THE EUROPEAN SYNCHROTRON

ESRF news

Number 70 July 2015

Shaping a sustainable future

Phase II upgrade officially launched

Industry targets breast cancer

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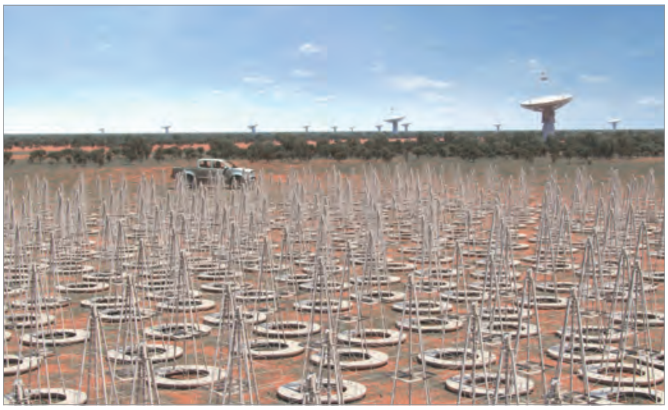
The European Synchrotron



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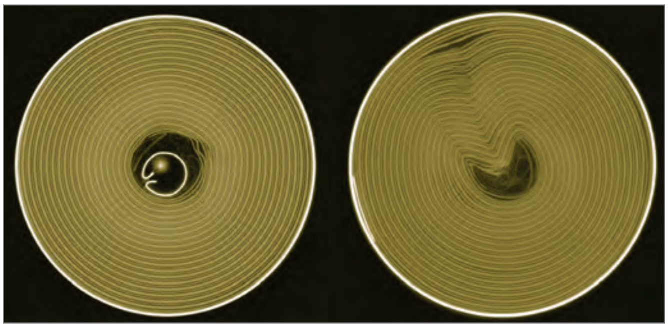

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ANA PRADAS DEL REAL

Tracking silver nanoparticles in the environment, p25.

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Shaping a sustainable future

In December this year, France will host the United Nations international climate change conference. During this crucial gathering it is hoped that 195 countries will be able to agree on the very ambitious objective of limiting global temperature rises to less than 2 °C above pre-industrial levels, beyond which it is estimated that catastrophic climate change will occur. Its success represents a major challenge for the whole planet that will benefit our, and future, generations, and therefore requires action from all stakeholders including governments, local authorities, civil society and industry.

Scientists have a major role to play in identifying the most effective way to understand, mitigate and eventually control global warming. As an international centre of excellence for research on materials and life sciences, which provides unique tools for fundamental, applied and industrial research, the ESRF can make important contributions to address the great new environmental challenges of the 21st century.

During the past 20 years, environmental sciences have increased their share of the research portfolios of synchrotrons in general, and at the ESRF more specifically. Indeed, the ESRF offers precisely the co-ordinated, shared and multifaceted approach required to shape sustainable development, uncover renewable materials and processes, improve energy management, and understand and mitigate pollution.

Taking action

This edition of *ESRFnews* is dedicated to recent research in the fields of energy and environment that has been made possible by the powerful synchrotron X-ray techniques available at ESRF beamlines. Such techniques are allowing users to: better understand how lithium-ion batteries work (p16); assess the long-term safety of high-level nuclear waste storage (p19); develop powerful catalysts for converting biomass into renewable fuels and chemicals (p21); gain deeper knowledge of new materials for solid-fuel combustion with inherent carbon dioxide capture (p22); and track the fate and toxicity of nanomaterials in waste-water treatment plants (p25).

We could have cited many further examples of research made possible by the cutting-edge scientific equipment at the ESRF, which includes the unprecedented nano-analysis capabilities of beamlines ID01, ID11, ID13, ID16A and ID16B. These beamlines and others allow scientists to characterise materials and processes at multiple scales and under realistic working conditions for future energy sources such as fuel cells, or for developing materials for hydrogen storage, solar cells, high-temperature superconductors and catalytic converters for cars.

"Environmental sciences have increased their share of the research portfolio"

Unveiling the innermost secrets of matter to make a better world is a quest that drives many of the scientists who use the ESRF in their research. With the launch of the Upgrade Programme Phase II – an innovative €150 m modernisation project to take place in 2015–2022 (see p8) – the ESRF is consolidating its pioneering role in synchrotron science. By paving the way to a new generation of light sources that will produce more intense, coherent and stable X-ray beams, the ESRF will enable even deeper exploration of matter to provide relevant answers to new environmental problems.

I wish to thank the ESRF users who have presented their exciting results in these pages and thus demonstrated the value of the ESRF in this vital area of research.

Francesco Sette, *ESRF director-general*

Didier Wermeille of XMaS shares his experience with students.

EPN trip tackles gender bias

Studies consistently show that females are underrepresented within science, technology, engineering and mathematics (STEM) subjects. Women typically make up 10–20% in academic STEM disciplines, a picture that is broadly similar in industry. Although there is no single explanation for this gender bias, recognised factors include a lack of female role models and stereotypes of scientists as males.

To help tackle some of these issues, in April the ESRF co-hosted 14 female students from the UK aged 16–17 years who had won a four-day long trip to Grenoble to experience the life of an international scientist on the EPN Campus. The competition, which was managed by the University of Warwick in partnership with the ESRF's UK-operated beamline XMaS and Institute Laue-Langevin (ILL), was launched in selected schools with entrants asked to write an essay on the legacy of the Nobel-prize winning X-ray crystallographer Dorothy Hodgkin.

The junior scientists toured the ILL and ESRF, where they met XMaS and other ESRF researchers. "They were extremely enthusiastic and were fascinated by the fact that so many different fields of research could be covered at the same central facility," says co-organiser and XMaS scientist Laurence Bouchenoire.

As a result of the experience, the students' perceptions about people working in STEM careers had clearly changed. "The trip made me more confident in pursuing my scientific aspirations as a female scientist and helped me to see that STEM industries were open to females," said A Couzens from Cardinal Newman Catholic School. "I am more likely to become a scientist because of this trip."

Tango shoots for the stars

The ESRF's in-house developed control system, Tango, has been chosen to manage the vast data and networking requirements of the Square Kilometre Array (SKA) – an international effort to build the world's largest radio telescope. The decision was announced in March, after the SKA collaboration made comparative tests of several major control systems addressing more than 140 criteria. Tango will now form the common framework for all SKA phase-1 work packages.

The SKA project, which has members from 11 countries including Italy, the Netherlands and the UK, envisions a distributed telescope with a collecting area of 1 km^2 that will address questions about galactic evolution, dark energy and whether we are alone in the universe.

South Africa's Karoo desert will host the core of the high- and mid-frequency dishes, with Australia hosting the low-frequency antennas. Once complete, the telescope will use thousands of dishes and up to a million antennas that will scan the sky in more detail and much faster than any previous system. The number of controlled points, the huge amount of data produced, the computation requirements and the network traffic to be handled represent one of the most challenging technological issues in the world, says the team.

"SKA's choice confirms that Tango is a mature solution with a clear roadmap for the next 10–20 years," says Andy Götz of the ESRF software group. "Branching out into astronomy will bring new applications, ideas and developers into the Tango community. We welcome the SKA community and look forward to working with them."

Tango has already become the *de facto* control system for European synchrotrons, and has also been adopted by the Laser Mégajoule fusion facility under construction near Bordeaux, the Onera wind tunnel in Toulouse, and the Extreme Light Infrastructure in the Czech Republic, Hungary and Romania.

SKA ORGANISATION

Tango, an open-source control system created at the ESRF in 1998, will help deal with the unprecedented demands of the SKA telescope.

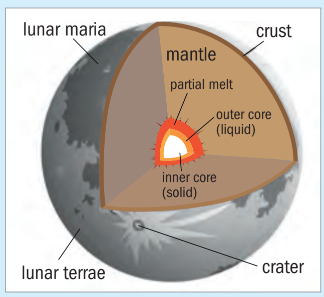
X-rays probe Moon's core

An international team led by researchers from the Institute of Mineralogy, Materials Physics and Cosmochemistry (IMPMC) in Paris have used high-pressure experiments at ESRF beamline ID28 to shed light on the structure and composition of the Moon's iron core.

Scientists already have valuable information about the moon's internal structure from seismic records obtained during the Apollo space programme, but these data alone are insufficient to reveal the properties of the lunar core. The new study, which involved measuring density and sound velocity in iron under pressures and temperatures similar to those of the lunar core, helps researchers interpret the seismic data and makes it possible to construct a direct model of the composition, structure, density and velocity of the lunar core (*PNAS* **112** 3916).

Iron, which is the major constituent of the cores of Earth-like or "telluric" planets, adopts a hexagonal compact structure under conditions of the Earth's inner core. But it is expected to have a face-centered cubic structure under the lower pressures of smaller planetary bodies, such as the Moon, Mercury or Mars. To investigate this, the team compressed iron samples to pressures up to 19 GPa in diamond anvil cells and heated them to 1150 K. The sound velocity was determined by inelastic X-ray scattering measurements, whereas the crystalline structure and its density were determined by X-ray diffraction.

The results indicate that the seismic velocities currently proposed for the inner core of the Moon are much lower than those found in face-centred cubic iron under simulated lunar-core conditions, and are also lower than those of other typical iron based alloys. "This study brings significant new constraints for the seismic model of the Moon's core and the cores of small telluric planets," says lead author Daniele Antonangeli of the IMPMC. "Thanks to the high brilliance of the ESRF source, and the large diversity of available techniques, material properties can be directly measured at these extreme conditions."



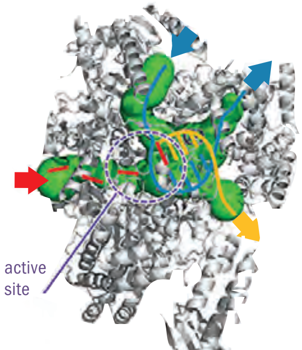
WIKI COMMONS / KELVINSONG

Like the Earth, the Moon has a distinct crust, mantle and core.

Viruses share replication machinery

A team from EMBL-Grenoble has used the ESRF to determine the first detailed 3D structure of the replication machinery of the La Crosse orthobunyavirus (LACV) – which is in the same broad group of viruses as influenza and can cause human encephalitis (DOI: 10.1016/j.cell.2015.05.006).

Stephen Cusack and Juan Reguera from EMBL-Grenoble, who jointly led the research, found striking similarities to influenza virus polymerase (whose atomic structure was previously determined by the same team) and now believe that all viruses within this group are related, opening up the possibility of quicker routes to developing treatments for the diseases they cause. Many dangerous human viruses use RNA as their genetic material instead of DNA, and have a specialised machine called the polymerase to replicate their RNA genomes. LACV and influenza viruses have a very specific way of replicating whereby they "snatch" a "cap" of genetic material from the host cell mRNA and insert it into their own.



Four internal tunnels (green) allow the vRNA template (blue) to enter and exit the polymerase active site, and allow the nucleotide building blocks (red) to enter and the product RNA (yellow) to exit.

"These viruses have evolved separately over millions of years, so they are genetically quite different," said EMBL PhD student Piotr Gerlach. "Yet their polymerases maintain the same structural architecture and carry out replication and transcription of the genome in the same fashion."

Using X-ray crystallography and cryo-electron microscopy, the team was able to identify that both ends of the vRNA attach to the polymerase to create a circle, and then sections of vRNA thread themselves through the polymerase before rejoining the circle again. "The ESRF MX beamlines, especially ID29 and ID23-1, were critical in measuring the X-ray data," Cusack told *ESRFnews*.

September proposal deadline extended

Since user operation began in 1994, the deadlines for submission of proposals for ESRF beamtime have remained unchanged: 1 March and 1 September for the spring and autumn proposal rounds, respectively. However, starting from 2015, the date of the autumn deadline will change to 10 September.

It is expected that this later deadline will give scientists more time to prepare proposals and result in a more even balance between the proposal rounds in terms of the number of proposals received and the work required by the evaluation committees. Typically, 10% fewer proposals are received for the September deadline than for the March deadline, resulting in a significant imbalance for the review committees.

User corner

At the last proposal submission deadline on 1 March 2015, 1062 proposals were submitted requesting a total of just over 15,000 shifts of beam time. Of these, 475 proposals totalling just over 7000 shifts were allocated, representing a success rate of 45%.

The next deadline for standard proposal submission is **10 September 2015** for beam time during the period March to July 2016. Please note the new date for the September round submission (see article above). Potential proposers are invited to check the status of open ESRF beamlines for the 1 September deadline (www.esrf.fr/UsersAndScience/UserGuide/Applying/beamline-status).

The ESRF User Meeting 2016 will be held on the EPN Campus from 8–10 February inclusive. In addition to the plenary session, the programme will include a range of tutorials and there will be three user-dedicated microsymposia on the themes of nanoscience: X-ray diffraction and coherence, dynamics of complex systems, and the future of (time-resolved) room temperature protein X-ray crystallography.

News from the beamlines

- Beamline **ID01** will finish its upgrade this year and will be fully opened for user proposals at the 10 September deadline. It currently already supplies coherent beams down to 100 nm in size. The new primary optics allow the beamline to work with Si(111) monochromators in channel-cut or double-crystal Bragg geometry, or with a larger bandpass delivered by a multilayer monochromator delivering a factor of 20 times more flux. Dark-field microscopy under diffraction conditions, currently under commissioning, will also be opened to users.
- Beamline **ID15** is currently undergoing upgrade. The new beamline will be on a canted section: ID15A will be dedicated to materials chemistry and materials engineering, while ID15B (formerly ID09HP) will be dedicated to high-pressure science. Radiation tests are foreseen for the end of November/beginning of December 2015, with the beamlines in user operation mode from summer 2016.
- Beamline **ID19** has a new multimodal monochromator designed for high-quality imaging applications that is now available for user experiments, offering double Bragg and double Laue Si(111) configurations in the energy range 10–200 keV and 30–200 keV, respectively. Pending completion of the control system, energy modifications currently require direct supervision of the local contact.
- The Rossendorf beamline (ROBL-CRG, **BM20**) is now operating as a pure XAFS spectroscopy beamline. The focus of ROBL-RCH is experiments involving actinides due to its dedicated radiochemistry installation, but will also include XAFS experiments for geosciences, chemistry, catalysis and similar fields that can make use of ROBL's superb detection limits and high level of specialisation in (bulk) XAFS spectroscopy. In the future, additional techniques such as high-resolution spectroscopy and scattering experiments at the mineral/water interface will be implemented.
- The X-ray side-branch at beamline **ID21** has been fully refurbished, offering a new end-station dedicated to micro X-ray diffraction and micro X-ray fluorescence 2D mapping. It is operated at fixed energy (8.5 keV) with a micrometre-probe in air. This new instrument complements the existing set of 2D micro-analytical techniques already available at ID21, namely micro X-ray fluorescence and micro X-ray absorption spectroscopy in the tender X-ray domain (2–9 keV) and micro-infrared spectroscopy in the mid-infrared region. Samples can be easily transferred from one instrument to another to allow combined identification and localisation of elements, species, molecular groups and phases. ID21 is therefore particularly well suited for the 2D characterisation of complex and heterogeneous samples.
- An aluminium flight tube has been installed to replace the plastic one at the **BM29** Bio-SAXS beamline to improve the vacuum in the beam path. Commissioning of a new *in situ* high-performance liquid chromatography (HPLC) system, to be used in parallel with the sample changer robot, is underway and the device will be available for external users from June 2015.

Phase II upgrade officially launched

A press conference held on 29 May in the presence of the ESRF Science Advisory Committee (SAC) marked the official launch of Phase II of the ESRF Upgrade Programme. During the next five years the ESRF will see the creation of an ultra-bright synchrotron source with a performance 100 times superior to present day facilities, opening a new chapter in synchrotron X-ray science.

Initiated in 2009, the ambitious ESRF Upgrade Programme will extend until 2022 and is being implemented in two phases. Phase I, which is scheduled to be completed on time and within budget by the end of 2015, saw the construction and refurbishment of 19 experimental stations and 8000 m^2 of new ultra-stable experimental-hall floor, in addition to enhanced instrumentation and accelerator infrastructure.

In addition to a new X-ray source, Phase II (which runs from 2015 to 2022) also includes the construction of new state-of-the-art beamlines, an ambitious instrumentation programme focused on high performance detectors (see p11) and an intensified "big data" strategy designed to exploit the enhanced brilliance and coherence of the Phase II machine. The user programme will be put on hold from the end of 2018 to June 2020 while the storage ring is dismantled and the new source installed and commissioned.

JARNIAS/ESRF

Local and national journalists attended a press conference in the ESRF visitor centre to learn about the Phase II upgrade.

The ESRF Upgrade Programme represents a qualitative leap that will open new fields of investigation at the nanometre level, with applications including: nanoscopy for the conception of new materials; science in extreme conditions; nano-imaging; structural biology and medicine; materials science; nanotechnologies; and environmental and energy sciences.

"The recently launched next phase of the upgrade programme is without question the most exciting development in the ESRF's history since its inception," said SAC Chair Des McMorrow. "It will create what is essentially a new synchrotron, with a low-emittance lattice delivering up to a factor of 100 increase in source brilliance and coherent flux. This will open up new scientific opportunities across the biological, physical and engineering sciences, and will future proof the ESRF's position as a world leader in the X-ray sciences."

ID30B enters user mode

User operation of the ESRF's macromolecular crystallography beamline ID30B began in June, with a micro-diffractometer (MD2S) and a SC3 sample changer in place. ID30B is operated by the ESRF/EMBL Joint Structural Biology Group and is located at the second branch of the canted ID30 setup. Its tunable undulator configuration allows the energy of the beam to be varied from 6–20 keV, while the beam size at the sample position can be changed between 20–200 μm.

ID30B is equipped with the latest generation of single photon counting pixel detectors, providing a high photon flux. Commissioning of an EMBL/ESRF-developed versatile sample changer in combination with a high capacity Dewar is ongoing. User access to this configuration, which will allow users to send samples or come to the ESRF with samples mounted either in SPINE standard pucks or in unipucks, will be available from September.

Twin inaugurations for Phase I beamlines

Two new ESRF beamlines – ID01 and ID31 – were inaugurated on 28–29 May during the first meeting of the ESRF's new Science Advisory Committee, signalling the last steps towards the completion of Phase I of the ESRF Upgrade Programme by the end of this year.

Beamline ID01 is devoted to X-ray diffraction imaging for nano-analysis, with the ability to study a wide variety of materials at scales ranging from nanostructures to bulk solids and offering imaging and strain studies using coherent X-ray diffraction methods and/or nanodiffraction. The beamline, which is also dedicated to SAXS, GID and GISAXS, is ideally suited to *in situ* studies of devices at the nanoscale. ID01 was identified as one of the most important beamlines for upgrade during Phase I of the ESRF Upgrade Programme and is already proving a powerful probe with which to characterise nanowires, semiconductor structures and other application-relevant systems.

JARNIAS/ESRF

Till Metzger, Harald Reichert, Des McMorrow, Francesco Sette, Tobias Schülli and Jean Susini cut the ribbon outside the hutches of ID01.

Beamline ID31 is a high-energy beamline that combines diffraction and imaging techniques with auxiliary methods for the investigation of buried interface structures and materials processing. The long beamline covers energies in the 30–150 keV region for the study of working devices *in situ* and allows users to reduce the beam size to as little as 200 nm. This will allow studies of less perfect, more realistic, interfaces in order to understand the interplay between microscopic material properties and macroscopic device performance – with particular relevance for research into advanced materials for fuel cells, organic solar cells, rechargeable batteries and catalytic materials.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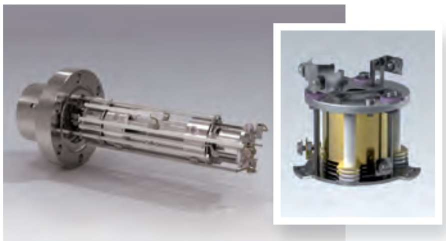
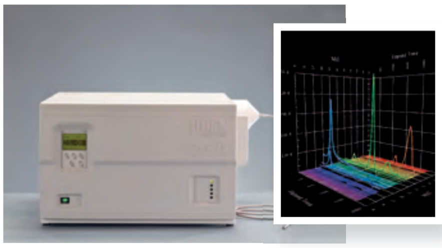
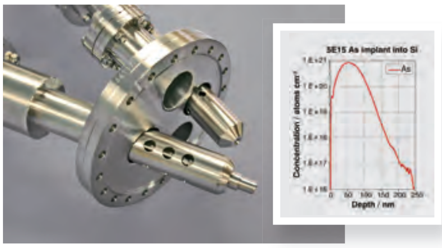






Phase II instrumentation on track

The new ESRF source will offer dramatic improvements in beam coherence and brightness, but only the right instrumentation can unleash its full scientific potential.

The X-ray flight tube at ESRF beamline ID02.

MCBRIDE/ESRF

The scientific output of the ESRF is underpinned by advanced instrumentation, which provides users with ever-improving X-ray beams and the necessary tools to exploit the beams for experiments. The ESRF stands out among synchrotron facilities in having a dedicated Instrumentation Services and Development Division (ISDD), which was established in 2009 to meet the challenges of the ESRF Upgrade Programme (ESRF UP). With ESRF UP PI on track for completion this year, the ISDD – which has recently come under new leadership (see box below) – is addressing the challenges of ESRF UP PII.

Taking place in 2015–2022, the recently launched Phase II upgrade will replace the ESRF storage ring with a novel design that provides a factor 100 increase in X-ray brilliance and coherence. The majority of the ESRF beamlines will reap immediate performance benefits, but new beamline instrumentation is vital to keep pace with the quantitative and qualitative improvements of the X-ray source – for example, requiring more advanced detectors.

"The expected generalisation of coherent scattering techniques to a wide variety of applications will require more efficient X-ray detectors that combine high sensitivity and spatial resolution," says Pablo Fajardo, who leads the ISDD's detector and electronics group. "The new detection systems should also be able to cope with the higher photon fluxes and exploit the time-resolution capabilities that the increased beam brilliance of the new storage ring will make possible."

Mapping the road ahead

The ISDD has two key objectives to maximise the PII opportunities. The first is to develop new instrumentation that exploits the new source properties to the full and thus opens the way for new scientific breakthroughs. The second is to consolidate ongoing instrumentation programmes that were initially foreseen within the scope of Phase I but suffered setbacks due to budget cuts. "It is crucial to scale these programmes back up to full capacity," says former ISDD head Jean Susini.

The proposed €20 m scientific instrumentation programme to be financed in the context of Phase II includes, in order of importance:

- The launch of a robust X-ray detector programme including development of two new 2D detector systems;
- A modernisation programme for beamline control and data analysis, which enable more efficient use of ESRF instruments;
- Upgrades to network bandwidth, storage solutions and other information technology to manage and process large volumes of data;
- Development of advanced metrology tools and methods for X-ray optics;
- Development of new expertise in mechatronics and online metrology to enhance the stability, speed and user-friendliness of the new end stations.

In addition, advanced project management will be critical for astute distribution of resources between projects, including the construction of the new accelerator with minimal disruption to user operation. "The plans in the frame of ESRF Upgrade Programme Phase II constitute the pillars of an instrumentation roadmap that will ensure the ESRF remains at the forefront of synchrotron radiation science and further strengthen our role as a nursery for synchrotron technology – even beyond 2022," says ESRF director-general Francesco Sette.

Matthew Chalmers

Michael Krisch appointed head of ISDD

C ARGOUD

Michael Krisch has been appointed head of the ESRF Instrumentation Services and Development Division (ISDD), replacing Jean Susini who has held the position since the division was created in 2009. The appointment came into effect on 1 June and will last for five years.

Krisch joined the ESRF in 1988 and completed a PhD in the Optics Group. He first worked at beamline ID16 and in 1996 was appointed scientist-in-charge at ID28. Since 2009, he has been head of the Dynamics and Extreme Conditions Group and in 2012 he took over the project management of the Phase I upgrade beamline UPBL6 at ID20.

"I am looking forward to working together with my ISDD colleagues to develop cutting-edge instrumentation and to provide optimum support for the accelerator complex and ESRF's beamline portfolio," says Krisch, who has appeared on more than 200 publications in the fields of X-ray instrumentation, X-ray spectroscopy and vibrational properties of condensed matter. "It is a challenging, but equally exciting period for us and our user community."

Pixirad-1 System

The **Pixirad-1 System** is the first commercial product of Pixirad Imaging Counters s.r.l.

The core of the X-ray imaging system is a new detector, based on chromatic photon counting, that has been realized coupling a pixelated large area ASIC, known as **Pixie-II**, to a matching pixelated sensor by flip-chip bonding technique. The Pixirad-1 System is able to deliver extremely clear and highly detailed images for medical, biological, industrial and scientific applications.

Pixirad-1 Detector Module options

ASIC[1]*:*

– Pixie-II read-out ASIC, 60 µm hexagonal arrangement

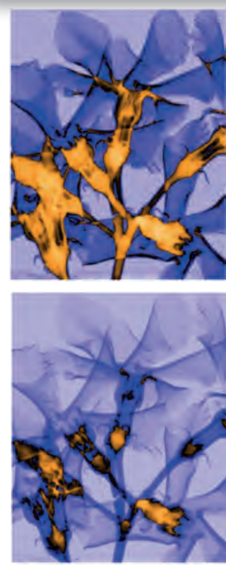
Sensors:

– 650 µm thick CdTe crystal Schottky type
– 750 µm thick CdTe crystal Ohmic type
– 500 µm thick GaAs crystal

[1] The Pixirad-1 Detector Module Unit is ready to use the new Pixie-III ASIC

Due to its architecture the Pixie-II ASIC is able to count incident X-ray photons according to their energy in order to produce two ‘color’ images from a single exposure.

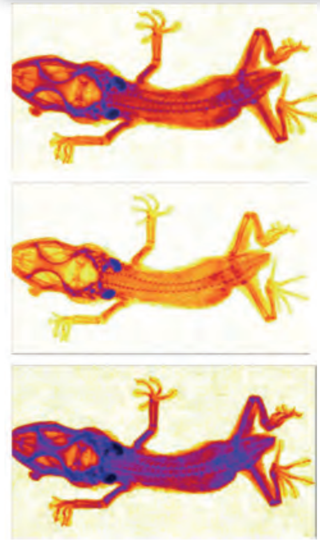
Low Energy Sensitivity



Images of a very low contrast object, taken with
a) 200 electrons global threshold corresponding to 1 keV (LOW counter, all photons)
b) at 6 keV threshold (1200 electrons). This image was taken in a single shot together with the previous one at 1 keV threshold

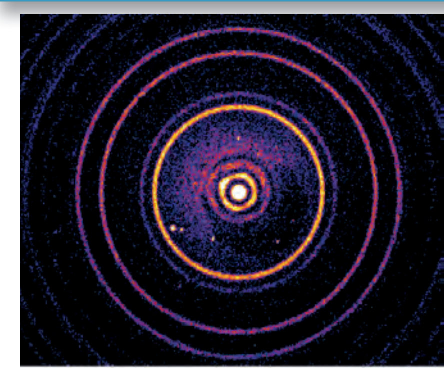
Chromatic Photon Counting

Three ‘colors’ from a single exposure



Images of a small dry animal obtained simultaneously by :
a) counting the X-ray photons with a low energy threshold (LOW COUNTER, all photons);
b) counting the X-ray photons with an higher threshold (HIGH COUNTER, high energy photons);
c) subtracting the previous pictures one from another (low energy photons)

X-ray Diffraction at 40 keV



Attenuated Beam and diffraction rings from a Ce02 powder (obtained at the Cornell Synchrotron on a 40 keV beam line)

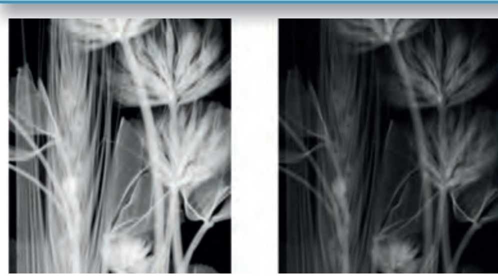
Pixirad-2 System

The new commercial product of Pixirad Imaging Counters s.r.l.

The **Pixirad-2** Detector Module Unit has 2 detector blocks (based on the **Pixie-II** ASIC) in a 2x1 pattern, with a global active area of 62 x 25 mm2.

The Pixirad-2 System is able to deliver extremely clear and highly detailed images for medical, biological, industrial and scientific applications.

Ohmic CdTe: Low Energy Sensitivity



Images of a very low contrast object, taken:
a) at 1 keV threshold (200 electrons, LOW counter, all photons);
b) at 6 keV threshold (1200 electrons) in a single shot

Pixirad-2 Detector Module options

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[1] The Pixirad-2 Detector Module Unit is ready to use the new Pixie-III ASIC

Cellular pump lights up optogenetics

Users have uncovered a light-activated ion pump that offers precise control of nerve-cell activity for the emerging field of optogenetics.

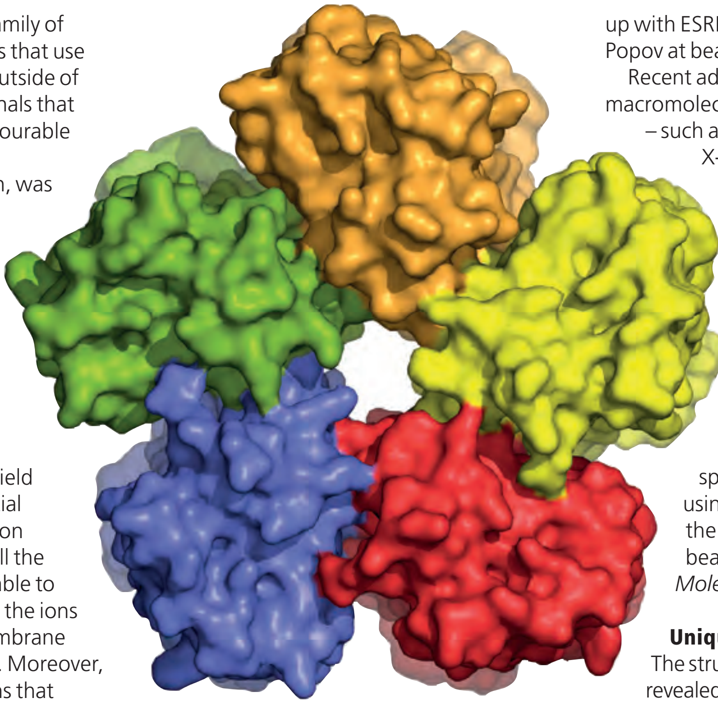
Microbial rhodopsins are a large family of photosensitive membrane proteins that use light to transport ions inside and outside of the cell, generating phototaxis signals that cause the cell to move towards favourable illumination conditions. The most studied system, bacteriorhodopsin, was discovered in 1971 but thousands more have since been identified in microorganisms as diverse as bacteria, algae and viruses, which exhibit various light-gated channels and light-driven proton and anion pumps.

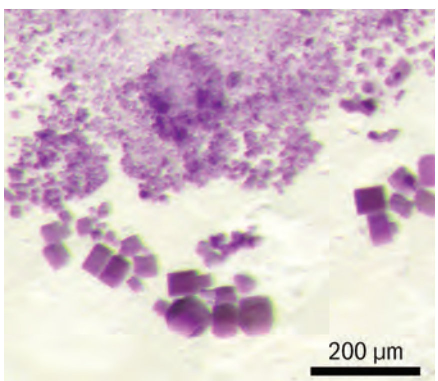
Incorporating these proteins into neurons has allowed precise control of neural impulses and lies at the foundation of the growing field of optogenetics, which has potential long-term applications such as vision and hearing regeneration. So far all the known light-driven pumps are unable to transport sodium and potassium – the ions whose motions across the cell membrane constitute natural neural impulses. Moreover, it was believed that the only cations that can be actively transported by microbial rhodopsins are protons.

That picture changed in 2013. While investigating the marine bacterium Krokinobacter eikastus, Hideki Kandori and co-workers at the Nagoya Institute of Technology in Japan unexpectedly discovered that during early periods of growth under illumination, K. eikastus cells are able to pump out sodium ions. It turned out that the corresponding ion transporter, dubbed KR2, belongs to a new type of microbial rhodopsin in which some of the most important ionizable amino acids are replaced with polar ones, and vice versa. Even when expressed in another commonly employed bacterium, E. coli, KR2 could still efficiently transport sodium ions. Yet it appeared that potassium ions, despite being very similar to sodium ions, were not transported by the KR2 pump.

Curious behaviour

This and other curiosities displayed by KR2 attracted the interest of our research group at Forschungszentrum Jülich in Germany, in partnership with colleagues at the Institute de Biologie Structurale (IBS) in Grenoble and the Moscow Institute of Physics and Technology in Russia. We had already carried out several structural studies of other microbial rhodopsins to investigate the molecular mechanisms behind their actions, but membrane proteins are notoriously difficult to handle and producing them is usually expensive and time consuming.





Top: Under physiological conditions five KR2 molecules spontaneously form a star-shaped complex, with each KR2 molecule being able to bind and transport a sodium ion across the membrane. Inside the proteins are the light-sensitive cofactors, retinals, which enable the pumping activity. Bottom: Crystals of KR2 used for structure determination.

The light-driven sodium pump KR2 is no exception. Since the protein could be produced only in batches of several milligrams, yet thousands of crystallisation conditions needed to be tested, we had to miniaturise and automate the process using robotic and imaging systems available at the IBS. The diffraction signal from the first crystals obtained, which measured less than 20 μm across, was too weak to be measured using in-house X-ray sources, so we teamed up with ESRF beamline scientist Alexander Popov at beamline ID23-1.

Recent advances at the ESRF's macromolecular crystallography beamlines – such as highly brilliant microfocus X-ray beams, automatic sample changers and convenient software protocols – greatly helped the search for the best crystals. After several months of trials at ID23-1 and ID29, we were able to improve the resolution of the diffraction data collected from around 20 Å to better than 1.5 Å.

Meanwhile we were able to extract information about KR2's spectroscopic characteristics using optical absorption spectra at the cryobench laboratory at ESRF beamline ID29S (*Nature Structural & Molecular Biology* **22** 390).

Unique features

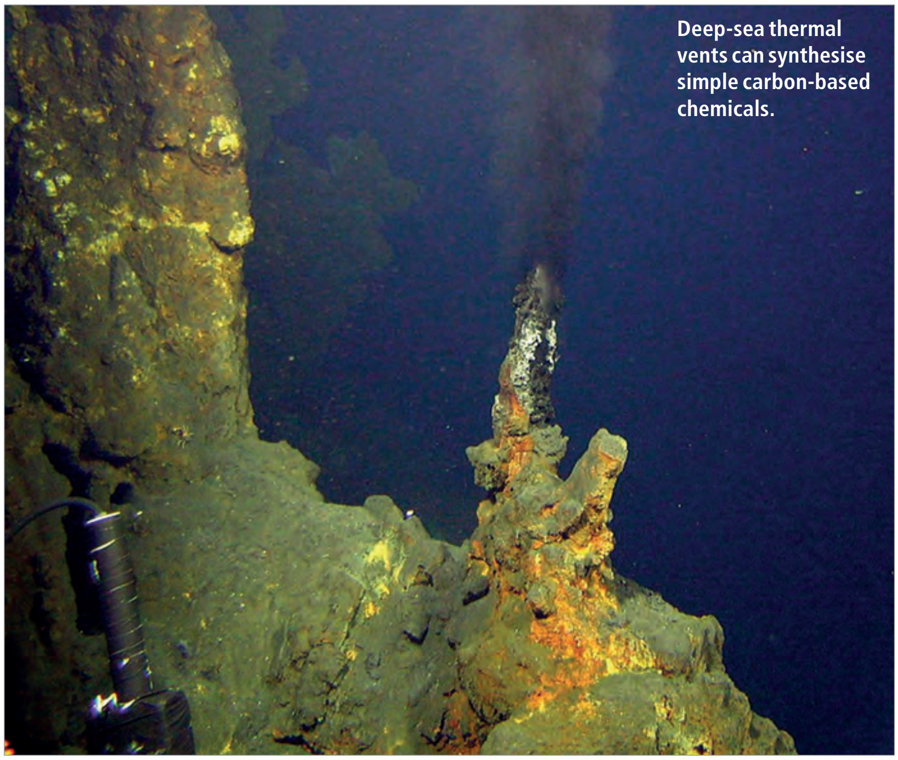
The structure of the KR2 sodium pump revealed a short protein helix that caps the outfacing opening of the pump like a lid. Another interesting feature of KR2 is the unusual structure of the inward facing ion-uptake cavity, which is unexpectedly large and protrudes from the protein surface. Hypothesizing that this structure could act as a kind of filter that allows KR2 to be more selective for sodium ions, we swapped specific amino acids at the relevant site using targeted mutations and found that KR2 indeed loses its sodium-pumping ability under such conditions. But we also found, in collaboration with optogenetics co-founder Ernst Bamberg at the Max Planck Institute of Biophysics in Frankfurt, that one of the mutations transforms KR2 into a light-driven potassium pump – the first of its kind.

For potential optogenetic applications, this result is especially interesting because transporting potassium ions from the cell is a natural neuron deactivation mechanism. Normally, activated neurons release the ions through passive potassium channels in the membrane, but a light-activated potassium pump would allow this process to be precisely controlled and therefore KR2 provides a highly effective off-switch for neurons. The next step is to find ways of integrating the pump into different types of cells. In combination with the light-activated Channelrhodopsin 2, which is a well known molecular off-switch, the KR2 potassium pump would form a perfect pair of tools for the precise control of nerve cell activity.

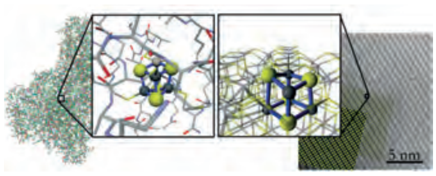
Ivan Gushchin and Valentin Gordeliy, Institut de Biologie Structurale, Grenoble.

Probing the origins of life

Hot vents on the seabed could have spontaneously produced the organic molecules necessary for life, suggest studies of minerals under extreme conditions.



Deep-sea thermal vents can synthesise simple carbon-based chemicals.



Schematic showing the ferredoxin centre of the CO dehydrogenase enzyme (left), and of the greigite surface showing its cubane structure (right). Greigite is found in hot vents and is structurally similar to Fe_4S_4 clusters found in ferredoxins.

How life emerged billions of years ago is among the biggest unanswered questions in science. One of the most promising theories suggests that increasingly complex carbon-based chemistry led to self-replicating molecules, and eventually to the appearance of the first cellular life forms. Research carried out at the ESRF has now revealed how one of these initial steps might have taken place. It proves that some of the key building blocks for organic chemistry were already being formed in nature before life emerged, and supports the hypothesis that life begun in undersea hydrothermal vents.

"There is a lot of speculation that hydrothermal vents could be the location where life on Earth began," says Nora de Leeuw of University College London (UCL) in the UK, who led the team. "There is a lot of carbon dioxide (CO_2) dissolved in the water, which could provide the carbon that the chemistry of living organisms is based on, and there is plenty of energy, because the water is hot and turbulent."

Carbon catalyst

The study shows how the surfaces of mineral particles inside hot vents have similar chemical properties to enzymes – the biological molecules that govern chemical reactions in living organisms. This implies that vents are able to create simple carbon-based molecules such as methanol and formic acid from dissolved CO_2 in the water, and then encourage these molecules to recombine into those usually associated with living organisms.

The team used supercomputer simulations to provide a molecule-by-molecule view of how CO_2 interacts with an iron sulphide mineral called greigite (Fe_3S_4), which is located on the inside surfaces of hot vents. They then compared the results to experiments performed at the ESRF's Dutch-Belgian beamline "DUBBLE" (BM26A). Using an electrochemical cell the team was able to replicate the conditions present in deep-sea vents, where hot and slightly alkaline water rich in dissolved CO_2 passes over greigite. Extended X-ray absorption fine structure (EXAFS) measurements allowed the researchers to investigate the precise conditions under which mineral particles act as catalysts in the conversion of CO_2 into organic molecules (*Chem. Commun.* **51** 7501).

"When we analysed EXAFS data of the greigite nanoparticles that were being used for the catalysis we found that the average bond distances, which are heavily influenced by the surface structure, were very similar to those modelled computationally, says coauthor Husn-Ubayda Islam, a PhD student at UCL who was sponsored by DUBBLE. "After building a very successful *in situ* electrochemical cell, we could look at the local structure of the greigite as the electrochemistry and catalysis occurred, which allowed us to gain significant insight into various phenomena."

Breaking bonds

The results show that the surfaces and crystal structures inside vents act as catalysts, which encourage chemical changes in materials that settle on them. Specifically, they break down the bonds between carbon and oxygen atoms so that these elements can combine with water to produce formic acid, acetic acid, methanol and pyruvic acid – simple carbon-based chemicals that pave the way for more complex carbon-based chemistry.

"Our studies set out to prove that iron sulphides, under certain relatively mild conditions, could act as catalysts for CO_2 reduction and conversion to organic molecules," continues Islam. "This was based on origin-of-life hypotheses, which have predicted for decades that iron sulphides deposited on sea beds from deep-sea vents could aid CO_2 conversion to larger organic molecules – in the same way that enzymes do in some living organisms, potentially providing a link to the first living organisms."

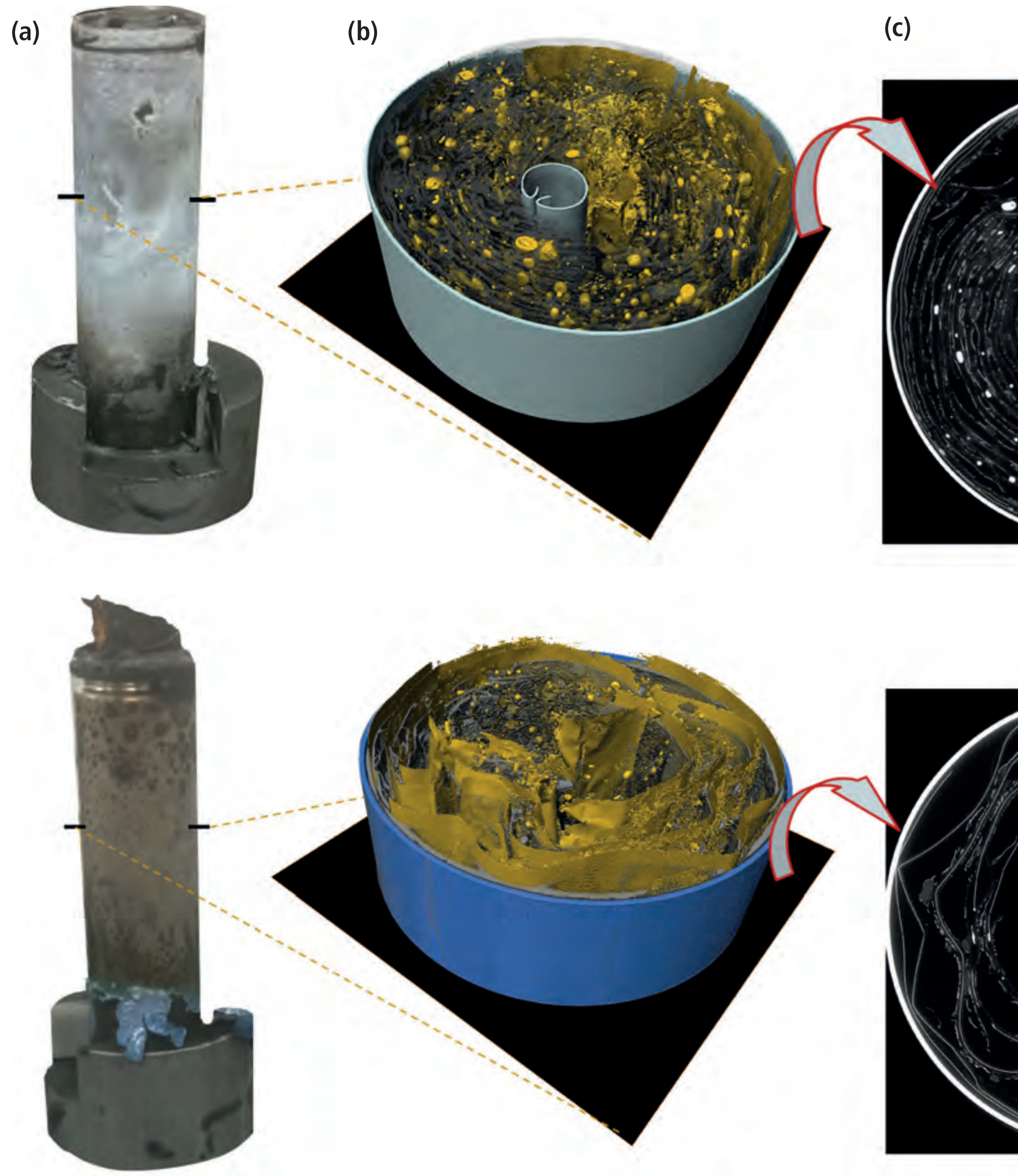
Practical applications

The study could also have practical applications, since it provides a method for creating carbon-based chemicals from CO_2 without the need for extreme heat or pressure. In the long term, the researchers claim, this offers a more environmentally friendly way to synthesise the raw materials for products such as plastics, fertilisers and fuels, which are currently produced from non-renewable raw materials. "If the process can be scaled up to be commercially viable, it would not only save oil but use up CO_2 – a greenhouse gas – as a raw material," says de Leeuw.

Matthew Chalmers

X-rays reveal workings

Lithium-ion batteries are key to building a clean and sustainable energy future. Powerful synch
in real time, with a recent study revealing for the first time what ha



In 2010, somewhere high above the Atlantic Ocean, a passenger on board an Air France Boeing B777 aircraft unknowingly dropped a lithium-ion battery down the side of a seat. When the seat was later reclined, the battery was crushed and caught fire. Although nobody was injured in this case, lithium-ion battery cargoes have since been linked to at least two fatal air crashes. Indeed, earlier this year three major airlines banned bulk shipments of the devices after tests carried out by the US Federal Aviation Administration found that overheating batteries could cause major fires.

Hundreds of millions of rechargeable lithium-ion batteries are manufactured and transported each year to power the modern world. Although failure is rare, understanding how the devices break down and potentially cause a dangerous chain reaction of events is vital for designing safer products – especially for applications such as hybrid electric vehicles, which require high-energy density batteries capable of operating under a range of demanding conditions.

Using the ESRF, a UK team from University College London (UCL), Imperial College and the National Physical Laboratory has shown for the first time how internal structural damage to batteries evolves in real time, and how damage can spread to neighbouring batteries. In conjunction with beamline staff at ID15A the team combined high-energy synchrotron X-rays and thermal imaging to map changes to the internal structure and external temperature of batteries as they were exposed to extreme heat (*Nature Communications* **6** 6924).

Thermal runaway

A battery has a range of critical temperatures, which, when reached, result in exothermic breakdown of its constituents. Once the rate of heat generation exceeds the rate of heat dissipation into the environment, the temperature rises and a sequence of detrimental events called thermal runaway can cause the battery to ignite. Preventing and mitigating thermal runaway is one of the greatest challenges for the safe operation of lithium-ion batteries, explains lead author Donal Finegan of UCL. "Following thermal runaway demands exceptionally high-speed imaging, and at ID15A we were able to capture 3D images in fractions of a second thanks to the very high photon flux and high speed imaging detector," he says.

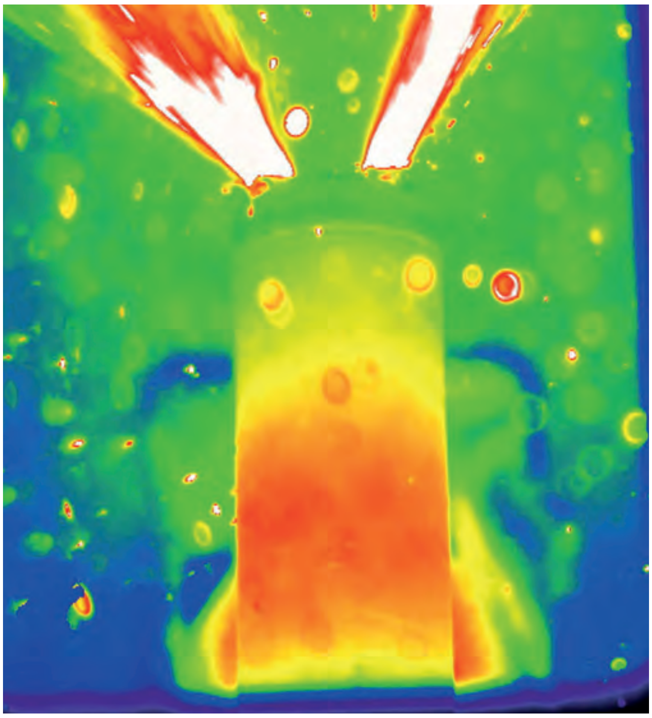
Although there have been studies of compositional and structural evolution of electrode materials at elevated temperatures, there is limited understanding of the change in the internal architecture of commercial cells leading up to and during thermal runaway and failure. Typically, researchers have only been able to analyse battery failure after the damage has occurred, but the high penetration depth of the ESRF X-rays allowed the UK team to study such processes *in operando* – offering insights into key degradation modes such as gas-induced delamination, electrode layer collapse and propagation of structural degradation.

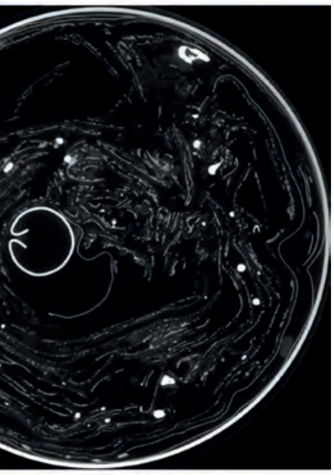
Support structure

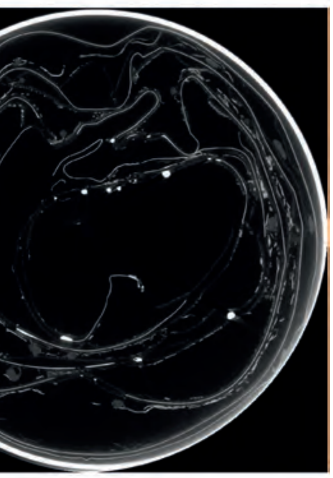
The researchers studied two distinct types of commercial lithium-ion batteries – those with and without an internal support – and monitored the effects of gas pockets, venting and heat on the internal layers as the battery shells were exposed to temperatures in excess of 250 °C (see figure). The battery that contained an internal support remained largely intact until the initiation of thermal runaway, after which the copper inside the cell melted (indicating temperatures of up to around 1000 °C) and heat spreading to

# of lithium-ion batteries

hrotron X-ray techniques at the ESRF are allowing users to follow the internal workings of cells
ppens inside a lithium-ion battery when it overheats and explodes.







Top: thermal imaging of a battery undergoing thermal runaway. Left: X-ray computed tomography reveals the effect of extreme heat on the internal spiral-wound layers of commercial lithium-ion batteries with (top row) and without (bottom row) internal supports. (a) external view of the cells after thermal runaway; (b) 3D reconstruction showing isolated copper phase (yellow), other broken-down material, battery casing and central cylindrical support; (c) greyscale slice from the XY plane.

the outside of the battery caused thermal runaway. The battery without an internal support, by contrast, exploded and caused the entire cap to detach and its contents to eject. Prior to thermal runaway, the tightly packed core of the unsupported cell collapsed, increasing the risk of severe internal short circuits and damage to neighbouring objects.

"Although we only studied two commercial batteries, our results show how useful our method is in tracking battery damage in 3D and in real time," explains co-author Paul Shearing of UCL. The destruction observed is very unlikely to happen under normal conditions, he adds, since the batteries under study were exposed to conditions well outside the recommended safe operating window.

The team now plans to study what happens with a larger sample size of batteries and in particular will investigate how changes at a microscopic level cause widespread battery failure. "Hopefully from using our method, the design of safety features for batteries can be evaluated and improved," says Shearing.

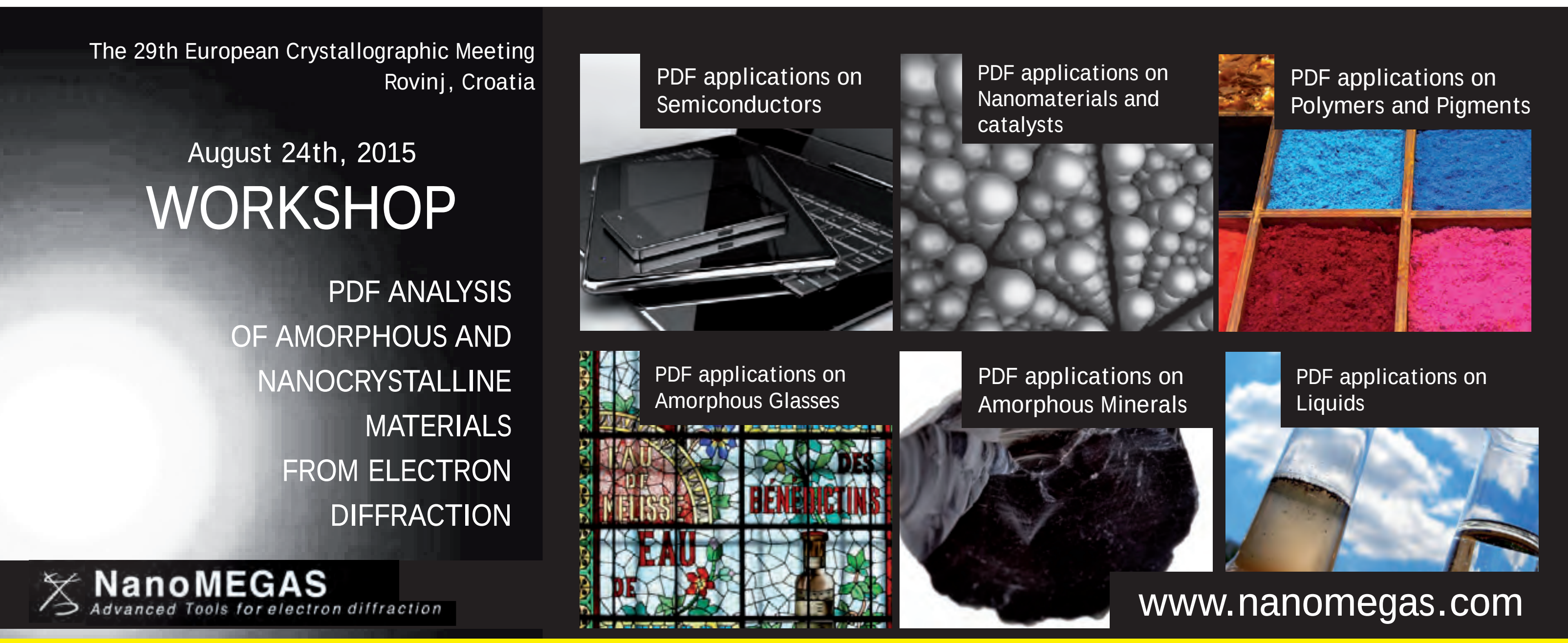
Matthew Chalmers

Advanced cells for charge and discharge studies

Two independent user groups at the ESRF's Swiss-Norwegian beamline (SNBL) have developed advanced cells with which to study the microscopic processes taking place within batteries as they undergo charge and discharge cycles.

A team from the Paul Scherrer Institut (PSI) has developed a cell that allows X-ray absorption spectroscopy (XAS) and X-ray diffraction (XRD) – two commonly used methods to study the reaction processes – to be carried out simultaneously, and seals samples against air and moisture. "The Swiss-Norwegian beamlines at the ESRF are one of the few beamlines worldwide to offer this possibility," says Claire Villevieille of the PSI. "The cell allows XAS at energies as low as the Ti-K edge and XRD of the material during cycling, and is very easy and quick to assemble." The researchers demonstrated the cell's potential by determining the reaction mechanism upon lithiation and delithiation of the model material $Fe_{0.5}TiOPO_4$, for which neither XAS nor XRD alone provide a complete picture of the underlying chemical processes (*J. Phys. Chem. C* **119** 3466). The cell will be especially useful for studying materials that involve the oxidization or reduction of more than one type of atom, explains Villevieille. "We will be able to test such materials with a special focus on the reversibility of the underlying reactions, which hopefully will help us develop advanced materials for the batteries of the future."

Meanwhile, a team from Technical University of Denmark has used the SNBL to demonstrate a new kind of capillary battery that will aid the development of improved lithium-air batteries – which offer higher energy densities than lithium-ion cells (DOI: 10.1039/c4ta04291c). "In the lithium-air battery research field we have an ideal reaction and a 'real' reaction, so in order to understand the chemical reactions taking place in the battery we need to investigate batteries as close to their running conditions as possible," explains lead author Mie Møller Storm. "Our study confirms the expected chemical reaction in the first cycle of the battery, but the results also show how X-ray exposure influences the diffraction peaks."



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Keeping nuclear waste safe

By bombarding thin films of uranium oxide with X-rays in the presence of water, users are exploring the long-term effects of storing spent nuclear fuel in geological repositories.

Nuclear power is a reliable, readily available, carbon-free energy resource and therefore presents an attractive solution to the increasing demand for electricity and the global drive toward low-carbon energy technologies. Today, more than 400 commercial nuclear reactors operate in 31 countries, providing 11% of the world's electricity. With these facilities together producing around 12,000 tons of high-level radioactive waste each year, it is essential that a safe and efficient storage strategy for such material is found.

Although there is no long-term radioactive waste storage facility currently in operation, many governments are considering adopting a "multi-barrier" approach whereby waste is encapsulated and then buried in a deep geological repository. Since the waste must be stored over timescales of tens of thousands of years, however, there is still a very real possibility that the stored fuel will one day come into contact with groundwater.

With uranium dioxide (UO_2) being the predominant component of nuclear fuel, the presence of groundwater would not normally be a cause for concern because it is insoluble in its stable state. However, once the fuel has been used during reactor operation, the presence of highly radioactive fission products results in extremely strong radiation fields that are able to cause radiolysis of the water. This process yields highly oxidising species that can transform UO_2 at the surface into the readily soluble UO_2^{2+} ion. In other words, this mechanism has the potential to cause dissolution of the spent fuel and release harmful radionuclides into the environment.

"High-level waste must be stored for tens of thousands of years."

Source-probe experiment

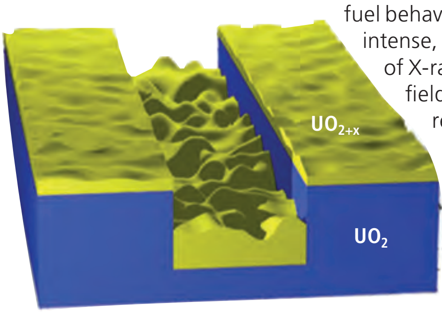
Detailed studies of the structure of spent fuel in such an extreme environment are prohibitively complicated, but there are other experimental routes that might shed light on fuel behaviour. We have used an intense, monochromated beam of X-rays to mimic the radiation fields present in a geological repository, with the aim of investigating their effect on a UO_2 / water interface (DOI: 10.1039/C4FD00254G).

This was first achieved on the ESRF's UK operated XMaS beamline in a joint project carried out between XMaS and Diamond Light Source in the UK, by exposing single crystal UO_2 thin films to ultra-pure water in the presence of a 15 keV X-ray beam.

As well as initiating radiolysis, the beam was also used to measure X-ray diffraction and reflectivity so that we could probe crystalline structure, surface morphology, oxide layer density and, ultimately, the dissolution of UO_2 as a function of exposure time. Modelling the reflectivity and diffraction data revealed a loss of crystalline UO_2, an increase in the UO_{2+x} hyperstoichiometric layer, a surface roughening, and eventually a loss in material due to dissolution (see figure).

These measurements help to refine predictive models of the spent fuel/water interface. Using engineered interfaces that are particularly sensitive to any change in surface structure, for instance, it is possible to investigate the role of individual variables. This information is important for validating theoretical models that attempt to predict the behaviour of fuels in contact with ground water over long timescales. Further complexity can now be introduced, for instance reproducing the effects of radiation damage, lattice deformation and fission product implantation. This research programme will take us towards a more complete understanding of the corrosion behaviour of spent nuclear fuel.

Ross Springell and Sophie Rennie, Interface Analysis Centre, University of Bristol, UK.



Along the X-ray beam path, the study found a loss of UO_2 material (blue) accompanied by an increase in thickness of a hyper-stoichiometric surface layer (UO_{2+x}) (yellow).

The International Energy Agency estimates that biofuels can provide up to 27% of the world's transportation fuel by 2050.

Harnessing the power of plants

An international team has used the ESRF to develop a powerful nano-alloyed catalyst for converting biomass into renewable fuels and chemicals.

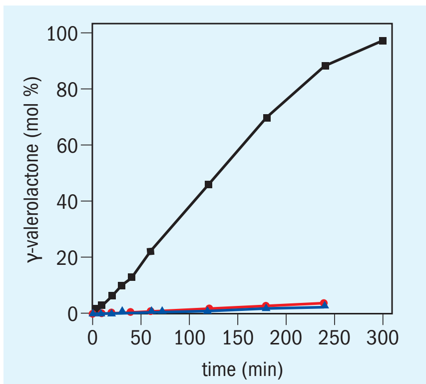
The modern world is built on crude oil, which is vital both as fuel and as raw material for chemicals and plastics. Yet it is also known that the use of crude oil is unsustainable, owing to its finite quantity and impact on the environment. Significant effort is therefore being put into finding ways to convert renewable resources such as agricultural or forestry waste into usable oil. Chemical building blocks derived from biomass, which includes straw, foliage and wood, can make a significant contribution to the demand for more "green" materials and fuels.

During the processing of biomass, a select number of chemicals form that serve as the basis for a wide range of renewable plastics, fuels and solvents. Catalysts are crucial for obtaining large yields of these intermediate products and for ensuring that they are properly converted, yet it has so far proved difficult to make the chemistry of the process fast and efficient.

Sustainable biofuels

A recent study at the ESRF's Dutch–Belgian "DUBBLE" beamline represents an important advance towards this goal. Wenhao Luo and colleagues at the Debye Institute for Nanomaterials Science in Utrecht, the Netherlands, together with researchers from University College London (UCL) in the UK and Lehigh University in Pennsylvania, US, have developed a new bimetallic catalyst that results in a greater yield of valuable chemical building blocks from biomass. It also produces fewer by-products and therefore less waste than conventional processing techniques (*Nature Communications* **6** 6540).

The research was carried out under the public–private partnership CatchBio, which is part of a €100 m investment programme funded by the Dutch government that aims to develop clean ways of processing non-edible biomass, and the team has already applied



Production of γ-valerolactone as a function of time during the hydrogenation of levulinic acid, using metal-on-titanium-oxide nanoparticle catalysts: monometallic gold (red); palladium (blue) and bimetallic gold-palladium (black).

"The improvement is unexpectedly excellent."

for a patent for its invention. "We worked closely together with a number of chemical companies that have already indicated being very interested in this catalyst," says Bert Weckhuysen of Utrecht University.

The new and reusable catalyst boosts the conversion of levulinic acid, a key platform molecule in biorefinery schemes that is derived from cellulose, into a more environmentally friendly building block called γ-valerolactone. Several metallic catalysts for this reaction have been trialed, with ruthenium nanoparticles showing good performance but also a tendency to deactivate with time. Nano-alloyed catalysts, by contrast, enable the structural and electronic properties of the metallic nanoparticles to be tuned in order to optimise catalytic activity.

Bimetallic benefits

The Netherlands-led team prepared bimetallic random alloys of gold-palladium and ruthenium-palladium supported on titanium dioxide. The gold-based system alone showed a 27-fold increase in activity compared with its monometallic counterparts, but the ruthenium-palladium system was found to control the desired conversion both quickly and specifically. "Gold or palladium on their own are not very good but in combination the improvement is unexpectedly excellent," says co-author Andy Beale from UCL. Extended X-ray absorption fine structure (EXAFS) measurements performed at the DUBBLE beamlines BM26A and BM23 played a crucial role in determining the nature and location of the catalytically active sites, adds Beale, allowing the team to determine the key to high catalyst efficiency.

Optimal performance was obtained with nanoparticles that measured approximately 1.5 nm in diameter and formed random alloys. Crucially, the results revealed that the higher efficiency of ruthenium-palladium catalysts is the result of a high degree of ruthenium dispersion in the palladium matrix.

"This structural information was determined by combining Fourier-transform infrared, X-ray photoelectron spectroscopy, scanning transmission electron microscopy and EXAFS data," explains Luo. "But it was the application of EXAFS in particular, recorded at both edges in the bimetallic samples, that allowed a detailed structural model of the active catalyst to be obtained."

Matthew Chalmers

Solid result for CO_2 capture

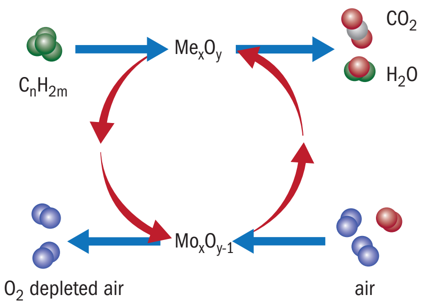
Bimetallic oxides present a promising new class of materials for solid-fuel combustion with inherent carbon dioxide capture.

SASKPOWER

Operational since 2014, a carbon capture and storage (CCS) facility implemented by SaskPower at the Boundary Dam power station in Canada can reduce carbon dioxide emissions by one million tonnes per year – equivalent to taking more than 250,000 cars off the road.

According to the Intergovernmental Panel on Climate Change, capturing and storing carbon dioxide (CO_2) is one of the most promising ways to reduce the effects of man-made greenhouse gas emissions. Indeed, studies by the International Energy Agency estimate that carbon capture and storage (CCS) technologies could contribute one sixth of the total reduction in CO_2 emissions deemed necessary to limit global temperature increases to 2 °C by 2050. Several large-scale CCS projects are already in operation in Canada, Norway and the US. However, the costs associated with such "first generation" technology – which is based mostly on amine scrubbing, whereby hydrocarbons are burned with air and CO_2 is then separated from the flue gas by passing it through an absorber – are very high, potentially limiting wider CCS adoption.

Chemical-looping combustion (CLC) is a next-generation CCS technology that could reduce the costs of CO_2 capture to as little as $7–15 per tonne. Instead of oxidising a hydrocarbon fuel in air, CLC uses a solid oxygen carrier, typically a transition metal oxide. The fuel is oxidised in a reactor, reducing the oxygen carrier such that the off-gas is a mixture of CO_2 and steam. Unlike in the conventional combustion process, CLC therefore inherently produces a pure stream of CO_2 (after the condensation of steam) and avoids the major cost of separating CO_2 from nitrogen in flue gases.



In chemical-looping combustion, a fuel is oxidized with oxygen derived from a metal oxide, producing CO_2 and H_2O (N_2 is absent). A pure stream of CO_2 is obtained via the condensation of steam, and the cycle is closed by regenerating the reduced metal oxide in air.

CLC was proposed in 1983 and was initially considered for gaseous fuels only. However, since coal is one of the major sources of anthropogenic CO_2 emissions, substantial research has gone into the development of CLC schemes that allow the use of solid fuels. Chemical-looping with oxygen uncoupling (CLOU) – in which molecular oxygen is released by the reduction of an oxygen carrier – is a promising way to transfer CLC to solid fuels. In this scheme, the fuel reacts with the oxygen released, producing a pure stream of CO_2 ready for compression and sequestration.

Two very attractive and extensively studied oxygen carriers for CLOU are CuO and Mn_2O_3, but both materials have drawbacks. Recently, we have overcome these shortcomings by developing bimetallic Cu-Mn oxygen carriers (DOI: 10.1039/C5TA01088H).

The materials are composed of a cubic spinel ($CuMn_2O_4$) phase and, depending on the mass ratio of Cu to Mn, also excess CuO or Mn_3O_4. We demonstrated the promising redox and oxygen release characteristics of the novel materials by cyclic redox experiments performed in a fluidised bed. Our results showed that the new oxygen carriers possess a substantially increased oxygen partial pressure when compared to CuO and a higher oxygen carrying capacity than Mn_2O_3, thus combining the advantages of both materials. We also were able to demonstrate that bimetallic Cu-Mn oxygen carriers combust charcoal fully – exceeding the performance of the current state-of-the-art material for solid-fuel CLC.

X-ray analysis

Since the improved reducibility of $CuMn_2O_4$ has been attributed to an electronic transfer between copper and manganese cations within the spinel lattice, we decided to further probe the material using X-ray absorption spectroscopy (XAS) measurements at the Swiss-Norwegian beamline "SNBL" (BM01B) at ESRF. This work allowed us to determine the oxidation states of the materials, without which we could not fully explain their remarkably improved characteristics compared to individual metal oxides.

Further *in situ* XAS experiments planned for the near future will provide insight into the reduction and oxidation mechanisms of these novel materials. Indeed, recent *in situ* XAS and powder diffraction experiments at SNBL have already allowed us to determine the reduction pathways and kinetics of another class of promising materials (Na^+-doped CuO-Al_2O_3 oxygen carriers) during repeated reduction–oxidation cycles.

The development of high-performance oxygen carriers for less costly CCS technologies is one of the key technological requirements to see large-scale CCS implementation in the mid-term future. Owing to their excellent redox and oxygen release characteristics, bimetallic oxygen carriers are highly promising materials for such architectures.

Christoph Müller, Davood Hosseini and Paula Macarena Abdala are at the Laboratory of Energy Science and Engineering, ETH Zurich.

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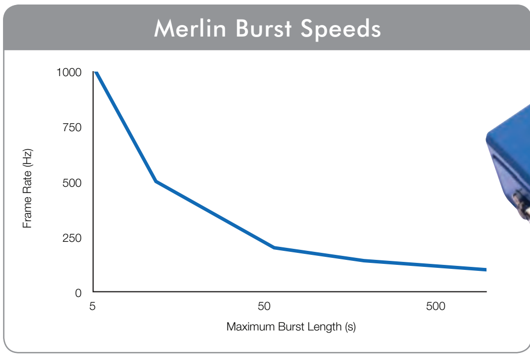

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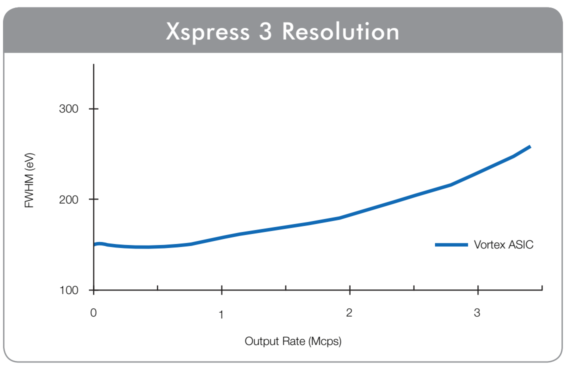

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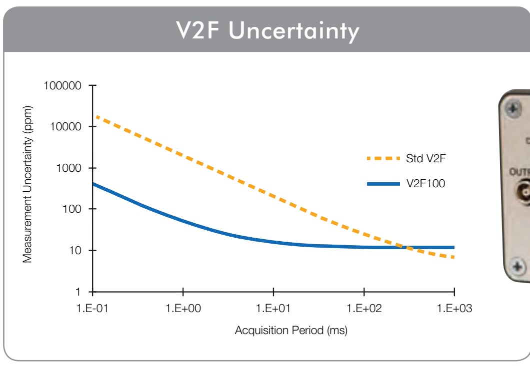

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Tracking waste nanoparticles

Multiscale X-ray techniques allow researchers to determine the fate and toxicity of nanomaterials released into the environment.

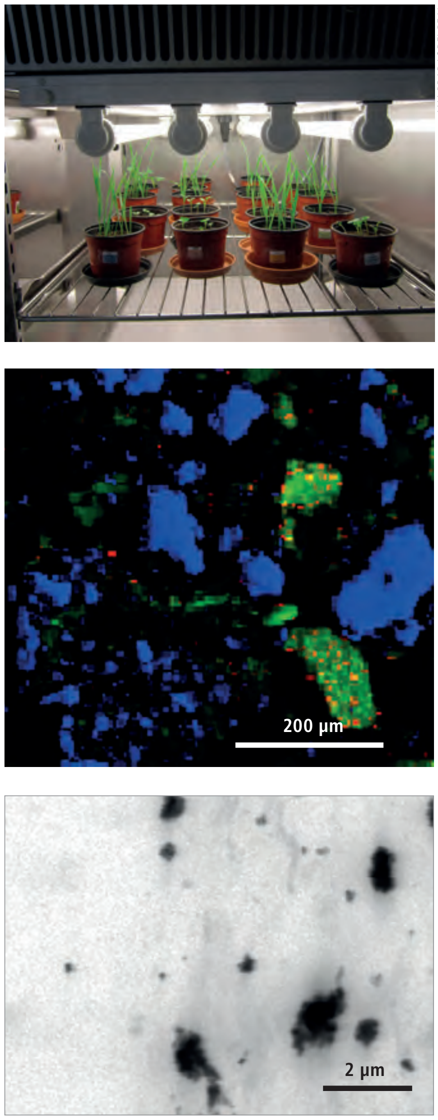
Silver nanoparticles (Ag NPs) are by far the most prevalent metallic nanoparticles in everyday use, being found in more than 240 consumer products including cosmetics, paints, fabrics and food containers. Ag NPs are popular for such applications due to their high antimicrobial activity, but what happens to these particles once they are released in the environment is not yet fully understood. Ag NPs leached from clothes during laundry, for instance, are transported by sewer systems to waste-water treatment plants that recycle water and produce sewage sludge as a waste product. Studies by one of the present authors, Ralf Kaegi, show that 90–95% of Ag NPs are retained in sewage sludge. Despite a lack of regulation concerning its nanoparticle content, around half of all sewage sludge produced in Europe is applied to agricultural soils as fertiliser.

Studying the fate of nanomaterials in the environment and evaluating their availability and toxicity in the long term is thus vital for safety and regulation purposes. This is a real analytical challenge because NPs have a low concentration and their interactions with natural constituents are poorly understood. Moreover, NPs in the environment may form amorphous and/or other weakly ordered secondary phases that are difficult to identify. By combining bulk and laterally resolved X-ray absorption spectroscopy and X-ray fluorescence imaging techniques, the ESRF is proving to be a powerful probe of such systems.

Entering the environment

Waste-water treatment plants are considered key markers for the flux of NPs entering the environment. Extended X-ray absorption fine structure (EXAFS) experiments performed at the ESRF's Dutch-Belgian "DUBBLE" beamline (BM01B) have previously shown that elemental silver NPs contained in waste water undergo a sulfidation process in sewer systems and treatment plants, leading to the formation of silver sulfides (*Water Research* **47** 3866). However, until now there have been no studies of the fate of Ag NPs in agricultural soils that have been amended with sewage sludge.

In a project funded by the French public–private partnership LabEx SERENADE, based in Aix-en-Provence, and in collaboration with the Swiss Federal Institute of Aquatic Science and Technology (Eawag), Ag NPs were deliberately introduced to a pilot waste-water treatment facility. The anaerobically digested sewage sludge was mixed with an agricultural soil, and crop plants (rape and wheat) were grown on this substrate at the Institut des Sciences de la Terre (ISTerre) laboratory in Grenoble. Silver speciation in the original sludge and in the amended soil before and after plant culture was studied using Ag K-edge bulk EXAFS at beamline BM30B, while the distribution of silver in the samples was studied using micro X-ray fluorescence (µXRF and µXANES) at ESRF beamline ID21 and nano-XRF at beamline ID16B. Complementary studies were performed using transmission electron microscopy coupled with energy dispersive X-ray spectroscopy.



ANA PRADAS DEL REAL

A multiscale analytical approach allowed users to track nanoparticles in sewage sludge and in amended soils where plants have grown: µXRF at ID21 (middle) shows the distribution of silver (red), sulphur (green) and silicon (blue), while nano-XRF at ID16B (bottom) shows nanoscale silver aggregates.

The results confirmed the transformation of Ag NP into silver sulfide (Ag_2S) in sludge and in soil samples, but EXAFS and µXANES data also revealed a less crystalline phase surrounding the Ag_2S. This species might be not as stable and insoluble as silver sulfide, whose solubility in its macro-crystalline form is extremely low, and therefore might be more available for take up by living organisms. Indeed, results from µXRF, µXANES and nano-XRF indicated a preferential association of Ag NPs with organic matter rather than with mineral components, although more research is needed to evaluate whether these species are stable in the long term and in particular during the turnover of soil organic matter. The extremely high sensitivity and resolution of ID16B also allowed us to detect diffuse zones of very low Ag concentration, suggesting some dissolution of Ag NPs and/or Ag_2S and the formation of secondary, possibly complexed, species. Our results therefore challenge the current idea that Ag NPs transform into a single crystallised form of Ag_2S that is highly insoluble and thus barely available for uptake by plants in soils (Pradas del Real *et al.*, in prep).

Holistic picture

This study demonstrates the great value of combining bulk-, micro- and nano-focused X-ray techniques with other lab-based methods. Additional techniques such as micro X-ray diffraction at beamlines ID21 and ID13, and nano X-ray absorption spectroscopy at ID16B, will provide even more precise structural characterisation of the system. Close collaboration between researchers in environmental sciences and ecotoxicology, and scientists and technical staff from several ESRF beamlines, especially ID21 beamline scientist Hiram Castillo-Michel, was essential for this project.

Overall, this ongoing study should allow unprecedented characterisation of the final form of Ag in soil in terms of particle size and speciation. Other results on soil microbiology, plant phytotoxicity and Ag bioavailability currently under acquisition will provide a holistic picture of the system. Such studies conducted under environmentally realistic conditions are urgently needed to evaluate the risk associated to the use of nanomaterials in consumer products.

Ana Pradas del Real and Géraldine Sarret are at ISTerre in Grenoble; Ralf Kaegi is at Eawag in Dübendorf, Switzerland.

A global approach to communication

The ESRF's new head of communications, **Delphine Chenevier**, describes her plans to increase the visibility of the ESRF.

Delphine Chenevier, who was appointed ESRF's head of communications in May, describes her career so far as "a succession of opportunities". She has always had an interest in research and innovation, but after completing a diploma in political science she did not have a specific career ambition. "I prefer to create networks and opportunities, and to work on projects," she says.

Delphine worked for more than 10 years in the office of the Mayor of Grenoble, and in 2008 co-ordinated Grenoble's bid to host the Winter Olympic and Paralympic Games in 2018. Two years later, she founded her own communications firm and undertook consultancy work with local clients such as Minatec and other GIANT partners. She then joined the office of the French minister for higher education, research and space science as a press and communications advisor.

The ESRF is a dynamic institute that is easy to settle into, she has found so far. "All the people are very welcoming and very open to communication," she says. "They see communication as part of a global strategy so they are very interested to promote their research."

Sense of purpose

Developing a global communications strategy that serves each of the ESRF's needs in the best way is one of Delphine's main goals. This translates into developing a public-relations strategy to promote ESRF science, supporting management's strategy and development of the ESRF, as well as facilitating internal communication and addressing the needs of the users and scientific community, she explains. "It's important to have a purpose and an aim for different targets, such as scientists, industrial partners, press, education and outreach and internal communications."

C ARGOUD

Delphine Chenevier in brief

Born: 1975, Auxerre.

Education: Graduate of Grenoble Institute of Political Studies (1996), with additional masters in economics, sport and recreation management (1998).

Career: Advisor then chief of the office of the mayor of Grenoble (1999–2010); founder of PR and communication company (2010–2012); press and communications advisor to the French minister for higher education, research and space (2012–2014); ESRF head of communications (2015–).

Family: Married, one child.

Interests: Climbing, mountains, sports in general.

> **"It's important to explain the ESRF's societal and environmental impact."**

Greater ESRF press activity is one of Delphine's aims, via press releases, press conferences and strong relationships with science magazines and large public media at a local, national and international level. While it is important to highlight the ESRF's scientific results, she says, it is also important to write a story to explain the societal, environmental and economic impact of the research, which conveys how an international institute like the ESRF can bring relevant answers to the new challenges the world has to face.

Another important goal concerns the ESRF's digital strategy, with plans for a new website, digital newsletters and enhanced social media to better target different audiences. "When you look at organisations like CERN or ESA they have a strong strategy on social networks and the Web, which is important because it strengthens the links between the user communities," she says. Another strand of her job will be public outreach and education activities, such as the synchrotron@school programme. "The public knows the ESRF as a landmark but they don't know what its impact is, so I'd like to explain: what is the ESRF, how does it work, and what is the social, environmental and economic impact. As a fundamental research institute, there is a very large field of applications for ESRF science."

Phase II opportunities

Delphine is also considering how to manage communications for Phase II Upgrade Programme, which will involve a shutdown of more than one year. It's important to create a story around the upgrade as CERN did with the LHC, she explains, and also to "personify" the Phase II upgrade by highlighting the people who work on this innovative project.

Delphine does not identify particular high or low points in her career so far, instead describing her CV as "a succession of great adventures". "I do my job with passion and I invest a lot," she says. "I like to develop a project and have pleasure in doing it in a collective manner with a team." She also enjoys a challenge: while a student she was a competitive sports climber and was member of the French climbing team.

"My most important challenge at the ESRF is to develop a global strategy and to share it, so that it's not just a thing done by the communication group but one that reflects the spirit of all the staff," she adds. "I would like that communication is seen as a support to the global strategy, not just an added-value thing but a very important aspect for the ESRF's development."

Matthew Chalmers

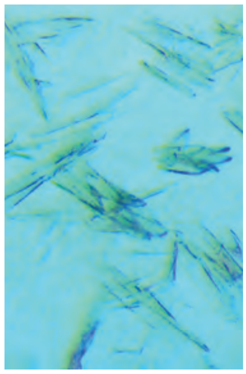
Partnership takes aim at breast cancer

A project carried out between the ESRF, ILL and AstraZeneca is investigating the structural characteristics of proteins relevant to the development of breast cancer.

Breast cancer is the most common form of cancer in women, accounting for roughly one in seven female cancer deaths worldwide. The condition can develop in many different ways but it begins when cells in the breast begin to divide and grow in an abnormal way.

Survival rates after diagnosis are improving, however, thanks to earlier detection, increased understanding of the underlying biology and better treatments.

Pharmaceutical firm AstraZeneca has pioneered life-saving breast cancer treatments for the past 50 years, and increasingly is undertaking research into novel hormonal and targeted agents. The firm is a long-standing client of the ESRF's macromolecular crystallography beamlines and, as part of its long-term development of new breast cancer treatments, has recently partnered with the ESRF and Institut Laue-Langevin (ILL) to work with a placement student for one year.



Katherine Moynihan, pictured working in the life sciences group at the ILL, used the ESRF to study the histone-reading protein TRIM24.

Purifying proteins

Katherine Moynihan, an undergraduate biochemistry student at the University of Bath in the UK, arrived on the EPN Campus in September 2014. She has been studying a protein called TRIM24, which is a histone reader involved in the regulation of gene expression. "TRIM24 is known to stabilise the interaction between an oestrogen receptor and chromatin, which leads to up-regulation of the oestrogen response elements that promote cellular proliferation," she explains. "Over-expression of TRIM24 has subsequently been linked to decreased survival in breast cancer patients."

Knowing that TRIM24 is involved in tumour growth in breast cancer, the interaction between TRIM24 and whole nucleosomes presents a potential drug target. Katherine's project therefore involves crystallographic and solution scattering studies of TRIM24 both in the native state and in complex with the nucleosome.

Specifically she has been working on a high-resolution crystal structure of the protein, using small-angle X-ray and neutron scattering experiments to study the nature of the interactions between whole reconstituted nucleosomes and different forms of TRIM24. The ILL was vital for producing deuterated TRIM24, while the team used the ESRF's MASSIF beamlime to obtain crystal data.

"We managed to grow crystals at conditions very close to those published and have diffraction data from MASSIF at 1.7 Å from the first crystals that I am currently refining," says Katherine. "The main issue has been difficulty in getting TRIM24 to bind to a nucleosome, so I've spent most of my project purifying proteins!" The experience of working in a real-life research environment has taught her much more than she has learned in university teaching labs, she adds. "It's very self directed and there is a lot of freedom. Before coming here I thought that a research career was probably not for me, but now I'm not so sure."

Industry collaboration

The ESRF and ILL are keen to establish collaborations with industry, both as partnerships and for proprietary services. "This project with AstraZeneca, in addition to training a young future researcher, shows off the complementarity of the ESRF's X-rays with the ILL's neutron techniques, which is increasingly important for providing a full view of bio-machines for applications in the biotechnology and pharmaceutical sectors," says Ed Mitchell, who heads the ESRF's Business Development Office. "Projects like this are benefitting massively from the ESRF's fully automated protein crystallography beamlines, and we are keen to get our teeth into more projects with industry partners."

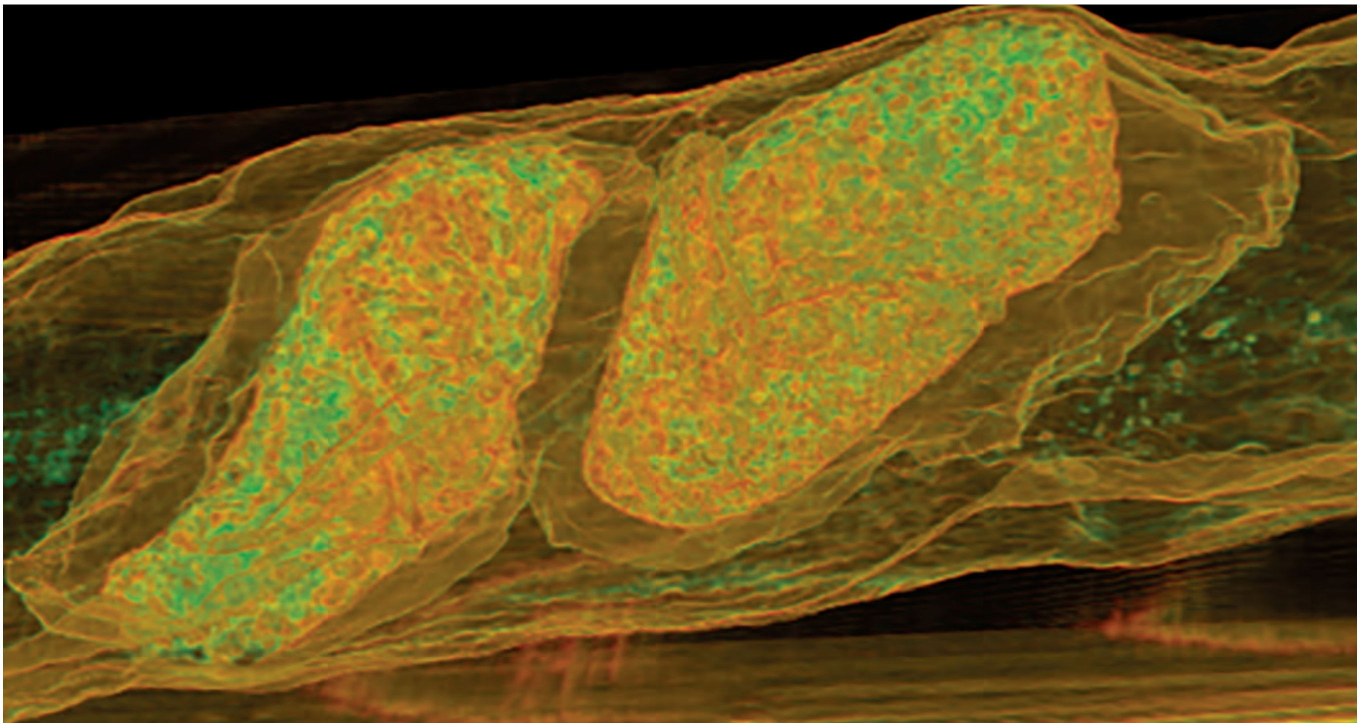
Matthew Chalmers

Movers and shakers

John Helliwell, emeritus professor of chemistry at the University of Manchester in the UK, has won the Max Perutz Prize of the European Crystallographic Association for "his long, generous and fruitful dedication to developing all aspects of the use of synchrotron radiation for crystallography and for his boosting support to global development of synchrotron and neutron facilities".

Helliwell, who was a key figure in the development of the ESRF, has been chair of the macromolecular crystallography working group and also chair of the ESRF's Science Advisory Committee. "The ESRF has always been a world-leading initiative and delivers, through all Europe's synchrotron radiation scientists working as a team, the most outstanding performances possible," said Helliwell, who will receive the prize at the 29th European Crystallographic Meeting in Rovinj, Croatia, on 23–28 August.

Des McMorrow was appointed Chair of the ESRF Science Advisory Committee (SAC) on 1 January 2015 for a period of three years, replacing Keijo Hämäläinen. McMorrow, who is a professor of physics at University College London and currently director of the London Centre for Nanotechnology, has spent his research career exploiting X-ray and neutron facilities to probe the structural and magnetic correlations that govern how electrons organise themselves in solids. He is co-author of the textbook *Elements of modern X-ray physics* and has a long-standing relationship with the ESRF. Having been a user of the ILL while a PhD student in the late 1980s, he started using the ESRF shortly after it opened, and has undertaken experiments on several beamlines. McMorrow, who was first appointed to the SAC as vice-chair in 2012, has also served on various ESRF beamtime allocation and beamline review panels and remains an active ESRF user.



Nanoparticle uptake in worms: This image shows a 3D rendering of the tiny roundworm *Caenorhabditis elegans*, which was exposed to cobalt nanoparticles in soil and then fixed in a quartz capillary. *C. elegans* is a model organism for exposure experiments because it has a short lifecycle and, being transparent, allows high-resolution imaging of its internal organs. The two distinct objects visible in the centre of the image are embryos in the uterus of a preserved adult hermaphrodite *C. elegans*, behind which abundant cobalt nanoparticles (green dots) can be seen in the creature's intestine. The image was created at the ESRF's nano-imaging beamline ID16A using phase contrast absorption X-ray nanotomography with a beam size of 20 x 37 nm, while the presence of cobalt was confirmed using X-ray fluorescence tomography. The study was carried out by users from the Norwegian University of Life Sciences (CERAD) and the University of Antwerp (Dag Anders Brede *et al.*, in prep).

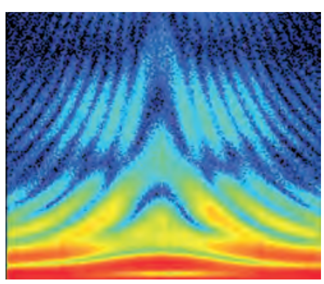
In the corridors

ESRF hosts African light source event

The ESRF will host the first African Light Source conference and workshop in Grenoble on 16–20 November in conjunction with the South African Institute of Physics, marking the first concrete steps towards the creation of a third-generation synchrotron on the continent. The event, which is open to all synchrotron users and "friends of Africa who support the vision for an African light source", will review the status of the African user base at international light sources and discuss a light-source roadmap for Africa – which is currently the only habitable continent without a dedicated synchrotron. South Africa has been involved with the ESRF since the beginning of user operation in 1994, and became a full member in 2013. Proponents of the African Light Source are keen to adopt the model of SESAME in Jordan, which presently is the only other international synchrotron facility apart from the ESRF. Details about how to get involved can be found at www.saip.org.za/AfLS2015.

Study puts the brakes on X-rays

Like all electromagnetic radiation, X-rays propagate at the speed of light – unless they travel through a dispersive medium. In the visible region, such nonlinear mediums can reduce the speed of electromagnetic waves to walking pace and even bring light to a complete standstill, but such "slow light" effects are much harder to achieve for X-rays. Kilian Heeg of the Max Planck Institute for Nuclear Physics in Heidelberg, Germany, and colleagues have now used the ESRF's nuclear resonance beamline ID18 to pass X-rays through a special cavity filled with layers of iron nuclei, observing delays of up to 35 ns in the outgoing beam that correspond to a factor 10,000 decrease in photon speed (*Phys. Rev. Lett.* **114** 203601). "These advances should pave the way for nonlinear optics and even quantum optics



in the X-ray regime," wrote Robin Kaiser of the University of Nice Sophia Antipolis in an accompanying Viewpoint, adding that the ID18 scheme "is an excellent candidate to explore the X-ray frontier".

Changes to ESRF computer network

The ESRF's open Wi-Fi network "ESRF_GuestLAN" has been shut down and replaced by a new network called EPN_Visitors, for use by all visitors to the ESRF, ILL, EMBL and IBS. The new network complies with current French legislation stipulating that any Internet activity must be attributable to an individual user, and is strongly linked to the EPN site entrance application: each time a visit is created, a PDF file containing a user name and password will be sent to the visitor by e-mail. It is also possible to disable Wi-Fi when creating a visit.

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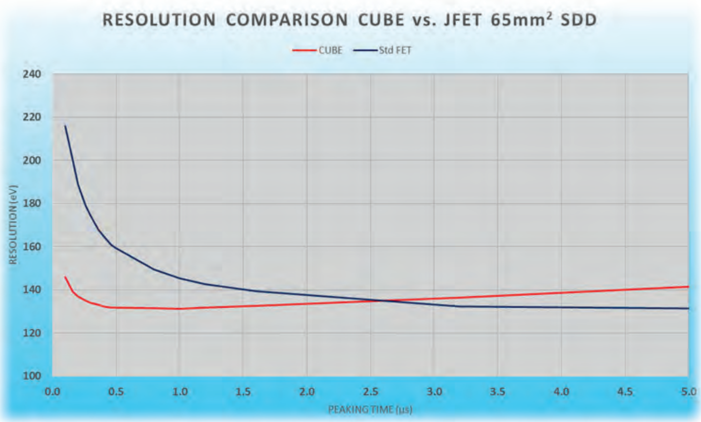

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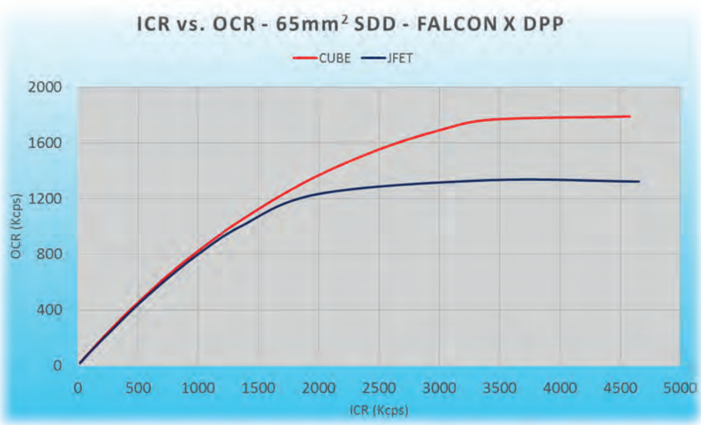

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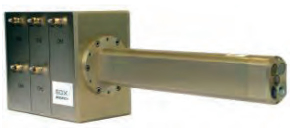
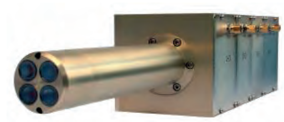
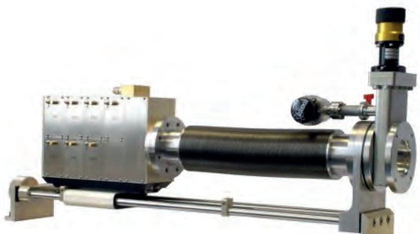




(4, 5 element focussed)



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