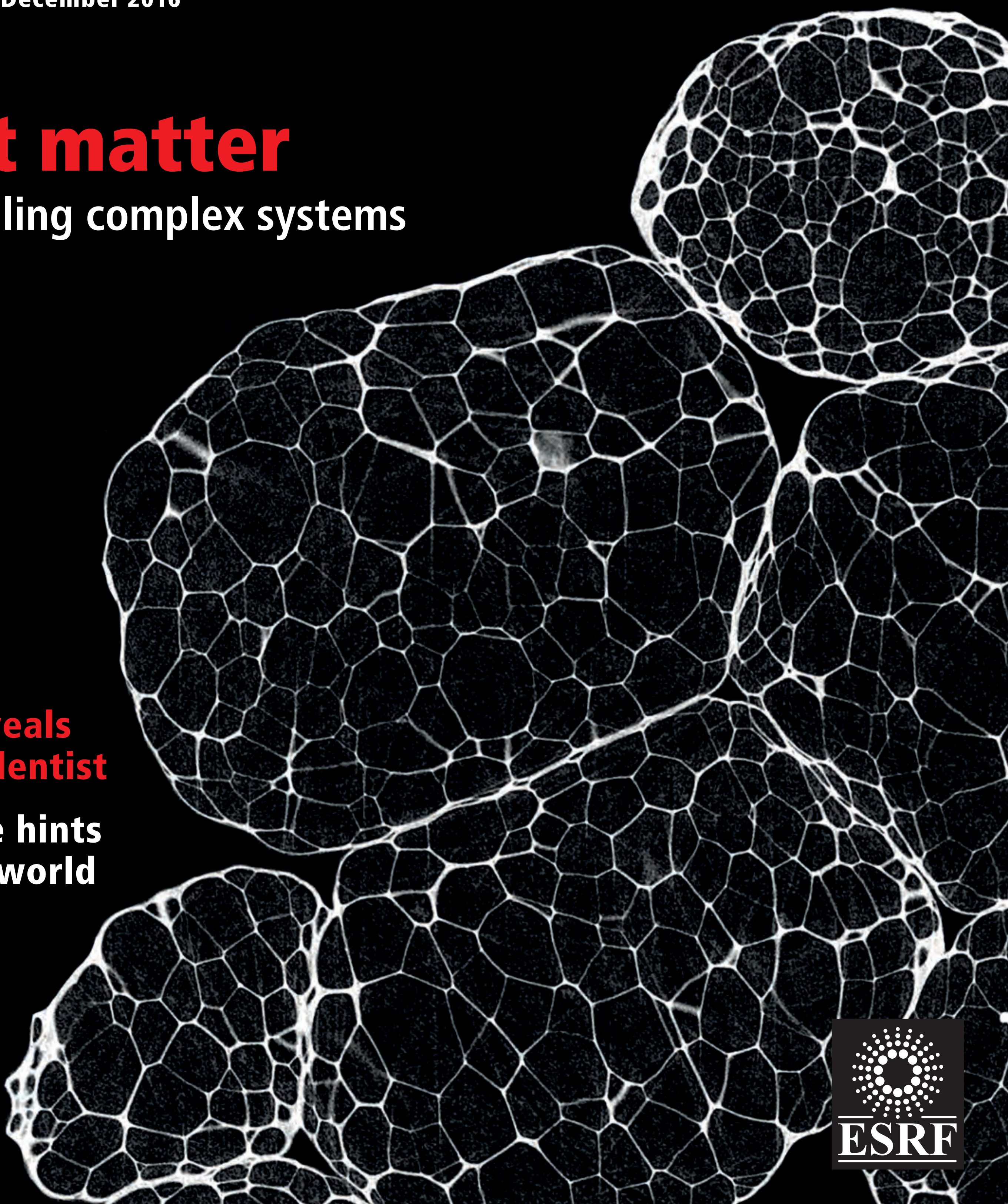
A LIGHT FOR SCIENCE

ESRF news

Number 74 December 2016

Soft matter

Untangling complex systems



Hair reveals trip to dentist

Microbe hints at RNA world

ESRF

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X - RAY SPECTROSCOPY

Exciting news.....
SGX Sensortech (MA) Ltd is now RaySpec Ltd!

SDD detectors for beam-line applications

Improved Resolution & Count Rates

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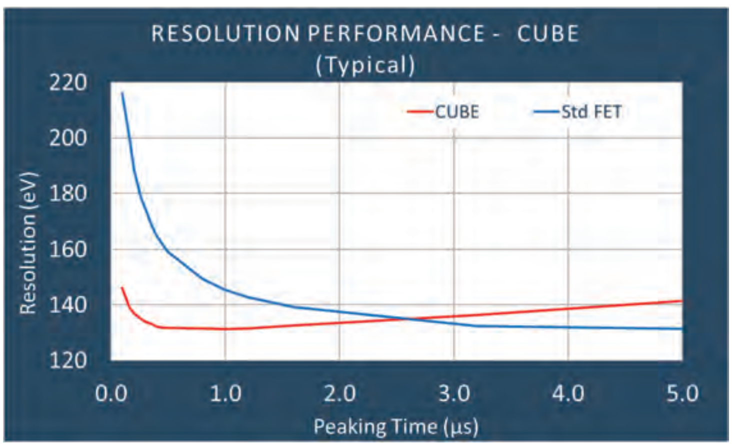

CUBE detectors offer improved resolution at shorter peaking times

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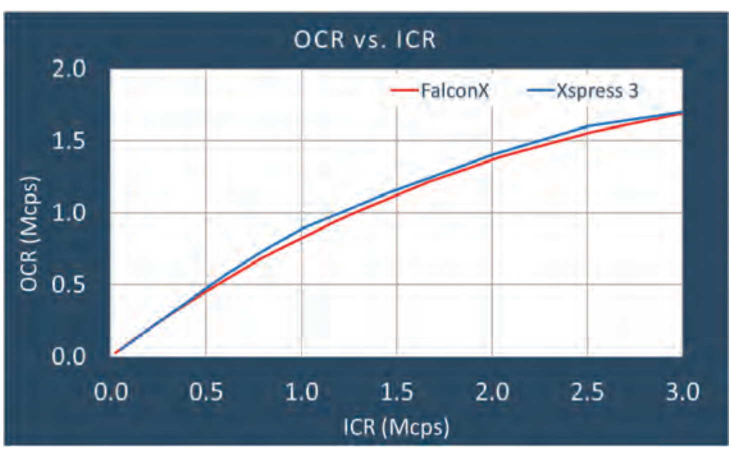
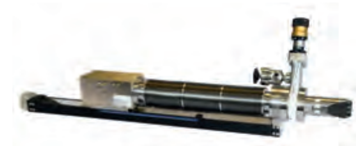


RaySpec SDD detectors - compatible with the latest Digital Pulse Processors from XIA and Quantum Detectors with input count rates >3Mcps

Single and Multi-Sensor SDD Detectors

Example of single sensor design



Example of multi-sensor design with UHV compatibility

Design Features

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Examples of Customised Designs

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4 Sensor Vertical Focused Array SDD

7 Sensor Circular Focused Array SDD

Windowless SDD design for UHV

Modular SDD Vacuum Compatible

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RaySpec Ltd
Sirius House, Watery Lane, High Wycombe, HP10 0AP, UK

Tel: +44 (0) 1628 533 060
Email: sales@rayspec.co.uk

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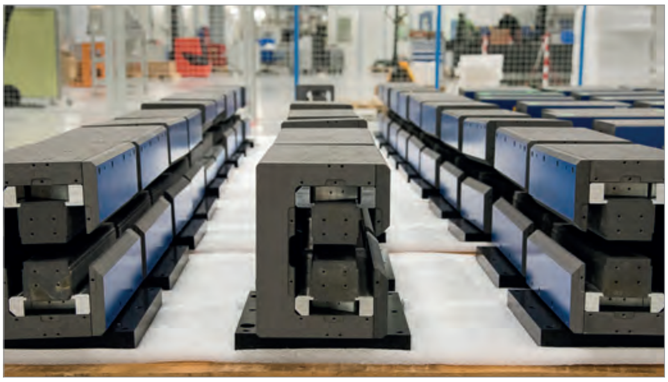
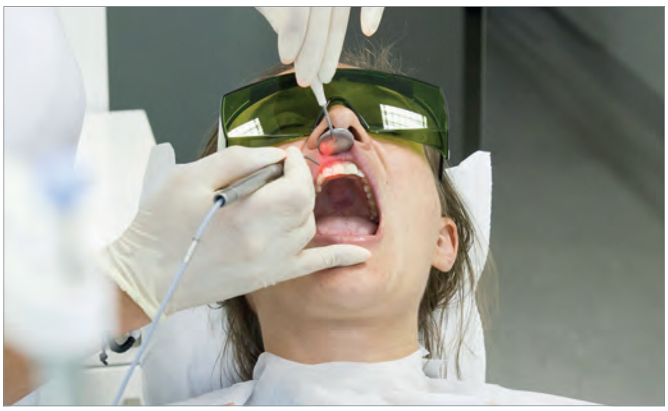
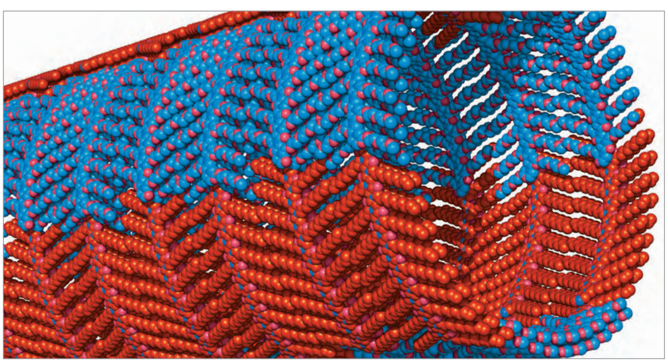


First EBS dipoles assembled in-house, p9.



X-ray analysis of a hair strand reveals past dental work, p12.



Scientists mimic self-assembly of natural proteins, p16.

On the cover: Microtomography of a cross-section of car safety foam (p28)

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ESRFnews

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Soft science, strong impact

Soft matter science is a truly interdisciplinary field of research, crossing the traditional borders between condensed matter, physical chemistry, biophysics, and bio, chemical and materials engineering. In simple terms, soft matter refers to anything that feels soft to the touch. More scientifically, it refers to a variety of physical systems that have a characteristic energy scale comparable to room-temperature thermal fluctuations, but have structures organised over much larger scales than regular solids. That definition includes liquids, liquid crystals, colloids, polymers, detergents, foams, gels, granular materials and of course many biological materials.

This year, the ESRF had the privilege of organising, together with the ILL and local institutions, the International Soft Matter Conference (p15). The conference helped to showcase the EPN Science campus's synchrotron and neutron activities, which are ideally suited to the investigation of the structural organisation of soft matter. Indeed, the ESRF has had a strong portfolio in the field from the outset. At present, activity related to soft matter corresponds to roughly one-eighth of accepted proposals, and in the last five years has generated more than 1000 publications.

One of the main reasons users studying soft matter come to the ESRF is that soft-matter systems of interest today are complex, and involve a hierarchy of structural scales: all of these can be accessed in reciprocal space by scattering methods, with nanobeams allowing us to see the larger range in real space. Other reasons are the high brilliance of the ESRF's X-rays, which allow users to investigate time dependent effects following an external perturbation, and the X-rays' high coherence, which allows the probing of equilibrium dynamics in these systems. Finally, soft-matter processes often take place at interfaces, which can be studied with surface-sensitive X-ray scattering.

"The EBS will bring a paradigm shift for soft matter research"

Probing the pathways of soft-matter, biomimetic and bioinspired self-assembly in the bulk and at interfaces (p16), the slow dynamics of arrested systems such as gels and glasses, and the elucidation of biological structure–function relations (p23) are some of the active areas of soft-matter research to which the ESRF has contributed – not to mention various industry collaborations (p21). Of growing importance is the establishment, jointly with the ILL, of the Partnership for Soft Condensed Matter (p25), which helps users to make better use of our infrastructure by offering sample preparation facilities, complementary characterisation techniques and a platform for scientific discussion and collaboration.

Until now, X-ray scattering has been the primary technique at the ESRF for soft matter research. That will change with the Extremely Brilliant Source, which will see the X-ray coherence boosted by two orders of magnitude. Indeed, the source upgrade, together with higher specification detectors, will bring about a paradigm shift for soft matter research, allowing unprecedented time resolution in dynamic studies and lens-less coherent diffractive imaging for the direct imaging of complex structures at the nanoscale.

Theyencheri Narayanan, *former head of soft matter group*
Jean Susini, *director of research for life sciences*


L'ORÉAL FOUNDATION

Prizewinner Caroline Bissardon at work at the FAME beamline.

ESRF user wins L'Oréal-UNESCO award

A regular ESRF user has won a coveted award that highlights the importance of women in science. Caroline Bissardon, a PhD student at the University of Grenoble–Alpes in France and the University of Swansea in the UK, received the €15,000 L'Oréal–UNESCO Award for Women in Science for her research into the impact of selenium in cartilage.

Selenium is one of the body's essential trace elements, and research has shown that too little can lead to the onset of osteoarthritis. Conversely, too much selenium in the body can be toxic. Bissardon, 25, has spent her PhD trying to understand the biochemical, biological and biomechanical impact of the element in cartilage tissue. Her research is based to a great extent on studies performed at the ID16A beamline and BM30B (FAME)", a French collaborating research group beamline, using techniques such as X-ray Fluorescence Microscopy, X-ray absorption near edge structure (XANES) spectroscopy and Fourier transform infrared (FTIR) microscopy.

"The subject of my thesis was quite unexplored until now", says Bissardon, who is starting a postdoc this winter at the ESRF with the University of Grenoble Alpes. "To gain recognition as prestigious as this means that my work is going in the right direction." She intends to use the prize money to attend conferences and establish international collaborations.

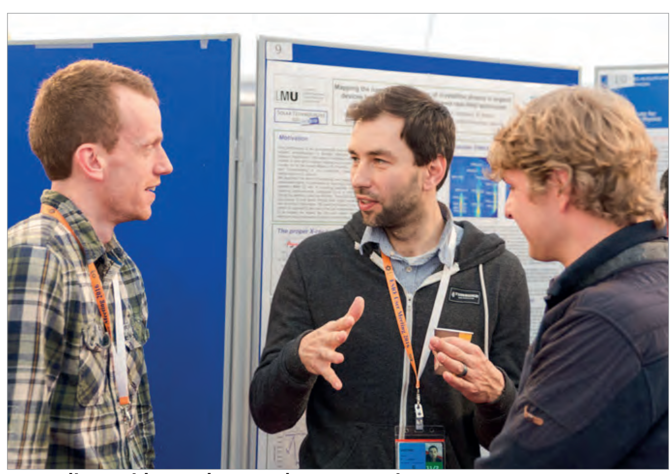
Named after the cosmetics company and the United Nation's cultural agency that founded them, the L'Oréal–UNESCO awards are designed to "promote and highlight the critical importance of ensuring greater participation of women in science". Bissardon's prize was one of 30 awarded to a "generation of young researchers" in France out of more than 1000 PhD and postdoc applicants.

User Meeting coming soon

Save the date and come to the 2017 ESRF User Meeting, which will take place from 6–8 February. The User Organisation Committee (UOC), which plays a central role in the meeting's organisation, says that the event will be a unique opportunity for users to talk with one another, ESRF staff and ESRF management about their science and ideas, for now and the future.

The UOC and the ESRF management have prepared a "rich and diverse" programme including keynote lectures, the Young Scientist Award, user poster clips, a poster session, the Director's report and user-dedicated sessions with tutorials and three microsymposia. The microsymposia, covering themes of resonant inelastic X-ray scattering, quantitative coherent X-ray diffraction imaging and "operando" structural studies in materials science, will give attendees an opportunity to present their research to fellow users and staff, and will explore the new science enabled by Phase I of the Upgrade Programme.

One of the plenary day sessions will be dedicated to the flagship ESRF upgrade, the Extremely Brilliant Source, with presentations on its status and the outcome of its dedicated workshop taking place in December.


C ARGOUD/ESRF

Users discuss ideas at last year's User Meeting.

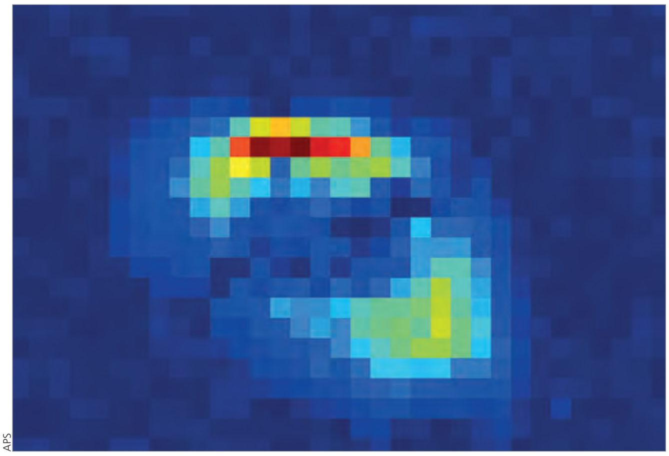
"The User Meeting is the best forum for users to network directly with each other and with ESRF staff about ideas for new science, new possibilities and new proposals," says Joanne McCarthy, Head of the ESRF User Office. She encourages users to promote the event by downloading its poster and sharing it among colleagues.

● More information, including the poster, is available at www.esrf.eu/UM2017.

Scientists see X-ray ghosts

The first demonstration of "ghost imaging" with X-rays has taken place at the ESRF beamline ID19. The new ability could lead to the development of low-dose medical X-ray diagnostics, and diffraction-imaging at X-ray free electron lasers (XFELs).

Ghost imaging is an indirect method of imaging in which most of the radiation never actually interacts with the sample. In its simplest form, it involves sending out two identical speckled beams. The sample transmits or scatters one of the beams to a single-pixel "bucket" detector; meanwhile, the second, reference beam is imaged by a pixel-array detector. The image is retrieved by correlating the bucket signals and the reference images, and repeating the experiment many times, using different yet correlated speckle patterns.


APS
Ghost image (diagonal shadow in centre) of a metal wire

This type of technique has already been used for visible light to improve satellite imagery of Earth, where the speckling results from atmospheric turbulence, but not for X-rays until now. The demonstration, by Daniele Pelliccia of the Royal Melbourne Institute of Technology in Australia and colleagues, suggests applications in medical diagnostics, where low X-ray doses are important, or in single-molecule diffraction at XFELs (DOI: 10.1103/PhysRevLett.117.113902).


S CLAISSE

EPN campus welcomes international students

The joint ESRF–ILL International Student Summer Programme on X-ray and Neutron Science ran from 29 August to 23 September on the EPN campus. The event, which consisted of a four-week experimental project, offered students a practical and theoretical introduction to working on large research instruments, as well as a social programme to discover the local culture and environment.

The 20 successful students were picked from more than 170 candidates and come from 10 different countries: Russia, Norway, Israel, Hungary, the UK, Spain, Italy, Germany, Denmark and South Africa. Mostly in the final stages of undergraduate science degrees, the students chose to spend the last weeks of the summer in Grenoble to gain an insight into working with world-class research technology.


COCKRELL SCHOOL OF ENGINEERING
Electrically darkened material.

Smart window saves energy

An international team of researchers has invented a flexible "smart window" that can control the heat and light transmitted through glass. The amorphous material, which the ESRF helped to characterise, could help to reduce heating bills for homes and businesses.

Some existing window coatings are amorphous metal-oxides, produced at high temperature and with dense, three-dimensionally bonded structures. Now the research team, led by Delia Milliron at the University of Texas at Austin in the US, has managed to create a linearly organised amorphous material made of chemically condensed niobium oxide.

From X-ray scattering at the ESRF beamline ID15B (now ID15A), the scientists were able to show that the atoms formed one-dimensional chains, allowing ions to flow in and out freely.

The material is flexible and electrochromic, meaning that a small electric charge can lighten or darken it, and control the transmission of near-infrared radiation. The team believes that it could be incorporated into windows, sunroofs and even curved glass surfaces (DOI: 10.1038/nmat4734).

Popular conferences

Two conferences organised by the ESRF at the end of summer proved popular. The 2016 International Workshops on Accelerator Alignment (IWAA) saw more than 130 participants gather at the synchrotron to join discussions on new and fast-changing alignment techniques. Meanwhile, the International Soft Matter Conference had some 700 attendees (see p15).

ESRF-10 will house spare accelerator equipment

New buildings take shape

The first of the new EBS buildings was completed in October. Known as ESRF-10, the temporary building will be used to store spare accelerator equipment, freeing up space in the technical zones surrounding the accelerator tunnel for the stockpiling of newly arriving girders. The foundations were laid in September and, in spite of bad weather, the building went up in a matter of days.

Work on the other EBS buildings is in progress. A total of eight new buildings will be constructed in the centre of the ring over the coming months to house the multitude of activities generated by the EBS.

Ground preparation for the main buildings, ESRF-01 and ESRF-02, kicked off in August and continued throughout the autumn.

The 1100 m² surface area of ESRF-01 will be used to assemble the girders for the new machine and, once this is completed, serve as a workshop. ESRF-02A and ESRF-02B, occupying an area of 500 m², will be used to store newly delivered components before and during the assembly phase.

Three temporary buildings, ESRF-11, -12 and -13 will be used as workshops to perform radiation activation measurements on the components removed from the tunnel and storage before final disposal. An extension to another existing building is also planned for November 2018 to provide a new location for the radio-frequency test stand and additional space to store radio-frequency components.

EBS seminar

The next EBS Seminar will be held on Tuesday 6 December at 3.00 p.m. in the ESRF Auditorium. Following on from Michael Borland's talk in November on the status of the Advanced Photon Source upgrade to the accelerator at the Argonne National Laboratory in the US, this month Ferdinand Willeke, Associate Director for Accelerator Systems at Brookhaven National Laboratory in the US, will give a status report on the NSLS II synchrotron.

MAC applauds progress

The fourth Machine Advisory Committee (MAC) meeting was held at the ESRF on 22 and 23 September. The panel of 14 experts listened to 15 presentations from staff on the current status of the EBS project, and provided advice accordingly.

The members were impressed by the progress made since the last MAC meeting in April, including the assembly of a prototype dipole magnet, thermal tests of the prototype girder, and ground preparations for the new EBS buildings. Procurement is at an advanced stage and the first deliveries are already being made on site.

The highlight of the event was a visit to the Chartreuse Hall where the committee was invited to inspect the newly delivered dipole magnets, and posters and a video detailed the complex dipole production process. The members also visited the mock-up of the ring tunnel in which the prototype girder is housed for tests.

"We see no showstoppers at this stage."

"The MAC is very pleased with the progress since the last meeting in April," says MAC chairman Richard Walker. "The project is on track and there haven't been any changes to the major project milestones. The MAC therefore sees no showstoppers at this stage for the successful and timely realisation of the project."

First dipole magnets assembled

The assembly of the first EBS "dipole" magnets has begun, following the successful test of a prototype last month. The dipoles, which are responsible for steering the electrons around the synchrotron, will be assembled entirely in-house from more than 60 tonnes of metal.

"Our team has valuable experience in permanent magnet systems and magnetic measurements, gained over years of building various types of insertion devices," says Joel Chavanne, head of the Insertion Devices and Magnets team. "Assembling the dipoles in-house will give us not only full control over the assembly process, but also a new experience in terms of large, series-based permanent magnet systems and their associated field tuning."

Rather than being just one magnet, each dipole actually consists of some 100 samarium–cobalt permanent magnets, distributed between five modules made of low-carbon steel. The overall assembly has pure iron poles, and a mass of 380 kg. Unlike electromagnets, which are used elsewhere in the synchrotron, the dipoles require neither power nor water cooling. This makes them compact enough to be squeezed into the EBS's tight magnetic lattice.

Over the summer, a magnet assembly area was set up in the ESRF's Chartreuse Hall in preparation for the dipoles' assembly, measurement and calibration. Everything has been designed by in-house experts – from the special assembly tool for inserting the magnets into the modules, to the stretched-wire measurement benches, which test the induced voltage and thus the magnetic field of each magnet.

A prototype dipole, consisting of its support, container blocks and magnets was delivered at the end of August, and assembly began in September. The engineers inserted the magnets into their modules before testing their magnetic fields and mounting them on their support. The whole five-block module was then tested. "All the measurements were within the expected range – with no bad surprises!" says Chamseddine Benabderrahmane, who is in charge of procuring the magnets. The prototype test served to refine the assembly, measurement and calibration process.

The dipoles are the first permanent magnets to be used in a fourth-generation storage ring, thanks to the lower cost of stable permanent magnet materials. However, the construction of large series of dipoles needs to be carefully controlled to reach the required magnetic field quality and so the magnets are being assembled in-house, taking advantage of ESRF expertise.

During September and October, material to construct the dipoles was delivered from as far afield as China, Germany, Italy and the UK. Consisting of more than 13,000 high-performance permanent magnets, 640 magnet modules and 128 dipole supports, the material is expected to be assembled within a year.

SANDRA CARDOT

C ARGOUD

Maxime Paulin (top left) and Chamseddine Benabderrahmane assemble a dipole. The empty dipole blocks arrived in September.

Engineers test girder's warp factor

ESRF engineers are performing tests on girders to see whether power cables can be channeled through them, without warping the metal.

Girders are primarily for supporting magnets, but they take up a lot of space in the tunnel. Ideally, the electric cables used to power the magnets would be channeled through them, but there is a risk that the heat generated in such a confined area will distort the girders and affect the alignment of the magnets above. Alignment tolerances require that movement is restricted to less than 50 μm in any direction, to ensure the accuracy of the beam positioning.

Over the last few months, ESRF engineers have constructed a mock enclosure within the Chartreuse Hall similar to the EBS's storage ring tunnel, to test a girder in normal ventilation conditions. The girder was equipped with 18 temperature sensors and 57 other sensors to detect any geometric changes in the girder. The cables were then powered on with different levels of tunnel ventilation.

The results showed that the temperature rose a maximum of 31°C in the cable bundle. There was no change in the horizontal plane of the girder, but vertically, it deformed into a near-symmetrical banana shape. Even with nominal current in the cables and maximum ventilation, the girder deformed by 35–40 μm, which, the engineers felt, was too large to be acceptable.

Thermal studies are now underway to evaluate a new configuration, in which the cables are not placed inside the girder but on two wide trays to one side, facing the cooling pipes.

Jean-Francois Bouteille, Head of Power Supplies for the EBS, is confident that a solution will be found. "Studies such as these show the vital role of the ventilation of heat sources around the girder," he says. "They will ensure that we choose the most effective solutions for the EBS, and save precious time during the installation phase."

SANDRA CARDOT

Girder tested for warping in the mock tunnel.

News from the User Office

The ESRF received 1154 proposals for the last submission deadline in September – another record for September and the third highest ever. Faster and more efficient experiments are ensuring that more of these proposals are being accepted, maintaining an acceptance rate of more than 40%. The proposals were reviewed during the Beam Time Allocation Panel meetings on 27 and 28 October.

The next deadline for the submission of standard proposals is 1 March 2017. The next deadline for long term proposals – those for completion by the shutdown of December 2018 – is 15 January 2017.

Joanne McCarthy, Head of the User Office

News from the User Organisation Committee

The ESRF User Organisation met in September to finalise the organisation of the ESRF User Meeting, which will take place on 6–8 February 2017 (see p6).

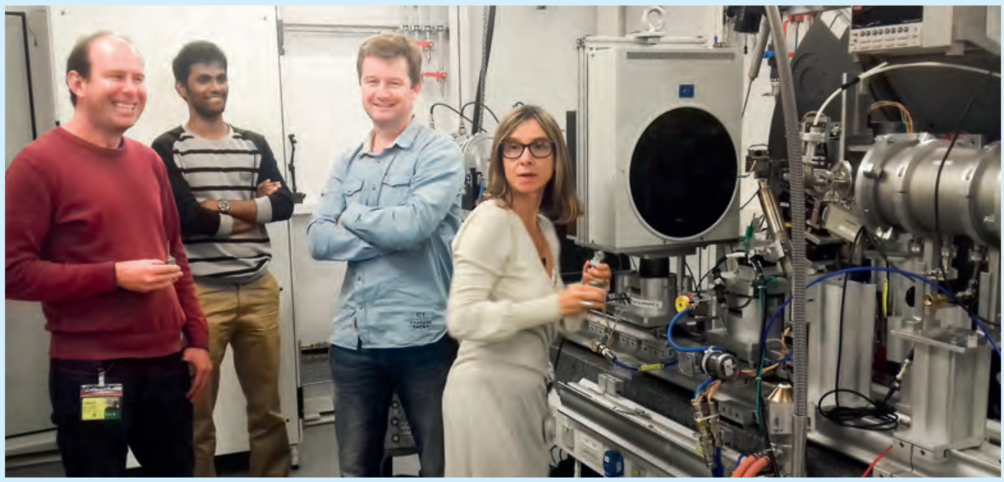
We would like to remind users that the User Organisation provides a direct link between them and the ESRF management. They are therefore welcome to contact us with any questions, comments or ideas. E-mail addresses for user-community representatives can be found at tinyurl.com/ESRFusers.

Finally, we are happy to announce that the User Organisation now has a Twitter account: @ESRF_UserOrg. Follow us and help to create a connected ESRF user network.

Paola Coan, chair of the UOC

News from the beamlines

- The full-field diffraction microscope on **ID01** is open for proposals. It allows imaging under diffraction conditions with a spatial resolution of 200 nm at an X-ray energy of 20 keV, with use of refractive lenses and a high-resolution detector at a distance of 6.5 m that can rotate in the horizontal plane. Typical samples include thin films or nanostructures *in operando* conditions.
- **BM01B** has been transferred from **BM01** (where it was part of a CRG split beamline) to a new bending magnet port on **BM31**. The Swiss-Norwegian Beamlines (SNBL) consortium will continue to operate both beamlines as a joint facility, open to academic and industrial users from Norway and Switzerland for its private time and to all ESRF public and industrial users for its ESRF time. After a brief commissioning period in July 2016, **BM31** is now in full operation. It has been designed specifically for *in situ* experiments, in which powder and EXAFS data can be collected quasi-simultaneously under identical experimental conditions.
- The **LISA-BM08** CRG beamline will carry out the first phase of its refurbishment in the first months of 2017. A new double crystal monochromator will be installed as well as the associated cryo-cooler. Si(111) and Si(311) crystal pairs will be available with an accessible energy range of 4.9–72 keV. Run 1 of 2017 will be dedicated to the installation and commissioning of the instrument, with users foreseen for April in Run 2. The new focusing mirrors will arrive in June and thus the first quarter of 2017 will be run without horizontal focalisation. Completion of the new optics layout is scheduled for Run 5 in 2017.



Users from the Sion campus, Ecole polytechnique fédérale de Lausanne, assisted by SNBL staff at BM01B/BM31.

- **ID11** has commissioned a new end station optimised for sub-micron beamsizes (0.15–0.6 µm) at high energies (30–60 keV). This greatly enhances its capabilities to exploit the smallest focal spot sizes.
- **ID15A** began commissioning in September and was reopened to users in November 2016.
- Since September 2016, Experimental Hutch 1 (EH1) at **ID20** has been available to users for non-standard experiments. Users can choose between a focused beam of size 40 µm (v) × 250 µm (h) and a highly collimated beam of size 0.4 mm × 2 mm with a vertical divergence of less than 10 µrad and a horizontal divergence of approximately 30 µrad. The experiments can use all high-resolution monochromator capabilities of ID20 and polarization conditioning using a double phase plate.
- Following the success of the **ID21** sulphur K-edge XANES database, a similar database composed of more than 30 K-edge XANES reference spectra of phosphorous compounds has been created by **ID21** users, collaborators and staff and is now available online at tinyurl.com/PspecXANES
- From October 2016 to May 2017, **ID23-2** will undergo a major upgrade. This will include the installation of a high-precision vertical axis goniometer with dramatically improved raster scanning speeds and precision, a new sample changer, and the addition of a new beam size of approximately 1.5 × 1.5 μm^2 FWHM. These upgrades should push the boundaries of the minimum crystal size that can be studied on **ID23-2**, and boost the speed and data quality of serial crystallographic experiments.
- New FLEX sample changer robots and high-capacity dewars (HCDs) are being installed on both **ID23-1** and **ID29**. Following the installation of new experiment tables to accommodate the HCDs during the summer shutdown, final installation on **ID23-1** was completed during the October shutdown, and **ID29** will be completed during the winter shutdown. The FLEX-HCD on these beamlines will be able to operate both with Spine and Unipucks for a maximum capacity of 312 sample holders. The **ID23-2** FLEX-HCD will only support Unipucks.
- A diffraction/diffuse scattering side station is now available for users at **ID28**. The new station can operate in parallel with the main inelastic scattering branch, providing high-quality mapping of reciprocal space. Diffuse scattering data contains information about the local environment of defects, short-range correlations, anomalies of lattice dynamics, etc. Tandem work with the inelastic scattering station will be mutually beneficial for both instruments. It uses a wavelength of 0.5–1 Å with a flux up to 10^{12} photons per second and a focal spot of 50 µm. A PILATUS 1M detector is used with a heavy load Euler goniostat, compatible for use with a cryostream, heat blower or diamond anvil cells.

Safety first?

Hair betrays toxic procedure

X-rays trace mercury to dental work carried out a year earlier.

Colette was no stranger to the dentist. Unnerved by the growth of a benign tumour in her mouth, she had opted to have all nine of her amalgam fillings removed, one at a time, in case they were leaking toxic levels of mercury. In May 2014, with the fillings long gone, she allowed Alain Manceau, an environmental scientist at Grenoble Alpes University in France, to cut a sample of her hair and take it to the ESRF. His group's analysis did indeed reveal a peak in mercury, consistent with that from dental amalgam. Strikingly, it could also precisely correlate the distance of that peak along the hair strand to the removal of Colette's last filling, 478 days earlier.

As it turned out, the peak indicated a mercury level of just 0.74 parts per million (ppm), far below anything considered dangerous. For that very reason, however, Manceau and colleagues' study sets a new benchmark for the techniques known as X-ray fluorescence (XRF) and X-ray absorption near-edge structure (XANES) spectroscopy. It demonstrates that it is possible to uncover the bonding environment of a metal at extremely low concentration, in an almost vanishingly small sample. The ability could pave the way for studies of how toxins like mercury are processed by organisms at a cellular level. "Our study is so advanced because of the ESRF," says Manceau. "We couldn't have done it at any other synchrotron facility."

Health risk

Mercury itself is a toxin high on the international agenda. Although concerns about its presence in amalgam fillings are generally deemed unwarranted, there is deserved unease about the metal leaking to the environment from gold mining and coal-fired power plants in particular. The World Health Organization believes that up to 17 in 1000 children living in traditional coastal villages suffer mercury-induced brain disorders, having eaten fish contaminated by industrial waste.

Regular chemical analysis can detect the presence of mercury in the body, and give an indication as to its source. The organically bonded mercury typical of seafood and drugs is thought to end up in the hair, while the inorganic and elemental mercury typical of electronics and amalgam fillings is thought to end up in the blood and urine. The detection of mercury in, say, the hair but not the urine might point to the ingestion of contaminated fish, but this would not be certain. Moreover, routine analysis requires a sample with a mass of at least several milligrams, which precludes higher resolution studies.

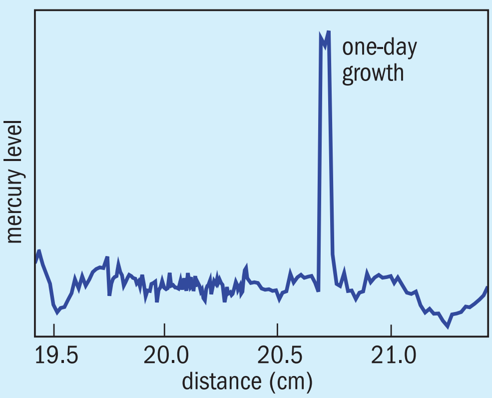
For their study, Manceau and colleagues came to the ESRF with hair samples from seven volunteers. The researchers performed nano-XRF at the ESRF beamline ID16B to map the elemental composition along the hair strands, and XANES spectroscopy with high spectral resolution at ID26 to address the chemical state of any detected mercury. Neither technique had been performed before for such low concentrations (down to 0.5 ppm) of an atomic species at such high resolution (the width of a human hair). "Based on our estimates, we thought we could do it," says Pieter Glatzel, who is in charge of ID26. "And it worked out."

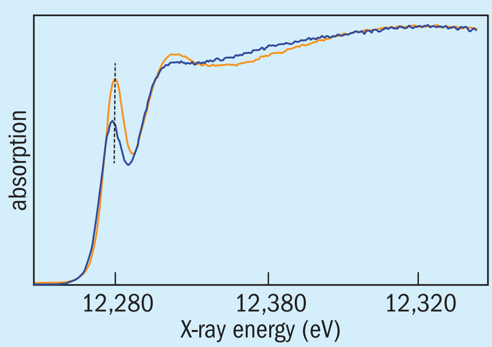
It was the nano-XRF that, in the case of Colette, allowed Manceau and colleagues to correlate a peak in mercury level some 20 cm along the hair's length with a dentist's removal of a filling more than a year earlier (top figure). A XANES spectrum (bottom figure) confirmed that the mercury was inorganically bonded, as it would be from an amalgam source – putting an end to the view that such mercury can only be detected in the blood and urine. Tests on samples from two residents of Pará, Brazil, who regularly ate fish from contaminated rivers, demonstrated that XANES could also detect organic mercury, specifically that bonded to methyl groups (DOI: 10.1021/acs.est.6b03468).

The researchers' method is unlikely to be used in the future to trace incidents of mercury exposure, except perhaps in high-profile forensic cases. But Manceau believes that it could "blaze a trail" for the study of mercury and other toxic metals at low concentrations at very small scales. Indeed, he already has ideas to study the way mercury is processed within rice grains and fish brain cells, which could help in the understanding of how toxins are amplified up the food chain. "It is fascinating, and totally new," he says.

Jon Cartwright

"We couldn't have done it at any other synchrotron."

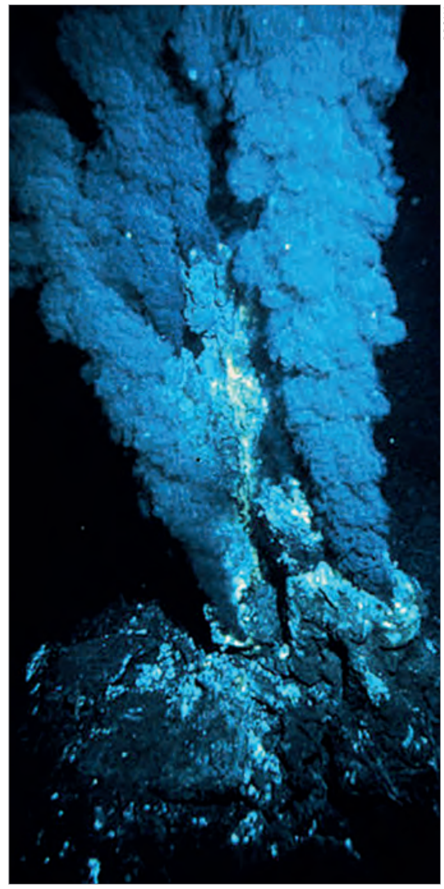




Top: Nano-X-ray fluorescence locates a peak in mercury concentration 20.7 cm along the hair strand, equivalent to 478 days' growth. Bottom: XANES spectra distinguish between inorganic mercury from dental amalgam (blue) and organic, methyl-mercury from fish (orange).

Secret from the abyss

A recent ESRF study of an organism that lives in hydrothermal vents helps trace our evolutionary past.



Microbes living in underwater vents reveal a possible link to the primordial 'RNA world'.

DNA makes RNA makes protein: crudely put, this is life how we know it. The DNA holds the genetic information, while the RNA takes a copy of that information so that a protein can be created from it. Proteins are the working agents of all living things, catalysing reactions that are essential for an organism's survival – including the formation of DNA and RNA.

It wasn't always thus. Most biologists agree that, in the earliest stages of evolution, DNA and proteins were absent, and life consisted solely of RNA. In this "RNA world", all genetic information was contained in RNA molecules, which could fold into catalysing shapes that forced other RNA molecules to duplicate themselves. At some point, nature found that organisms could become more complex if the catalysing role was handed over to proteins and, ultimately, the genetic role was partially handed over to DNA.

The RNA world is an attractive hypothesis, not least because it answers the question of whether it was the proteins or the nucleic acids – DNA and RNA – that came first. Nonetheless, smaller chicken-and-egg questions remain. In today's DNA, RNA and protein world, for example, two quintessential processes – the synthesis of new DNA, and the transcription of DNA onto RNA – involve a protein, specifically a polymerase, latching onto a strand of DNA and copying its information. For synthesis, this polymerase comes in a special type of architecture known as a "Klenow" fold, while for transcription, it comes in a "double-psi beta-barrel" (DPBB) fold. One would assume evolution started off with just one polymerase architecture. So what came first?

The question is one that has bothered Marc Delarue of the Pasteur Institute in Paris, France, and colleagues. By studying tiny organisms known as archaea with the ESRF, however, they now believe they have found an answer.

Archaea are a group of life midway between bacteria and eukaryotes – the latter representing plants, animals, fungi and many other organisms. Often living in extreme environments, such as deep hydrothermal vents, they are thought to have evolved little over the ages, and therefore offer a window into what life might have been like, billions of years ago. They also host a class of DNA polymerase known as PolD, the only class for which the structure is still unknown.

This year, Delarue and colleagues managed to crystallise parts of PolD from the archaeon *Pyrococcus abyssi*, and took their samples to the ESRF beamline ID23, where they performed macromolecular crystallography X-ray diffraction to obtain the crystal structure. "They have some of the best beamlines in Europe," says Delarue. Remarkably, the catalytic core of PolD was unlike other known DNA polymerases. Instead, it closely resembled the DPBB fold seen in RNA polymerase. The similarity suggests that both DNA and RNA polymerase evolved from a common ancestor – one that was shaped much like the RNA polymerase seen in life today (DOI: 10.1038/ncomms12227).

The work will help to paint a finer picture of the transition from the RNA to DNA world, and it might help in the understanding of later evolution, too. The researchers are now planning to examine the structure of the entire archaean PolD complex, which they believe shares some building blocks with DNA polymerase from eukaryotes, a more advanced form of life. "It looks like evolution was very opportunistic and made use of existing 'units' and merely combined them in many different combinations, until one of them worked better than the others," says Delarue. "It would be interesting to learn how these common bricks are arranged."

Jon Cartwright

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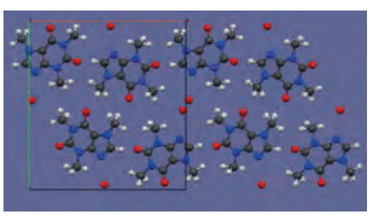
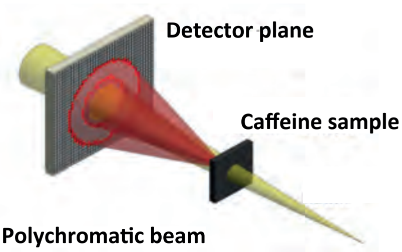
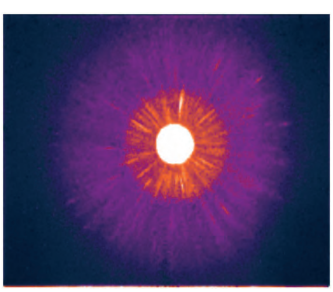
X-ray Diffraction of the crystal structure of caffeine using PIXIRAD and a white X-ray beam

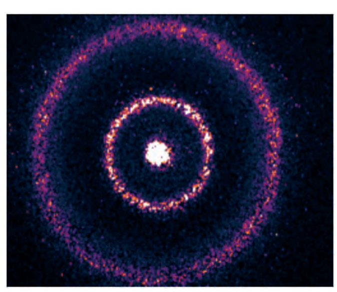


Caffeine structure

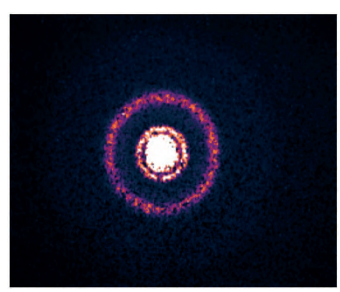




Caffeine diffraction image when using the full spectrum of the X-ray beam



The two caffeine Debye-Scherrer rings for a window energy selection of 9-10 keV



The same rings for a window energy selection of 21-22 keV

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Into the hub of soft matter

SERGE CLAISSE/ILL

Many developments to discusss

The International Soft Matter Conference held in Grenoble in September shone a light on a thriving field.

There are many international conferences focused on various areas of soft matter, but when it comes to the field as a whole, the International Soft Matter Conference (ISMC) has become one of the standard-bearers. This year saw its fourth edition, the ISMC2016, which was held at the Alpexpo convention centre in Grenoble, France, 12–16 September. Co-organised by the ILL, the ESRF and local institutions, under the auspices of the SoftComp consortium, the conference brought together some 700 scientists, including more than 200 students, from over 30 countries.

Soft matter is a highly interdisciplinary field. It comes as no surprise, therefore, that the ISMC's scientific programme covered a broad range of fundamental and applied aspects of soft matter and complex systems, including conventional areas such as colloids and polymers, soft matter technology, microfluidics, dynamical processes, soft glasses and cellular biophysics. From over 800 submitted abstracts, we had more than 150 talks and nearly 500 posters – including the plenary lectures, keynote presentations, lunchtime sessions and award ceremonies.

With such a large number of excellent contributions, it is nearly impossible to pick out highlights. Among the central themes, DNA-guided self-assembly featured in many presentations. Nature has optimised complex self-assembly processes over millions of years of evolution– now soft matter scientists are striving to do the same, with fewer ingredients and, of course, much less time. As highlighted in the conference's opening plenary lecture by Jasna Brujic of New York University in the US, DNA linkers can control interactions between particles, and drive the self-assembly process along predictable pathways. The following plenary lecture, by Francesco Sciortino of the University of Rome in Italy, illustrated how one can exploit the specificity of the interactions between complementary DNA base pairs to build a wide range of nanostructures that replicate computer models, with exciting new physics and potentially novel applications. Indeed, pathways of self-assembly of amphiphilic molecules such as surfactants, block-copolymers, and lipids have been an active area of research, and now the interest is shifting towards biological and bio-inspired molecules such as peptides, synthetic peptoids and so on (see p16).

Active research

Another common theme was active matter – self-propelling particles that mimic the collective patterns of motion seen in the swarming and flocking of bacteria, insects, birds and other creatures. The particles self-propel with use of a chemical motor, similar to the chemotaxis employed by microorganisms. These are nonequilibrium systems that break time-reversal symmetry. In the closing plenary lecture, Mike Cates of the University of Cambridge in the UK illustrated the challenges involved in building a complete statistical-mechanics model of these systems, even defining proper intensive thermodynamic variables. The ultimate goal of active matter research is to reach a comprehensive understanding of the complex spatio-temporal behaviour exhibited by living organisms.

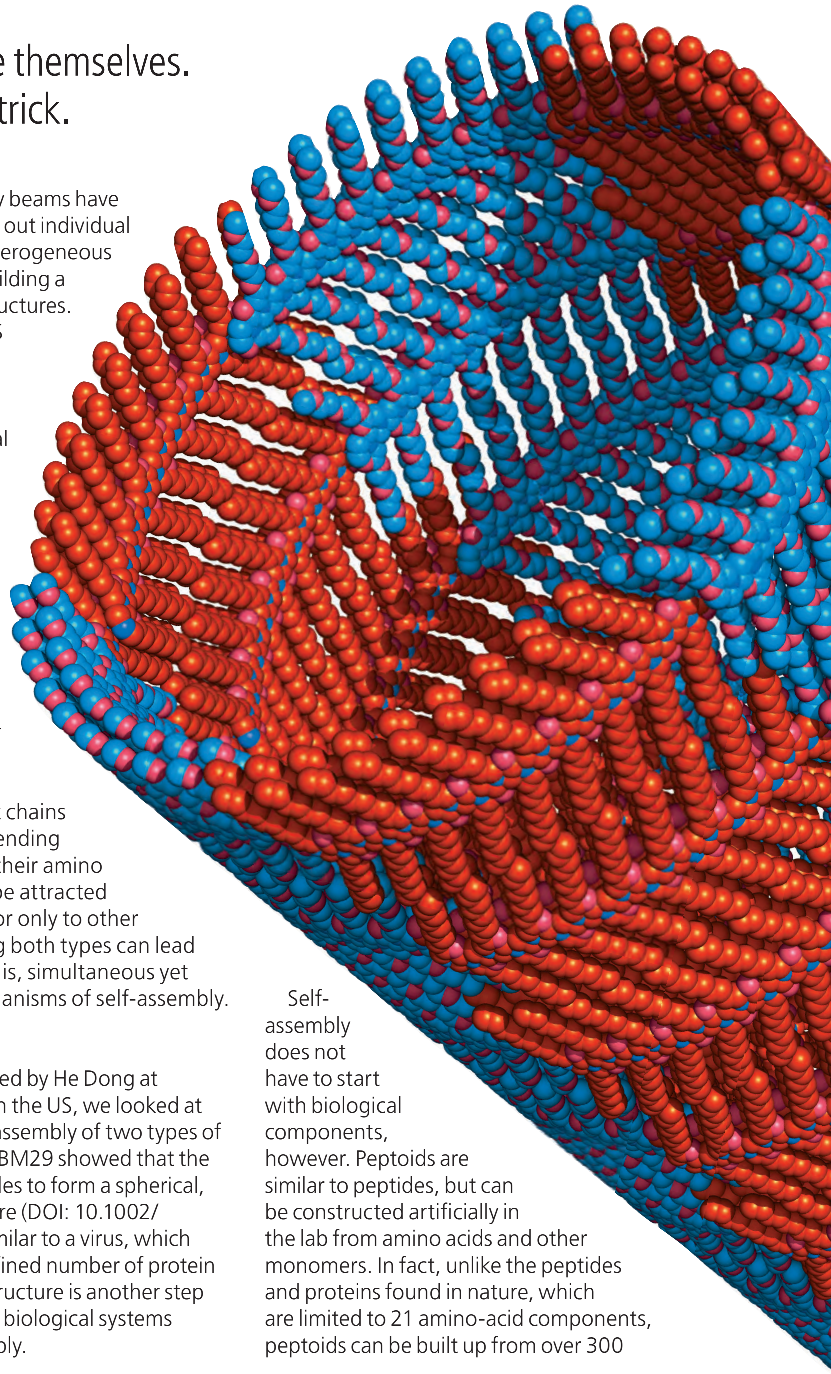
The other plenary speakers were the SoftComp founder Dieter Richter of the Jülich Research Centre in Germany; Matthias Ballauff of the Helmholtz Centre Berlin in Germany; Igor Muševičof the University of Ljubljana in Slovenia; Regine von Klitzing of the Technical University Berlin in Germany, who won the Pierre-Gilles de Gennes prize, sponsored by the *European Physical Journal E*; Joao Cabral of Imperial College London in the UK; and the Soft Matter Lectureship awardee Damien Baigl of the École Normale Supérieure in Paris, France.

As expressed by many participants, the conference overall was a great success in terms of the quality of presentations, the technical organisation and the scientific exchanges. It made a great showcase of the ESRF and ILL, though of course it would not have been possible without the active involvement of colleagues from Grenoble–Alpes University, the French National Centre for Scientific Research, and the French Alternative Energies and Atomic Energy Commission, as well as sponsorship by various other institutions and private companies including the Nanoscience Foundation in Grenoble, the Grenoble Alpes Métropole and the International Union of Crystallography. We look forward to the next ISMC in 2019; the venue will be announced by the SoftComp consortium soon.

Theyencheri Narayanan, ESRF, and Peter Lindner, ILL

The natural route

In nature, complex nanostructures assemble themselves. The ESRF is helping scientists to master this trick.



The creation of a protein is a slick operation. From a score of possible amino-acid building blocks, it self-assembles via a neat interplay of hydrogen bonds, electrostatic attractions and water-repelling "hydrophobic" interactions. The resultant structure is so distinctive – and so specific to the protein's function – that a mutation in just one of the amino acids is enough to cause serious diseases, from sickle-cell anaemia to cancer.

Scientists have long aspired to this kind of complexity in the lab. In an attempt to mimic nature, they have managed to induce self-assembly among simple synthetic materials, such as polymers, colloids and surfactants. Granted, the structures produced have had distinct overall shapes – spheres, cylinders and so on. But a closer look at these structures has too often exposed the rudimentary character of the self-assembly, with the component parts haphazardly distributed and the function ill-defined. When it comes to nanostructures, then, there has been a large gap between what is achieved in nature, and what is achieved in the lab.

That gap could soon be closed. Our group is one of several that is developing self-assembly to the point where it can create nanostructures of truly protein-like complexity, opening up new avenues in nanotechnology and biomedicine. If we can master the process, it might be possible to create nanostructures with highly specialised functions – targeting diseased cells, boosting or hindering bodily reactions, regenerating tissue, or simply creating new materials for niche products.

Synchrotron science has been key to understanding self-assembly, and the ESRF has long played a unique role. At the beamline ID02, a combination of wide, small and ultra-small angle X-ray scattering (WAXS/SAXS/USAXS) has given a unique "bottom up" view of structural organisation, even in real time (see "Caught in the act", below). At ID13, micro and nano X-ray beams have allowed users to pick out individual domains within a heterogeneous sample, gradually building a map of local nanostructures. Meanwhile, BioSAXS at BM29 has allowed the straightforward observation of small amounts of biological macromolecules in solution, in their natural state.

One way to manage nature-inspired self-assembly of nanostructures is to start off with biological building blocks – in particular peptides, which are protein fragments containing just short chains of amino acids. Depending on the chemistry of their amino acids, peptides can be attracted only to themselves, or only to other peptides. Combining both types can lead to orthogonal – that is, simultaneous yet independent – mechanisms of self-assembly.

Going viral

This year, in a group led by He Dong at Clarkson University in the US, we looked at the orthogonal self-assembly of two types of peptide. BioSAXS at BM29 showed that the process led 12 peptides to form a spherical, star-like nanostructure (DOI: 10.1002/smll.201600910). Similar to a virus, which consists of a well-defined number of protein subunits, the nanostructure is another step towards engineering biological systems predictably and reliably.

Self-assembly does not have to start with biological components, however. Peptoids are similar to peptides, but can be constructed artificially in the lab from amino acids and other monomers. In fact, unlike the peptides and proteins found in nature, which are limited to 21 amino-acid components, peptoids can be built up from over 300

Caught in the act

The high brilliance of X-rays at the ESRF makes it possible to shoot pulses just a few milliseconds in length and still obtain structural information. This has allowed the beamline ID02 to "film" self-assembly in real time. For the past few years, Stéphane Bressanelli of the CNRS Laboratory of Molecular and Structural Virology in Gif-sur-Yvette, France, and colleagues have been using ID02 to watch proteins self-assemble around RNA to form virus particles, potentially helping us understand how viruses propagate (DOI: 10.1021/ja403550f). Similarly, in 2015 Frank Schreiber of the University of Tübingen in Germany and colleagues watched the crystallization of a protein, which could help in the understanding of diseases, such as Alzheimer's (DOI: 10.1021/ja510533x).

Filming self-assembly could have applications outside of biology, too. In recent years, together Jan Skov Pedersen's group at Aarhus University in Denmark and Theyencheri Narayanan, scientist in charge of ID02, have used this ability to watch surfactant micelles self-assemble and change shape – results that could help to develop consumer detergents (DOI: 10.1021/acs.jpclett.6b00767).

to complexity

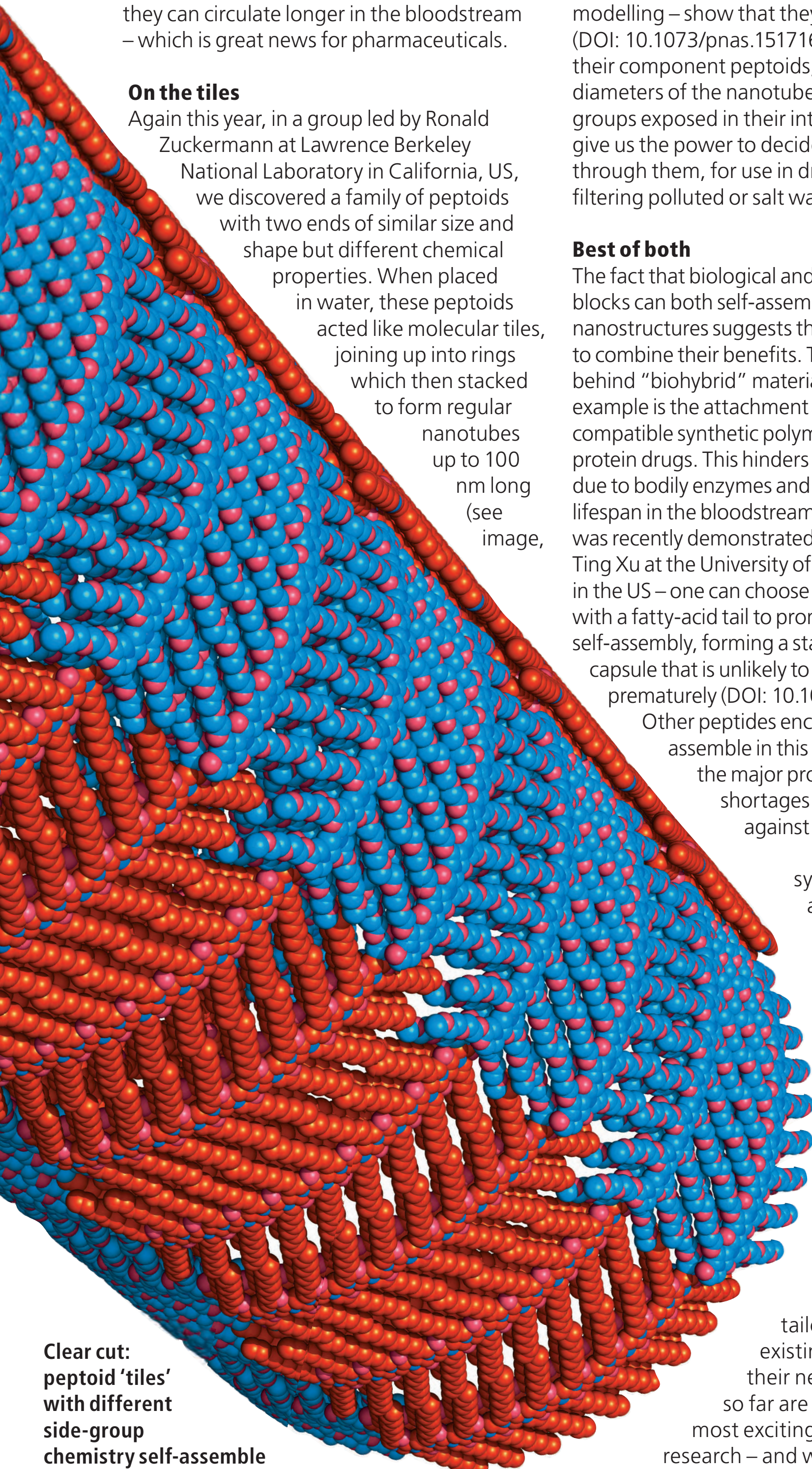
different building blocks – meaning there is almost no limit to the nanostructures that can be created. Peptoids are also insensitive to the enzymes present in the body. This suggests they can circulate longer in the bloodstream – which is great news for pharmaceuticals.

On the tiles
Again this year, in a group led by Ronald Zuckermann at Lawrence Berkeley National Laboratory in California, US, we discovered a family of peptoids with two ends of similar size and shape but different chemical properties. When placed in water, these peptoids acted like molecular tiles, joining up into rings which then stacked to form regular nanotubes up to 100 nm long (see image, below). BioSAXS at the BM29 beamline was again particularly useful, as it enabled us to accurately determine the dimensions of the nanotubes, and – with help from computer modelling – show that they were hollow (DOI: 10.1073/pnas.1517169113). By altering their component peptoids, we can vary the diameters of the nanotubes and the chemical groups exposed in their interiors. That could give us the power to decide what passes through them, for use in drug delivery, and for filtering polluted or salt water.

Best of both
The fact that biological and synthetic building blocks can both self-assemble into novel nanostructures suggests there may be a way to combine their benefits. This is the thinking behind "biohybrid" materials. A simple example is the attachment of a biologically compatible synthetic polymer to peptide or protein drugs. This hinders the degradation due to bodily enzymes and prolongs the drug's lifespan in the bloodstream. Alternatively – as was recently demonstrated by a group led by Ting Xu at the University of California, Berkeley, in the US – one can choose a synthetic polymer with a fatty-acid tail to promote compact self-assembly, forming a stable nano-sized capsule that is unlikely to discharge a drug prematurely (DOI: 10.1021/bm5005788). Other peptides encouraged to self-assemble in this way could tackle the major problem of antibiotic shortages (see "The fight against resistance", right).

Self-assembly, synchrotron science, and biological and synthetic building blocks: together these comprise a fantastic toolbox to create new functional nanomaterials. The breadth of combinations means that scientists will be able to build up entirely new nanostructures from scratch, or tailor those already existing to better suit their needs. The results so far are some of the most exciting in soft matter research – and we are still only at the beginning.

Reidar Lund, University of Oslo, Norway.



Clear cut: peptoid 'tiles' with different side-group chemistry self-assemble into regular nanotubes. Credit: Ronald Zuckermann/LBL.

Self-assembling against resistance

Antimicrobial resistance is the tendency of bacteria and other microbes to adapt during exposure to antibiotics and other antimicrobial drugs, and is a serious global health problem. Nearly half a million people are thought to develop drug-resistant tuberculosis every year, and resistance is thought to be affecting the fight against HIV and malaria, too. But there is one class of antimicrobials that is much less susceptible to resistance than conventional medicines: antimicrobial peptides (AMPs). These already exist throughout nature, in humans, animals and even bacteria themselves, and kill a broad class of pathogenic bacteria by disrupting their cell membranes.

The problem with AMPs is twofold: they can be toxic to non-pathogenic cells, and, like other peptides, they suffer from an instability towards enzymes that can lead them to break apart before they are able to kill any harmful bacteria at all. In recent studies at the ESRF's BM29 beamline led by He Dong at Clarkson University in Potsdam, US, however, we have found that the toxicity and instability issues can be significantly reduced by designing AMPs that self-assemble into nanostructures (DOI: 10.1039/C4CC08808E); the effect is even better if the peptides are conjugated with polyethylene glycol (DOI: 10.1039/C5RA24553B). Importantly, the conjugation also appears to dramatically reduce the destruction or "haemolysis" of red blood cells, thus significantly decreasing the AMPs' toxicity.

Tuberculosis: getting harder to beat.

Positioning loads accurately to the micrometer: hexapod at PETRA III

Since 2010, PETRA III (Fig. 1) is the most brilliant storage-ring-based X-ray light source in the world and it provides international scientists with excellent experimentation facilities. In particular, this benefits researchers investigating very small samples or those requiring tightly collimated and very short-wavelength X-rays for their experiments. The high-energy radiation of up to and above 100,000 electron volts with high light intensity offers versatile capabilities, for example in the broad field of materials research for the inspection of welded seams, or for the examination of fatigue symptoms in workpieces. In some cases, this involves accurately positioning really heavy loads down to the micrometer. At the heart of the P07 beamline, which delivers the high-energy X-ray radiation required for materials research, is therefore a heavy-duty Hexapod. Thanks to its accuracy, it facilitates in-situ measurements of material properties under realistic process conditions.

Fig 1: *PETRA III offers scientists from around the world superlative experimentation facilities. In particular, this benefits researchers investigating very small samples or those requiring tightly collimated and very short-wavelength X-rays for their experiments, e.g. in the field of materials research (image: DESY/Reimo Schaaf)*

Hexapods are parallel kinematic positioning systems (Fig. 2), available in many versions with travel ranges of up to a few hundred millimeters. With precision below a micrometer, they can position loads weighing from a few kilograms to a few hundred kilograms, or even several tons. Their advantages compared with serial, i.e. stacked systems, are that they have much better path accuracy, repeatability and flatness. In addition, the moved mass is lower, enabling better dynamic performance, which is the same for all motion axes. Depending on the geometry of the Hexapod, rotations from a few degrees up to 60° and translations of a few millimeters to several centimeters are possible.

Short-wave X-ray radiation for materials research

The PETRA III short-wave X-ray radiation penetrates very deeply into the material and thus is also capable of passing through material of greater thickness. This enables welded seams to be inspected and fatigue symptoms in workpieces to be measured as an aid to quantifying the anticipated durability and service lives, or to analyze new metal alloys. Here effects can be proven down to the level of domain or crystal structures.

Fig 2: *Basic design: In parallel-kinematic systems, all actuators act directly on the same platform. (image: PI)*

The opportunities that proceed from this in respect of materials research are leveraged by Helmholtz-Zentrum Geesthacht (HZG) at the High Energy Materials Science Beamline (HEMS), P07, for example when conducting in-situ measurements of the material properties that occur during reshaping processes such as welding, pressing, rolling or stamping. The application of a mechanical load causes tensile and elongation stresses to occur inside the material. The investigation involving X-ray radiation then indicates the chronological sequence of effects occurring within a material at a crystalline level in micrometer-sized domain areas.

Powerful positioning system for the experimentation chamber

At the heart of the described experimentation chamber is a Hexapod, developed by Physik Instrumente (PI). Dr. Norbert Schell, the scientist in charge of the HEMS Beamline, illustrates the context: "For an increasingly large range of in-situ investigations of 'real' processes, that is processes that actually occur in industry (but of course not only there) – associated with the cutting of workpieces, the coating of surfaces for the hardening or improvement of tribological properties, reshaping, welding, heat treatment as well as combinations of these techniques – it is our ultra-rigid Hexapod with its tremendous load-bearing capability and micrometer precision positioning that makes it possible for the first time for us to conduct scientifically rigorous examinations of the structural changes that occur at an atomic level. This is highly interesting, and is also important in helping us to understand the processes that are occurring and that, ultimately, enable customized materials to be optimized."

The parallel-kinematic custom model, the M-850K (Fig. 3), delivers micrometer-precision positioning for loads of up to one ton in every orientation. It stands ±20° approx. 700 mm high and has a diameter of 800 mm (top platform with large aperture) and 900 mm (bottom). The lower platform is installed on a 360° rotation table and the cabling was designed to be dragchain compatible. With its high load capacity of up to one ton, the Hexapod can carry the entire measurement setup including the equipment where mechanical forces are applied. The Hexapod positions even these large masses over distances of 400 mm to a precision of ±1 micron, and performs rotational motions of ±20° with a resolution to 0.5 µrad. Inside the experimental hutch, this enables complete engine blocks, turbine components, sinter furnaces and cryogenic chambers as well as welding fixtures or other machining units to be aligned precisely for the planned investigations and to be moved accordingly during the analysis.

Fig 3: *Hexapod in the experimentation station EH3 on the P07 beam guidance unit on PETRA III: With its high load capacity, it can carry the entire measuring setup including the structure where mechanical forces are applied – in this figure a chamber for the laser-welding of titanium aluminides. (image: PI / HZG)*

PI (Physik Instrumente) in brief

In the past four decades, PI (Physik Instrumente) with headquarters in Karlsruhe, Germany has become the leading manufacturer of nanopositioning systems with accuracies in the nanometer range. With four company sites in Germany and ten sales and service offices abroad, the privately managed company operates globally. Over 700 highly qualified employees around the world enable the PI Group to meet almost any requirement in the field of innovative precision positioning technology. All key technologies are developed in-house. This allows the company to control every step of the process, from design right down to shipment: precision mechanics and electronics as well as position sensors.

Author: Dipl.-Phys. Birgit Schulze, Marketing & Products at PI (Physik Instrumente)

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Shear success for opal films

Structural colour developed at the ESRF may soon be found on everything from banknotes to clothing.

The colouring of natural opal (pictured) can now be achieved by shearing a film of plastic beads.

The ancient Greeks believed they sprung from the tears wept by Zeus after he defeated the Titans. Science suggests that they grew over millions of years, as rainwater gradually washed silica into the crevices of underground rocks. Whatever the process, natural opal doesn't come about easily.

People desire them all the same, of course, and largely because of their deep ethereal colouring. The appearance is an example of structural colour, which results from the interference of light as it scatters off different aspects of the gemstone's microscopic composition. Structural colouring can also be found in peacock feathers, scarab beetles and various butterflies – but rarely in manmade objects, which mostly rely on dyes to absorb different wavelengths of light. Various industries would like to get their hands on structural colour, partly because of its unique appearance, and partly because it would avoid the pollution associated with making synthetic dyes.

Their wish may soon be granted, now that Jeremy Baumberg at the University of Cambridge in the UK and colleagues have developed a new fabrication technique that promises to make the manufacture of structural colour an industrial process. Simply by shearing plastic beads between a pair of rollers, the researchers claim to be able to make kilometres of opalescent film at a time. If their hopes are realised, the other worldly sheen may soon be sported on everything from car tyres and bank notes to medical bandages and clothing. "It's completely different to what anyone's done before," says Baumberg.

Chemists have been able to make opals artificially in the past, by allowing microscopic silica beads to settle in acidified water. But the process takes several months, and speeding it up is tricky because the beads all too easily "crash out" of the nascent structure, forming a disordered lump at the bottom of the vessel. Baumberg and colleagues wanted to find a way to quickly encourage them into the ordered state from which iridescence arises.

Just under a decade ago, they had the idea of ditching the solvent altogether. Instead, they coated microscopic plastic beads with a softer, stickier polymer, the consistency of chewing gum. At first they tried squeezing the beads between heavy plates, then they tried extruding them from a nozzle – but they could not get the ordering to last. Finally, they tried squashing the particles into a film, before subjecting them to an oscillating shear between several rollers. The particles neatly slipped into a permanently ordered state.

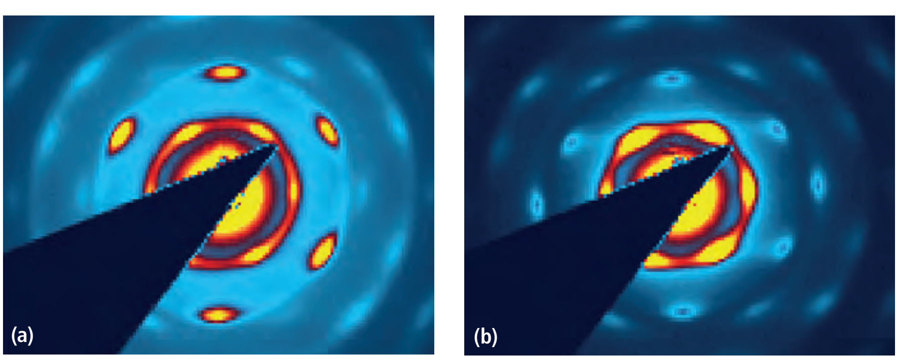
The researchers wanted to hone the technique to produce the best opalescence possible – hence coming to the ESRF. Using small angle X-ray scattering (SAXS) at the BM26B beamline to observe the long-range order between the beads (see figure below), they tried shearing the beads in the same direction, and then in two directions, while oscillating the rollers. The SAXS data showed that shearing in two directions gave the best result (DOI: 10.1038/ncomms11661). "The samples were fantastic," says Baumberg.

Easily tailored

The structural colour of the films depends on the size of the plastic beads and their spacing, so can easily be tailored from the infrared to the ultraviolet. But the films are elastic too, and since stretching them makes the beads separate, the colour changes as a result. That suggests a raft of applications, from the aesthetic to the practical: clothes that change colour when worn; car tyres that turn green when inflated to the right pressure; bras and bandages that indicate when they are correctly fitted; even banknotes that display an anti-counterfeit pattern when stretched.

The researchers have spun out a company, Phomera Technologies, which is now scaling up the fabrication process in China, and claim to already have 150 companies interested, including Nike, Mars and Unilever. If progress continues apace, the opalescent sheen may go from being a jewel of the natural world to a commonplace colouring of many manmade objects.

Jon Cartwright



Left: SAXS data show that order in the opalescent films is greater when they are sheared in two directions (a) rather than one (b).

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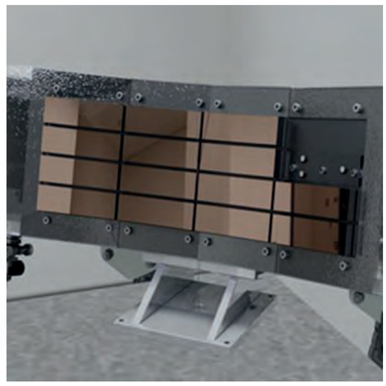

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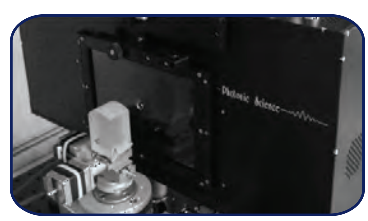

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Soft at heart?

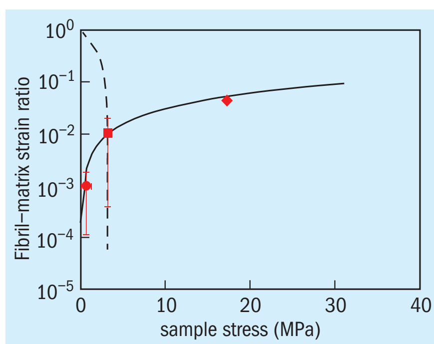
Slugging it out

An ESRF study ends the debate over how sea cucumbers turn soft. Drugs for people who suffer from skin hardening could follow.

Most creatures can be scared stiff, but the sea cucumber is scared soft. Alarmed by a predator, the slug-like organism softens the collagen in its body wall, forms a hole and expels its guts. The predator is distracted – leaving the sea cucumber to slither to safety, where it gradually grows some new innards.

Scientists have long been fascinated by this phenomenon, and not just because of its grisly nature. In most animals the stiffness of collagen hardly changes, but sea cucumbers, starfish, sea urchins and other echinoderms have the peculiar ability to turn it soft or stiff at will. The special variant is called mutable collagenous tissue (MCT), and it is key to the creatures' survival. While softening allows it to eviscerate itself, the sea cucumber will also stiffen its MCT to lock itself inside rocky crevices, out of harm's way. "That change in stiffness gives it a lot of flexibility, in terms of where it can squeeze itself," says biophysicist Himadri Gupta of Queen Mary University of London (QMUL) in the UK.

MCT consists of two components, and there has been a debate as to which provides the softening ability. Some scientists believe it arises from the special makeup of the collagen fibrils, which give the MCT its texture, while others believe that it arises from the protein-based matrix, which binds the fibrils together. Now, an ESRF study by Gupta, together with his PhD student Jingyi Mo, neurobiologist Maurice Elphick and others, demonstrates that it is indeed the latter. The result could resonate way beyond the depths of the ocean, as the tough nature of collagen is implicated in certain human health problems, such as the skin-hardening disease scleroderma.



Strain derived from SAXD patterns (red dots) match a prediction for matrix (solid line) rather than fibril (dashed line) softening.

> **"Human collagens could be made soft, too."**

Gupta's group brought a sea cucumber to the ESRF beamline ID02, where they could perform small-angle X-ray diffraction (SAXD). They isolated samples of the middle layer of the skin, which is composed entirely of MCT, and dipped them into one of three vials: one containing potassium-rich sea water, which naturally makes MCT stiffen; one containing calcium-rich seawater, which naturally makes MCT soften; and one containing regular sea water, which has no effect. They placed individually treated samples into a purpose-built micro-mechanical chamber, which could apply a tensile stress to them while in the beamline.

Using a computer model, the researchers predicted two stress–strain relationships for the samples, depending on whether the softening in MCT arises from the fibrils or the matrix. They then measured the actual strain in each component as a function of applied stress, by measuring the shift in SAXD peaks generated by the fibrils' periodic banding (see figure). The data clearly matched the relationship for softening in the matrix, rather than in the fibrils (DOI: 10.1073/pnas.1609341113). "We have conclusively proved it," says Mo.

Although the researchers cannot yet say with certainty how the matrix changes the MCT's mechanical properties, there is past evidence that it depends upon cells secreting certain proteins or peptides that either cause stiffening or softening. Indeed, previous work by Elphick has identified a softening peptide (DOI: 10.1371/journal.pone.0044492), and he speculates that a human variant of it could provide a therapeutic basis for those suffering from scleroderma. "Human collagens could be made soft, too, inspired by how MCT works in echinoderms," he says.

Jon Cartwright

A soft spot for the ESRF

Many companies approaching the synchrotron have one type of science in mind.

What do washing liquid, margarine and diesel fuel all have in common? Not much to the average consumer – but for scientists, they are all examples of soft matter.

In fact, a high proportion of manufactured products are one form of soft matter or another. Whether undisputedly squishy, like processed food, or at the harder end of the spectrum, like plastic composites, these types of products all have characteristic properties lying somewhere between the classic solid and liquid states – which, scientifically at least, makes them soft. According to Michael Sztucki, the ESRF's industry liaison for soft matter, roughly one in five of the companies approaching the ESRF have soft-matter studies in mind.

Fortunate, then, that the ESRF is well geared up for this kind of work. While most industry scientists may head straight to ID02 to perform ultra small-, small- and wide-angle X-ray scattering (USAXS/SAXS/WAXS), or to ID13 to perform micro- and nano-diffraction, several other beamlines also come in handy – ID19 for micro computed-tomography and ID16A for nano computed-tomography, for instance. "Having a wide range of beamlines and, critically, sample environments to watch soft matter under real conditions, opens the door for industrial research and innovation," says Sztucki. "Our facilities are pretty much unmatched for soft-matter experiments."

Fuelling innovation

One of the latest industrial soft-matter collaborations with the ESRF has been on the subject of diesel fuel with the oil company Total in collaboration with C2P2, a joint laboratory of the CNRS, CPE Lyon and the Claude Bernard University Lyon 1 in France. Diesel consists of various naphthenes, aromatics and paraffins, the last of which have a tendency to crystallize in frosty weather, clogging up fuel injectors. Certain polymers can be added to control this crystallisation, but a lack of clarity over how they work is preventing further development.

"Our study needed highly brilliant X-rays. The ESRF provided that."

PhD student Arthur Zarrouki and other researchers from C2P2 laboratory visited ID02 to perform USAXS, SAXS and WAXS on diesel samples as they were cooled down *in situ*, to better understand how polymer additives affected the crystal structure. "A kinetic study of the cooling of diesel requires a rapid measurement, and hence highly brilliant X-rays," says Zarrouki. "The ESRF provided that."

Sometimes a collaboration with the ESRF can lead to a completely new product. This was the case with Ariel Excel Gel, an invention of the consumer-goods company Procter & Gamble (P&G). Drawing on ID02 SAXS data, P&G scientists were able to pioneer a stable liquid-crystal microstructure that enabled the washing detergent to be manufactured with very little water, without the use of an environmentally unsound organic solvent. The detergent even cleaned at 15°C, saving energy.

Soft matter collaborations are not always one-off. The consumer-goods company Unilever has a long-running collaboration with the ESRF to find healthier alternatives to the saturated fats found in oily food products, such as processed meat, chocolate and margarine. One possibility are the cholesterol-lowering sterols γ-oryzanol and β-sitosterol, which are found in sunflower oil. A few years ago, SAXS and WAXS data taken at the ESRF beamline ID02 showed Unilever scientists that, within sunflower oil, the sterols self-assemble into hollow tubules – suggesting they might lend oily foods a similar texture to saturated fats.

The researchers wanted to know whether the sunflower oil was strictly necessary for the tubules' formation. Returning to ID02 recently, therefore, they performed SAXS and differential scanning calorimetry on samples with different amounts of oil. The data showed that, in samples containing less than a third or so of oil, the tubules collapse – giving the Unilever team an idea how little oil they can get away with.

One thing is for certain, there is no limit to the number of soft-matter products whose chemistry and structure could be improved based on data taken with the ESRF's instrumentation. Says chemist Eric Robles, P&G's global coordinator for synchrotron research: "There are only very few capabilities that the ESRF doesn't have."

Jon Cartwright

An essential partnership

Soft matter studies with X-rays and neutrons have a much-needed ally.

The PSCM takes up the second floor, and some of the ground floor and basement, of the EPN campus's Science Building.

DIEGO PONTONI/ESRF

Imagine starting a long-awaited study at the ESRF, only to discover that your precious biological samples degraded on the flight to Grenoble. Or imagine that, once here, you suddenly have an idea for a novel sample environment that doesn't exist yet. Or perhaps you have a long-term vision for your soft-matter work – one involving not just synchrotron and neutron science, but sustained assistance and complementary techniques.

These are all real situations at the EPN campus for which the Partnership for Soft Condensed Matter (PSCM) has been indispensable. A joint ESRF–ILL initiative, the PSCM comprises a dozen laboratories in the EPN campus's Science Building, which adjoins the ESRF and ILL experimental halls (see photo right). Here visitors can find more than 30 laboratory tools, including atomic force and optical microscopes, rheometers, calorimeters, ellipsometers and light scattering apparatuses – not to mention various sample preparation facilities and technical expertise. The PSCM is dedicated to advanced soft-matter research, with applications ranging from nano-materials and environmental science, to energy and biotechnology.

Stronger science

The PSCM concept was born in 2004 when an ILL scientific output study suggested that soft matter research could be further strengthened if neutron experiments were complemented with conventional laboratory techniques onsite. With the ESRF on board, a number of leading European scientists and representatives of the two facilities – notably Giovanna Fragneto of the ILL and Theyencheri Narayanan of the ESRF – began to make the concept a reality. The entire process took about a decade, from the organisation of the first joint Soft Matter User Meeting in 2006, to the completion of the PSCM implementation phase at the Science Building this year.

The PSCM instruments are shared between the ESRF and the ILL, and access is designed to be easy and transparent. That can be ad hoc for a single beamtime, or recurrent over multiple beamtimes. There was the occasion, for example, when visitors came to rescue a jeopardised experiment by making and checking some new liposome samples; or when our engineers designed, produced and installed for some visitors a specialist "magnetic actuation" sample environment in a particular beamline. The PSCM is also a hub for networking and scientific exchange, and contributes to the organisation of soft matter seminars, colloquia, workshops and large international conferences. A dedicated office is reserved for scientific visitors willing to explore and test the PSCM environment.

Besides ad hoc visitors, the PSCM caters for those who need longer-term continuous access, for the development of particularly complex projects. In this case, their hosting institutions can freely apply for Partner status to the PSCM Steering Committee, which makes a decision based on independent, expert scientific evaluations in a confidential manner. Four PSCM Partner institutes have already started their programmes, with results going from "one shot" texture analysis exploiting photon energy (DOI: 10.1002/anie.201603784), to the imaging of keratin bundles in cells using a combination of X-ray techniques (see "Seeing in cells" below).

The PSCM's support is ever-growing, and its large community of ILL users is being joined by an expanding range of ESRF users. The PSCM welcomes all users who find its services useful – regardless, in fact, of whether their samples are technically "soft" enough. We have those studying biomimetic nano-systems, bulk and interfacial self-assembly, complex fluids, bio-minerals, colloids, polyelectrolytes and nanoparticles. The PSCM will do its best to support everyone, and hopes to be expanding its services in preparation for the upcoming ESRF Extremely Brilliant Source (EBS), from its inception in 2020 onwards.

Diego Pontoni, ESRF–PSCM coordinator

"The PSCM is dedicated to advanced soft-matter research."

• For further information visit **tinyurl.com/PSCMinfo** or e-mail **pscm-support@esrf.fr**.

Teamwork helps to see inside cells

X-ray imaging of biological cells is a promising complement to fluorescence and electron microscopy. But while resolution is verging on the nanometre scale, the doses risk destroying samples.

This year, scientists at the first official Partner of the PSCM, the Institute for X-ray Physics at the University of Göttingen in Germany, demonstrated at the ID13 beamline that doses could be lowered by combining two X-ray techniques, ptychography and nanodiffraction, on the same sample. The data gave the group, which was led by Sarah Köster, structural information on the number of 10 nm-thick single keratin filaments making up bundles inside a cell, without inflicting strong radiation damage (DOI: 10.1021/acsnano.5b07871).

Jon Cartwright

A story of length scales

Jean Daillant, director of France's SOLEIL synchrotron, explains why he finds soft matter so interesting.

SOLEIL

Jean Daillant in brief

Born: 1963, Chalon sur Saône, France.

Education: Ecole Normale Supérieure de Saint-Cloud and Université Pierre et Marie Curie; PhD, Paris-Sud University (1989).

Career: Research scientist, CEA (1989); deputy director, LURE (1999); head of LIONS, CEA, (2003) head scientific council, SOLEIL (2006); director, SOLEIL (2011).

> **"The ESRF brought possibilities you couldn't previously imagine."**

Jean Daillant was doing his doctorate at the University of Paris–South in France when the buzz really began to grow around synchrotron science. It was 1989, and he was using lab-generated X-rays to study how a liquid spreads when it wets a surface – a phenomenon that, back then, was considered important in the performance of all sorts of coatings, from paints to pesticides. His instrumentation could see the average fluctuations of liquid molecules – but over at the German Electron Synchrotron in Hamburg, physicists Peter Pershan and Jens Als-Nielsen were demonstrating that synchrotron X-rays had the potential to see much finer details. Suddenly, says Daillant, "there was excitement about the possibilities X-rays could open up".

In 1994, having joined the French Atomic Energy Commission (CEA), Daillant had his first opportunity to get in on the excitement. Now focused more on the surfaces of bulk liquids, which govern how a liquid interacts with a neighbouring solid or gas, he visited the newly opened ESRF – the only synchrotron in Europe that had enough flux to observe the correlations of fluctuations, known as capillary waves, among water molecules on the surface of water. "It was clear that with the ESRF, we had the tool we needed," he says. "It brought possibilities that you could not imagine doing before."

Promising start

It also brought the start of Daillant's career in synchrotron science, specialising in soft matter. Although capillary waves had been studied at large scales before, using the scattering of laser light, the synchrotron allowed Daillant to see down to nanometre scale, where the subtle effect of van der Waals forces could be observed. It was a line of research he would pursue for about a decade, during which time he became a joint director of the now decommissioned Laboratory for Electromagnetic Radiation (LURE) synchrotron in Orsay, France. In 2006, SOLEIL, the French national synchrotron, was opened near Paris, and Daillant chaired its scientific council; five years later, he would be named its director. Being optimised to cover the entire electromagnetic spectrum, from hard X-rays up to terahertz and infrared wavelengths, SOLEIL was designed to complement the ESRF, which was primarily designed for X-rays of high energy.

By this time Daillant had turned his attention towards a topic that has occupied him to this day: lipid membranes – bilayers of fatty-acid molecules – in water. This was a "natural extension" of his previous work, he says, as the membranes act like almost pure, two-dimensional surfaces; for other, biology-focused scientists, they have potential implications for the way biological cells transmit nutrients. In a recent study, conducted at the French Collaborating Research Group beamline BM32, he and his colleagues from the University of Strasbourg in France have found that electric fields cause lipid membranes to lose tension but gain rigidity – a surprising result, due, he thinks, to a capacitor-like accumulation of charges on either side of the bilayer (DOI: 10.1103/PhysRevLett.116.228101). Indeed, he believes that the result might help scientists to understand how electric fields boost the permeability of cell membranes during gene therapy.

Finding time

A director's duties are many, of course, and it is not always easy to squeeze in the science. "I try to do some research in the early morning, before 9 o'clock," he says, "and participate in experiments from time to time." Between the many meetings that he must attend, he keeps an eye on recent developments in soft matter. At the moment, for instance, he is particularly intrigued by research into nano-confined fluids.

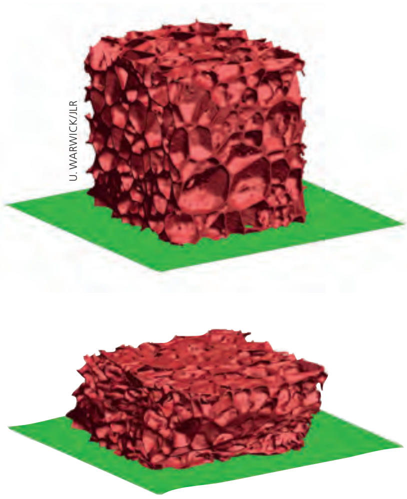
"In some sense, soft matter is a story of different length scales. So you have the famous Debye length for the electrostatic interaction, which can be microns to angstroms, and you have van der Waals forces, which are at the nanoscale, and you have the molecular length and so on. What's interesting about nano-confined fluids is that all these length scales are confined to be of the same order of magnitude, and you start to have some very interesting effects."

So what is it that attracts Daillant to soft matter in general? "What I personally find interesting," he says, "is this interplay between very precise experiments and statistical physics. At synchrotrons, you really can get a very precise description, and use that along with theory to understand quite complex systems."

Jon Cartwright

Foam boosts car safety

Jaguar Land Rover and the University of Warwick team up with the ESRF to observe how safety foams deform *in situ*.


U. WARWICK/JLR


MALCHICK743 (ALAMY)

Microtomography (above) reveals how safety foam disperses energy under load for car headrests (right).

Energy absorption is important for the safety of car passengers. Besides the seat foam used for comfort, modern vehicles have a denser foam such as expanded polypropylene (EPP) inside headrests and bumpers that decelerates passengers in such a way as to minimise any stresses on them. Ideally, a foam would do this by deforming in a controlled manner, reducing the maximum forces experienced by the occupant.

Researchers from the University of Warwick in the UK and the car manufacturer Jaguar Land Rover (JLR) have tried to optimise the use of EPP foam by studying its energy-absorption properties under deformation at the ESRF. The work was carried out by Craig Carnegie, a JLR-sponsored engineering doctorate student at Warwick, who developed new material models to improve a computer-aided design process.

At the beamline ID19, assisted by ESRF scientist Alexander Rack, the researchers performed microtomography, which enabled them to continually image their EPP foams (see figure above) as they were slowly compressed with a dedicated press facility. Incorporating the images into a three-dimensional computer model, the researchers could analyse them to understand how to improve the foam, and how much of it to use in a vehicle for optimal performance.

"As an automotive manufacturer, we've been following the progress of this research closely," says Mark Blagdon, a materials engineer who led the project from JLR. "The facilities available at the ESRF will allow us to improve the use of polymer foams as energy absorbers in our vehicle range."

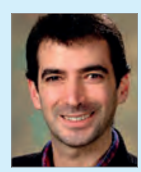
Materials scientist Darren Hughes at Warwick believes the benefit of the ESRF was the ability to track the deformation *in situ* and in great detail. "It clearly showed the response that the EPP material provides," he adds.

Movers and shakers

Helmut Schober Is the new director of the ILL, taking over from Bill Stirling. The appointment comes as part of a whole new management team at the ILL, including **Mark Johnson** as the new director of science and Associate British Director, which came into effect on 1 October. A physicist who has studied fullerenes, liquid and glass dynamics and neutron instrumentation, Schober was previously the ILL's director of science and Associate German Director.

Robert Feidenhans'l will be the next chair of the management board of the European X-ray free electron laser (XFEL) in Hamburg, Germany, after the retirement of the current chair Massimo Altarelli at the end of this year. Feidenhans'l, who is currently heading the Niels Bohr Institute at the University of Copenhagen in Denmark, has previously chaired the XFEL council, and has been involved at major synchrotron facilities including the PSI in Switzerland, DESY in Hamburg and the ESRF.

Amina Taleb-Ibrahimi is the new director of matter sciences at the SOLEIL synchrotron near Paris, France. A condensed-matter physicist, Taleb-Ibrahimi has been the director of research at the French National Centre of Scientific Research (CNRS) and the deputy science director of Very Large Research Infrastructures at the CNRS Physics Institute; previously, she oversaw the development of the photoemission beamline Cassiopée at SOLEIL. She began her new role on 1 September.



Laurent Chapon is the new director of physical sciences at Diamond Light Source on the Harwell campus in the UK. Chapon has worked at Harwell before as group leader for the crystallographic and engineering group at the ISIS neutron facility, as well as being a regular user of Diamond, working on new materials and multiferroics. In 2011 he moved to the ILL and, in 2013, became its diffraction group leader. He began his new role on 14 November.

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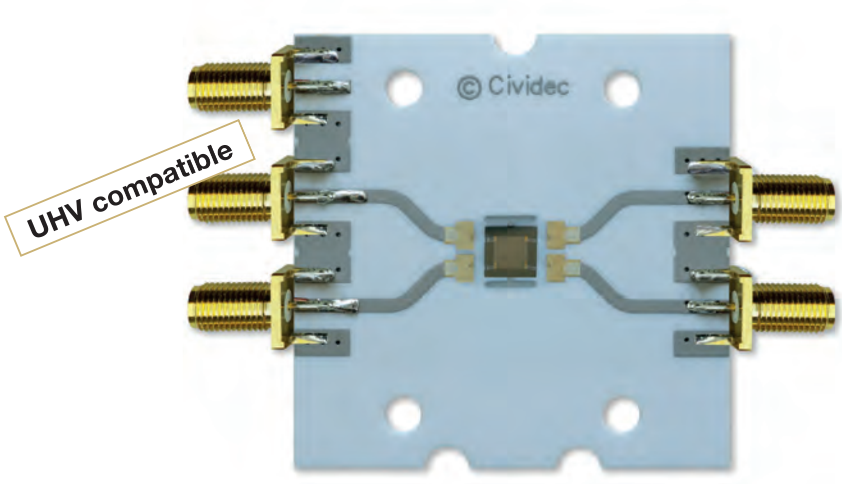

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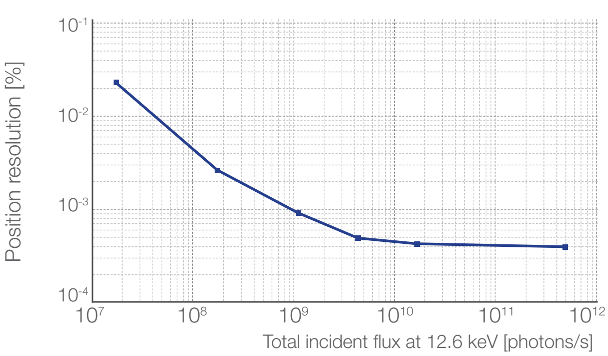

Figure: Measurements at Diamond Light Source Ltd., UK, show the measured position resolution at 1 kHz bandwidth for various beam intensities of the 12.6 keV photons. A position resolution of better than 0.1% of beamsize is obtained even for an incident flux as low as 10^9 photons/s.

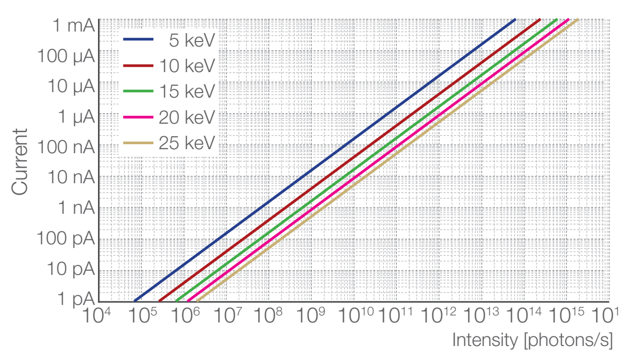

Figure: The detector response as a function of the photon energy and intensity demonstrates the wide dynamic range of the XBPM System in combination with the C8 Electrometer Amplifiers.

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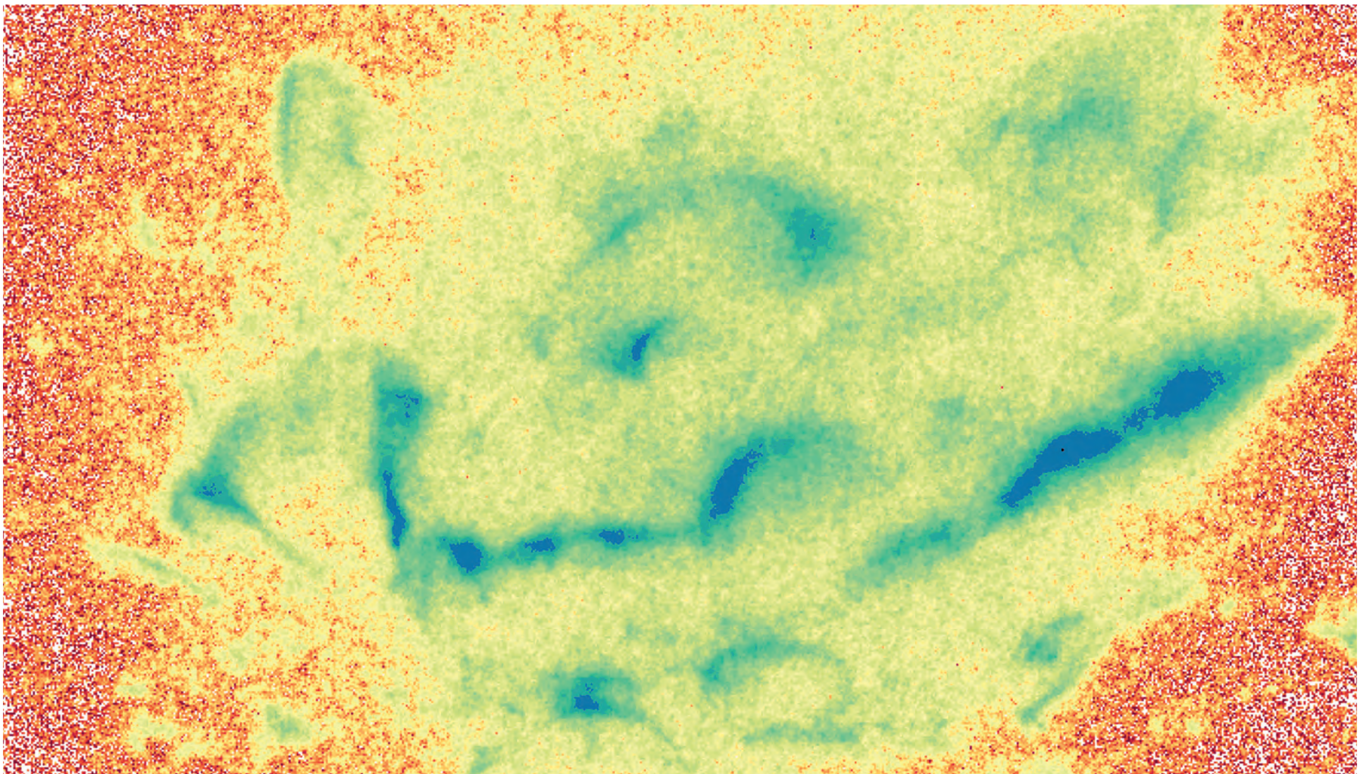


DR TAO ZHOU/ID01

Unstable: A silicon battery electrode absorbs lithium ions, forming regions of high order (blue) and low order (yellow). This image of diffracted X-rays, recorded at one angle with the new Full Field Diffraction X-ray Microscope (FFDXM) on the ESRF's ID01 beamline, suggests that the lithium ions are not being absorbed everywhere in the same manner – a trait that could lead to electrode instability. Silicon is a promising electrode material because it has the potential to outperform current materials in terms of capacity and charging speed, but currently undergoes strong structural changes that can lead to rapid degradation. FFDXM, which was made possible thanks to the ESRF upgrade, helps to study this by resolving structural information over a large surface area.

In the corridors

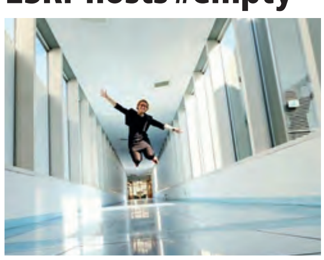
EUROPEAN XFEL

XFEL starts commissioning

How many officials does it take to fit a two-metre length of tube? In the case of the European X-ray free electron laser (XFEL), the answer is 11 – one for each member country. The officials – together with some 350 other politicians, administrators and scientists from around the world – oversaw the "symbolic" installation of one of the final sections of beamline at the site in Hamburg, Germany, to mark the beginning of commissioning in October. The first experiments by external scientists are due to begin in summer 2017.

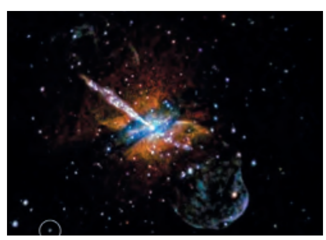
• Meanwhile, XFELs are getting ever more popular. The UK's Science and Technology Facilities Council issued a report in August calling for the construction of a UK XFEL by 2020. The move marks a strong turnaround for the country: in 2009 it pulled out of the European XFEL, only to re-join in 2014. A UK XFEL is expected to cost £500 m (€550 m), and could open by 2026.

ESRF hosts #empty



@STEPHV38

Instagrammers were overjoyed to visit the the ESRF in October, as part of an #empty event to expose scientific and cultural institutions to the younger generation. A rare opportunity for the public to go behind the scenes at the synchrotron, #empty brought together science, digital art and a certain playfulness.



ASA/CXC/UNIV. BIRMINGHAM/M BURKE ET AL

Mysterious X-rays come and go

Astronomers have glimpsed mysterious objects in neighbouring galaxies that, for about an hour, emit X-rays with a luminosity of up to 10^{34} watts – making them brighter than neutron stars. Trawling through archival footage from the Chandra and XMM-Newton space observatories, the astronomers found the objects on the outskirts of the Virgo galaxy NGC 4636 and the Centaurus A galaxy, NGC 5128. No-one knows for certain what the objects are: they are unlikely to be neutron stars or magnetars because the flares last too long. The astronomers believe they could be intermediate-mass black holes, though cannot explain the reason for the phenomenon.

Rare flower on site

ERNDH

The EPN campus upped its biodiversity credentials this summer after a few specimens of a rare orchid were spotted. *Spiranthes autumnalis*, known commonly as Autumn lady's-tresses, is a small grey–green orchid producing white flowers just 5 mm long with a green interior, in a helix around the stem. Though not currently threatened with extinction, the species may become so, according to the international Convention on International Trade in Endangered Species of Wild Fauna and Flora, as its natural habitats are disappearing due to urbanisation and environmental change.