

ESRF news

Number 79 June 2018

INSTRUMENTATION

The ESRF's strong hand

The story of colour photos

Serial MX: a new phase

Synthetic skin

ID02

LENGTH	99
MAX ENERGY	20
MIN BEAM SIZE	10
TECHNIQUES	4
SAMPLES/HOUR	100

ID32

LENGTH	120
MAX ENERGY	1.6
MIN BEAM SIZE	3
TECHNIQUES	4
SAMPLES/HOUR	0.01

ID30B

LENGTH	110
MAX ENERGY	20
MIN BEAM SIZE	20
TECHNIQUES	1
SAMPLES/HOUR	15

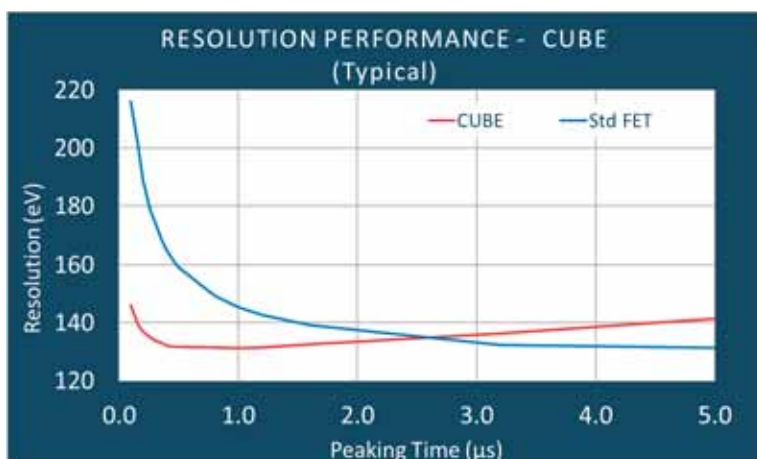
ID16A

LENGTH	185
MAX ENERGY	34
MIN BEAM SIZE	0.013
TECHNIQUES	2
SAMPLES/HOUR	0.25

SDD detectors for beam-line applications

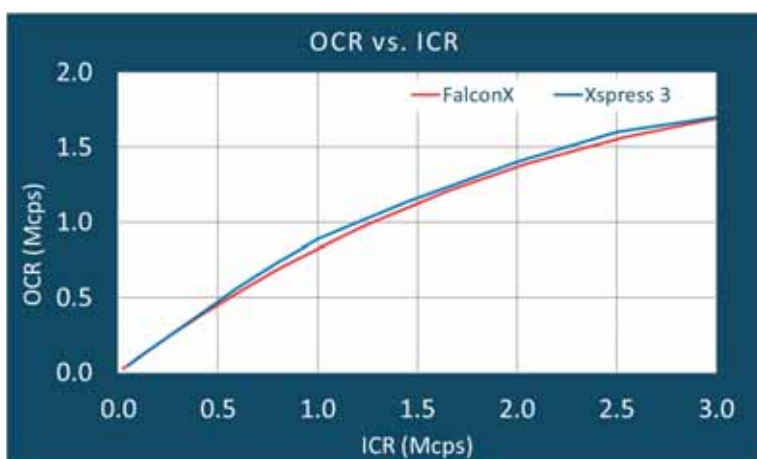
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Single and Multi-Sensor SDD Detectors



Example of single sensor design



Example of multi-sensor design with UHV compatibility

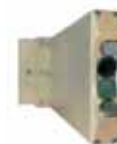
Design Features

- 1 to 19+ channels
- Sensors with active areas of 10, 30, 65, 100 & 170mm²
- Resolution from 126eV
- P/B >15k
- Focused or planar sensor arrangements
- High count rate to >4Mcps
- Windows: Thin Polymer / Beryllium / Silicon Nitride / Windowless
- Custom collimation and application specific designs
- High solid angle
- Slide options: manual / adapted for translation tables
- Gate valve and bellows available for UHV compatibility

Examples of Customised Designs



4 Sensor 'Beam Through' Detector
Circular Focused Array SDD



4 Sensor Vertical
Focused Array SDD



7 Sensor Circular
Focused Array SDD



Windowless SDD
design for UHV



Modular SDD
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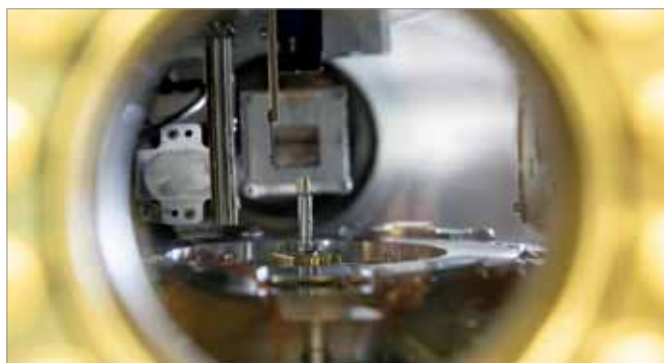
The European Synchrotron



Original recipes for colour photography, p12.



Chameleon-like skin synthesised, p14.



The focus of EBS instrumentation, p16.



On the cover:
Unique beamlines
at the ESRF, p19

ESRFnews Number 79 June 2018

EDITORIAL

- 5 An instrumental role

IN BRIEF

- 6 Russian membership ratified
6 Plastic precursor made from CO₂
7 New way to phase crystals
7 Dino flies like a pheasant
7 ESRF strengthens Israel bond
7 ID17 user wins award
7 New User Organisation chair

ESRF–EBS NEWS

- 8 Insight: the alignment process

10 USER CORNER

FEATURES

- 12 The birth of colour photos
14 Synthetic skin revealed

FOCUS ON: INSTRUMENTATION

- 16 The four pillars of the EBS
19 Brighter horizons
23 Science without borders

PORTRAIT

- 27 Ada Yonath

INDUSTRY

- 29 A fresh take on deodorants

BEAUTY OF SCIENCE

- 30 Crystal-clear alloys

IN THE CORRIDORS

- 30 Comet puzzle solved; DESY plans ahead;
LHC starts 2018 run; ESRF scientists speak at festival

Beamline Instrumentation

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An instrumental role

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The first international conference on Synchrotron Radiation Instrumentation (SRI) in Hamburg, 1982, arrived at the turning point for synchrotrons. Already light sources had a long history, with the first detection of synchrotron radiation using a table-top apparatus at the US company General Electric in 1947, and the construction of the electron-positron collider AdA at the Italian particle-physics lab Frascati in the 1960s, being two key milestones. But the 1980s was a time for bright – literally bright – ideas, for “third-generation” synchrotrons whose X-rays would be powerful enough to uncover secrets at the very heart of matter. And at the 1982 conference, there was talk of the possibility of a first such facility in Europe – the ESRF.

Unique to the ESRF was its full exploitation of insertion devices in the storage ring, to force the electrons to wiggle and thereby generate extremely brilliant X-rays. After operations began in 1994, techniques that were only just beginning to take form at second-generation sources – inelastic X-ray scattering, resonant inelastic X-ray scattering, X-ray emission spectroscopy, X-ray magnetic circular dichroism and X-ray photon correlation spectroscopy, for example – came to maturity. In other areas, the ESRF trod entirely new ground: X-ray phase contrast 3D-imaging and microscopy; a multi-technique extreme-conditions programme; a comprehensive industrial programme for macromolecular crystallography; and hard X-ray techniques for material science and palaeontology.

Between 2009 and 2015, the ESRF’s Phase I Upgrade Programme enabled the construction or total refurbishment of 19 experimental stations, offering new and often unique opportunities that are to this day world-leading in many areas (see pp19–20). But today, once more the ESRF is in the spotlight, commissioning a brand new storage ring based on an innovative hybrid multi-bend achromat design that will reduce horizontal emittance by a factor of 30, while maintaining a large dynamic aperture. That’s not all. The Extremely Brilliant Source (EBS) project also includes the construction of four new beamlines, several major refurbishments, and an ambitious Enabling Technologies programme resting on four pillars of instrumentation (see pp16–17).

It is part of the ESRF’s core mission as the European flagship for research using synchrotron radiation to share its expertise with stakeholders and the scientific community at large. The Frelon camera, single-crystal thin-film scintillators and crystal analysers for spectroscopy are just a few examples of the instrumentation that is produced in ESRF laboratories, and can be purchased via the Business Development Office or companies with which licence agreements have been established (see pp23–24).

This is the third year of the ESRF–EBS project, and what an extraordinary year. Exactly 30 years after the signature of the ESRF convention in Paris, on 10 December 2018 the iconic ESRF storage ring will be switched off for an unprecedented 20-month shutdown. We’re looking forward to discussing the project at this year’s SRI conference in Taipei, Taiwan, this month.

Michael Krisch, *Head of the ESRF Instrumentation Services and Development Division*

“Part of the ESRF’s core mission is to share its expertise.”



DELPHINE CHENEVIER/ESRF

Daniel Verwaerde of the CEA, Mikhail Kovalchuk of the NRC-KI and Antoine Petit of the CNRS shake hands.

Russian membership ratified

Russia officially became a member of the ESRF on 22 March, following a protocol of accession to the ESRF Convention entering into force that was signed back in 2014. To mark the occasion, the president of the National Research Centre Kurchatov Institute (NRC-KI) in Moscow, Mikhail Kovalchuk, the former administrator general of the French Alternative Energies and Atomic Energy Commission (CEA), Daniel Verwaerde, and

the president of the French National Center for Scientific Research (CNRS), Antoine Petit, took part in a ceremonial signing of the new statutes of the ESRF Company at the French Ministry of Research and Higher Education in Paris.

The signing took place in the presence of the French minister of higher education and research, Frédérique Vidal, the aide to the president of Russia, Andrei Fursenko, the chair of the board

of the Russian Foundation for Basic Research, Vladislav Panchenko, the chair of the ESRF Council, Miguel Ángel García Aranda, and the ESRF director-general, Francesco Sette.

Russian scientists have a long history of collaboration with the ESRF since the beginning of the facility in 1988. Last year, 92 Russian academic institutions and research laboratories were involved in experiments carried out at the

ESRF, demonstrating expertise in many fields of science: soft and hard condensed-matter, structural biology, medical and life sciences, applied materials science, chemistry, earth sciences, environment and cultural heritage, among others. Membership of the ESRF gives scientists in Russia even more scope to explore these areas, and will also help to nurture the Russian synchrotron user-community in general.

Catalyst puts CO₂ into plastics

The ESRF has tested a catalyst that enables the production of ethylene – an industrial precursor of many plastics – without resorting to fossil fuels. Even better, it sucks up carbon dioxide in the process.

Currently, ethylene is produced by cracking oil-derived hydrocarbons with steam at high temperatures. Its industrial output of some 150 million tonnes a year is more than for any other organic compound, largely because it can be made into polyethylene for plastic packaging. It is also used as a hormone to force the ripening of fruit in agriculture.

Created by Enrico Andreoli and colleagues of Swansea University in the UK, with help

from scientists at the University of Nebraska in Lincoln, US, the new catalyst is based on copper with a polyamide additive. In an electrochemical environment, using electricity from renewable sources, the catalyst converts carbon dioxide and water into ethylene. “We knew it was working, but we didn’t know why,” says Andreoli. To find out, his team visited the ESRF beamline ID03, which allowed the researchers to monitor the crystalline structure of the materials during ethylene production. “The polyamide doubles the efficiency of ethylene formation, achieving one of the highest rates of conversion ever recorded in standard bicarbonate water solutions,” Andreoli



Can plastic be green?

explains (*ACS Catal.* **8** 4132).

The next step for the team is to find industrial partners for the research, so that the process can be developed at a larger scale. “CO₂ emissions are a massive

issue, and we have found a way to put them to good use by making materials we need in very large amounts,” Andreoli says. “Industry will benefit from this as much as our planet.”

New phase for serial MX

A team of scientists at the ESRF, together with DESY and the Hamburg Centre for Ultrafast Imaging in Germany, has demonstrated that the damage induced by a macromolecular crystal during a synchrotron crystallography experiment can be exploited to determine the crystal's phase, and hence the molecule's structure.

It is often impossible in a macromolecular crystallography experiment to obtain a complete dataset from a single crystal before that crystal is destroyed by radiation damage. For that reason, scientists turn to serial crystallography, scanning multiple positions on a single crystal, or multiple crystals. To fit the various subsets of data together, in addition to the intensity of reflections, the scientists need to know the crystal phases—but these are hard to come by. One method for obtaining phases involves the introduction of heavy atoms, which bind to the macromolecular proteins and act as markers.

An alternative, in development at the ESRF since 2002, is to make use of the radiation damage itself. Because certain parts of a molecule are known to be more



STEF CANDE

Max Nanao exploits crystal damage at ID23-2.

susceptible to radiation damage, it is theoretically possible to allow the molecule to undergo just enough X-ray exposure for those parts to be altered, so that its phase can be discerned. "It's like burning holes in specific places," explains Max Nanao, scientist-in-charge of the ESRF's ID23-2 microfocus beamline.

Now, studying insulin

and thaumatin crystals at ID23-2, Nanao and colleagues have shown that the signal from radiation damage is big enough to overcome the variations between multiple crystals, so that the crystals can be phased. Key to the results was new software the team developed in 2016 to identify which subsets of data can be merged (*Acta Cryst. D* **74** 366).

Dino took to the skies, almost

Archaeopteryx, a bird from the late Jurassic period, has long been mooted as the first free-flying dinosaur. Now a study performed at the ESRF suggests that it did indeed fly—but only for short-distance hops, like a modern pheasant. Dennis Voeten of the ESRF—

together with colleagues from

the ESRF, Palacký University in the Czech Republic, CNRS and Sorbonne University in France, Uppsala University in Sweden, and the Bürgermeister-Müller-Museum in Solnhofen, Germany—used phase-contrast synchrotron microtomography at the ID19 beamline to compare the wing bones of three

Archaeopteryx specimens with those of modern birds. The match was "closest to birds like pheasants that occasionally use active flight to cross barriers or dodge predators," Voeten explains. The surprise result implies that avian flight must have originated before the late Jurassic (*Nat. Commun.* **9** 923).

Israel–ESRF links reinforced

In March, scientists attended the Israel Academy of Sciences and Humanities in Jerusalem for a workshop to strengthen links between scientists in Israel and at the ESRF. Discussions centred on the importance of synchrotron science to the Israeli scientific community, and included a talk by the Nobel laureate and long-term ESRF user Ada Yonath of the Weizmann Institute of Science (see p27).



ID17 user wins award

A collaborator on the ESRF's ID17 beamline has been awarded a prize for the best scientific paper at the European Congress of Radiology two years running. Giacomo Barbone, a medical-physics PhD student in the group of Paola Coan (see below) at Ludwig Maximilian University in Munich, Germany, presented a paper about advanced tomography on ID17 to study the effects of Alzheimer's disease on the brain. He was praised for the quality of his presentation and its impression on the audience.



New UOC chair

The ESRF User Organisation Committee has a new chair: Michela Brunelli, a scientist on the ESRF's Dutch–Belgian beamline, DUBBLE. Brunelli takes over from the former chair, Paola Coan of the Ludwig Maximilian University in Germany. The aim of the committee is to promote ESRF research through discussion within the user community, be a direct link between the users and the ESRF management, and to organise the ESRF's annual User Meeting.

Daniel Schirr-Bonnans, a technician in the ESRF's Survey and Alignment Group, checks the alignment of an EBS sextuple magnet.



Insight: The alignment process

Why is alignment so important?

For the new machine to function correctly, and to make good its promises of boosted X-ray brightness and coherence, each piece of equipment needs to be precisely positioned on the EBS girders. The most sensitive magnets, for example, need to be aligned to within 50 µm of theoretical specifications – that's half the width of a human hair.

How hard is that?

Components can be aligned initially with a metal positioning jig, but this is nowhere near precise enough. Also, jigs give no assurance of the location of critical interior parts of the machine – the centres of magnets, for

example, or the insides of vacuum chambers – which are inaccessible once assembled. The solution is a process called fiducialisation – from a Latin word meaning “trust”.

What does fiducialisation involve?

Machined into the girders, and on the surfaces of major components, are small inverted cones, which form a network of reference points. The challenge is to adjust the alignment of the components and girders until these numerous reference points precisely match the coordinates on the EBS blueprint. Once all the reference points match the blueprint – and there are more than 1200 of them – the EBS team can be sure

that everything, including the interior features, are in perfect alignment. Of course, this means measuring the distances between the reference points, and to do this the technicians place spherical reflectors inside the cones (see image, above), and expose them to a sophisticated laser tracking system. It's a painstaking process.

Why?

Because components have to be aligned in layers, like a Russian doll. First, a bare girder is levelled and a reference network installed and measured on the girder itself. In the next step, a magnet is installed on the girder with a jig to within half a millimetre, before its centre is aligned by laser tracking.

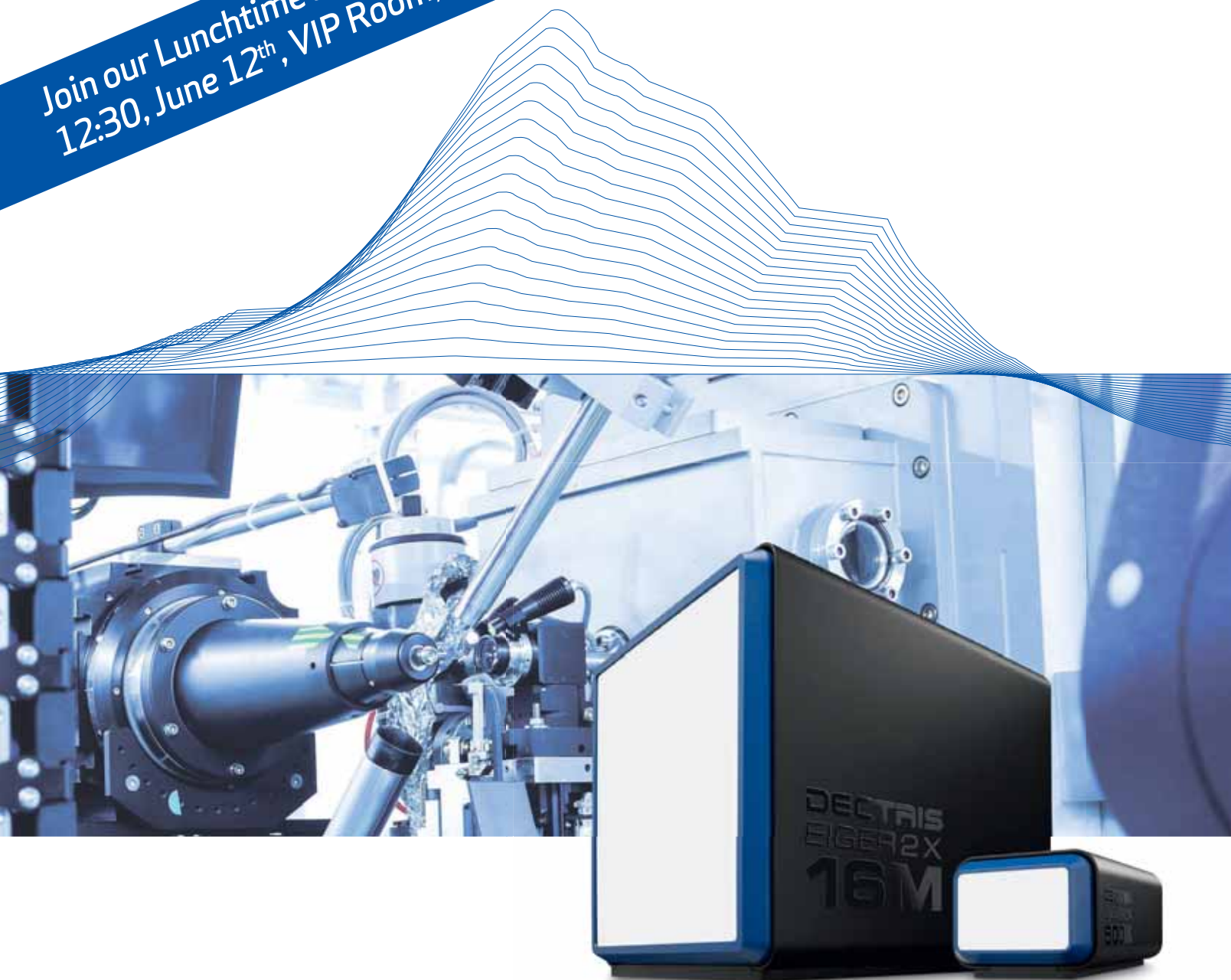
Then the magnet is opened up and the vacuum chamber installed – a tricky process, as the gap between the edges of the vacuum chamber and the poles of the magnet is less than a millimetre. Inside these chambers are beam-position monitors, which are also linked (fiducialised) to external reference points for alignment. Only once this is done can the magnets be reassembled, and a final alignment check performed.

How long does all that take?

The alignment of one complete girder takes two people three days to complete. With 128 girders in total, and six to eight pairs of hands, the EBS team has got its work cut out.

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News from the User Office

At the last proposal submission deadline on 1 March, 1240 proposals were submitted, requesting a total of 16,360 shifts of beam time. That brought the total number of proposals received for beam time in 2018 to a record-breaking 2544. On 26 and 27 April, 117 external scientists met together at the ESRF to evaluate these new proposals and provide recommendations for beam-time allocation: we thank them for all their hard work on this important task.

Most panel members will now begin a well deserved break, only meeting again in April 2020 after the EBS shutdown to review the first batch of proposals aiming to exploit the new source. (Next submission deadlines: 15 January 2020 for Long Term Project proposals, and 1 March 2020 for standard and MX BAG proposals). Only the Structural

Biology panel will continue to be active, reviewing proposals for the Cryo-EM facility, which will continue to run as normal during the long shutdown. Applications for time on the Cryo-EM facility can be submitted as rolling applications at any time.

In the operation year 2017, the ESRF delivered a total of 20,408 shifts for user experiments (16,578 to the public programme and 3830 to the private CRG programme). These were delivered in more than 2628 experiment sessions, with more than 9000 user visits made by more than 5000 different scientists. Operation year 2018 will finish prematurely on 10 December 2018, but we aim to provide as much time as possible to users before the shutdown begins.

Joanne McCarthy, Head of the User Office

News from the User Organisation Committee

The ESRF User Organisation is currently working on the format of the next User Meeting, which will take place in Grenoble on 4–6 February 2019, during the long shutdown of the ESRF storage ring. We encourage users to contact us with any suggestions for topics that they would like to see for tutorials and user-dedicated microsymposia.

Meanwhile, the User Organisation is undergoing a number of changes. Representatives of the Complex Systems and Biomedical Sciences user community, Roberta Angelini (CNR-ISC Rome, Italy), and of the X-Ray Nanoprobe user community, Paola Coan (Ludwig Maximilians University, Munich, Germany) have ended their terms on the User Organisation. Calls have opened for nominations for new members to represent these two communities, and

elections will be held, allowing the respective communities to choose their new representative. Until then, Roberta and Paola will still be available as representatives.

We thank Roberta and Paola for the fruitful work that they have accomplished in recent years, which has shaped new and lively ESRF User Meetings. On 8 April, Michela Brunelli took over the role from Paola Coan as chair of the User Organisation Committee (see p7).

Any users who have questions, comments or ideas are welcome to contact the User Organisation at any time via e-mail.

Representatives of each user scientific community and their contact details can be found at www.esrf.eu/UsersAndScience/users_org.

Michela Brunelli, Chair of the User Organisation Committee

News from the beamlines

- The Swiss-Norwegian CRG, **SNBL**, is continuing the preparation of its beamlines for the restart in 2020. In anticipation of the new Extremely Brilliant Source (EBS), SNBL's combined spectroscopy/diffraction end-station was already successfully transferred from BM01B to a new infrastructure on **BM31** (see *ESRFNews*, December 2016, p10). As a next step, a new high heat-load monochromator designed and manufactured by Instrument Design Technology IDT-LTD in the UK has been installed on **BM01**. This should ensure stable operation and an immediate restart in 2020 for this highly requested and efficient crystallography beamline.

- The refurbishment of **BM08 (LISA)** has been completed and the beamline is now back to full operation. The X-ray optical elements have been completely renewed and a new experimental hut has been created. The beamline now operates with a liquid-nitrogen-cooled monochromator equipped with two pairs of crystals [Si(311) and Si(111)] giving access to an energy range of about 4–90 keV. A first cylindrical mirror is used for beam collimation and a second toroidal mirror is used for focusing. A



The new 14-crystal high-resolution spectrometer on BM16 (FAME-UHD).

long bench has been installed in the new experimental hut with a wide space available for standard equipment and/or users' instrumentation. Tests show a beam size of 200 μm , a flux in the range 10^{10} – 10^{11} ph/s and crystal rocking curves close to theoretical values. The beamline is now open to users with a very intense experimental program.

- Since January, a new 14-crystal high-resolution spectrometer under its helium box (CE certified) is fully operational on CRG beamline **BM16 (FAME-UHD)**. Designed and built by the beamline team and equipped with bent crystals made mostly at the ESRF Crystal Analyser Laboratory, the spectrometer (see photo) allows the measurement of an XAS signal in fluorescence mode with a ~ 1 eV energy bandwidth.

XAS measurements can now be achieved on diluted elements, down to one part per million or lower (*J. Environ. Qual.* **46** 1146).

- In the context of the ESRF–EBS Upgrade, a new French CRG beamline will be built on port **BM07**, replacing the present **FIP-BM30A** beamline dedicated to macromolecular crystallography (MX). The future beamline, named **FIP2**, will be a flexible and highly automated beamline designed to host a large variety of MX experiments, including anomalous phasing, spectroscopy coupled diffraction, *in situ* diffraction, etc. The construction of the infrastructure of **FIP2** on port **BM07** should start by the end of April 2018 so that the beamline is available following the restart after the long shutdown.

- When beam time is awarded through the public user

programme, users of the **Structural Biology group** beamlines can now choose to request the reimbursement for transport costs of sending a dewar of samples to the ESRF in place of the reimbursement of the travel and subsistence costs of a user. Most Structural Biology MX beamlines offer Remote Access data collection, while MASSIF-1 and the Cryo-EM offer Mail-In services. The possibility of obtaining financial support for sending samples instead of users to the ESRF is therefore a move that aims to continue to support the structural biology community as these modes of access to the beamlines evolve. See www.esrf.fr/MXDewarReimbursement.

- The **Structure of Materials group** has refurbished and made available for the user programme a prototype of an X-ray image intensifier. The system has a rather coarse pixel size of around 100 μm , so is suitable for fast diffraction experiments or time-resolved (Bragg diffraction) X-ray imaging. Due to the short afterglow of the phosphor used (700 ns), it is compatible with high-speed acquisition schemes (up to MHz) and can resolve the bunch-structure of the ESRF synchrotron in 4-bunch mode.

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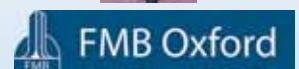
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How colour was born

The ESRF exposes secrets behind the earliest colour photographs.

He may not be a household name, but Louis Ducos du Hauron is believed to be one of the inventors of full-colour photography. The precise ingredients that went into making each of his images has always been something of a mystery, but now, thanks to ESRF techniques, researchers have been able to shed light on them for the first time.

"Through energy tunability, and an ability to analyse on the microscale, this study makes excellent use of the synchrotron to answer questions that could not be answered utilising traditional analytical methods," says Art Kaplan, a leading conservation scientist at the Getty Conservation Institute in Los Angeles, US, who was not involved with the work.

Photography has a foggy past, but it certainly became popular in the first half of the 19th century with the introduction of the metallic daguerreotype process by the French inventor Louis Daguerre, and the salted-paper process by the British inventor Henry Fox Talbot – both of which produced black-and-white images. Although others toyed with adding colour pigments, it was Ducos du Hauron, a French pianist and scientist, who first captured the world as our eyes see it, by producing full-colour photographs.

Radical recipe

His method began with taking three simultaneous black-and-white negatives of a scene, through green, orange and purple filters. In the next step, he printed

"The synchrotron has answered a question that could not have been answered by traditional methods."

a positive monochrome image for each of these negatives in the complementary colours – red for green, blue for orange and yellow for purple – on semitransparent films, light-sensitised by dichromate. Finally, he assembled the prints on top of one another to create a coloured photograph. If the method sounds simple in principle, in practice it was anything but, and Ducos du Hauron spent more than four decades refining it after his first attempts in the early 1860s.

Together with his brother and assistant Alcide, Ducos du Hauron recorded many of his recipes and developments. Unfortunately, it has not always been clear which recipe is behind which surviving photograph. This is a problem not only for historians, but also for curators who want to know which methods of restoration and conservation are safe. "If you know what materials are there and how they were used, you can choose one strategy for restoration over another," says Marine Cotte, the scientist in charge of the ESRF's ID21 beamline.

Big team

To determine the best conservation methods, curators at the Musée d'Orsay in Paris teamed up with researchers from the Centre for Research and Restoration of the Museums of France and the École Nationale Supérieure Louis-Lumière, also in Paris, with additional contributions from the museums of Agen and Nicéphore Niépce in Chalon-sur-Saône, the Academy of Sciences and independent conservators. The team studied huge volumes of letters, books and patents, as well as 27 of Ducos du Hauron's photographs; of these 27, the researchers sampled microscopic fragments from three photographs and sent them to Cotte's group at the ESRF for additional infrared and X-ray microanalysis, including X-ray absorption near-edge structure spectroscopy.

The power of the ESRF data was to

Lessons from the masters

The early photographers may have struggled with colour pigments, but painters have had just as much trouble obtaining a decent white. From ancient times until the 19th century, white pigment generally came from allowing lead to corrode in vinegar, although the quality – and cost – depended on the precise recipe. To determine which grades of lead white were used by the old masters, researchers at Parisian museums have sent samples of oil paintings to the ESRF beamlines ID21 and ID22 for analysis by X-ray diffraction. The white in a painting by Leonardo da Vinci, *The Virgin and Child with Saint Anne* (right), appears to have undergone extended treatment in vinegar; conversely, the white in a painting by Jean de Beaumetz, *Calvary with a Carthusian monk* (far right), shows signs of treatment with hot water, to remove impurities (*Anal. Chem.* **89** 13203).



identify and locate different components in what were highly complex samples. One important result was that Ducos du Hauron had apparently added castor oil to his mix, probably to improve plasticity, which according to Cotte may be why some of his photographs have turned yellow over time. Another result from a sample dated 1884 was that collodion (otherwise known as nitrocellulose) was present – not in the underlying film as described by the photographer in a patent 14 years later, but on the surface, probably functioning as a protective varnish, the researchers say (*Angew. Chem.* **130** 1). The results come at a particularly active time for the history of art, with 2018 being labelled as the European Year of Cultural Heritage (see “Preserving culture”, right), and with another ESRF study exploring the origin of oil pigments (see “Lessons from the masters”, opposite).

Kaplan says the identification of pigments

and additives is all “important information”, although he points out that the current results may not allow the unambiguous dating of prints. For instance, although it may appear that the specific location of collodion – on the surface, or in the underlying film – could mark a print as having been made before or after Ducos du Hauron filed his patent, that relies on the shaky assumption that the photographer was consistent in his approach. “It’s not uncommon for an artist to step back and utilise older materials during his development of improvements,” Kaplan explains.

Nevertheless, Kaplan believes that the study is encouraging, and looks forward to more synchrotron studies of this kind. “This being one of the earliest, successful colour processes makes it very intriguing in better understanding the early history of colour photography and the development of the pigment process,” he says.

Jon Cartwright

Preserving culture

Culture must be preserved for its vital importance to individuals, communities and societies. That’s according to the European Commission (EC), which has named 2018 the European Year of Cultural Heritage. The year will see a series of initiatives and events across Europe to encourage people to become more involved with their cultural heritage, including special funding under Horizon 2020 and other EC research programmes.

“Cultural heritage shapes our identities and everyday lives,” an EC statement reads. “It surrounds us in Europe’s towns and cities, natural landscapes and archaeological sites. It is not only found in literature, art and objects, but also in the crafts we learn from our ancestors, the stories we tell to our children, the food we enjoy in company and the films we watch and recognise ourselves in.”

This view of Agen in France is one of Ducos du Hauron’s most memorable colour photographs.



Epic dermis

Scientists have successfully mimicked biological skin, and it even changes colour.



There is a lot more to skin than being thin and rubbery. Aside from protecting us against disease, regulating temperature and healing itself, skin has special mechanical properties – it is soft yet strong, and tough yet flexible. It also has the particular characteristic of stiffening when it is stretched, which helps to prevent injury.

Now, for the first time, scientists have tested a synthetic material that mimics these properties at the ESRF – and it even changes colour, like a chameleon. Future versions of the material could be used for adaptive camouflage, or for more realistic prostheses. “Our materials may be programmed to cover a wide range of mechanical and colour characteristics,” says study author Dimitri Ivanov of the Lomonosov Moscow State University in Russia.

The mechanics of biological skin arise from the fact that it is made up of two contrasting proteins: a scaffold of stiff collagen fibres, interwoven with elastin. When the skin stretches, its stiffness initially rises exponentially before switching to a linear response. This is tricky to mimic in synthetic elastomers and gels, the stiffness of which tend to increase steadily.

Together with Ivanov, Sergei Sheiko of the University of North Carolina at Chapel Hill and colleagues have solved the problem with their development of a “bottlebrush” polymer, consisting of a backbone with side chains. The backbone forms a soft matrix, while the flexible side chains turn rigid when forced together.

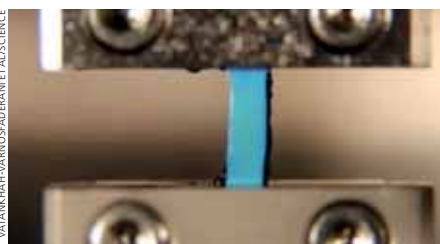
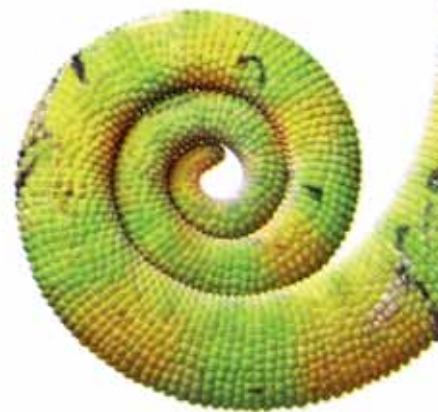
The result is an elastomer that stiffens intensely when stretched, very much like biological skin. What’s more, stretching reduces the distance between the side chains, meaning that the elastomer’s iridescent colour shifts from turquoise to dark blue (*Science* **359** 6383).

Several of the researchers brought samples of the material to the ESRF beamline ID02, for analysis with ultra-small-angle X-ray scattering. The material was characterised while static, and also when stretched, making use of an *in situ* stretching apparatus. “The need to use synchrotron radiation, and in particular the ESRF, is explained by the fact that the structure of these materials exhibits organisation at very different spatial scales,” says Ivanov, one of the users. For instance, the colour results from molecular separations on the sub-micron scale, while the structure of the bottlebrush polymers themselves can be seen on the ångström scale.

The initial results bode well for medical implants, which need to have similar mechanical properties to biological tissues to reduce the chances of inflammation or rejection. And the material could be used for more than artificial skin: many other tissues, including the intestinal wall and the heart muscle, share the special stiffening mechanics. The precise properties of the material can be tuned by altering the make-up of the polymers. “This approach is similar to coding our hereditary information in DNA strands,” Ivanov explains.

Jon Cartwright

“The ESRF allowed us to see the organisation at very different spatial scales.”



Left: In its regular state, the elastomer is light blue. Stretch it, and its colour deepens.

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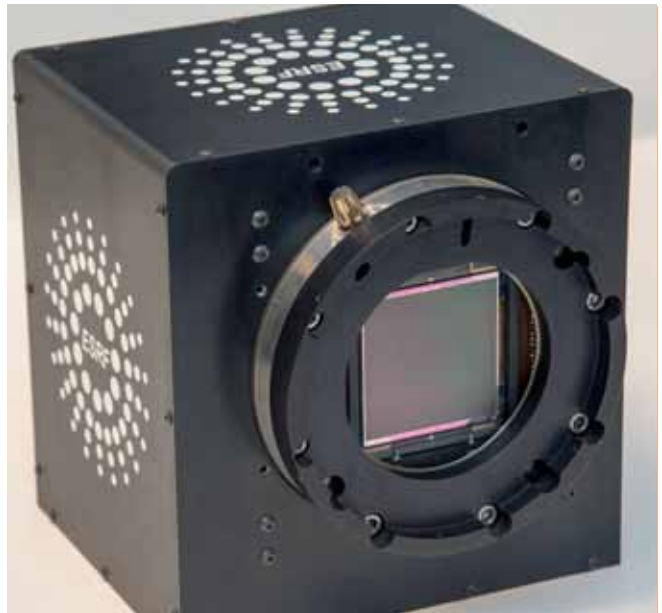
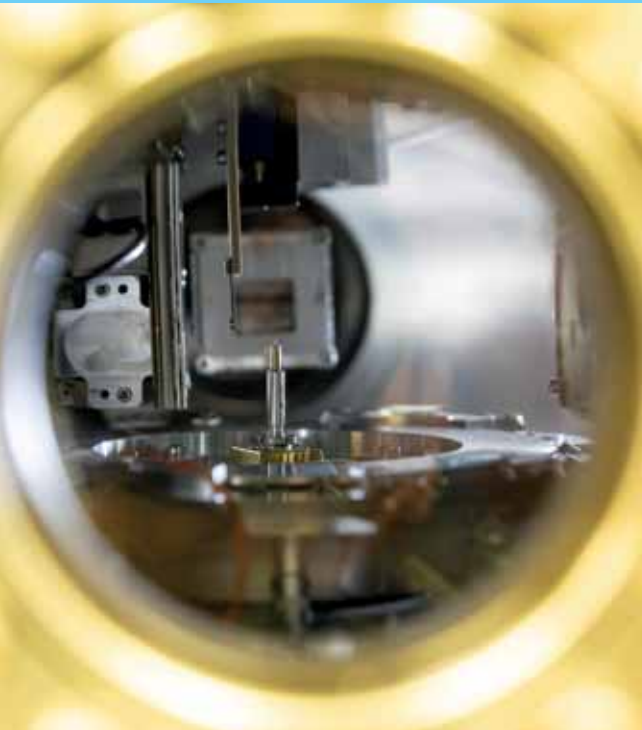
The performance of the ESRF's Extremely Brilliant Source upgrade w

OPTICS

With both coherence and brilliance 100 times greater than the current source, the ESRF's Extremely Brilliant Source (EBS) upgrade will set a new benchmark for fourth-generation synchrotrons when it launches in 2020. So far as actual beamline performance goes, however, none of this will be much use without optics of matching quality. Optics transform the raw X-ray beam into a form best suited for a particular experiment, by tuning characteristics such as shape, energy, divergence and polarisation. Get the optics wrong, and a beamline could lack precision and stability, and even degrade over time.

That is why the ESRF-EBS project has a special development programme to address optics and metrology. The programme intends to minimise the spatial and temporal perturbation of X-ray wave-fronts and transfer X-ray flux to samples as efficiently as possible. It requires input from optical and mechanical modelling, systems design, process development, and testing outside and inside beamlines. Crystal optics, multilayer optics, mirror optics, metrology projects, refractive lenses and bent-crystal analysers will see particular attention.

The ESRF has a strong pedigree in optics. Only last year, the ID16A beamline set a new resolution record for high-energy X-rays, thanks to the installation of a new, multilayer mirror (see p19). Besides nano-focusing, techniques such as imaging and coherent scattering rely strongly on the quality of beam manipulation. But the impact of the optics and metrology programme will be far-reaching, giving a lift to the ESRF-EBS's entire beamline portfolio.



DETECTORS

Few aspects of instrumentation capture the attention of users so much as detectors, a change in which can instantly affect the amount of data generated in an experiment by orders of magnitude. The ESRF is always improving its detectors, and in the next year or two, several ongoing projects will come to fruition, such as the next-generation Frelon camera (see p24). For the EBS, however, a dedicated detector development plan (DDP) aims to go further. Aside from developing advanced new detector systems, the DDP involves collaborating with external laboratories to access other detectors already in development, as well as improving related technologies that will help maintain the quality and productivity of ESRF-EBS beamlines.

The detectors themselves are being developed along two lines. The first, SMARTPIX, will be a photon-counting hybrid pixel detector with a 55 μm pixel pitch, based on the MEDIPIX3RX readout circuit developed by the international Medipix3 Collaboration, of which the ESRF's Detector and Electronics Group is a partner. SMARTPIX will be faster and more flexible than previous versions, and it will also have the potential for greater temporal resolution. The second line of development is a series of large-format detectors for high-energy diffraction. These include fast, high-dynamic-range detectors, charge-integration detectors and high-dynamic-range detectors for indirect detection.

That's not all. To supplement detector development, the ESRF is advancing three strategic technologies: X-ray sensors, which convert the X-ray photons into a measurable signal; control and data-acquisition, which manages the throughput of data; and energy-dispersive detection technology, which involves the use of silicon drift diodes and germanium detectors to record the number and energy of X-rays emitted from a sample.

the future

will rest on four pillars of instrumentation, as Jon Cartwright reports.

CONTROL AND MECHATRONICS

With the launch of the EBS, many users will see data-acquisition times drop from milliseconds down to microseconds, and lower. That poses an issue for control systems, which will need to be more responsive in order to keep up, while maintaining synchronisation with the EBS storage ring.

It isn't purely about timing, however. As more and more experimenters look towards the nanoscale, and want to perform their experiments more efficiently, control systems also need to become both more precise and more automated. This is where mechatronics can help. The ESRF already has plenty of expertise here, for example with the almost fully automated MASSIF beamlines at ID30, which can be set up to evaluate and probe macromolecular crystals without human intervention. For the EBS, a mechatronics task force has been created to work between groups within the ESRF's Instrumentation Services and Development Division, and to provide a platform to share expertise in modelling, mechanics, electronics and software. A mechatronics laboratory is also forthcoming.

Various areas will see the benefits. Currently in development, a new double-crystal monochromator – the optical component that filters bundles of X-rays down to a single wavelength – will incorporate mechatronic concepts to improve precision. Similarly, the Nano-positioning and Active Stabilization Stage, or NASS, project – part of a collaboration with engineering physicist Christophe Collette at the University of Brussels in Belgium and colleagues – is working on a prototype for a new generation of highly integrated end-stations.



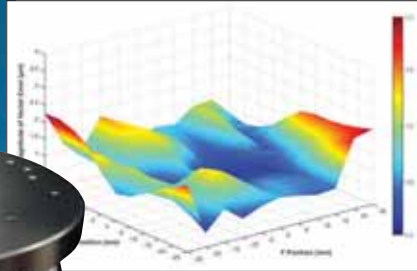
SOFTWARE

Faster experiments at higher resolution can only mean one thing: more data. So much data, in fact, that making sense of it will be a challenge in itself. To adapt their measurement strategies in real-time, users will need new, sophisticated ways of visualising data. They will also need new ways of processing it onsite or online, as many of them will lack the appropriate computing facilities at their home institutions.

Fortunately, solutions are in the pipeline. The foundations for a new beamline control system, BLISS (which stands for BeamLine Instrumentation Support Software), have been laid, and it is already being deployed on the ESRF beamlines ID15 and ID31, as well as those of the Structural Biology Group. Meanwhile, software engineers have been busily developing SILX, a library of common data-analysis routines, generic applications and software. Now in its sixth release, SILX is being used in an increasing number of areas, and has been demonstrated to cut data-analysis times dramatically.

Myriad other changes behind the scenes will help funnel the data deluge. For example, a new ESRF data policy requiring metadata to be systematically recorded is being implemented; it is present on 11 beamlines already and will become the standard on a further 10 beamlines every year, until they are all covered. In the future, the policy could extend to acquiring and storing data in the flexible "HDF5" format, and standardising its management, archiving and publication.

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Brighter horizons

The ESRF has long been pushing the frontiers of X-ray instrumentation, as its current beamlines show.

About a decade ago, former ESRF scientists Giulio Monaco and Simo Huotari, together with ESRF engineer Christian Henriquet, were faced with an intractable problem. How to improve both the energy resolution and photon flux of inelastic X-ray spectroscopy (IXS), when each parameter comes at the expense of the other? "All they had in front of them was a pencil and a blank sheet of paper," recalls Christoph Sahle, the current scientist in charge of the ID20 IXS beamline. "There were no other instruments in the world they could refer to."

This is a common story at the ESRF, which has been at the forefront of X-ray instrumentation for about 30 years. Staying ahead in this regard is the way that the synchrotron has consistently delivered such high-quality science, in the form of more than 30,000 papers. But while attentions are now focused on the launch of the ESRF's major upgrade, the Extremely Brilliant Source (EBS), it is almost easy to forget that much of the current set of instrumentation on offer is still first class.

IXS at the ID20 beamline (left) is one example. Its predecessor was at ID16, which, like other IXS beamlines the world over, attempted an unsatisfactory compromise between energy resolution and photon flux in the same instrument, only allowing minor modifications between experiments depending on the most important criterion. Monaco, Huotari and Henriquet's answer was not one but two specialist spectrometers – one for extremes of resolution, and the other for extremes of flux. Installed at ID20 as part of the ESRF's Phase I Upgrade Programme in 2012, with user operation the following year, these dual spectrometers set records for resolving power and largest-detectable solid angle for an IXS instrument. "Thanks to the high resolution, we can measure electronic and magnetic excitations, and track their dispersion with unprecedented accuracy," says Sahle. "And thanks to the the large solid angle, we can measure non-resonant IXS signals from samples that were thought impossible to measure just five years ago."

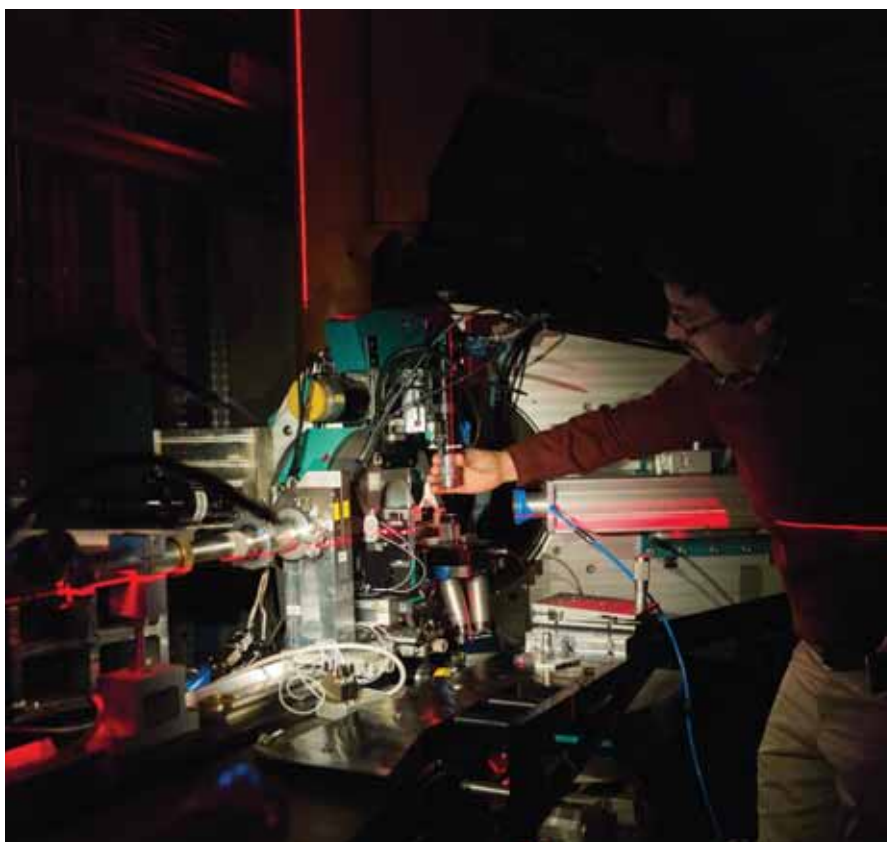
Many options

Many other ESRF beamlines host unique experimental abilities (see "One of a kind", overleaf). Like ID20, ID32 explores the electronic and magnetic structure of materials, and also has record-breaking energy resolution. But ID32 is targeted at lower energy, "soft" X-rays, where the "3d" electronic states of transition metals from scandium to zinc on the periodic table, and the "4f" electronic states of the rare-earths, can be probed. This makes ID32 ideally suited for studies of high-temperature superconductors and other materials with complex magnetic and electronic ordering. The presence of the bright red, 12 m-long spectrometer of ID32 is hard to miss in the ESRF's Belledonne experimental hall, which has special foundations to prevent vibrations compromising the tight mechanical tolerances. More than five times the length of its predecessor on ID08, the ID32

One of a kind

Unique ESRF beamlines include:

- **ID01** (right) offers perhaps the easiest-to-interpret data on the structure, chemistry and mechanical properties of materials
- **ID02** uncovers changes in the structure of soft matter and biological systems from the ångström to micron scales, in sub-millisecond time-steps
- **ID15A** is unrivalled in its ability to pinpoint material changes inside real devices in working conditions
- **ID16A** holds the record for the highest resolution of images taken deep within samples
- **ID20** investigates the electronic and magnetic properties of functional and extreme states of matter, at record-breaking energy resolution
- **ID24** can obtain the local and electronic structure of a material in extreme conditions, in just 0.1 nanoseconds
- **ID31** has a unique portfolio of X-ray techniques to study real devices in their working environments
- **ID32** maps variations in the electronic and magnetic structure of materials at the highest energy resolution
- **MASSIF-1** records the structural data of protein crystals automatically, day-in, day-out



PERREJANET

spectrometer arm can move six tonnes of instrumentation over 100° in angle without breaking the vacuum, in part thanks to an engineering collaboration with the Italian company Cinel.

Another hard-to-miss beamline is ID16A, which at some 200 m is the ESRF's longest. Last year, it became the world's first high-energy beamline to focus towards 10 nm, a spot size that verges on the diffraction limit. The achievement was made possible by the installation of a new pair of elliptically profiled X-ray mirrors, figured and polished by JTEC Corporation in Japan before coming to the ESRF's Multilayer Laboratory for coating with 120 layers of tungsten and boron carbide. On the opposite side of the storage ring, ID01 also functions as a nanoprobe, albeit for diffraction rather than imaging, but its special feature is its ease of use – helped recently by the development of online analysis software that can swiftly provide optimised feedback on terabytes of data. "We may not be winning the nanometre race, but we think that we're winning the practicality race," says Tobias Schulli, ID01's scientist-in-charge.

When it comes to boosting throughput,

"The ESRF has led the way in X-ray instrumentation for nearly 30 years."

however, the MASSIF beamlines at ID30A-1 and -3 surely win hands down. With its robotic sample changer from the Swiss mechatronics company Stäubli, MASSIF-1 in particular can collect millions of diffraction images for macromolecular crystallography every month, with practically zero human intervention. It is operated by the ESRF and the EMBL Joint Structural Biology Group, and provides a unique service to users, especially those from the pharmaceutical industry who need to sift through masses of datasets to find instances where test drugs have successfully bound to target proteins.

Special combination

Meanwhile, on the border of the life and physical sciences, ID02 has established itself as the go-to beamline for science involving transient structures over vastly different length scales – from a few ångströms to several microns (more than four orders of magnitude), at a time resolution down to the sub-millisecond range. "It is this combination that makes ID02 unique for elucidating static and transient hierarchical structures with small-angle scattering," says Theyencheri Narayanan, scientist-in-charge. Key to the beamline's function is the hugely variable distance to the detectors, made possible by a vacuum tube 34 m long and 2 m wide, co-developed by the ESRF and the Spanish company Added Value Solutions.

Versatility is not always a benefit. Before the Phase I upgrade, ID15 was a multi-purpose high-energy beamline, but under ever-increasing demand it was redesigned as a more specialist beamline (ID15A) for

chemistry and materials engineering. The beamline is equipped with a state-of-the-art Pilatus CdTe detector, especially designed for ultrafast measurements with very high-energy X-rays. Combined with a very intense, focused beam, it can obtain space- and time-resolved maps of the interior of real devices, such as catalytic reactors, batteries and fuel cells, in working conditions. Meanwhile, in its special engineering hutch, ID15A can probe deeply into very big objects – the turbine blade of an aeroplane engine, for instance.

Experimental conditions are also critical to ID24 – in this case extremes, be they of pressure, temperature or magnetic field. The beamline's real distinction, however, is speed. It was the world's first beamline for energy-dispersive extended X-ray absorption fine-structure (EXAFS) spectroscopy to record a full spectrum with one X-ray pulse, taking just 100 ps. "This is what has made it so famous," says Sakura Pascarelli, the scientist-in-charge. Most famously, in 2016, the ESRF's Raffaella Torchio and co-workers were able to observe the effects of a shock wave in transit through an iron sample, after the sample was blasted with a 35 J laser – an experiment that no-one else has subsequently been able to repeat.

A new high-power laser facility, with an energy of 100 J, is set to come online as part of the EBS upgrade, allowing for science at yet more extreme conditions. Over the next couple of years, the EBS will dramatically improve all other aspects of instrumentation too (see pp16–17). Until then, however, if you want to experience much of the best in X-ray instrumentation, the ESRF is still the place to be. *Jon Cartwright*



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Science without borders

The ESRF's expertise in instrumentation has impact beyond the EPN campus.

Engineers, as Melissa Rivadeneira knows all too well, do not always make the best marketers. Passionate about their craft, focused on extracting the very best from the available technology, they are more comfortable talking about pixels and microns than euros and cents. Rivadeneira's challenge is to make them see the fruits of their labours outside the ESRF. "We want to share this technology with industry, and with other synchrotrons," she says.

There are good reasons for this. From the smallest X-ray intensity-monitors to complete beamlines, the ESRF is already home to much of the world's best in synchrotron instrumentation (see pp19–20), a lot of which has been the product of decades of in-house research and development. Sharing it with other light sources and industries improves the quality of research that can be performed worldwide. It also brings in welcome revenue. Last year, the ESRF made about €0.5 m from technology transfer, all of which goes towards hiring staff, buying better equipment and generally improving the service that the facility provides for users.

Rivadeneira (above), 24, has been supporting the ESRF's technology-transfer efforts for nearly two years, bridging the gap between the Business Development Office (BDO) and the Instrumentation Services

and Development Division. Born in Quito, Ecuador, she came to Grenoble after high school and trained in electronics and software before obtaining a masters in technology and innovation management. After that she spent three years as an apprentice engineer for the CEA, before joining the ESRF. She sees her present job as simplifying the process of tech transfer and valorisation in general – that is, exploiting technology beyond its original purpose – to minimise the time that engineers have to be involved. "Because of my technical background, they are happy to talk to me, and tell me in their words what they are developing," she says.

"Sharing technology with others improves the quality of research worldwide."

Ed Mitchell, head of the BDO, believes Rivadeneira has brought a new perspective to how technology transfer is performed at the ESRF. "She'll often look at a process and say, why on Earth are you doing it this way – why don't you do it like this?" he says. "And she's usually right." He hopes that valorisation is something that the ESRF can grow in the future. "We're one of the few light sources in the world that has a dedicated instrumentation division," he says. "So we do a lot of development, and we tend to be at the leading edge of new technologies."

Obviously, some pieces of instrumentation are worth more than others. Designed by Jean-Claude Labiche, a former ESRF engineer, Frelon cameras have for nearly a quarter of a century been the go-to imaging detectors for fast readout and low noise (see "Swansong for Frelon", overleaf). The ESRF has even been approached in the past to share the engineering of almost complete beamlines. But these are big-ticket items that tend to be one-offs. At the other end of the scale, the scintillators produced by the ESRF's liquid-phase epitaxy lab are relatively inexpensive, but have sold in their hundreds.

The scintillators are a great example of in-house technology that can't be found elsewhere. Essential for converting X-rays into visible-light photons, which can be detected



Swansong for Frelon

The ESRF may have been a revolutionary light source when it switched on in 1992, but, according to Jean-Claude Labiche, imaging detectors were stuck in the dark ages. Every time a one-megapixel image was recorded, the best detector of the time needed a sluggish 20 seconds to refresh itself. “It meant that only 0.1% of the beam could be used,” recalls Labiche.

A new recruit to the ESRF’s detector group, Labiche saw two options: continue to develop the then-common technology of gas-filled detectors, or turn to CCD cameras, which at that time were alien to synchrotrons and used only by astrophysicists. He opted for the latter. By 1994, his prototype fast-readout, low-noise (Frelon) camera – “Frelon” being the French word for “hornet” – was demonstrating

recording times of 0.2 seconds, a 100-fold increase. It was a fast, plug-and-play system that was ideally suited to ESRF users, because if anything went wrong they could simply request an in-house replacement. As Labiche explains, the sensor inside the Frelon could have been bought by anyone – the ESRF’s ingenuity was in the integrated electronics, which enabled the fast data acquisition.

Today, about a quarter of ESRF beamlines use Frelons, and they are sold to other synchrotrons worldwide. And upgrades have continued apace. In May this year, the latest incarnation delivered image recording in a lightning 12 milliseconds – although this may be a developmental swansong, as Labiche has just left the ESRF. “It’s a very, very big improvement, but now it’s finished,” he says. “I’m in retirement!”

What next for the Frelon? Theoretically CMOS detectors will supplant CCDs eventually because of their selective, and hence even faster readouts, but these are still too expensive for the relatively niche world of synchrotrons. As a result, Labiche reckons the Frelon has another 10 years – for which users of the ESRF and other light sources will surely be grateful.

**“Upgrades
have continued
apace.”**

on a CCD or CMOS detector, scintillators are made the world over, but only at the ESRF are they produced consistently and with resolutions down to 300 nm. “The real asset of our scintillators is to have sub-micron resolution, which is quite difficult with some other commercial products,” says Thierry Martin, head of the detector unit.

The epitaxy lab produces about two scintillators a day on average. As the ESRF only needs about 10–15 a year, the rest can be sold to other light sources. But Martin’s is just one of several specialist labs at the ESRF; others – some of which are devoted to crystal analysers, mirrors and metrology, crystal polishing, and multilayers – are also able to share their expertise with external clients. Other valuable

ESRF instrumentation includes the Dynaflo Cryostat for sample cooling, Maxipix detectors for fast-readout photon-counting and beam viewers. Almost all software is available, too – although in this case for free, according to the ESRF’s open-source policy.

New horizons

The ESRF upgrade, the Extremely Brilliant Source (EBS), is opening up new possibilities for technology transfer, with two patents having already been filed. One of these is for the radio-frequency “fingers” that ensure the continuity of the electrical conductivity between vacuum chambers, to reduce the danger of sudden electrical discharges. The other is for the EBS’s “hot swap” system,

which allows power-supply components to be removed safely without turning off the power.

Indeed, according to Rivadeneira, almost all ESRF technology has the potential to be sold or licensed. When she first came to the ESRF, her dream was to create her own company that would help research and development in Ecuador, by building links between research organisations in France and universities in her home country. Now, however, that dream is on hold, as even the technology transfer officer is realising that there is more to be found at the ESRF than she had at first appreciated. “I just want to grow, and develop more technical skills,” she says. “I want to learn everything that I can from here.”

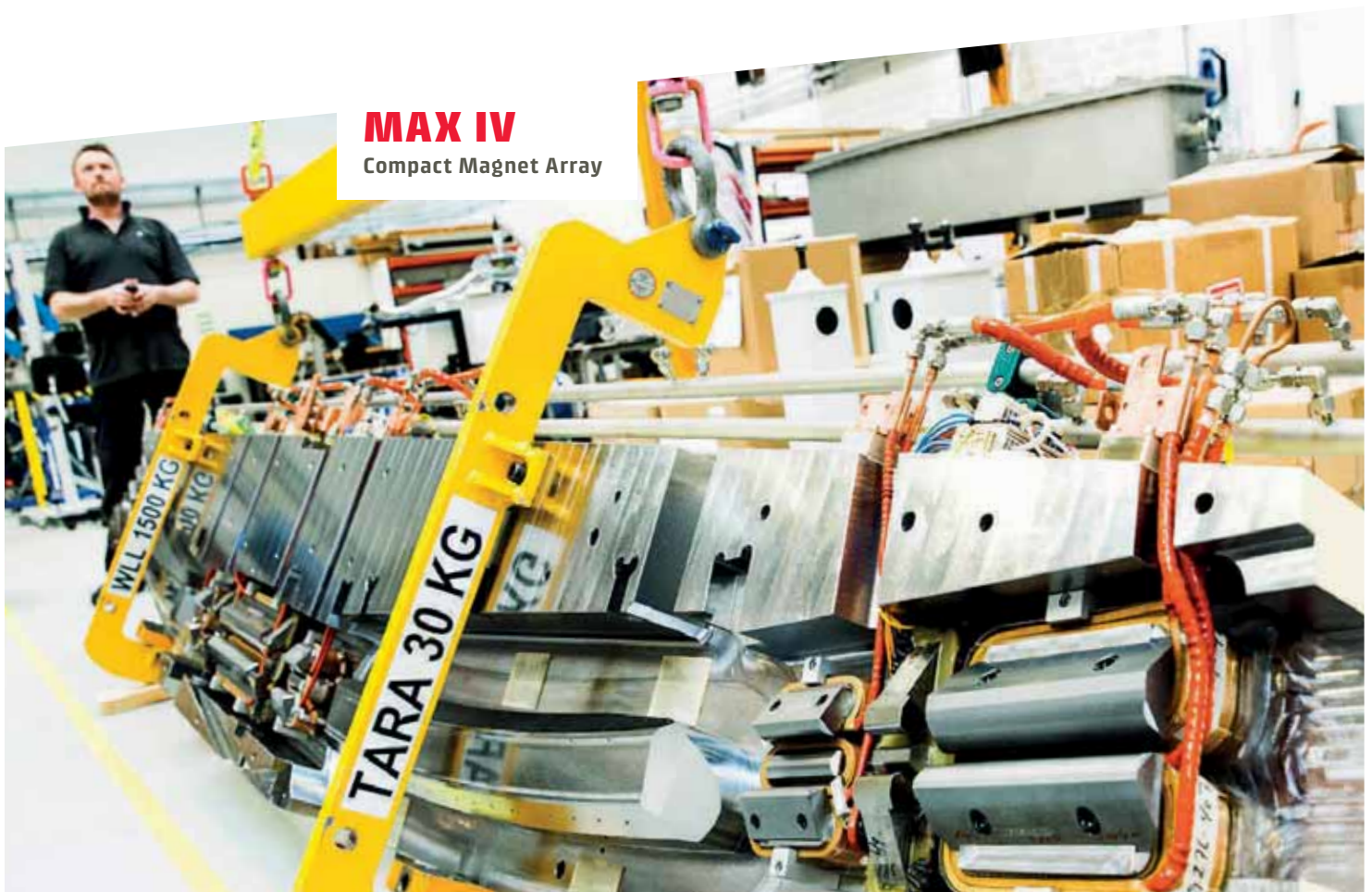
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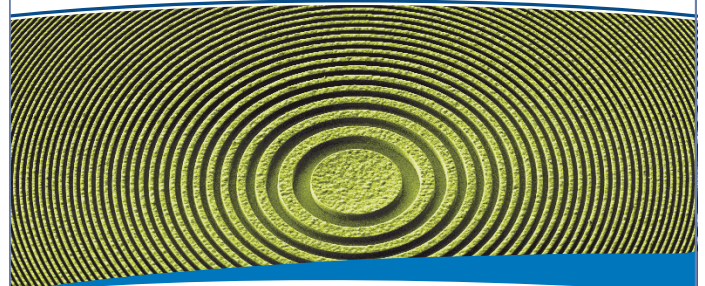
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Celebrating the best

Ada Yonath, winner of the 2009 Nobel Prize in Chemistry, talks to Jon Cartwright about the importance of instrumentation.

JC: How important has the ESRF been for your research?

AY: The ESRF is among the main sites where we have collected our data over the years. Actually, most of our publications are based on data that we've collected at the ESRF, and our group was among the first to use the facility when it opened in 1994. Of course, we've taken measurements at many synchrotrons around the world – in the US, Germany, the UK and Japan – but the ESRF is among those that provide a very high-quality beam.

JC: Is it true that you have given guidance on structural-biology instrumentation to the ESRF?

AY: Before coming to the ESRF we took measurements in several international high-quality facilities. There I gained a lot of experience and was happy to share it with ESRF staff, who were always open to new ideas. I'm extremely happy to see that the ESRF maintains itself as a state-of-the-art facility. An example close to my scientific interest is its recent installation of the cryo-EM facility. It's led to a resolution revolution in structural biology.

JC: You won the Nobel Prize in Chemistry in 2009 for your work on the ribosome, which is also studied in the context of antibiotics. It seems that only in the past year or so the topic of antibiotic resistance has made regular headlines in the media, despite it being a long-term problem. Do you think policy and research is now headed in the right direction?

AY: I hope so. Of course, as always, there are many scientific and medical concerns about current research and policy. This is why we're still researching how to design the next generation of antibiotics, which will be species-specific – therefore with reduced resistance – and eco-friendly.

JC: Is that what you're working on right now?



Ada Yonath in brief

Born: 1939, Jerusalem.

Education: BSc chemistry (1962), master's biochemistry (1964), PhD X-ray crystallography (1968).

Career: Postdoc, Carnegie Mellon University, US (1969); Massachusetts Institute of Technology, US (1970); scientist, Weizmann Institute of Science, Israel (1970); lecturer, Tel-Aviv & Ben Gurion University, Israel (1971); consultant, The Open University, Israel (1971); visiting scientist, University of Alabama, US (1974); visiting scientist, University of Chicago, US (1977); visiting professor, Universidad Austral de Chile (1978); visiting professor, Max-Planck Institute for Molecular Genetics, Germany (1979); senior scientist, Weizmann Institute (1974); associate professor (1984); chair, department of structural chemistry and structural biology (1989); head, Max-Planck Research Unit, Germany (1986); director, Mazer Center for Structural Biology, Weizmann Institute (1988); Kimmel Professor (1988); director, Kimmelman Center for Biomolecular Assemblies (1989).

“Most of our publications are based on ESRF data.”

AY: Yes. Specifically, how to deal with resistance to antibiotics that act by paralysing the translation of the genetic code.

JC: Tell me about a historical figure whom you find inspirational.

AY: Marie Curie. The reason is her curiosity and desire to understand natural processes, her passion for her investigations and her high human values.

JC: There are only four female Nobel laureates in chemistry: you, Marie Skłodowska Curie, Irène Joliot-Curie and Dorothy Crowfoot Hodgkin. You have the additional accolade of being to date the only female Israeli laureate in any category. How do you feel when we focus on these facts in the media?

AY: Actually, almost all of my interactions with students, scientists and policymakers have nothing to do with female, Israeli etc. issues. And that's the way I like it. But the media is more interested in fashionable topics like feminism, therefore publicises it more, giving the impression that most of my lectures are centred on these issues.

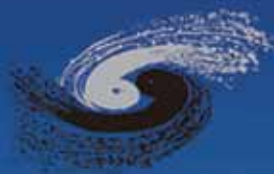
JC: When you are not at work or spending time with your family, what do you do to relax?

AY: I have some other interests and activities, such as swimming, reading and listening to music.

JC: The ESRF is celebrating its 30th anniversary in September. Will you be having a glass of champagne with us?

AY: Sure, if time permits. Thanks for the invitation!

Jon Cartwright



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Deodorants: a fresh approach

L'Oréal visits the ESRF to probe the mechanism of antiperspirants.

Deodorants have been in popular use for well over a century, so it is perhaps surprising that no-one knows for certain how they work. Those that function as antiperspirants are generally based on aluminium salts, and one theory is that these salts have an astringent effect, shrinking the sweat pores. Another theory is that the salts affect how sweat is secreted by sweat glands in the first place. Yet the third, and most widely held, theory is that the dissolved salts diffuse into the sweat ducts where they hydrolyse in contact with sweat, forming an aluminium hydroxyde "plug".

The precise mechanism is important for a cosmetics company such as L'Oréal, because it helps direct researchers towards more effective products. "For our experiments, the ESRF was the best choice," says Jean-Baptiste Galey, a researcher at L'Oréal's research and innovation centre in



Aulnay-sous-Bois, France.

To understand the mechanism of antiperspirants, Galey and his colleagues developed a T-shaped microfluidic device to mimic a sweat pore: a channel 50 µm wide for the pore itself, connected to a channel 400 µm wide for the skin surface. Into the pore end of the device the researchers injected a

model of human sweat, consisting of saline solution with additional serum albumin, a protein found in human sweat but in this case taken from cows. Into the other end they injected an aluminium salt, aluminium chlorohydrate.

At the ESRF beamline ID13, Galey and colleagues could study the structure and composition

of the resultant material using small-angle X-ray scattering, resolved in space and time. The results supported the theory of plug formation, but it was not that simple: the growth of the plugs seemed to occur only where diffusive and hydrodynamic flows achieved concentrations of proteins and aluminium polycations with balanced electrical charges. The presence of sweat proteins seemed to be crucial, too, since they "feed" the plug by being constantly brought by sweat flow. (*Soft Matter* **13** 3812).

"These results open up perspectives to find new antiperspirant agents with an improved efficacy based on the size and charge of reactive species found in our work, and on their ability to react with sweat proteins and proteins present at the surface of sweat pores," says Galey. *Jon Cartwright*



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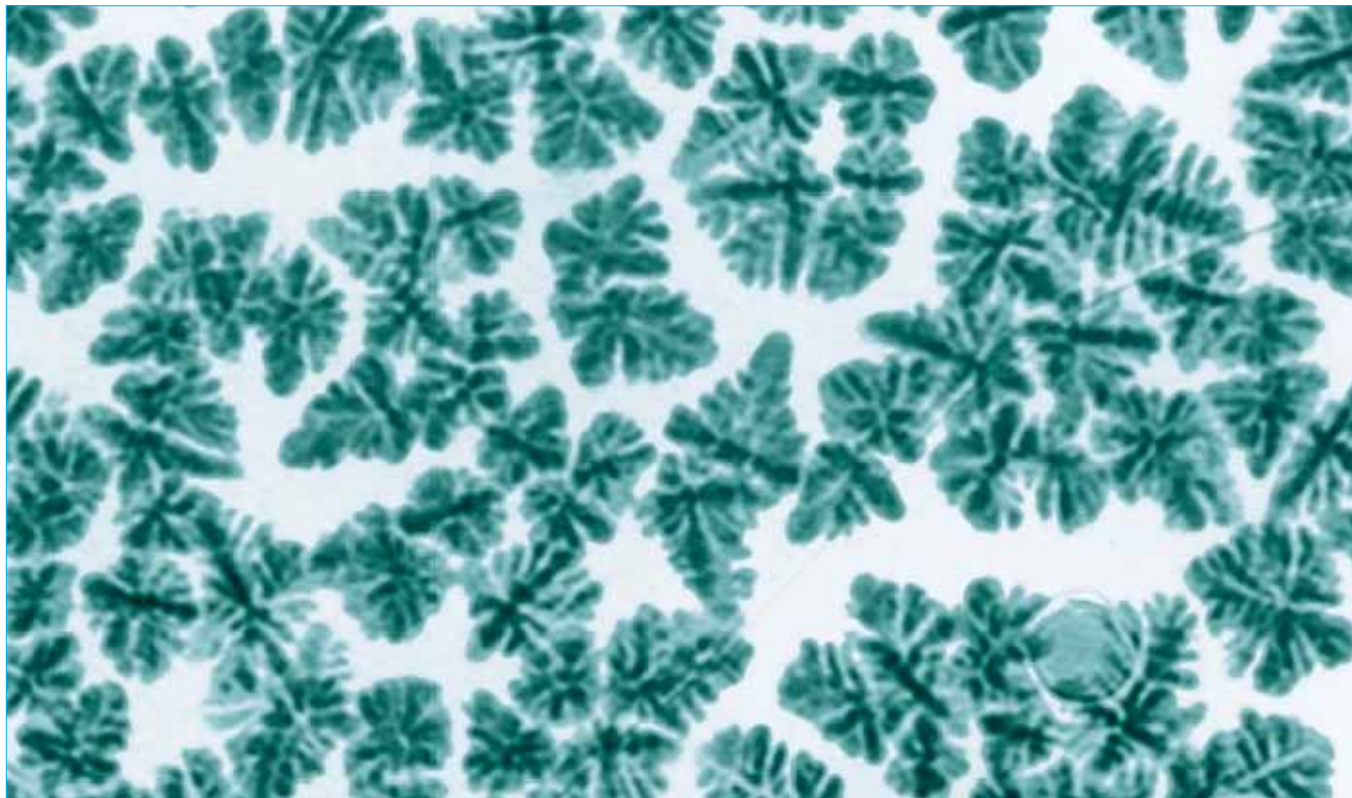
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Crystal clear: The way solid crystals nucleate during the cooling of a liquid metal alloy is critical to obtaining the best final material properties. Occurring over very short timescales and at the micro- to nanoscale, however, crystal nucleation is hard to study. Enzo Liotti at the University of Oxford in the UK and colleagues have managed to do so using a bespoke solidification rig at the ESRF's ID19 beamline, with help from a computer algorithm that automatically spots the onset of new crystals in the image data. The ID19 image above (falsely coloured) is typical of the growth of aluminium crystals in one of the researchers' aluminium-copper alloys (*Sci. Adv.* **4** eaar4004).

In the corridors

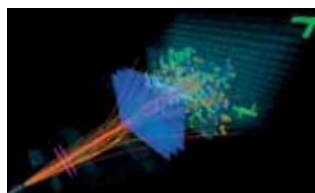


Comet puzzle solved

Comets are famous for their visible tails, which are formed due to their interaction with the solar wind. Less well known is that – despite being cold – comets sometimes also emit X-rays. Now an international team of scientists thinks it knows why. In the lab, Alexandra Rigby at the University of Oxford in the UK and colleagues directed a burst of plasma towards a solid sphere to simulate the solar wind striking a comet, and found that, thanks to turbulence, the temperature of electrons in the plasma rose to about a million degrees; in the presence of a magnetic field (similar to the Sun's), they generated X-rays (*Nat. Phys.* doi:10.1038/s41567-018-0059-2).

DESY looks to 2030

The German laboratory DESY has set forth a strategy for the 2020s that includes an expansion of the European X-ray free-electron laser, the building of a centre for computer science and the construction of its next-generation synchrotron, PETRA IV. According to the document, *DESY 2030: Our strategy for the future*, a detailed concept for PETRA IV will be presented by next year, with a technical design report following in 2021. The first user experiments could come as soon as 2026. DESY is a member of the Helmholtz Association of German scientific research centres, which have an annual budget of €4.5 bn.



LHC starts 2018 run

Machine operators of CERN's Large Hadron Collider (LHC) injected 1200 bunches of protons into the accelerator at the end of April, marking the beginning of the 2018 physics season. As in previous runs, the LHC experiments will be searching for deviations from the Standard Model of particle physics, which provides the best explanation so far of all known particles and forces, but which does not account for (among other things) dark matter and dark energy. This will be the last year of collisions at the LHC before the machine enters a long shutdown until spring 2021 for upgrades, and the operators hope to gather more data than ever before.

ESRF goes for a pint

ESRF scientists have put aside any reservations about British imperial units to participate in Pint of Science, a global festival that engages people in science in the comfort of their local pub. The festival has run for five years, but this year is the first that ESRF scientists have participated, with Tobias Schüllli and Steven Leake of the ID01 beamline giving talks in Grenoble on the topic of "light, the universe, crystals and the nano-world". The festival was initiated as a single event in 2012 by two researchers at Imperial College London in the UK, but quickly became popular: it now reaches 40 cities in France, and some 300 cities in total worldwide.



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