# ESRF news

December 2019

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Common user questions answered

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Tomography seduces the world of modern art

## HIDDEN WAVES

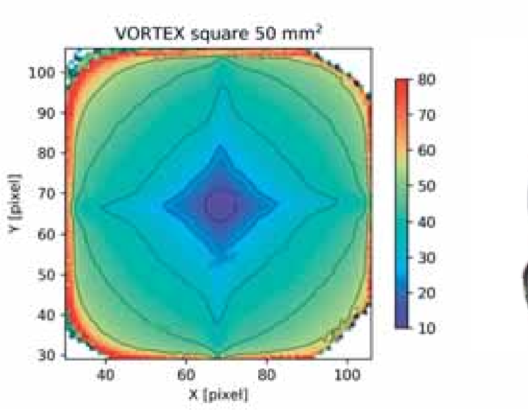
**The strange signals coming from high-temperature superconductors**

ESRF

# Exciting Innovations for Research Scientists

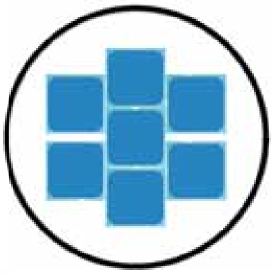
# The VORTEX® ME-7 SDD

## 7 x 50 $mm^2$ SDD sensors in a probe diameter of 38.1 mm



VORTEX ME-7 SDD Rise Time Mapping

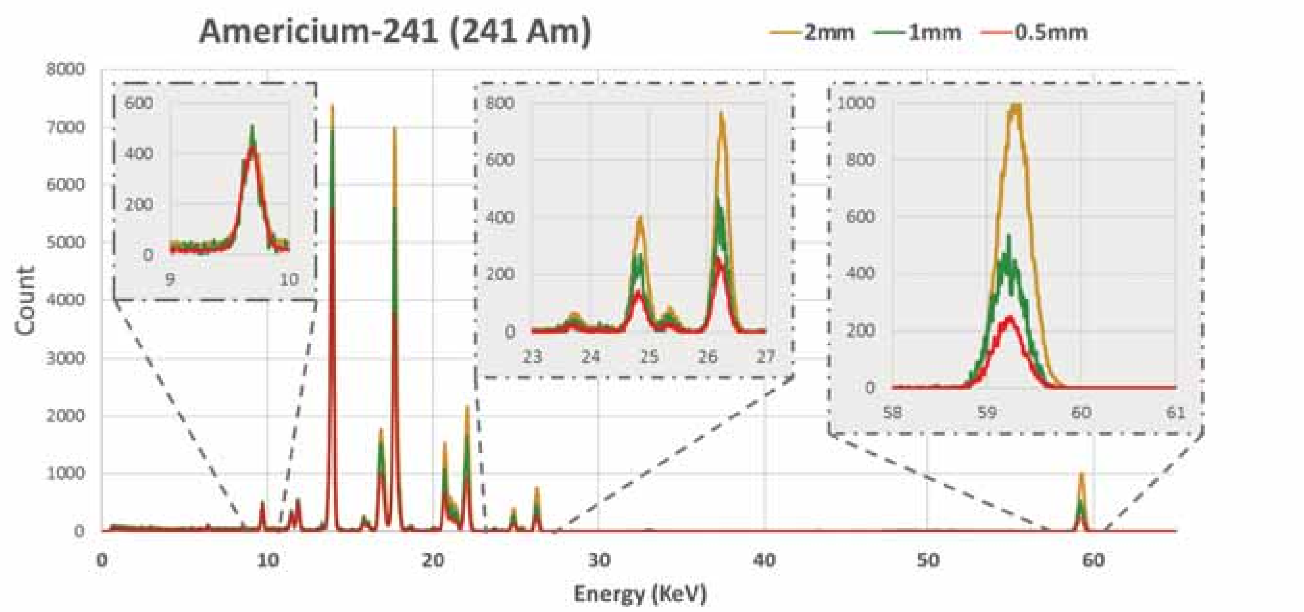
VORTEX ME-7 SDD— 38 mm diameter Detector - the same as the current VORTEX ME-4 SDD



38 mm

## AND

# The VORTEX® 2mm thick SDD Sensor



## Contact us to discuss the future

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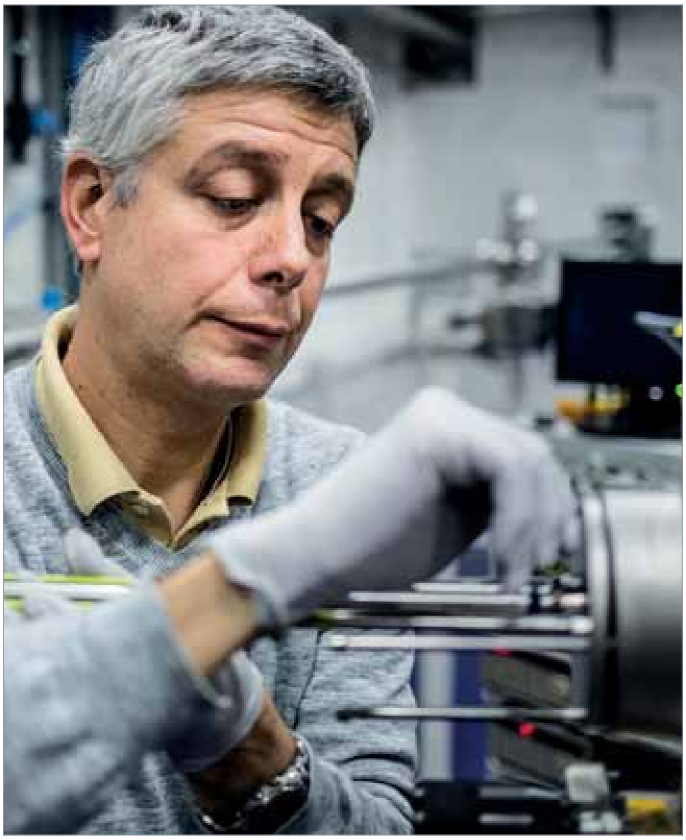

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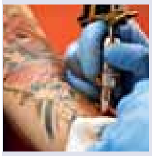
## EDITORIAL



## NEWS

## INDUSTRY

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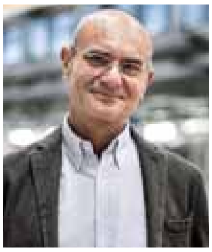
## BEAUTY OF SCIENCE

# Unveiling complexity



**Francesco Sette**
ESRF director-general

The 2019 Nobel Prize in Physics has been awarded to James Peebles for his work on the understanding of the universe, and to Michel Mayor and Didier Queloz for their observation of the first exoplanet orbiting a Sun-like star. Such discoveries have given new impetus and interest to the exploration and understanding of the complexity of the universe.

The 2019 Nobel Prize in Chemistry has been presented to John B Goodenough, M Stanley Whittingham and Akira Yoshino for the development of lithium-ion batteries, which have become a ubiquitous feature of all consumer electronics and electric vehicles, and have opened new perspectives for the gigantic challenge of sustainable energy production, storage and management.

These studies have inspired many scientists and have opened new fields of research that require specific state-of-the-art tools to manage and unveil complexity, both before the discovery itself and after, for the numerous applications that follow.

Synchrotron radiation facilities offer unique tools to push back the boundaries of scientific investigations into new materials and living matter. Synchrotron light sources also play a key role in stimulating innovation and enhancing competitiveness for industry.

> “Both Nobels recognise research areas that the ESRF contributes to”

Both areas of research recognised by the 2019 Nobel Prize Committee are an integral part of the ESRF science programme, and ESRF users contribute to them by making use of cutting-edge and often unique X-ray characterisation techniques. Diffraction tomography and high-speed imaging allows researchers to understand how lithium-ion batteries operate and fail, and so to help advance a wireless and fossil-fuel-free society. Meanwhile, ESRF extreme-temperature and pressure facilities allow scientists to better understand the chemical composition and atomic structures of extrasolar worlds.

Now, the ESRF is preparing to launch an innovative new tool: the Extremely Brilliant Source (EBS). The EBS will be the first high-energy fourth-generation synchrotron, with X-ray performance increased by a factor of 100 compared with third-generation light sources. It will open new perspectives for X-ray science and contribute to the understanding of complexity in living and condensed matter. These new X-ray-based investigation tools will help scientists tackle global challenges for a sustainable economy and society. The countdown has started for the restart of the machine and the beamlines (see p19). See you in 2020 for new discoveries!

**ESRF news**

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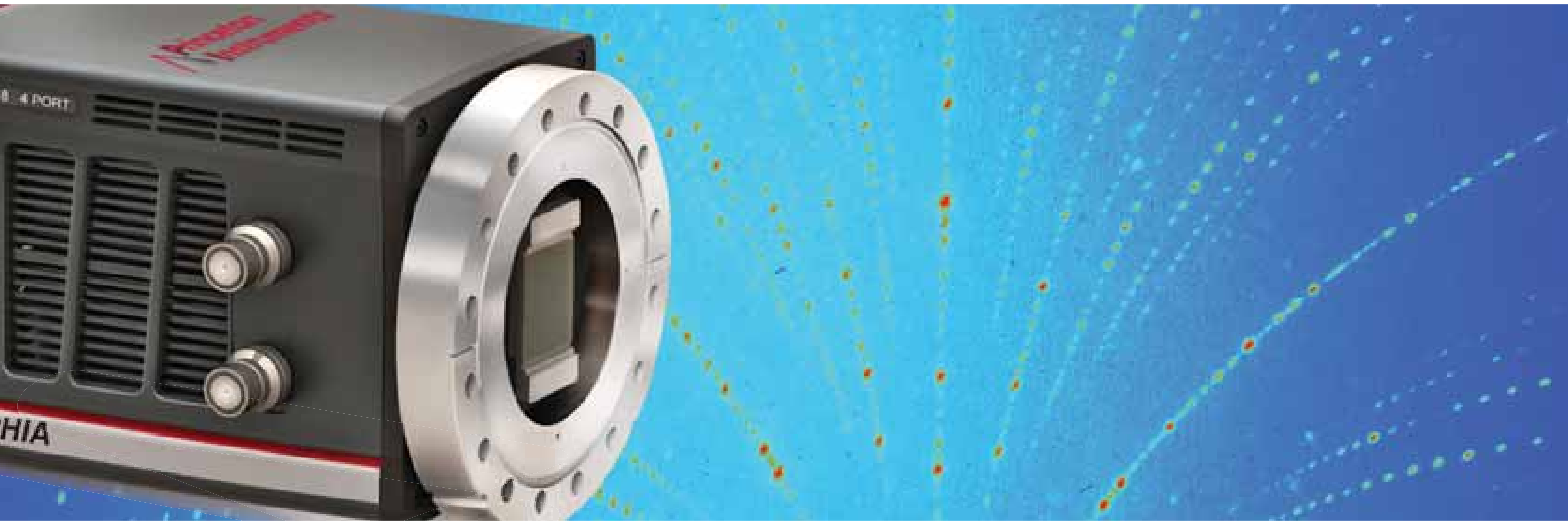
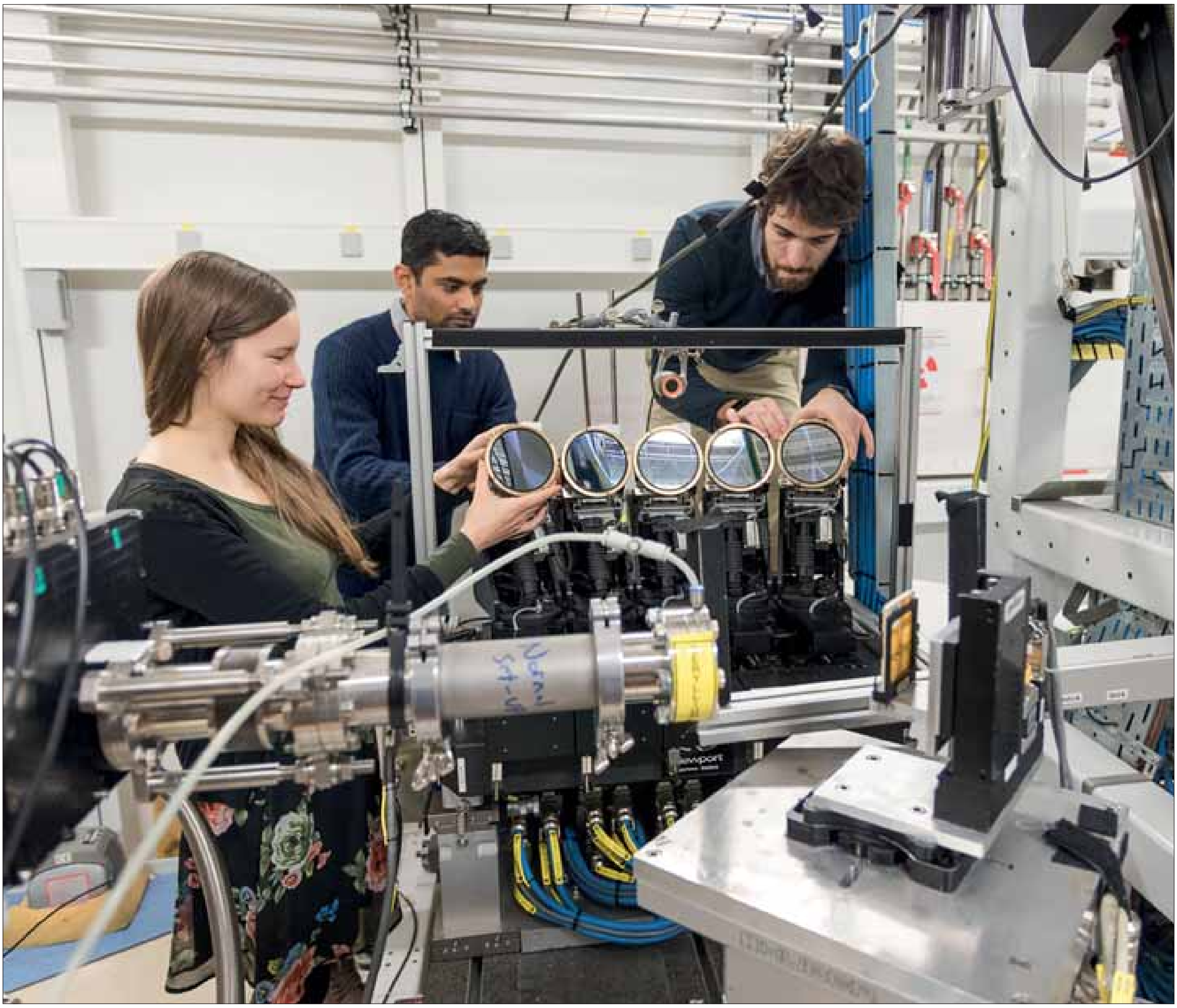




ESRF/P JAYET

# Students needed for InnovaXN

> *"This is a special opportunity for ambitious students"*

Students will soon be invited to apply for a doctoral training programme that matches synchrotron X-ray and neutron research to the needs of European industry. Known as InnovaXN, the programme will give students the opportunity to understand challenges in industrial research and development while exploiting the advanced characterisation techniques of the ESRF synchrotron and the ILL neutron source in Grenoble.

"This is a special opportunity for ambitious students to get experience and training in a modern mixed-science, industry and technology environment, opening a wide range of career paths," says Jean Susini, the ESRF director of life-sciences research and the co-director of InnovaXN.

InnovaXN officially launched in October this year, having received more than 60 project proposals from European companies and their partners. Currently, the programme's selection committee is whittling this number down to 20 PhD thesis topics. The first of two recruitment calls for applicants opens in February next year, and will close at the end of March. Taking place mostly at the ESRF and the ILL, and co-supervised by those facilities' scientists with scientists from industry and academia, the work will begin in September and last for 36 months.

InnovaXN is supported by the European Commission as part of its Marie Skłodowska-Curie Actions (MSCAs), a Horizon 2020 programme that supports young scientists in their early careers. As a "COFUND" MSCA, the project's funding is split, so that the Commission provides half of the salary costs, while the remainder is provided by the ESRF, the ILL and the industrial partners.

- Visit www.innovaxn.eu

# Continent-sized oceanic crust could exist in Earth's interior

Unusual seismic signals from the Earth's lowermost mantle could be generated by continent-sized regions of buried oceanic crust, according to a study conducted at the ESRF. The study, which was made possible thanks to the ESRF's large volume press, suggests that huge volumes of crust have sunk nearly 3000 km – almost to the Earth's core – due to plate tectonics.

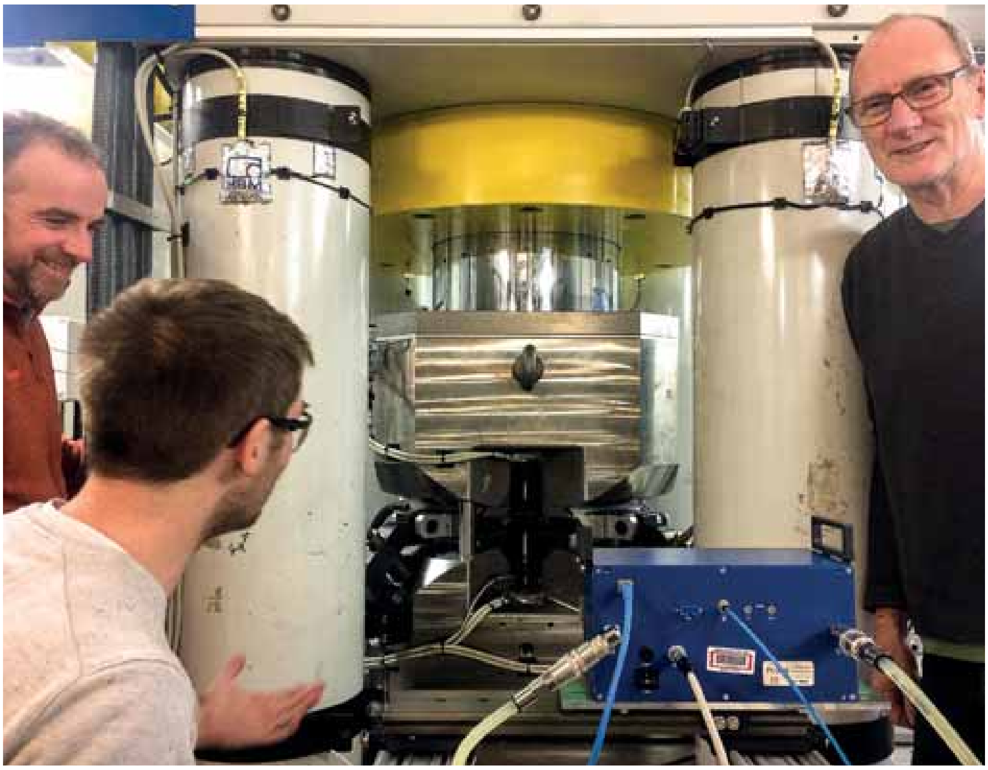
For about three decades, scientists have known that seismic waves originating in the Earth's lowermost mantle travel through huge regions in which their velocity is unusually low. One idea is that these regions are a different type of mantle that formed along with the rest of the Earth 4.5 bn years ago, but another is that they began life as oceanic crust and gradually sank. Oceanic crust is made largely of basalt, a very dense rock that is prone to sink, or subduct, when it is forced into lighter rock at a collision with another tectonic plate. Once under the extreme temperatures and pressures of the Earth's interior, basalt forms minerals including calcium silicate perovskite (CaPv), which is believed to be the third most abundant mineral at depths below 660 km.

> *"The team took advantage of the large volume press at ID06, a unique facility in Europe"*

The trouble with testing this theory is that CaPv does not exist in ambient conditions, making its seismic properties hard to study. As a result, a team including scientists at University College London (UCL) and the University of Leeds in the UK, as well as the University of Oslo in Norway, and the University of Bayreuth in Germany, took advantage of the large volume press at the ESRF's ID06 beamline – a unique facility in Europe – to expose samples of CaPv to pressures of greater than 100,000 atmospheres and temperatures higher than 1000 °C. Meanwhile, the team could record the samples' crystal structures via X-ray diffraction and measure the time taken for ultrasonic waves to travel through at different pressure–temperature combinations.

The speed of the waves was lower than expected, and suggested that CaPv at extreme pressures and temperatures could be the source of the low-velocity seismic waves recorded from the lowermost mantle (*Nature* **572** 643). "[The] continent-sized regions of anomalously low velocity seen at more than 2500 km in depth beneath the Earth's surface can be explained as areas with moderate enrichment of recycled oceanic crust," says group member John Brodholt of University College London.

Left to right: ESRF scientist Wilson Crichton together with Andrew Thomson and John Brodholt of UCL at work on ID06's large volume press.



ANDREW THOMSON/UCL

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ESRF

## ESRF streamlines future science

The ESRF has launched a project to capitalise on the new scientific opportunities that will be opened up by its forthcoming Extremely Brilliant Source (EBS). Backed by the European Commission under its Horizon 2020 programme for research and innovation, STREAMLINE will make key updates to the facility's scientific strategy, renew its business plan and revisit access modes, including a new access service package. It will also support and train new and existing user communities.

The project is necessary because of the huge step-change in capability of the EBS upgrade. As the world's first high-energy fourth-generation light source, the ESRF–EBS has been designated a future "landmark" facility by the European Strategy Forum on Research Infrastructures, and will light up synchrotron science with X-ray beams up to 100 times brighter and more coherent than before. The benefits will be seen across all the ESRF's 44 beamlines, especially the new state-of-the-art EBSL beamlines and those delivering micro- and nano-beams. There will be more users, more samples and more data through faster and wholly new experiments. New services could be created, too.

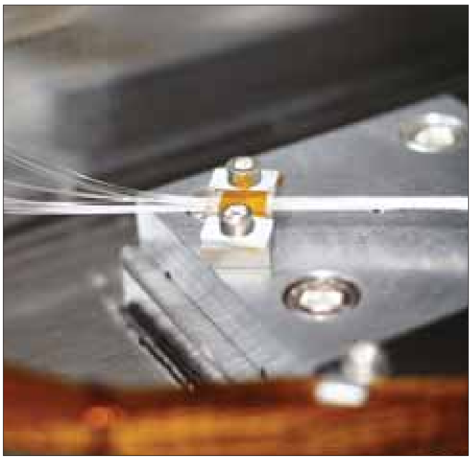
Ultimately, the goal of STREAMLINE is to show what is possible with a fourth-generation synchrotron, for industry and academia alike. The results of the project will be shared with other European light sources, many of which are planning EBS-type upgrades in coming years.

Advertising feature

# Crystal parallelism: monochromator upgrade at ESRF

*Flex structure for easy alignment of the Ø 4 mm sensor heads (image copyright ESRF).*

*Fiber connectors air side (image copyright ESRF).*



*155 μm thin HV and radiation hard fibers (image copyright ESRF).*

*As the front runner in upgrading an existing synchrotron into a low emittance fourth-generation synchrotron, ESRF will be able to bring x-ray science into new research areas only imaginable today. More than 9000 researchers in the 22 partner countries visit the ESRF every year and look forward to benefiting from its current upgrades.*

In addition to the Extremely Brilliant Source, new beamlines are being designed and old beamlines refurbished to facilitate the ever-growing demand of faster, more precise structural determination, with higher resolution. Resolution relies, of course, not only on high-brilliance beams but also on high-precision mechanics and position metrology. Ultimately, it comes down to the stability and position accuracy of the sample with respect to the shape and direction of the beam. *attocube systems*, a known pioneer of precision, supports this cause.

How does one reach the most precise movements and movement detection? One first needs reliable positioning and position sensing with precise enough resolution and repeatability. The laser interferometer from attocube offers nm resolution, while the nano positioner has a repeatability down to ±25 nm, independent of the travel range.

Sensor heads and the stages can handle a variation of extreme environments and a combination of the extremes, such as 10 MGy radiation in UHV while being cooled down to mK temperatures. Laser interferometers and piezo nano positioners from attocube can be found everywhere in a synchrotron facility, from where the beam is generated, through the undulators, monochromators, focusing optics, shutters, and all the way to the sample position. An illustrating example is the current development at the ESRF.

Spectroscopy beamlines at the ESRF are equipped with a generic model of double crystal monochromator (DCM), originally acquired in the 1990s. Now after more than 20 years, the then pioneering technology cannot meet the increased challenges. With the ongoing EBS upgrade, specifications like position and angular stability, thermal stability, cooling system, vibration, control and feedback, will need to be newly defined. As a consequence, new DCMs are being redesigned.

Most critical for the performance of the DCM is the parallelism of the two crystal planes. To maintain crystal parallelism to within defined specifications over the entire scan range is perhaps the most challenging aspect of the entire DCM design: intrinsic dynamic behavior and thermal gradients make it impossible to envisage such stability uniquely through passive mechanical design. Therefore, there are several requirements that have to be met when detecting the smallest shifts and changes of the aligned crystals:

- precise detection of the crystals' rotation in the nanorad range
- vacuum compatibility
- long life inside a vacuum chamber with scattered X-rays.

With the IDS3010 and the specific sensor heads, attocube offers a solution which is easy to integrate and guarantees the required precision.

## Precision and resolution

During Bragg angle scans, the crystals are moved with a required stability of the parallelism between crystals of 15 nrad.

Although standard integrated optoelectronic (and other type of) encodes for positioners, goniometers and rotators meet these numbers, the resolution and stability at the moved object is far away from such numbers. This changes completely when using a combination of precise positioners – for the movement – and an accurate displacement sensor for sensing the movement of the object.

Measurements at attocube's labs showed that when using an external sensor the setup enables the positioning of targets with a repeatability 100 times better than with an integrated one – detected in a standard laboratory environment. By detecting the positioner's movement and the target movement, the IDS3010 closes the feedback loop at exactly the point that is, in general, the point of interest.

## Vacuum compatibility

To gain the best stability an "undisturbed" beam is preferred. This is guaranteed by bringing the measurement tool as close as possible to the crystals. The IDS3010 separates the sensor unit (sensor head) where the detection is done only optically, from all electrical components. Both parts are only connected via a fiber which can easily be led into a high or ultra-high vacuum chamber. Thus, it is possible to place the sensor heads directly behind the main crystal plate.

## Radiation hardness

Most encoders offering the requested precision are not made to be placed inside a chamber with scattered radiation. Their lifetime decreases drastically. By separating the electronics from the detection head and only placing these inside the chamber the sensitive electronics are out of the direct line of primary scattering. Although the sensor heads and fibers are designed to be able to handle a radiation dose of up to 10 MGy, local shielding is also implemented as extra precaution.

Based upon the severity of the specifications for parallelism stability, the DCM's online metrology system, using multiple radiation resistant IDS3010 interferometer heads, has been integrated to monitor the angular drifts of the 2nd crystal and allow correction of its position in real time.

*For more information, visit* **www.attocube.com/en**

attocube
WITTENSTEIN Group

# Xenocs now just steps away from the ESRF

**Laboratory SAXS/WAXS instruments provider Xenocs is now neighbour to the EPN Campus**

### Size, shape and structure of materials at the nanoscale

Xenocs is a market-leading provider of complete instruments for structural characterisation of materials using X-ray scattering techniques (SAXS/WAXS/GISAXS/USAXS). Typical information obtained from the measurements is the size, shape and structure of materials at the nanoscale and how this structure changes with parameters such as temperature and stress. Applications include polymer research, nanoparticles and colloids, structural biology, cosmetics or renewable energy, to quote just a few. With the progress in instruments and software over the last two decades, most measurements and analysis can now be done in a matter of seconds to minutes.

### New facility in an ideal location

“*We are thrilled to work in this new facility, compliant with the latest energy-saving standards, which provides our staff with a stimulating work environment and enough space to centralise both R&D and production in one location,*” Xenocs CEO Peter Høghøj explains. “*The new facility is ideally located and offers our customers the opportunity of combining a trip to the synchrotron with a visit to our state-of-the-art laboratory where they can meet our SAXS scientists and try out the latest solutions for characterisation at the nanoscale in the lab.*”

### Xenocs step by step

Created in 2000 as a spin-off from ILL, Xenocs started its activities by developing multilayer X-ray optics. The company moved a step further a few years by launching the Genix X-ray sources embedding the optics. In 2010, Xenocs launched the Xeuss product line in response to the market request for a complete in-lab X-ray scattering equipment. In 2014, the company launched the Nano-inXider, a compact vertical SAXS/WAXS instrument. In early 2017, Xenocs acquired SAXSLAB, a Danish company known for their expertise in small angle X-ray scattering instruments and technologies. Last August, with more than 100 SAXS systems installed worldwide, Xenocs moved its headquarters to the new building called ‘Steps’, a reference to the step-by-step growth of the company.

**Web** www.xenocs.com
**Email** application@xenocs.com

# User Meeting nearly here

The next ESRF User Meeting will take place between 3 and 5 February next year. Arriving three months into the electron-beam commissioning of the new Extremely Brilliant Source (EBS), the meeting will allow users to discuss how they can best exploit the fourth-generation machine in preparation for the proposal submission deadline only a few weeks afterwards, on 2 March.

The meeting will include four keynote speakers; a director's report on the status of the upgrade and the new flagship beamlines; three user-dedicated microsymposia; eight user tutorials; and an evening poster session over cocktails, during which the annual Young Scientist Award will be presented. It is regarded as an ideal setting to network, share research ideas and learn about new opportunities.


ESRF/P JAYET

# Hard X-rays get spectrography

An international collaboration of synchrotron scientists has demonstrated phonon spectroscopy with spectrographic imaging for hard X-rays at the ESRF for the first time, potentially opening a new window for the study of electronic and magnetic excitations.

Spectrographic imaging is a common tool for infrared through to soft (low-energy) X-ray electromagnetic radiation. For visible light, it is commonly associated with Newton's prism, which splits white light into its constituent rainbow of colours, or wavelengths, which can then be exploited individually; diffraction gratings perform the same function for soft X-rays. In the hard (high-energy) X-ray regime, however, conventional diffraction gratings are inefficient. As a result, scientists are limited to selecting specific wavelengths with devices such as monochromators, which act as crude filters, throwing away most of the beam's intensity.

Working with researchers at the Argonne National Laboratory's Advanced Photon Source in Illinois, US, and DESY in Hamburg, Germany, Alexandr Chumakov, Dimitrios Bessas and Rudolf Rüffer at the ESRF have managed to apply spectrography to hard X-rays on the ID18 beamline with diffraction elements made of special, asymmetrically cut crystals in combination with focusing optics. Using these spectrographic components, they then studied nuclear inelastic scattering in phonon spectroscopy of alpha-iron – an extensively studied allotrope of iron that gives cast iron and steel their ferromagnetism – and discovered some unusual, previously unknown soft atomic dynamics (*Phys. Rev. Lett.* **123** 097402).

"This approach is a fascinating way to gain in quality – energy resolution – while maintaining or even gaining quantity – count rate," says Chumakov.

*"It's a fascinating way to gain quality while maintaining quantity"*


ERSF/P JAYET


THOR/FLICKR

## Tattoo needles deposit metals in the body

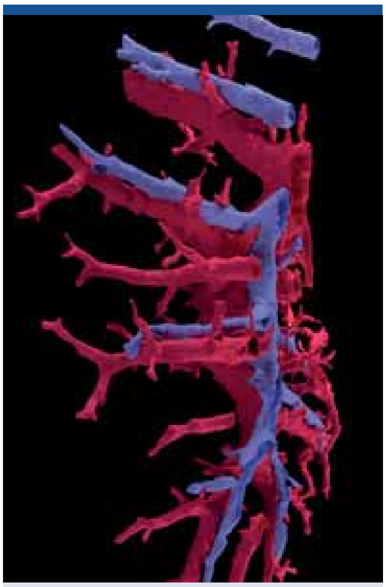
An investigation based on ESRF data has shown that the needles used by tattoo artists can abrade during operation, depositing toxic metals into the body. The abrasion is caused by inks containing titanium dioxide, an abrasive material.

"It is beyond doubt that the metal particles [in the body] derive from the tattoo needle as result of pure mechanical grinding," says Bernhard Hesse, a researcher at the synchrotron access-facilitation company Xploraytion in Berlin, Germany, and an ESRF visiting scientist.

Two years ago, Hesse and colleagues found evidence on the ESRF ID21 and ID16B beamlines that nanoparticles present in tattoo ink can migrate to the lymph nodes, a critical part of the body's immune system (see *ESRFnews* December 2017, p7). The result was a warning about the dangers of tattooing, but also raised a question, as the particles included iron, chromium and nickel, despite those metals not being present in normal tattoo inks. Led by researchers at the Federal Institute for Risk Assessment in Germany, the group recently speculated that the metals came not from the inks, but the needles.

To test their theory, the scientists took needle samples to ID21 and ID16B, where they could perform nano X-ray fluorescence and nano X-ray absorption near-edge structure. They found that needle wear rose significantly upon tattooing with titanium dioxide when compared with carbon black, another pigment (*Part. Fibre Toxicol.* **16** 33).

"There's more to tattoos than meets the eye," says ESRF scientist Hiram Castillo, one of the study authors.


S BAYAT

## X-ray tomography reveals airway collapse

Synchrotron phase-contrast imaging has been used to study the closure of airways in lungs for the first time. The ESRF results point to the thickness of fluid lining the airways as a cause of closure, and could help in the development of treatment strategies for people suffering from acute respiratory distress syndrome (ARDS).

ARDS is a life-threatening condition in which the lungs cannot supply the body with enough oxygen, and is usually the result of another severe condition, such as pneumonia or a chest injury. Mechanical ventilation is the standard treatment, but this involves stress on the lung tissue and can actually worsen the damage. Clinicians believe the root problem lies in the collapsibility of small airways in the lungs, but these are not visible enough in the coarse resolution of traditional computed tomography scans. To avoid this limitation, therefore, Ludovic Broche of Uppsala University in Sweden and colleagues made use of the high-resolution, phase-contrast version of the technique at the ID17 beamline.

In their ARDS model, the researchers uncovered a mechanism of airway closure called compliant collapse, in which fluids lining the airway walls thicken, inducing compressive forces. Moreover, they found evidence that airway closure and reopening is dependent not only on the air pressure, but on the timing of ventilation. That suggests airway closure could be prevented by reducing the time during which respiratory pressure is lowered in the breathing cycle, the researchers say (*Crit. Care Med.* **47** e774).

# 3D chemistry comes to fossils

An ESRF-developed technique that uses hard X-rays to image the chemical speciation of ancient materials has been tested for the first time, on a 53-million-year-old ant. Known as 3D X-ray Raman imaging, the technique has revealed the chemical composition of the insect at a resolution of 10 µm.

"It's the first scientific case for this technique after five years of development, not only from the technical point of view but also from the data-analysis side," says Christoph Sahle, the scientist in charge of ID20 and one of the study authors.

Developed on ID20, 3D X-ray Raman imaging involves inelastically scattering X-rays off a material, so that a small portion of the X-ray energy is transferred to the material's electrons. The signal from the electrons is then captured by a new spectrometer, which allows the chemical composition of the material to be interpreted in three dimensions. That makes it more attractive than X-ray fluorescence imaging, which can only image thin sections of materials, or 3D X-ray computed tomography, which provides a 3D insight into a material's interior density at the expense of any information on molecular composition, especially that of lighter elements.

The team behind the demonstration of the new technique was led by the IPANEMA CNRS laboratory at the Université Paris-Saclay in France, and included three other CNRS labs (IMPMC, ISYEB, and LCPMR in Paris), three synchrotron facilities (the ESRF and SOLEIL in France, and SLAC in the US) and the University of Lausanne in Switzerland. Applying it to the ant, which had been well-preserved in ancient amber, the researchers found chitin, a complex sugar that constituted the insect's exoskeleton. The results also revealed a difference in preservation between the part of the insect that had first been caught in the resin and the part that had been submerged later, after its death (*Sci. Adv.* **5** eaaw5019).

"This collaboration, bringing together synchrotron facilities from around the world, illustrates the interest of seeking new and original methods for studying ancient and heritage objects," says Loïc Bertrand, the IPANEMA director.

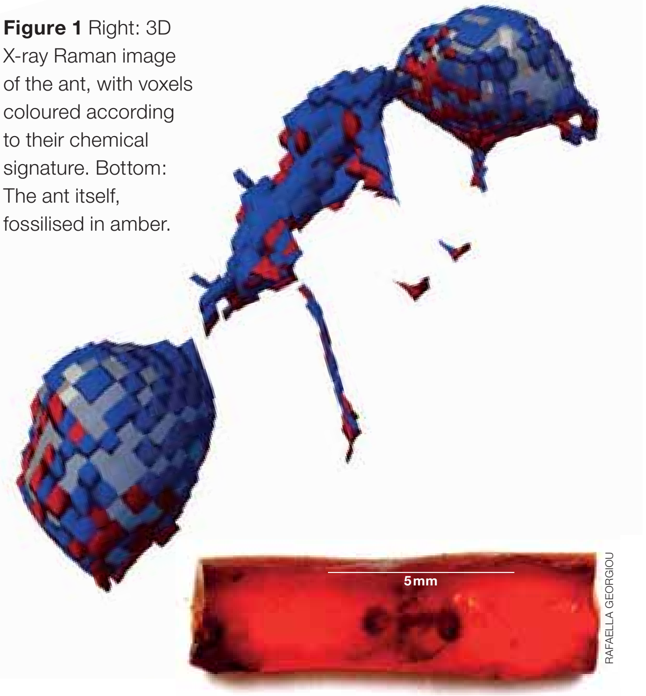


**Figure 1** Right: 3D X-ray Raman image of the ant, with voxels coloured according to their chemical signature. Bottom: The ant itself, fossilised in amber.

RAFAELLA GEORGIOU

# Plutonium state unexpected

An international team of scientists studying ESRF data have discovered a new and stable "pentavalent" (V) state of plutonium. The existence of the unexpected state could change predictions of how plutonium behaves in the environment over millions of years.

Kristina Kvashnina, a physicist from the Helmholtz Zentrum Dresden-Rossendorf laboratory in Dresden, Germany, who is based at the ESRF's ROBL beamline, got the first hint of the discovery while preparing plutonium-dioxide nanoparticles from different precursors. When she and her colleagues tried to make them from plutonium in the VI oxidation state, they found a strange intermediate state. High-energy-resolution fluorescence detection at the ROBL and ID26 beamlines confirmed the state as plutonium V; measurements repeated months later confirmed the state's long-term stability.

The results have been published in *Angewandte Chemie* in what the journal calls a "very important paper", a designation given to less than 5% of manuscripts (*Angew. Chem. Int.* doi:10.1002/anie.201911637).

"The chemists were in complete disbelief, but the results were quite clear," says Kvashnina.

> *"Chemists were in complete disbelief"*

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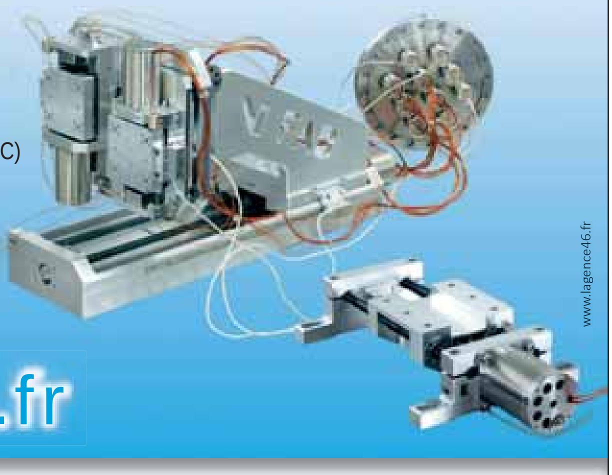
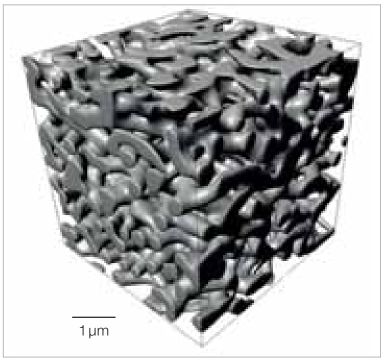


# Beetling towards eco-paint

AkzoNobel, makers of Dulux paint, join the University of Sheffield to mimic nature in search of the perfect white.

ANDREW PARNELL



COMMUN. CHEM. 2 100

**Figure 1** Above: The white scales on *Cyphochilus* are due to structural colouring. Left: 3D X-ray nanotomography at ID16B reveals the ratio of material to void that delivers this structural whiteness.

Pure white, vanilla white, cornflower white, chalk white, goose white, ultra white – the international coatings manufacturer AkzoNobel produces almost countless shades of white paint. Based on its research at the ESRF, however, it soon might need to add another to its list: beetle white. The research has shown how to synthesise the unusual "structural" whiteness of a tropical beetle, with the potential to create paints with a much lower carbon footprint.

Most paint gets its whiteness from microscopic particles of titanium dioxide. Thanks to its high refractive index, titanium dioxide scatters light very strongly from its surface, but its manufacture is also harmful to the environment, contributing nearly 75% of the carbon footprint of every tin of paint. In biological organisms, however, whiteness usually comes from light scattering from within microstructures and interfering with itself. The shell of the tropical beetle *Cyphochilus* is a good example of this structural white, and scientists such as Daragh McLoughlin of AkzoNobel's research centre in Slough, UK, have been keen to investigate its makeup as a potential titanium-dioxide substitute.

## Enter the void

In previous studies, researchers have established that *Cyphochilus*'s shell has an exceptionally thin and aperiodic structure, consisting of a common biological material known as chitin and plentiful voids. Now, in partnership with a team at the University of Sheffield in the UK and other institutions, McLoughlin has made use of 3D X-ray nanotomography imaging at the ESRF's ID16B beamline to understand how that structure forms (see fig. 1). "It's the unique combination of the high intensity of the X-rays available from the beam, coupled with the advanced analysis facilities in Grenoble, that allowed the 3D nano-tomography to be achieved with such amazingly good resolution," says David Elliott, the manager of materials characterisation at AkzoNobel in Slough.

When compared with simulations, the results suggested that the beetle's structure forms from liquids "unmixing" in certain conditions – a process known as spinodal decomposition – and pinpointed optimal whiteness at a 25% chitin filling. That meant that the researchers could go on to create their own synthetic structural white, by quenching a polymer in a solvent. The ESRF's ID02 beamline allowed them to confirm the formation mechanism as the polymer layer dried and became structured. The resultant film was 94% reflective – even more than the shell of *Cyphochilus* itself (*Commun. Chem.* 2 100).

More work will need to be done on the polymer to see whether it is suitable as a titanium-dioxide substitute. But Andrew Parnell of the University of Sheffield can already see how it might help save the environment in more ways than reduced carbon emissions. "Ideally, we could even recycle plastic waste, structure it just like the beetle scale and then use it to make super white paint," he says. "This would make paint with a much lower carbon footprint and help tackle the challenge of recycling single-use plastics." ■

**Jon Cartwright**

# ELI-ALPS[1] receives NanoESCA III

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| | |
|---|---|
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| $\Delta t_{TOF}$ | 88 ps |
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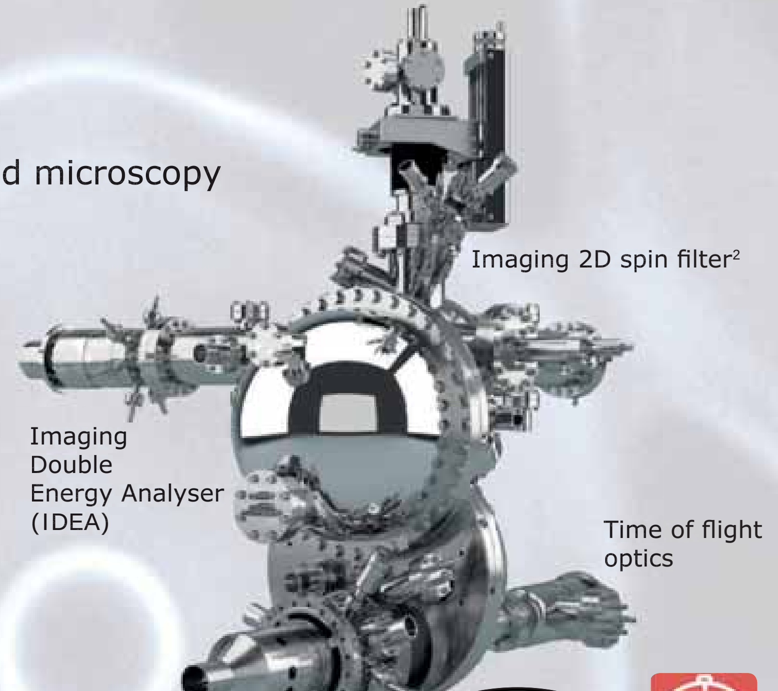
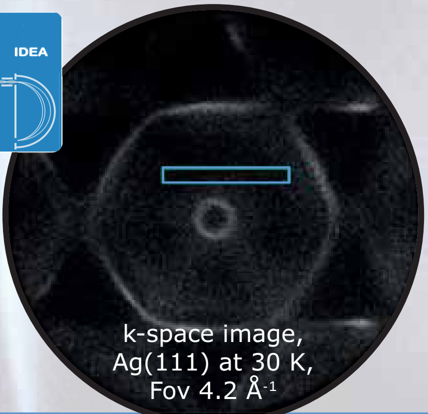


Imaging 2D spin filter[2]

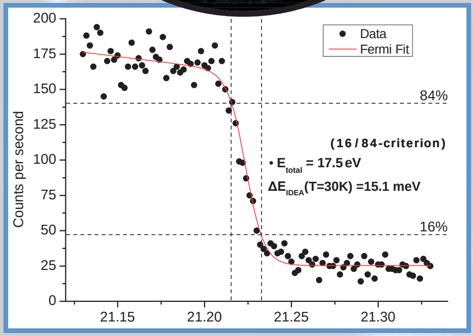
Imaging Double Energy Analyser (IDEA)

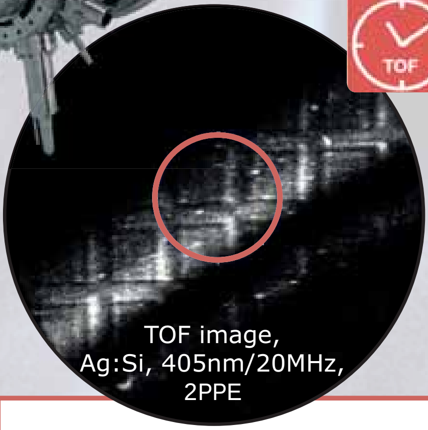
Time of flight optics

PEEM optics

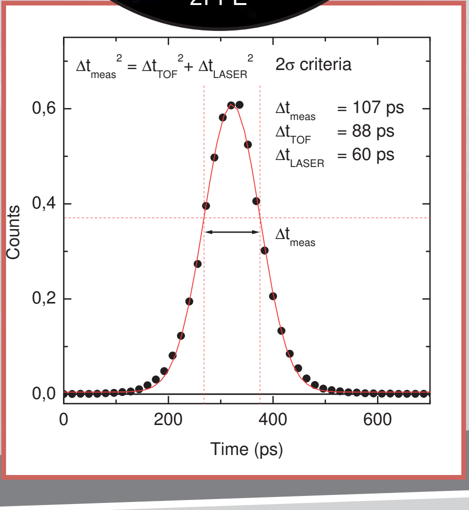


k-space image, Ag(111) at 30 K, Fov 4.2 Å$^{-1}$





TOF image, Ag:Si, 405nm/20MHz, 2PPE



Spin assymmetry contrast of an iron poly crystal (4.9eV UV source, dE 100meV, FoV 66µm, 400 sec acquisition time)

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[1] ELI-ALPS: www.eli-alps.hu

[2] Protecting German Patents: DE102005045622B4, DE102010010981B4, legal and technical rights secured by sublicensing from the Surface Concept GmbH

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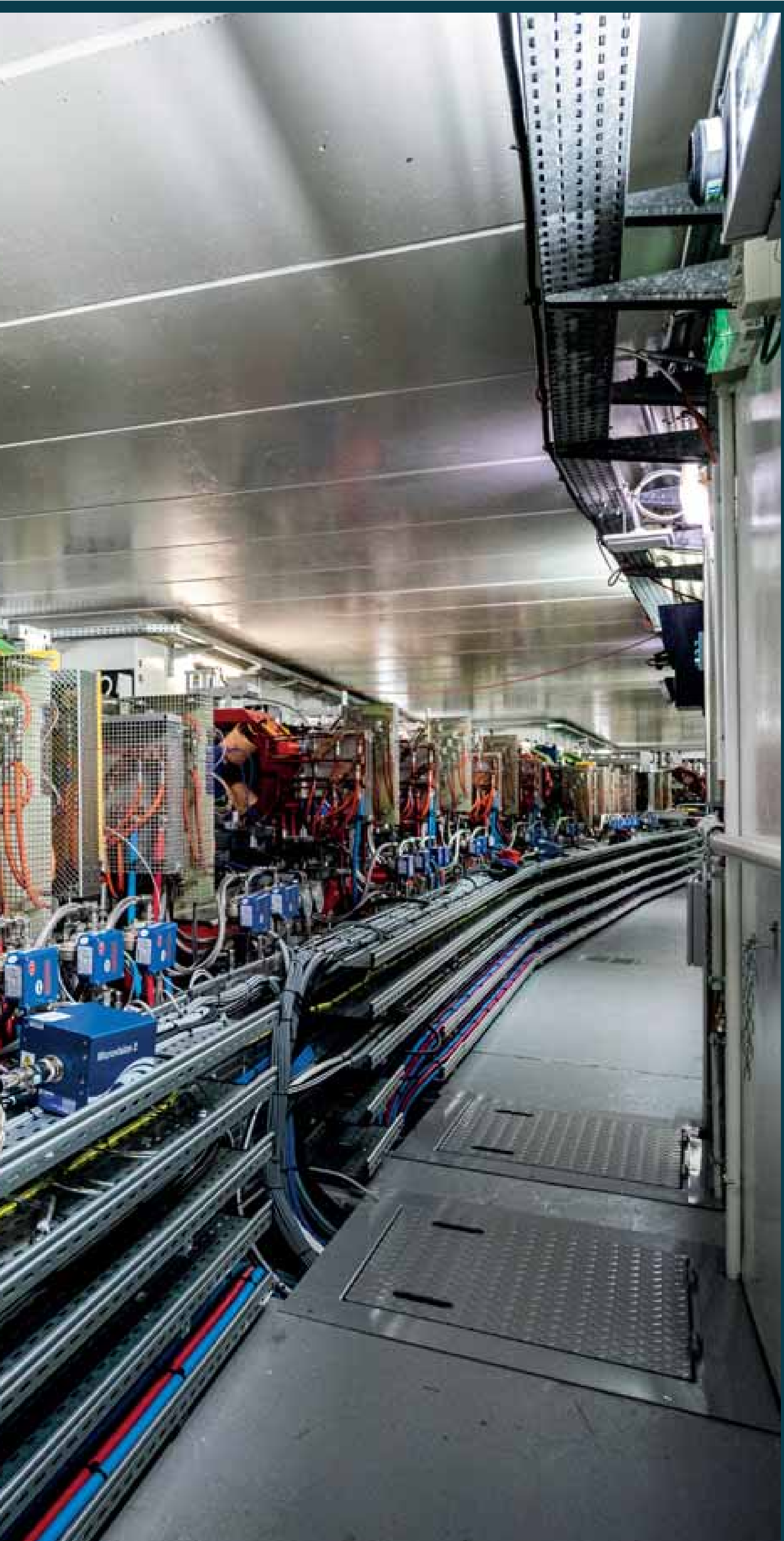

# Power on

The EBS's electromagnets have been activated for the first time since their installation in the tunnel in May. Representing the initial signs of life for the new fourth-generation machine, the magnets were powered on in turn and ramped up to operation settings in order to test and debug any teething problems in their new power supplies before commissioning.

A big change for the EBS is that each magnet in the storage ring is powered individually, rather than within a family of 32 or 64 as in the previous machine. While improving control over the magnets, this also results in nearly 1000 separate power supplies. In order to maintain the high reliability of these power supplies, ESRF engineers have come up with a concept called hot-swapping. This means that in the case of an electric failure in one of the units, another unit steps in automatically to maintain current in the magnet while the faulty unit is changed – avoiding any interruption in the X-ray beams.

This innovative way of powering the magnets, together with an efficient design for the electromagnets themselves, and the use of permanent magnets where possible, will slash the power consumption of the accelerator by 1 MW. ■

**Anya Joly**


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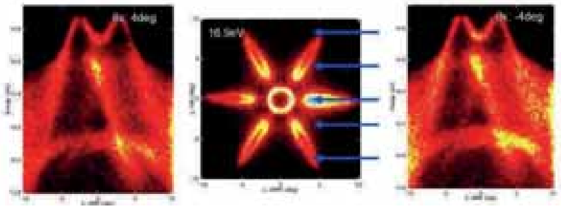

# Beam me up

ESRF accelerator physicists are preparing to send electrons into the hotly anticipated new synchrotron source.

As the final concrete roof beam is lowered onto the storage ring tunnel, the EBS installation groups prepare to hand the EBS baton to the ESRF's accelerator physicists. With the first electron beams due to enter the new ring in early December, the beam dynamics group is eager to make a start. "We've been working towards this day for six years," says Simone Liuzzo, one of the five members of the group. "An opportunity like this doesn't come along twice in the life of an accelerator physicist."

In preparation for beam commissioning, staff are testing equipment and power supplies before starting up the injector system, which involves a linear accelerator sending electrons into a six-gigaelectronvolt booster and on into the storage ring. "Commissioning the booster should be more straightforward than the storage ring as fewer things have changed," says Nicola Carmignani, who will oversee the work along with his colleague Thomas Perron.

Once the booster is up and running, injecting the first electrons into the 844-metre-circumference ring will be a key milestone for the team. They will use diagnostic equipment to painstakingly monitor the very first turns that the electrons make, and tune the individual strengths of the electromagnets that steer the electron beam onto its ideal trajectory.

"We've been working towards this day for six years"

Wisely, they are already testing this process out on a simulator of the EBS, developed with the accelerator control team. The simulator is a virtual synchrotron control system that allows the team to test and tune properties of the electron beam as if they were working on the machine itself. "It's really useful for getting to know the EBS machine before commissioning starts," says team member Lee Carver.

Following the first turns of the electrons, the team will ramp up the current and optimise the parameters of the beam. "The commissioning programme is dense," says Simon White, the beam dynamics group head. "But we're focused on delivering an electron beam with nominal current, lifetime and emittance so the beamlines can resume their programmes with optimal conditions."

With machine commissioning to run from 2 December until 2 March, when six months of beamline commissioning begins, the team won't get a chance to pause until the EBS officially opens to scientific users on 25 August 2020 (see Insight, p21). But that won't be the end of the simulator. Once the EBS is running smoothly, the team will use it to test further machine developments. ■

**Anya Joly**

Members of the beam dynamics group prepare to commission the EBS from the ESRF's control room. From left: Simone Liuzzo, Nicola Carmignani, Lee Carver and Simon White.


XIA
FalconX8

# The final countdown

In this special Insight, the ESRF directors of research Harald Reichert and Jean Susini answer common user questions about the EBS launch.

C ARGOUD/ESRF

Full steam ahead: Harald Reichert and Jean Susini (right) say they have always planned to restart the maximum number of beamlines.

**When will the world's first high-energy, fourth-generation light source be up and running?**
The launch will be a three-step process. The first step is expected to commence on 2 December, shortly after this issue goes to press, when the first electrons will be injected into the new EBS storage ring. Our colleagues in the Accelerator Division will then spend three months commissioning the ring, so that beamline commissioning can begin on 2 March next year, including some limited operation with long-term, "friendly" ESRF users. Finally, normal user operation will resume almost six months later, after the summer shutdown, on 25 August.

**Will all the beamlines be restarted?**
Hopefully all of them, including the collaborating-research-group (CRG) beamlines, along with all the support labs. Since the beginning of the EBS upgrade project, the overall strategy has been to restart the maximum number of beamlines. The only beamlines not restarting immediately are the ones that are in shutdown for a major upgrade (ID03 and ID29, which will become the EBSL2 and EBSL8 flagship beamlines) or refurbishment (ID27, ID24 and BM23).

The restart of beamline operation is by itself a very complex process involving almost everyone at the ESRF. If any beamlines should have problems, these will be addressed during the beamline commissioning period from March to July next year, prioritised by management according to the availability of resources.

**What about bending-magnet (BM) beamlines?**
Indeed, these won't have a source when the commissioning of the other, insertion-device beamlines starts in March. Those that requested a two-pole wiggler source, which constitute about half of all the BM beamlines, will start commissioning after the first shutdown at the end of April, when these sources are being installed. The remaining BM beamlines, which requested a single-bend source, will begin commissioning in June after the second shutdown.

**Will all the beamlines actually be able to cope with the new source?**
It's worth remembering that the installation of the EBS is the second phase of an upgrade programme that began back in 2009. About two-thirds of the ESRF public beamlines were already upgraded in phase 1 with the EBS in mind. Among the others, some are in the process of refurbishment, some require modification of their optics, and some will be able to cope with the new source until future upgrades are completed. A major part of the activities during the shutdown is the installation of a new generation of high-performance 2D detectors on the scattering/diffraction beamlines.

There is, however, another big change that will help the beamlines to get the most out of the EBS: their control systems.

KEY DATES

**2 December 2019**
Storage ring commissioning

**2 March 2020**
Standard and long-term proposal submission deadline

**2 March 2020**
Beamline commissioning

**25 August 2020**
Full EBS user operation

**What does that mean in practice?**
Many users will be familiar with the ESRF's existing beamline control system, SPEC, which has been in place for decades. The new control system, BLISS, will have many new advanced features, such as continuous complex scans, but entails a learning curve for ESRF staff and users alike. Think of it like a major upgrade in your laptop's operating system, with a different layout and a slightly different syntax. If you are a user who has written your own data-acquisition protocols for SPEC in the past, these will have to be individually ported to BLISS, as they are beamline specific. This process might take some time: there are hundreds if not thousands of them in total. To begin with, however, only about half of the beamlines will be running BLISS; the rest will be upgraded later, gradually.

**Is the control system integrated with the new data policy?**
Yes. But certain essential features of the new data policy, such as the metadata catalogue and the electronic log book, will be implemented on as many beamlines as possible, regardless of whether

ESRF scientist Alexandra Joita Pacureanu at work on the ID16A nano-imaging beamline. ID16A is one of many beamlines that were built in phase 1 of the upgrade programme with EBS capabilities in mind.

STEF CANDÉ/ESRF

they have been upgraded to BLISS, by the official launch of normal user operation in August. The exceptions are the CRG beamlines, for which these features will come later.

Data management is a challenge for all big scientific infrastructures. In the ESRF's case, a combination of increased brightness and flux, and faster detectors, means that the data volume will strongly increase. In the ESRF strategy, data management has been identified as one of the most crucial issues to be dealt with in the future. It will take time to work out the best ways to manage, transfer, store and process data.

> *"We challenge our users to explore what is possible with a source that is 100 times brighter and more coherent"*

**Will we see a change in the number and duration of experiments? And can we submit long-term proposals?**

We know from phase 1 of the upgrade that optimised experiments are generally much shorter. Therefore, we are expecting shorter experiments and more of them, which pose some challenges for the operation budget of the ESRF and the increasing number of users travelling to the facility. In parallel, therefore, we are exploring new access models, such as mail-in or remote access in areas beyond structural biology.

Regarding long-term proposals – yes please! The deadline is 2 March 2020.

When submitting proposals for the ESRF–EBS, we challenge our users to explore what is possible with a source that is 100 times brighter and 100 times more coherent. Think what you could study now that you could not study before.

**Is the ESRF management excited yet?**

Actually, we were already excited two years before the shutdown, because that was when we did all the planning, including making the best of the long shutdown. Once the EBS is up and running, we will see whether everything we set in motion actually works. Naturally, there will be problems – but the challenge then is solving them. What excites us most is the science to come from our users. ■

**Jon Cartwright**

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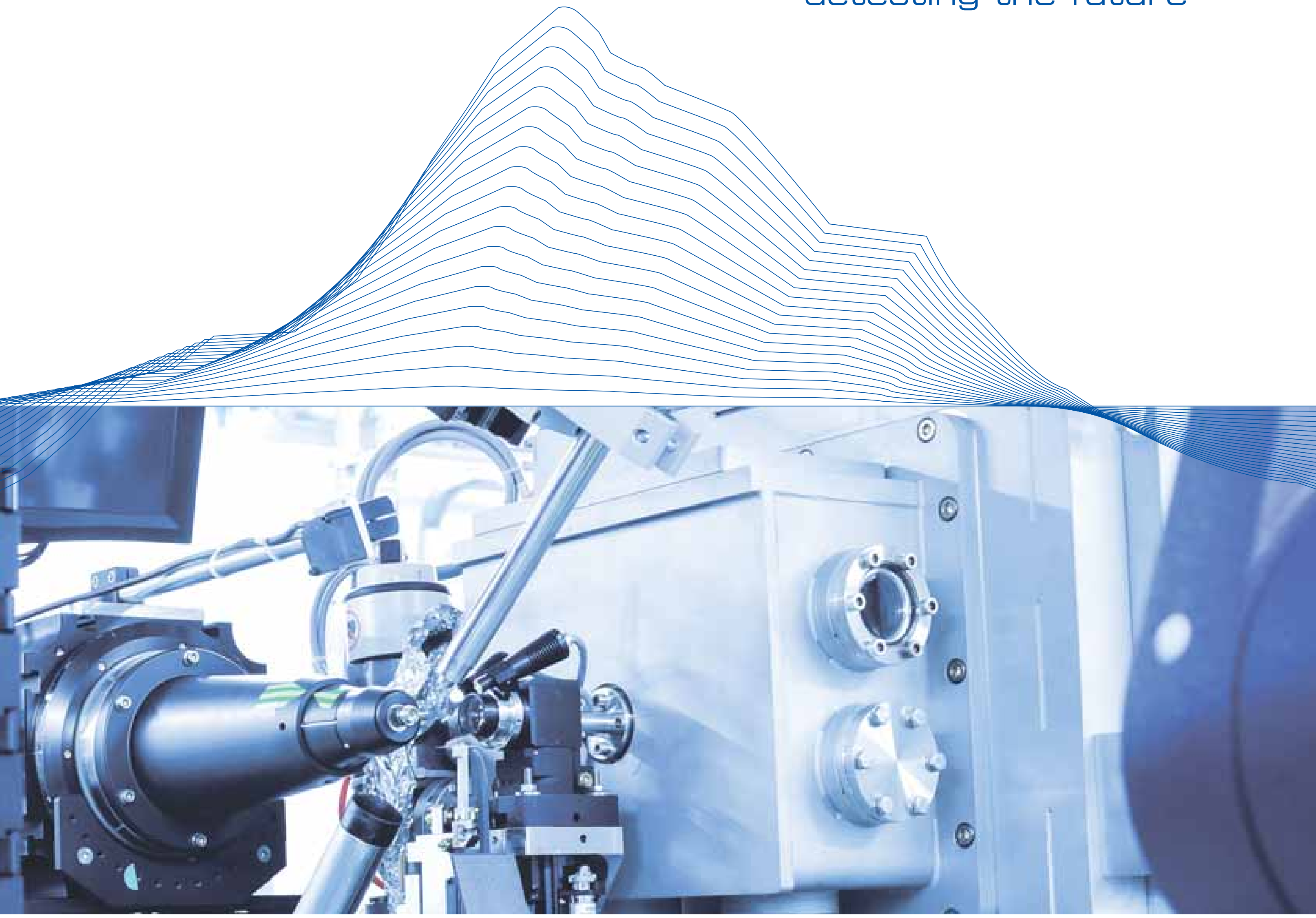

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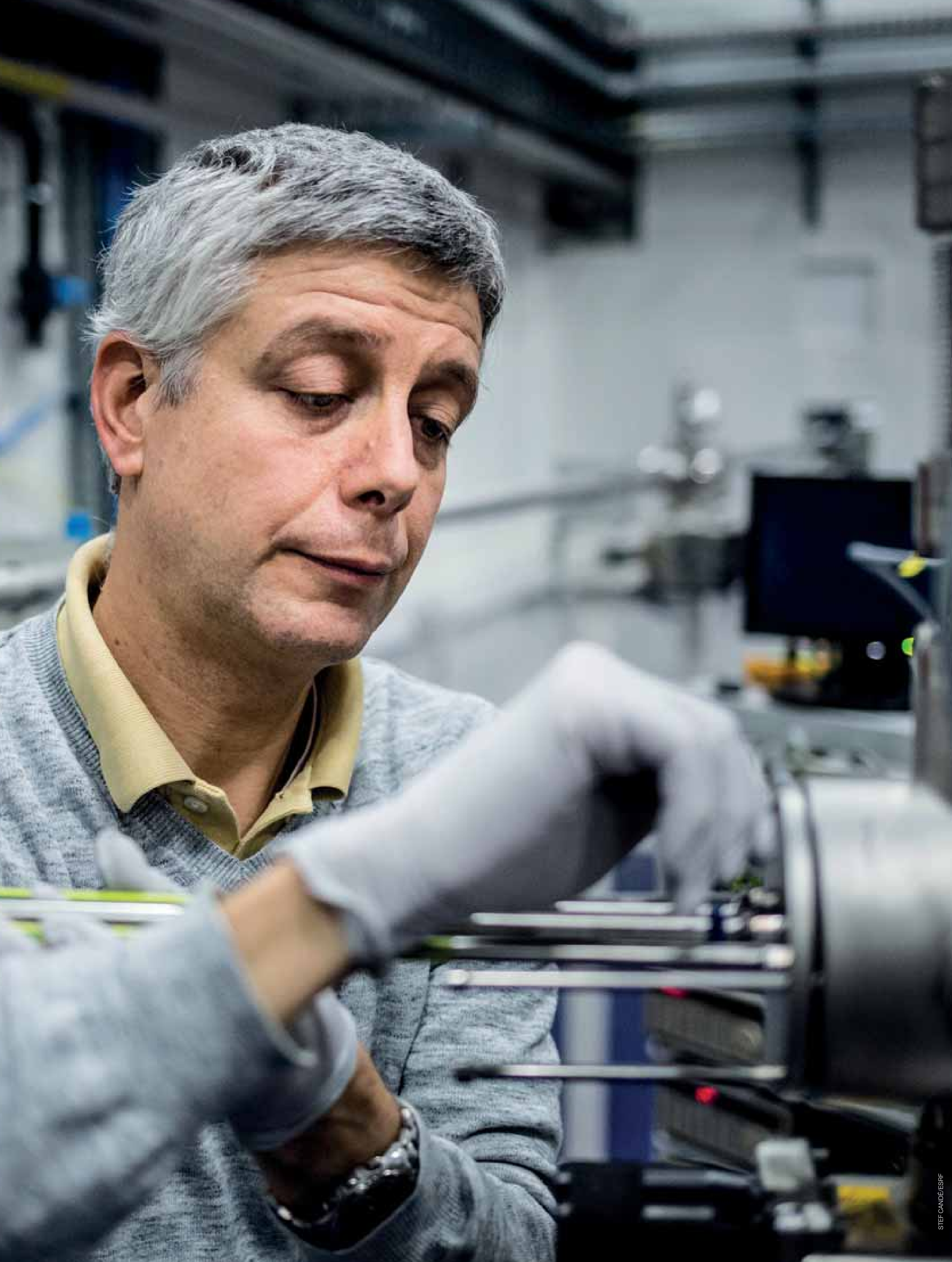
STEF CANDÉ/ESRF

# MAKING WAVES

## Scientists dream of room-temperature superconductivity. Could the ESRF observation of charge-density fluctuations help make it a reality?

THE term "holy grail" is overused in science. Yet if there is one object that truly merits its use, it is the room-temperature superconductor. A material that would, in ambient conditions, conduct electricity with zero resistance. A material that would allow super-fast electronics, levitating trains and, perhaps most importantly, lossless transmission of energy over vast distances.

A material that has evaded discovery for more than a century.

To study superconductivity, you need patience. Big developments do not come often. Many scientists who are initially attracted to the field by the prize of finding something world-changing leave years later, frustrated at the lack of progress. "For some people it's a never-ending story," says Giacomo Ghiringhelli (opposite), a physicist at the Politecnico di Milano in Italy and a long-time ESRF user.

But now this story has taken a twist. Thanks to the efforts of Ghiringhelli and others who have been perfecting a observational technique known as resonant inelastic X-ray scattering (RIXS) at the ESRF, scientists are realising that certain superconductors exhibit a peculiar phenomenon besides superconductivity – waves and fluctuations of electric charge. No-one is sure yet what causes these waves and fluctuations, nor does anyone know if they are behind the superconductivity itself. But when all the obvious paths have been explored long ago, any new clue in the search for room-temperature superconductivity is welcome indeed.

"I happen to think charge-density waves are an essential feature of the story," says Steve Kivelson, a theorist at Stanford University in California, US, who has not been involved in the research.

The superconductivity story began in 1911, when the Dutch physicist Heike Kamerlingh Onnes discovered that the electrical resistance of mercury suddenly disappeared beneath a temperature of 4.2 K (–269 °C). The observation could not be readily accounted for, even with the advent of quantum mechanics in the 1920s. At any temperature, suggested the theory, random thermal fluctuations ought to keep the current in check. Only in 1957 did three theorists at the University of Illinois Urbana-Champaign in the US – John Bardeen, Leon Cooper and Robert Schrieffer – manage to describe the phenomenon, by showing how an electron deforms the atomic lattice through which it moves. The deformation allows the electron to pair with the next one in line, and in this way all the electrons begin moving as a single cohort, or condensate, which prevails over thermal fluctuations with ease.

BCS theory, as it came to be known (after the surnames of its originators), was very good at describing how superconductivity could arise in metals at temperatures close to absolute zero, and certainly less than about 30 K (–243 °C). Starting in 1986 with Georg Bednorz and Alex Müller at the IBM Zurich Research Laboratory in Switzerland, however, scientists began to alight on materials that superconducted at temperatures over 35 K (–238 °C), where BCS theory no longer applied. Unlike their frigid counterparts, these high-temperature superconductors were typically insulators made of doped copper oxides, or "cuprates". Within a year, cuprates were transitioning at temperatures over 93 K (–180 °C) – warm enough to be cooled by liquid nitrogen rather than liquid helium.

> "You get a new telescope, and you start to see more. And RIXS is like a new telescope"

The rising transition temperatures of superconducting materials in the late 1980s and early 1990s gave impetus to the hope that one could soon be found operating in ambient conditions. But by the mid 1990s, the transition temperatures (at ambient pressure) were plateauing, at

▸


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about 135 K (–138 °C). The problem was that there was no overarching theory of superconductivity that could direct physicists to their holy grail. After a flurry of initial proposals, theorists had settled into one of two camps: one in which electrons paired through chemical valence bonds become mobile thanks to doping; and another in which doping allows for a ripple in antiferromagnetic alignments of electrons, drawing the electrons together. To this day, however, there is no theoretical consensus.

> “Maybe a property of the charge-density waves plays a role in the conduction process”

## Doping for success

One feature that many theorists broadly agreed on was what should happen when cuprates are doped. Doping is crucial to making ordinary cuprates into superconductors, and involves the introduction of impurities that either boost or reduce the numbers of conduction electrons. These extra electrons, or the extra “holes” left by their removal, can move around inside the material irrespective of the position of the doping atoms, and arrange themselves into energetically favourable patterns. The patterns, theorists believed, would have a well-defined shape and size, and would generate waves of charge density misaligned with the lattice.

“Scientists thought, if the charge-density waves were an intrinsic phenomenon, they might play a role in the superconductivity of cuprates,” says Ghiringhelli. “But first one had to find experimental evidence.”

In the mid 1990s, indirect evidence for charge-density waves came from techniques such as neutron scattering. For indisputable proof, however, scientists had to wait until 2012, when Ghiringhelli and co-workers employed RIXS at various beamlines, including the ESRF’s ID08, to observe the waves directly in the so-called Y123 family of cuprates below the superconducting transition temperature (*Science* **337** 821). A relatively new technique, RIXS had been pioneered in the 1990s by Ghiringhelli and his Milan colleague Lucio Braicovich at the ESRF – with support from the ESRF’s Nick Brookes and in collaboration with the Paul Scherrer Institute in Switzerland – and allowed the study of electronic structure with unprecedented accuracy and sensitivity. Within a few years, the Milan group and others had found charge-density waves in almost all the known cuprate families.

There are good reasons to believe that charge-density waves are not involved in superconductivity. One is that there is no accepted theoretical mechanism in which the waves force the conduction electrons to pair up and create a condensate. Another reason is that the waves appear to be in competition with superconductivity: generally speaking, materials with more prominent waves have lower superconducting transition temperatures than those in which the waves are slighter. “[The waves] are interesting, but I think they’re a sideshow,” says theorist Jan Zaanen at the University of Leiden in the Netherlands.

Other theorists are less dismissive. Kivelson points out that cuprates have another odd feature, in their resistivity. In ordinary conducting materials, resistance rises with temperature due to increasing electron scattering – but only up to a certain point, when the average distance between scattering events becomes on par with the quantum-mechanical uncertainty in the actual electron positions. Then, the resistance levels out. Not so in cuprates: the resistance continues to rise linearly, even after this point. Kivelson calls it “bad metal” behaviour. “I claim it means the picture of individual electrons scattering is wrong for cuprates – that the entire conduction process is different.” Maybe, he says, some property of the observed charge-density waves plays an essential role in the process of conduction.

In August this year, the Milan group (together with co-workers at the ESRF and the Chalmers University of Technology in Sweden, as well as the University of Rome “La Sapienza”, the University of Naples Federico II and the CNR-SPIN laboratory in Italy) reported a result that lent support to this idea. Working on so-called YBCO and NBCO cuprates at the ESRF ID32 beamline – the ESRF’s flagship station for RIXS, which Ghiringhelli and Braicovich helped to develop – the researchers discovered previously unseen charge-density modulations extending beyond the superconducting transition temperature. Though much fainter than normal charge-density waves, and more limited in extent, these fluctuations demonstrated that charge-density modulations of some sort underlie both of the unusual phases of cupra-

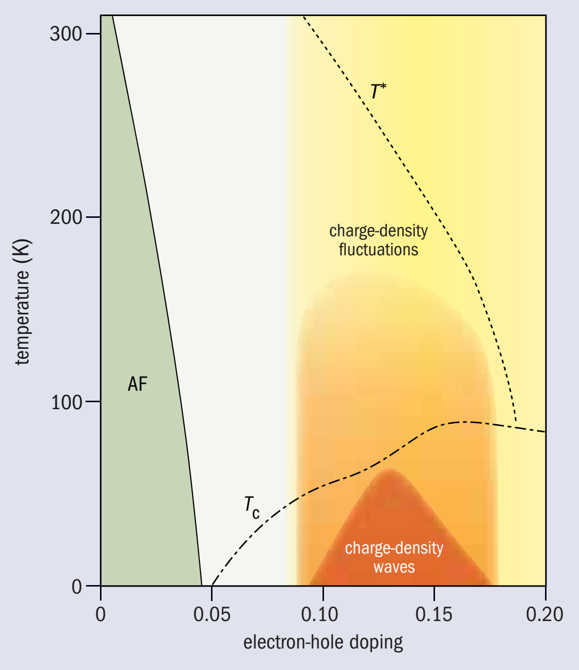

**Figure 1** Cuprate superconductors have several typical regions in their phase diagram. At low levels of electron-hole doping, they are insulating and antiferromagnetic. Then, as doping increases, they exhibit various degrees of conduction depending on temperature: superconduction at relatively low temperatures below $T_c$; middling “pseudogap” conduction at temperatures below $T^*$; and “strange” or “bad” metal conduction at higher temperatures. In recent years, Giacomo Ghiringhelli of the Politecnico di Milano in Italy and colleagues have shown that most of the conducting regions are pervaded by charge-density modulations of some sort. Below $T_c$, these are three-dimensional charge-density waves (red); above $T_c$, the waves peter out into “quasi-critical” two-dimensional waves (orange); and finally they become short-range fluctuations (yellow), which persist even above $T^*$. What the modulations do in the low doping region of 0.05 to 0.08 is currently uncertain, although they are known to disappear in undoped cuprates.

STEF CANDÉ/ESRF

## ROOM TEMPERATURE: WITHIN SIGHT?

For about 60 years, metallic hydrogen has been touted as a potential room-temperature superconductor. The material is predicted to form at very high pressures in the underworlds of gas giants, such as Jupiter. There are claims of its creation in labs here on Earth, too, although these are disputed.

Hydrogen-rich compounds, which are believed to be physically similar, could be more promising. In 2015, a group including Mikhail Eremets and Alexander Drozdov at the Max Planck Institute for Chemistry in Germany, found evidence that hydrogen sulphide became superconducting at pressures of 150 GPa and temperatures beneath 203 K (–70 °C); a year later, they confirmed the superconductivity at the ESRF's ID18 beamline using Mössbauer spectroscopy (*Science* **351** 1303). Then this year, Eremets, Drozdov and colleagues set a new record at 250 K (–23 °C) and 170 GPa with lanthanum hydride. Aside from their high transition temperatures, these hydrides are attractive because, unlike cuprates, they appear to be describable with conventional BCS theory.

The goal is to find compounds that are as close to metallic hydrogen as possible. In 2017, Paul Loubeyre and colleagues at the French Alternative Energies and Atomic Energy Commission enlisted the ESRF's ID27 beamline to compress iron in a laser-heated diamond-anvil cell in an environment of excess hydrogen. With help from beamline responsible Mohamed Mezouar (above), they took X-ray diffraction measurements to show that the sample transformed into iron pentahydride ($FeH_5$), which contained layers of pure atomic hydrogen (*Science* **357** 382). That material has not yet been tested for superconductivity, but this year Dmitrii Semenok at the Skolkovo Institute of Science and Technology in Moscow, Russia, and colleagues used ID27 to attempt to synthesise thorium decahydride ($ThH_{10}$). They succeeded – and found that it turned superconducting at a temperature of around 160 K. Unlike other superconducting hydrides found to-date, $ThH_{10}$ stabilised at a relatively low pressure of 85 GPa (arXiv:1902.10206).

> "Suddenly, high-temperature superconductors are making headline news again"

tes: the superconducting phase, and the bad-metal phase (see fig. 1, p27).

Kivelson believes that there could be deeper physics linking all these phenomena. "We have to understand the problem holistically," he says. "It's not as simple as the charge-density wave being a mechanism of superconductivity, or the superconductivity being a mechanism of the charge-density wave. Instead, both of them are reflections of the same underlying physics."

Suddenly, high-temperature superconductors are making headline news again. In 2015, following some two decades in which they barely managed to raise superconducting transition temperatures at all, scientists began to find hydrated metals that turned superconducting at high pressures and at temperatures 70 K higher than the previous record. As well as these materials (see "Room temperature: within sight?", above), ESRF scientists have been exploring superconductivity in a newfound family of nickel-based compounds, which are analogous to cuprates.

Now, the results on charge-density fluctuations give scientists another window through which to study superconductivity. Ghiringhelli finds it hard to speculate how the fluctuations will ultimately shed light on the phenomenon, if at all. But one thing is becoming clear, even among sceptics: the importance of RIXS, as perfected at the ESRF's ID32 beamline.

"You get a new telescope, and you start to see more," says Zaanen. "And RIXS is like a new telescope." ■

**Jon Cartwright**


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# EVENTS

### CARAC2019: Accelerate your R&D

**28–29 November 2019**

From the publication of this issue, there are just a few days left (until 15 November) to register for this materials characterisation event for industry. The unique opportunity will see all the characterisation platforms based in Grenoble open their doors to people with a passion for innovation, with a business or a scientific background. Attendees will be able to meet scientists with worldwide expertise in materials characterisation, visit all the facilities, learn about cutting-edge techniques, and find solutions for R&D-acceleration and business support. R&D managers, scientists, innovation managers and product-developer managers will be present.

### EBS workshop: Sample modulation by high photon densities

**11–13 December 2019**

The EBS's very high photon densities will in many experiments modify the sample or its environment in a way that affects the collected data. Such effects include structural modifications, bond breaking, photopolymerisation, transport phenomena, redox processes, heating, change of macroscopic properties (e.g. electrical conductivity) or external conditions (e.g. ionisation of surrounding gas molecules). This workshop will make the entire ESRF user community aware of these problems by emphasising all aspects of sample alteration, which may in some cases actually be desired. Assessment of beam damage, determination of acceptable doses, plausible mechanisms as well as mitigating strategies will be discussed.

### EBS workshop: Cultural and natural heritage

**22–24 January 2020**

Whether it is using X-ray tomography to study ancient fossils, or micro-analysing fragments from impressionist paintings, the cultural and natural-heritage community is increasingly exploiting ESRF instruments. In this workshop, recent achievements and discoveries in palaeontology, archaeology and art science, based on the application of a large variety of X-ray techniques, will be highlighted. The emerging new opportunities offered by the ESRF–EBS as well as the new and improved beamlines will be discussed. Moreover, two satellite sessions will be proposed: one as an introductory session to explain the basic principles of the ESRF to non-experts, and one comprising tutorials on data analysis and practical X-ray tomography. Weather permitting, a tour of the street art in Grenoble will be also be organised on the final day, to end the workshop.

### User Meeting 2020

**3–5 February 2020**

See p11.

# BEAUTY OF SCIENCE

FABIEN CHAUVEAU & MARLENE WIART/CNRS

## Fifty shades of X-rays

Though it looks like an angry baboon, this image is actually an X-ray phase-contrast rendering of a section of a rat's brain. It is one of a dozen renderings taken at the ESRF beamlines ID19 and ID17 with the technique – which involves measuring the refraction of light, rather than absorption – exhibited in October at the Musée des Confluences in Lyon, France. The exhibition, "Virtual microscopy: Organs in fifty shades of grey", is the result of a collaboration between the ESRF and several research centres in Lyon. It will come to the ESRF later this year.


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FAST-PS-M
FAST-PS
FAST-PS-1K5
NGPS