

A LIGHT FOR SCIENCE

ESRF **news**

Number 77 December 2017

**Extremely
Brilliant
Source**



Green light for new beamlines

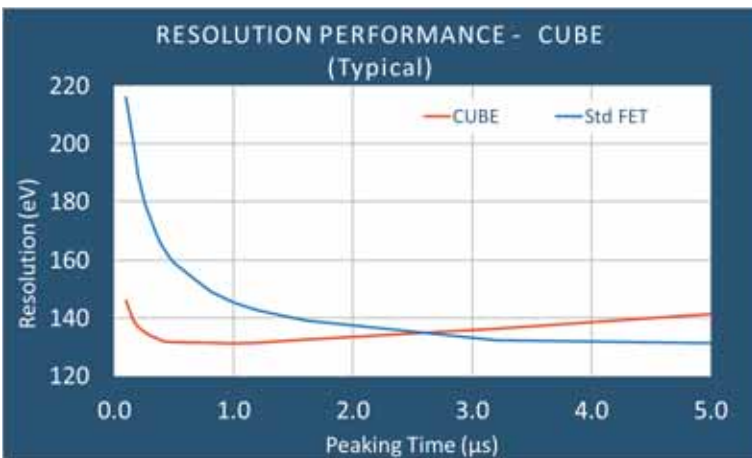
Algae turn fat to fuel

Cryo-EM arrives

SDD detectors for beam-line applications

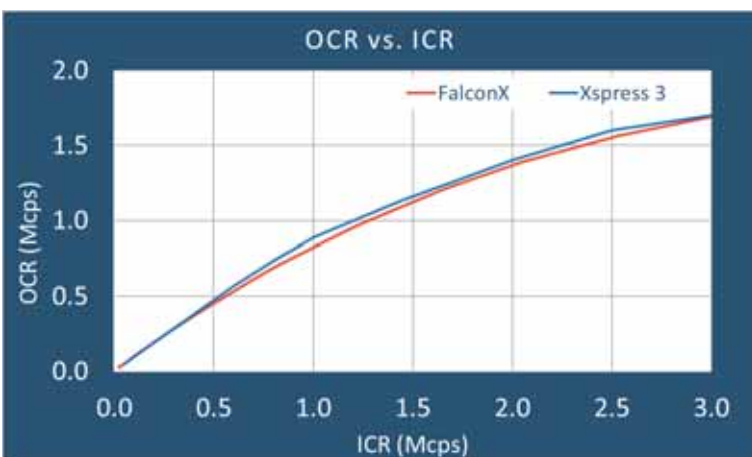
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Example of single sensor design



Example of multi-sensor design with UHV compatibility

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4 Sensor Vertical Focused Array SDD



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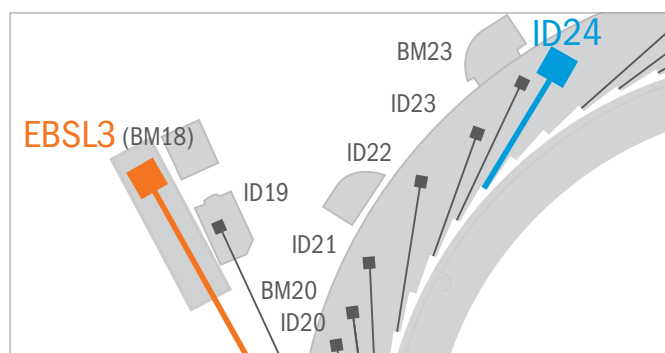
The European Synchrotron



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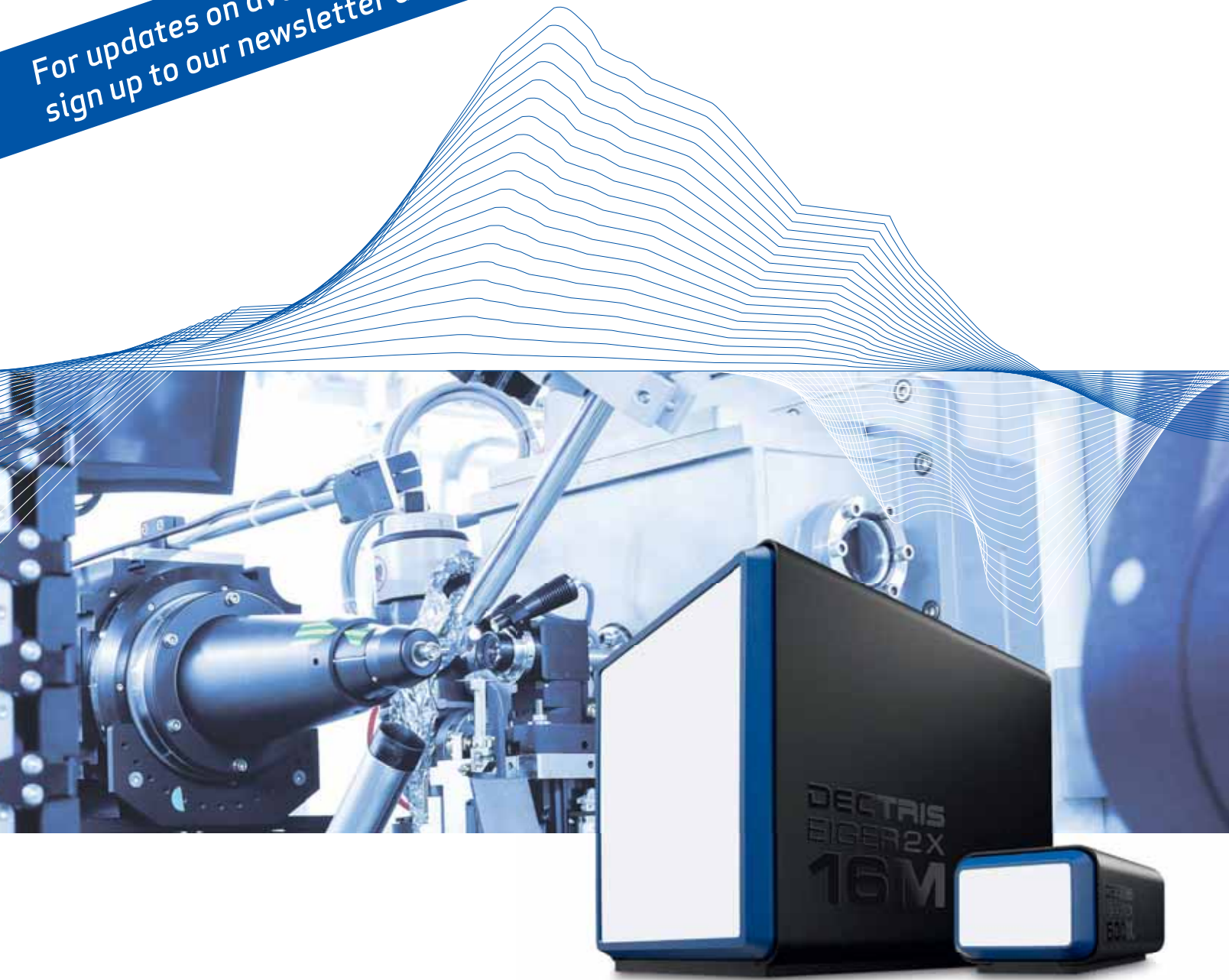
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A new record

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In September, a paper was published in the journal *Science* reporting a potentially groundbreaking step towards sustainable sources of hydrocarbon fuels. Using ESRF data, the authors managed to uncover how a recently discovered enzyme has the seemingly unique ability to convert fats into hydrocarbons, in the presence of sunlight. The results suggest that the enzymes could be exploited to generate diesel, petrol or even jet fuel (p12).

This *Science* paper was special in another way: it was one of those that inched the ESRF past a milestone of 30,000 publications since our facility first opened its doors in 1994. Among those publications are many that have since become regarded as breakthroughs – the discovery of the structure of the ribosome, for instance, won its authors Ada Yonath and Venkatraman Ramakrishnan, both long-term ESRF users, a share of the 2009 Nobel Prize in Chemistry. The publications reflect the diversity and vibrancy of the ESRF user community, too. Researchers from over the world, from all cultures and disciplines, come to the ESRF to push back the frontiers of science, to unlock the secrets of materials and to better understand living organisms. For over two decades this research has enriched scientific culture, as well as furthering society by boosting the economies and competitiveness of ESRF member states and beyond.

Such progress has been possible through a continuous and dynamic research and development programme, which focuses on constantly improving the instrumentation and technology of the X-ray source and beamlines. Together with the constant support of its international partners, the ESRF has become one of the leaders in the field of synchrotron and accelerator physics and technology.

One aspect of the ESRF R&D programme is the new cryo-electron microscopy (cryo-EM) platform, inaugurated in November this year. A collaborative initiative among the ESRF and its partners on the EPN science campus – the European Molecular Biology Laboratory, the Institut de Biologie Structurale and the Institut Laue-Langevin – this new state-of-the-art cryo-EM facility will provide opportunities to the international community of structural biologists. It will complement the ESRF macromolecular-crystallography beamlines and the other EPN campus structural biology facilities, and help us piece together the atomic details of the molecular machines of life (p13).

More broadly, the experimental programme of the ESRF's Extremely Brilliant Source (EBS) upgrade is entering a new operational phase, with the launching of the construction of four new flagship EBSL beamlines (pp19–25) – identified by the ESRF Science Advisory Committee and validated by the ESRF Council last June – complemented by important progress in the detector-development and data-infrastructure programmes. The four new EBSL beamlines, combined with a programme of refurbishment (pp16–17), will underpin research addressing the major challenges facing our society today, including defining the next generation of sustainable materials, developing new drugs, unravelling the complex mechanisms of living organisms, unlocking the secrets of our planet and environment, and reconstructing historical artefacts and fossils in 3D.

While all this is going on, the ESRF maintains its close involvement in European activities. It plays an important role in the ATTRACT programme for shared progress in detectors and imaging, in the SESAME programme for the Middle East's light source, through collaboration with Russia on the development of its fourth-generation synchrotron source, and with the new LEAPS (League of Electron Accelerator-based Photon Sources) initiative aiming to strengthen European leadership in X-ray science at synchrotron and XFEL facilities (pp6–7). The history of the ESRF has shown that international scientific collaboration can create tremendous breakthroughs and build bridges between nations: thanks to the constant support of our community, we are looking forward to setting new records.

Francesco Sette, *ESRF director-general*

"Many publications have become regarded as breakthroughs"

Giant LEAPS for integration

Last month the ESRF, together with 15 other facilities in Europe, launched the League of European Accelerator-based Photon Sources (LEAPS), to offer a “common vision” of using scientific excellence to solve global challenges, and boost European competitiveness and integration.

One of the main aspects of LEAPS is an agreed roadmap for the development of next-generation light sources and instrumentation, and tackling big data. Serving a combined 24,000-strong user community, the initiative also aims to maximise the strengths of individual facilities through coordinated specialisation, and to expand industry services. Meanwhile, it will capture and map socioeconomic impact, and improve training and outreach programmes. “LEAPS will use the power of its combined voice to ensure that member light-source facilities continue to be world-leading, to act as a powerful tool for the development and integration of skills with a view to address 21st-century global challenges, and to consolidate Europe’s leadership in the field,” reads the initiative’s mission statement.

ATTRACTing tech to the market

Large research facilities are home to swathes of cutting-edge technology, most of which is never seen outside of the scientific community. A new pan-EU initiative, ATTRACT, hopes to change that by helping to bring the detector and imaging technology to market, to boost sectors such as information technology and sustainable materials.

ATTRACT proposes a new co-innovation paradigm among industry, business, investors, innovation specialists and European research infrastructures. The ESRF, the European Molecular Biology Laboratory, the Institut Laue-Langevin and CERN are among the initiative’s nine partners, which will arrange for project proposals to be reviewed by an independent panel in order to receive €100k each in seed funding. The first launch for open calls is in spring next year with a final assessment in 2020. Details at attract-eu.org.



Kvashnina installs crystal analysers on ID26.

Kvashnina bags ERC grant

Kristina Kvashnina – a long-time ESRF user, a former ESRF staff member and current collaborating research group beamline scientist – has received a prestigious starting grant from the European Research Council (ERC) to pursue her work on actinide and lanthanide nanomaterials. The grant of €1.5 million is the maximum that can be awarded, and is designed to help early-career researchers in any scientific field who show the potential for “excellence”.

A theoretical physicist by

training, Kvashnina defended a PhD on soft X-ray spectroscopy of lanthanide and actinide systems at Uppsala University in Sweden, based on data taken at the Advanced Light Source at Lawrence Berkeley National Laboratory in California, US. Afterwards, she moved to the ESRF, where she has studied the same systems using the hard X-rays at beamlines such as BM20, the German Rossendorf (ROBL) beamline. Helmholtz-Zentrum Dresden-Rossendorf are the backers of the ROBL beamline, and have also

prepared samples of lanthanide and actinide nanomaterials for Kvashnina’s research.

To study the samples, Kvashnina has needed the ROBL beamline’s rare ability to safely handle radioactive materials, its X-ray emission spectrometer, and its functioning with low-energy X-rays in the region of 3–7 keV, to reach the appropriate actinide and lanthanide absorption edges.

“The ESRF is the only place in the world where we can carry out this research,” she says.

P&G signs up with the ESRF and ILL

Procter and Gamble (P&G) signed a “master collaboration agreement” with the ESRF and the Institut Laue-Langevin (ILL) in September, making the international consumer-goods company a strategic research partner with the X-ray and neutron facilities. The agreement establishes basic terms that will streamline future collaborations.

P&G scientists first came to the ESRF in 2004, when they used its beamlines to understand and optimise the microstructure of Clairol Perfect 10, a hair colourant that worked in a record-breaking 10 minutes. Since then, the scientists have used the ESRF to investigate everything from fabric detergent and conditioner, to skin products, nappies, polymer films and toothpaste.

“We have been working with the ESRF and ILL for more than a decade now to reveal the



Representatives from P&G, the ESRF and the ILL after signing the agreement.

fundamental microstructure of our materials and products, which helps the entire development process, from manufacturing to performance-optimisation,” said Eric Robles, a P&G research fellow. “Looking forward, we will co-develop new capabilities that allow us to continue to push the

boundaries, to deliver even more breakthrough benefits.”

Francesco Sette, the ESRF director-general, applauded the agreement. “The long-term partnership with P&G, a company that strongly invests in research for its development, is a great example of commitment to innovation,” he said.

Tattoo nanoparticle danger

Think before you ink.



Nanoparticles present in tattoo ink can migrate to the lymph nodes, a critical part of the body's immune system, according to research performed at the ESRF. The nanoparticles include pigments and titanium dioxide, as well as chromium and nickel, which are toxic.

"When someone wants to get a tattoo, they are often very careful in choosing a parlour where they use sterile needles that haven't been used previously," says ESRF scientist

Hiram Castillo-Michel, one of the authors of the work. "No one checks the chemical composition of the colours, but our study shows that maybe they should."

Previously, the chemical dangers of tattoos have been studied only outside of the body, although it has long been noted that the lymph nodes of tattooed people are sometimes enlarged and take on the tattoo's colour, implying that at least micro-sized particles are present. According to study

author and ESRF postdoc (now visiting scientist) Bernhard Hesse, however, the discovery of tattoo nanoparticles in lymph nodes is concerning because particle behaviour on that scale in the body is poorly understood. "That is the problem: we don't know how nanoparticles react," he says. The study was performed using X-ray fluorescence measurements at ESRF beamlines ID21 and ID16B (*Sci. Rep.* **7** 11395).



User Meeting: 5–7 February.

ESRF gears up for User meeting

The 2018 User Meeting is set to take place from 5–7 February, and is open to all ESRF users. According to the User Organisation Committee (UOC), the meeting will include a "rich and diverse" programme of events, including keynote lectures, the Young Scientist Award, a poster session and director's report, as well as a range of scientific tutorials. Microsymposia will be held on the subjects of high-pressure science at the ESRF, metallurgy and materials processing, and the use of synchrotrons to understand neurological diseases. For more information, see the UOC story on p10 or visit www.esrf.eu/UM2018.

SESAME staff exchanges begin

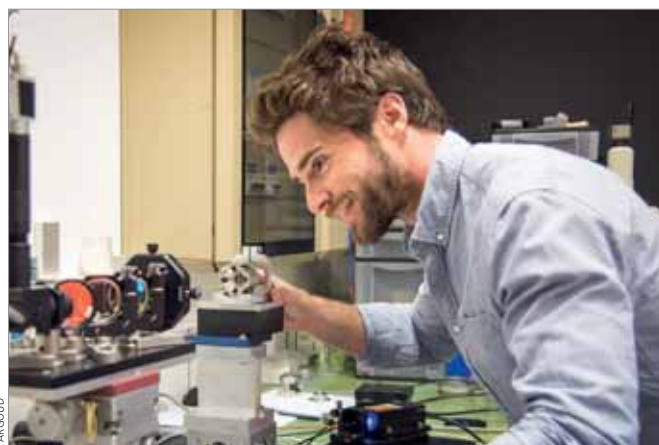
The ESRF welcomed its first scientist from the Synchrotron-Light for Experimental Science and Applications In the Middle East (SESAME) in July, as part of an EU project coordinated by the ESRF, known as OPEN SESAME, to help establish the new Jordan-based light source. Mahmoud Abdellatif is one of 16 SESAME scientists who have either exchanged or have scheduled exchanges with European facilities as part of the project. Ultimately, 65 staff will be exchanged.

A beamline scientist, Abdellatif was at the ESRF for three weeks, during which he completed technical specifications for a diffractometer and decided what set-ups and techniques his experimental station will enable. He was mentored by Andy Fitch, beamline scientist at ID22, among other ESRF staff. "This experience will help me to figure out the best design for our beamline so that we can exploit it to the maximum with the scientists in the community," Abdellatif said.

Carbonates explain carbon's descent

Most people are familiar with the carbon cycle above ground, but tectonic movements actually circulate a lot of the carbon below ground. In fact, recent estimates place some 90% of the Earth's carbon in the mantle and core – though quite how it is transported there has been a mystery.

Valerio Cerantola of Bayreuth University in Germany (now a postdoc at the ESRF) and others have shed some light on the matter, by performing experiments on iron carbonate at pressures over 100 GPa and temperatures over 2500 K, using the ESRF beamlines ID27, ID18 and ID09A (now ID15B). The carbonates are believed to exist in the lower mantle because they are found in diamonds originating in that region, but their behaviour at



Cerantola exploits the high-pressure lab at the ID18 beamline.

extreme conditions is poorly understood.

The researchers found that, at higher pressures and temperatures, different phases of iron-bearing carbonates

containing CO₄ tetrahedra are preserved by reduction and oxidation reactions, suggesting that these could be the lower mantle's carbon-carriers (*Nat. Commun.* **8** 15960).

Success for mock-up cell

Engineers at the ESRF have successfully built an entire EBS cell, consisting of girders, magnets, vacuum chambers and other components. The mock-up went entirely to plan, and paves the way for the assembly of the EBS proper (see story, right).

The EBS storage ring will consist of thousands of components, all of which have been designed in three dimensions with computer software. Experience from previous projects, however, has demonstrated a possibility of conflicts between components during assembly due to design or machining faults, especially when space constraints are very high. In the EBS, many components have been designed with less than half a millimetre of room for error. The Machine Advisory Committee recommended the building of a mock-up cell at a meeting last year, and work began in February this year in the ESRF's Chartreuse Hall. "The mock-up is the best tool to anticipate and to tackle technical issues before the busy assembly phase," says Laurent Eybert, one of the managers of the mock-up. "It is also a demonstrator and a useful training tool for all people involved in the project."

The mock-up space was divided into two zones: one for the assembly of vacuum vessels on dedicated tables, and the other for four girders, anchored to the concrete floor in the alignment they will ultimately have in the tunnel. The engineers prepared a detailed procedure for assembly, outlining the necessary control points, timescales, labour, tools and layout configuration. By 12 September, all the magnets were installed and aligned,

and all the vacuum chambers were connected together and inserted in the magnets, and pumped. "No major issues were discovered," says Eybert.

"Many components have less than half a millimetre of room for error."

The mock-up was the first time that engineers from the different groups and divisions – alignment and geodesy, technical infrastructure, vacuum, insertion devices, power and so on – have worked together in the same place for the new EBS machine. "We've had many meetings, but never before have we all been working together, to do the assembly," says Eybert. In fact, adds Eybert, working together allowed the team to get to know one another, and to know precisely who was responsible for what.

The engineers baked the vacuum chambers at temperatures of up to 150 °C for 48 hours to achieve a level of vacuum better than 1×10^{-10} mbar, and checked that the main components still remained within the alignment tolerances. In the next step, input facilities such as piping and cabling will be installed to fully validate their design, before issuing calls for tender.

Right: ESRF engineers and technicians make adjustments to the mock-up cell.



For more upgrade news,
check out the EBS blog:
<http://ebs.esrf.fr>

Girder assembly begins

Assembly of the EBS has begun in earnest, as ESRF engineers and technicians prepare the machine's 128 girders with all the components they need to form the new storage ring. Working in a separate, dedicated building, ESRF01, the engineers began last month and hope to complete up to four girders per week, so that the girders are ready to be moved into the location of the existing storage ring by the shutdown at the end of 2018.

Like that of the existing ESRF, the EBS storage ring is made up of 32 cells, each of which is composed of one straight

section and one arc. One arc is composed of four girders, and these have to support all the components for maintaining the electron beam – principally magnets, magnet supports and vacuum chambers. There are many steps to the assembly, including the installation of pre-positioning templates, alignment and checking of alignment. It takes engineers and technicians three weeks to assemble the four girders in one arc, although they are speeding up the process by running three assembly lines in parallel. "This assembly represents a

complete, real arc," says Jean-Claude Biasci, the manager of the assembly. "For us technical people, it's the opportunity to validate all of the components and the assembly procedure, including girder connections and bake-out."

The exact rate of girder assembly will depend on the stock of components and the availability of labour. Once assembled, the girders will be ready to be moved into the ESRF tunnel, where they will only need to be aligned and have their vacuum chambers connected together.

Insight: The hybrid multi-bend achromat lattice



One cell of the EBS's HMBA lattice, supported on its four girders. There are four dipoles (blue) and three dipole-quadrupoles (pink), making seven bending magnets in total. Also shown are quadrupoles (red) and sextupoles (green).

What is the lattice?

The hybrid multi-bend achromat (HMBA) lattice is the composition of magnets that will guide and focus the EBS's electron beam. Currently, the ESRF has a "double bend achromat lattice", so called because it relies on two bending magnets (dipoles) per cell, and because electrons of different energies are bent and focused in the same way – "achromatically" – resulting in very collimated and stable beams. There are also achromat lattices based on three or four bending magnets: the HMBA will have seven. Pioneered by the ESRF, the HMBA will be key to the EBS's status as a "fourth-generation" synchrotron source, reducing horizontal emittance by a factor of 30 compared with the existing machine.

Why is that so important?

Emittance describes how tightly the electrons are confined in the beam. If the emittance is smaller, the electrons are more

closely packed, and the resulting synchrotron X-rays are brighter and more coherent – in other words, they approach the properties of a laser beam. The HMBA will boost coherence by some two orders of magnitude. Such properties are the driving force of the EBS upgrade, opening up new fields of investigation in fundamental research, and allowing far better industry diagnostics to promote innovation.

How will the HMBA make these gains?

Having seven bending magnets per cell gives far more control over the electron beam, but that is just the start. Many magnets are innovative: some dipoles are made of permanent magnets (see *ESRFnews* July 2017, p9), for example, while some magnets simultaneously bend and focus the beam. Compared with the current lattice, there will also be 25 further magnets per cell to enact subtle corrections.

Will all this actually fit in the current ESRF tunnel?

Remarkably, yes. With roughly twice as many magnets in the same space, each has had to be engineered to be more compact and generate magnetic fields up to three times stronger than existing models. The vacuum chambers have had to be redesigned for limited space in and around the magnets, too, while the mechanical tolerances of some components have shrunk to just 100th of a millimetre. It is a complex and revolutionary design that has inspired the composition of other big light sources around the world, and won the ESRF's Pantaleo Raimondi the prestigious Gersch Budker Prize from the European Physical Society Accelerator Group earlier this year. It "shows Raimondi's ability to foster new ideas, his deep understanding of accelerator physics and [his] mastering of technological aspects," the jury said.

News from the User Office

In September, for the penultimate deadline before the shutdown for the ESRF's Extremely Brilliant Source (EBS) upgrade at the end of 2018, an all-time record of more than 1300 proposals was submitted, requesting a total of almost 18,000 shifts of beam time. Despite this high submission, faster and more efficient experiments should still ensure an acceptance rate of around 40%. These proposals were reviewed during the Beam Time Allocation Panel meetings on 26 and 27 October, when 126 external scientists met at the ESRF to evaluate them and provide recommendations for beam-time allocation. We are very grateful to our committee members for all their hard work in reviewing and discussing this very high number of proposals.

The next deadline (and last one before the EBS shutdown) for submission of standard proposals is 1 March 2018, for beam time between August and December 2018. There is no Long Term Project submission deadline in January 2018.

Proposers are reminded by the Beam Time Allocation Panels of the importance of submitting experiment reports for all beam-time allocations previously used, and of citing these in the relevant section of the proposal form. 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. Continuation proposals must have an experiment report submitted for the original proposal.

Joanne McCarthy, Head of the User Office

News from the User Organisation Committee

The User Organisation Committee (UOC) met in September at the ESRF to finalise the organisation of the User Meeting. The 28th edition of the event will take place on 5, 6 and 7 February 2018 at the ESRF and all users are invited to attend.

Along with management and the User Office we have prepared a diverse and rich programme including keynote lectures, the Young Scientist Award, a poster session, the Directors' report and user-dedicated sessions with 12 tutorials on a range of different scientific topics and three microsymbiosia. During the plenary session, an update on the EBS upgrade and the plan of future related activities will be given. Further information on the 2018 User Meeting, and a download of the event poster

(which we encourage you to circulate), can be found at esrf.eu/UM2018. In particular, we invite users to send nominations for the Young Scientist Award. This year, besides receiving €1500, the winner will also feature in a short promotional video about his or her research, produced by a professional company.

We would like to remind all users that the UOC has regular meetings over the year, the agenda and minutes of which are available upon request. Users are welcome to contact us at any time via e-mail. Representatives of each user scientific community and their e-mail addresses can be found at www.esrf.eu/UsersAndScience/users_org. *Paola Coan, chair of the User Organisation Committee*

News from the beamlines

- The ESRF has installed and commissioned a Titan Krios cryo electron microscope (**cryo-EM**) equipped with a GATAN K2 detector for studies in Structural Biology (see p13). This new facility, ESRF "beamline" **CM01**, became operational in mid-November 2017 and has been open for proposals since 28 September. Applications for microscope access are made as Rolling Access Proposals and reviewed by the Beam Time Allocation Panel C10 (Structural Biology beamlines). Initially, only projects that have been previously evaluated using cryo-EM and for which suitable proof of the sample quality is available will be considered. This proof has to be included in the application for access.

- On the **SNBL** CRG beamline **BM01**, just prior to the summer shutdown, a group of users from the Department of Materials Science and Engineering at the Norwegian University of Science and Technology (NTNU) had the first beamtime using a new *in situ* heating setup for thin films (see photo, right). The setup is designed to mimic the processing parameters typically used in thin-film production by the chemical solution deposition method. The group has a long history of



Anders Bank Blichfeld of the NTNU using the new *in situ* heating setup on BM01.

working with piezoelectric thin films and hopes that the new *in situ* characterisation tool will help to understand the nucleation and growth mechanisms, and improve the tuning and designing of processing protocols to obtain better materials.

- On **ID15A** the new station for Materials Engineering (ID15A-EH2) is ready for users. Techniques available are ultra-fast imaging/tomography, energy dispersive diffraction and

angular dispersive diffraction. In the Materials Chemistry station (ID15A-EH3) the DRIFTS spectrometer has been installed since the beginning of the summer, allowing combined XRD-DRIFTS measurements to be made.

- Additional laser PSS systems have been installed on both **ID19** and **ID31** to allow shock as well as additive manufacturing experiments to be performed.

- In addition to bulk XAFS spectroscopy, the **ROBL** CRG beamline **BM20** has now a Johann-type spectrometer with five analyser crystals available, which can be used for high-resolution XANES, XES and RIXS. Furthermore, surface diffraction (CTR, RAXR), powder diffraction and single-crystal diffraction are currently being developed and available to expert users upon request. Note, however, that ROBL will be closed from the summer shutdown 2018 onwards for the building of a new radiochemical hut. After the EBS upgrade, all techniques will be available in an alpha-lab environment dedicated to actinide research.

- The infrared station at **ID21** stopped operation at the end of September after 13 years of successful activities, mainly in

the fields of biology and cultural heritage. The microscope will still be operational but using the internal Global source, and the hut will be fully modified as part of the refurbishment of the ID21 X-ray platform. The X-ray branches will be temporarily closed from December 2017 to March 2018 for the first parts of this refurbishment.

- The Structural Biology beamlines **FIP-BM30A** and **ID30B** hosted practical sessions as part of the MXIS 2017 training course. MXIS is a practical course dedicated to *in situ* protein crystallography, with a special emphasis on ligand screening. The workshop was organised in two parts, each one lasting for three days. Part 1 took place 8–10 November at the CNRS campus in Montpellier, with full lectures and practicals on the topics of plate pre-coating for "dry" co-crystallisation and *in situ* screening on a lab source. Part 2 took place on the EPN Science Campus in Grenoble, 29 November – 1 December, with full lectures at the Institut de Biologie Structurale and practical sessions at the ESRF on the topics of *in situ* diffraction for crystal screening and structure resolution, and *in situ* data processing.

Positioning solution for analytical-methods: mechanical system approaches limit of technical feasibility

At the X-ray light source PETRA III at the DESY research centre (German Electron Synchrotron) in Hamburg, Germany, the Helmholtz-Zentrum Geesthacht Centre for Materials and Coastal Research (HZG) operates the Imaging Beamline P05, which includes two experimental hutches, one for nanotomography and one for microtomography. In the nanotomography hutch, X-ray optics for three-dimensional micrographs with resolutions around 100 nm are used. The set-up also includes microscopy optics for visible light, used for further magnification of the X-ray micrographs and their transfer to a camera.

With the aim to carry out as many different experiments as possible, the HZG provides two different X-ray optics configurations: an imaging set-up, in which the sample is positioned in front of the objective optics, and a cone-beam set-up, in which the sample is placed in the diverging beam behind the optics. In both cases, high mechanical stability and precision positioning are essential to obtain micrographs of high quality.

However, thanks to the close co-operation of the clients with the engineers and developers from PI (Physik Instrumente), this complex task could be solved in a practice-oriented manner.

A particular challenge was how to configure the control, which was based on an industrial controller. The challenge consisted in controlling almost 50 axes independently of one another while ensuring collision protection. The entire system was finally integrated into the TANGO interface customary for beamlines.

The base: granite platform supported by air bearings

To minimise the effect of vibrations and securely fasten the individual components and stabilise them, relative to one another, a granite base 6.8 m in length forms the basis of the instrument. Another four moving granite platforms driven by linear motors are arranged on this base on air bearings. This makes it possible to position all components with high speed and precision: the sample stage, the X-ray optics and the detector. The substructure itself, which weighs several tonnes, is also mounted on air bearings. This allows the entire assembly to be moved out of the X-ray beam with minimal effort when the second experimental station is to be used,



Mounted on the combined tilt and Z stage (consisting of three differentially controlled lifting elements) is an air-bearing supported rotation stage. It rotates at a velocity of up to 36°/s and works with flatness deviations of less than 100 nm at a resolution of 0.5 μrad (Image: PI / HZG)

while maintaining a stable position as soon as the air flow is switched off.

Complex sequences during sample positioning

The basis of sample positioning is a horizontal positioning unit that moves the sample stage into the beam. It has a travel range of 20 mm, can be subjected to a load of 300 kg and works with a repeatability of 30 nm.

This displacement unit is equipped with three lifting elements that perform the height adjustment, tilt correction and orthogonal alignment, relative to the beam. It is based on three identical, symmetrically arranged and position-controlled stepper motors, combined with worm gears and spindle drives. Mounted on this Z stage is an air-bearing supported rotation stage. In developing this stage, the designers had to push the limits of technical feasibility: what was required was a really “pure” rotary motion of the sample with minimal wobble, radial runout or eccentricity. Only in this case can sharp pictures over 360° be made that all refer to the same volume element and can all be clearly assigned when reconstructing the picture. This is why the rotation stage, which rotates at a velocity of 36°/s,

works with flatness deviations of less than 100 nm at a resolution of 0.5 μrad. The air bearing does not produce any friction, which over time would lead to a deterioration of these values.

Parallel kinematics for the sample holder and the optics

The actual sample holder is located in the aperture of the rotation stage on the moving platform of a six-axis parallel kinematic system. The samples are positioned with six degrees of freedom. Essential features are the freely selectable pivot point of the parallel-kinematic system and its high stiffness. A six-axis parallel-kinematic system of this type is also used for the positioning of the optics. In nanotomography, which allows three-dimensional micrographs with resolutions below 100 nm, this machine is used to align compound refractive lenses (CRL) in the beam with high precision.

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Algae turn fat into fuel

The ESRF snapshots a reaction that could provide cleaner hydrocarbons.

A lush carpet of algae stretches over a placid freshwater lake. In the environmental movement, this is the kind of treasured picture the word “green” was intended to evoke. Yet it could prove to be a source of a product that usually brings to mind all the dirtiness of crude oil: hydrocarbon fuel.

That is the suggestion of a group of biochemists led by Fred Beisson at the Institute of Biosciences and Biotechnologies of Aix-Marseille (BIAM), a joint institute of the French Atomic Energy Commission, the French National Centre for Scientific Research and the University of Aix-Marseille. Previously, the researchers discovered that certain algae, when illuminated by sunlight, are able to convert fatty acids into hydrocarbons. Now, with help from the ESRF, the researchers have found out how – and they believe the answer could lead to a clean source of biofuels. “It’s amazing to discover that microalgae might hold a key to the fuel of tomorrow, especially as regular petroleum is in fact heavily derived from ancient algal deposits,” says Beisson.

Fatty acids are chemically similar to hydrocarbons, except for the addition of a pair of oxygen atoms at the end of the carbon chain. Removing this carboxylic head normally requires expensive and energy-intensive processes, such as electrolysis, and corrosive reactants, such as sodium hydroxide.

Since the 1970s, there have been hints that some microalgae are able to remove the carboxylic head, although it has been equally possible that any hydrocarbons generated came from contaminating bacteria, or even from organic solvents used in the analysis. Given the prospect of a clean source of fuel, Beisson and colleagues wanted to find out for sure. Last year, they performed controlled tests on several pure cultures of microalgae, and found that a freshwater species, *Chlorella variabilis*, could indeed convert fat to fuel, with sunlight as an input (*Plant Physiol.* **171** 2393).

To explore how the microalga does it, Beisson and colleagues filtered its proteins into a number of fractions, and tested each for an ability to convert fat. The researchers then used a standard technique known as proteomics to identify the proteins inside the most highly active fractions. Among the 10 proteins common to the active fractions analysed, only one of them was unknown. The researchers therefore made a bacterium express the gene encoding this protein, and

found that, under blue light, the bacteria could indeed generate hydrocarbons from its own membrane fatty acids. The protein was a new photoenzyme, which the researchers named fatty photodecarboxylase.

“Seeing is believing. The structure of the complex was important.”

The discovery was exciting: besides photosynthesis, reactions involving light-driven enzymes are incredibly rare in living organisms. To confirm the enzyme activity, Beisson and colleagues sent crystals of the photoenzyme in complex with a common fatty acid to the ESRF’s MASSIF-1, the only beamline in the world able to automatically screen crystals and probe a crystal’s best

Cryo-EM launched

ESRF facility delivers high-resolution structures without crystals.

For 10 years, you have been investigating the structure of a biological complex comprising dozens of proteins and nucleic acids. Classical macromolecular crystallography has given you high-resolution structures for some of the components, but only a three-dimensional structure of the entire complex at close to atomic resolution will reveal its detailed function and interactions. Unfortunately, although you have a pure sample of the complex, it refuses to be crystallised.

If this situation is anything like your own, the ESRF's new cryo-electron microscopy (cryo-EM) facility could help. In a cryo-EM experiment, a solution containing the biomolecule or complex of interest is applied to a sample holder, or "grid", as a thin layer. The grid is flash-frozen in liquid ethane to vitrify the sample, which is then imaged by the cryo-EM with low doses of electrons to minimise radiation damage. Because particles are imaged directly, there is no need for crystallisation – although, if available, any high-resolution crystal structures of the complex's individual components can be used to guide the interpretation of the cryo-EM image.

In October this year, the Noble in Chemistry was awarded to three scientists, Jacques Dubochet, Joachim Frank and Richard Henderson, for pioneering developments using cryo-EM. Over the last decade or so, the average resolution that can be obtained using this technique has dramatically improved from 15–20 Å to around 8 Å, with many of the most recent depositions in the Protein Data Bank exhibiting atomic resolution of 3–4 Å. This leap has been made possible by an enormous progress in direct-detection technology for cryo-EM cameras, the

technology in the microscopes themselves (electron delivery, for example) and in cryo-EM sample preparation.

As a European leader in large-scale structural-biology infrastructure, the ESRF has embraced this cutting-edge technique and invested in a cryo-EM facility, CM01, that will be operated in a similar fashion to its regular X-ray beamlines. A collaboration with the European Molecular Biology Laboratory, the Institut de Biologie Structurale and the Institut Laue-Langevin, CM01 was inaugurated last month, and will remain operational during the long ESRF shutdown for the Extremely Brilliant Source upgrade in 2019/2020. CM01 is a state-of-the-art Titan Krios G3 cryo-EM for the 3D characterisation of single particles of biological macromolecules. In addition to a GATAN K2 direct detection detector, this most powerful and flexible high-resolution microscope is also equipped with a Volta Phase Plate, delivering high-resolution imaging of macromolecules of less than 150 kDa in mass, and a GIF quantum LS energy filter to enhance the quality of the images by producing less background noise and better contrast. The high frame-rate K2 detector can record many images in a "movie mode", thereby correcting for electron beam-induced sample drifts and enhancing both low- and high-resolution frequencies.

As with any ESRF "beamline", CM01 was extensively characterised and tested prior to its inauguration. The first large biomolecule studied with the microscope – the tobacco mosaic virus, which infects tobacco and other plants – produced an excellent description of its three-dimensional structure at a resolution better than 4 Å. The result augurs extremely well for the new facility and its aim of producing structural information of high scientific impact on the form and function of large macromolecular complexes.

• Proposals for experiments on CM01 can be submitted via a Rolling Access application pathway. See tinyurl.com/ESRFcryoEM.

Christoph Mueller-Dieckmann and Eazhisai Kandiah, ESRF



Chlorella variabilis lives in a freshwater environment.

diffraction volume. The data revealed the fatty acid wrapped around part of the photoenzyme that leads to where a common biochemical, flavin adenine dinucleotide (FAD), can be held. Beisson and colleagues believe that, under blue light, the photoenzyme helps FAD to steal an electron from the fatty acid, which responds by detaching its carboxylic head to make a pure hydrocarbon (*Science* **357** 903). "If seeing is believing, then the structure of the complex was indeed important," says BIAM's Pascal Arnoux, who worked closely with the ESRF beamline operation manager Didier Nurizzo.

In nature, explains Beisson, *C. variabilis* probably converts some of its fatty acids to hydrocarbons for use as a hydrophobic barrier, or for signalling purposes. By engineering a photosynthetic microorganism to produce more of the photoenzyme, this mechanism could be encouraged for the industrial generation of hydrocarbons for fuel. This would be an all-in-one solution in which a ready-to-use fuel is effectively produced by a cell factory, via photosynthesis, from carbon dioxide. Alternatively, the photoenzyme itself could be exploited to convert fatty acids derived from plant oils, such as rapeseed and palm.

With regular plant or microalgal fatty acids, the hydrocarbons that result would be 15–17 carbon atoms long, like diesel. But Beisson is musing on the possibility of shortening the fatty acids first, to generate hydrocarbons more like petrol (6–10 carbon atoms) or jet fuel (10–13 carbon atoms). "Jet fuel is of particular interest, because cars can always be powered by electricity, but not planes," he says.



CM01 was inaugurated in November.

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Extremely brilliant progress

The ESRF's upgrade, the Extremely Brilliant Source, is on track to deliver exciting new scientific and industrial opportunities.

This year has been an important one for the ESRF's upgrade programme, the Extremely Brilliant Source (EBS). On 27 June the ESRF Council, representing the 22 partner nations of the synchrotron, gave the green light for the construction and commissioning of new beamlines, which together with the new source and enabling technology make up the ESRF-EBS programme's three pillars. Four new beamlines will be designed for the full exploitation of the enhanced performance of the first fourth-generation high-energy synchrotron. They will address major challenges facing our society, including the development of the next generation of drugs, biomaterials and sustainable materials, and provide deep insights into the complex mechanisms governing living organisms. They will elucidate our recent and ancient past, as manifested in historical artefacts and fossils. What's more, they will provide unique opportunities for applied and innovation-driven research.

The choice of the four EBS beamlines (which are described in detail on pp19–25 of this issue) was the result of an extensive selection process. The entire ESRF user community put forward 48 expressions of interest, and the ESRF Science Advisory Committee then ranked the final eight proposals for new beamlines. The construction of the new beamlines is planned such that it will minimise the disruption of user service. First steps towards the implementation of the new EBS beamlines will be taken in spring 2018 with the delivery of Technical Design Reports for the construction of the two first beamlines, at BM18 and ID29.

Just the start

The four EBS beamlines will be complemented by a deep refurbishment of three more beamlines considered during the selection process – ID10, ID24 and ID27 – and the implementation of two user platforms: the high-power laser facility (HPLF) and the cryo-electron microscope (cryo-EM). Within the framework of continuing beamline refurbishment, further beamlines will be modernised too, following the outcome of the regular beamline reviews: ID18, ID21, ID23-2 and possibly more. Furthermore, significant investment is foreseen for the upgrade of detectors and the replacement of the

spectroscopy monochromators in order to enable a maximum number of beamlines to take full advantage of the EBS source.

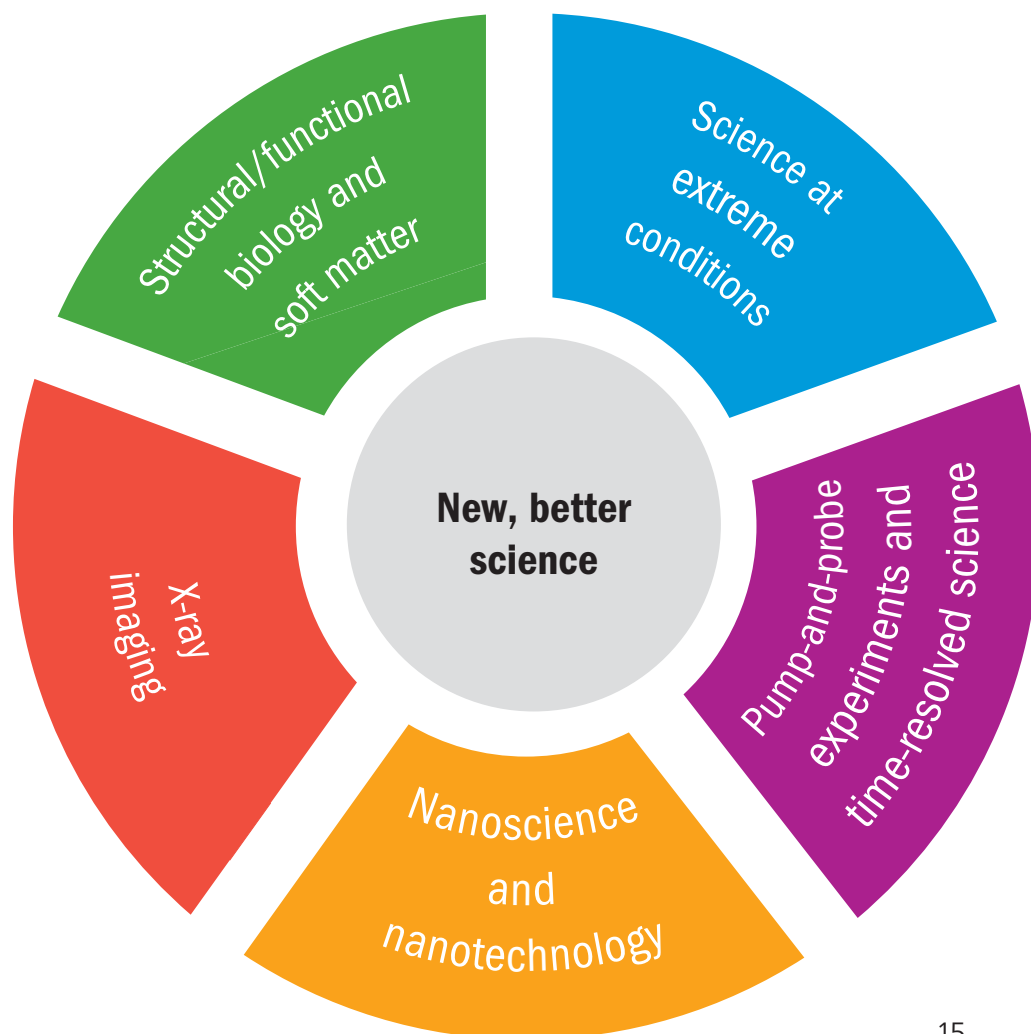
Meanwhile, there is a significant investment in the third pillar of the ESRF-EBS programme: experiment control, data management and the development of advanced data-analysis tools for our users. At the heart of this initiative is the new data policy endorsed by the ESRF Council in November 2015, which guarantees data archival for 10 years (and metadata indefinitely) as well as open access to data after an embargo period of three years, which can be extended upon request. The implementation of the new beamline control system, BLISS, will take place throughout the duration of the ESRF-EBS programme.

After a 20-month shutdown, starting at the end of 2018, the ESRF will come back with an extremely brilliant source that will

"Scientists will carry out experiments that are currently impossible."

enable scientists to carry out experiments that are currently impossible. We will ensure that the ESRF beamlines and their performance continue to evolve after the EBS project, to capture all opportunities to optimise the science that can be developed with the new source.

Harald Reichert and Jean Susini, ESRF



ESRF–EBS: the

The makeup of the Extremely Brilliant Source (EBS) has become clear. This diagram shows the layout of the upgrade, including new beamlines

EBS flagship beamlines

Four entirely new beamlines will be built between 2018 and 2022:

EBSL1 Coherence applications (see p19)

EBSL2 Hard X-ray diffraction microscopy (see p21)

EBSL3 Phase-contrast tomography (see p23)

EBSL8 Serial synchrotron crystallography (see p25)

EBS refurbishment beamlines

Also over the 2018–2022 period, a refurbishment programme will be carried out in parallel to the regular refurbishment, to exploit the high performance of the new EBS storage ring for other beamlines (see details opposite):

ID10 Surface science

ID24 High brilliance X-ray absorption spectroscopy

ID27 Nano X-ray diffraction in extreme conditions

New platforms

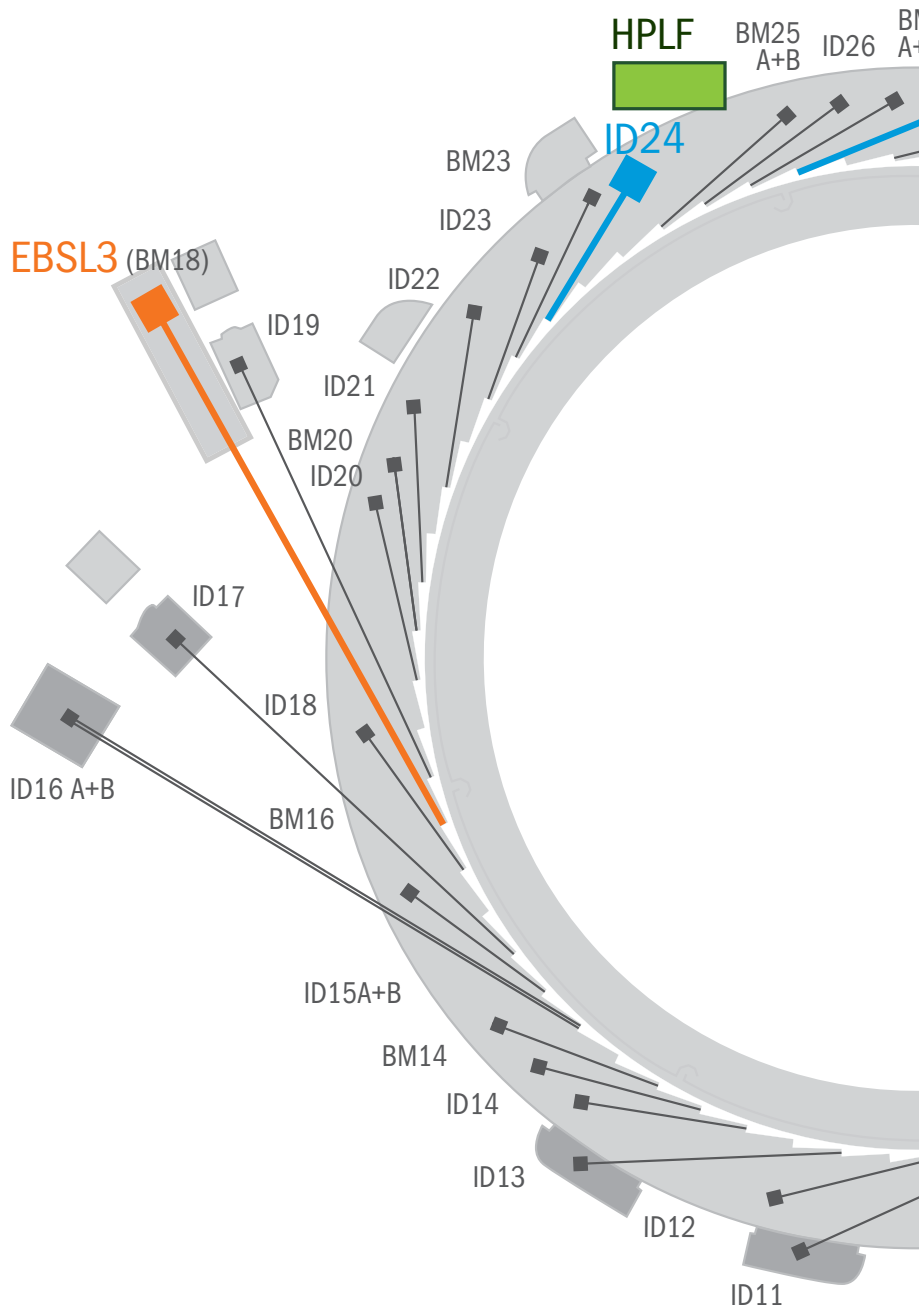
Finally, as part of ongoing efforts to provide improved services to users, the ESRF is strengthening its expertise with two cutting-edge platforms:

Cryo-EM for determining structures of biological macromolecules without crystallisation (see p13)

High-power laser facility (HPLF) for the creation of exotic states of matter at ultra-high pressures and temperatures, such as those found in exoplanets, or for the study of matter under very high strain, such as that in demand by industry.

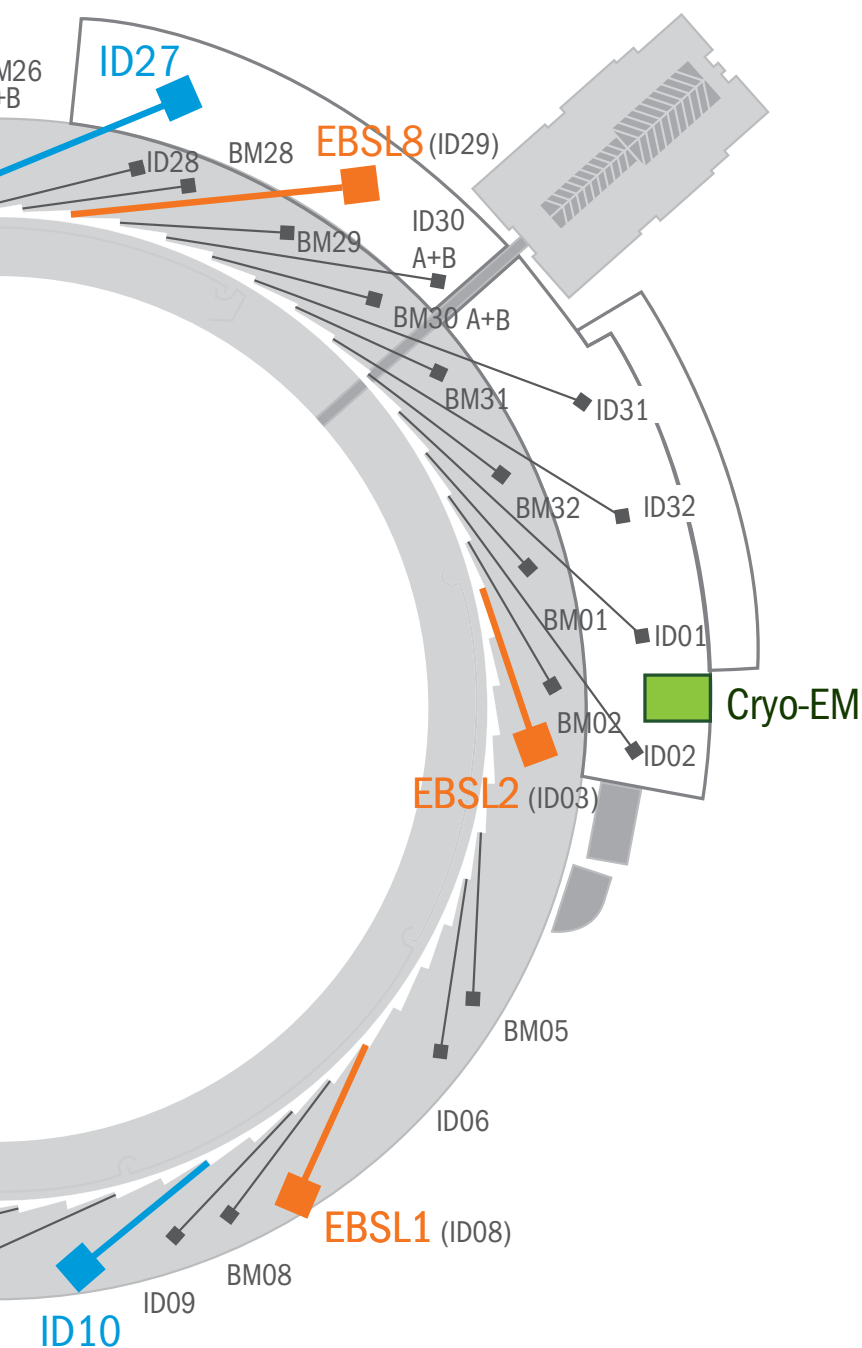
Instrumentation and data

Properly exploiting the EBS's outstanding X-ray properties involves major upgrades to instrumentation: faster and more efficient detectors; better optics; and new end-station designs integrating mechatronic concepts. But upgrades to data handling are just as crucial. Users will need to visualise data in real time to adapt their measurement strategies, and the wealth of data they record means much of it will need to be processed on site, at least in part. On top of cutting-edge data storage, backup and processing facilities, therefore, will be a new beamline control system, BLISS, a new ESRF data policy to systematically record metadata, and the extensive adoption of a common software library for data analysis, SILX. In fact, the improvements are already coming: SILX software was recently shown to cut the analysis time of an experiment at the ID01 beamline from 10 hours down to two minutes.



The future is near

Earlier now that the ESRF Council has approved several key aspects of the new design. **Jon Cartwright** takes a look.



ID10 Surface science

The new ID10 will have two experimental hutches: one with a multipurpose diffractometer and beam deflector for studies of liquid surfaces, and the other for developing the work of the former ID03 beamline, performing surface X-ray diffraction with a focus on surface chemical reactions and surface dynamics. Such dynamics data will now shrink to the millisecond range, which will allow catalytic ignition and electrochemical dissolution processes to be studied at unprecedented time resolution, and which will open up new fronts on the dynamics of nanoparticles at fluid interfaces and macromolecules in model cell membranes. A new large-area pixel detector means that determining surface and interface structures will get roughly 100 times faster. Meanwhile, improved optics will open up new possibilities for *in situ* and operando studies and sub-micron objects, as well as dynamic studies of buried interfaces.

ID24 High brilliance X-ray absorption spectroscopy

Here, one branch of the existing ID24 beamline – used for energy dispersive X-ray absorption spectroscopy (XAS) – will be converted for use with scanning extended X-ray absorption fine structure spectroscopy (EXAFS). Using a novel double-crystal monochromator, which is challenging from an engineering point of view, the new EXAFS branch will provide a variable spot size of 1 mm to 1 μm , a variable flux of up to 4×10^{13} photons per second and a time resolution down to a second. Today, *in situ* XAS applications in the geosciences are limited to conditions relevant to the Earth's lower mantle, but the new ID24 will allow investigation of the local structure of melts at the inner core boundary, at pressures and temperatures exceeding three megabars and 5000 K. Functional materials and natural samples will also be explorable at unprecedented concentrations and timescales.

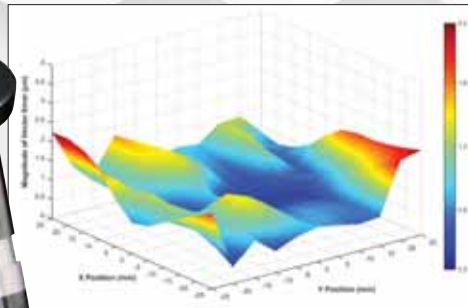
ID27 Nano X-ray diffraction in extreme conditions

Extending the existing ID27 beamline from 50 to 120 metres, the new ID27 will be a sharper, more flexible nano-focussed probe, unique in the world in terms of photon flux and focussing capabilities. Coupled with the most advanced tools to monitor the most extreme conditions of pressures and temperatures, the upgraded capabilities will largely surpass current barriers and open unique routes to probe new material properties. It will benefit many areas of research, from planetary science to fundamental physics, including solid-state chemistry, materials science and biology. For example, the small beam size will allow scientists to probe pressures higher than found at the centre of the Earth, while fast signals will capture information from short-lived samples, such as those held at above 3,000K in liquids. Materials engineering and the pharmaceutical industry will be well served.

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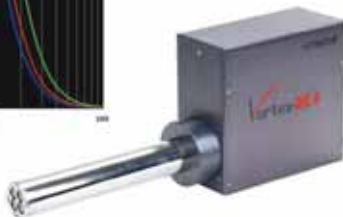
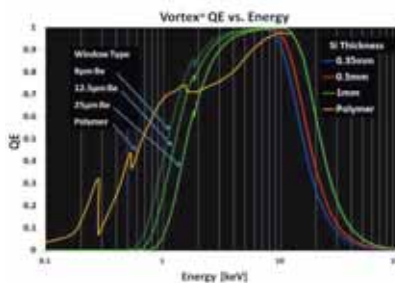
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The ideal match

For XPCS, a technique reliant on coherent X-rays, the EBSL1 beamline is a dream come true.

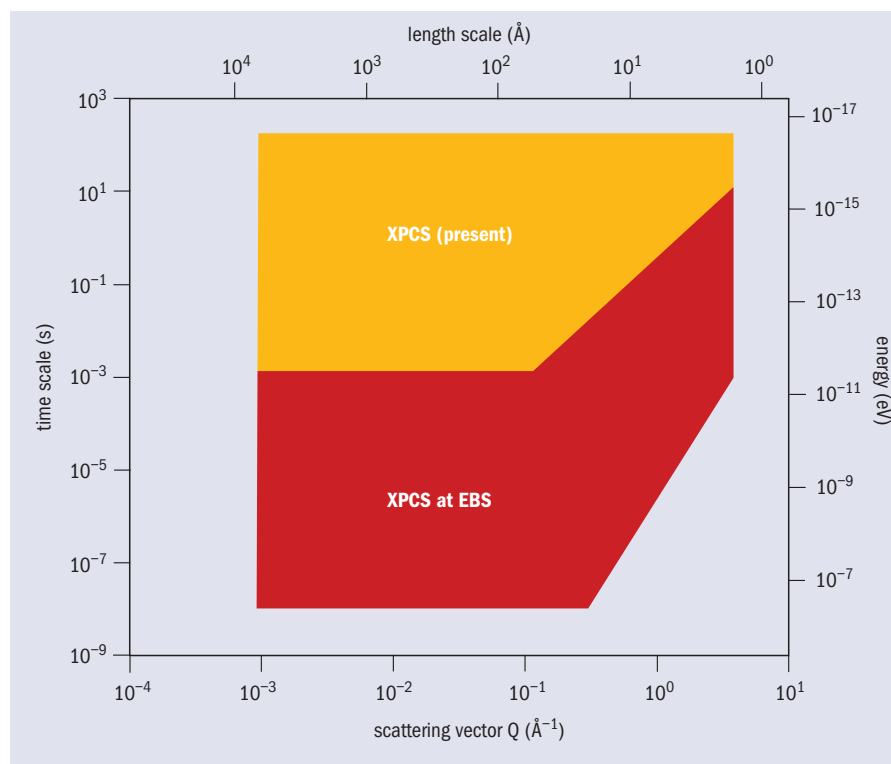
The boost in coherent flux of the ESRF's Extremely Brilliant Source (EBS) upgrade will directly benefit many X-ray techniques. Perhaps none more so, however, than X-ray photon correlation spectroscopy (XPCS). With this technique, the smallest accessible timescale improves with the square of the coherent flux. While the EBS coherence rises 100-fold, therefore, the key performance factor of XPCS advances 10,000-fold. The EBS is now set to exploit this potential with a new XPCS beamline, EBSL1.

The ESRF has always been an XPCS pioneer, being the first synchrotron in Europe to open a station for the technique in the 1990s. XPCS is used to study slowly changing phenomena (currently on the scale of 10^{-2} – 10^4 seconds) down to the nanometric and atomic scale. It can track dynamics over time, and as a function of external parameters, including temperature, illumination conditions, electric and magnetic fields, and pressure. Scientists routinely use it to measure fluctuations in a variety of colloidal, soft and hard condensed-matter systems on mesoscopic scales; at the ESRF, scientists have led the way in the observation of slow dynamics (lasting longer than 10 seconds) in alloys and glasses on atomic length scales. XPCS is the only technique to observe atomic dynamics in deeply supercooled melts and in structural glasses.

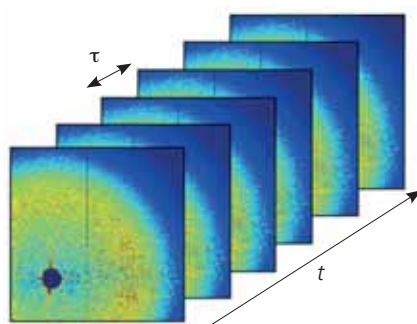
Massive boost

Yet, XPCS at the ESRF is currently operating during just half of the available beamtime at ID10, and it is strongly limited by the small fraction of coherent flux available from a third-generation storage ring. Operating at low photon energies of around 8 keV also limits experiments with radiation-sensitive samples, and sample environments requiring a higher transmission power.

Situated at the ID08 port, the new EBSL1 will easily surpass these former limitations. The EBS's boost in coherence and its extension into photon energies of 35 keV will open the door to new groundbreaking experiments, covering 11 orders of magnitude in timescales at both the nano and atomic



In XPCS, coherent light scattering from a sample forms a speckle pattern that reveals information about the intrinsic dynamics. Above: The dynamical range of XPCS before and after the EBS upgrade. Below: With the new EBSL1 beamline, the acquisition time (τ) between speckle patterns will be 10,000 times faster, allowing the observation of much more rapid dynamical processes.



level. For the first time, it will be possible to explore microsecond fluctuations in biological systems (such as proteins and water-based soft matter) and in hard materials (such as high-temperature superconductors, piezoelectrics and magnetic systems). Thanks to the use of high energies, XPCS at the EBS will unveil the particle motion of complex materials under pressure (polyamorphism in liquids and glasses), in confinement and at buried interfaces. Many of these processes are characterised by spatial and dynamical heterogeneities that evolve on the microsecond timescale. For the first time, these phenomena will be experimentally accessible over a broad length scale, ranging from single particles to particle clusters.

EBSL1 will be unique worldwide, but it will not just be a platform for XPCS. It will also revolutionise coherent X-ray diffraction imaging (CXDI), a lensless microscopy technique for three-dimensional imaging of

"The key performance factor of XPCS improves 10,000-fold."

thick specimens at nanometre resolution. CXDI at EBS promises three times higher resolution in tomographic imaging, bridging the gap between the atomic resolution of transmission electron microscopy and other electronic techniques, and the sub-micron resolution of optical microscopes. It will offer unique possibilities to study pathological changes of bone structure, human cells and growth processes of bio-minerals and micro-particles with 5–10 nm resolution.

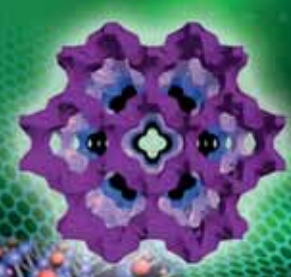
Before EBSL1 is up and running, major technical challenges, including stability requirements and detector development, will have to be solved in order to exploit the extraordinary increase in coherent flux with small diffraction-limited X-ray beams. But one thing is for certain: the instrument will open a new horizon in many disciplines across physics, material science and biology. *Beatrice Ruta, Institut Lumière Matière, Université Lyon 1 and CNRS, France*

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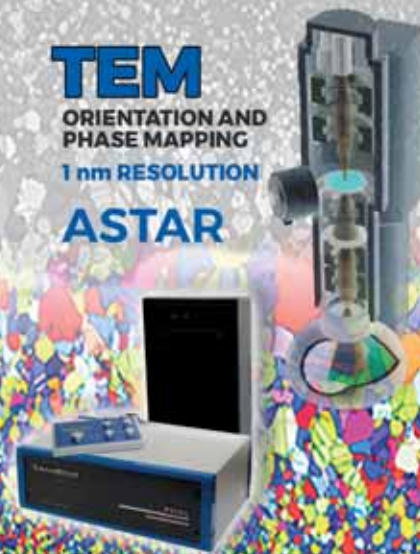
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ORIENTATION AND PHASE MAPPING

1 nm RESOLUTION

ASTAR


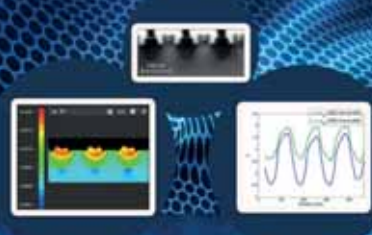


5nm Orientation image courtesy
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Tilting the scales

EBSL2 will allow users to track structural dynamics across length scales in real time.

Most hard materials – from rocks to metals, to semiconductors and even bone – have complex internal structures, often comprising several layers of substructures interwoven across multiple length scales. What's more, these hierarchical structures define many of the properties of the material. Bone is a classic example: its unexpectedly high toughness stems from the way "hydroxyapatite" nanoparticles self-assemble into highly oriented fibres up to the macroscopic scale. Understanding the material means understanding the dynamics of how the individual sub-structures interact – but this requires direct access to every length scale, within the same sample and at the same time.

This is the idea behind the hard X-ray microscope: a single instrument offering comprehensive full-field and time-resolved imaging in two, three and four dimensions of the phases, grains, domains, stress fields and defects spanning length scales from 1 mm to 10 nm.

What makes hard X-ray microscopy unique is the broad range of problems it can tackle. Using high energy X-rays we can probe sub-structures within large samples or even full devices. By combining complementary imaging techniques with the versatility of X-ray optics, we can seamlessly switch between fast overviews of microscopic features and detailed images of individual defects. In this sense, the hard X-ray microscope is analogous to a transmission electron microscope, albeit one that leverages the penetrating power of X-rays to see orders of magnitude further into the samples.

Hard X-ray microscopy was developed

"We will capture real-time movies of structural dynamics."

jointly between the Technical University of Denmark (DTU) and the ESRF as part of an Advanced Grant from the European Research Council. Six years on, at the end of 2016, this collaboration resulted in a prototype instrument at ID06. First results have provided new insights into some longstanding and fundamental problems in metallurgy, energy and biological materials. In one case, we visualised the multi-scale structure of dislocation networks in diamond, an important industrial material and, more recently, an ideal sandbox for quantum-information technologies. Crucially, its photonic properties are largely at the mercy of these defects – their intermingling networks, and the long-range strains they create.

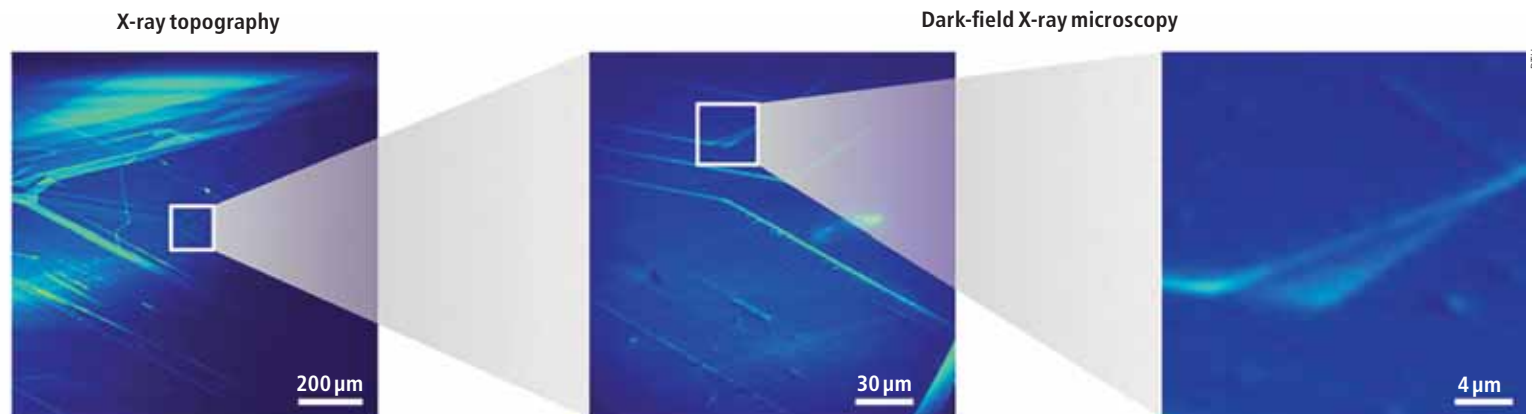
At the coarse scale, we used diffraction X-ray topography to map the location of every dislocation in the crystal. This allowed us to find a specific set of dislocation lines of interest, in this case a group of three containing kinks, including one with a clear stacking fault. Next, simply by inserting an X-ray objective lens into the beam of Bragg-

diffracted X-rays, we created a magnified, full-field image of the individual dislocations within the crystal – a technique known as dark field microscopy. Coordinated movements of the sample and optics then enabled us to quantitatively map the local crystal strain and symmetry with a resolution of 100 nm. These maps showed how dislocations and stacking faults self-organise inside the material to create buried networks characterised by long-range strain fields and lattice distortions (see figure).

Now, however, this result is set to be just the tip of the iceberg. One of the ESRF-EBS upgrade beamlines, EBSL2, located on the ID03 port, will improve spatial resolution 10-fold, while making experiments hundreds or even thousand of times faster. This means we are no longer limited to seeing static structures, but can capture real-time movies of structural dynamics as they happen across multiple length scales. In the context of diamond, it could mean tracking the 3D motions of dislocations as they form networks under stress. More broadly, it allows us to pursue much more aggressive dynamics problems, such as nucleation and material failure.

The ability to extend 3D imaging into the temporal regime is a game-changer for materials science, providing the opportunity to truly guide and validate multi-scale materials models. Such models are essential to emerging approaches to the computational design of materials.

Henning Friis Poulsen and Hugh Simons, Technical University of Denmark, and Carsten Detlefs, ESRF



A multi-scale analysis of an artificial diamond shows the dislocation network, individual dislocation lines, and a single stacking fault embedded deep within the crystal. After the EBS upgrade, with the EBSL2 beamline, it may be possible to track the 3D motions of these features with sub-second resolution as the material is stressed.



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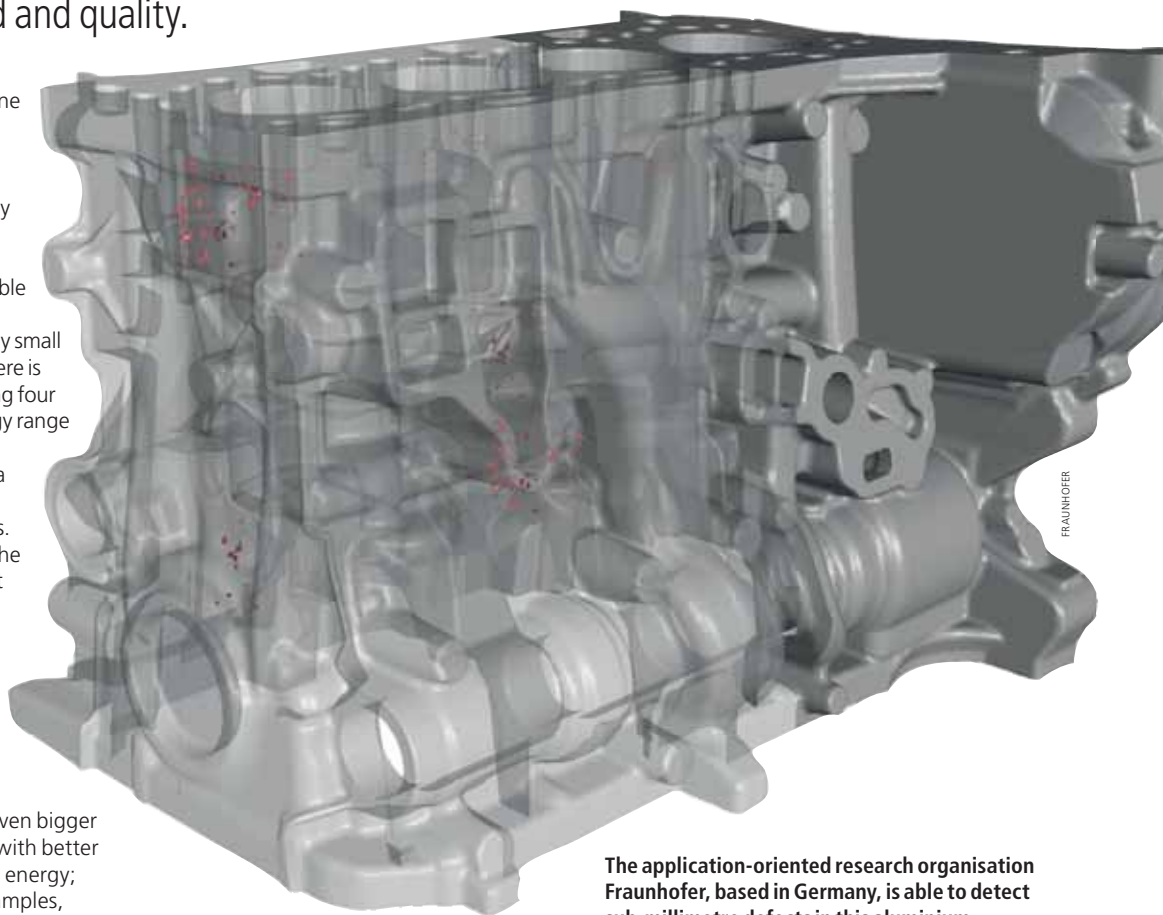
Bigger, faster, stronger, better

Thanks to EBSL3, microtomography could be targeting objects the size of car engines at unprecedented speed and quality.

Synchrotron microtomography is one of the primary three-dimensional characterisation techniques at the ESRF. Simple, non-destructive and – compared with conventional X-ray microtomography – fast and high-quality, it has evolved steadily over the past 20 years, and is now available on several beamlines. Where once there was a tool that could scan only small samples using X-ray absorption, there is now a tool for sample sizes spanning four orders of magnitude, over an energy range 10–250 keV, with multiple contrast mechanisms and in times down to a tenth of a second, even in complex *in situ* or *in operando* environments. Indeed, versatility has been key to the success of the microtomography at the ESRF, and explains the breadth of applications covering material science, cultural heritage, biology, biomedical research, geology, and notably industry.

Despite this success, however, synchrotron microtomography is reaching its limit. Industrial and academic scientists want to scan even bigger samples, at higher resolution and with better phase-contrast capabilities at high energy; they want to scan large series of samples, sometimes with multiple resolutions and with high throughput.

An entirely new beamline, EBSL3 – together with a refurbishment of the ESRF's present iconic microtomography beamline, ID19 – will eclipse these limits. Making the very best of the ESRF's Extremely Brilliant Source (EBS) upgrade, EBSL3 will be a 220 metre-long beamline that will perform hierarchical imaging (a voxel size of 100 μm down to 1 μm) of medium to large objects, using propagation phase contrast with high levels of automation. Thanks to the EBS's exceptional coherence, high-quality propagation phase contrast of up to 350 keV will be possible. A 40 metre-long



The application-oriented research organisation Fraunhofer, based in Germany, is able to detect sub-millimetre defects in this aluminium engine cast using high-energy conventional X-ray tomography. With the EBS's EBSL3 beamline, it would be able to detect defects in the 20-micrometre range, and then examine them on a microscopic scale.

experimental hutch will allow propagation distances up to 35 metres, in order to obtain high levels of phase contrast even at maximum energy and low resolution. All this will be possible for samples 100 times bigger than today (up to 2.5 metres vertically by 1.5 metres horizontally), for weights up to 300 kg. The whole beamline is designed to be rapid, versatile, easy to use, largely automatic, and robust, to ensure high level of reliability and high throughput.

Parallel upgrade

While EBSL3 is being built, the upgrade of the upgrade of ID19 will lead to it becoming an undulator-based beamline that reaches higher resolutions with higher throughput, as well as at high energies in the sub-micron range. X-ray imaging is one of the ESRF's best industrial tools, and so EBSL3 and ID19 have been designed together with industrial needs in mind. High throughput will allow rapid surveys of, for example, pills and small metallic devices on ID19, or batteries, mechanical items and food products on EBSL3. On the other hand, the large, high-energy beam and hierarchical capabilities of EBSL3 makes it ideally suited to study small aspects of big

samples, such as defects in aluminium engine casts (see figure), or the microscopic structure of large composite devices, with world-leading sensitivity and resolution.

Academic fields, from cultural heritage to biomedical applications, will greatly benefit too. Today we can scan a *Tyrannosaurus rex* arm, a cat mummy, or a small human organ; tomorrow we will be able to scan a complete *T. rex* skull, a complete human mummy in its wooden sarcophagus, or even a complete human body. Inside any of these we will see things no one has ever seen before without destroying the specimen, down to the micrometre scale.

This large-scale project will continue to make the ESRF's synchrotron microtomography capabilities truly unique, benefitting a host of industrial and academic scientific topics – including many we are not yet even aware of.

Paul Tafforeau, ESRF

“These synchrotron microtomography abilities will be truly unique.”

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The serial approach

With EBSL8, synchrotron serial crystallography is coming to the ESRF with full force.

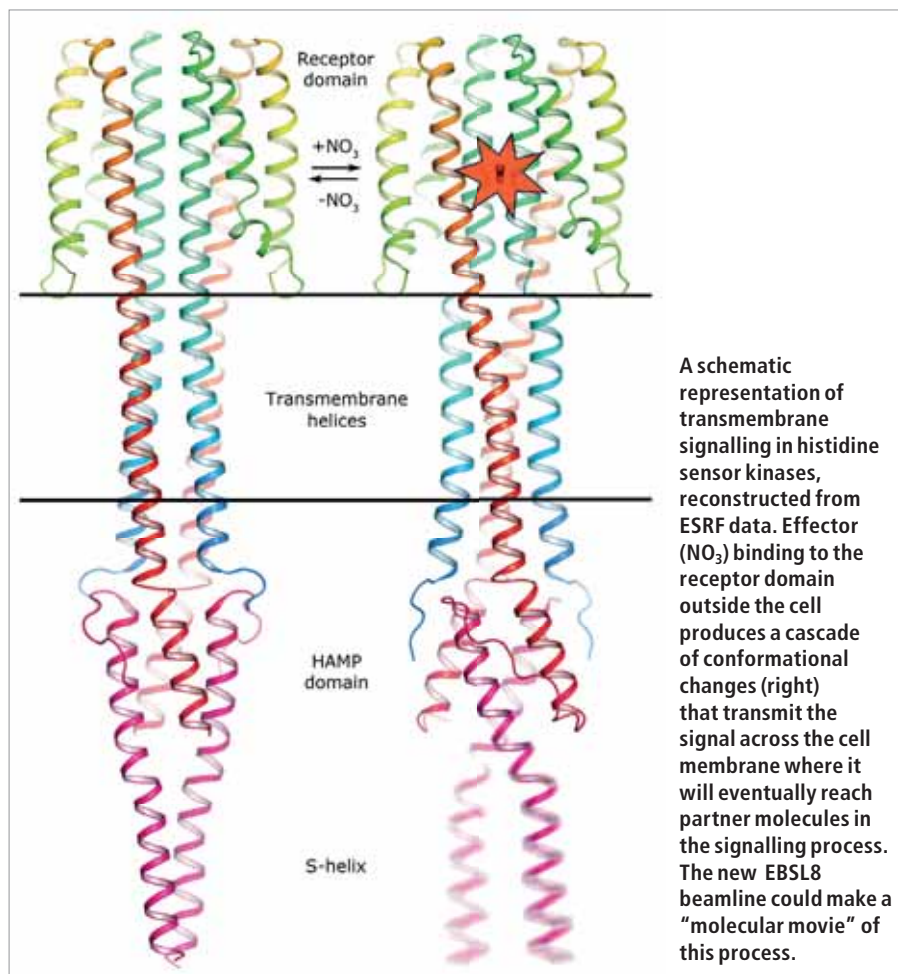
The harder you look, the more damage you do: this unalterable truth of X-ray crystallography has always prevented crystals of biological macromolecules from divulging all their structural information before they are destroyed by radiation. Serial crystallography (SX), performed at either X-ray free electron lasers (XFELs) or synchrotron sources, helps overcome this limitation. The technique involves taking diffraction data from numerous microcrystals – hundreds of thousands, in some cases – in order to assemble a complete dataset, piece by piece.

SX was developed at XFELs, where just a single femtosecond pulse is usually enough to destroy a crystal and where it is known as serial femtosecond crystallography (SFX). The first successful applications involved sending crystals through the pulsed X-ray beam in a liquid jet, at a density optimised for a maximum “hit rate” when crystal and X-ray beam coincide. Later, investigators found that less crystalline material was needed when the crystals were contained in a viscous liquid, or even on a fixed target containing specially designed crystal holders from which, in favourable cases, multiple diffraction images could be collected per crystal.

Now, following successful proof-of-principle synchrotron experiments at the ESRF and elsewhere, the ID29 beamline is being rebuilt for synchrotron serial crystallography (SSX) as part of the ESRF’s Extremely Brilliant Source (EBS) upgrade. Although an ESRF team led by Daniele de Sanctis is still finalising the technical details, the new beamline, EBSL8, promises to carry out entirely new types of experiment at a synchrotron source.

The beamline will have a variable focal spot size of 0.5–10 µm, tuneable over the energy range 10–30 keV, with a flux of some 10^{16} photons per second. That equates to a brilliance many orders of magnitude higher than current macromolecular crystallography beamlines. The smallest beam size will allow SSX data collection from micron or even sub-micron sized crystals.

SX has several advantages over conventional crystallography. One is that SX facilitates data collection at room temperature rather than the usual cryogenic temperatures, which sometimes obscure functionally important conformations. Also, SSX at EBSL8 will allow exploration of the potentially mitigating effects on radiation damage of very fast, very high dose rate data-collection at synchrotron



sources (*Acta Cryst.* **D70** 1248). Perhaps the biggest advantage, however, is that SX allows time-resolved experiments to be carried out. The most exciting recent example of this has been the SFX studies on photosystem II – the enzyme that splits water molecules in photosynthesis – where researchers have obtained structures of different states in the photocycle (*Nature* **513** 261).

Crucial timescale

The EBSL8 SSX beamline will produce similar insights on many biological systems. While the exploration of femto- and nano-second timescales will remain the domain of XFELs, EBSL8 will take full advantage of the EBS upgrade to allow crystallographic studies at sub-millisecond time resolution, a timescale on which many conformational changes in biological macromolecules take place.

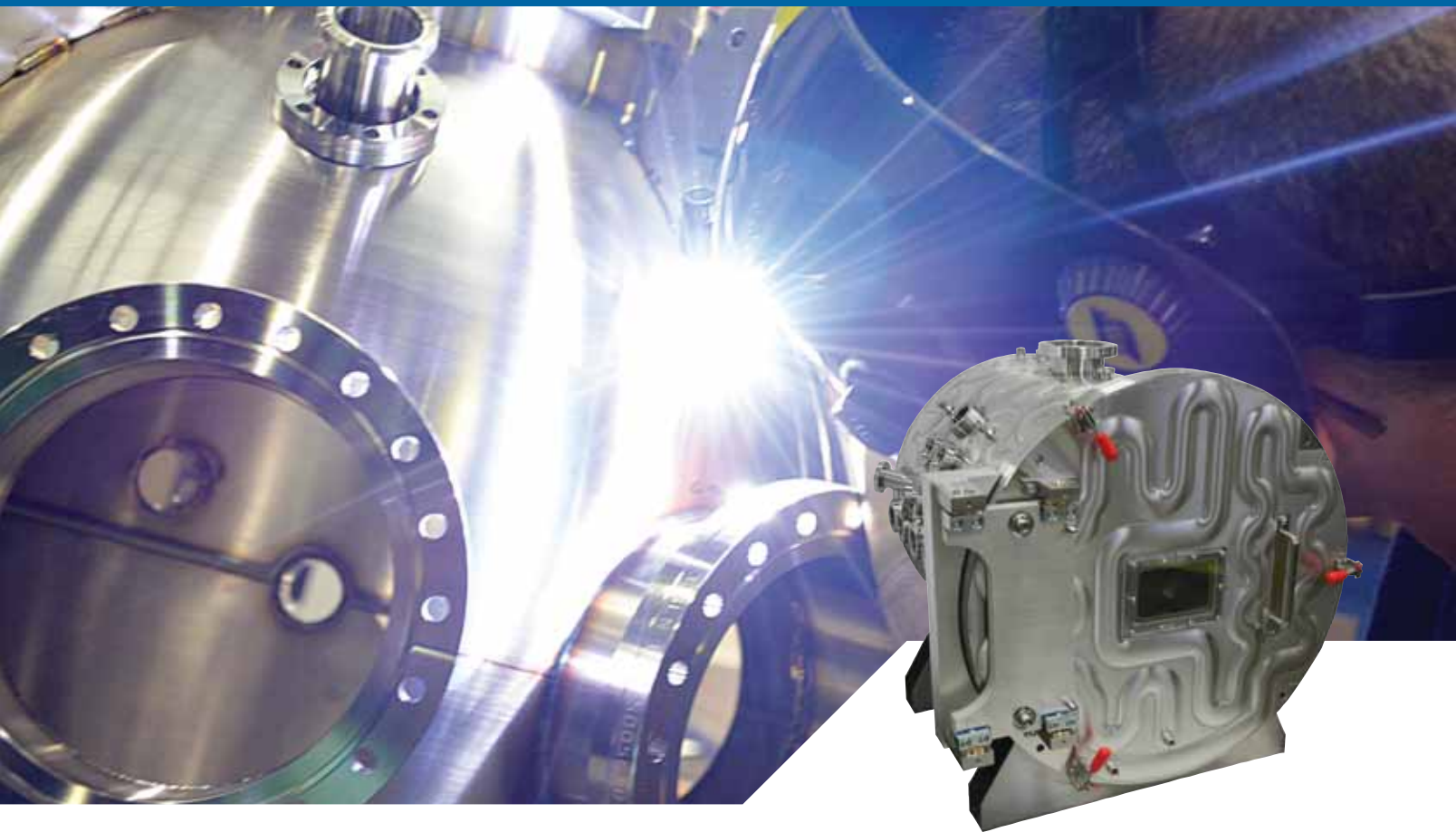
Other potential experiments on the new beamline include optimised “mesh and collect” experiments, in which a complete data set is compiled from diffraction images from hundreds of crystals contained on the same sample support. EBSL8 will also naturally expand industrial applications with microcrystal-based rational drug-design

programmes that should require lower consumption of both potential drug molecules and their biological targets. Moreover, SSX-derived room-temperature crystal structures will provide more information of the dynamics of drug binding.

Examples of other systems where SSX can provide crucial structural information abound. These include bacterial sensor histidine kinases, which play an important role in signal transduction. This year, a group led by Valentin Gordeliy at the Research Centre Jülich in Germany, including scientists at the ESRF, determined crystal structures of a large fragment of the *Escherichia coli* nitrate/nitrite sensor NarQ in its ligand-bound and ligand-free states. The results revealed a signalling mechanism based on a cascade of conformational changes that are transmitted from outside to inside the cell (*Science* **356** 1043, see figure). EBSL8 will make it possible to study this signalling mechanism in much greater detail, including the production of a “molecular movie” of the conformational changes involved.

Andrew Leslie, MRC Laboratory of Molecular Biology, Cambridge, UK, and Gordon Leonard, ESRF

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The beauty of small things

Titia Sixma of the Netherlands Cancer Institute reveals how modern techniques are helping us see life's tiniest components in ever more detail.

A toxin that gives people traveller's diarrhoea may not sound like the most glamorous topic for a PhD. For a true structural biologist like Titia Sixma, however, the interest always lies in the finer details: a molecular structure of 30 beta strands in five-fold symmetry, within an asymmetric heterodimer. "It's a beautiful protein, it's gorgeous," she says.

In fact, Sixma's *Escherichia coli* protein structure revealed valuable information as well as beauty, for its unusual architecture suggested ways in which it could enter human cells. This was all the more important since the diarrhoea toxin is closely related to another that results in cholera, and so perhaps it was little surprise that the crystallography results, taken at the European Molecular Biology Laboratory's beamlines at the Hamburg synchrotron HASYLAB, found their way into the journal *Nature*. That was way back in 1991, three years before the ESRF was inaugurated – a time when structural biology was on the cusp of a rapid expansion. Sixma, a chemist by training, was lured by the prospect of hard data in a field traditionally swamped by complexity.

New horizons

Sixma continued to study the human process targeted by the toxin in her postdoc, before quickly taking up a group-leader position at the Netherlands Cancer Institute in Amsterdam, where she has stayed since. "I was lucky because structural biology was expanding, and positions were appearing," she recalls. There still are relatively few structural biology groups in the Netherlands, yet Sixma has come to be regarded as one of the leading scientists in her field.

One of the reasons for that is her work on DNA mismatch repair, for which she came to the ESRF in the late 1990s to make use of its crystallography beamlines BM14 and (now decommissioned) ID14. Mismatch repair happens in



Titia Sixma in brief

Born: 1962, Utrecht, the Netherlands.

Education: BSc chemistry, University Utrecht (1987); PhD structural biology, University of Groningen (1992).

Career: Postdoc Yale University, New Haven, Connecticut, US (1992–1994); group leader (1994–present) and division head (2008–present) Netherlands Cancer Institute; affiliate professor (2004–present) of structural biology, Erasmus University Rotterdam.

"Having access to crystallography and cryo-EM is very powerful."

everyone's cells when they divide, to repair the few mistakes that occur during DNA replication. But sufferers of Lynch syndrome have mutations in the genes encoding the mismatch-repair proteins, and therefore have a predisposition to colon cancer. Sixma has studied how the proteins work, to understand how these enzymes find these very rare errors and then only repair the newly synthesised strand (*Elife* doi:10.7554/eLife.06744)

Another high point in Sixma's career has been the study of the targets of molecules, such as nicotine, that affect signal

transmission in the nervous system. In humans and other mammals, these targets are membrane proteins known as nicotinic acetylcholine receptors (nAChRs), and biochemists have long tried to uncover how they bind to important ligands. In the early 2000s, Sixma and others used the ESRF to reveal how an analogue of the nAChRs' ligand-binding domain binds its ligand: so called acetylcholine-binding protein, found in snails (*Neuron* 41 907). "There were many things known about the receptors before, but with our structure everything fell into place, and

stood out in three dimensions," she says. Today, the structures of the mammalian receptors are only just being solved, although Sixma is no longer pursuing the topic.

Now head of biochemistry at the Netherlands Cancer Institute, Sixma is well aware of the strong collaboration between the Netherlands and the ESRF. They share an ESRF block allocation group with structural biologists in Belgium, and the two countries have a dedicated collaborating research group beamline at BM26, DUBBLE. They have been coming regularly to ESRF for many years. Recently, however, Sixma has been "extremely pleased" with the ESRF's automated MASSIF beamlines. "At the moment our default is to send things to MASSIF first. It's really efficient. We got a couple of really nice structures back recently with minimal effort."

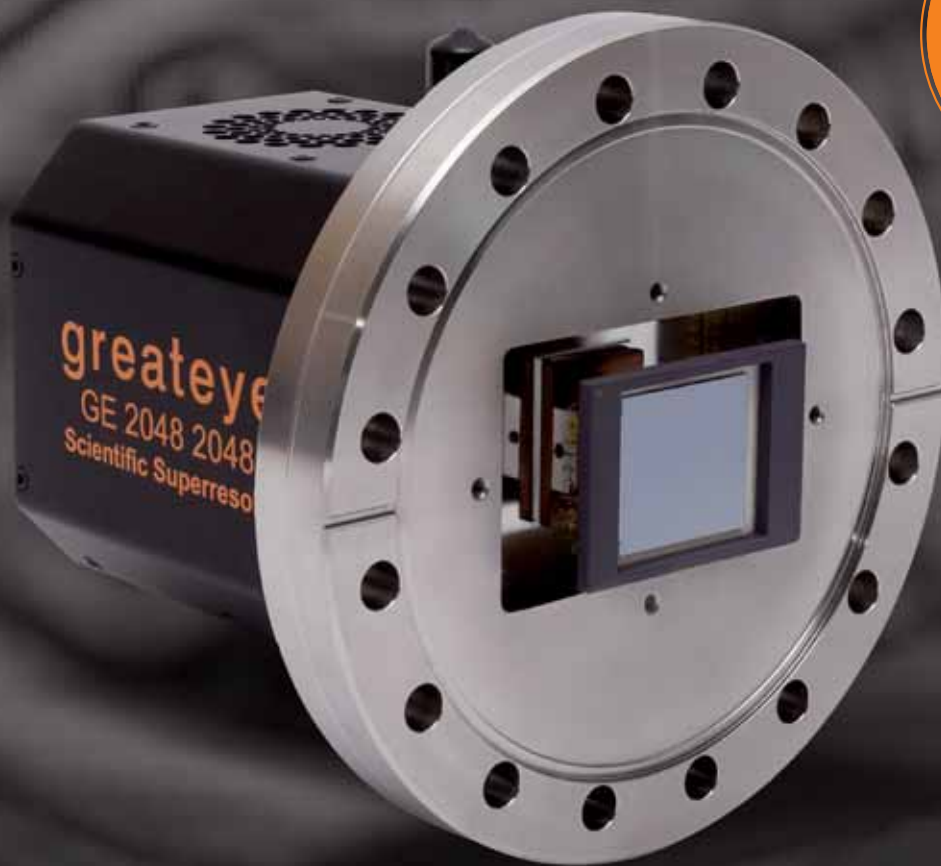
Cool ideas

With the advent of cryo-electron microscopy (cryo-EM, see p13) at the ESRF, Sixma is eager to further another of her key research areas: ubiquitin conjugation, a process in which one or more ubiquitin proteins hang on to a target protein to change its fate. In the last few years, she and her colleagues have determined a number of structures of deubiquitinating enzymes to understand how they are regulated via "allostery", or the transmission of binding effects from one site to another. This could help in targeting them for cancer or neurodegenerative drugs, but some of the latest proteins are proving hard to crystallise. "We've been banging our heads against the wall for too long, not getting crystals of the larger and more transient complexes, where we want to capture the molecules in action," says Sixma. "With cryo-EM we still have to trap the right transient state, but at least then we don't need to crystallise them. Having access to both crystallographic methods and cryo-EM is very powerful." *Jon Cartwright*

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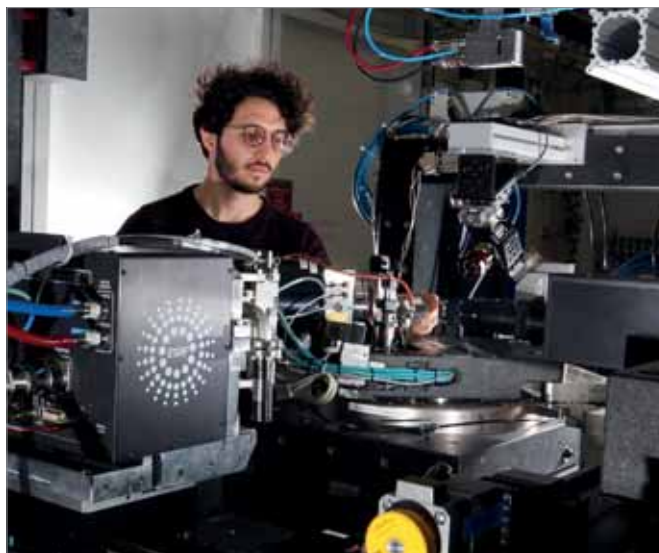
Steel dives into dark-field microscopy

Research centre for the application of steel turns to the ESRF to improve alloys.

In traditional metallurgy, microstructures can only be studied in two dimensions in the finished product. "When I heard that we could follow metallurgical reactions live and in 3D, I was immediately convinced that this was the way to go," says Roger Hubert, the chief scientific officer at OCAS, a metallurgical research centre in Belgium.

Meanwhile, OCAS has funded a postdoctoral researcher, Can Yildirim, to investigate the effects of nitrogen alloying in steel at the ESRF. Nitrogen can boost various steel properties such as strength and hardness, but this strongly depends on controlled nucleation and growth of nitride phases. Upon cooling down, the solubility of nitrogen decreases and excess nitrogen precipitates as iron nitrides. These precipitates are desirable, so long as their phase, morphology and size can be controlled.

To explore these iron-nitride precipitates, Yildirim turned to ESRF beamline ID06, where he could perform dark-field X-ray



Yildirim at work on ID06.

microscopy. Unlike bright-field microscopy, where a sample is seen on a bright background thanks to direct illumination, dark-field microscopy detects only light that is scattered from Bragg diffraction. The result is a non-destructive technique that allows 3D mapping of orientations and stresses within

crystalline materials from 100 nm to 1 mm, even at high temperatures. "We're basically able to see the internal structure of steel alloys and follow which processes occur on a sub-micron scale in *operando* conditions," says Yildirim. The technique will become even more powerful with the Extremely Brilliant

Source upgrade of the ESRF.

Yildirim's model samples are used to design the methodology, and especially the heat-treatment cycle management in alloys. In conventional steel processing, nitrogen alloying is challenging due to the element's limited solubility during casting and solidification. Alternatively, nitriding as thermochemical treatment on the final material (also called case hardening) can be used to significantly improve surface and bulk properties. Thanks to dark-field X-ray microscopy, Yildirim and others can look inside the material, whether it is inside a furnace or other sample environment, and see how the microstructure evolves during processing. That is what makes it ideal for industrial clients such as OCAS.

In July, Yildirim and his colleagues also conducted a first experiment on non-grain-oriented electrical steel samples, and were moving on to a second experiment at ID06 as this issue went to press.

Jon Cartwright

Movers and shakers



Edith Heard has been selected to be the fifth director general of the

European Molecular Biology Laboratory (EMBL). Currently the director of the Genetics and Developmental Biology Unit at Institut Curie in Paris, and chair of epigenetics and cellular memory at the Collège de France, also in Paris, Heard has research experience in epigenetics and developmental biology, as well as chromosome and RNA biology. She will take over from the EMBL's existing director general, Iain Mattaj, in January 2019.



James Naismith has been appointed director of the new Research Complex

at Harwell (RCAH), a multidisciplinary lab that has facilities for researchers in the life and physical sciences, including those using the adjacent Diamond Light Source. A long-standing member of Diamond's board of directors, Naismith is an expert in using X-ray crystallography of proteins to investigate the pathways of disease. He retains a position at the University of Oxford, where he is also co-interim leader of the new Rosalind Franklin Institute.



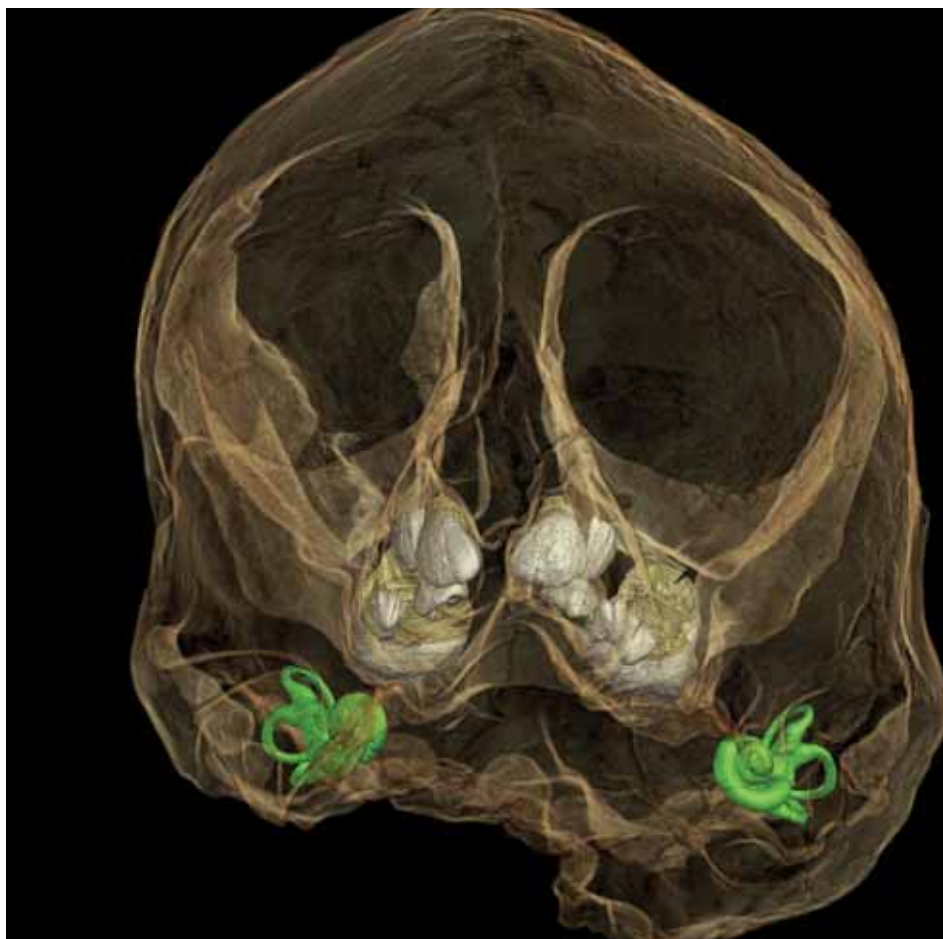
ESRF users **Paul Shearing** (left) of University College London in the UK and **Donal Finegan**

of the National Renewable Energy Laboratory in Colorado, US, have won a coveted "Safety and Security" award from *The Engineer* magazine for their research on the failure of lithium-ion batteries. Judged by a panel of leading UK engineers, the awards draw attention to work that is "innovative, collaborative and likely to have an impact in their field of application", and are designed to snapshot of some of the trends and technologies defining modern engineering.



Michael Hahn, former leader of the ESRF vacuum group, has taken sabbatical

leave to become section leader of vacuum technology at the Paul Scherrer Institut (PSI) in Villigen, Switzerland. At the PSI he will be in charge of vacuum systems of large research infrastructures, consisting of proton machines, a synchrotron light source and a free electron laser under construction. He will also work on the definition and preparation of the forthcoming improved Swiss Light Source, SLS-2, which is expected to be built over the period 2021–2024.



Family resemblance:

Meet Alesi, a 13 million year-old fossilised ape who – thanks to research carried out at the ESRF – has given strong clues about the common ancestor of all living apes and humans. Alesi's skull was dug up in the Turkana basin of northern Kenya three years ago, and although it looks like that of a gibbon, it is actually that of a primate in the genus *Nyanzapithecus*. Performing X-ray microtomography at ESRF beamline ID19 on Alesi's teeth, scientists revealed that the primate was just one year and four months old when it died; meanwhile, in the inner ears (highlighted green) the microtomography revealed cochleae (for hearing) and semi-circular canals (for balance). These structures, and the size and shape of the teeth, suggest that Alesi was an ape, and specifically an evolutionary cousin to that line of ancestral apes from which humans split off some seven million years ago (*Nature* **548** 169).

In the corridors



First users at XFEL

First experiments began at the European X-ray free-electron laser (XFEL) in Hamburg, Germany, on 20 September, nearly three weeks after the facility was inaugurated. Highlighting the range of methods available at the XFEL, the experiments include the use of different spectroscopy methods to track ultrafast reactions and electron movement in model molecules, the probing of organic light-emitting diodes, and the investigation of nitrogen and oxygen recombination in the muscle tissue protein myoglobin. "This is a very important event, and we are very happy that the first users have now arrived," said Robert Feidenhans'l, the European XFEL's managing director.

China launches X-ray telescope

China's most recent foray into space-based science is an X-ray satellite, the Hard X-ray Modulation Telescope (HXMT), which launched in June. Designed to study some of the universe's most energetic phenomena, such as gamma-ray bursts, neutron stars and black holes, HXMT actually carries three X-ray telescopes operating at different energies, from 20 to 200 keV. It is the latest of a series of instruments launched as part of China's highly active space programme, including probes of dark matter, microgravity and long-range quantum entanglement.



Briefest X-ray flash

A group led by Zenghu Chang at the University of Central Florida in Orlando, US, has smashed its own record for the fastest X-ray pulse. Lasting just 53 attoseconds (10^{-18} seconds), the pulse was 14 attoseconds shorter than the group's previous record set in 2012, and is equivalent to the time it takes light to travel one-thousandth the diameter of a human hair. It was generated by interacting an intense infrared laser with a noble gas, and is thought to be important because it reaches the "water window", where carbon atoms absorb strongly but water does not (*Nat. Comm.* **8** 186).

X-rays help restore wind instruments

Scientists at the Paul Scherrer Institut (PSI) in Switzerland have used X-ray and neutron tomography to guide the restoration of more than a dozen 16th-century Italian wind instruments. The resultant three-dimensional images revealed the internal structure of the components made of wood, metal and leather, in particular the shape of the finger holes and the borehole, and their deterioration over hundreds of years. "Using this data, we can now reconstruct for the first time what they would have sounded like when first played," said Giulia Festa at the University of Rome Tor Vergata.



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