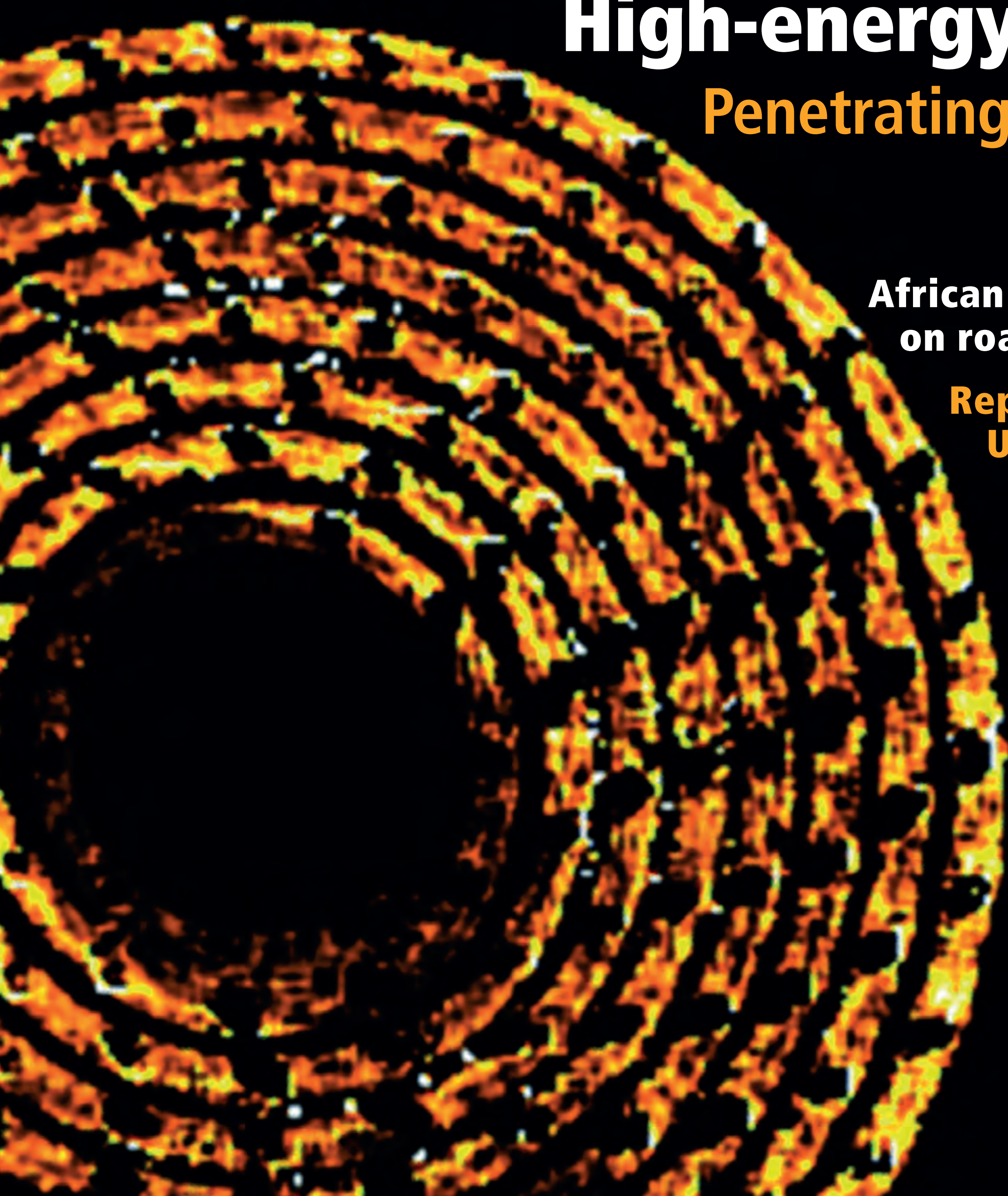
THE EUROPEAN SYNCHROTRON

ESRF news

Number 72 March 2016

High-energy X-rays
Penetrating materials



African Light Source on road to success

Reports from the User Meeting

ESRF

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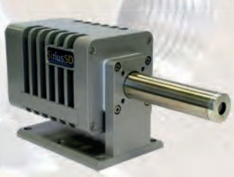
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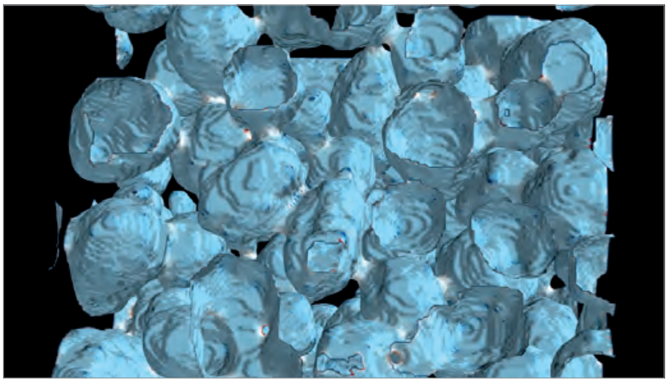
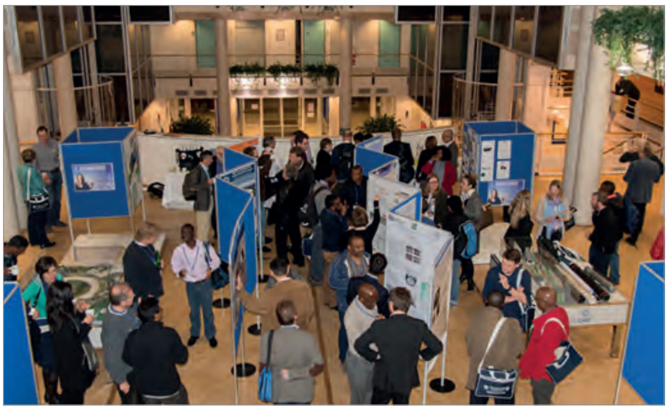


The 2016 User Meeting saw keynote speakers focus on materials, p12.



C ARGOUD

ESRF hosts the first African Light Source conference and workshop, p16.

ISTOCKPHOTO.COM

High-energy X-rays target industrial methane conversion, p22.

ESRFnews Number 72 March 2016

On the cover: *In situ* high-energy X-ray studies of a commercial battery during operation (p24)

ESRFnews

Going from strength to strength

2015 marked an important year in the evolution and role of the ESRF.

First, excellent operation of the accelerator complex and outstanding beamline performances contributed to record values both in operational statistics and in the number of submitted proposals, with a steep increase of submissions from ESRF users targeting opportunities offered by the new beamlines.

Second, 2015 saw the end of the ESRF Upgrade Programme Phase I, which has been completed on time, within budget and with minimal impact on the operation of the facility and the ongoing user programme. This €180 m project, which began in 2009, has involved the construction and/or refurbishment of 19 beamlines, many specialised in nanoscience, in addition to the construction of a new 8000 m^2 ultra-stable experimental hall and the upgrade and renewal of facilities and support laboratories.

The third major achievement of 2015 was the tremendous progress made towards the new ESRF–Extremely Brilliant Source (EBS), launched in May. The aim of this revolutionary €150 m project is to construct and commission the new ESRF–EBS storage ring over the period 2015–2020. About 90% of the existing infrastructure will be reused, and a greatly improved energy efficiency will reduce electricity costs by 20%. During the period 2016–2022, new public and CRG beamlines will be constructed and/or adapted to the new source. Similarly, extended instrumentation and IT programmes will be implemented. The new Science Advisory Committee, along with ESRF scientists and users, has already defined plans for implementing the public and CRG beamline portfolios, setting the scene for the next few years of experimental activities.

"The launch of ESRF–EBS and the delivery of ESRF UP Phase I are critical steps for the ESRF's future"

The launch of ESRF–EBS and the delivery of ESRF UP Phase I are critical steps for the ESRF's future. With performances multiplied by 100 in terms of brilliance and coherence, ESRF–EBS will offer unprecedented tools for the exploration of matter, for the understanding of life at the macromolecular level and for the excellence of European and world science.

High-energy focus

One of the ESRF's defining features, and the focus of this issue of *ESRF News*, is its very high-energy X-ray beams. The ESRF storage ring circulates electrons at an energy of 6 GeV, which is higher than most European synchrotron sources and comparable to that of the other three high-energy synchrotrons in the world. Each step up in electron energy leads to a steep increase in the brightness of the high-energy part of the spectrum, and the pioneering use of in-vacuum and cryogenic undulators makes the ESRF beamlines among the best in the world for high-energy applications.

The ESRF has eight dedicated high-energy beamlines (p19) that allow users to penetrate deep into samples ranging from metals and alloys (p25) to industrially relevant catalysts (p22) and advanced nuclear fuels (p24). High-energy X-rays are also increasingly relevant to the pharmaceutical industry (p21), and for investigations into hydrogen storage materials, thick layers and other systems too numerous to describe here. ESRF–EBS will enhance these capabilities even further, especially concerning valuable *in situ* and *operando* studies, allowing users to address ever more closely the scientific and industrial challenges facing society.

Francesco Sette, *ESRF director-general*

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The main accelerator tunnel of the European XFEL.

ESRF shares expertise with European XFEL

The European XFEL nearing completion in Hamburg, Germany, has joined forces with the ESRF to design a new end station at the facility devoted to materials imaging and dynamics (MID). The contract to the main supplier for MID was placed in January and the instrument is expected to be ready for user operation by the end of 2017.

It is a technologically demanding project that involves positioning an instrument that weighs several tonnes to within a few dozen microns and dealing with very large vacuum forces. The ESRF supplied engineering assistance for the study and design of the unique MID instrument, as well as knowledge transfer and training. The project was initiated in 2012 and has now reached the construction stage.

"The completion of the project was only possible thanks to the long-standing expertise and experience of the ESRF, the pooling of professional know-how as well as good communication with the European XFEL," says project leader Muriel Magnin-Mattenet. "Nine people from the ESRF worked on the project throughout the design phase as part of the technical review committee."

Anders Madsen, scientist in charge at MID, says the choice to collaborate with ESRF engineers represents a two-to-threefold return on investment. "The ESRF delivered such a complete and detailed set of drawings valid for manufacturing that the manufacturer will be able to start cutting metal almost on day one, saving us thousands of euros in engineering and production costs."

INFN signs agreement with ESRF

An important collaboration agreement between the ESRF and the INFN (Istituto Nazionale di Fisica Nucleare) was signed on 8 February in the presence of the Italian and French ministers of research. The agreement will result in an exchange of expertise and the strengthening of technical assistance between ESRF and INFN focused on the construction and installation of the ESRF–EBS storage ring.

The agreement reinforces links between ESRF and INFN that date back to the very beginning of the ESRF and to the design of the existing storage ring. INFN's pioneering work on aluminium vacuum chambers for its DAFNE facility has helped in all aspects of the design and construction of the ESRF–EBS vacuum system. As Francesco Sette said: "This agreement will be of great importance at a time in which the ESRF and its European partners work together to invent and construct the new ESRF–EBS storage ring."

ESRF/P JAYET

The ESRF welcomed the French and Italian ministers of research to witness the signing of the new agreement.

Italian Minister for Education, Universities and Research, Stefania Giannini, said that the collaboration agreement emphasises the primary role played by Italy in important European research infrastructures. "Italy, together with France and Germany, is among the main contributors to the ESRF, a model of international cooperation, which welcomes every year thousands of scientists."

"This partnership between the ESRF and the INFN is a historical one, because it is at the heart of immense scientific progress," said French Minister of State for Higher Education and Research Thierry Mandon.

President of the INFN, Fernando Ferroni, gives a full account of the collaboration between the ESRF and INFN on p27 of this issue.

Bank backs ESRF–EBS

The European Investment Bank (EIB) has decided to support the ESRF–EBS project (the revolutionary new storage ring to be implemented at the ESRF) to the level of €65 m. ESRF–EBS represents a total investment of €150 m over the period 2015–2022 and will result in a new X-ray source within the existing infrastructure.

The funding has been guaranteed by InnovFin Large

Projects, a financial product developed under the new Framework Programme for Research and Innovation in the European Union, Horizon 2020. A finance contract was signed at the ESRF on 11 December in the presence of EIB and European Commission (EC) officials. "This first signature with InnovFin Large Projects in France renews our commitment not only for basic research but also for public health, which is a major issue of Horizon 2020," said EC commissioner for research, innovation and science, Carlos Moedas.

EIB vice-president Ambroise Fayolle said that supporting innovation is one of the priorities of the European Union's bank. "The EIB is proud to be helping the European synchrotron with its upgrade and international development project."

Landmark for ESFRI roadmap

The ESRF has gained "landmark" distinction in the 2016 ESFRI Roadmap on Large Scale Research Infrastructures, which was launched on 10 March at an event in Amsterdam.

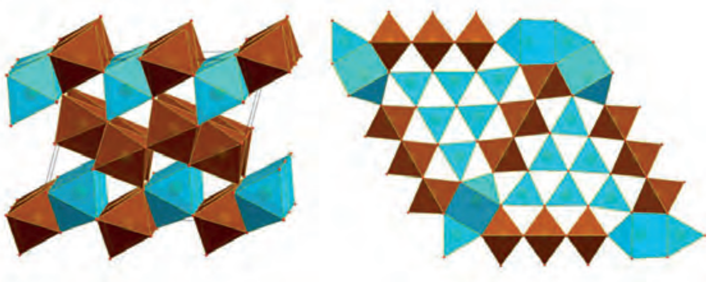
Since 2006, the European Strategy Forum on Research Infrastructures (ESFRI) has presented several roadmaps to support European policymaking.

The 2016 Roadmap, which has been under development since 2014, consists of 21 ESFRI projects with a high degree of maturity – including six new ones – and 29 ESFRI "Landmarks". These are research infrastructures listed in previous ESFRI roadmaps that have become pillars of scientific excellence and competitiveness in the European Research Area. Landmark status was granted to the ESRF–EBS, along with ILL's upgrade programme and CERN's Large Hadron Collider, among others. "This marks the ESRF upgrade as a facility of pan-European importance and a key part of the European Research infrastructure landscape," says ESFRI chair John Womersley, who is chief executive of the UK's Science and Technology Facilities Council. "It also re-emphasises the need for ongoing support from the member states to ensure that the ESRF can continue to deliver the highest quality science."

Oxygen recycled in Earth's mantle

Studies of iron oxides under extreme conditions point to the existence of a novel oxygen recycling mechanism in the Earth's interior, according to a study by an international team at the ESRF.

Using diamond anvil cells and laser heating, the team from the University of Bayreuth in Germany, the ESRF, PETRA III and the University of Chicago applied pressures of over 100 GPa and temperatures above 2500 K to samples of iron oxide (Fe_2O_3) to see how it behaves in the mantle. Fe_2O_3 is one of the main building blocks of mantle phases and is important for industrial iron production. Despite its simple chemical composition, it exhibits unusual structural and electronic transitions at elevated pressures and temperatures that have so far eluded explanation.



Experiments reveal chemical complexity in iron oxides at high pressures, which transform to produce Fe_5O_7 (left) and $Fe_{25}O_{32}$ (right).

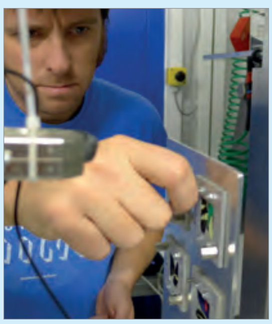
The team used single-crystal X-ray diffraction at ESRF beamline ID09A to show that Fe_2O_3 is chemically unstable at high pressures and temperatures (DOI: 10.1038/NCOMMS10661). When heated at pressures above 60 GPa it is transformed into the known post-perovskite structure, which then releases oxygen and forms a novel compound Fe_5O_7. The team then performed experiments on Fe_3O_4 and found that this iron oxide also decomposes to form previously unknown $Fe_{25}O_{32}$. Overall, the study suggests the presence of an oxygen-rich fluid in the deep Earth's interior that can significantly affect geochemical processes.

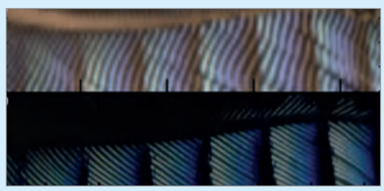
The results therefore have important implications not only for fundamental high-pressure chemistry, but also for the understanding of Earth's evolution, says the team. "This possible mechanism of oxygen recycling in the Earth's interior is a game changer," says author Leonid Dubrovinsky of the University of Bayreuth. "Any global models of the past and future of the Earth, including models of evolution of climate, should take into account the new findings."

Birds master nanotechnology

Scientists from the University of Sheffield in the UK have discovered that birds alter the nanostructure of their feathers to create the vivid colours of their plumage. Using X-ray scattering at ESRF beamline ID02, the researchers studied the feathers of the jay, which are made of a nanostructured keratin material that can change colour from ultraviolet through to blue to white.

The results showed that the Jay demonstrates amazing control over the size of the holes in this sponge-like structure: larger holes mean a broader wavelength reflectance of light, which creates white, while smaller holes result in blue (*Scientific Reports* **5** 18317).





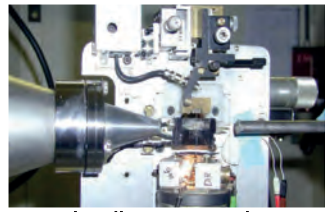
Andrew Parnell aligns samples on the ESRF's ID02 beamline. The colour of the jay feather reconstructed from a SAXS scan (top) and the same region observed using optical microscopy (bottom).

In contrast to colours formed using pigments, colours created from structure do not fade over time. "This discovery means that in the future, we could create long-lasting coloured coatings and materials synthetically," explains team member Andrew Parnell. "Current technology cannot make colour with this level of control and precision – we still use dyes and pigments. Now we've learnt how nature accomplishes it, we can start to develop new materials such as clothes or paints using these nanostructuring approaches."

Prions generated by a copper switch

Prions are unique infective agents that convert proteins into misfolded isoforms in the brain, causing a group of transmissible neurodegenerative disorders affecting humans and animals. Despite the importance for pathogenesis, however, the insoluble nature of prions makes it difficult to characterise the mechanism of prion formation.

Now, using extended X-ray absorption fine structure spectroscopy at ESRF beamline BM30B (FAME), a team of scientists from Italy has revealed the structural events underlying this important conversion. Data allowed them to identify a novel copper-mediated mechanism that acts as a switch that turns the normal protein into its pathological alter ego. The findings suggest that copper bound to this region could stabilise it and prevent misfolding events (*Scientific Reports* **5** 15253).



A muscle cell was mounted vertically in a trough between a loudspeaker motor and a force transducer.

Stimulating muscles *in situ*

Experiments on ESRF beamline ID02 have revealed a mechanically sensitive structural switch that links muscle performance to its external load. Using X-ray diffraction from single skeletal muscle cells, an international team from the University of Florence in Italy, King's College London in the UK and the ESRF has shown that muscle contraction is controlled by two switches. Previously, it was believed that actin-containing filaments were the only elements governing this vital muscle regulatory process.

The very high brilliance and collimation of the ID02 beam, combined with the sensitivity and resolution of its 2D detectors, allowed the team to record X-ray signals from a single muscle cell, timed to within a millisecond and with very high spatial resolution.

The results show that force generation against high loads requires a second permissive step linked to a change in the structure of the thick filament, offering a promising approach for new therapeutic opportunities (*Nature* **528** 276).

Soft matter event comes to Grenoble

The 4th International Soft Matter Conference (ISMC2016) will be held in Grenoble on 12–16 September.

The triennial event will be held at Grenoble's Alpexpo conference centre and is being co-organised by the ESRF, ILL and other research institutes in the area. Narayanan Theyencheri, scientist in charge at ESRF beamline ID02, is conference co-chair. Early bird registration is open until 1 June (www.ismc2016.org).

Top-up mode to boost operation

ESRF users are soon to experience a major change in the way the facility's accelerators operate. Whereas presently, electrons are injected into the storage ring every 3–8 hours followed by a smooth beam decay, a new injection mode will see the storage ring being topped up with electrons every 10–30 minutes or so. Top-up operation, which has been studied since 2012, is a key deliverable of the ESRF Upgrade Programme Phase I and is also important for the operation of ESRF–EBS.

The near constant beam lifetime offered by top-up operation reduces current and heat-load variations on beamline optics, reducing drift and the need for compensation systems. Top-up operation also produces a smaller vertical emittance and smaller insertion-device gaps, resulting in brighter beams for users.

Preparing the machine for top-up operation requires significant modifications to be made to the ESRF's accelerator complex, in particular improving the efficiency and reliability of the injector complex so that it can handle more frequent injections. "We've recently completed major upgrades to the injector, including a new buncher on the Linac, two additional RF cavities in the booster and the development of a sequencer that performs injection automatically," explains operation manager Jean-Luc Revol. "New diagnostic tools have also been implemented in the booster, as well as feed-forward correction systems that limit disturbance to the stored beam caused by the injection itself."

RF engineer Alessandro D'Elia with one of the additional cavities installed in the booster to improve the efficiency and reliability of the injector complex.

The team is now focusing on limiting disturbance to the beam due to cleaning, which takes place after injection to remove unwanted electrons from certain parts of the storage ring. One way to avoid this, which is currently undergoing tests, is to install a fast magnet called a stripline kicker that allows just a single electron bunch to be injected without parasitic electrons that could pollute adjacent bunches.

Another approach, for which the necessary hardware has been installed and commissioned, is to allow the cleaning process to take place in the booster rather than in the storage ring.

These modifications will be finalised during the first half of 2016. Once validated, user operation in top-up mode beginning with 16-bunch and four-bunch filling will commence.

Rock steady result for ESRF–EBS girder

A prototype of the girder that will form the support for the ESRF–EBS storage ring has been put through a series of tests to check its stability against motion. ESRF–EBS requires 128 of the seven-tonne girders, each of which will be mounted with precisely aligned magnets and vacuum chambers before it is installed in the storage tunnel. As a result, it is vital that the alignment is not compromised during transportation from the assembly area to a girder's precise location in the storage ring tunnel. The ESRF has set a maximum limit of 50 µm of movement during the entire transportation process.

Mechanical engineers devised three movement tests for the prototype girder and checked the alignment of the magnets before and after each test with a precision of 10 µm. The first test saw the girder being lifted half a metre by a large system of jacks and lowered back into place. For the second test, the girder was first lifted, displaced sideways and then lowered onto new feet attached to rollers, then driven a distance of a few tens of metres across the experimental hall floor by a hydraulic motor. In both tests, the teams found no significant movement of the magnets.

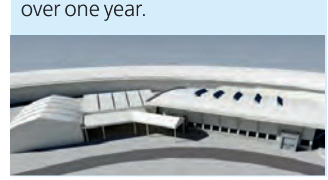
Finally, the girder was driven outside the experimental hall, lifted several metres by a crane and placed onto the back of a truck. It was then driven around the exterior of the storage ring at a speed of 15–20 km/h, with a maximum speed of 30 km/h. "Even taking into account maximum measurement errors we got a total magnet movement of 44 µm," says David Martin, who is responsible for alignment. "We can safely say that assembling the girders and transporting them with the same care observed in these tests will cause no significant deterioration in the overall alignment."

Mechanical technician Marc Lesourd measures the acceleration as the prototype girder is lifted to test for movement of the attached dummy magnets.

ESRF–EBS prepares for new buildings

The construction of three new buildings for ESRF–EBS has been approved (artist's impression below). Located inside the storage ring, the new structures will consist of one permanent building (B1) and two temporary buildings, B2A and B2B, in addition to a loading/unloading zone. With an area of 1145 m^2, B1 will be used to assemble the 128 girders and their associated hardware for the future storage ring. Once installation begins in spring 2019, the building will be left free to store equipment or serve as a workshop. Temporary pre-fabricated buildings B2A and B2B will be used for storage before and during the assembly stage while a fourth temporary building will serve as a base for construction staff.

Work to prepare the grounds is scheduled to begin in May, with construction estimated to begin in August and lasting just over one year.



News from the User Office

Following the record number (1135) of proposals received for the September 2015 deadline, 455 have been allocated beam time in the 2016/I scheduling period. The next Beam Time Allocation Panel meetings to review proposals submitted for the 15 January (long-term projects) and 1 March (standard and MX BAG proposals) deadlines are 28 and 29 April.

Proposers are reminded by the Beam Time Allocation Panels of the importance of submitting experiment reports for all beam time allocations previously used. It is also expected that proposal forms include more than one proposer. If this is not the case, the proposer should include a comment on who will carry out the work in the technique section of the experiment methods template. The need for synchrotron radiation and for the specific beamline(s) requested should be clearly explained, and any reference in the proposal to previous experiments should always be accompanied by the proposal number of that experiment.

Resubmitted proposals should be clearly marked as such, and it is mandatory to clearly indicate what aspects of the proposal have been modified or improved. Reviewers advise that proposers omitting these details or resubmitting an unchanged proposal are unlikely to be awarded beam time.

The 26th ESRF User Meeting took place on the EPN Science Campus on 8–10 February. More can be read about this in the dedicated article on pp12–13.

News from the User Organisation Committee

The User Organisation would like to thank all the users who participated in the 2016 User Meeting and contributed to a pleasant and dynamic atmosphere. Participants have recently been sent a link to an online questionnaire that will allow us to evaluate the meeting, and we kindly invite all participants to complete the survey. Your feedback is important for improving future editions of the event, and the questionnaire is also an opportunity to propose new ideas for keynote speakers and topics for the 2017 user dedicated microsymposia.

The 2016 User Meeting took place at a special time for the ESRF. Last year saw the completion of Upgrade Programme Phase I and the launch of a second and new phase, the ESRF–EBS. Last year, the ESRF opened a call for expressions of interest to identify new experimental programmes and beamlines for this ambitious project, and these are being taken into consideration.

We would like to remind all users who have questions, comments or ideas 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

News from the beamlines

- The soft interfaces and coherent scattering beamline **ID10** installed a new diffractometer at the surface scattering end station in December. The diffractometer is combined with a double crystal deflector dedicated to studies of liquid surfaces and interfaces, which allows the X-ray beam to be tilted downwards (at large angles) without movement of the surface. The deflector will speed up the data collection, decrease the liquid surface agitation and increase the *q* range for reflectivity measurements on liquids. The new instrument has been fully operational for user experiments since 24 February.

C ARGOUD

The newly installed diffractometer at beamline ID10, which will allow faster data collection.

- Work on upgrade beamline **ID15** continues, with completion of the infrastructures and assembly and installation of components underway. ID15 will reopen for user applications in the March proposal round, boasting a new detector specifically designed for high-energy diffraction experiments. The large (2M) CdTe Pilatus 3X detector will be shared between ID15 and **ID31**.

- Ptychography and ptychographic X-ray computed tomography are now available for users at the nano-imaging beamline **ID16A**, offering the ultimate spatial resolution of the beamline. Ptychographic tomography is a coherent imaging method that can reconstruct the 3D distribution of the electron density in the sample with a resolution below 30 nm. As such, it completes the framework of complementary nano-imaging techniques available at the beamline, which also comprises holographic X-ray nanotomography and X-ray fluorescence microscopy. The instrument has also been upgraded to operate at cryogenic temperatures, which is crucial for life-science applications.

- **ID19** now offers extended time-resolved X-ray imaging capabilities thanks to recently installed cameras: a Shimadzu HPV-X2 megaframe-rate camera is available for acquisition speeds up to single bunch imaging, while an image-intensified camera PI-MAX4 from Princeton Instruments is aimed at shock and impact studies. Furthermore, a deep-cooled "pco.edge gold" camera allows dose-sensitive samples to be imaged at moderate acquisition rates. All FReLoN and pco.edge cameras can now be operated with extended dynamic range by a dedicated accumulation mode.

- A new double phase plate setup was installed on **ID20** and **ID26** during the winter shutdown to allow greater control over the polarisation of the X-ray beam. The system allows the incident beam to be rotated from horizontal or linear polarisation to an arbitrary direction, or to be converted into circularly polarised X-rays. Experiments that combine variation of the incoming beam polarisation with resonant inelastic X-ray scattering, X-ray emission spectroscopy and X-ray Raman spectroscopy will help disentangle contributions to the scattering signal from different excited electronic states.

TOYAMA *Pioneering New Horizons in Science*

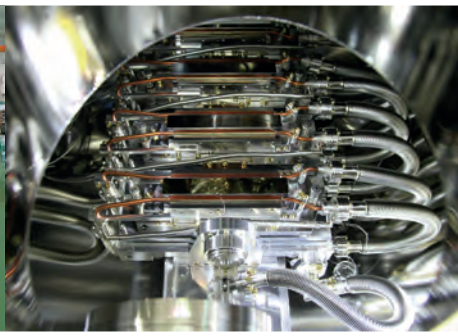
Soft X-ray Beamlines

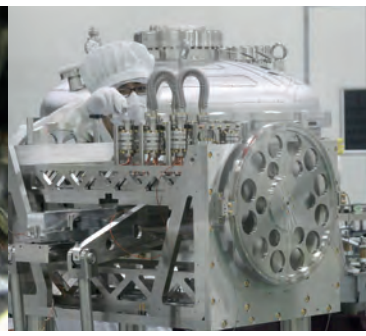

cPGM for MAX IV

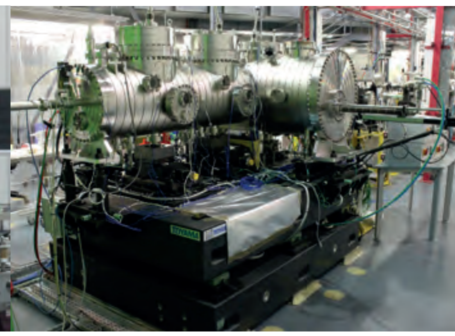
Soft X-ray Monochromators

Toyama monochromators are in use at all the Japanese synchrotrons and also at Soleil and ALBA. Three of the latest generation of cPGM monochromators have recently been delivered to MAX IV, together with new design of gas filter exit slit. Our complete range of soft x-ray monochromators features:

- VLSPGM, cPGM, SGM and NIM designs
- Adaptable configuration including up to four mirrors and gratings
- Combined grazing and normal incidence available in the same instrument
- Excellent energy resolution and energy stability
- FEA is used to optimise design for high vibrational stability
- Precision exit slits; gas filter exit slits for high harmonic rejection
- Mechanism is decoupled from the vacuum chamber





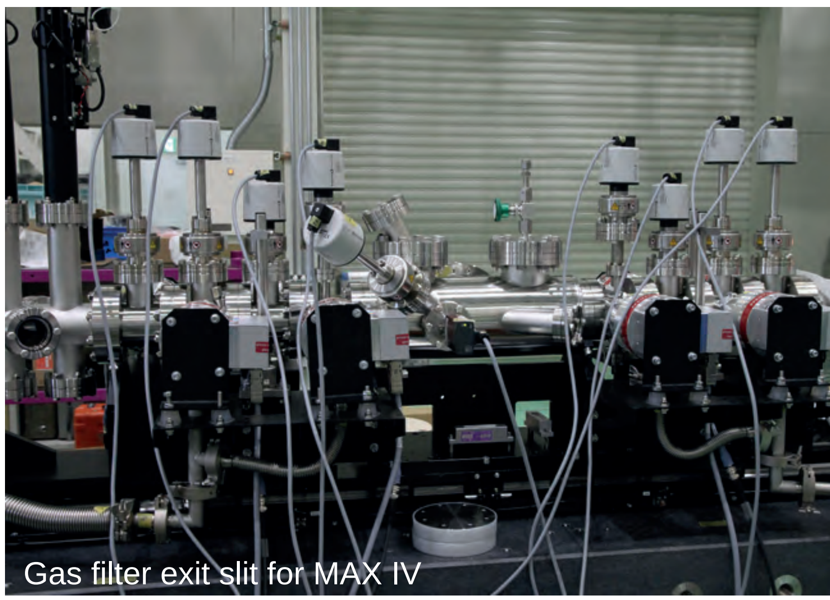


Soft X-ray Beamlines Components

In addition to monochromators, Toyama also specialises in the design of components for soft x-ray beamlines, from single components to complete beamline design. Our capabilities include:

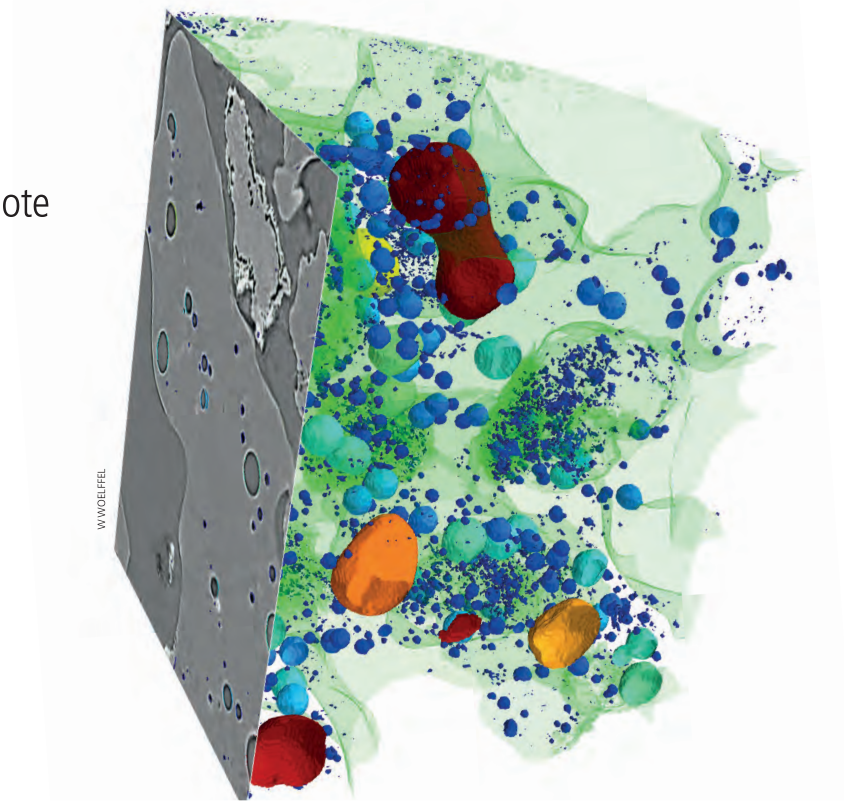
- Full beamline design including optical design, together with thermomechanical and vibrational FEA
- Mirror and KB systems including mirror benders
- Beam monitors
- Masks and collimators
- Front end components
- End stations for electron spectroscopy

We have the technology and experience to turn your ideas into reality


Gas filter exit slit for MAX IV

Users put materials in the spotlight

The 26th ESRF User Meeting held 8–10 February brought together more than 350 synchrotron scientists for keynote lectures, tutorials, dedicated symposia and poster clips.



An *in situ* furnace at beamline ID19 allowed users to make 3D movies of glass formation, in which raw materials are transformed into a silicate liquid that traps bubbles and crystalline defects.

Emmanuelle Gouillart of the CNRS and Saint Gobain, France, uses synchrotron X-rays to make movies of materials as they evolve from one phase to another. Her aim is to understand the rapid transformations that take place during production or under operating conditions, which have important consequences for materials manufacturing.

"It's amazing to be able to observe the evolution of liquids at a temperature of 1000 °C, and the ESRF is really one of the very few places in the world where this is possible," said Gouillart during her keynote lecture kicking off the plenary session of this year's User Meeting. "*In situ* 3D imaging is a unique way to witness turning points of such transformations from the inside."

During the past few years, ESRF staff and users at beamline ID19 have overcome many technical challenges to make *in situ* tomography a reality. Today, the technique offers 3D imaging with a time resolution as short as one second while maintaining a spatial resolution at the micron level. Moreover, explains Gouillart, a variety of dedicated set-ups are now available on the beamline to reproduce conditions such as heating, mechanical loading and freezing. This allows researchers to tackle diverse applications including food engineering, metallurgy and ceramic processing.

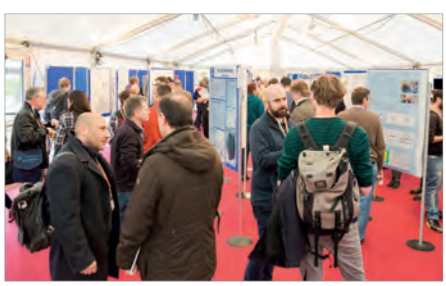
Gouillart and co-workers are focusing on glass formation in realistic environments. Watching a movie of sodium carbonate and silica heated at 800 °C, for instance, her team recently quantified the reactive regions of each grain and observed several reaction mechanisms leading to sodium silicates. "The geometry of reactions and the origin of crystalline defects are big factors in the glass industry." The data sets produced by *in situ* tomography are huge, but it's worth it to be able to watch what happens inside materials as they form, says Gouillart. "Although it's an industry that evolves over long timescales, our work has already triggered R&D projects for Saint Gobain."

Magnetic divergence

Sticking with the materials-science theme, structural chemist Andrew Goodwin from the University of Oxford in the UK described new developments in our understanding of supramolecular reactions. "We try to use flexibility and disorder to identify materials with new and interesting properties, such as more responsive materials that react to their environment," he says.

One example is zinc dicyanoaurate, which has a tetrahedral structure and exhibits extreme negative linear compressibility (see panel right). Another is the family of silver and gold cyanides, which, despite their strong chemical similarities, have different structures. Synchrotron X-ray powder diffraction at the ESRF's Swiss–Norwegian beamline allowed the team to obtain a microscopic understanding of the unusual physicochemical behaviour at play in both systems – with the help of a neat magnetic analogy.

"From a structural viewpoint we can map the structure of the gold and silver structures onto simple fictitious spin models: with gold you get a triangular ferromagnet, whereas with silver you get a triangular antiferromagnet," explains Goodwin. The team recently used this magnetic analogy to understand a system that contains both a silver and a gold atom, for which theorists predicted the corresponding ground state would be a ferro-quadrupolar phase (DOI:10.1038/NCHEM.2462). For instance spin vortices in the spin system translate into screw dislocations in the gold–silver system, says Goodwin. "Such dislocations have the



Winner of the 2015 Young Scientist Award: Andrew Cairns

The ESRF User Organisation has awarded the title of Young Scientist of the Year to chemist Andrew Cairns, 27, for his groundbreaking studies of negative linear compressibility (NLC). On behalf of the ESRF User Organisation, Andrew was presented the award by Massimo Altarelli, managing director of the Europen XFEL, during the 26th ESRF User Meeting on 8 February.

Cairns completed a masters degree in chemistry at the University of Oxford in 2011, and then embarked on a PhD using synchrotron techniques to explore how very low-energy interactions in materials can give rise to extremely unusual properties and complex disordered structures. During this time he received several accolades including being named as a finalist for the 2013 Reaxys Inspiring Chemistry Prize and winning a 2013 Margaret C Etter Student Lecturer Award from the American Crystallographic Association.

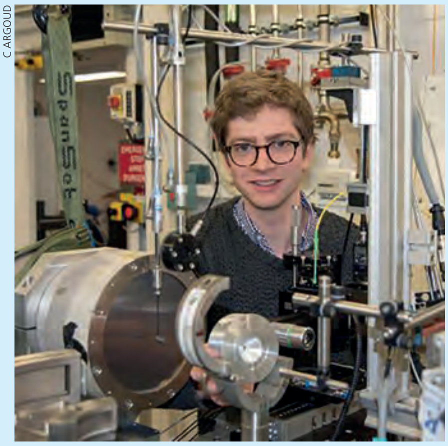
"Being named Young Scientist of the Year is a huge surprise and a great honour," he said on receiving the award.

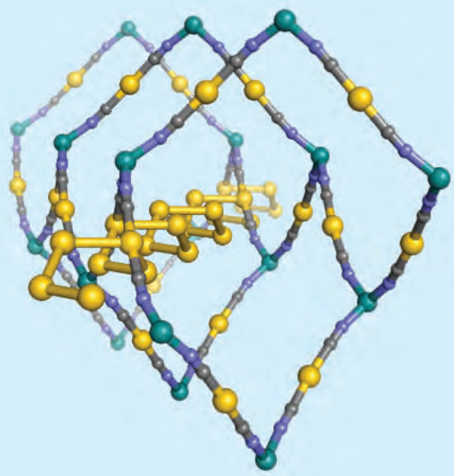
Wine-rack science

Negative compressibility is a highly counterintuitive phenomenon whereby a material expands under pressure rather than shrinks. At first, this effect appears to violate basic thermodynamics, but the laws of physics do not dictate that a material must shrink in every direction when put under pressure. Just imagine a foldable wine rack, says Cairns: its collapsed (low-volume) state is narrower but taller than its expanded (high-volume) state. "At the molecular level, a similar process takes place inside NLC materials."

Until recently, very few NLC materials were known. Using the ESRF's Swiss–Norwegian beamline, however, Cairns and co-workers demonstrated record-breaking negative linear compressibility in zinc dicyanoaurate, showing that this material expands in one direction 10 times faster than the typical contraction observed in common engineering materials under pressure (*Nature Materials* **12** 212).

C ARGOUD





Top: Andrew Cairns at beamline ID27. Bottom: Zinc dicyanoaurate, which is made up of zinc (teal), gold (yellow), carbon (grey) and nitrogen (blue) atoms, increases in size by a factor 10 when compressed due to the presence of responsive gold "nano-springs" embedded within the structure.

The work is part of a broader research programme involving a class of materials called molecular frameworks, which is similar to playing with Meccano. "Molecular frameworks are made up of metal nodes with certain shapes, which for us act like hinges, linked together with molecules that act as struts," says Cairns. "For NLC we're usually looking for materials that resemble the famous wine rack, for example."

NLC materials have numerous potential applications, including pressure sensing, pressure-driven actuators, artificial muscles and bullet-proof clothing. "I'm attracted to exotic materials that break the rules," he says. "They do the opposite to what's expected of them, and for me, that's fascinating."

Cairns has recently taken up a postdoc position at ESRF beamline ID27, where he will broaden his experience of high-pressure experiments. Commenting on his former student's success, Andrew Goodwin at the University of Oxford described him as a supremely talented young researcher. "Andrew is a careful experimentalist with a sharp analytic mind and excellent scientific instincts, but above all he is a creative researcher with no fear of the tricky and rewarding science between disciplines."
Kirstin Colvin

• Physicist Davide Orsi at the University of Parma, Italy, won the prize for best poster at this year's User Meeting, describing 2D dynamical arrest transitions in nanoparticle-phospholipid layers.

potential to store information in the same way as proposed for spin vortices but at room temperature."

The third keynote lecture was delivered by experimental biogeochemist Liane Benning from the German Research Center for Geosciences. Her group's research concerns reactions that control the nucleation, growth and crystallisation of mineral phases from solution. These can follow a series of complex stages that are difficult to assess, but new high-resolution *in situ* and time-resolved synchrotron X-ray methods are allowing these processes to be quantified under more realistic conditions. "This knowledge is key to understanding many natural systems, with a huge impact on Earth's element cycles and is also crucial for making novel materials," she explains.

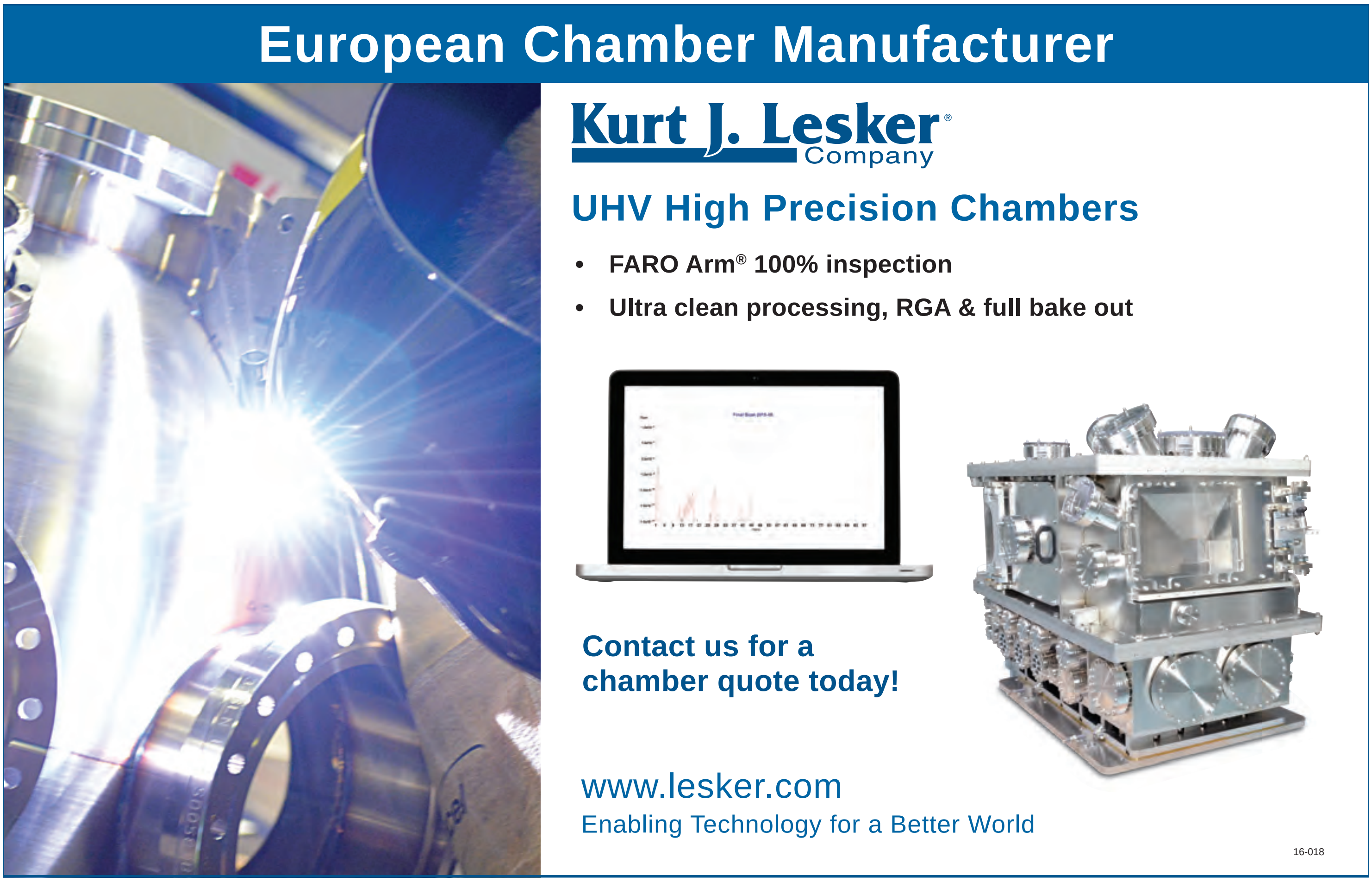
Recently, her team used SAXS and PDF analyses to determine how gypsum forms. Gypsum is the precursor material used in plaster, 100 million tonnes of which are used each year in Europe alone, and it is made via heating to produce the mineral bassanite. "It would save a lot of money if we could find a way to make this process more efficient, so we tried to find out how gypsum forms and saw that bassanite was actually being formed first as a nanophase and that this happened before gypsum crystallised," said Benning. "We have the toys but we still need much more experimental and theory knowledge."

Landmark year

The annual User Meeting is a key event in the ESRF calendar, allowing users, staff and management to share results and shape the future of their science. The final day of the meeting was dedicated to user-dedicated micro-symposia covering nano X-ray diffraction and coherence, dynamics of complex systems, and the future of room-temperature protein crystallography.

During the directors' report, delegates heard about the record numbers of proposals in 2015 and the large number of papers published in high-impact journals. In just over 20 years, experiments at the ESRF have led to 26,831 refereed papers, a tenth of which have appeared in journals with an impact factor greater than seven. The session also updated users on the progress and first results from the upgrade beamlines ID02, ID16 and ID32.

ESRF director-general Francesco Sette described 2015 as a landmark year that saw the completion of the ESRF Upgrade Programme Phase I and the launch of the ESRF–EBS Programme, among other important developments. "I would like to thank all ESRF users and staff for making this possible, and for contributing to a highly successful meeting," he said.
Matthew Chalmers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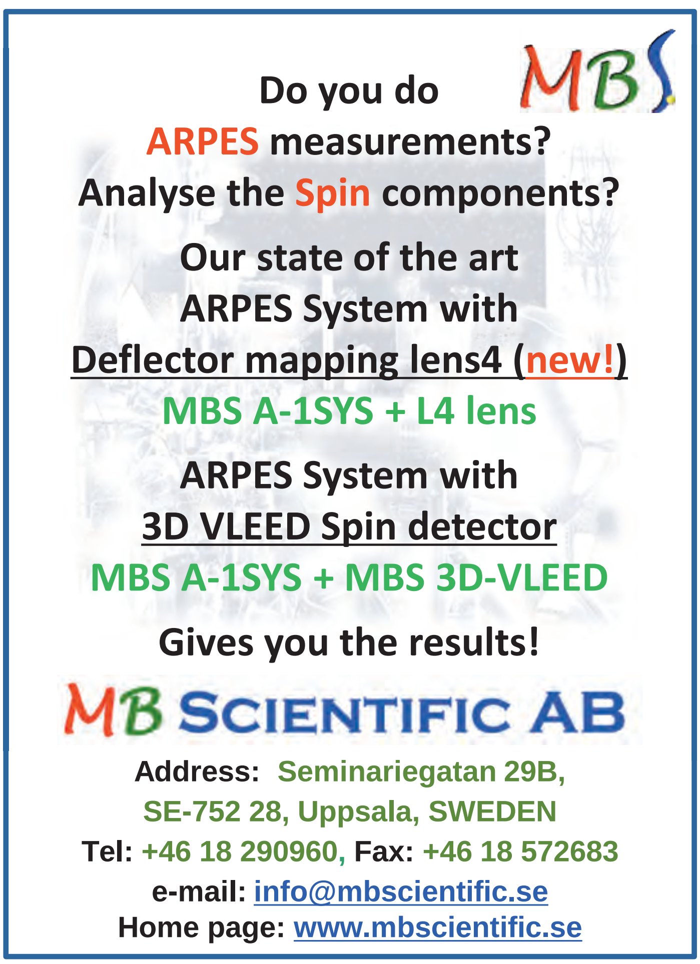
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Instruments That Advance The Art

Bringing big data into line

A bold new data policy at the ESRF will see all data preserved and made available to everyone after a period of three years.

B LEBAYLE

The ESRF has two large tape libraries that are fully automated with robots.

Generating new knowledge from scientific investigation is the primary business of large-scale research facilities such as the ESRF. Data lies at the core of this process, but until recently, the importance of managing this valuable raw material has been largely overlooked. Today, with data volumes at scientific laboratories rising at an unprecedented rate, the need for a clear data policy has become increasingly urgent. The ESRF alone collects around 2PB of data per year and is expected to produce 15PB per year by the end of the decade.

In addition to the technical challenges of storing and processing large datasets, the era of "big data" raises important ethical issues. Concerns about how to preserve data and make it available to others has been under discussion for almost 20 years, not just in science but also in industry and broader society. This has led recently to the creation of the Research Data Alliance, for example, and there are also plans for better data management among the European Commission's Horizon 2020 projects. Indeed, scientific publishers are increasingly requesting that both publications and the original data set are linked with persistent identifiers such as DOIs.

Metadata matters

Against this rapidly evolving backdrop, the ESRF has decided to implement a data policy that advocates full and open access to scientific data collected through publicly funded research. Currently, data at the ESRF are only kept on disk for 50 days before being deleted, and the onus is on users to retain a copy of it and manage the data. The new data policy will see the ESRF take on this role.

Following the model of the existing PaNdata data policy, the ESRF identifies itself as the custodian both of raw data and metadata. The latter is fundamental for organising and indexing data, and the ESRF will therefore aim to collect metadata automatically for all experiments carried out on all its beamlines. This will help facilitate data analysis and long-term archiving, and will also enable the ESRF to certify data and collect statistics about publications. Crucially, it will allow scientists who were not part of the original experiment to verify the results and eventually re-use data to obtain new insights.

Open access

The metadata and a link to the data itself will be stored in a searchable catalogue called ICAT. The associated experimental team will have sole access to the data set for an embargo period of three years, which can be extended if necessary. After the embargo, the data will be released into the public domain with open access under the "CC-BY-4" license, which demands that the original experimental team is correctly cited in all ensuing publications. All data collected at the ESRF will therefore be traceable, verifiable and re-useable. Metadata and raw data will be archived for a period of 10 years, with the option of extending this for more sensitive and unique data sets.

We have already started to implement the ESRF's new data policy. Two beamlines are now collecting metadata and storing it in the metadata catalogue, and nine further beamlines will be depositing data into the archive before the end of the year. The aim is for all ESRF beamlines to follow suit by 2020, when the ESRF's new storage ring (ESRF–EBS) enters operation.

Implementing the data policy is a huge task, involving beamline scientists, software engineers and system administrators. A cross-divisional team has started working on the necessary machinery, which includes metadata capture, electronic log-books, persistent identity management, web-based user interfaces and upgrades to the tape libraries. Physically, the data will be stored on tapes within existing libraries, and the main cost of implementing the data policy will be the required human resources.

The adoption of the data policy by the ESRF Council on 1 December 2015 has kick-started a new era of professional data management and associated services. This will benefit not just ESRF users, but the scientific community at large.

Rudolf Dimper, head of the technical infrastructure division; Andy Götz, head of the software group.

Further information

- ESRF data centre: **www.esrf.fr/news/general/data-centre/index_html**
- PaNdata: **http://pan-data.eu/**
- ICAT: **https://icatproject.org/**

Event puts African Light S on the road to success

There are 47 synchrotron light sources around the world, but none on the African continent. A roadmap launched at the ESRF in November aims to change that picture.

Discussions about building a third-generation synchrotron on the African continent started almost 20 years ago. During this time there has been a steady growth in the number of advanced light-source users from African institutions, but many have had to leave the continent to pursue their careers.

Building a modern synchrotron in Africa would contribute significantly to the African science renaissance by developing skilled personnel and a new generation of young scientists. It would accelerate the growth of African industries and the advancement of research that is relevant to countries on the continent. If Ebola and malaria are to be conquered, for example, Africa must take the lead in identifying target proteins in the disease-causing viruses and parasites to develop appropriate drugs and vaccines.

Southern beginnings

The first proposal for such a facility came from researchers in South Africa, which is by far the largest light-source user community. In 1994, Trevor Derry and Jacques Sellschop from the University of the Witwatersrand became the first synchrotron users from Africa when they carried out studies of diamond surfaces at the newly opened ESRF. Two years later, Giovanni Hearne, also from the University of the Witwatersrand, used the ESRF to study materials under extreme conditions. The experience led him to urge South Africa's National Research Foundation (NRF) to investigate the feasibility of constructing a South African light source in partnership with neighbouring countries.

In 2004, the NRF, in conjunction with the South African government and the South African Institute of Physics, commissioned a report calling for new flagship projects to complement those in astronomy like the Square Kilometre Array. South African light-source users quickly began to establish organisations, in particular the Synchrotron Research Roadmap Implementation Committee (SRRIC).

"We already have the model of the ESRF, SESAME and CERN."

In collaboration with the SAIP in December 2011, the SRRIC convened a synchrotron science workshop in Pretoria attended by a number of international partners, including ESRF director-general Francesco Sette. Implementing one of the workshop's most important recommendations in May 2013, South Africa signed a medium-term membership agreement with the ESRF to contribute at a level of 0.3%.

The first call for a pan-African light source came in 2002 from the African Laser Centre (ALC), which oversees more than 30 laser laboratories across Africa. After many discussions, a formal movement towards an African Light Source (AfLS) was launched in July 2014 and an interim steering committee was elected. This, in turn, led to the first ever AfLS conference and workshop, which was held at the ESRF in November last year. This highly successful meeting achieved several major outcomes, crucially defining a roadmap to guide future activities (see panel on right).

Towards an African Light Source

The main goals in the next three years are to train large numbers of African researchers and students in synchrotron methods, establish formal partnerships with international light sources and promote the involvement of industry. We also aim to enhance Africa's light-source "feeder infrastructure", such as sample preparation facilities, and to promote the project among policymakers and the public. Finally, we aim in this period to develop an AfLS non-site specific pre-conceptual design report.

The main mid-term goals, to be met within the next five years, are to study the feasibility of constructing an AfLS and to develop a detailed business plan. Finally, in the longer term, the goal is to complete a Technical Design Report that includes site selection and details of the storage ring itself. Then, when the project is approved, hopefully within the next 10 years, we can begin construction.

The main hurdle will be to convince African governments of the necessity of an AfLS to help solve problems on the African continent. However, we already have the model of the ESRF, which is a collaboration of 18 European governments plus South Africa, Israel and Russia. More recently the SESAME light source undergoing completion in Jordan, which is

a collaboration between Bahrain, Cyprus, Egypt, Iran, Israel, Jordan, Pakistan, Palestinian Authority and Turkey. UNESCO serves as the umbrella organisation for SESAME and also for CERN, which was established in 1954 and now has 20 member states. Thanks to models like the ALC, the ESRF, SESAME and CERN, the road already is partially paved for an African advanced light source.

Sekazi Mtingwa is a partner at TriSEED Consultants, US; Simon Connell is at the University of Johannesburg, South Africa.

Source

The Grenoble resolutions

- Advanced light sources are the most transformative scientific instruments, similar to the invention of conventional lasers and computers.
- Advanced light sources are revolutionising a myriad of fundamental and applied sciences, with an accompanying impact on sustainable industry.
- The community of researchers around the world are striving collaboratively to construct ever more intense sources of electromagnetic radiation, specifically derived from synchrotron light sources and X-ray free-electron lasers (XFELs), to address the most challenging questions in living and condensed-matter sciences.
- The African Light Source is expected to contribute significantly to the African science renaissance, the return of the African science diaspora, the enhancement of university education, the training of a new generation of young researchers, the growth of competitive African industries, and the advancement of research that addresses issues, challenges and concerns relevant to Africa.
- For African countries to take control of their destinies and become major players in the international community, it is inevitable that a light source must begin construction somewhere on the African continent in the near future, which will promote peace and collaboration among African nations and the wider global community.

Top left: Francesco Sette, ESRF director-general, and Sidiki Zongo from Burkina Faso, who is a student at the University of South Africa, with the traditional African "speaking" stick. Top right: Simon Connell and Sekazi Mtingwa holding the African "summoning" stick, which opened the conference and workshop. Bottom: participants of the workshop at the ESRF. Credit: C Argoud.

ESRF hosts first AfLS conference and workshop

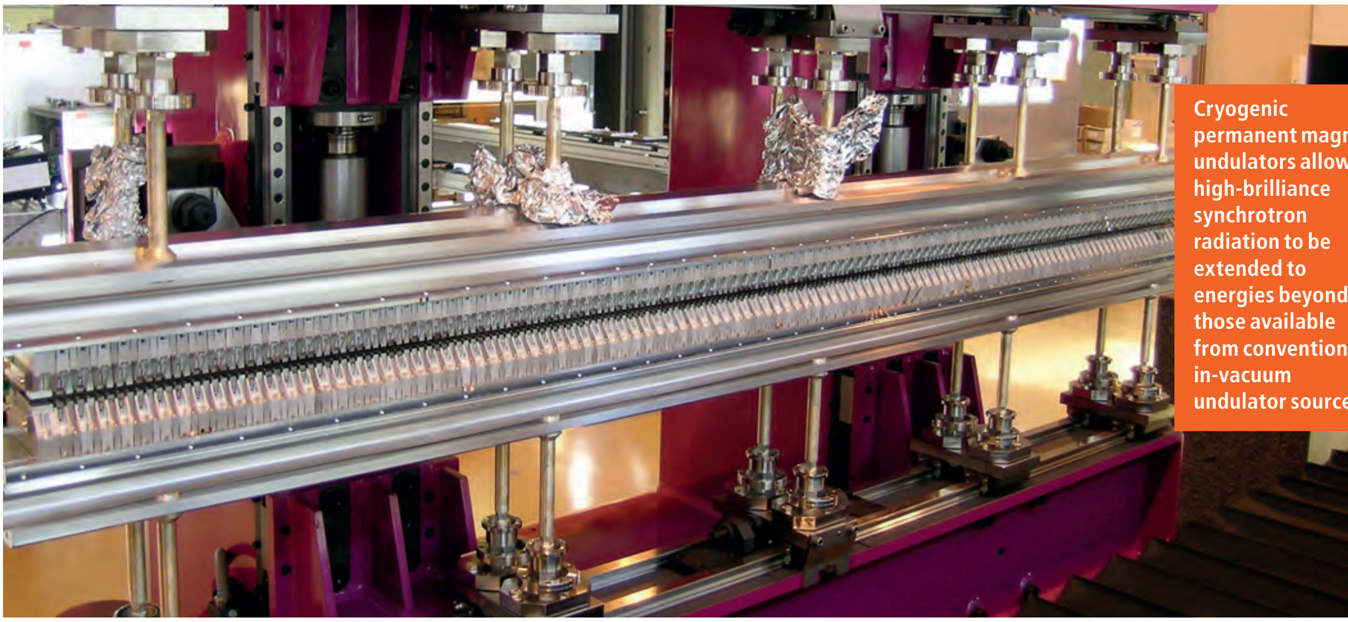
More than 80 scientists and government officials convened at the ESRF on 16–20 November for the first African Light Source (AfLS) conference and workshop. The event showcased the immense contribution of light sources to research and industry across numerous fields, and highlighed particular research relevant to the social, environmental, economic and scientific challenges confronting Africa.

The event concluded with the establishment of a set of resolutions (above) and a roadmap with short-, medium- and long-term goals. A fully mandated steering committee was elected to drive the roadmap forward, with the aim of strengthening the synchrotron user community in Africa.

"If African countries want to take control of their destinies, be competitive socially, politically and economically and become major players in the international scientific community in the years to come, access to a nearby light source will be an absolute necessity," said AfLS proponent Sekazi Mtingwa. "The election of a steering committee and the drawing up of a clear roadmap give us an official mandate to discuss the feasibility of constructing an African Light Source somewhere on the African continent as a collaborative project akin to SESAME."

ESRF director-general Francesco Sette said that a global discussion towards a collaborative African scientific project is highly relevant in the current international climate. "Science is an inescapable driver to peaceful relations among cultures and nations, a force for sustainability and a necessity in confronting the major challenges facing society today," he said. "I'm convinced that the 'Grenoble Resolutions' will mark the beginning of an exceptional adventure for African science and for the international scientific community."

Matthew Chalmers



J CHAVANNE

Cryogenic permanent magnet undulators allow high-brilliance synchrotron radiation to be extended to energies beyond those available from conventional in-vacuum undulator sources.

Surfing the X-ray frontier

The ESRF has eight dedicated high-energy beamlines offering users the ability to probe deep into large-volume samples under industrially relevant conditions.

High-energy X-rays available at the ESRF and a handful of other third-generation synchrotrons have revolutionised studies of the bulk behaviour of materials. With typical energies in the region 80–300 keV, which is around 10 times higher than conventional sources, high-energy X-ray beams penetrate deep into matter – up to a range of several centimetres in the most common transition metals and alloys. This is of great value to users who need to probe thick samples, and in particular for characterising industrially relevant systems.

Experiments using high-energy synchrotron radiation are complementary to neutron probe techniques, which have intrinsically low absorption in most materials. Indeed, the first use of very hard X-rays for condensed matter studies was initiated at the Institute Laue-Langevin in Grenoble in 1969. Following many years of operation of high-energy X-ray beamlines worldwide, involving imaging, inelastic scattering and diffraction techniques, many new fields of research have emerged across the sciences such as *in situ* materials chemistry, and surface and interface science.

Flat spheres

In addition to their large penetration depth, high-energy X-rays have large, almost flat Ewald spheres. This allows users to collect diffraction or scattering intensities along lines and planes in reciprocal space with varying crystal orientation, as well as offering very short exposure times for the detection of weak scattering signals in large-volume samples. A final key advantage of high-energy beams is their low absorption, which is crucial for studies of biological or fragile samples. Lower energy X-rays, by contrast, often lead to failure in such experiments due to the dominance of radiation damage induced by the much stronger photoelectric effect.

Strong growth

Of particular relevance for industry and other scientists studying real-world systems, high-energy X-rays are not impeded by complex or bulky sample environments such as tensile testing rigs, diamond anvil cells, cryostats and furnaces. They are therefore ideally suited for *in situ* or *operando* studies, which is an area that has witnessed particularly strong growth at the ESRF in recent years, with users working on catalysis, metallurgy, energy storage, materials synthesis and many others technologies.

While in the past only a few dedicated high-energy beamlines were available to users, many scattering beamlines have now moved towards higher photon energies. The ESRF–EBS programme will see our high-energy beamlines fed by undulators in order to take full advantage of the 40-times higher brightness of the source. This will improve the time resolution of experiments to the point where users can study irreversible reactions in strongly scattering or high-contrast samples using single-shot X-ray diffraction or imaging techniques. With new detectors to meet these challenges, ESRF–EBS will also allow coherence to be exploited in the high-energy regime.

Harald Reichert, ESRF director of research; Veijo Honkimäki, head of the structure of materials group.

High-energy beamlines at the ESRF

- **ID11 (18–140 keV)** Materials science beamline dedicated to moderate- to high-energy diffraction and/or imaging studies of a wide variety of systems.
- **ID15A (20–750 keV)** Dedicated to high-energy studies in materials chemistry and engineering, especially *in situ* catalysis experiments.
- **ID16B (6–65 keV)** Nano-analysis end station linking distribution, concentration and speciation of trace elements to morphology and crystallographic orientation.
- **ID17 (25–115 keV)** Dedicated to large field-of-view imaging, radiation biology and radiation therapy both *in vitro* and *in vivo.*
- **ID19 (6–250 keV)** Microtomography beamline devoted to 3D and phase-contrast imaging for a wide variety of topics including palaeontology.
- **ID22 (6–80 keV)** Dedicated to high-resolution powder diffraction for structural, dynamic and *in situ* experiments.
- **ID27 (20–90 keV)** Premier X-ray powder and single crystal diffraction station dedicated to research at extreme pressures and temperatures.
- **ID31 (20–150 keV)** Portfolio of hard X-ray techniques including reflectivity, wide- and small-angle scattering and imaging for time-resolved studies in materials processing.

X-ray steps to better health

Experiments using high-energy synchrotron X-rays are directly connected to the multi-billion euro global pharmaceutical industry.

Public health depends increasingly upon a continuous stream of pharmaceutical drug development. Antibiotic resistance and the spread of new hazards such as Ebola, for instance, are in direct competition with our limited stock of medicines. High-energy X-rays play a key role in drug development, which begins with the identification of an active pharmaceutical ingredient (API).

Once a new API has been developed, which involves laborious screening of candidate molecules against biological receptors or infective agents such as viruses, the question is how best to deliver the API to the target. Since the API must be placed into a form that is stable under the required storage or transport conditions, the delivery process begins immediately after synthesis. The API must also be made biologically available in the most efficient manner, which is often a case of controlling solubility but might incorporate more complex mechanisms such as controlled release kinetics.

Recent experiments carried out by independent user groups showcase the ESRF's capabilities in the key stages of drug development, from probing the role of hydrogen bonds in an organic compound to monitoring the formation of pharmaceutical co-crystals *in situ* (see orange panel).

Intellectual property

High-energy synchrotron X-rays also underpin a booming industry related to intellectual property. Each step in the formulation of an API can be patented and protected, right down to the particular polymorph formed. Although molecular structure can be determined using NMR and other analytical methods, determining and predicting the crystal structures adopted by molecular materials remains a serious challenge. This is especially important in pharmaceuticals because patents only apply to crystal structures that have been clearly identified.

Polymorphism is extremely common for organic small molecules, and for a given API a polymorph may show wildly different physical properties and even pharmaceutical activities. Seemingly esoteric experiments at synchrotrons are therefore directly connected to a multi-billion euro global industry.
Simon Kimber, scientist at beamline ID15A.

From drug discovery to final formulation

- **Understanding polymorph stability and structure**
  Ayoub Nassour of the CNRS and colleagues used charge density analysis at beamline ID11 to understand how hydrogen bonds in the compound 2-methyl-1,3-cyclopentanedione influence the crystal structure. Very high-energy X-rays allowed structural data to be collected with sub-Ångstrom resolution, revealing how delocalisation along a certain direction drives the formation of a short hydrogen bond that helps explain the predominant crystal packing (*Acta Crystallographica* **B70** 197).

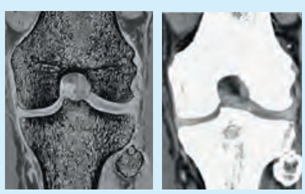
- **Tracking co-crystal formation during bulk processing**
  Ivan Halasz of the Ruđer Bošković Institute in Croatia and co-workers used X-ray powder diffraction at beamline ID15B (now ID15A) to monitor structural and chemical transformations during mechanochemical milling in real-time and *in situ*. Milling is widely used in the production of pharmaceutical co-crystals, which are key to improving the solubility, bioavailability and tableting properties of the API, and the team observed the formation of never-before-seen intermediate phases (*Angewandte Chemie, International Edition* **52** 11538).

- **Characterising crystalline APIs**
  Maxwell Terban of Columbia University in the US and co-workers, including scientists from GlaxoSmithKline, used difference atomic pair distribution function (PDF) methods at beamline ID15B to detect and characterise nanoparticles of a pharmaceutical compound suspended in a solvent at very dilute concentrations. The work paves the way to studies of crystalline APIs in amorphous matrix formulations, or inorganic nanoparticles embedded in a glass matrix (*Nanoscale* **7** 5480).

- **Evidence for polyamorphism in paracetamol**
  Yen Nguyen Thi of the BAM Federal Institute for Materials Research and Testing in Germany and co-workers used sophisticated sample environments and *in situ* X-ray scattering and spectroscopy at beamline ID11 to trace the crystallisation behaviour of paracetamol from organic compounds. Different solvents promote the formation of different polymorphs, and PDF analysis showed that paracetamol exists in several different amorphous forms, which is important for patenting (*CrystEngComm* **17** 9029).

High-energy X-rays for medical imaging

Researchers at ESRF beamline ID17 are exploiting high-energy X-rays to produce stunning images of the human body. Unlike conventional hospital sources, high-energy X-rays (60 keV and above) are able to penetrate and thus image bone while still revealing details of cartilage and other soft tissue. A 2014 study led by researchers from Ludwig Maximilian University of Munich used a phase-contrast technique to obtain unprecedented images of the human knee (pictured left, compared to conventional CT pictured right). Although the sample came from a cadaver, results show that the required X-ray dose is tolerable in live patients. "It's like having an



MRI and a hospital X-ray CT scan combined at the same time," explains ID17 scientist-in-charge Alberto Bravin. The ID17 high-energy imaging programme is proceeding in two directions, he says. The first is to study different organs in order to expand the range of biomedical applications, while the second aims at achieving a virtual histology in 3D that will help understand at the micron level the physiological effects related to certain neurological diseases. "These features are invisible using conventional techniques." *MC*

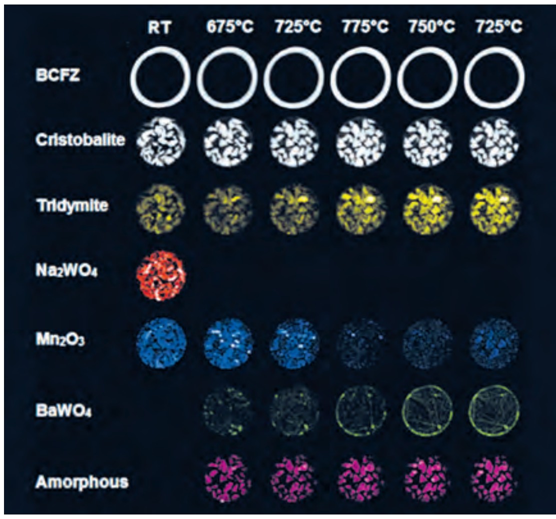
Catching catalysis in action

X-ray diffraction computed tomography has allowed users to investigate the chemical evolution of a working catalytic reactor for the conversion of methane into ethylene.

Ethylene is an important chemical that is used for the production of a wide range of products including plastics, detergents and antifreeze. With the decreasing use of energy-intensive and expensive naphtha crackers in the past few years, however, new ethylene production processes are required. Fortunately, thanks to the reduction of gas flaring and the exploitation of shale gas by hydraulic fracturing, there has been a dramatic increase in the availability of methane.

The challenge is to find environmentally friendly and cost-effective processing technologies that can directly convert methane into light olefins such as ethylene. A potentially viable route is the oxidative coupling of methane (OCM), which was discovered in the 1980s. One of the drawbacks of the reaction is the total oxidation of methane and/or the conversion of carbon molecules into CO_x, but it is possible to overcome this using catalytic membrane reactors in which the flow of oxygen to the catalyst is regulated by an external membrane. Such devices make processing cheaper and more efficient.

Membrane reactors consist of two main components: an OCM catalyst surrounded by a dense oxygen-transport membrane made from ceramic hollow fibre. Traditionally, researchers have characterised such integrated reactor systems *ex situ* using techniques such as SEM and powder X-ray diffraction, but there has been a lack of *in situ* and *operando* studies. The problem is that you need very hard X-rays in order to penetrate the reactor and therefore acquire a useful chemical signal.



Phase maps of the evolution of the catalyst as a function of temperature, showing five identified crystalline phases and one amorphous phase – in addition to the membrane itself (top row).

Synchrotron solution

Recently, we used high-energy X-ray diffraction computed tomography (XRD-CT) at ESRF beamline ID15A to investigate the chemical evolution of a working catalytic reactor for the OCM reaction. This first ever solid-state study under operating conditions was based on a state-of-the-art OCM catalyst (Mn-Na-W/SiO_2) and a "BCFZ" oxygen transport membrane. XRD-CT allowed us to obtain spatially resolved chemical information and also to observe the catalyst and membrane for the first time in their active states (*Chem. Commun.* **51** 12752).

Our results reveal that the catalyst undergoes a solid-state chemical evolution during the OCM experiment: not only are the active components of the catalyst changing but also the SiO_2 support itself changes with time. More importantly, at high temperatures a new phase ($BaWO_4$) was observed to form and grow at the interface between the catalyst particles and the membrane. This is important because the loss of tungsten could impact the long-term stability of the catalyst and can lead to the formation of a layer at the membrane wall that blocks the flow of oxygen to the catalyst bed.

The reactors have been assembled at pilot scale within an EC FP7 funded project called DEMCAMER, and research is ongoing to improve the stability and performance of the reactor under a newly awarded EC Horizon 2020 project called MEMERE with the aim of full industrial scale-up.

Simon Jacques, University of Manchester, and Antony Vamvakeros, University College London, UK.

INDUSTRY CASE STUDY

BP characterises catalysts for Fischer-Tropsch reaction

The petrochemical industry produces vast quantities of useful hydrocarbons each year, so even the slightest improvement in catalytic efficiency can bring major economic and environmental benefits. BP recently joined forces with the ESRF and University College London (UCL) in the UK to investigate ways to boost the Fischer–Tropsch process, which converts carbon monoxide and hydrogen gas into hydrocarbons such as diesel or kerosene.

Pierre Senecal of UCL, who is nearing the end of a two-year postdoc sponsored by BP, has visited the ESRF several times to take advantage of the high-energy X-rays at beamlines ID11, ID15 and ID31, which can probe deep into the samples. "In this project we are looking at mm-sized catalyst pellets that are prepared in exactly the same way that they will be put into an actual reactor," he says. "Crucially we are doing this *in operando*."

Reconstructed images of cobalt phases in mm-sized samples observed under reduction of a cobalt catalyst precursor.

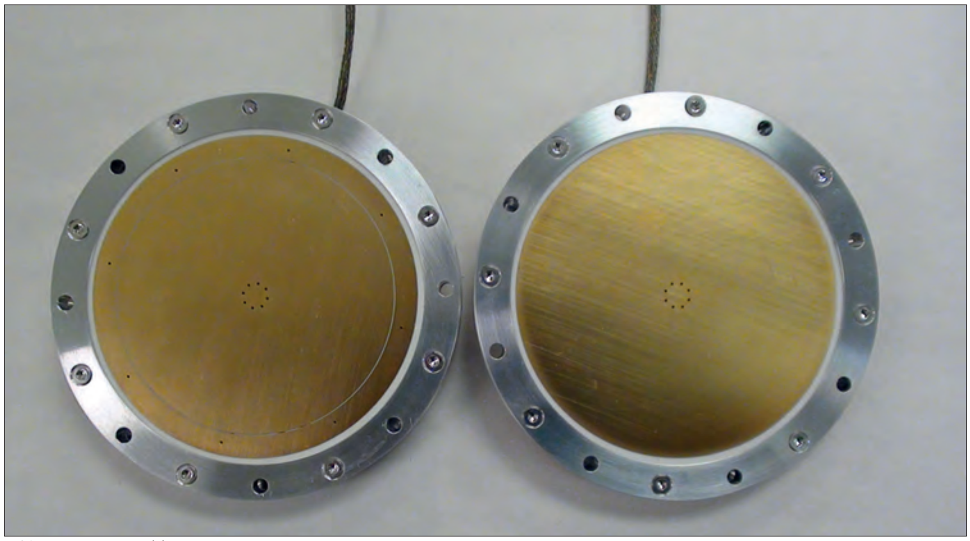
First the team tested the approach using a basic cobalt-on-alumina catalyst. X-ray diffraction computed tomography revealed the different phases inside the catalyst as it was reduced from cobalt into cobalt oxide and eventually cobalt metal, which is the active phase for the Fischer–Tropsch reaction. The main advantage over laboratory-based XRD, explains Senecal, is that the technique also gives the position of the different phases. This reveals whether the catalyst is getting reduced first on the outside surface of the pellet or deeper into it, helping researchers to compare catalysts.

In the second part of the project, which is now being prepared for publication, the team applied the technique to a new catalyst that is under investigation by BP. The aim is to try different combinations of elements and doping levels to optimise the process, explains project leader Andy Beale of UCL. "We're trying to put spatial information into determining catalysis efficiency and the fact that we can do chemical imaging in real time is a real advantage." *MC*

Positioning in a vacuum

Vibrational stability in beamline optics continues to evolve to comply with the improved brilliance of low-emittance storage rings around the world. This requires the vibrational performance of the beamline to be characterized, monitored and often compensated for during operation to nanometre levels of performance.

Vacuum versions of Queensgate's nanopositioning and nanosensing products are used to providing active damping to reduce vibration and to measure vibration to 0.1 nm. We also produce custom designs and variants where an off-the-shelf solution is not available.



NCG-1-AL-UHV capacitive sensors.

Material selection for UHV-prepared products

Providing products to operate at UHV, frequently at cryogenic temperatures and potentially needing to be rad-hard, requires that we select materials carefully. The higher the vacuum the more important it is to source materials that have low outgassing. This often means that we avoid materials such as brass, zinc, lead, etc, as not suitable for UHV. Polymers must also have low outgassing, so our standard PVC wiring is replaced with Kapton-insulated wires, and all bonding agents are selected using the NASA outgassing database.

Vacuum preparation

The preparation of materials and components is also important because the manufacturing process can introduce problems under vacuum conditions. Parts are therefore ultrasonically cleaned to remove oils and carbon residue from any metalwork. Prior to installation, we recommend a bake-out to remove any solvent residues.

Sensors

The NanoSensor® is a non-contact position measuring system based on the principle of capacitance micrometry. The sensors are non-self-heating and maintain their zero position when powered down. Resolutions as small as 7 picometres (RMS) can be achieved. Measurement ranges from 20 µm up to 11 mm are available as standard, with frequency responses up to 5 KHz and linearity down to 0.02 %.

DPT-D range of actuators.

The NX NanoSensor® is a series of high-resolution capacitive sensors, constructed of either super invar where temperature coefficient is critical or lower-cost aluminium. They are available in a number of shapes and sizes to operate over different ranges and with a UHV variant, which can be used to 10^{-9} Torr.

The NC NanoSensor® is a new series of high-resolution capacitive sensors developed specifically for UHV to 10^{-10} Torr. Their ceramic gold construction has removed all adhesive bonding agents and the construction also means that they can be baked out at higher temperatures and be used in high-radiation environments.

Closed-loop actuators

The DPT-D range of actuators are designed with capacitive feedback control to give precise positioning. A system comprising a DPT-D actuator with fully programmable NPC-D-5200 digital closed-loop controller is ideal for the most demanding applications. The capability to move loads of up to 60 kg over the full travel range with low electronic noise, high linearity and fast settle time gives confidence that the actuator is positioned with precision, speed and accuracy. High-thermal-stability super-invar construction gives superior positional stability with UHV compatibility to 10^{-9} Torr. The DPT-E range of actuators offers a low-voltage replacement for the DPT-C range, and hence provides an upgrade path for existing users.

Open-loop actuators

The MTP range of actuators offer an open-loop alternative with up to 105 µm displacement. They have a rugged stainless-steel construction and are internally pre-loaded to provide high pulling forces. The MTP actuators can be used with a Queensgate NanoSensor® to provide a sub-nanometre precision closed-loop system, and allows the sensor to be mounted in the most appropriate position for system accuracy. There is a comprehensive range of accessories available for both the MTP and DPT ranges of actuators.

Stages

Vacuum-compatible versions of Queensgate flexure-guided piezo-actuated stages are available and assembled using Kapton wiring. The stages can be used to position optics with sub-nanometre precision.

As part of a vacuum nanopositioning/nanosensing system, a feedthrough and airside cable is required. Suitable feedthroughs can be provided by Queensgate or sent to be calibrated as part of a complete system calibration.

Queensgate specialise in offering product solutions with the highest stability, performance and reliability, to meet individual customer requirements. Our engineers, scientists and manufacturing experts provide the best solution for challenging problems. We can advise on the best use of standard products and are specialists in delivering custom systems. Products are engineered to last, with many systems in continuous operation in excess of 20 years. Queensgate offers a number of products, including flexure-guided stages, closed-loop actuators and nanoposition sensors, as well as high-quality control electronics.

If you have a positioning application where you require a nanopositioning solution, please contact us.

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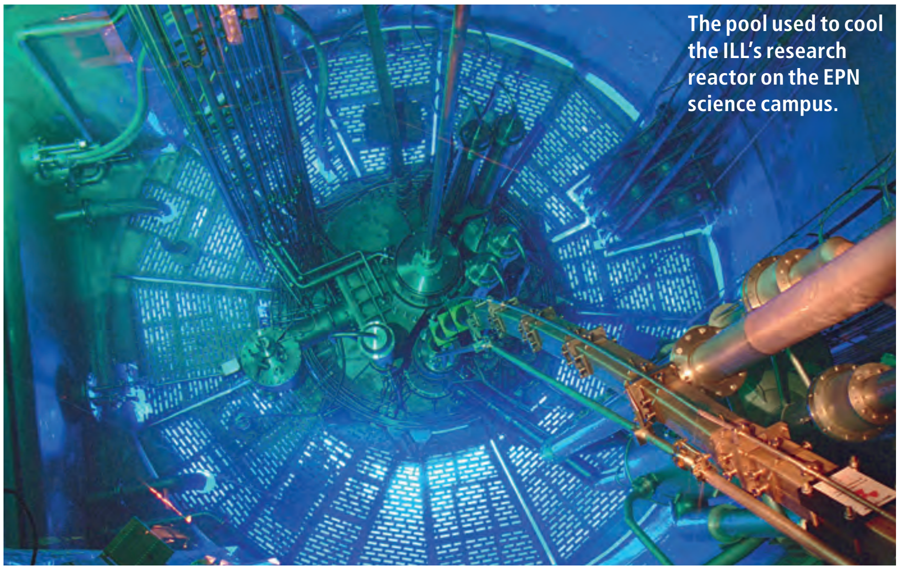
Contact
Web www.nanopositioning.com/
Email info@queensgate.com

Towards safer nuclear fuels

Tracking the microstructure of uranium-molybdenum particles helps manufacturers develop low-enriched fuels for research reactors.



ILL/JL BAUDET

The pool used to cool the ILL's research reactor on the EPN science campus.

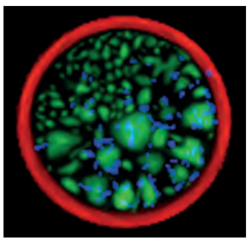
Neutron sources are a vital tool in many fields, ranging from basic science to industrial challenges such as material doping for microelectronics and radioisotope production for medical imaging. More than 20 such sources are operational, most taking the form of small nuclear reactors.

The most powerful research reactors use highly enriched uranium, in which the fraction of the fissile isotope uranium-235 can be as large as 93%. However, non-proliferation issues have led to a worldwide initiative to convert these reactors so that they operate with low-enriched nuclear fuels containing less than 20% uranium-235. Doing so without incurring significant loss in performance represents a significant challenge.

Promising alternative

The most promising nuclear-fuel candidate is based on high-density uranium-molybdenum (UMo) particles dispersed in an aluminum matrix, which are processed to form nuclear fuel plates measuring about 1 mm thick. Thanks to high-energy synchrotron X-rays, we are now able to monitor the microstructure of UMo particles both at different scales and during different manufacturing steps, ranging from initial atomisation through to the final fuel plate after hot-rolling.

In 2011, the present authors used the ESRF to obtain the first quantitative microscale characterisation of as-fabricated UMo fuel plates. Conventional X-ray beams are not able to provide quantitative phase information due to the large difference in absorption between UMo particles and the aluminum matrix.



The results of XRD-CT analysis on an atomized UMo particle (diameter 20 µm) showing UO_2 (red), UMo (green) and U(C,O) (blue).

Using high-energy 87 keV X-rays and X-ray diffraction at former ESRF beamline ID15B, however, we were able to reveal the crystalline phases present in the UMo particles. Our results allowed the composition of the fuel to be optimised at trace level, enabling us to propose improvements to the manufacturing process (*Z. Kristallogr. Proc.* **1** 29).

More recently, we used nano X-ray diffraction computed tomography at ESRF beamline ID22NI (now ID16B) to carry out complementary studies at a spatial resolution of 0.2 µm. By producing images of the microstructure of the atomised metallic particles at the nanoscale, we were able to to investigate whether the most abundant impurity phase, U(C,O), influences the formation of small uranium-molybdenum grains during solidification, which currently limits the performance of research-reactor fuels. Rather than act as a nucleation site for UMo grains, the results showed that U(C,O) forms during UMo grain solidification and growth (*Appl. Phys. Lett*. **105** 084103).

Extended collaborations

Our studies have enabled extended collaborations with industrial teams in Europe and Korea that are seeking their own solutions to low-enriched reactor fuels. The development of such fuels in Europe is proceeding under the support of Euratom and in close collaboration with the US Department of Energy, with the objective to provide a robust solution by 2025.

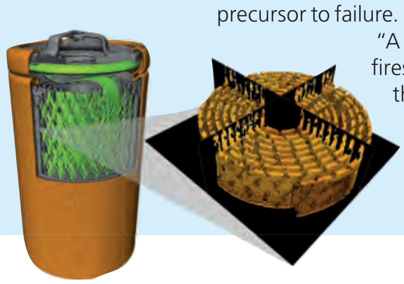
Indeed, the ability to probe the micro- and nanostructure of fuel systems is also extremely promising for investigating the evolution of irradiated fuels used in nuclear power plants.

Hervé Palancher is at CEA-Cadarache in France; Anne Bonnin is at Paul Scherrer Institut, Villigen, Switzerland.

High-energy study reveals batteries in operation

The unique high-speed imaging capabilities of ESRF beamline ID15A have revealed the inner workings of a commercial lithium primary battery for the first time (DOI: 10.1002/advs.201500332).

Donal Finegan of University College London (UCL) and colleagues used high-speed synchrotron X-ray CT to capture a series of tomograms during operation and discharge of the cell, finding a large degree of volume change among electrode materials. This leads to a loss of electrical contact, which can lead to higher local temperatures and is often a precursor to failure.



"A number of aircraft fires have been linked to the failure of lithium-ion batteries, so we were motivated to see which parts of the battery were working under normal operating conditions," explains co-author Paul Shearing of UCL. The team, which recently used the ESRF to perform similar studies of a cell under more extreme loads, now plans to push the capabilities of the technique further to span a range of spatial and temporal scales as well as different thermal and mechanical abuse problems.

"These problems occur over many time and length scales, with the most rapid processes occurring in milliseconds," says Shearing. "The energy of the beam was critical because we are looking at off-the-shelf cells encased in a steel container." *MC*

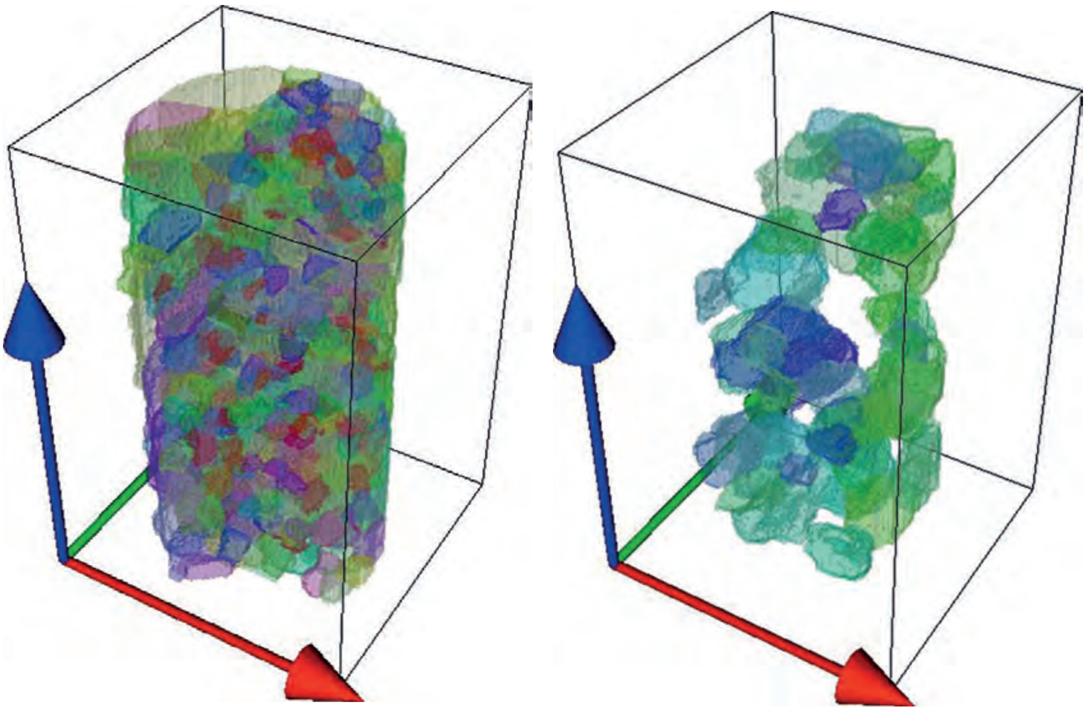
Twin study aids metallurgy

Experiments at beamline ID11 reveal the development of twins in hexagonal metals, which is crucial for understanding the onset of plasticity in engineering materials.

As every physics student learns, materials stretch or compress under a mechanical load. For small loads, materials behave elastically and return to their initial shape, while larger loads can cause a material to undergo plastic deformation and change shape. In a practical engineering situation, it is vital that a material is in the elastic regime in order to prevent the failure of components.

Two groups of users have recently taken advantage of the high-energy X-ray beams at ESRF beamline ID11 to investigate the microscopic processes that determine the transition from elasticity to plasticity in hexagonal close-packed metals, which include titanium, zirconium and magnesium alloys. The studies took advantage of new techniques called diffraction computed tomography (DCT) and 3D X-ray diffraction (3DXRD), which allow users to see the grains inside of materials by looking at diffraction from their crystal lattices.

"The results of such experiments ultimately allow researchers to design metals with strong preferred orientation for taking loads in particular directions," says ID11 scientist Jonathan Wright.



The 3D grain structure of a titanium alloy (left) and a twin network inside it (right), where the colours represent the crystallographic orientations of each grain.

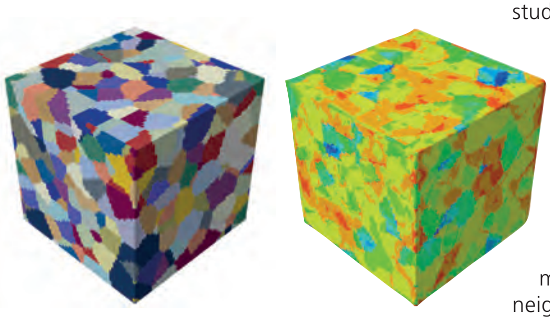
Deformation by twinning

While many metallic materials deform due to dislocation slip, some undergo an additional deformation mode called twinning. Twinning arises from reshuffled atoms that induce a reorientation of the local crystal structure and a shear strain, but the precise criteria for twin nucleation are not known.

Using DCT, Laura Nervo of the University of Manchester in the UK, who is also co-funded by the ESRF, and colleagues were able to study the neighbourhood of each twinned grain and identify twin networks for the first time (*Acta Materialia* **105** 417). The team studied a titanium alloy before and after deformation, finding that twins develop 3D networks and that the formation of twins is promoted by microtexture regions in which grains are well aligned (see above). "Such a result is vital for validating new plasticity models that attempt to incorporate twinning," explains team leader Michael Preuss of the University of Manchester.

Since no single grain can deform without neighbouring grains having to accommodate the deformation, non-destructive methods are vital when studying the onset of plasticity. "We need to be able to detect micron-wide features such as twins, but at the same time study large enough volumes so that we can make statistical comparisons," explains Preuss. "This is only possible at a synchrotron source such as the ESRF."

The work has strong parallels with another recent study at ID11 based on a zirconium alloy. Hamidreza Abdolvand of the University of Oxford in the UK and co-workers investigated the stress in individual grains of the metal, rather than analysing the grain neighbourhood, and for the first time were able to measure the load sharing between twin and parent pairs statistically and in 3D (*Acta Materialia* **93** 246).



3D grain maps revealing deformation in a zirconium alloy, showing grain shapes (left) and their associated stress fields (right).

Zirconium and its alloys are extensively used in nuclear reactors on account of their low neutron absorption cross-sections, but many fundamental questions regarding their deformation mechanisms remain unanswered. Since twinning can cause drastic changes to the material's texture during processing, understanding the deformation mechanism of twins is essential for predicting the in-service performance or lifetime of materials.

Unique and efficient

Abdolvand and co-workers used 3DXRD, which is a complementary technique to DCT that offers higher-resolution measurements of strain and orientation and therefore provides a unique opportunity to study a large number of twins with various different neighbourhoods.

"The capability of running *in situ* deformation experiments along with the use of high-energy and high-intensity X-ray beams, and the assistance of experienced scientists at ID11, make the ESRF a unique and efficient place to run 3DXRD experiments," says Abdolvand. "This data set has led us to propose new constitutive equations for the accurate modelling of deformation in hexagonal-close packed polycrystalline materials."

Matthew Chalmers

Accelerating synchrotron science

President of the INFN, **Fernando Ferroni**, describes the long-standing collaboration with the ESRF and how Italy's expertise in accelerator physics will help make ESRF–EBS a reality.

Particle physicist Fernando Ferroni has been president of the Istituto Nazionale di Fisica Nucleare (INFN) since October 2011. Founded in 1951 and headquartered in Rome, the INFN is Italy's coordinating institute for nuclear, particle and astroparticle physics. It manages large facilities across four national laboratories, including a 1 GeV electron-positron collider in Frascati called DAFNE, a 70 MeV cyclotron near Padova for radioisotope research and accelerators in Florence dedicated to cultural-heritage studies. Italy's national light source, the 2.4 GeV Elettra facility in Trieste, falls outside the INFN's remit.

It's a complicated structure, admits Ferroni, with most INFN researchers being located in the physics departments of 21 Italian universities. In the 1950s it built Europe's first synchrotron, a 1.1 GeV machine that was the most powerful in Europe at the time, and the first electron-positron collider was also built in the INFN's Frascati laboratories.

INFN

Fernando Ferroni in brief

Born: Rome, 1952.

Education: Laurea in Fisica, 1975.

Career: Research associate, INFN-Rome (1977–1982); assistant professor, University of Rome (1982–1988); associate professor, University of Ancona (1988–1991); visiting scientist, CERN (1991–1992); associate professor, University of Rome (1992–1999); visiting scientist, SLAC (1998–1999); professor, University of Rome (2000–)

Family: Married.

Interests: Mountain activities.

> "This collaboration with the ESRF will engage a new generation of physicists."

Strong links

Italy's pioneering accelerator R&D underpins a new agreement between the INFN and the ESRF that was signed earlier this year. It will see the two institutes collaborate on key technologies for the ESRF's next phase of development: ESRF–EBS, a groundbreaking new storage ring that will be 100 times brighter than the existing source.

"The experience we have developed over many decades with DAFNE and many other accelerators means that the INFN is able to contribute significantly to the construction of ESRF–EBS," says Ferroni. "The INFN is very attentive to make available its expertise and technology to all undertakings in which accelerators are used for science in general, and for applications to the benefit of society."

Collaboration between the ESRF and INFN goes back almost 40 years. As early as 1978, more than a decade before construction of the ESRF started, researchers at INFN's Frascati laboratory were preparing memos about how to correct chromaticity in the new machine. Throughout the 1980s the two institutes collaborated closely on designing the optics for the ESRF storage ring, with INFN researchers contributing technical material for the ESRF's 1984 report, the *Green Book*.

History is now being repeated thanks to a new agreement that was formally endorsed by ministers from France and Italy at the ESRF on 8 February. "The ESRF requirement is to be a very brilliant machine, which means stretching components to the limits of technology," says Ferroni. DAFNE has a lot to teach us here, he adds, as does the Italian electron-positron collider "Super B factory" that was cancelled before construction could begin.

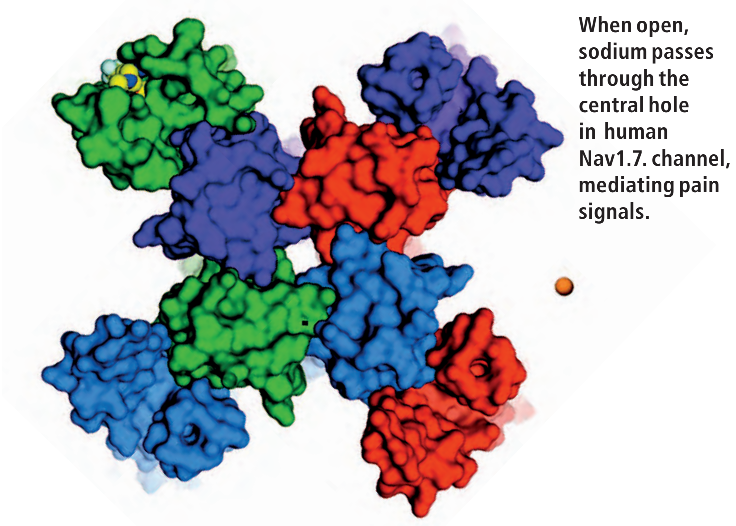
The key to that machine, says Ferroni, was to find out how to make the beams as narrow as possible – a problem that was solved by former INFN director of accelerators Pantaleo Raimondi, who is now director of the ESRF's Accelerator and Source Division. "Pantaleo had the idea for ESRF–EBS while he was designing the B factory at Frascati," says Ferroni. "One way is to increase the current and the other way is to make the beam pipe smaller. The latter has never been pushed to the limit, but he found out how to get these very narrow beams." In doing so, Raimondi had found an effective match between the two labs. "The vacuum chamber of DAFNE, which is made from aluminium, is seen as a good model for ESRF–EBS so we took charge of designing the beam pipe for ESRF–EBS, and the first of these have already been tested."

Reaching out

In addition to the ESRF, the INFN is heavily involved in the construction of the European XFEL in Hamburg, Germany, to which Italy is a member at the level of 3% and has made a €35 m contribution towards construction. The organisation is also building part of the superconducting accelerating cavities for the European Spallation Source in Sweden, and is working in collaboration with the SESAME light source in Jordan to develop resonant cavities and X-ray detectors.

"Although this latest agreement with the ESRF is quite high profile, we are always cooperating with each other – this is the beauty of physics," says Ferroni. "As the ESRF and E-XFEL have shown, it's very difficult to build something on your own because at some point you need someone who knows more things than you do. This collaboration with the ESRF will engage a new generation of physicists and entail lots of discussion and mutual understanding. It is the start of something that is very important."

Matthew Chalmers



When open, sodium passes through the central hole in human Nav1.7. channel, mediating pain signals.

A new view on pain relief

Genentech researchers have determined the structure of a previously unknown drug-binding site that could accelerate the development of pain treatments.

The opening of voltage-gated sodium (Nav) channels initiates action potentials in muscle cells and neurons, making them essential elements of the electrical signalling that occurs throughout our bodies. The human body contains nine Nav channel isoforms, with mutations in these channels associated with migraine, epilepsy, pain, cardiac and muscle paralysis syndromes. In particular, mutations of Nav1.7 (which is expressed primarily in sensory neurons) have been linked to extreme pain disorders. The loss of function of Nav1.7 has also been shown to cause a complete inability to feel pain, making it a promising target in the pain-perception pathway.

Human Nav channels are large membrane proteins that contain a central pore module surrounded by four peripheral voltage-sensing domains (VSDs). Available Nav channel blockers used to treat neurological and cardiovascular disorders lack isoform selectivity and thus therapeutic utility because they bind within the highly conserved central pore module. Recently, however, a new class of antagonist has been reported that seems to target a unique binding site on the fourth voltage-sensor domain (VSD4), raising hopes that an isoform-selective inhibitor can be found.

Unfortunately, the inherent complexity of human Nav channels makes it difficult to determine their 3D structures. To overcome this, researchers from US biotech firm Genentech exploited a simpler bacterial sodium channel by fusing portions of Nav1.7 VSD4 onto it, allowing them to purify, crystallise and determine the structure of potent aryl sulfonamide inhibitors in complex with the human Nav1.7 VSD4 receptor site (*Science* **350** 6267).

To understand the mechanism of Nav1.7 inhibition by this novel class of antagonist, the researchers pursued high-resolution structural studies at ESRF beamline ID29. High-quality X-ray diffraction data revealed that the anionic "warhead" from the inhibitors directly engages the voltage-sensing "S4 helix", which effectively traps VSD4 in its activated state and inhibits the Nav1.7 channel. Remarkably, the drug receptor site is partially submerged within the membrane.

Thanks to this novel crystallisation strategy and the ESRF, the structure of VSD4 from human Nav1.7, in complex with potent isoform-selective small molecule antagonists, has been determined. It is hoped that these structures will accelerate the development of new treatments for pain that target Nav1.7 and aid drug design efforts aimed at other voltage-gated ion channels.

Unilever targets product stability

Hair conditioners, used daily by millions of people to detangle hair and ease combing and styling, are based on a dispersion of liquid crystalline phases that work with the flow of water to lubricate and protect hair fibres.

The stability of such products is vital for guaranteeing consistent performance, which is especially important to firms such as Unilever given the wide range of formulations needed to serve consumer needs. In order to validate the stability of the company's hair conditioner products, including a variety of ingredients and their permutations, Unilever products were shipped to the ESRF last year to undergo a three-month stability trial.

"The validation of stability on a routine basis can only practically be done with characterisation techniques that measure physical properties of the bulk product, such as rheology, pH, light scattering and calorimetry," explains research scientist Cesar Mendoza at Unilever Port Sunlight in the UK. "However, it is difficult to understand what and how underlying microstructures influence the bulk properties and any changes that occur over time."

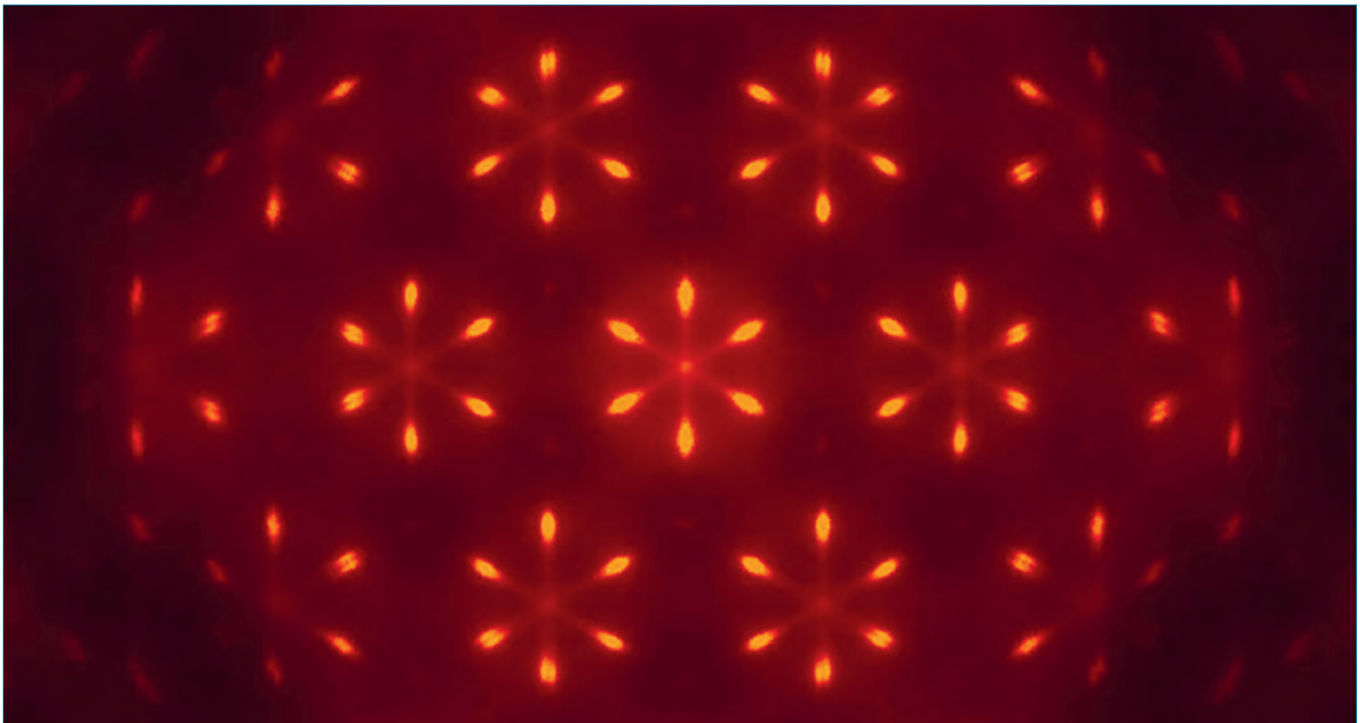
Using small angle X-ray scattering at ESRF beamline ID02, the team monitored the microstructure evolution of the products at critical intervals during the trial period. Although the details of the results are confidential, the experiment proved instrumental in providing a mechanistic understanding of how a hair conditioner microstructure evolves with the passage of time, from factory to consumer, says Mendoza. "This gives our R&D a better insight into key quality and performance attributes."

Movers and shakers

Giacomo Ghiringhelli from the Politecnico di Milano in Italy has won the Kai Siegbahn Prize 2015 for his seminal contributions to spectroscopy at the ESRF's ID08 and ID32 beamlines, as well as at the Swiss Light Source. Ghiringhelli has a long-standing relationship with the ESRF since 1994. He worked initially as an undergraduate and then PhD student on beamline ID12B, and has been a regular user since 2001. The €3000 prize, which was awarded at the University of Uppsala in Sweden last September, specifically recognises his outstanding, innovative work in the experimental development and scientific exploitation of resonant inelastic X-ray scattering (RIXS) in the soft X-ray regime. Ghiringhelli has used this and other synchrotron techniques to study high-temperature superconducting cuprates, and he also played an important role in the 10m-long spectrometer for the European RIXS facility ERIXS that is now operational at beamline ID32.

Biophysicist and ESRF user **Venki Ramakrishnan** took up the five-year long post as President of the Royal Society on 1 December. A co-recipient of the Nobel Prize for Chemistry in 2009, and currently deputy director of the MRC Laboratory for Molecular Biology in Cambridge, Ramakrishnan has used the ESRF's structural biology beamlines to study how genetic information is translated by the ribosome to make proteins and the action of antibiotics on this process. There have been 60 presidents of the Royal Society since it was founded in 1660, including such figures as Isaac Newton, Humphry Davy and Ernest Rutherford.

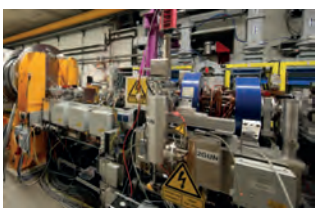
Beauty of science



Aperiodic structure: This image reveals the structure of an alkane-urea compound called n-dodecane/urea, a type of composite crystal that contains two or more subsystems with incommensurate lattice parameters. Such organic host–guest materials have an aperiodic structure and thus exhibit different degrees of freedom than those available in normal 3D crystals, giving rise to a unique range of temperature and pressure phases. The image, obtained by diffuse X-ray scattering at ESRF beamline ID23, arises from complex symmetries in n-dodecane/urea at low temperatures. The degree of freedom corresponding to sliding along the direction of the urea channel gives rise to monoclinic ordering for the alkane, resulting in six-fold flower shaped Bragg peaks. Understanding phase transitions in aperiodic crystals is of fundamental interest because most of the rules of solid-state physics are based on periodic systems. (Principal investigators: C Mariette, L Guérin, P Rabiller, B Toudic, Université de Rennes, France. Data collection and reduction: A Bosak and A Popov, ESRF.)

In the corridors

European XFEL injects electrons



The injector for the European XFEL in Hamburg, Germany, accelerated its first electrons at the end of 2015, marking a milestone in the development of the facility. This first stage of the superconducting particle accelerator, which has been under construction since 2013, produced a series of tightly packed bunches of electrons that passed through the 45 m-long injector beamline at near-light speed. When the facility is complete, the energy of the electrons will be increased along a 2 km-long linear accelerator and used to generate extremely short bursts of intense synchrotron X-ray light. DESY, which is the European XFEL's largest shareholder, is responsible for the construction and operation of the electron injector as well as the rest of the linear accelerator.

First light at MAX IV

Researchers at the MAX IV facility in Sweden have observed the first visible synchrotron radiation emitted by electrons passing through one of the machine's 140 bending magnets. The visible portion of the radiation, which was steered by mirrors to the outside of the shielding walls around the storage ring, appeared as a bright blueish glow through a silica window. In the coming months the MAX IV team will continue to tune the machine, further characterise the electron beam properties and install insertion devices. The official inauguration of MAX IV will take place on 21 June.

Synchrotrons shrunk to size

The world's first compact synchrotron for high-brilliance X-rays has been put into operation by researchers at the Technical University of Munich in Germany (TMU). The Munich Compact Light Source, measuring just 5 × 3 m^2, can generate intense X-rays and will form part of the Center for Advanced Laser Applications – a new €70 m joint research project between TMU and Ludwig Maximilian University that is due to open this year. The diminutive synchrotron, which generates X-rays by colliding a laser with high-speed electrons, was developed and installed by Lyncean Technologies – a private company founded in 2002 specifically to develop a compact X-ray source.

ESRF joins Instagram

In February, the ESRF joined Instagram, the mobile photo-sharing platform used by more than 300 million people worldwide. Among its first posts was this 25-year old photo of the 1991 User Meeting in full swing at the World Trade Center of Grenoble, when construction of the ESRF was just about to finish. Follow us at www.instagram.com/esrf_synchrotron/.

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Highlights

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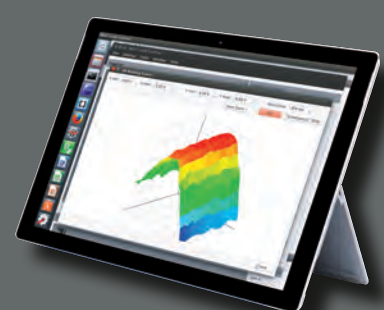

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