cylinder cell, which allowed pressures beyond 100 GPa to be reached thanks to better alignment of the diamonds. This design and its variations have been used widely for experiments up to a few megabars. Many varieties of DACs are derived from the basic piston-cylinder Mao-Bell and opposite-plate Merrill-Bassett designs.

Synchrotron frontier

Applications of synchrotron radiation in highpressure research in the late 1980s opened a vast new frontier. Synchrotron radiation provides a powerful high-energy beam for penetrating the hard and dense anvil or gasket

Double-stage diamond anvil cells and advances in laser here matter at pressures and temperatures his



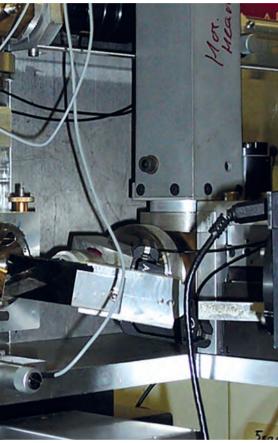
A DAC mounted at ESRF beamline ID09 for a single-crystal Xheating system that rotates in synchrony.

materials that surround a sample chamber. The low-emittance and high-brilliance X-ray beams are also ideal for focusing them to micron or less dimensions to probe miniature samples at ultrahigh pressures.

While low-temperature measurements at high pressure with DACs depend on the ability of a cell to fit in an appropriate cryostat, heating samples at multi-megabar pressures is a major challenge. The laserheated DAC proposed by Bassett and Li-chung Ming in 1974 is one of the most fundamental tools in high-pressure mineral physics research, since it is the only method that allows access to the entire range of pressures (up to 350 GPa) and temperatures (6000 K) encountered in terrestrial planetary interiors. Since 2009, however, the development of the portable laser heating technique by Reini Boehler's group at the University of Mainz and our group at the University of Bayreuth, also involving

rs the terapascal regime

eating techniques are enabling synchrotron users to study wher than those in the centre of the Earth.



ray diffraction experiment with a double-sided laser

"Synchrotrons have opened a vast new frontier."

ESRF beamlines ID18, ID24 and ID09, has significantly advanced the application of this method at synchrotron facilities. In particular, single-crystal, high-pressure, high-temperature crystallography beyond 100 GPa and thousands of degrees Kelvin is now starting to become possible (see right).

Systematic studies of matter at megabar pressures and above have revealed new types of structures of elemental materials and led to exciting discoveries of pressure-induced insulator-to-metal and superconducting transitions. Samples as small as 10 μ m are now routinely characterised at pressures above 300 GPa, but until very recently the design of DACs and the strength of the cell materials have limited achievable pressures to approximately 400 GPa.

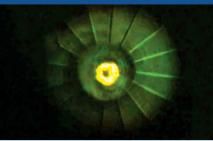
The double-stage DAC

A breakthrough came in 2012 with the development of the double-stage DAC and the use of high-strength nanocrystalline diamond (NCD) to make secondary stage anvils (*Nature Communications* **3** 1163). Unlike a conventional DAC, in which the force is exerted immediately on the sample, a double-stage DAC (ds-DAC) first transfers the force to the secondary anvils and then to the sample. This is the principle of disproportionation: the force is applied stepwise to a smaller and smaller area in order to increase pressure on a sample placed between the tips of the secondary anvils.

We have demonstrated that pressures in the range over a half a terapascal can be achieved in the ds-DAC, offering a chance to look at phenomena that could not be observed previously. For example, studies of osmium in a ds-DAC at pressures 770 GPa at the ESRF, PETRAIII in Germany and the APS in the US, recently allowed our group to see structural changes that have been interpreted as a manifestation of the interaction of core electrons in this most incompressible elemental material (Nature 525 226). Several groups worldwide are now working on the development and application of ds-DACs. Last year, for example, Takehiko Yagi and colleagues at the University of Tokyo, Japan, achieved pressures beyond 300 GPa and claimed 430 GPa. These and other works confirm the extreme efficiency of the ds-DAC design and the importance of materials used as secondary anvils.

It is clear that the ds-DAC technique is only in the beginning of its development. It requires well characterised pressure standards, advances in loading of gases and liquids, the application of heating and other improvements. Experiments in the terapascal pressure range can now be realised, but only on very small samples measuring a few microns across. The ability to probe samples with sub-micron X-ray beams, which we hope will become available at the new ESRF–EBS facility, will take us deep into this unexplored territory.

Natalia Dubrovinskaia and Leonid Dubrovinsky, University of Bayreuth, Germany.



A single crystal compressed to 140 GPa and double-sided laser heated at 2250 K, corresponding to the core-mantle boundary.

Extreme single-crystal X-ray diffraction

Performing single-crystal X-ray diffraction (SC XRD) in a diamond anvil cell (DAC) is difficult because the samples are very small. The body of a conventional DAC also prevents free rotation of a sample necessary for structural studies, while the DAC and the pressure-transmitting medium complicate the diffraction pattern. Finally, stresses in the DAC tend to destroy the single crystal.

The past decade has seen considerable progress, however. The use of inert gases as a pressure medium preserves single crystals, for example, while specially designed anvils and DACs allow a larger part of the reciprocal space to be covered. Combined with the efforts of ESRF beamline scientists Michael Hanfland and Marco Merlini, this has pushed the limit of high-pressure SC XRD to the megabar pressure range. However, experiments on single crystals at variable pressure and temperature remain extremely challenging.

The major problem is that SC XRD requires the sample to be rotated while measuring the intensities and positions of the diffracted X-rays using a 2D detector. If the sample is laser-heated, the lasers must be focused at the same spot from which the diffraction is being collected and the whole heating system must therefore rotate synchronously with the DAC. Another problem arises from inhomogeneity in the temperature distribution within the laser-heated spot.

Our team at the University of Bayreuth recently developed a portable double-sided laser-heating system that can be rotated with the DAC. The system has recently been employed at ESRF beamlines ID09A and ID27 and also at PETRAIII in Hamburg, revealing, in particular, totally unexpected compounds in iron oxides at lower-mantel conditions (DOI: 10.1038/ncomms10661). *Maxim Bykov and Elena Bykova, University* of Bayreuth, Germany.

Advertising feature

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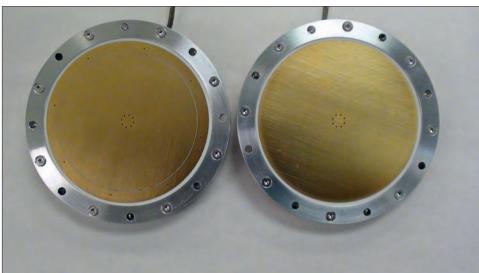
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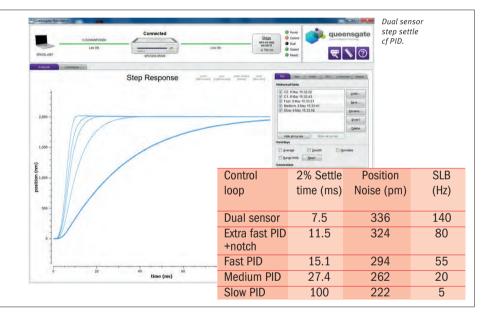
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Going underground

ESRF users are closing in on the thermal state of our planet's mysterious interior.

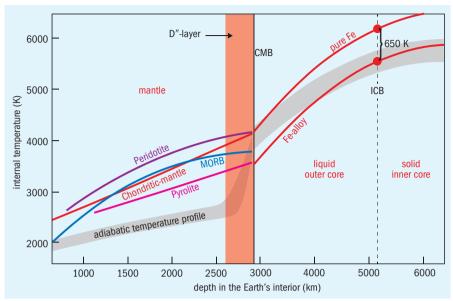
To geophysicists, the Earth is a massive heat engine that generates the geomagnetic field and is responsible for global plate tectonics and volcanism. In order to understand such crucial processes, we need to determine the amount of heat permanently available to activate them. This requires knowledge of the Earth's temperature as a function of depth, called the geotherm. Although careful analysis of seismic data helps to constrain some properties of the deep Earth's interior, seismic waves are too weakly affected by temperature to provide direct constrains on the geotherm. Instead, we have to compare seismology with measurements of minerals subjected to extreme conditions of pressure and temperature. In the past 15 years, synchrotron X-rays have driven major advances in our understanding of this domain.

In the Earth's solid mantle, heat is transported slowly via convection and the geotherm slope is dictated by the equations of state (EoS) of mantle minerals, which describe how the density of a material evolves with pressure and temperature. For instance, the density of minerals increases by around 50% from the Earth's surface to the core-mantle boundary (CMB) due to a huge pressure difference of 135 GPa. Measuring the EoS requires a combination of high-flux, hard X-ray diffraction and laser-heated diamond anvil cells. Since the hot and compressed zone measures only a few micrometres across, the X-ray beam must be of similar dimensions to provide useful data. Only a few synchrotrons worldwide can provide such a beam, and ESRF is one of them.

Temperature jump

Measurements of the EoS refined during the past 15 years suggest that the temperature rises from around 1400 °C just below the Earth's surface to 2500 °C at a mantle depth of 2500 km. But this rather smooth geotherm is followed by a steep increase of temperature

when approaching the hot core 200–300 km above the CMB, where seismic revealed ultra-low seismic velocities that could be due to mantle partial melting. Understanding this temperature jump is a major



The Earth's temperature profile (grey) inferred from X-ray diffraction at the ESRF. Just above the core-mantle boundary (CMB) seismic measurements indicate partial melting (the D"-layer), suggesting that the temperature should resemble the melting curve of relevant geological materials. Going deeper, the melting of pure iron was recently reported at 6230 K at the inner-outer core boundary (ICB). The melting curve of iron alloys constrains the temperature profile in the core.

goal because it is directly linked to the amount of heat permanently escaping the core, which drives our planet's internal dynamics.

In order to constrain the temperature in this mantle region, researchers measure the melting curve of the major geological materials present at these extreme conditions. Several user groups at the ESRF and other synchrotrons in Japan and the US are undertaking such studies, suggesting a CMB temperature of around 3800°C (*Earth Planet. Sci. Lett.* **304** 251; *Science* **344** 892; *Science* **329** 1516; *Science* **343** 522). These results suggest that the temperature increases by around 1300°C in a thin layer at the base of the mantle.

Deeper into the Earth's interior, seismic data reveal another crucial boundary between the liquid outer core and a solid inner core (the ICB), where the pressure is around 330 GPa. The ICB temperature corresponds to the crossing between the geotherm and the melting curve of the iron-based alloy that constitutes the core. However, the melting temperature of pure iron (which is the main core constituent) at such pressures has been highly controversial. Recent measurements at the ESRF finally reached agreement with data obtained by laboratory shock-wave compression and ab initio calculations. This experiment, which involved laser heating in a diamond anvil cell coupled with rapid X-ray diffraction, enabled researchers to detect the melting of iron before it reacts with surrounding materials and suggests that pure iron melts at 6000 °C at 330 GPa (Science 340 464).

However, the core contains other constituents such as silicon, sulphur and oxygen, which decrease its melting temperature by approximately 650 °C, as measured by other ESRF experiments (*C. R. Geosci.* **346** 130). This provides a fixed point to the geotherm at a temperature of 5350 °C and a pressure of 330 GPa, which can be extrapolated to infer a CMB temperature of 3750 °C – in good agreement with the estimate on the mantle side. Yet this value still remains uncertain due to uncertainties in the composition of the core, and also the difficulty in making precise measurements of the temperature of the laser-heating spot.

Thermal history

Although synchrotrons have improved our knowledge of the Earth's inner temperature, the thermal history of our planet remains largely unknown. This is important for understanding how the Earth became structured into distinct layers and how it sustains the geodynamo. Improving our knowledge of these processes will require extensive modelling of mantle dynamics over geological periods, demanding a better understanding of transport properties under extreme conditions. Presently, the role of pressure on thermal conductivity, atomic diffusivity and viscosity, which control the thermal evolution of the Earth, is barely constrained. This raises new challenges for synchrotron facilities.

Denis Andrault, Université Blaise Pascal; Agnès Dewaele, CEA Arpajon; Guillaume Morard, Université Pierre et Marie Curie, France.

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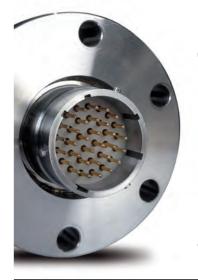
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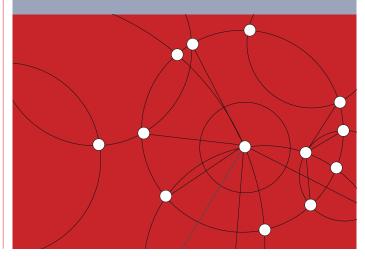
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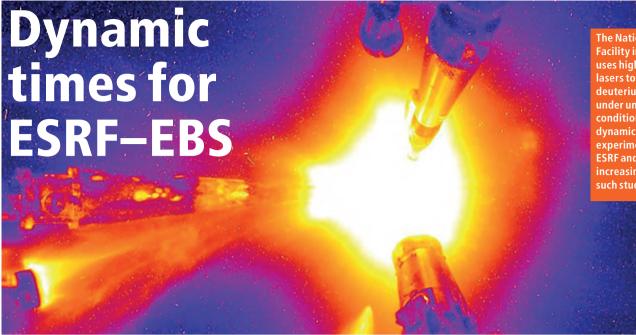
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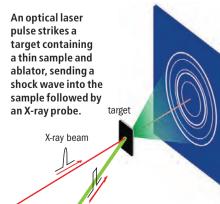
The National Ignition Facility in the US uses high-power lasers to compress deuterium-tritium under unprecedented conditions, but dynamic compression experiments at the ESRF and XFELs are increasingly allowing such studies.

In conjunction with the European-XFEL, ESRF–EBS will provide European researchers with two world-leading facilities for the study of dynamically compressed matter.

The dynamic compression of matter, whereby pressure is rapidly applied to substances by impact, explosion or illumination, plays a central role in many natural and technological systems. Impact cratering by asteroids is a wellknown example, but it also occurs in medical therapies such as the removal of kidney stones, inertial confinement fusion energy and even the strike of a mantis shrimp's claw.

Dynamic compression is also a primary technique to study the properties of matter under extreme pressure, density and temperature. In dynamic compression, changes in pressure travel through matter in the form of a shock wave. Although similar to a sound wave, shock waves have a very high pressure such that the highest-pressure part of the wave overruns any lower pressure part of the wave in front of it, producing a sudden jump in pressure. The conditions obtained in shocked samples can be much more extreme that those obtainable in a static compression experiment, but they last only for a period of a few nanoseconds - about the time it takes for a sound wave to travel across a tiny sample.

Modern shock-compression science began



60 years ago, when researchers found that solid iron undergoes a structural phase transition from the familiar body-centredcubic structure to a hexagonal-close-packed structure when shock pressures exceeded 10 GPa. Studying such phenomena at the lattice level, however, has long been limited by the lack of X-ray sources with sufficient brightness.

Synchrotron studies over the last 25 years show that the structural behaviour observed in even simple metals at high pressures is extremely complex, and this complexity is believed to continue to multi-TPa pressures. Dynamic compression alone can obtain the extreme pressures and temperatures of interest, but if we are to truly probe the behaviour of materials at extreme conditions we also need improved structural diagnostics.

Complementing XFELs

X-ray free electron lasers (XFELs) have recently offered dynamic compression science an almost ideal X-ray source: monochromatic, micro-focussed, high-energy X-ray pulses as short as one ten-thousandth of a nanosecond. With a peak brightness one billion times higher than a synchrotron, XFELs are perfectly suited for pump–probe experiments whereby an optical laser compresses a sample followed by interrogation by the XFEL beam. This allows extremely high-quality diffraction and scattering measurements that are unaffected by any smearing from atomic motion.

While some studies of shock-compressed matter have recently been performed at synchrotrons, the signal obtained is generally poorer than is obtainable at an XFEL. That situation is about to change, however, thanks to the lattice upgrades planned on thirdgeneration synchrotrons such as the ESRF. In particular, an anticipated 100-fold increase in flux on beamline ID27 following ESRF–EBS will make single X-ray pulse experiments much more feasible.

The first ultra-fast synchrotron spectroscopy experiments on laser-shocked iron were recently performed at ESRF beamline ID24 by a team from France and the UK (*Scientific Reports* **6** 26402). This summer will see five experiments performed at ID24 to investigate the behaviour of a wide range of materials compressed with a high-energy optical laser provided by the CEA. A particular aim is to determine whether the quality of the spectroscopic data obtainable from a synchrotron, for which the X-ray bandwidth can be chosen, is better than that from an inherently monochromatic XFEL.

The start-up of the European-XFEL in Germany in 2018, in conjunction with ESRF-EBS in 2020, will provide European researchers with two world-leading facilities for the study of both statically and dynamically compressed matter. With fortuitous timing, high-power, highrepetition-rate diode-pumped solid-state lasers are also becoming available that will deliver 100 J, 10 ns pulses at a rate of 10 Hz. When combined with the ability to shape the laser pulse, such lasers will enable exotic compression paths that are perfectly suited for dynamic compression studies at both the European-XFEL and the ESRF. Malcolm McMahon and Stewart McWilliams. School of Physics & Astronomy and Centre for Science at Extreme Conditions, The University of Edinburgh, UK.

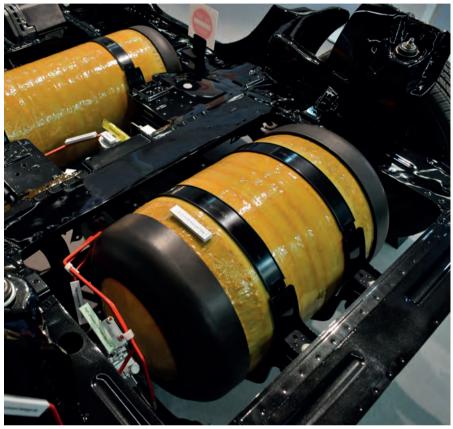
Materials synthesis under pressure

Extreme pressures and temperatures transform the chemistry and properties of materials, opening new routes to superconducting, storage and energy technologies.

The chemistry of materials is governed by the behaviour of their outermost electrons, which determine the bonding, reactivity and structures of molecules and solids. High pressure drastically reduces the molar volume and average atomic distances of any chemical species, modifying its outer electron shells and inducing profound modifications to its properties. Gaseous oxygen, for instance, transforms from a dense liquid into an electrical insulator at 5 GPa and becomes a superconducting metal at 96 GPa, while conductive materials such as lithium and sodium undergo even more surprising transformations into insulators.

A well known example is the high-pressure synthesis of diamond and cubic boron nitride, which are the super-hard abrasives of choice for cutting and shaping hard metals and ceramics. Under ambient conditions these materials possess a "soft" hexagonal structure with graphite-like planes interacting weakly, but they undergo a phase transition to rigid cubic structures with strong "sp3" chemical bonds.

Understanding high-pressure chemistry is vital to guide researchers to new materials and compounds, but without synchrotron X-rays it would be very difficult to probe samples under such drastic conditions. As diamond anvil cells can only handle a tiny quantity of material, a very intense and highly focused high-energy X-ray beam is therefore crucial for *in situ* chemical studies at high pressure.



A major target of extreme-conditions research is to identify materials with a very large storage capacity, which have applications in hydrogen fuel cells for transport applications.

INDUSTRY CASE STUDY Extreme pressure aids pharmaceutical formulations

To become a drug, a pharmacologically active compound must be prepared in a specific form that can be manufactured, packaged, stored, transported, administered and delivered to a target in the body. Our team from the Russian Academy of Sciences has performed a series of high-pressure experiments at the ESRF over the past 15 years to explore new pharmaceutical formulations with improved bioavailability and to understand the processes that take place during tableting (Current Pharmaceutical Design, in press). We first focused on paracetamol, for which we had obtained the orthorhombic form by direct hydrostatic compression of the monoclinic polymorph (J. Therm. Analys. Calorim. 68 437). Later, in collaboration with researchers at US pharmaceutical firm Pfizer, we started



to explore the effect of pressure on an antidiabetic drug called chlorpropamide. Hydrostatic pressure experiments using diamond-anvil cells performed at the SNBL beamline allowed us to compare the results of compression between dry samples and the same samples immersed in a fluid, finding that the pressure-induced polymorphic transition is solvent-assisted and therefore can be avoided by using dry samples (*Intern. J. Pharm.* **327** 51). We have since revisited this system at SNBL in collaboration with Vladimir Dmitriev and

also at ESRF beamline ID15B with Michael Hanfland and Damian Paliwoda. Based solely on a thermodynamic phase diagram, it was not possible to predict which phase will be formed at a given combination of temperature and pressure. Instead, there exists a complex interplay of nucleation and growth kinetics that leads to the formation of different phases depending on the choice of the starting polymorph, the hydrostatic medium or the compression method (submitted to CrystEngComm). Thanks to experiments such as these, pharmaceutical companies can achieve a much better control of polymorphism in products, obtain new forms with improved bioavailability, and avoid undesirable transformations during processing. The development and control of drug forms is also crucial for intellectual property.

Elena Boldyreva, Novosibirsk Institute of Solid State Chemistry and Mechanochemistry, Russian Academy of Sciences.

"The challenge is to find new metastable polymorphs that exist at ambient conditions."

Searching for metastability

In the past decade, many breakthroughs in high-pressure chemistry have been achieved at large synchrotron facilities. Examples that derive from the ESRF's outstanding X-ray beam characteristics include the *in situ* synthesis and structural characterisation of a 3D form of fullerene (*Science* **283** 1720) and a cubic polymorph of the ternary compound BC₂N with outstanding mechanical and thermal properties (*Appl. Phys. Lett.* **78** 1385). Just like the transformation of graphite into diamond, these exceptionally hard materials are recoverable at ambient conditions.

Another example of the power of highpressure chemistry, which was obtained by reacting carbon dioxide with silicon dioxide under high pressure, is a new silicon carbonate phase that opens the potential to synthesise a novel class of materials with applications in CO_2 segregation and storage (*PNAS* **108** 7689). The challenge for these particular materials is to find metastable polymorphs with equivalent physical properties that could be exploited under ambient conditions.

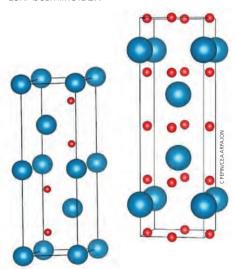
A similarly high-impact application of extreme materials is superconductivity. Following the recent discovery that hydrogen sulphide becomes a superconductor at a temperature of 203 K under high pressure (see panel, right), researchers are now searching for a new class of hydrides with outstanding physical properties. Iron hydrides are of particular interest because ab initio calculations predict that they possess both a high-temperature superconducting transition temperature and an exceptionally high hydrogenstorage capacity. In 2014, a team of users from France in conjunction with ESRF staff compressed and heated a mixture of hydrogen and iron to synthesise two novel hydrides, FeH₂ and FeH₃ (see right, Phys. Rev. Lett. 113 265504).

The ESRF–EBS source will afford higher photon flux and spatial resolution from submicrometres down to tens of nanometres, allowing studies of dynamical phenomena. This will have a profound impact on extremeconditions chemistry. The design, synthesis and *in situ* characterisation of new materials that can perform reliably under thermomechanical extremes and are central to addressing future challenges in fossil fuel, fission, fusion and other technologies. For instance, new materials synthesised under extreme conditions are good candidates for confining a hydrogen plasma at temperatures of several millions of degrees in future fusion reactors such as ITER, which is a fantastic challenge.

High-flux future

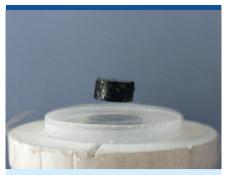
The high flux of the new machine will also allow studies into the synthesis of harder and more resistant BCN materials, thanks to the ability to study micron-size samples at several megabars and thousands of degrees in laser-heated diamond-anvil cells. It will also open new possibilities to tailor novel materials with unique chemical and physical properties. A recent example is the synthesis of bulk cubic Mg_{0.4}Fe_{0.6}N, for which ESRF users brought together unreactive starting materials and used pressure to remove the barrier to reactivity (Angew. Chem. Int. Ed. 54 15109). A particular incentive for such nitride synthesis is the potential application of MgN and FeN systems in spintronics, in which the spin of electrons is exploited for information processing and storage.

Finally, ESRF–EBS could take the "holy grail" of high-pressure science within reach: the formation and structural characterisation of metallic hydrogen, which 80 years ago was predicted to be a superconductor at ambient temperature. Confining and bringing highly reactive micron-size samples of hydrogen to the necessary pressures remains a huge challenge, but ESRF–EBS will allow small hydrogen samples to be characterised in unprecedented detail and therefore shed new light on the physics of element one. *Mohamed Mezouar, scientist in charge at ESRF beamline ID27.*



Intense X-ray microbeams at ESRF beamline ID27 led to the discovery of new iron hydrides under extreme conditions. The structure of FeH₂ (right), with alternate planes of iron and hydrogen atoms, is remarkable because the hydrogen molecules could become metallic at much lower pressure than pure hydrogen.

Focus on: matter at extremes



The Meissner effect, discovered in 1933, causes a magnet placed above a superconductor to levitate due to the expulsion of magnetic field lines during the superconducting transition.

Superconductivity confirmed in hydrogen sulphide

Last year, physicists in Germany discovered that hydrogen sulphide (H_2S), which is normally associated with the smell of eggs, becomes a superconductor at a recordbreaking temperature of 203 K when put under pressures of 150 GPa. The team, led by Mikhail Eremets at the Max Planck Institute for Chemistry in Mainz, confirmed the superconducting state by measuring the electrical resistance and magnetic susceptibility of tiny H_2S samples in a diamond anvil cell (*Nature* **525** 73).

Earlier this year, members of the same team in conjunction with ESRF staff used nuclear resonant scattering at ESRF beamline ID18 to establish this claim beyond doubt, marking an important step towards room-temperature superconductivity. Since the expulsion of an external magnetic field is a defining characteristic of a superconductor (via the Meissner effect), the researchers placed a thin tin-119 sensor inside an H₂S sample under pressure and used synchrotron Mössbauer spectroscopy to monitor the magnetic states of nuclei under an external field of about 0.7 T.

When H₂S is in the normal superconducting state, or if the superconducting state has been partially destroyed, the magnetic field penetrates into the sample volume and splits the nuclear levels in tin to produce "quantum beats" in the time spectra. When H₂S is in the superconducting state, however, the magnetic field does not penetrate the tin and the corresponding time spectra show an exponential decay (Science 351 1303). The small size of samples required for highpressure experiments makes it difficult to study the superconducting properties of H₂S at such high pressures, but the resonant character of nuclear scattering ensures only data acquired from the tin sensor with zero background from the sample environment. The proposed method should also allow studies of superconductors under pressures up to 300 GPa. Matthew Chalmers

Two positions – Time resolved X-ray science; ultrafast diffraction and spectroscopy

The ELI (Extreme Light Infrastructure) Project is an integral part of the European plan to build the next generation of large research facilities. ELI-Beamlines is a cutting edge laser facility currently being constructed in Dolni Brezany, next to Prague in the Czech Republic. At ELI Beamlines, Research Program 4 (RP4) develops applications in Molecular, Bio-medical and Materials (MBM) Sciences. Specific research areas under development are time resolved X-ray diffraction and spectroscopy, atomic and molecular science, Coherent Diffractive Imaging, time resolved VUV magneto-optical ellipsometry and advanced optical spectroscopy methods (femtosecond Stimulated Raman Scattering and 2D spectroscopy).

The junior researchers/postdocs will work predominantly on the following topics:

- participate in the development of the ELI beamlines scientific end stations for time resolved X-ray diffraction and spectroscopy
- take an active role in the X-ray diffraction and/or spectroscopy research at RP4, including the commissioning of the scientific end stations
- participate in relevant experimental and theoretical work in the labs of national and international collaborators as well as at suitable research facilities

Requirements:

- PhD in Physics, material science, chemistry, structural biology or related field. or equivalent capabilities
- a few years of experience from X-ray diffraction and/or X-ray spectroscopy research, ideally using both continuous and pulsed X-ray sources
- experience from designing, building and implementing X-ray instrumentation, in particular X-ray optics, pulsed laser research and using liquid jet sample delivery systems is an advantage

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Shocking materials

Gas-gun experiments demonstrate the ESRF's ability to take multi-frame, high-resolution phasecontrast images of extreme physical behaviour.

The high-velocity impact between two materials is a fundamental event that occurs in numerous natural and man-made processes, such as high-speed machining or upon failure in high-performance vehicles and aircraft. The forces that result produce a range of unique structural changes that evolve rapidly, often faster than the speed of sound. Traditionally, researchers study these changes using highspeed cameras and interferometry, but the limited penetration-depth of such techniques leaves much of the material's underlying microstructure obscured.

ESRF beamline ID19 is ideally suited for such studies. Over the past year, our group at the Institute of Shock Physics at Imperial College London, UK, has been using a portable single-stage helium-driven gas gun to accelerate a 13 mm-diameter projectile to velocities approaching 800 ms⁻¹. This generates pressures up to 160,000 times atmospheric pressure in shock waves travelling at several kilometres per second, which rapidly sweep through the mm-scale samples producing conditions that persist for only fractions of a second.

In order to make meaningful measurements in this limited time, free from the effects of shock-front blurring or image "ghosting", the X-ray probe must be similarly quick. This is achieved using a single, approximately 100 ps-duration X-ray bunch, which can be readily isolated during the sparsely populated 4- or 16-bunch modes of the ESRF. After scattering through the sample, the single bunch is detected by a fast, high light-output scintillator optically relayed to a pair of intensified CCD cameras that are synchronised to both the gas gun and the synchrotron radio-



The Imperial College gas gun installed on ESRF beamline ID19.

frequency system. This approach allows us to capture sequential single-bunch phase contrast images of shock waves and ensuing deformation features in profile. The resulting images (see below) are sub-ns snapshots of the very first moments of shock compression and wave evolution in condensed matter systems, and have enabled us to study a variety of extreme physical phenomena – such as dynamic tensile failure and cavity collapse – at the ESRF for the first time.

Stellar origins

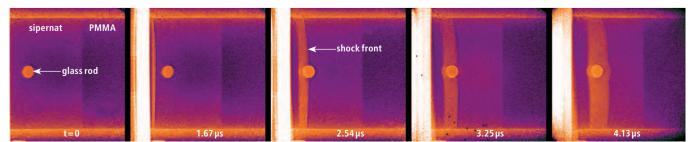
One theme of research that has emerged from such experiments is the study of astrophysical chondrite-like powders under high-velocity impact. Chondrites were some of the earliest objects to form in our solar system, and understanding the manner in which they were compacted under impact can help researchers unravel the conditions in the protoplanetary disk. Using the portable gas gun at ESRF beamline ID19, powder mixtures comprising a bimodal mixture of solid density SiO₂ chondrules and highly porous nanoscale SiO₂ matrix have been impacted at velocities exceeding 600 ms⁻¹, generating average pressures in the mixtures of between 1–2 GPa.

By staggering the images in multiples of the bunch spacing we were able to construct time sequences showing several stages of a supersonic compression wave as it traversed the powder bed. These images provide a first

"ID19 is ideally suited for such studies."

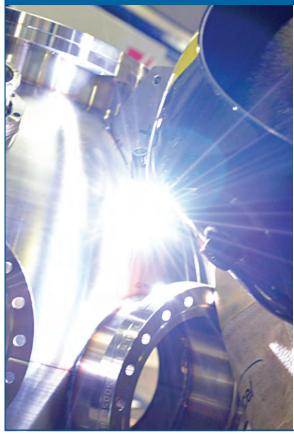
glimpse of the highly irregular compaction in this strongly heterogeneous system, helping researchers characterise the local subsurface variations in shock velocity and density. Until now, this has only been revealed through simulations.

With the majority of models for porous systems derived from continuum measurements, the ability to make direct observations at the scale of the individual chondrules will have a major impact in validating asteroid and planetary-impact models. One such example can be seen below, showing a time series of X-ray images of a shock wave striking an embedded glass rod. The rod produces a well-defined disturbance in the compression front consisting of shock, re-shock and release events. Such a geometry is directly amenable to 2D simulation, and thereby provides new capabilities for validating material models through matching them to microscopic in situ behaviour. Daniel Eakins, Institute of Shock Physics, Imperial College London, UK.



Time series of a model powder configuration showing the interaction of a shock wave in silica powder with an embedded 1 mm diameter glass rod.

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Elements of success

Paul Loubeyre describes the importance of extreme-conditions research for materials science and how the ESRF is helping the decades-long search for metallic hydrogen.

It all began with a newspaper article in Le Monde in 1979. Paul Loubevre was in his second year of a physics degree in Paris when he read the news that helium had been transformed from a gas into a solid under high pressure at ambient temperature using a diamond anvil cell (DAC). Given the importance of low-temperature helium for condensed matter physics, Loubeyre suspected that studies of helium at megabar pressures would also reveal interesting physics. So he got in touch with the author of the study and set about determining the phase diagram of helium at high pressure, which became his thesis topic.

The ability to load gases into DACs was a "game changer", says Loubeyre. "It was when the extreme-matter field was starting out, and it also coincided with the emergence of thirdgeneration synchrotrons." Following his degree, Loubeyre started to work on quantum effects in helium under pressure and was led guickly to the textbook element: hydrogen. In 1992, the then director of the ESRF. Yves Petroff. invited Loubeyre to carry out singlecrystal diffraction on hydrogen to infer its equation of state at megabar pressures. Loubeyre became a regular user of ESRF beamlines, winning the ESRF Young Scientist Award in 1996.

Mastering hydrogen

Hydrogen has been a focus of Loubeyre's research ever since. "Pressure breaks down the molecular nature of hydrogen and so it becomes more quantum than helium," he explains. "It's an exciting system with many applications such as planetary models, inertial confinement fusion as well as being a model system of fundamental interest."

Mastering the properties of dense hydrogen could pave the way to novel schemes for energy production, storage



Paul Loubeyre in brief

Born: 1958, Clermont Ferrand, France.

Education: Ecole Normale Supérieure: MSc physics (1982) and PhD (1987, University of Paris VI) **Career:** Research scientist, CNRS, University Paris VI (1982–1998); CEA (1998–present).

Family: Married, three children.

Interests: Family, music, reading.

"You could probably characterise any extreme state using the ESRF."

and transport. A promising example, says Loubeyre, is a class of superhydrides materials with unusual hydrogen stoichiometry and very high temperature superconductivity. The challenge is to recover these at ambient pressures, he says.

Currently, Loubeyre is working towards the production of metallic hydrogen, which is predicted to form at a pressure of around 450 GPa. "There are other groups worldwide doing this, but we are all stuck below 400 GPa with current DACs," he says. Recently, Loubeyre's team achieved pressures of 600 GPa on gold at ESRF using a novel toroidal design that is now being applied to hydrogen. "Hydrogen's phase diagram is hugely complex, with calculations predicting intriguing structures," he explains. "Although spectroscopic measurements can define the boundary lines of the phase diagram, X-ray determination of the structures remains a challenging project that will be within reach following the ESRF-EBS upgrade." A new multichannel collimator at beamline

ID27 has recently enabled Loubeyre's group to measure the structure factor of fluid hydrogen under pressures of a few GPa.

Stuck at pressure

Although electrical and magnetic measurements can be carried out in the laboratory, compressed hydrogen samples above 400 GPa are so small that probably only synchrotrons will be able to fully characterise their transport properties, says Loubeyre, who uses infrared absorption as the signature of the transition to metallic hydrogen. Synchrotron nuclear resonance scattering could then be used to obtain a magnetic characterisation of its superconducting nature, as recently demonstrated at the ESRF for the compound H₂S. "With a bit of imagination you could probably characterise any extreme state using the ESRF," says Loubeyre. "That is why synchrotrons are so essential to the high-pressure community worldwide.'

There have already been claims that metallic hydrogen has been produced in multi-shock compression at laser facilities, and Loubeyre says that the transition from a molecular fluid to a plasma is now clear. But the jury is still out for the transition to the metallic state. "The hydrogen problem illustrates the fact that the dynamic and static communities are converging in their investigations," he adds.

Loubeyre and collaborators have recently pioneered highpower laser shock methods at the ESRF. By coupling these with X-ray absorption spectroscopy measurements at ID24, the team obtained first results on the local and electronic structure of warm dense iron. "The ESRF-EBS should help to gain a full microscopic characterisation of dynamically compressed matter up to TPa pressures," he says. "However, static compression will still be the path to reveal the subtle physics of metallic hydrogen". Matthew Chalmers

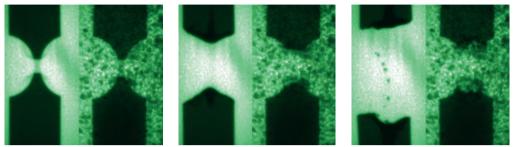
Industry

Imaging reveals electrical breakdown

French energy firm Mersen has teamed up with the ESRF and researchers from the University of Grenoble to study the breakdown of electrical fuses in situ for the first time.

Fuses can be found in most electrical circuits, where they protect electrical components against current surges. Despite their widespread use, however, nobody has ever watched what happens to a fuse as it explodes. Now, thanks to sophisticated single-bunch imaging techniques at ESRF beamline ID19, a team of researchers has tracked the ultrafast processes that take place in fuses during electrical breakdown, helping French energy firm Mersen develop advanced compact fuses for renewable energy applications. "Most fuse development to date has been based on postmortem analysis and theory, never on direct observation, so this is a huge step in terms of understanding electrical arcing in situ," says Xavier Just of the SIMaP laboratory at the University of Grenoble.

Generating power from wind turbines and other intermittent sources relies on DC converters and fuses, but these tend to be bulky and complex compared to familiar AC versions. "In DC circuits, the fuse has to manage current-interruption without profiting from the voltage passing through zero," explains



Breakdown of an electrical fuse at time intervals of 25, 100 and 500 µs (left to right) following a current spike, showing the effects of sand (right half of each panel) in the arc-quenching channel.

Mersen's Jean-Louis Gelet. "Also, there is a big interest in reducing the dimension of the fuse because long fuses increase the inductance of the circuit, which is detrimental to DC inverters."

Beyond laboratory sources

Electrical arcs and the interaction between arc and silica sand as a guenching material are at the heart of Mersen's knowledge, says Gelet. With fuse breakdown typically taking place on timescales much less than a millisecond inside an opaque medium. in situ observations were thought impossible. But last year, Just and co-workers got in touch with ID19 beamline scientist Alexander Rack and realised that synchrotron X-ray

3D microtomography could take them far beyond the capabilities of laboratory X-ray sources.

Earlier this year, the SIMaP team spent three days at the ESRF recording data from commercial and test fuses to understand the interaction between electrical arcs and matter. Beamline ID19 allowed the team to measure the speed and lifetime of "burn-back" an important process whereby the electrical arc damages the electrode material and creates silica cavities in the arc channel (see image). Although previous post-mortem studies had shown various phenomena, they revealed little about the temporal characteristics of the process.

"We didn't know before whether the silica cavity is formed in one go or if it forms in steps with the burn-back," says Just. "The results change this picture a lot."

In addition to characterising the dynamics of the system, the team determined the impact of electrical and materials parameters (such as the influence of grain sizes, porosity and utility of silicates) and is now planning further measurements at ID19 to investigate the transition from pre-arcing to arcing at even faster rates. "Both the quality of the ESRF beams and ultrafast recording are necessary for Mersen's investigations," says Gelet. Matthew Chalmers

Movers and shakers



John Womersley, chief executive of the UK's Science and Technology

Facilities Council (STFC), which manages the UK's involvement in the ESRF, has been selected as the next director general of the European Spallation Source (ESS) under construction in Lund, Sweden. Womersley has led the STFC, since 2011 and is currently chair of the European Strategy Forum on Research Infrastructures. He will step down from the STFC and take over from current ESS director-general Jim Yeck on 1 November.



Wim Bras, project leader at the Dutch-Belgian CRG beamline "DUBBLE", has

position as director of the soft condensed matter and biology group in the neutron science division of Oak Ridge National Lab in the US. Bras, who started his career in the UK's Daresbury Lab and has worked at the ESRF for the past 20 years, has made numerous contributions to X-ray scattering and the development of combined techniques, and is the co-author of more than 200 publications. He expects to start in his new role in August.



ESRF user Ari-Pekka Honkanen from the University of Helsinki,

Finland, has won the Finnish Physical Society's inaugural Young Scientist Award. The €1000 prize was based on Honkanen's master's thesis, titled X-ray diffraction properties of spherically bent crystal analysers. His research took place during a summer internship at ESRF at the inelastic X-ray scattering beamline ID16 (which has since moved to ID20 as part of the ESRF Upgrade Programme), and so far the work has led to the publication of two peer-reviewed articles.



In March, Ori Lavi and Avner Okun, winners of the ESRF EIROforum prize at the 2015 European Union

Contest for Young Scientists, made a four-day visit to the ESRF and ILL. The students, both aged 18 and from Israel, were recognised for their work on the structure of the large ribosomal subunit of Deinococcus radiodurans in complex with antibiotics. In April, the ESRF also hosted a team of six high-school students from the Lycée Viette in Montbéliard near Grenoble following their success in the French national physics Olympiads competition.





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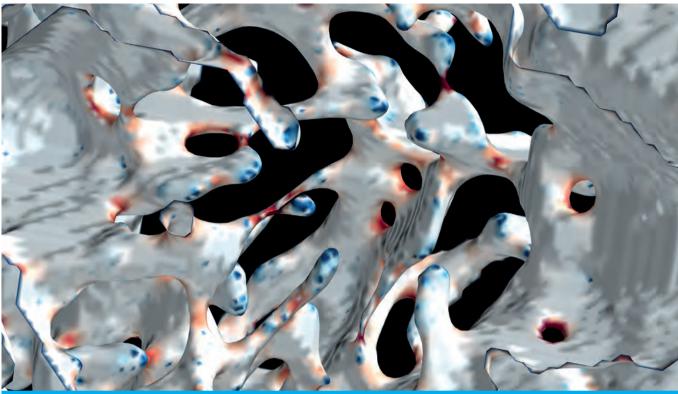
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Beauty of science



Topological imaging: This image, which was obtained at ESRF beamline ID19, shows a surface rendering of the copper phase in an ESRF using 3D X-ray microtomography, which is an ideal tool for quantitative studies of the complex topology of multiphase materials. electrical conductivity. The grey-level volume was processed by Emmanuelle Gouillart of the SVI laboratory to extract the two phases using denoising and segmentation algorithms from scikit-image.

In the corridors



Neutrons rule out parallel universe

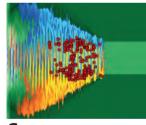
A team of physicists from France and Belgium has used intense neutron beams at the Institut Laue-Langevin in Grenoble to test if our universe is merely a 3D "brane" floating around in a higher dimensional bulk - a popular hypothesis for theories attempting to go beyond the standard model of particle physics. If true, this scenario could cause particles, especially uncharged neutrons, trapped within one brane to occasionally tunnel quantum mechanically into an adjacent brane and disappear from our world. The team used a helium-3 counter to detect neutrons swapping back from the hidden dimension, and despite drawing a blank, was able to improve the limit from previous experiments by four orders of magnitude (Phys. Lett. B 758 14).

Animal in the machine



hours of 29 April, CERN's Large Hadron Collider (LHC) was temporarily knocked out

of action by what the Geneva lab described as a "severe electrical perturbation". The culprit? An unsuspecting beech marten that had stumbled into an over-ground 66 kV transformer and caused a short circuit. The incident, which cost the small creature its life, occurred just as LHC operators were preparing for the machine's 2016 run and generated media reports across the world. Not to be outdone. however, the ESRF tweeted an image from the experimental hall floor showing its own animal attraction



Gamma-ray source in sight

Numerical simulations have allowed physicists at the University of Texas in Austin, US, to demonstrate a potential source of collimated gamma rays that could be used to drive experiments or medical therapies. The models show that a strong PW-class laser pulse striking a structured plastic target generates a beam of multi-MeV photons with high-conversion efficiency, due to unprecedented quasistatic magnetic field of 0.4 MT (yellow, orange and blue above) driven in an over-dense plasma. Electrons liberated by the pulse are accelerated and emit synchrotron radiation that could constitute a

gamma-ray source with an output in the TW range, says the team (Phys. Rev. Lett. 116 185003).

X-rays used in evidence



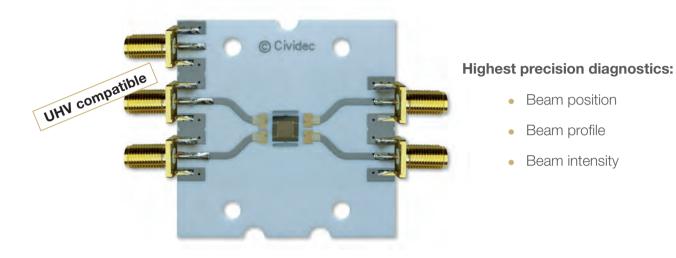
Synchrotron X-rays have helped police in Australia establish the scene of a

murder. Scientists at the Australian Synchrotron used X-ray powder diffraction to trace microscopic brick fragments found on the victim's clothes to their origin. Robert Fitzpatrick from the Commonwealth Scientific and Industrial Research Organisation and colleagues were able to identify the origin of the bricks by analysing the minerals inside the fragments, allowing them to locate the individual brick that was the source of the particles. The analysis was carried out four years ago but it is only now that the team is able to reveal the underlying details.



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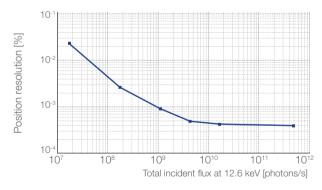


Figure: Measurements at Diamond Light Source Ltd., UK, show the measured position resolution at 1 kHz bandwidth for various beam intensities of the 12.6 keV photons. A position resolution of better than 0.1% of beamsize is obtained even for an incident flux as low as 10⁹ photons/s.

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- Wide dynamic range.

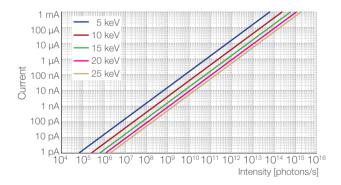


Figure: The detector response as a function of the photon energy and intensity demonstrates the wide dynamic range of the XBPM System in combination with the C8 Electrometer Amplifiers.

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