A LIGHT FOR SCIENCE

ESRF news

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A light for science


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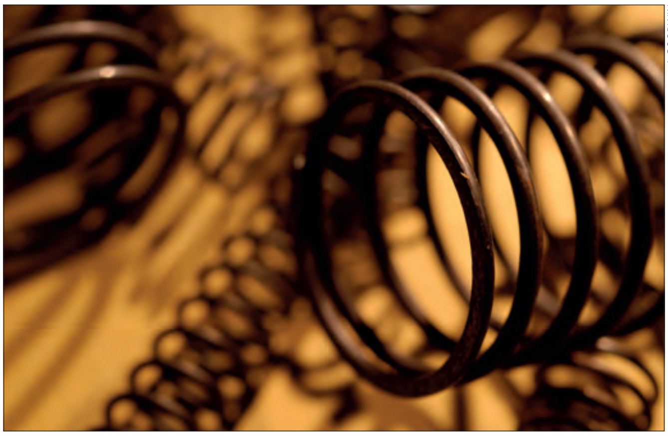
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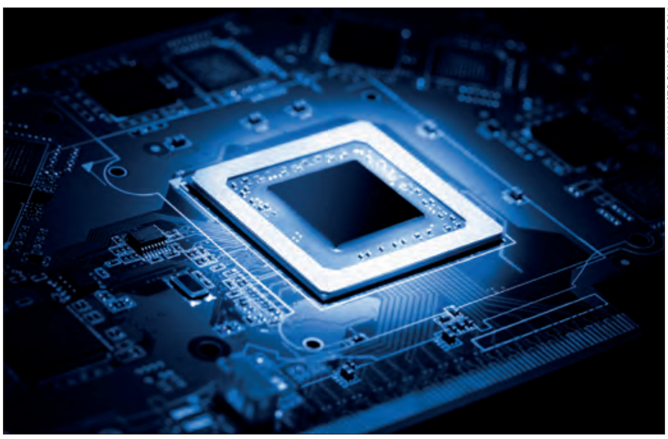
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Mastering materials

The development of existing technologies and the creation of new ones with reduced environmental impact rely on advanced materials with specifically tailored properties. A deep knowledge of the structure of materials is crucial for understanding material properties and, in the case of artificially engineered materials, for validating theoretical predictions.

Complementary to static structure determination is the study of the structure and morphology of materials during their synthesis or under realistic working conditions. These *in situ* studies provide fundamental information about different phases in a material's lifecycle – the steps that underpin its formation or production. Synchrotron-based techniques provide a deep understanding of each step at the atomic or molecular level, allowing researchers to optimise and control a material's overall performance. *In situ* X-ray studies also provide information about the intrinsic dynamics of a material's structure during mechanical or chemical degradation, offering ways to block or delay such processes.

In recent years, ESRF staff and users have developed numerous new techniques to resolve the structure of materials with superior spatial and temporal resolution. The coupling of synchrotron probes with more standard laboratory techniques allows precise control of sample conditions, making it possible to relate variations of a material's properties to modifications in its underlying structure.

The sample environment group at the ESRF has developed a broad range of instruments that allow users to alter the sample temperature from a few Kelvin to a few thousand Kelvin, or to employ magnetic fields in excess of 30T, for example. Mobile infrared spectrometers are now available for installation at beamlines, as are scanning probe microscopes that are compatible with the electrically and mechanically noisy environment of an X-ray beamline. These microscopes are not only able to image material surfaces *in situ*, but are valuable tools with which to align nanosized X-ray beams with nanoscale objects. They also allow users to apply a force on a nano-object in order to induce a mechanical response.

"Advanced materials will continue to take centre stage"

This issue of *ESRFnews* is dedicated to advanced materials. The examples illustrate only a small subset of the full range of materials studied at the ESRF and the experimental techniques on offer, but provide a snapshot of some of the latest advances in this important field. The commissioning of new facilities, such as the new electrochemistry and catalysis laboratories, bear witness to the ESRF's dedication to providing users with the best possible support. These particular facilities will provide tools for controlling processes occurring at the interface between a conducting solution and a conducting substrate, which are relevant for many technological applications such as batteries, fuel cells and photovoltaics.

With new and upgraded beamlines becoming available to the user community that target specifically the structure of materials down to the nanoscale, research on advanced materials will continue to take centre stage at the ESRF.

Roberto Felici, *ESRF head of structure of materials group*
Harald Reichert, *ESRF research director*

SESAME

CERN will provide the magnets for SESAME's storage ring.

EC backs SESAME

The European Commission is to contribute €5 m towards SESAME, the third-generation light source under construction in Jordan. The agreement allows CERN to work with SESAME researchers to develop magnets for the facility's storage ring, paving the way for first beams in 2015.

SESAME's injection systems are based on hardware donated from the BESSY source, but its storage ring will be completely new. "We are responsible for providing the magnets of the new storage ring, namely 16 bending magnets, 32 focusing quadrupoles, 32 defocusing quadrupoles, 64 sextupoles/correctors, plus spares," says CERN magnet engineer Attilio Milanese. "We've been working on this project with colleagues in Jordan since several months, so the technical work is well advanced." The magnet designs are now complete and CERN is ready to issue calls for tenders.

"SESAME is one of the most important projects in the world right now," said CERN director general Rolf Heuer, "with its close parallels to the origins of CERN, I am very happy that we are able to make this important contribution to the young laboratory's success."

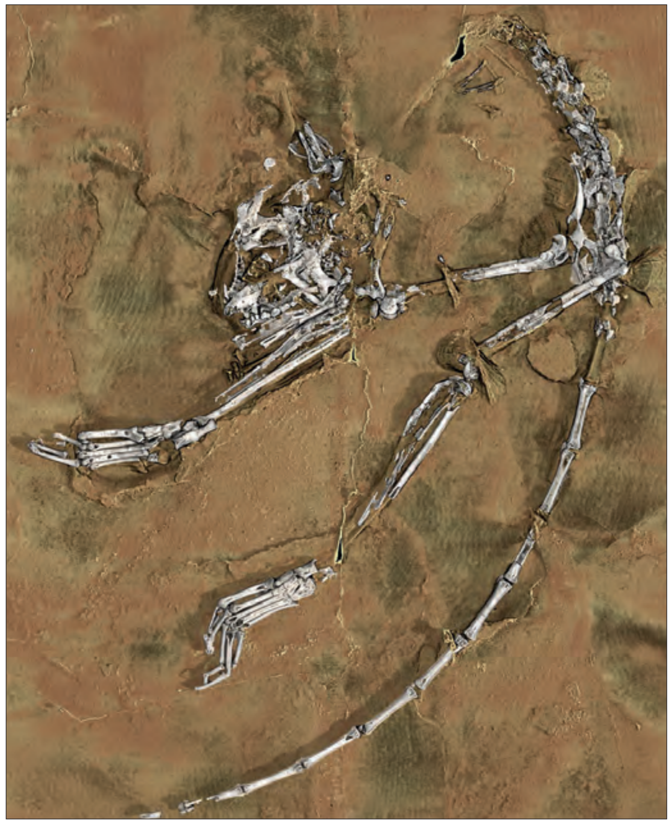
Oldest human and ape ancestor revealed

The earliest primate skeleton ever found has provided new insights into our evolutionary past, thanks to X-ray imaging at the ESRF. The 55 million-year old fossil, which represents a previously unknown genus and species *Archicebus Achilles*, is thought to have come from a pivotal "branch split" in primate and human evolution that put monkeys, apes and humans (the anthropoids) on a separate evolutionary lineage to tarsiers.

Archicebus differs radically from any other primate known to science, according to Christopher Beard of the Carnegie Museum of Natural History in Pittsburgh, one of the authors of the research. "It looks like an odd hybrid with the feet of a small monkey, the arms, legs and teeth of a very primitive primate, and a primitive skull bearing surprisingly small eyes."

Archicebus belongs to a separate branch of the primate evolutionary tree that is closer to the lineage leading to modern monkeys, apes and humans. Team leader Xijun Ni of the Chinese Academy of Sciences in Beijing says the result represents a big step forward in our efforts to chart the course of the earliest phases of primate and human evolution. "It will force us to rewrite how the anthropoid lineage evolved," he said.

The fossil was excavated by a local farmer from sedimentary rock strata in an ancient lakebed in central China's Hubei Province. Computed tomography scanning at the ESRF's ID17 beamline allowed the team to piece together the two separate parts and digitally reconstruct 3D images of the fragile creature (*Nature* **498** 60). Extrapolating from statistical analyses, *Archicebus* is thought to have weighed only about 20–30 g and be smaller than today's smallest living primates, overturning conventional wisdom.



ESRF/P TAFFOREAU

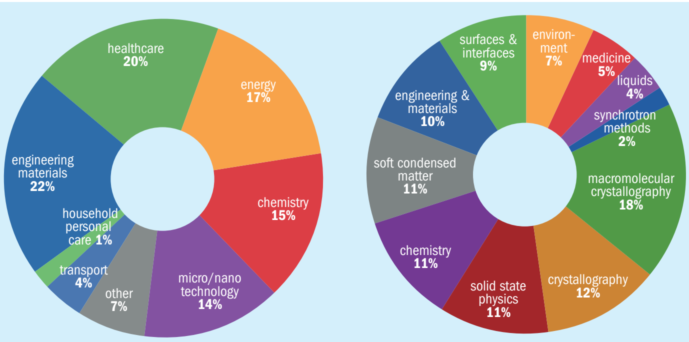
3D image of *Archicebus Achilles*, which is 7m years older than the oldest fossil primate skeletons previously known.

"For several years the ESRF has developed imaging facilities enabling us to non-destructively study fossils buried in rock with a level of detail and contrast unique in the world," says coauthor Paul Tafforeau of the ESRF X-ray imaging group. "We've been able to reveal microstructures that would normally require partial destruction of the specimens."

X-rays appeal to industry

Synchrotrons are making an increasing impact in industry, a survey of ESRF users undertaken in February reveals. Of 775 academic users who responded, 39% stated that the results of their experiments have applications in industrial R&D. Almost half said that their research group has direct links or collaborations with industrial R&D centres, while one third stated that their research benefits from industrial sponsorship.

For those participants who indicated a link with industry, engineering materials was the most common industry sector followed by healthcare, energy, chemistry and micro-/nano-technology. The majority of the ESRF's beamlines are involved in industry-linked research, and the ESRF actively encourages companies to apply for public-access beam time themselves and via academic partnerships.



Industry sectors represented by the ESRF survey (left) and the distribution of all science domains represented at the ESRF (right).

According to Tobias Schülli, scientist in charge of ID01 where more than 50% of experiments concern applied materials and devices, industrialists are attracted to the ESRF because of the superior level of detail that synchrotron X-rays can resolve compared to standard laboratory techniques. "We can, for example, map the mechanical strain within micrometre-sized electronic components to identify defects," says Schülli. "This eventually helps to fine-tune manufacturing processes."

I GINZBURG

A PSB technical platform.

Biologists celebrate decade of success

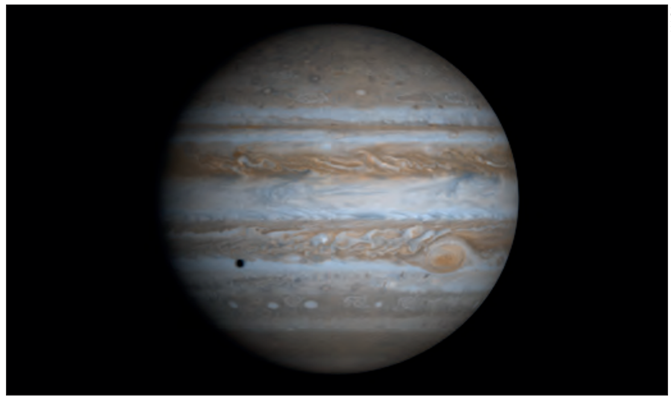
The discovery of the 3D structure of the flu virus polymerase has opened new possibilities for anti-flu drugs, and is just one example of the successes of the Grenoble Partnership for Structural Biology (PSB) during the past decade.

In 2003, the ESRF joined forces with nearby institutes EMBL and ILL along with three local research centres and universities to pool knowledge and equipment in structural biology.

On 4 June more than 150 specialists from across Europe gathered in Grenoble to mark the PSB's tenth anniversary. Providing unique tools ranging from sample expression and crystallisation to structure resolution and imaging, the PSB has helped drive the explosion in structural biology research in Grenoble in recent years.

Noble chemistry under pressure

NASA



Understanding the composition of Jupiter and other "gas giants" sheds light on planetary evolution in the solar system and beyond.

Noble gases tend to avoid chemical reactions because their outer electronic shells are fully occupied. At high pressures and temperatures, however, noble elements such as xenon exhibit rich chemistry that provides clues to the inner workings and evolution of planets.

Using laser-heated diamond anvil cells at the ESRF's ID27 beamline, Chrystèle Sanloup of the University of Edinburgh and co-workers have discovered that xenon reacts with water ice at pressures above 50 GPa and temperatures of 1500 K – conditions found in the interiors of Uranus and Neptune. *In situ* X-ray diffraction revealed the presence of a hexagonal lattice with four xenon atoms per unit cell and several possible distributions of oxygen atoms. The team combined the diffraction data with *ab initio* calculations in order to fully solve the crystallographic structure of the compound (*Phys. Rev. Lett.* in press).

"We wanted to see if pressure could be used as a variable to synthesise xenon compounds," explains Sanloup. "The field of noble gas chemistry has witnessed amazing advances in the last decade with over 100 xenon compounds synthesised, essentially by UV photolysis. But the pressure variable has seldom been used, with only a couple van der Waals compounds discovered so far."

The discovery of a pure xenon oxide phase extends the chemistry of xenon in natural conditions to pressures relevant for the giant planets, says Sanloup, and adds further evidence for the sequestration of noble gases at depth thought to explain the depletion of elements such as neon in Jupiter's atomsphere.

The team now plans to explore the reactivity of xenon with other materials at extreme conditions. "We have used the ESRF before to study the reactivity of xenon with silicon oxide to look for potential xenon phases stable at depth in the Earth that could explain the atmospheric deficiency in xenon," Sanloup told *ESRFnews*. "The next step will be to target other light oxides and to try to map the pressure-temperature-chemical space of xenon."

Users' corner

At the last proposal submission deadline on 1 March, 1011 new proposals were received. It marks another record number of proposals received per operational beamline.

The next deadline for submission of standard proposals is 1 September for beam time during March–July 2014. Proposers must use the most recent "Experiment Methods" template available on the user guide web pages and respect the two-page length limit. Review panels will reject proposals that do not respect the limit. Proposers should also ensure that they submit experiment reports for all relevant previous proposals.

To confirm the status of open ESRF beamlines for the 1 September deadline, proposers are invited to check here and on the Beamline Status web page. Users are also reminded to make sure that all new publications resulting from data collected either entirely or partially at ESRF are registered in our database via our easy-to-use interface: www.esrf.fr/UsersAndScience/Publications/publication-notification-form.

News from the beamlines

- **ID01** will be closed for users due to upgrade works from December 2013, with user operation to resume in November 2014. Depending on their requirements, users may consider ID13, ID03, ID10 or ID02 as alternative beamlines and should discuss with the respective beamline scientist before submitting proposals.
- Considerable progress has been made at the Swiss Norwegian Beamline station **BM01B** in allowing users to follow chemical and catalytic processes *in situ* using quasi-simultaneous XAFS, powder diffraction (PD) and Raman spectrometry. The recent monochromator upgrade allows a full XAFS scan to be taken in a few seconds, with similar durations for full high-resolution PD measurements thanks to a new 12 MP flat-panel CMOS. The system is fully integrated for user operation.
- **ID02** will close down to be upgraded in July 2013, with user operation expected to restart in Spring 2014. The new beamline with 30 m variable sample-detector separation will have high-resolution and ultra small-angle scattering capabilities. It will be available for the next call of proposals.
- **ID08,** which is being upgraded and moved to ID32, will be open for proposals for the XMCD branch in the coming September round. Partial user operation is expected in the first half of 2014, with the move towards full user operation (including the opening of the RIXS branch) for the March 2014 proposal round. Please contact beamline staff before submitting proposals. See p13 for a detailed report.
- The nano-imaging beamline **ID16A-NI** and nano-analysis beamline **ID16B-NA** will open for users during the scheduling period 2014-I (proposal deadline 1 September 2013). ID16A-NI will offer sub-50 nm beams with mild monochromaticity (1%), while ID16B-NA will offer a multi-modal approach designed to accommodate different micro-analytical techniques combined with X-ray imaging. See p12 for a detailed report.
- The high-resolution powder diffraction beamline **ID31** will close in December 2013 and will be moved to ID22. It will re-enter user service in April 2014 with an energy range extended beyond the current limit of 63 keV. Scientists wishing to submit proposals should remember to request beamline ID22 for proposals submitted from the 1 September deadline onwards.

The ESRF welcomes South Africa

South Africa has become the 20th country to join the ESRF, and researchers there are already planning new experiments.

C ARGOUD

ESRF director general Francesco Sette and secretary to the ESRF Council Itziar Echeverría tie the knot with Nithaya Chetty of the National Research Foundation of South Africa.

Scientists in South Africa have been involved with the ESRF since the beginning of user operation in 1994, when Trevor Derry of the University of Witwatersrand carried out the first synchrotron studies of diamond surfaces in collaboration with teams from the ESRF and the Netherlands. Derry hasn't returned to the ESRF since then but, like many researchers in South Africa, he now expects to take full advantage of the country's newly agreed membership.

On 21 May, South Africa representatives signed a medium-term arrangement that will enable the country to participate in the ESRF at a level of 0.3% for the next five years. "This agreement strengthens a relationship that has already produced numerous important results – notably in palaeontology, materials science and macromolecular crystallography – and builds ties between South Africa and the international synchrotron radiation community," said ESRF director general Francesco Sette. Nithaya Chetty, group executive for astronomy at South Africa's National Research Foundation (NRF) said the agreement was "a solid achievement that makes us extremely happy".

Proposal time

When physicist Giovanni Hearne of the University of Johannesburg undertook high-pressure experiments at the ESRF in 1999, he recalls having been so impressed by the facility's research capabilities that on returning home he immediately wrote to the then president of the NRF suggesting that South Africa seek ESRF membership. "Just over a decade later it seems that 'we have seen the light', which has involved the tireless efforts of our synchrotron research community," he told *ESRFnews*. "I will obviously try to exploit this wonderful opportunity."

Fellow University of Johannesburg physicist Bryan Doyle, who completed a post-doc at the ESRF at the turn of the millennium, is also planning to submit proposals in light of the new arrangement. "I do not think that the importance of us joining the ESRF can be overstated," he says. "It makes it much easier to convince people who have not yet discovered the enormous benefits of synchrotron radiation to become users."

Subterranean treasure

Diamonds (see box below) are not the only treasure lying beneath South African soil. In 2008 paleoanthropologist Lee Berger of the University of Witwatersrand uncovered a new species of early human ancestor in the Malapa Nature Reserve: *Australopithecus sediba*. In February 2010, he took the 1.9 million year-old-fossil to the ESRF, and the high-resolution X-ray scans at ID17 of the fossil's brain case enabled Berger and co-workers to shed light on the transition from *Autralopithecus* to *Homo*.

Sasol, the South African energy company based in Johannesburg, has a long-standing industrial partnership with the ESRF to synthesise hydrocarbons via the Fischer-Tropsch reaction. The ESRF allows Sasol scientists to study the reaction under realistic conditions to gain a deeper understanding of the company's proprietary catalysts.

Africa is the only continent that does not yet have a synchrotron, and the new agreement is part of a broader NRF plan to fund access to other synchrotrons worldwide. "For some years now, we have seen a new growth trajectory in science in South Africa," said Thomas Auf der Heyde of South Africa's Department of Science and Technology. "The agreement between the NRF and ESRF serves to further build human capital and research capacity for sustainable growth and development."

The new agreement is imperative if South Africa is to become internationally competitive across a broad spectrum of disciplines, adds Hearne. "Not only do we now have convenient access to one of Europe's most powerful research tools but it will likely lead to new collaborations with European colleagues and students," he told *ESRFnews*. "We are now limited only by our imagination."

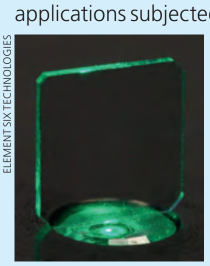
Matthew Chalmers

Diamond optics demand crystal perfection

Most materials either absorb or transmit X-rays, making it difficult to build components that manipulate X-ray beams. Silicon's high degree of crystal perfection has made it the material of choice, but diamond has several advantages including a higher heat conductivity, lower thermal expansion and lower absorption. Only synthetic materials made using high-pressure high-temperature techniques can achieve the necessary single crystalline quality (pictured).

An industrial partnership initiated between Element Six Technologies in Johannesburg, the University of Johannesburg and the ESRF over a decade ago was the first source of high-quality diamonds for X-ray optical elements, says physicist Simon Connell of University of Johannesburg. "The ESRF was certainly a very significant early player and has remained a key player in the development of diamond optical elements."

Lower quality diamonds have long been used as windows, filters and polarising elements, explains Connell. But it is only in the last five years or so that diffracting elements such as beam splitters and monochromators, especially those in imaging or coherent related applications, have been possible. "For applications subjected to the highest intensity radiation, in particular for X-FELS, diamonds may turn out to be the only solution," he says.



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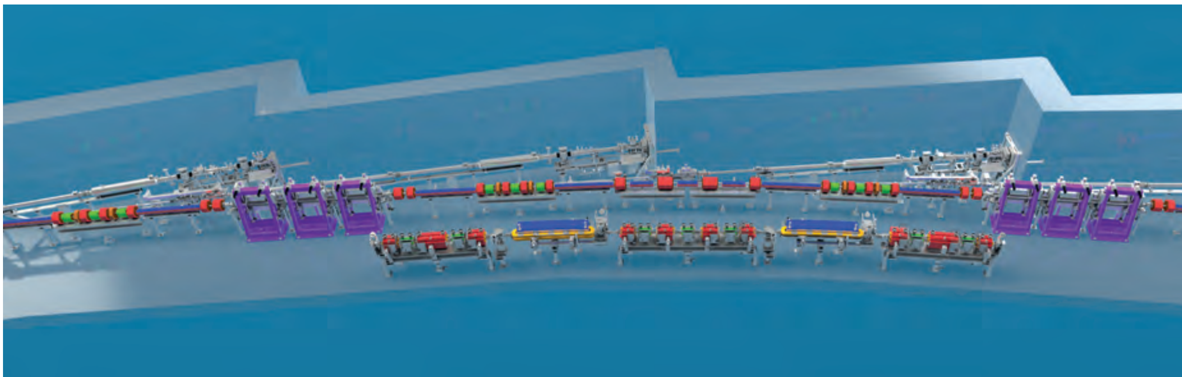

Machine upgrade pushes boundaries

With the technical design study for the ESRF Phase II upgrade underway, the Accelerator and Source Division faces the challenge of transforming a lattice concept into an operational machine, describes *Jean-Luc Revol* on behalf of the Phase II project team.

The ESRF is at an advanced stage of an ambitious upgrade. The first phase, based on new and improved beamlines and instrumentation, is two-thirds complete. The second phase, foreseen for 2015–2019, is designed around a new storage ring lattice that will reduce the horizontal spread of emittance of the ESRF's electron beam 4 nm to less than 0.2 nm – boosting the brilliance and coherence of its X-rays to values never before achieved at a synchrotron.

Much progress has already been made towards this goal. The ultra-low vertical emittance and orbit stability provided by the new beam-positioning electronics and the fast orbit feedback system, for instance, are fully compatible with the requirements of a next-generation storage ring. But a smaller horizontal emittance is a recurrent request from the ESRF beamlines: highly brilliant nanobeams allow users to extract maximal information about materials.

Since December last year, when the ESRF Council endorsed the management's proposal to launch the technical design study of the new machine lattice, members of the Accelerator and Source Division have been working hard to ensure that a lattice that works perfectly on a computer can be built, installed and operated reliably in practice.

Conceptual design

The new design maximises the use of existing infrastructure, with all insertion-device and bending-magnet beamlines being kept in addition to the present injector chain. The storage ring itself will be rebuilt from scratch while preserving its 32-cell periodicity, beamline positions and energy. Achieving this within the same tunnel puts very strong constraints on the lattice design and engineering.

The Phase II lattice – technically a "hybrid 7-bend achromat" – achieves its record low emittance thanks to an increased number of bending magnets (dipoles) and stronger focusing magnets (quadrupoles and sextupoles). It comprises 33 magnets per cell, which is about twice as many as are in the present machine, and the quadrupoles must have a very small magnet bore to achieve the necessary magnetic gradients. The magnetic design of the high-gradient quadrupoles is well advanced and prototypes will be built during the second half of 2013. Sextupoles and the combined dipole-quadrupole magnet are presently under study.

> **"We are working within very strong constraints."**

The dipoles in the existing storage ring are electromagnetic, but the ESRF's 20 years of experience with permanent magnets (for instance in insertion devices) allows us to design high-performance, compact permanent-magnet dipoles. Permanent magnets take up less space on the lattice, and lead to significant savings in the cost of operating the machine.

The magnet aperture defines the shape of the vacuum chamber, which is another major challenge presented by the Phase II machine. Since the new lattice will pack more magnets closer together, in practice we will have just 3–4 m of "drift space" per cell compared to 8 m presently. The smaller magnet bore means a narrower vacuum chamber, requiring distributed pumping, and additional pumping will be provided by lining the entire storage ring with non-evaporable getter coatings developed at the ESRF. The increased number of dipoles also means that we have to install more bending-magnet radiation absorbers along the beam path.

All this has to be achieved while also leaving space to extract the X-rays from the chamber. The strong dipoles in the existing lattice make it relatively easy to separate the light from the path of the electron beam, but the very small dipole angle of the new machine requires a much more complicated chamber design. Moreover, the lack of space restricts where we can install vacuum chamber hardware such as flanges, bellows and pumps and also the diagnostic equipment. We therefore expect to have to design and build more complicated chambers than those in the present machine.

Machine multitasking

These are just a few of the many exciting challenges involved in building the world's first ultimate storage ring – which has to be carried out while continuing actions from Phase I of the ESRF upgrade and, of course, while providing full access to users for experiments. Nine workflow packages have been identified to undertake the task. The ESRF's plans are based on pushing existing technologies to their limits, but R&D will also be undertaken into permanent-magnet designs, high-gradient quadrupoles and low-conductance vacuum chambers.

The technical design study will be complete by autumn next year. Should the green light for construction be given, the procurement and pre-assembly phase will begin in 2015 and the machine will then be stopped for approximately one year starting in August 2018 to install and commission the new ring. User access would begin late 2019, opening a new chapter in synchrotron science.

New halls ready for beamlines

Construction work on the Belledonne and Chartreuse experimental hall extensions (EX2 project) are complete. The two new halls were delivered in mid-June, one month behind schedule, with the laboratory and office building expected to be ready at the end of July. The inauguration of the new premises took place on 24 June as *ESRFnews* went to press.

An important milestone was reached at the end of April with the pouring of the final layer of the high-quality concrete slabs in the Belledonne and Chartreuse halls. This layer, made out of a special concrete mix and steel mesh, is 30 cm thick and required 3500 tons of concrete in total. Together with precise temperature control in the halls, the monolithic slabs will provide stable conditions for the new nano-beam capabilities of the ESRF Upgrade Programme.

"First measurements throughout May confirm that the slabs are within specifications concerning their planarity and vibration stability," says Rudolf Dimper, head of the ESRF's Technical Infrastructure Division. "The drying process appears to generate less shrinkage than expected and so far no micro-cracking could be observed. This is an important, successful and very promising milestone indeed."

The regionally funded CPER project (Contrat Project Etat Region) is also proceeding well. Completion of the new site entrance is scheduled for the end of the year, while the complete refurbishment of the site restaurant is underway and the first floor of the restaurant building is almost complete. The new chalet located next to the guesthouse is also finished and can be booked online for social events.

"All works for the restaurant extension will hopefully be finished this autumn, while the science building, under the supervision of the ILL, will be complete in the next few months," says Dimper. "The ESRF is looking forward to relocating important laboratories into this new building which, given the many common activities between the ILL and ESRF, will play a pivotal role on the site."

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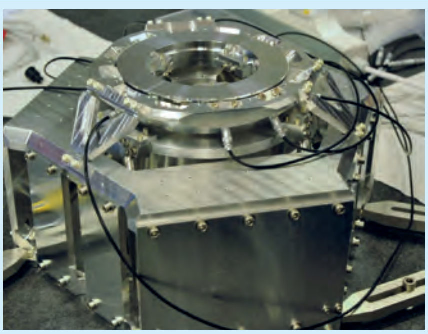
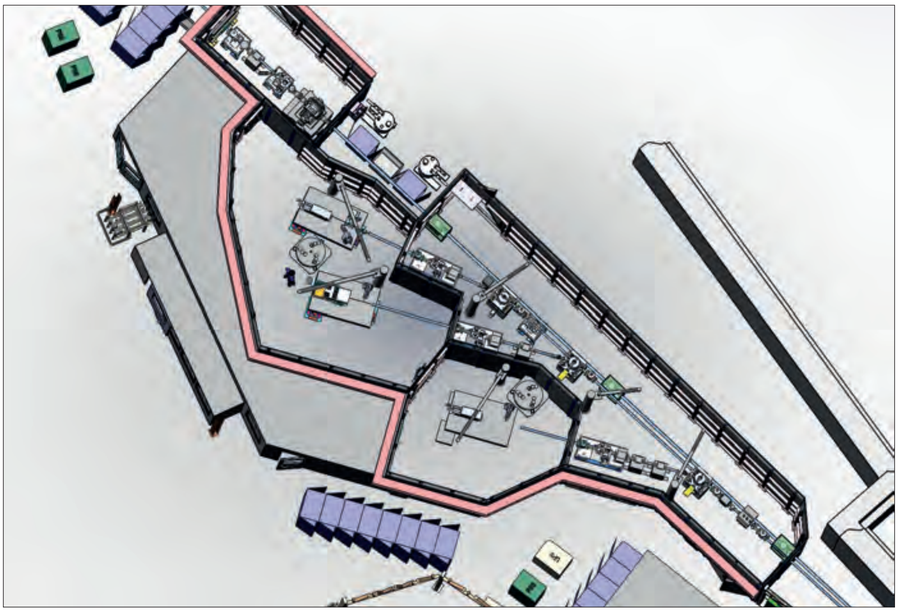
The exterior of the new laboratory and office building and the high-quality concrete slab of the Belledonne experimental hall extension.



ID16A's nano-positioning system, based on piezo-driven real-time correction of positioning errors, has been commissioned.

Nanoscience on track

The ESRF's nano-imaging and nano-analysis upgrade project, UPBL4 NINA, will provide high-brilliance beams focused down to nanometre sizes at two beamlines located in a satellite building 180 m from the source. The nano-imaging end station ID16A-NI, currently being commissioned, will open for user operation in spring 2014. ID16A-NI will offer beam dimensions of less than 50 nm with mild monochromaticity, allowing quantitative 3D characterisation of the morphology and the elemental composition of specimens in their native state by combining coherent imaging techniques and X-ray fluorescence analysis. Aiming at life science applications, the instrument will operate in a cryogenic environment. The final optics elements of the nano-analysis beamline ID16B-NA are being installed and the beamline will open for business in February 2014, offering users the ability to combine different micro-analytical techniques (such as X-ray fluorescence and X-ray absorption) with X-ray imaging. Based on the existing ID22NI end station, ID16B-NA will offer an improved lateral resolution, larger capability for *in situ* experiments and a monochromatic nano-beam that is tunable within a large energy range (5–70 keV).



The three fixed energy beamlines of MASSIF, also showing the ID30B optics hutch (top).

Structural biology suite takes shape

The ESRF's structural biology upgrade project will provide a state-of-the-art facility for studying the structure of proteins in solution (BM29 beamline) and a unique resource for macromolecular crystallography (MX) based on second-generation automation. The hub of the project – a new sample-evaluation and sorting facility MASSIF (massively automated sample selection integrated facility) – will allow crystals to be distributed to the best suited of seven end stations: MASSIF-1/-2/-3, ID23-1/-2, ID29 or ID30B. MASSIF will reduce the problem of inter- and intra-sample variations in modern MX experiments, allowing academic users and the pharmaceutical industry to evaluate 1000 samples per day per end station.

The first (BM29) of the end-stations that will make up the structural biology upgrade project was handed over to users in July 2012. Since then, this small-angle X-ray scattering facility has been in full operation while continuing to improve options for users, including a newly installed online high performance liquid chromatography (HPLC) set-up. All major optical elements have been installed and commissioned on the three fixed-energy beamlines for MASSIF, and the end stations for MASSIF-1 and MASSIF-3 are currently being equipped and commissioned. "First external user experiments at MASSIF-1 are planned for October 2013, while those at MASSIF-3 are planned for early 2014," says scientist in charge Christoph Müller-Dieckmann.

For MASSIF-1, ray-tracing studies indicate that users can expect a beam diameter of around 100 μm containing 5×10^{13} photons per second. This end station will be equipped with a robotic arm that will perform the dual roles of sample changer and goniometer, and will offer users advanced protocols for automated sample evaluation and data collection. MASSIF-3 is expected to produce a beam diameter of less than 10 μm containing 2×10^{13} photons per second. A new pixel detector recently ordered for this end station is expected to arrive in October 2013.

In parallel, construction of the tunable beamline ID30B, which will replace ID14-4, has begun in the new Chartreuse experimental hall extension. Radiation tests for the optics hutch are under way and delivery of the experimental hutch is due in December 2013, with first users expected from the middle of 2014.

ID32 branches out

An upgrade beamline for soft X-ray absorption spectroscopy and high-energy resolution resonant inelastic X-ray scattering is being constructed on ID32, replacing the ID08 beamline that is due to be closed in early October 2013. With sophisticated sample environments and tunable X-ray beam sizes ranging from microns to hundreds of microns for dichroism studies such as XMCD, together with a very high-performance resonant inelastic scattering instrument, the beamline will provide new facilities for users to study the electronic and magnetic properties of materials.

The new beamline will have two branches. One, providing advanced sample preparation facilities and state-of-the-art techniques for soft X-ray dichroism experiments, has at its core an ultra-high vacuum fast-scanning superconducting magnet. The magnet – which has a 9T field parallel to the X-ray beam that can be swept at a rate of 8T per minute and a 4T field orthogonal to the beam with a sweep rate of 2T per minute – has already been installed and successfully used for user experiments at ID08. This branch will be open for user proposals in the September 2013 round. Commissioning is scheduled to start in March 2014, allowing some experiments to be carried out before the 2014 summer shutdown.

The second branch at ID32 will provide users with very high-energy resolution for soft X-ray resonant inelastic scattering experiments. "This branch should be open for user projects for the March 2014 round, with experiments scheduled in the period September 2014 to February 2015," says scientist in charge Nick Brookes.

Phase II science update

Phase II of the ESRF Upgrade Programme, based around a new storage ring (see p11), will enable new experiments thanks to highly brilliant nanobeams with an unprecedented level of coherence. The outcomes of five workshops held during the 2013 Users' Meeting in February to discuss the science potential of the Phase II machine were presented to members of the ESRF's Science Advisory Committee on 30 May. The workshop summaries will be made available on the ESRF website.

ID20 enters operation

The upgraded ID20 beamline, which is dedicated to high-resolution inelastic X-ray scattering, was inaugurated on 30 May and users have since carried out the first experiments using the new RIXS spectrometer. This large state-of-the-art instrument is the centerpiece of one of ID20's two end stations. The other end station hosts an even larger Raman spectrometer (pictured), which will be operational in October 2013. Together, these tools offer unprecedented bulk information about samples.

"Beamline commissioning, in particular on the RIXS spectrometer, has been continuing during the last run with very good results," says ID20 engineer Keith Martel. "Assembly and installation of the Raman spectrometer is almost complete and should be commissioned with beam in July, after a number of planned user experiments on the RIXS spectrometer in June."

Right: ID20 scientist Laura Simonelli inside the new Raman spectrometer at the inauguration of the beamline on 30 May.

T TRENIT

Science *in situ*: the ESRF puts

From semiconductor chips to synthetic fuels, the ESRF offers users increasing scope to study the structure and morphology of materials during synthesis and under realistic working conditions.

Quantum computers, rather than process information via vast arrays of transistors, aim to manipulate the states of individual electrons. The semiconductor industry offers a promising route to such "qubits" based on coupled quantum dots, in which electrons are trapped in a quantum well within a semiconductor nanostructure. The properties of the dot are defined by the structure of the well and therefore depend strongly on the local atomic structure.

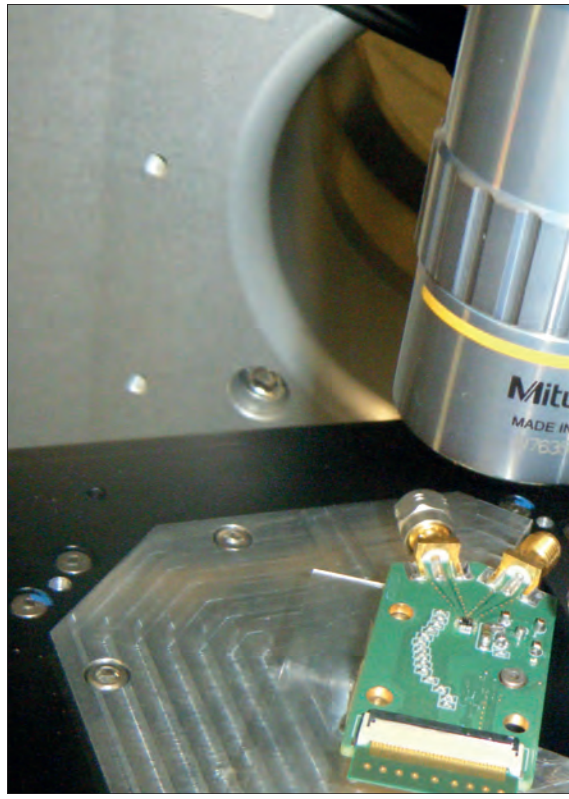
Until now, the role of structural defects such as atomic steps has been difficult to probe. But X-ray nanodiffraction at the ESRF has recently revealed structural information at length scales similar to the size of the confined electron wavefunction, allowing researchers to characterise the buried quantum well within an integrated quantum dot or other semiconductor device.

Paul Evans and colleagues at the University of Wisconsin-Madison in the US used nanodiffraction at the ESRF's ID01 beamline to study a fully processed chip built for a prototype quantum computer. Co-author Tobias Schülli, who is scientist in charge at ID01, says that there was no particular reason for choosing a quantum chip as opposed to a conventional one. "We were simply developing small-beam methods and were investigating structures that are supposed to be homogeneous," he says. "But nobody had ever checked the homogeneity at such scales, so we decided to look at a circuit board that the Madison group had in their laboratory."

The researchers were surprised at what they saw. Variations of just two atomic layers can be sufficient to ruin the electronic properties of a chip, and that's exactly the kind of detail Evans and co-workers were able to resolve directly. The results show that the structure of the silicon quantum well is influenced both by the epitaxial growth and subsequent metallisation processes used to produce the quantum dots (*Adv. Mater.* DOI: 10.1002/adma.201201833).

"The ESRF's strength is that we can look deep into buried structures without having to prepare the sample – we didn't even take the chip off the board," says Schülli. "Imperfections in 2D thin films are important and the fact that we can easily extract such parameters is very intriguing." One semiconductor fabrication company has already undertaken experiments at ID01 to exploit the technique, and another has expressed interest.

The work follows diffraction studies of a fully processed transistor carried out at ID01 (*Nano Letters* **11** 2875).



A prototype chip for a quantum computer in position at the E

These studies are not strictly *in operando* in the sense that the chips were functioning at the time that they were studied, says Schülli, but the technique is highly promising for allowing manufacturers to characterise the active regions buried deep within fully processed semiconductor devices.

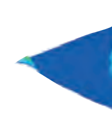
X-ray movie puts semi-solid casting in the spotlight

Semisolid casting allows complex components to be forged from a partially solidified metal slurry, and is promising for the production of lightweight metallic components. But semi-solid metals have unusual flow properties that until now have remained largely hidden. On shearing, for instance, they can switch from being an elastic solid to a viscous liquid.

To shed light on the underlying flow dynamics, Simon Zabler of the University of Würzburg in Germany together with colleagues from the ESRF and the Karlsruhe Institute of Technology injected a semi-solid aluminium germanium alloy into a thin channel while recording high-resolution radiographs at the ESRF's ID15 beamline. The very high photon flux density was sufficient to film the process at more than 500 frames per second, allowing researchers to observe for the first time single particle trajectories and a rapid break-up of solid skeletons (*Acta Mater.* **61** 1244).

The team developed a dedicated *in situ* setup to control the temperature and injection force of the semi-solid alloy as it was directed into a bottleneck channel. Advanced optical flow analysis allowed the researchers to track and to quantify particle and liquid motion separately, pictured (colour denotes the direction of the flow and brightness denotes its speed).

Compared to previous experiments carried out by the same group, the new results show a multitude of dynamic effects. "We believe that this is the first visual proof of the thixotropic break-up during semi-solid injection," concludes the team.



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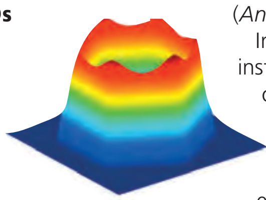
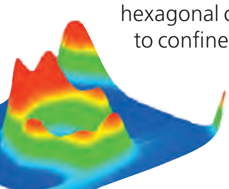
SRF's ID01 beamline.

Nanowires light up LEDs



Nanowires are also promising materials with which to exploit the counterintuitive behaviour of quantum mechanics. Compared to the planar semiconductor architecture of LEDs, nanowires are more "area effective" and can fit within the building blocks of nanophotonic devices such as light-emitting diodes (LEDs) and lasers, explains the ESRF's Gema Martínez-Criado. "Nanowires allow more sensitive control of parameters such as the geometry and composition of the quantum well."

Core–multishell nanowires with a hexagonal cross section are predicted to confine charge carriers in 2D at the hexagon corners – a potentially useful property for LEDs that until now had never been observed. Using energetic X-ray nanobeams at the ESRF's ID22 beamline, Martínez-Criado and co-workers imaged such confinement in gallium arsenide/indium gallium arsenide multi-quantum-well nanowires by combining optical luminescence with X-ray fluorescence spectroscopy (*Nano Letters* **12** 5829).



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"This allowed us to correlate directly the properties of the material with their function," says Martínez-Criado. "Our work represents a step toward not only the validation of theories of quantum confinement, but also the realisation of nanostructures with spectroscopic properties that could prove advantageous in nano-devices."

In situ catalysis

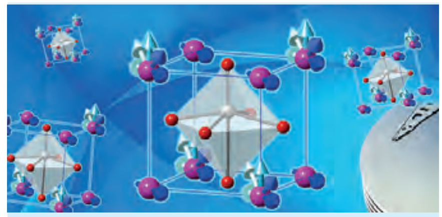
High-energy diffraction at the ESRF offers a powerful tool for industry to track processes inside a working chemical reactor. A better understanding of each step in reactions under industrial conditions can help chemical manufacturers improve efficiency and reduce costs.

Last year, users at ID15B undertook *in operando* studies of the zeolite catalyst SAPO-34 in a custom built mini-reactor. This material is key to the methanol-to-olefin process, whereby inexpensive feeds such as coal and biomass are converted into useful hydrocarbons, but the build-up of coke in its zeolite cavities causes the catalyst to deactivate. Real-time structural data allowed the team to correlate growth in size of the unit cell of the zeolite catalyst with its deactivation at different positions in the reactor. Diffraction patterns were collected every second as the reactor setup was moved through the beam (*Angew. Chem. Int. Ed.* **51** 7956).

In collaboration with several European institutes, ESRF researchers are also carrying out *in situ* studies of the catalytic reactions occurring in car exhausts, in which noble-metal nanoparticles convert unburned hydrocarbons and other exhaust gases into water, carbon dioxide and nitrogen. These studies are carried out at different levels ranging from model systems to the "real" reactor, explains Roberto Felici, scientist in charge at ID03. For example, experiments at ID03 have revealed that palladium nanoparticles under exposure to carbon monoxide form a carbide phase that may play an important role in the reaction. At ID15, the same phase has been observed in a real car exhaust.

"Nanoparticles exhibit a number of characteristics that are not present in bulk form, and these experiments give us the possibility to design nanoparticles with higher selectivity for a particular reaction and with a longer lifetime," says Felici. "The *in situ* catalysis experiments now being undertaken at the ESRF have never been done before, and they provide hugely important information about the numerous individual steps underpinning a working catalyst."

Matthew Chalmers



Multiferroic memory

The magnetically driven memory inside hard-drives is extremely robust, but it requires a lot of energy to write and erase data. Electrically driven "flash" memory consumes less energy, but currently is not as stable as magnetic memory and offers lower storage densities. Users of the ESRF's XMaS beamline (BM28) are turning to advanced multiferroic materials – which couple electric and magnetic properties – to develop more efficient data storage.

Philip Ryan of Argonne National Laboratory in the US and co-workers used the unique sample environment at XMaS to combine a high magnetic field and a high electric field at the extremely low temperatures at which europium titanate reveals dual magneto-electric behaviour. Nanometre-thin europium titanate was previously known to change from being a paraelectric antiferromagnet to a ferroelectric ferromagnet when stretched at temperatures close to absolute zero. Under strain, the two co-existing magnetic interactions compete, raising the possibility of switching between the two states by applying a small electric field.

Ryan and colleagues observed such a switch for the first time using X-ray resonant magnetic scattering, and could explain the underlying intrinsic mechanism using first principles calculations (*Nature Communications* **4** 1334).

"XMaS gives you the freedom to measure complicated diffraction geometries in these very specific environments that you can control and change," said Ryan. "From a potential application point of view the results here suggest that the key requirements for the valuable coupling of these two properties might not be as unique and rare as first thought. This can only increase the chances of being able to replicate them together at higher, more commercially appropriate temperatures."

The measurements were all done *in situ* with both electric and magnetic fields applied on the sample, adds XMaS beamline co-ordinator Laurence Bouchenoire. "Other examples of advanced materials research carried out at XMaS include studies of liquid crystals and organic photovoltaic devices."

Failure is not an option

Techniques developed at the ESRF allow engineers to study crack propagation in composite materials for next-generation aircraft engines and nuclear safety vessels.

In 2012 the European Aviation Safety Agency ordered the inspection of all Airbus A380 super-jumbos after fatigue cracks were discovered on the giant plane's wings. The cracks made headlines, but in fact cracks are present in all aircraft. A typical plane can contain thousands of them, mostly microscopic and detectable only with sophisticated eddy-current techniques.

"Few engineering structures can be guaranteed to be defect free, so the whole basis of aircraft safety is about working out the maximum size of the defects that we can tolerate," explains Phil Withers, director of the International Centre for Advanced Materials at the University of Manchester in the UK. Once a crack is detected, rigorous mathematical models and test data allow engineers to predict how long it will take for the crack to grow from being safe to producing a catastrophic failure. This tells airlines how often they must inspect an aircraft to assure its safety.

Novel composite materials such as titanium silicon carbide are lighter and can handle higher temperatures than traditional metals.

3D crack-tip microscopy

Crack propagation in metallic materials is well understood. But aircraft manufacturers are increasingly turning to more complicated composite materials that are lighter, stronger and can operate at higher temperatures. Lower weight reduces fuel consumption, while higher engine operating temperatures allow aero-engines to be more efficient. The challenge is to understand how cracks propagate in such materials. Electron microscopy reveals the surface features of micro-cracks, but synchrotron X-rays penetrate tens of millimetres into a sample where the behaviour of cracks can be very different.

"The wonderful thing about the ESRF is that it provides two pieces of information: imaging, to see how the crack grows, and diffraction, which tells us about the local stresses that it grows under," Withers explains. "ID15 is one of the few beamlines in the world where you can switch effortlessly between the two modes to form a kind of crack-tip microscope."

In conjunction with Rolls-Royce, Withers and co-workers have been using the ESRF since the early 1990s to investigate novel composite materials such as titanium reinforced with silicon carbide fibres. This material can operate at higher temperatures than titanium alone, making it a promising candidate for jet engine parts including new "bladed ring" designs. The A380 and Boeing's 787 Dreamliner already employ polymer-based composites in the fuselage frame and skin, but only metallic composites can handle the hostile conditions inside an aero-engine.

Composites typically contain three distinct phases: a metallic matrix, non-metallic fibres and the interface between them. In this system, cracks do not propagate in a planar manner, but instead divert along the interfaces. Provided the interfaces are not too strong the fibres themselves don't break, explains Withers. Once the crack goes past the fibres, they stop the crack from growing – just like extra stiches in a material stop it from tearing. "Understanding that sequence tells us how to engineer the material," he explains.

Withers and co-workers recently studied cracking in titanium-silicon-carbide at ID15. The ability to monitor cracks under load at high temperatures allowed the Manchester team to evaluate the potential of these materials under realistic conditions (*Proc. R. Soc. A* **468** 2722; *Acta Materialia* **60** 958).

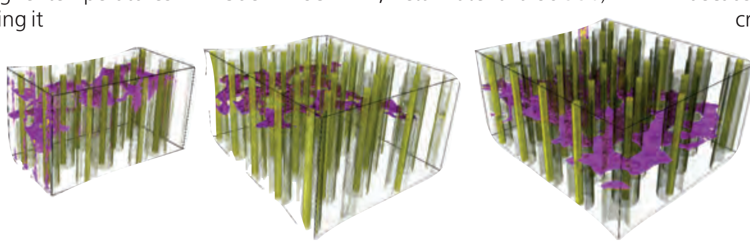
"I'd guess we're talking 15 years before such high-temperature composites could enter production," says Withers. In the search for tough ceramics that can operate at even higher temperatures the team is also looking to natural systems – namely seashells, which owe their toughness to multiple layers. Ceramics also show potential to be turned into self-healing materials that repair themselves upon damage.

Nuclear issue

Better knowledge of crack propagation transfers directly to other industries in which failure is unacceptable, says Withers, notably the nuclear industry. Here, the team exploits the unique power of ID15's X-rays to penetrate up to 30 mm of steel. Such measurements are used to support sophisticated model predictions in order to make advanced safety cases for reactor systems.

"Engineers are always looking for devious new ways to ensure that cracks don't grow or, if they do grow, to find new ways of stopping them," remarks Withers. "ID15 is so useful because it allows you to both see how the cracks progress but also to map the local stresses that causes them to grow. That allows you to make realistic estimates of the lifetime of existing components and to design safer, more crack-resistant materials for the future."

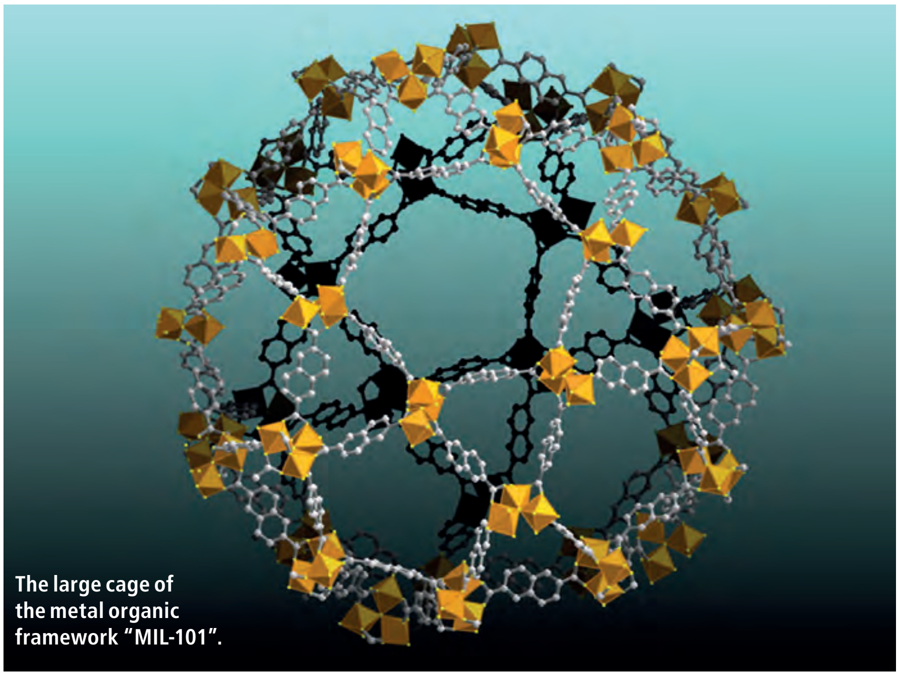
Matthew Chalmers



3D crack-tip microscopy shows a crack (purple) growing in a composite material containing silicon carbide fibres.

Molecular sponges flex their muscle

Metal organic frameworks have an enormous capacity to change shape in response to an external stimulus, with applications in gas storage and artificial muscle.



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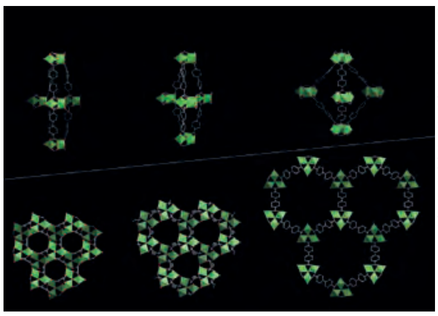
The large cage of the metal organic framework "MIL-101".



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Opposite: MIL-88 with diphenyl dicarboxylate in its various forms: as-synthesised dry solid (left), anhydrous (middle), and fully hydrated (right). The cell volume increases by more than 300% and is fully reversible from the dry to the hydrated forms.

Metal organic frameworks are the most porous materials known, exhibiting internal surface areas in excess of 7000 m^2 per gram. These molecular sponges, which comprise a metallic array joined by soft organic linkers, can expand and contract while preserving their single-crystal nature. "It's a bit like a wine rack," explains Vladimir Dmitriev of the ESRF's Swiss Norwegian beamline (SNBL). "It allows a material to absorb a lot of gases, and is relatively easy to synthesise."

The capacity of metal organic frameworks (MOFs) to hold large volumes of gas close to ambient conditions has enormous potential for fuel storage and waste-gas management. MOF pioneer and long-time ESRF user Gérard Férey of the Instiut Lavoisier at the University of Versailles in France says that there has been an exponential growth in this field since 2000, with thousands of new solids being discovered every year. "MOFs are truly multifunctional materials," he told *ESRFnews*. "They exhibit a lot of fantastic properties, such as mixed electronic-ionic conductivity, ferromagnetism and luminescence, for use in such things as storage, catalysis, encapsulation and drug delivery."

Synchrotron first

In the early 1990s Férey's group was among the first to use a synchrotron to study the hybrid materials. "The vast majority of researchers in this field are not so familiar with very large facilities, but I was convinced of the power of synchrotron radiation for studying structural evolution in real time under the action of a stimulus," he says. "In particular this allowed us to understand the capture of CO_2 at the molecular level during its introduction."

With the help of the ESRF, the Versailles team has developed numerous metal-organic 3D structures that can open or close in response to pressure, temperature, light or the influence of gases and solvents. One cubic metre of the material "MIL-101", for example, can trap nearly 400 cubic metres of carbon dioxide at 25 °C in pores measuring 3.4 nm across. In 2011, Patricia Horcajada at Versailles and co-workers carried out single-crystal microdiffraction of iron(III) terephthalate at the ESRF's ID13 beamline, revealing that the amount of swelling in this material can be tuned by changing the functional groups grafted onto the organic spacer – and paving the way for the use of MOFs for separation or drug-delivery (*J. Am. Chem. Soc.* **133** 17839).

Last year, Andrew Goodwin of the University of Oxford and co-workers used the ESRF's SNBL station to demonstrate record-breaking negative linear compressibility in a MOF-like material called zinc dicyanoaurate – a rare property that holds promise for artificial muscles, actuators and sensors. Normally, a material shrinks when pressure is applied all around it, but this particular MOF expands in one direction 10 times faster than the typical contraction observed in common engineering materials (*Nature Materials* **12** 212).

Biologically, explains Goodwin, muscles translate an electrical impulse into a mechanical contraction or expansion. But MOF chemistry shows researchers how to design materials that behave in a similar way in response to a different sort of stimulus, such as a change in temperature, pressure or the presence of a particular chemical species. "We're not really trying to engineer prosthetic limbs per se, but rather to translate the fundamental concept of muscular action onto the atomic scale for use in smart functional materials," Goodwin told *ESRFnews*. "Our work has really set a new benchmark in terms of what sort of mechanical response is possible in MOFs, and it shows how particular geometric motifs used in engineering and biology, such as atomic-scale springs and helices, can be used as design elements to create MOF-based materials with extreme properties."

Tricky experiments

MOFs represent a huge new family of promising solids. According to Férey, many companies are interested in his group's hybrid structures in the domain of molecular medicine, and chemical firm BASF is beginning to commercialise MOFs produced at the ton scale. "It's mainly for gas storage but the company now has the know-how to develop new applications, such as cars fuelled by natural gas stored in our aluminum-based MOFs," he says.

The quality of the ESRF's crystallography and its ability to provide *in situ* studies are what make the ESRF so appealing for MOF research. "These are tricky experiments," says Goodwin. "The X-ray intensity available at ESRF, together with the high precision of the diffractometers, is what really allows us to build up a picture of how the materials function."

Matthew Chalmers

Eyeing up tomorrow's superconductors

CERN researchers have teamed up with the ESRF to study superconductors that will enable future high-field magnets for particle accelerators.

The Large Hadron Collider (LHC) at CERN, Switzerland, is the most powerful particle collider ever built. It was designed to circulate 7 TeV beams of protons in opposite directions around a 27 km circumference ring and bring them into collision at four points. Last year, by sifting through the debris of trillions of such collisions, physicists discovered a new elementary particle – the Higgs boson.

None of this would have been possible without superconductors. The LHC's 1232 dipole magnets have to carry currents in excess of 10 kA to produce magnetic fields strong enough to bend the high-energy protons around the ring. The feat is achieved using niobium-titanium alloy superconducting cables that lose their electrical resistance when cooled to –264 °C using liquid helium.

In the quest to delve even deeper into the structure of matter, the LHC is due to be upgraded in the coming years. A luminosity upgrade scheduled for the end of the decade would involve replacing the focusing magnets close to the LHC's collision regions with stronger ones, boosting the collision rate. A more audacious energy upgrade, potentially taking place in the 2030s, would require all of the LHC's bending magnets to be replaced with more powerful ones. To help improve the superconducting cables required for the new magnet technology, CERN has hooked up with researchers at the ESRF.

"Static tomography of superconducting wires with reduced size at the BESSY synchrotron demonstrated the potential of the technique, but we also realised that we needed higher X-ray energies to study the strongly absorbing samples," says Christian Scheuerlein of CERN's Technology Department. "Eventually I came into contact with Marco Di Michiel at ID15, who made the first state-of-the-art micro-computed tomography images of superconducting wires."

Fine structure

To the naked eye the LHC's superconducting cables look like any other conductor. In fact they comprise around 8000 extremely fine niobium-titanium filaments. This alloy is malleable and can be drawn to final size, but superconductors envisaged for more powerful accelerator magnets, such as Nb_3Sn and Bi-2212, are brittle. Usually the brittle superconducting phase is obtained from precursor materials (a powder, for instance) during heat treatment of the magnet coil. Phase changes and porosity formation during the processing stage can degrade the microstructural and micro-chemical homogeneity, and thus performance, of the finished superconductor.

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CERN

Top: 3D visualisation of the porosity formed during the processing of a niobium tin superconductor. Bottom: Advanced superconducting cables power machines such as CERN's Large Hadron Collider.

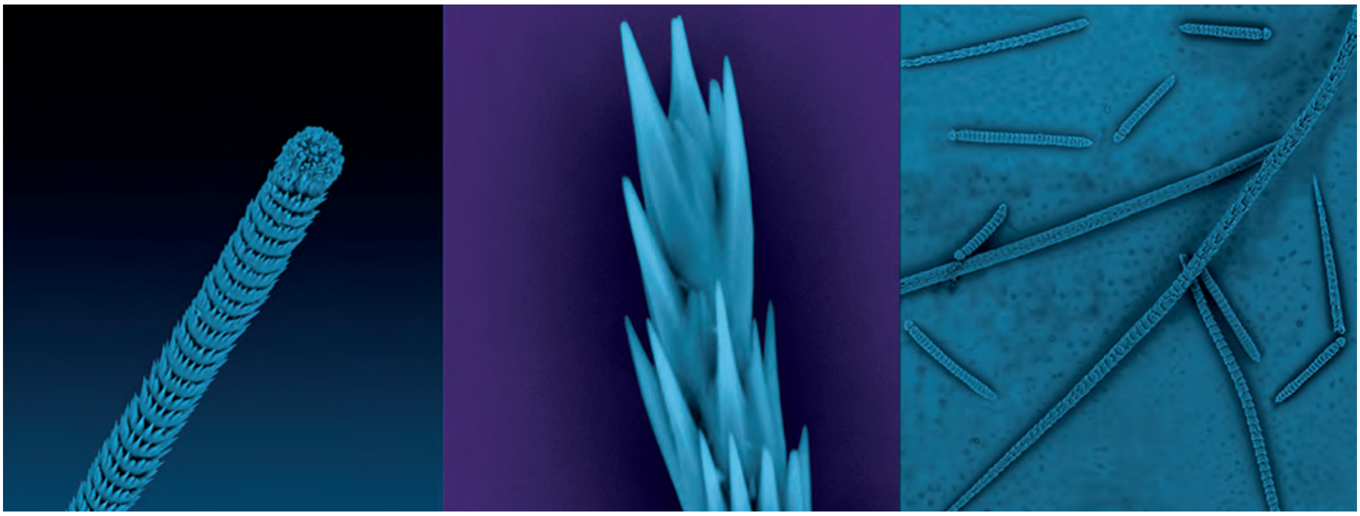
The ESRF's high-intensity beams provide unique opportunities to study superconducting materials and processing techniques. Scheuerlein and colleagues submitted their first proposal to the ESRF in 2006: an *in situ* study of void growth during the processing of niobium tin superconductors using fast micro-computed tomography (CT) and X-ray diffraction.

Synchrotron advantage

The main appeal of high-energy synchrotron beamlines, explains Scheuerlein, is the possibility to characterise the sample inside a furnace or in other auxiliary equipment such as cryostats or tensile rigs, and to allow observations of the inside of the superconductor with good spatial and temporal resolution. "Some superconducting samples are notoriously difficult to prepare destructively, for instance for microscopic studies," he says. "With non-destructive micro-CT we can be sure that the microstructural features that are visualized are not sample-preparation artefacts."

One of the group's most important micro-CT results at ID15 concerned the high-temperature "cuprate" superconductor Bi-2212. "The *in situ* studies of the porosity formation during the processing of Bi-2212/Ag wires carried out at the ESRF in collaboration with researchers at the National High Field Magnet Laboratory in Florida, US, have helped us better understand how the porosity, which can block the current, redistributes during the entire heat treatment cycle in this very promising superconductor," says Scheuerlein. "Controlling the porosity formation allows us to drastically increase the critical current density."

The CERN/ESRF team plans to continue its *in situ* studies of the processing and degradation of different superconducting composites, using fast micro-CT and X-ray diffraction as well as newer methods such as X-ray diffraction tomography. One target, explains Scheuerlein, is the influence of the process gas on the phase evolution during the processing of Bi-2212 superconductors, while another aim is to investigate the degradation of superconductors under operating conditions. "At ID19 we want to study in more detail the influence of the very complex Bi-2212 micro-structure on the current flow through Bi-2212/Ag composites by acquiring static tomograms and subsequent quantitative analysis." The ESRF's high-energy beams also stand to boost the team's understanding of the processing and degradation mechanisms in advanced superconductors such as MgB_2 and YBCO.
Matthew Chalmers



Spicules from the simple sea creature *Herdmania momus* contain large single crystals vaterite of higher quality than those in the synthetic vaterite used in previous structure determinations.

Sea squirt solves crystal conundrum

The ESRF has helped crack the crystal structure of vaterite, a rare form of calcium carbonate that has perplexed scientists for almost a century.

Calcium carbonate is one of nature's most abundant raw materials, much of it formed from the remains of microscopic sea creatures. It comes in three anhydrous crystalline polymorphs. Calcite, which has a trigonal symmetry, is the most stable and abundant form. Aragonite, shown to be orthorhombic by none other than William Bragg a century ago, is metastable and transforms into calcite on geological timescales. The third form, vaterite, named after German mineralogist Heinrich Vater, is the rarest and least stable form of anhydrous calcium carbonate.

Vaterite is an important constituent of cement, where the addition of water transforms it into calcite, and also in the paper industry where it is used as a filler to provide colour or texture. Unlike the other calcium carbonate polymorphs, however, the crystal structure vaterite has eluded researchers for almost a century.

Although vaterite has been found during oilfield drilling, in gallstones and even in a meteorite, geological vaterite is rare and unstable. The synthetic version, a powder, yields only nanosized crystal grains. Biogenic vaterite is produced as a minority component in green turtle eggshells, freshwater pearls and the scar tissue deposited by mollusks to repair their shells. But one creature in particular, a solitary filter-feeding marine invertebrate called *Herdmania momus*, has a body and tunic spicules made entirely of pure vaterite.

Now, thanks to X-ray powder diffraction at the ESRF and high-resolution transmission electron microscopy, this humble "sea squirt" has given up vaterite's structure – with unexpected results. The data show that vaterite does not have one crystal form but two different structures that coexist within a pseudo-single crystal (*Science* **340** 454). "We never envisaged this scenario," explains Boaz Pokroy of the Technion-Israel Institute of Technology, who led the study. "It was a total surprise, but at the same time it made so much sense knowing the years of conflicting results from different groups publishing on the structure of vaterite."

Structural riddle

Researchers thought they had solved vaterite's structure as long ago as 1925, when X-ray diffraction revealed hexagonal symmetries that made it distinct from calcite or aragonite. As better tools came along, including Raman spectroscopy, this picture needed to be refined. In the 1950s vaterite was linked to a unit cell with pseudo–hexagonal-orthorhombic symmetry, but in 1963 researchers reported eight weak reflections that were attributed to a superstructure rotated 30° from the main hexagonal one. Since then, the debate about vaterite's structure has centered on whether its symmetry is hexagonal or orthorhombic, with researchers unable to reconcile diffraction data with optical spectroscopy and also with density functional theory. "In latter years there have been more attempts to solve the structure of vaterite within the concept that it is a single structure," says Pokroy.

In 2011 Pokroy and co-workers grinded a multitude of *Herdmania momus* spicules obtained from the Mediterranean and the Red Sea into a fine powder and placed it in the ESRF's ID31 beamline. None of the previously reported vaterite structures could fit the high-resolution diffraction spectrum, they found. While a hexagonal structure gave the best fit to the data, says Pokroy, it did not explain all the observed diffraction peaks. "ID31 really proved that all the structures were simply wrong – our results change the concept of vaterite."

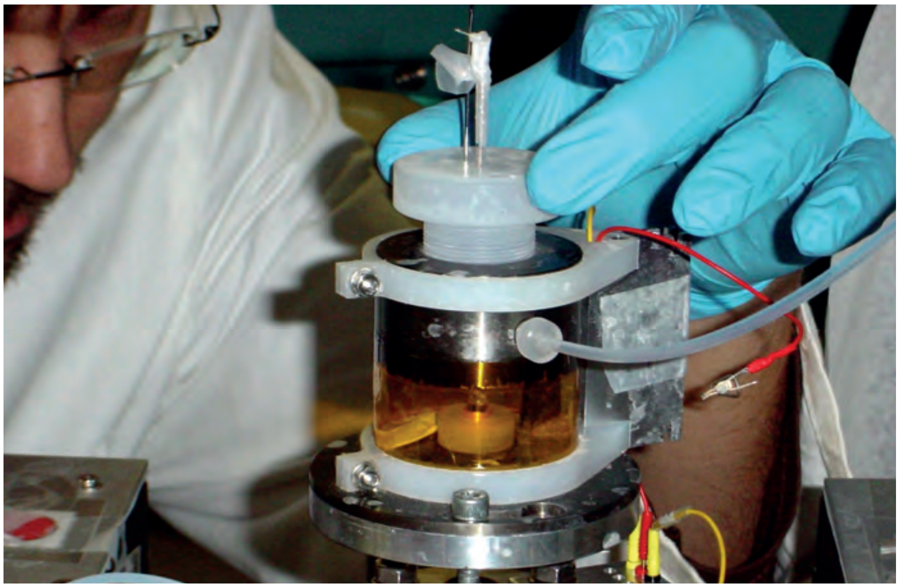
To ensure that the data had not come from other structures, such as aragonite or calcite, the team used aberration-corrected high-resolution transmission electron microscopy at the Technion to examine very small volumes of vaterite. This confirmed its dual-crystal structure, but the precise nature of the second structure – which is less abundant than the larger hexagonal form and visible only in certain orientations – remains unknown.

This year, the team intends to remedy that situation using diffuse scattering at the ESRF's ID13 beamline in collaboration with Christian Riekel. The microfocussing capabilities of this beamline allow a single *Herdmania momus* spicule to be studied, with higher statistics allowing the team to map the diffraction spots coming from the second crystal. "Although this study is a basic scientific work to address a 100-year-old conundrum, it could also tell us how vaterite forms in the process of biomineralisation," says Pokroy. "And if we understand that then it might give us some insight into how to stabilise other metastable forms of calcium carbonate to make them useful."

Matthew Chalmers

Smooth deposits aid chips

Anomalous copper deposition revealed at the ESRF will help engineers tailor high-tech production processes for semiconductor microchips.



J STETTNER

Mounting a copper sample in the ESRF's ID32 beamline.

Copper's superior electrical conductivity makes it the material of choice for electrical contacts. Not only can copper be drawn easily into wires but by discharging copper ions from solution under an applied voltage (a process called electrodeposition) copper can be fashioned into ultra-small connections for chips and circuit boards.

The relentless miniaturisation of electronics demands reliable electrical interconnects at the nanoscale, which requires precise control of the electrochemical deposition processes and hence an improved understanding of the underlying physics and chemistry. The smallest features on today's chips can be as little as 100 atoms wide, so the slightest defect can severely degrade or cause a loss of the electrical contact.

In contrast to copper epitaxial growth under vacuum conditions, however, a fundamental understanding of electrodeposition from solution under high voltages is largely lacking. Specifically, the influence of the solid-liquid interface and the applied electrode potential on the elementary surface processes is unclear. Industrial plating processes have long used small amounts of chloride in the acidic plating bath to optimise growth behaviour and deposition properties, explains ESRF user Olaf Magnussen of Kiel University in Germany, but until now it has been a mystery how this simple inorganic species influences copper growth at the atomic scale.

Growing on chloride

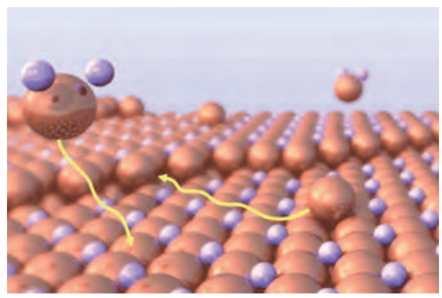
Last year, in collaboration with ESRF staff, Magnussen and co-workers used surface X-ray diffraction at ID32 to carry out *in operando* studies of electrochemical copper growth in a chloride-containing acidic solution. They found that minute quantities of chloride in the electrolyte solution have a decisive influence on the structure of the growing films, which in turn determines their electrical behaviour (*Phys. Rev. Lett.* **108** 256101).

The experiments were performed using an electrochemical three-electrode cell in which contact with the electrode was established by a droplet of electrolyte. The high-speed surface scattering setup at ID32 allowed the researchers to study copper growth at a rate of several monolayers per second, shedding light on how atoms attach to the growing surface during electrodeposition. By increasing the voltage the team found that the copper films grew smoother, implying reduced defects and cavities in the deposits. "This actually was a big surprise," explains Magnussen. "Other metals, for example gold, grow rougher at higher voltages and this is also what current theory predicts."

The anomalous behaviour of copper, the team concludes, is due to the arrangement of the atomic layer of chloride ions present on the surface. Copper atoms moving across the surface have to push through this layer and they become more mobile at higher voltages, where the chloride is more weakly bound. "We suspect that it is due to the electrostatic energy resulting from the interaction of the charge chloride absorbates with the strong electric field at the electrochemical interface," says Magnussen.

The novel concept of a voltage dependent surface mobility mediated by absorbed adlayers, he explains, provides a simple conceptual framework that allows microscopic physical growth theories to be extended to liquid phase deposition processes. The results contribute to a better fundamental understanding of growth and surface transport in electrochemical environments, and have significant practical implications, according to the team. For instance, knowledge about whether the electrode surface transport is normal or anomalous allows manufacturers to assess the roughness of the deposit that may be expected at certain deposition potentials.

"Our results have significant practical implications."



J GOLKS

Graphical representation of the growth process, during which deposited copper atoms move across the surface through a layer of chloride.

"Choride is the simplest additive and real industrial additives are more complicated," says Magnussen. "Our work is still at the very fundamental level but it provides insight into how these additives work." The ESRF is important, he adds, because it allows researchers to study deposition at high rates *in operando*, approaching conditions in industry. "We want to extend synchrotron techniques to get the full 3D structures of the additives. Such techniques may also be useful in other areas such as fuel cells."

Matthew Chalmers

Seeking the bigger picture

Senior advisor at the Research Council of Norway and chair of the ESRF's Administrative and Finance Committee **Aase Marie Hundere** makes the most of her research experience in advanced materials.

Aase Marie Hundere's career has spanned academia, industry and, in her current role, science policy. But one thing has remained constant: an interest in advanced materials. Aase's PhD research in the early 1990s concerned the properties of ceramics such as aluminium nitride, specifically how its thermal conductivity changes due to different production processes. It was a natural step from her undergraduate degree in inorganic chemistry, she explains. "I was interested in the links between the fundamental properties of materials and how they are synthesised."

On finishing her PhD, Aase became a research scientist for Elkem – one of Norway's largest companies – where she focused on silicon- and aluminium-based materials for lining the furnaces used in large-scale metal production. Metal production is one of Norway's biggest industries, along with energy and fisheries, and materials science is one of the largest areas of scientific research in the country.

"Going from the laboratory where you focus very much on the details into more applied settings was very interesting," she recalls. "I came from a family of engineers and I wanted to go from the academic world to something more practical and relevant."

Seeking a broader overview of research, in 2003 Aase took up a position as an advisor to the Research Council of Norway, which had just launched a new programme in nanotechnology and advanced materials. "Also, there was a lot of travel in my previous job and I wanted to start a family," she says. "Norway is very good at supporting you while you have small children, but you still are compared internationally so I think it's tough to be a woman in science."

C ARGOUD

Aase Marie Hundere in brief

Born: Norway, 1966.

Education: PhD materials science, Norwegian University of Science and Technology, 1994.

Career: 1995–2003 Research scientist, Elkem; 2003–present Senior advisor at the Research Council of Norway.

Family: Married with two children.

Interests: Skiing, running and walking the dog.

> **"Long-term challenges in industry can be very stimulating for academia."**

Representing Norway

Since 2005 Aase has represented Norway at the ESRF and is the current chair of the ESRF's Administrative and Finance Committee (AFC). The task of the AFC is to make recommendations to the ESRF Council based on budgetary and economic considerations. Norway participates at the ESRF via the "Nordsync" consortium, which contributes 4% of the total ESRF budget and also includes Finland, Denmark and Sweden. The country also has a 2.5% stake in the European Spallation Source currently under construction in Sweden and a similar stake at CERN.

The ESRF's Swiss-Norwegian beamline (SNBL) is funded separately, and Aase has also represented the research council on the board of the SNBL. The SNBL has become a "home laboratory" to Norwegian synchrotron users where they can build relationships and equipment with local contacts, she explains. "It's a very strategic way to carry out research."

Some of Norway's 140 or so synchrotron users travel to the nearby MAX-lab in Sweden to carry out experiments in areas such as surface science. Will more use be made of the new MAX-IV machine when it comes online?

"Of course MAX-IV will cover a larger part of synchrotron science, and it is much closer than Grenoble, but Norway's commitment to the ESRF and the SNBL will continue," says Aase. "It's also very important for Norwegian researchers to collaborate in an international environment, because we find that researchers who do so are more successful."

Materials for industry

Last year, Norway's research council launched a new 10-year-long programme in nanotechnology and advanced materials, NANO2021, which is Aase's main responsibility for the coming years. One of its goals is to stimulate Norwegian industry to apply the knowledge that is built up in universities in new areas, which is something Aase is looking forward to focusing on. "Also, given the new infrastructure provided by the ESRF upgrade, I want to understand how we can take full advantage to make industry more aware."

At the end of the day, she says, research funding needs public acceptance. "It's a balance with blue sky research, but we also know that long-term challenges in industry can be very stimulating for academia."

Matthew Chalmers

ESRF to boost European nanoelectronics

A major new public–private programme funded by the French government will further open up the ESRF to companies developing advanced micro- and nano-electronics.

Today's microprocessors pack billions of transistors into a piece of silicon roughly the size of a postage stamp. This remarkable feat is possible thanks to complementary metal–oxide–semiconductor (CMOS) and silicon-on-insulator (SOI) techniques. But chip manufacturers need to push advanced lithography down to the atomic scale if they are to continue the relentless miniaturisation of transistors, which demands new technologies.

In April 2012, the French National Research Agency and the CEA established an eight-year long €460 m public–private programme called Institut de Recherche Technologique (IRT) "Nanoelec" to develop 3D assembly for increasingly complex chips, and silicon nanophotonics for faster communication within and between chips. IRT Nanoelec is one of seven major programmes established in France to leverage high-tech industry through public and private sector research. The ESRF together with the Institut Laue-Langevin (ILL) and the CEA are to share €6.5 m to help industry meet these goals.

The aim is to make it as simple as possible for companies to access beam time at large facilities. Based on experiments carried out by industrial partners and the ESRF, the programme will define preparation and characterisation processes for specific materials and devices. "The ESRF allows characterisation of microelectronic devices but it's still difficult for enterprises to access the ESRF," says Michel Wolny of the CEA and Director of IRT Nanoelec. "IRT will help the ESRF to open its capabilities to industry thanks to specific investments and networking among the 17 IRT partners."

Some partners of the IRT Nanoelec initiative

- *Manufacturing companies* – STMicroelectronics, Mentor Graphics, Schneider Electric, Soitec, Bouygues, Presto Engineering and Ineo.
- *Public bodies* – CNRS, Inria, Université Joseph Fourier, Grenoble INP and Grenoble Ecole de Management.
- *Research institutes* – ESRF and ILL.
- The Minalogic cluster.

"This is an important project for us"

Industry on board

IRT Nanoelec partner Soitec uses X-ray diffraction and reflectivity techniques to characterise materials, but until now this has mostly been carried out using conventional diffractometers, explains IRT project co-ordinator for Soitec, Yves-Matthieu Le Vaillant. "The use of the synchrotron allows intense radiation and focused beams, resulting in a better accuracy at the sub-micron lateral scale," he told *ESRFnews*. "It will allow unique observations of amorphous silicon dioxide layers and possibly III-V semiconductors for the solar industry."

For 3D integration of chips, explains laboratory manager Nadine Bicaïs of STMicroelectronics, the ESRF provides a unique tool to characterise large-area silicon wafers. "Thanks to the high brilliance provided by the ESRF, X-ray based techniques allow us to observe large embedded features while maintaining state-of-the-art resolution."

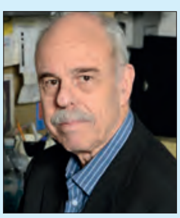
"This is an important project for us," says Ed Mitchell of the ESRF's Business Development Office. "Once we gain experience and expertise in working with these firms we can apply it to companies across Europe, thus extending the benefits to all ESRF funding states."

Matthew Chalmers

Movers and shakers

User awards

Metin Tolan of TU Dortmund University has won the German Research Foundation's 2013 Communicator Award for his innovative approaches to communicating physics. Tolan, who has carried out thin-film experiments at the ESRF's ID10 beamline since 1998 and is also director of the Delta electron storage ring at the Dortmund Synchrotron Radiation Centre, beat 48 others to the €50,000 award. To help bring X-rays to life, he often appeals to James Bond movies such as *Moonraker* and *The World is Not Enough*, he told *ESRFnews*. "I calculated how intense the X-ray sources must be in order to achieve the results shown [such as seeing through walls], and concluded that they could only be produced by a synchrotron."

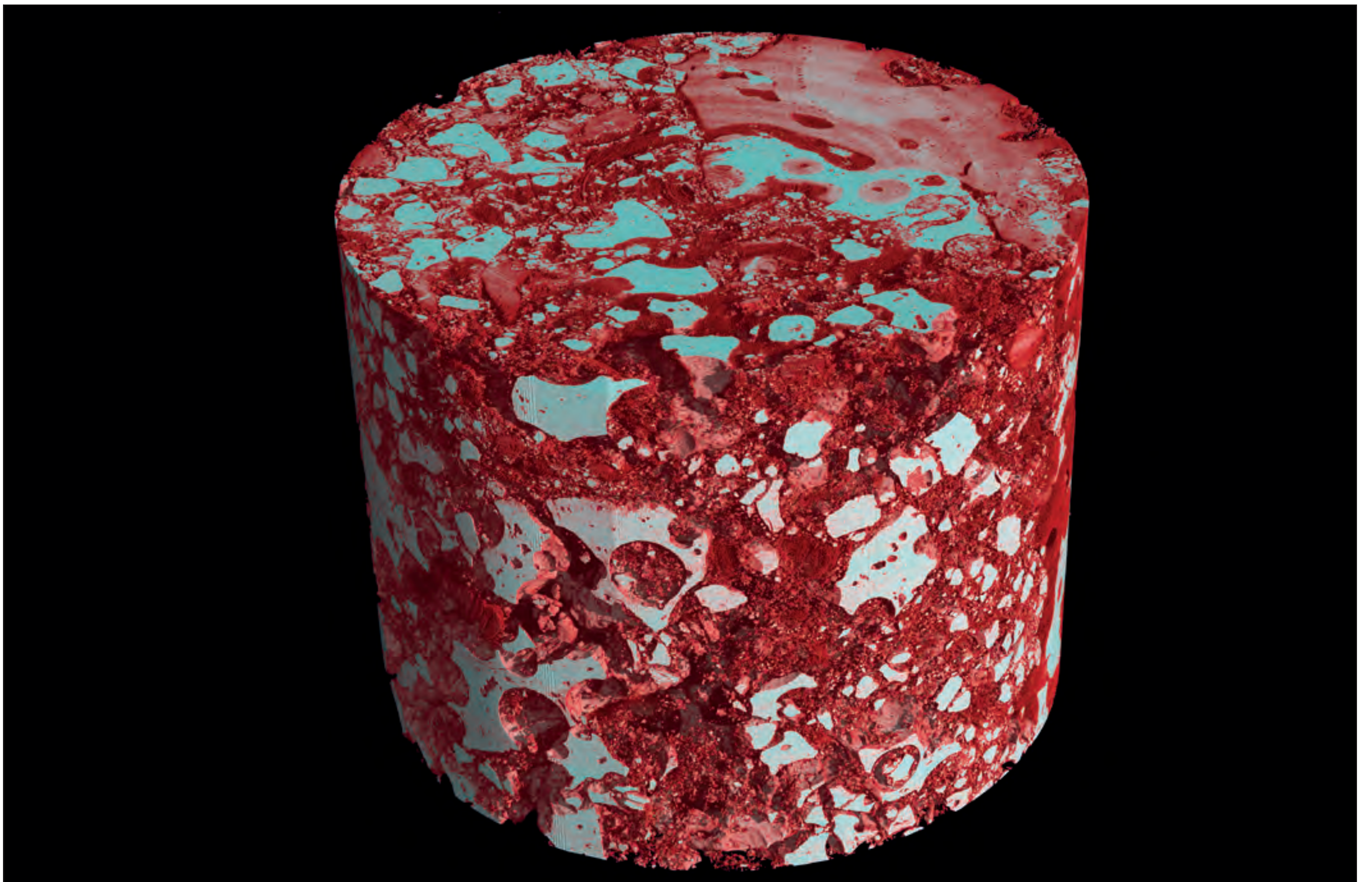


Nathan Nelson of Tel Aviv University, who has been an ESRF user since 1999, has won the 2013 Israel Prize in the field of Life Sciences for his research into molecular biology and proteins. The ESRF played a key role in the work that led to the award, specifically allowing him to solve the structure of the plant photosystem I (PSI) complex. "Each lecture I give on this structure, I always end up not only by thanking the ESRF but also emphasising the excellent service and lack of bureaucratic demands from the ESRF administration," he told *ESRFnews*.

Destined for Diamond

The ESRF's **Jorg Zegenhagen** is to become physical science co-ordinator at Diamond Light Source in the UK, where he will oversee science and developments at more than 20

beamlines. He was in charge of the ESRF's ID32 beamline, which was closed for user operation on 5 December 2011 as part of the ESRF Upgrade Programme. Zegenhagen, who takes up his new position in September, joined the ESRF in November 1999 and helped establish hard X-ray photoelectron spectroscopy (HAXPES) internationally, organising the first meeting of the HAXPES conference series at the ESRF in 2003.



A RACK/ESRF; M STILLER, C BERLIN, C KNABE/UNIVERSITY OF MARBURG

Bioceramic jawbone: This 2.8mm-diameter cylinder represents a regenerating-bone biopsy with bioceramic particles taken from the jawbone of ESRF beamline scientist Alexander Rack. Among their many surgical applications, bioceramic materials are used to help jawbone regenerate following the loss of a tooth due to infection. This sample, imaged six months after implantation, was scanned using high-resolution computed-tomography at ID19 and shows the integration between a commercial bioceramic (white) and new bone tissue (red). Images such as this allow researchers to determine how the degradation speed, microporosity and other properties of bioceramic particles impact bone regeneration.

In the corridors

X-ray scanners reveal too much

US TRANSPORTATION SECURITY ADMINISTRATION

The US Transportation Security Administration has removed all "backscatter" X-ray scanners from its airports following criticism that the technique reveals too much unnecessary detail to machine operators. Instead of relying on the absorption and transmission of X-rays, backscatter screening maps the patterns of X-rays scattered off elements within an object. Since elements with low atomic numbers scatter X-ray photons more strongly, the systems are good at imaging organic material such as drugs or explosives. US airports will use alternative full-body scanners based on millimetre-wave technology with software that produces a generic outline of the body and highlights suspicious material using coloured boxes.

Netherlands at ESRF

More than 25 Dutch companies were represented at the ESRF on 26–27 June as part of an industrial fair to bring together high-tech industry and R&D partners from the GIANT Grenoble campus. The ESRF is one of eight members of GIANT, which groups more than 10,000 industrial and academic researchers on one site and generates some 5000 publications and 500 patents every year.

External perception

ESRF

The ESRF's slogan "A light for science" is clear and understandable to those outside the ESRF, but does not convey enough about the ESRF's mission. These are two conclusions to have emerged from a survey of scientists and policy makers undertaken by communication specialist Marzena Lapka of CERN. The focus of the study was CERN, but Lapka also contacted 13,720 ESRF staff and users (2.5% of who responded). Her results suggest that surprisingly few CERN scientists are familiar with the ESRF. "What's interesting, though, is that once respondents who were unfamiliar with the ESRF visited the ESRF website, they decided that it was an important organization," says Lapka. "So it might be a communication issue."

No Windows 8

Having weighed up the pros and cons of Microsoft's latest operating system, Windows 8, the ESRF has decided to stick with Windows 7 until the release of Windows 8.1 later this year. An ESRF team concluded that Windows 8 has no added value for the ESRF at present and would require both an upgrade of ESRF infrastructure and extensive user training. Consequently, PCs running under Windows 8 are not supported and will not be connected to the ESRF network.

Multi-element X-ray detectors for beam-line applications

SGX Sensortech have a distinguished heritage in the manufacture of Silicon Drift (SDD) and Si(Li) detectors. Previously known as e2v scientific and Gresham Scientific, SGX specialises in producing detectors from standard designs through customised assemblies to complex multi-element detectors. All detectors are designed to meet the highest specifications, including the largest single active area (100mm^2) available of its kind.

Applications include:

- Extended X-ray Absorption Fine Structure (EXAFS)
- X-ray Absorption Near Edge Structure (XANES)
- Total Reflection X-Ray Fluorescence (TXRF)
- Particle Induced X-Ray Emission (PIXE)
- Micro X-ray fluorescence (µXRF)
- X-Ray Fluorescence (XRF)

Features

- **Available in 1 to 13+ channels**
- **Individual sensor areas 10mm^2 - 100mm^2**
- **Resolutions from 127 eV**
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- **Windows: Thin Polymer / Beryllium**
- **Focused or planar sensor arrangements**
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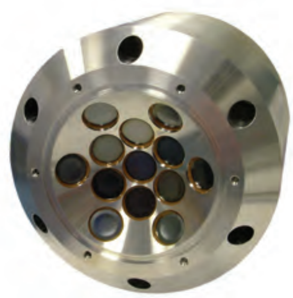
- **High solid angle**
- **Vacuum compatible (including UHV)**
- **Slide options: manual / adapted for translation tables**



Also available is a choice of close packed 3 x 100mm^2 or 6 x 100mm^2 sensor arrays

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