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EDITORIAL
5 International Year of Crystallography 2014

IN BRIEF
6 Science Building inaugurated
6 10,000th protein solved
6 Users’ corner
7 Supervolcano secrets
7 Glass phase cracked
7 Fossil fish sheds light on the face

REPORTS FROM THE USERS’ MEETING
8 Reports from the plenary session
10 Workshops target Phase II science

UPGRADE NEWS
13 Powder diffraction moves to ID22

FOCUS ON: YEAR OF CRYSTALLOGRAPHY
14 A century of X-ray crystallography
16 The genius of the Braggs
18 IUCr president looks to the future

FEATURE
21 Copper clue to Parkinson’s disease

PORTRAIT
23 Bill Stirling: new ILL director

INDUSTRY
24 Bright times for Dutch–Belgian industry

BEAUTY OF SCIENCE
26 Toothpick reveals its structure

IN THE CORRIDORS
26 Digital X-ray lines
26 100 years of flight
26 Eyeing up CERN’s future
26 Grenoble on camera

On the cover: A diffraction pattern from a crystal of synthetic DNA (credit G Leonard/ESRF).
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Celebrating crystallography

It is a little over a century since the birth of modern crystallography. In 1912, Max von Laue directed an experiment showing that X-rays are diffracted by crystals, followed shortly afterwards by the work of William and Lawrence Bragg (father and son) who showed that X-ray diffraction can be used to accurately determine the positions of atoms within a crystal (see pp16–17). The significance of these achievements was realised immediately: von Laue received the Nobel Prize for Physics in 1914 and the Braggs the year after. To mark these seminal experiments, which have led to a revolution in our understanding of matter, the General Assembly of the United Nations adopted 2014 as the International Year of Crystallography (IYCr).

Crystallographic research primarily provides information about the structure of a sample, which is intimately linked to the properties and functionalities of materials and molecules at all dimensions. The impact of crystallography can therefore be felt everywhere in our daily lives. It underpins the development of new materials ranging from toothpaste to aircraft components, for example, and is increasingly vital for drug development and nano- and biotechnology.

Today, crystallography encompasses studies of both crystalline and non-crystalline materials, and the technique permeates structural science at the molecular level across physics, biology, chemistry, mineralogy and the geosciences. It is also proving a valuable tool for understanding and preserving cultural-heritage artefacts. One of the aims of the IYCr is to strengthen the links between crystallography and the other sciences.

Synchrotron impact

The highly stable X-ray beams produced by storage rings, which are some 12 orders of magnitude more intense than those produced by conventional sources, have revolutionised crystallographic research and opened numerous new research avenues. Well established methods such as powder diffraction have undergone a renaissance with the availability of synchrotron radiation, while new techniques such as time-resolved studies of chemical reactions also became possible. Furthermore, the exponential growth of known protein structures that has opened up the field of structural biology would not have been possible without the rapid data-collection times offered by synchrotrons. It should also be noted that a facility such as the ESRF does not only provide scientists with X-rays, but also with advanced optics and sample environments that allow researchers to study matter under a vast range of conditions – including industry-relevant settings that have widened and advanced crystallographic research.

The significance of synchrotron-based crystallography is also clearly evidenced by the award of Nobel prizes in 2003, 2006, 2009 and 2012 for breakthroughs obtained using synchrotron radiation and crystallographic methods. Like several other major photon laboratories, the ESRF is planning a machine upgrade that will deliver ever brighter and more coherent X-ray beams. These facilities will be joined by X-ray free-electron lasers that produce even brighter pulsed radiation, further enhancing the links between crystallography and synchrotron radiation and ensuring that X-ray crystallography is only going to become more pervasive in the coming decades.

Although the experiments carried out at the ESRF’s many different beamlines might seem diverse and without a common foundation, most are associated with one of the many scientific commissions of the International Union of Crystallography, which is co-sponsor of the IYCr (see pp18–19). The rise of synchrotrons during the past two decades has led small-angle X-ray scattering and X-ray spectroscopy to become part of the union’s commissions, which bring coherence to the crystallographic community. The IYCr is the perfect opportunity to boost awareness of crystallography and to showcase the enormous impact of synchrotron radiation in this field.

Sine Larsen, University of Copenhagen, IUCr President 2008–2011 and former ESRF research director
10,000th protein structure solved

In February 2014, the number of crystal structures of biological macromolecules that have been solved from diffraction data collected at the ESRF passed 10,000. These structures play an essential role in understanding fundamental biology and support a wide range of applied research, particularly in the development of pharmaceuticals. The 10,000th structure, solved by EMBL Grenoble scientists, was a cap-binding domain of the tickborne Orthomyxovirus virus polymerase, which is a promising drug target (pictured).

When the first atomic co-ordinates based on ESRF diffraction data were deposited in the Protein Data Bank in 1996, solving a protein structure was a long and laborious process. However, the construction of dedicated beamlines coupled with continuous improvements in beam properties and automation has allowed the number of structures solved to increase almost exponentially.

Now numbering 10,000, these structures represent a staggering 10% of all such structures ever solved. The tally includes some outstanding achievements, such as the structure of plant photosystem L, which is the source of most molecular oxygen in our atmosphere, and the structure of the ribosome, that details how proteins are produced from our genetic information.

A particular recent highlight has been the determination of the structures of G-protein coupled receptors, which contribute to numerous signalling pathways and are the target of around 30% of all pharmaceuticals. The information that these crystal structures provide is already contributing to a new generation of more effective drugs. However, great these achievements are, they should not outshine the large number of structures that contribute enormously to the broader research goal of understanding the structure of proteins and how these translate into biological mechanisms.

Just under half of the ESRF’s 10,000 crystal structures were solved using the ID14 beamlines that closed at the end of 2013 and are to be replaced with new beamlines on ID30 that are expected to come online this year. This will allow the number of structures solved to continue to increase and enable ESRF users to tackle the increasing complexity of the biological systems being studied.

Text contributed by Matthew Bowler, EMBL Grenoble and the ESRF

Science Building inaugurated

A brand new facility shared by the ESRF and ILL was inaugurated on 21 February in the presence of French Minister for Higher Education and Research Geneviève Fioraso and local and regional dignitaries. The 5000 m² building, construction of which was funded by France’s Contrat de Projets État-Région (CPER), will house new joint laboratories and scientific partnerships, notably for soft condensed matter, and provide a platform for industrial R&D teams to make use of unrivalled analysis tools. The E41.1 m CPER project also includes the new Institute of Structural Biology building, a new site entrance and refurbishments to the common restaurant.

Users’ corner

Deadlines for the next Beam Time Allocation Panel meeting to review proposals submitted for 15 January (long-term projects) and 1 March (standard proposals) are 24 and 25 April, respectively. The next deadline for standard proposal submission is 1 September.

Users are reminded to ensure that all new publications resulting from data collected either entirely or partially at ESRF are registered in our database via our quick and easy-to-use interface: www.esrf.fr/UsersAndScience/Publications/publication-notification-form.

The 24th ESRF Users’ Meeting & Associated Workshops took place on 3–5 February 2014. More can be read about this in the dedicated articles on pp8–11.

News from the beamlines

ID03 has added an electrochemical flow cell to its impressive collection of in situ experimental setups, allowing electrode surfaces to be studied in their native environment – an aqueous electrolyte. The setup allows the rapid change of up to eight different electrolytes and precisely controlled potentials. So far, it has been trialled in in situ structural studies of bimetallic fuel cell catalysts, palladium single-crystals during electrochemical oxidation, silicon-based lithium-ion batteries, germanium electrodeposition on gold surfaces and other relevant electrochemical systems.

The BM14 team, together with EMBL Grenoble, has developed a new goniometer head type that enables users to expose crystals to X-ray while still in their native crystallisation plate. Such room-temperature experiments are critical for screening the crystallisation nano-hits in order to design experimental protocols to optimise crystal production. In parallel, the team has developed a new sample environment device, called REX, with local company ARINAX. The nozzle changer device, which enables users to remotely swap the crystal environment on the goniometer from a controlled humid air stream at 300K to a cryogenic gas stream during dehydration experiments at 100K, constitutes a major improvement for users who typically have had to carry out this procedure manually.

ID19 continues its ambitious refurbishment programme. A new multimodal monochromator has been installed, offering for the first time a double bendable Laue silicon monochromator with an energy range of 45–200 keV, and will be available for experiments by September. In parallel, a monochromator that uses Bragg reflection on large silicon crystals will be commissioned, offering an energy range of 10–150 keV. Finally, a double multilayer is under development while the existing single-bounce multilayer remains in operation. Users are encouraged to contact ID19 staff before submitting proposals that require the monochromator.

A new high-speed pixel array detector, the Dectris Pilatus3 2M, and a fast translation table have been installed on the MX microfocus beamline ID23-2. The setup has been commissioned and demonstrated data collection at frame rates of up to 200 Hz. EDNA/BEST data collection strategies have been modified for the new detector, which has been available for user experiments since the end of January.

The high-resolution powder diffraction beamline ID31 closed in December 2013 and is being moved to ID22 (see p13). It will re-enter service in April with an energy range extended to at least 80 keV. ID22 will recommence operation in standard high-resolution powder diffraction mode with the current ID31 diffractometer. Later in the year a new powder diffractometer will be delivered, while in early 2015 a large 2D medical-imaging detector will also become available to complement high-resolution experiments.
**Predicting the next supervolcano**

Apart from a giant meteorite, the biggest natural catastrophe facing Earth’s inhabitants is a supervolcano. These vast pools of magma eject thousands of times more debris than ordinary volcanoes, although they are much less frequent. An event 600,000 years ago in Wyoming, US, sent more than 1000 km$^3$ of ash and lava into the atmosphere, producing major changes in global climate and creating a giant crater in the middle of what today is Yellowstone National Park.

In order to better understand when the Yellowstone and other supervolcanoes will erupt, an international team has used the ESRF to reproduce the conditions inside the magma chamber of a supervolcano. Speck-sized rock samples were compressed to pressures of up to 360 GPa between the tips of two tungsten carbide anvils at ID27 and heated to 1700°C with a resistive furnace.

This allowed Wim Malfait of ETH Zurich, Switzerland, and co-workers to accurately probe the state of the system and the change in density when magma crystallises into rock. Results show that supervolcano eruptions may occur spontaneously, driven only by magma pressure without the need for an external trigger. The processes inside a supervolcano are different from those in conventional volcanoes. Not only is its magma chamber much larger, it is plastic: its shape changes as a function of the pressure as it gradually fills up, allowing the pressure to dissipate more efficiently and also ensuring that supervolcanoes do not erupt very often. The new study shows that the pressure resulting from the differences in density between solid and liquid magma is sufficient in itself to crack more than 10 km of the Earth’s crust above the magma chamber (Nature Geoscience 7 122).

“The magma penetrating into the cracks will eventually reach the Earth’s surface, even in the absence of water or carbon dioxide in the magma,” says co-author Carmen Sanchez-Valle of ETH Zurich. “As it rises to the surface the magma will expand violently, which is the well known origin of a volcanic explosion.”

**Facing up to our anatomical past**

A 410-million-year-old armoured fish has helped researchers understand the origin of our most important anatomical feature: the face. Microbeam X-ray scans of the fossil creature *Romundina* at the ESRF’s ID19 beamline reveal the individual steps through which the face assembled during the evolutionary transition from jawless to jawed vertebrates — a dramatic transformation that effectively turned the face inside out.

In embryos of jawless vertebrates, blocks of tissue grow forward on either side of the brain, meeting in the midline at the front to create a big upper lip surrounding a single midline “nostril”. In jawed vertebrates, however, the tissue grows out between the left and right nasal sacs, creating two nostrils rather than a single large hole in the middle. This process also makes the front part of the brain longer, which is why our noses are positioned at the front rather than far back between our eyes.

*Romundina* exhibits a mixture of primitive and modern features that make it a valuable intermediate fossil, says the team. It has separate left and right nostrils like we do, but they are located far back behind an upper lip like that of a jawless vertebrate. Images of the skull’s internal structure also reveal that the brain had a short front-end, again similar to that of a jawless animal (DOI: 10.1038/nature12980).

“In effect, *Romundina* has the construction of a jawed vertebrate but the proportions of a jawless one,” says one of the study’s lead authors Per Ahlberg of Uppsala University in Sweden. “This shows us that the organization of the major tissue blocks was the first thing to change, and that the shape of the head caught up afterwards.”

**Glassy mystery is solved at last**

It’s a question that has puzzled physicists for the past 50 years: why do glasses have a higher heat capacity than crystals? Although glasses are disordered and crystals are ordered, they should have the same heat capacity because both have the same number of atoms, the same atomic mass and the same chemical bonding. In the late 1950s, however, scientists found that at temperatures of around 10K glasses need much more heat to warm up than crystals. This has long been attributed to additional states that exist in the spectrum of atomic vibrations at certain frequencies, attributed to additional states that exist in the spectrum of atomic vibrations at certain frequencies, but attempts to explain how disorder causes these anomalies have been unsuccessful.

Using novel X-ray techniques developed on the ESRF’s nuclear resonance (ID18) and inelastic scattering (ID28) beamlines, Aleksandr Chumakov of the ESRF and co-workers found that disorder does not change the heat capacity after all. The team studied silicon dioxide, the main ingredient of window glass, because it has several glassy and crystalline polymorphs with different structures and densities. By comparing the spectra of atomic vibrations, Chumakov and colleagues found that both glassy and crystalline samples have the same number of additional states but in some samples these states are located at lower energies and therefore provide a higher heat capacity (Phys. Rev. Lett. 112 025502). Previous studies, he points out, had compared typical glasses to typical crystals and therefore combined the disorder effect with a density effect.

“It’s really very simple,” explains Chumakov. “Atoms in an ordered state occupy a small volume whereas in a disordered state they occupy a bigger volume. The techniques developed at the ESRF, combined with the very high energy resolution required, were crucial in reaching this conclusion.”
User science takes the stage

Cutting-edge synchrotron experiments spanning medical research, next-generation electronics and molecular self-assembly were the focus of the 2014 ESRF users’ meeting, reports Matthew Chalmers.

Why is a pediatric oncologist giving a talk at the ESRF? That was the question asked by Michael Grotzer of Zurich University in Switzerland as he kicked off the 2014 ESRF users’ meeting on 4 February. “It’s because synchrotrons can do things that we simply cannot!” he continued. Grotzer described a potentially revolutionary cancer treatment called microbeam radiation therapy (MRT) that is currently under development at the ESRF and elsewhere. Although the technique is yet to be applied to human patients, experiments show that multiple, parallel micro-X-ray beams can safely deliver much higher radiation doses to animal tissues than the maximum doses tolerated in standard hospital radiotherapy and radiosurgery.

The conventional way to kill tumours is to use fractionated, low-dose photon-beam radiation therapy over a period of several weeks. However, the technique is not effective against certain tumours – in particular an aggressive childhood cancer called diffuse intrinsic pontine glioma, which occurs in the brain-stem where the body’s most vital functions are controlled. Chemotherapy is also ineffective for this type of tumour, says Grotzer, as are novel drugs that target the affected cells’ signalling pathways.

“Treating diffuse intrinsic pontine glioma of childhood – one of the most devastating of pediatric malignancies, with around 600 cases diagnosed in Europe each year – is an excellent vision for clinical MRT, but one has to carefully discuss epidemiology, rationale and ethical considerations,” he said. “This is a huge, long-term project based on the finding that it is possible to deliver high doses of radiation in arrays of small beams without destroying normal tissue.”

Tests on ducks, mice, rats and rabbits have produced positive results. More recently, adult pigs that were irradiated at the ESRF as piglets were found to be indistinguishable from un-irradiated littersmates, explained Grotzer. “You can see in the histology the signs of the tiny channels from MRT, but the pig behaved absolutely normally, which is extremely important to know,” he said. “Healthy brain appears to be resistant to this type of radiation, even in relatively high doses.” The next step, he said, is to test the technique on naturally occurring tumours in larger animals such as cats and dogs. The first such test was carried out at the ESRF in November 2013 using a cat that was suffering from an irremovable malignant nasal tumour. “Pets are a vastly underused resource for research because many people would try anything for their pets to live, plus there are strong similarities between pet and human cancers,” explained Grotzer. MRT was first developed by researchers at Brookhaven National Laboratories in the US, and has been a focus of the ESRF’s ID17 biomedical beamline since 1992 with collaborators from Switzerland and other European countries. There is now a vast body of knowledge concerning the effects of different configurations of beam spacing, dose and beam orientation, and major progress has also been made in the technical preparation of clinical trials, said Grotzer. He cautions that human trials are some time away but insists that the technique is possible, pointing to the example of proton-beam therapy – which is now applied at several facilities around the world to target tumours that are difficult to access. “Proton therapy at the Paul Scherrer Institute (PSI) in Switzerland started out as a science experiment, and, after years of development, we now have patients from all over Europe coming to the PSI,” said Grotzer. “MRT is also a long road, but it is definitely worth investing in.”

New insights into cell repair

A completely different, if more traditional, synchrotron-based approach towards cancer treatment was presented by ESRF user Titia Sixma from the Netherlands Cancer Institute in Amsterdam. Sixma and her team are using techniques including SAXS to understand the interactions between proteins that regulate DNA repair, which are vital for protecting us from skin cancer. When DNA damage occurs due to excess UV irradiation, polymerase gets stuck in its task of copying the DNA and a small regulatory protein called ubiquitin comes to the rescue. Using the ESRF, the Dutch team is investigating this vital signalling process at the atomic level, with combined biophysical techniques recently demonstrating that...
The 19th ESRF Young Scientist Award (YSA) has gone to Mathieu Le Tacon of the Max Planck Institute for Solid State Research in Stuttgart, Germany, for his outstanding work on high-temperature superconductors. Le Tacon, who is 33 years old, beat 27 other candidates to the prestigious €2000 award.

The research that marked him out took place over a period of 18 months, during which he co-authored eight papers in high-impact journals (see Nature Physics 10 S2). It began with the discovery, along with Giacomo Ghiringhelli and Lucio Braicovich from the Politecnico di Milano, of a new electronic phase in “cuprate” superconductors.

Cuprates were discovered in 1986 and caused great excitement because they become superconducting at temperatures some 100° higher than conventional superconductors. But unlike conventional superconductivity, in which the electron–phonon interaction causes electrons to pair up and condense, the mechanism of high-temperature superconductivity has eluded physicists.

Using resonant X-ray scattering at the ESRF’s ID08 beamline and elsewhere, Le Tacon and co-workers identified the new cuprate phase as an incipient charge-density wave. In most compounds this phenomenon arises from electron–phonon interactions, but understanding its origin in the more complex cuprates represents a major challenge. Adopting an approach developed by Alexei Bosak and Michael Krisch at ID28, Le Tacon and colleagues first made a “diffuse scattering” map of the crystal (see figure), which told them precisely where they should employ inelastic scattering to study the underlying phonon activity.

ubiquitin is flexible relative to its target protein. Sixma and co-workers are also investigating how ubiquitin is removed from its targets by enzymes, and recently used the ESRF’s ID14 beamline to reveal the elongated structure of the “USP7 C-terminal” domain. “The SAXS data showed that these domains bend back to the catalytic domain, affecting the enzyme’s conformation and catalytic activity as shown by mutagenesis and binding data,” explained Sixma. “We propose that the protein exists in a two-stage equilibrium between an active and an inactive state, which opens perspectives for finding drugs that target the allosteric activation of this important regulator.”

Core science drivers

Human health is one of the key science drivers of the ESRF Upgrade Programme, as are energy, the environment and new materials and nanotechnology. Discussions at the 2014 users’ meeting spanned all these areas.

Mathieu Le Tacon (front) and Alexei Bosak mount a sample at ID28 and, below, diffuse scattering from a YBCO-type high-temperature superconductor showing where electron–phonon coupling takes place.

Moshe Deutsch of Bar-Ilan University in Israel explained how atomic-scale micro-X-ray diffraction studies at the ESRF are revealing the emergence of order at liquid interfaces, for instance in a novel class of ionic liquids that are promising “green” replacements for solvents and other working fluids.

Time-resolved SAXS at the ESRF, meanwhile, is being used to elucidate structural changes in amphiphilic systems in order to control the self-assembly of unilamellar vesicles, which are promising encapsulation and delivery systems, described Michael Gradzielski of Berlin Technical University in Germany.

Participants at the meeting also learned about new applications of ferroelectric tunnel junctions, which exhibit large changes in resistance that are correlated with the direction of the ferroelectric polarization. Agnès Barthélémy of Paris University and co-workers are using the ESRF to study this memristive effect in detail, and claim that it could form the basis of future “neuromorphic” computational architectures.

The quality of the science being carried out at the ESRF is clear from its publication record, with the total number of published papers standing at 23,300. 2013 was also a record year for publications in very highly reputed journals with 292 registered so far, said ESRF Director General Francesco Sette. “The first phase of the Upgrade Programme will make our beamlines on average five times brighter and 10 times faster with beams 100 times smaller – a factor 5000 improvement,” Sette said, adding that there was an aggressive programme taking place worldwide to push storage rings towards much brighter beams.

“The ESRF council has acknowledged that the rapid implementation of ESRF UP Phase II is vital to maintain its leadership in X-ray science, and we are doing everything possible to make this dream machine happen.”
Extreme science forges ahead

The study of matter under extreme pressures and temperatures is a flourishing field of science, and one in which the ESRF is at the forefront. Experiments are routinely performed at pressures up to 100 GPa and span temperatures ranging from 10–5000K, revealing new and often surprising material behaviour. At a workshop held at the ESRF on 3–5 February, 70 researchers working across high-pressure research in Europe and beyond discussed the state of the art in this rapidly evolving field.

It is clear that the ability of third-generation synchrotrons to provide atomic-scale insights is making a major impact in the three key areas of high-pressure research: hard condensed matter, planetary science, chemistry and biology. Currently, the ESRF offers structural investigations on around a dozen beamlines, notably the flagship high-pressure facilities ID09A and ID27, and the EDXAS_S branch of the upgrade beamline ID24. Synchrotron capabilities are also complemented by laser facilities such as the National Ignition Facility at Livermore Laboratory in the US, LULI in France and the X-ray free-electron laser LCLS at Stanford, the latest results from which were also presented at the February workshop.

The focus of discussions was the opportunities on offer from a proposed upgrade of the ESRF storage ring (ESRF UP Phase II). The significantly greater photon flux can be generated only in small volumes or in dynamic, transient processes. This will allow studies of warm dense matter, which is defined as the boundary between condensed-matter and plasma physics, and enable investigations of stellar and planetary interiors.

Static compression made possible by double-stage diamond anvils, or via dynamic compression using lasers with nanosecond pulse durations, will extend the detailed and routine characterisation of solids from the 100 GPa to the TPa pressure range. Furthermore, the increased coherence of the x-ray beam at high energies will enable techniques such as phase contrast-, coherent diffraction- and ptychographic imaging, allowing structures to be resolved at a spatial resolution of a few tens of nanometres and therefore probing nucleation, diffusion of species and phase separation.

Penetrating planets

The Earth’s interior is largely mysterious. We know from measuring seismic waves that it comprises a solid-iron inner core at a temperature above 5000 °C and pressure of more than 3.3 million atmospheres (330 GPa), surrounded by an outer liquid-iron core and a cooler lower and upper mantle. By subjecting tiny samples to extreme conditions for a few microseconds at a time, X-ray techniques at the ESRF allow users to extract much more detailed information.

Last year, ESRF users at ID27 discovered that the Earth’s core is 1000 °C hotter than previously thought by measuring the melting point of iron at different pressures. Since the temperature difference between the mantle and the core drives thermal motion, the result helps account for why the Earth has a magnetic field (Science 346, 164).

In another step towards understanding the Earth’s internal engine, ID18 users last year exploited a highly sensitive Mössbauer technique to obtain electrical conductivity profiles of the Earth’s mantle, providing important complementary data to seismic profiles (Nature Communications 4, 1427).

Meanwhile, in situ X-ray diffraction studies of xenon at ID27 have revealed that this normally inert element exhibits a rich chemistry at high temperatures and pressures, with implications for our understanding of Jupiter and other gas giants (Phys. Rev. Lett. 110, 265501).

And closer to home, ID27 users have recently reproduced the conditions inside the giant magma chamber of a supervolcano, such as the one lying beneath Yellowstone National Park in the US (see p7), and found that supervolcano eruptions may occur spontaneously driven only by magma pressure (Nature Geoscience 7, 122). Matthew Chalmers

Change of phase

The Phase II machine would likely lead to breakthroughs in three main scientific areas: structural complexity at extreme conditions, synthesis and characterisation of novel materials, and planetary science. For example, the combination of high pressure, low temperature and very high pulsed magnetic fields, together with the enhanced brilliance of the new source, will be ideal for probing quantum critical phenomena. These include the suppression and re-emergence of superconductivity, new spin structures of antiferromagnetically coupled sub-lattices and magnetic quantum phase transitions.

Offering measuring times at the nanosecond level, which is compatible with dynamic shock compression using lasers, the upgraded machine will enable systematic structural and spectroscopic studies at pressures in the TPa regime and temperatures above 10,000 K. The outstanding characteristics of the proposed new source will allow us to determine the phase diagram and melting line of iron alloys, for instance, which are important for a detailed understanding of Earth’s inner core (see box).

It will also probe the melting, metallisation and dissociation of silicate compounds in previously unchartered pressure–temperature conditions. These are not only relevant to the complex dynamics of the Earth’s mantle, but extend the ESRF’s high-pressure studies to other planets including those lying outside our solar system.

Michael Krisch, Mohamed Mezouar and Sakura Pascarelli, ESRF
Functional materials challenge Moore’s law

For decades, electronic device engineers have sought to reduce the size of the basic building blocks of microelectronics devices—a trend famously quantified by Moore’s law. However, with the physical limits of miniaturisation now in reach and semiconductor processing costs rising dramatically, a parallel evolution in device engineering is taking place. This “more than Moore” approach is based on greater 3D chip integration and the inclusion of materials with increased functionalities, which demands full knowledge of the interaction between different materials. The magnetic properties of a functional layer, for instance, can be influenced by boundary conditions imposed by the silicon environment and by the elastic strain induced during integration into a chip.

Synchrotron techniques including spectroscopy, resonant inelastic X-ray scattering, X-ray magnetic circular dichroism, and many variants of diffraction imaging are proving increasingly valuable for developing next-generation microelectronics. This was the focus of discussions at a workshop on functional materials for electronics held at the ESRF on 3–5 February, which was attended by more than 80 expert users, beamline scientists and device engineers working in fields ranging from graphene to multiferroics, and from photovoltaics to semiconductors for high-power applications.

By combining structural imaging and spectro-microscopic techniques, researchers can analyse systems in which multiple materials and device technologies are integrated into one chip. For example, ESRF users have recently used techniques at five different beamlines, including microbeam fluorescence mapping and scanning diffraction mapping, to study the variation in efficiency of photovoltaic cells that have undergone the same production processes.

Across Europe, research and development in microelectronics engineering is orienting away from Moore’s law towards ever more complex chip integration. For existing production facilities, synchrotron techniques therefore potentially offer greater returns on investment and open new fields of innovation. Indeed, a number of electronics and semiconductor firms are beginning to access the ESRF directly to solve specific device-engineering problems.

Once Phase II of the ESRF Upgrade is in operation, it will deliver much brighter and more coherent X-ray beams that will further increase the resolution and sample throughput of diffraction-imaging methods. By combining these methods with spectroscopy on a single instrument, for example, researchers can better understand also the ageing processes in devices and yield information about device reliability. In a world where such devices will play a role in areas such as human health or self-driving individual transportation, synchrotron techniques are set to play a vital role.

Tobias Schülli, scientist in charge at ID01

Structural biology: past, present and future

Some 100 participants attended a joint ESRF–EMBL workshop on 3–5 February devoted to the past, present and future of structural biology at the ESRF. Coinciding with the 10,000th deposit in the Protein Data Bank based on ESRF data (see p6), the event began fittingly with a celebration of ID14—one of the most productive beamlines in the world for structural biology. ID14, which was closed late last year and will be replaced by a new suite of beamlines being built during Phase I of the ESRF Upgrade Programme, had operated for 15 years and delivered 130,000 hours of beam time for external user experiments.

The ESRF’s unrivalled structural-biology output owes much to its long record in experiment automation. The three end-stations of the new MASSIF upgrade suite will provide an ideal environment for high-throughput experiments, strengthening links between structural studies and rational drug design, while the new data-collection interface MxCuBE2 streamlines experiment design and execution. The ESRF also provides users (via the ISPyB interface) with real-time results of data processing and analysis, and recent progress in this area will allow even more efficient use of the ESRF’s structural biology facilities.

Although the ESRF is still putting the finishing touches to Phase I of the upgrade, a strong theme of the workshop concerned the potential for structural biology research at a much brighter storage ring (ESRF UP Phase II). It is clear that a supercharged source would make possible the construction of beamlines with flux densities at the sample position five orders of magnitude greater than now, which will give rise to new types of experiments such as serial crystallography. Using this technique, which was inspired by recent successes at XFELs, users can build complete datasets from single-shot diffraction images taken from hundreds or thousands of individual microcrystals.

A brighter source will also allow the study of protein dynamics and lead to advanced data-collection protocols that take into account information contained in previously collected partial datasets.

Should ESRF UP Phase II go ahead, it would shape the study of structural biology at synchrotrons for decades. It would allow us, for example, to determine the structures of membrane proteins and other targets that produce only very small crystals, and allow users to combine information about protein dynamics to study the action of biological macromolecules. This latter aspect could provide important information for drug-design protocols, including those aimed at agents to combat cardiovascular conditions and neurodegenerative diseases associated with aging populations.

Gordon Leonard, Christoph Müller-Dieckmann (ESRF); Andrew McCarthy, Adam Round (EMBL Grenoble)
Powder diffraction reborn at ID22

In user service since 2002, the ESRF’s ID31 beamline has established itself as one of the world’s best facilities for powder diffraction. Thanks to the extension of the experimental hall as part of the ESRF Upgrade Programme, the beamline closed on 18 December to make way for a long, hard-energy nanofocus beamline. Powder diffraction is now being transferred to a new home at ID22, where it will benefit from substantial refurbishment.

Powder diffraction is one of the oldest and simplest X-ray techniques, yet also one of the most versatile and widespread. It begins with a powdered or polycrystalline specimen that consists of a large number of tiny, randomly orientated crystallites. When irradiated by X-rays (or, for that matter, neutrons or electrons) some of the crystallites will be favourably orientated such that they satisfy the Bragg condition, causing X-rays to be diffracted towards the detector.

Since each pure substance produces a distinct diffraction signature depending on its underlying atomic arrangement, powder diffraction can be used to identify and determine the abundance of certain compounds and mixtures in a sample. This makes powder X-ray diffraction highly attractive for qualitative and quantitative analysis, such as ensuring quality control in pharmaceutical production or phase identification in mineralogy. Indeed, a powder diffraction instrument is currently operating on the surface of Mars, having been fitted to NASA’s Curiosity rover to analyse minerals.

Powerful technique
Modern powder diffraction has advanced beyond this analytical approach. Thanks to synchrotrons, we can gain a deeper understanding of the physical and chemical factors that control a material’s properties. At the ESRF, excellent signal to noise ratios, accurate peak positions and narrow diffraction peaks allow users to characterise complex crystal structures, particularly for substances for which a suitable crystal cannot be obtained. Advanced sample environments, meanwhile, allow users to follow structural responses to changes in temperature or pressure, or to follow the influence of gas adsorption, chemical and electrochemical reactions, and electric or magnetic fields.

The importance of powder diffraction for materials research has led many synchrotron facilities to operate dedicated beamlines optimised for different applications. Several of the ESRF’s “CRG” beamlines, where X-rays are produced by a bending-magnet rather than an undulator, have powder diffraction capabilities – as do the ESRF’s high-pressure beamlines ID27 and ID09A.

The ESRF’s high-resolution powder diffraction beamline was originally built on a bending-magnet port (BM16), where it entered service in May 1996. When the beamline was moved to ID31 in March 2002, the X-ray intensity increased greatly thanks to the use of three ex-vacuum undulators, which also enhance the angular resolution and broaden the energy range of the beam. The move to ID22 will bring further benefits for powder-diffraction users at the ESRF. For one, the change to a high-β (even-numbered) section of the storage ring as opposed to a low-β (odd-numbered) section, results in a smaller horizontal beam divergence, increased flux on the sample and narrower diffraction peaks at low scattering angles (the value of β, which relates the beam size, emittance and divergence, alternates around the storage ring due to different focusing schemes). The most significant change to the new beamline is the provision of an in-vacuum undulator. This will provide greater flux at hard energies – particularly in the 31–40 keV range as exploited on ID31 for routine operation – and reinforce the beamline’s role for hard-energy, high-resolution powder diffraction. It will also be possible to extend the operational range up to 80 keV, allowing access to the absorption edges of heavy elements that were inaccessible for anomalous-scattering studies on ID31.

Open for business
The new beamline is scheduled to begin operations in April. Initially, the ID31 diffractometer will be installed, coupled with new beamline optics, but a new diffractometer offering a higher load-carrying capacity and faster scanning speed will be delivered later in 2014 in preparation for the future availability of improved hard-energy pixel detectors. Also planned is a large 2D medical-imaging detector and focussing via a translocator, allowing faster data collection. This will especially impact experiments that employ the “pair distribution function method”, whereby data collected at large diffraction angles and at hard energy can be used to characterise materials with poor crystallinity. Additionally, a strain diffractometer will be installed to allow users to map residual strain in engineering components. Strain mapping was already a key part of the scientific programme of ID31, with photon energies of up to 63 keV allowing users to study aerospace components made from lighter aluminium- and titanium-based alloys.

ID31 is the only high-resolution powder diffraction beamline in the world built on a dedicated undulator source that allows routine operation at energies above 30 keV, yet maintains complete wavelength tunability from soft to hard X-ray energies. The upgrade will bring increased X-ray intensity and improved resolution which, combined with beamlines in the Structure of Materials group and several CRGs, will keep the ESRF at the forefront of materials research.

Andy Fitch, scientist in charge at the ESRF’s high-resolution powder diffraction beamline
The rise of X-ray crystallography

Synchrotrons and free-electron lasers are shaping the next century of crystallography.

1611 Kepler notes six-fold symmetry and structure of snowflakes
1723 Capeller publishes his Introduction to Crystallography
1815 Hauy introduces translational symmetry
1895 Röntgen discovers X-rays Nobel, 1901

1912 Max von Laue shows X-rays diffracted, proving their wave-like nature Nobel, 1914
1912 William Lawrence Bragg finds formula linking diffraction pattern to crystal structure (also 1913)
1913 William Lawrence and William Henry Bragg solve structure of salt and diamond Nobel, 1915

1916 Debye and Scherrer invent powder analysis

1922 Davisson and Thomson discover diffraction of electrons by crystals Nobel, 1937
1924 Bernal solves the structure of graphite
1929 Lonsdale proves planar structure of benzene

1946 Sumner receives Nobel for showing that enzymes can be crystallised
1946 Hodgkin solves penicillin, one of several biochemicals solved during her career Nobel, 1964
1947 First observation of synchrotron radiation (General Electric)

1952 First synchrotron beamline, Cornell
1953 Crick, Watson and Wilkins decipher structure of DNA Nobel, 1962
1957 First protein structures solved by Kendrew (myoglobin) and Perutz (haemoglobin) Nobel, 1962

The rise of X-ray crystallography

Synchrotrons and free-electron lasers are shaping the next century of crystallography.

1947 First observation of synchrotron radiation (General Electric)
1953 Crick, Watson and Wilkins decipher structure of DNA, Nobel, 1962
1957 First protein structures solved by Kendrew (myoglobin) and Perutz (haemoglobin), Nobel, 1962
1981 SRS in the UK becomes first dedicated X-ray storage ring
1984 Deisenhofer, Huber and Michel solve 3D structure of membrane protein, Nobel, 1988
1985 ESRF, the first third-generation synchrotron, enters operation
1988 Brockhouse and Shull share Nobel for neutron diffraction
1994 Structure of lysozyme solved
1994 Deisenhofer, Huber and Michel solve 3D structure of membrane protein, Nobel, 1988
1994 MacKinnon solves structure and function of ion channels, Nobel, 2003
1997 Boyer and Walker receive Nobel for ATP structure using synchrotron light
1998 MacKinnon solves structure and function of ion channels, Nobel, 2003
1997 Shechtman discovers quasicrystals, Nobel, 2011
1994 Deisenhofer, Huber and Michel solve 3D structure of membrane protein, Nobel, 1988
1997 Boyer and Walker receive Nobel for ATP structure using synchrotron light
2005 First soft-X-ray free electron laser, FLASH, enters operation
2009 Yonath, Steitz and Ramakrishnan win Nobel for ribosome structure using synchrotron X-rays
2009 First hard-X-ray free-electron laser enters operation at SLAC
2005 First soft-X-ray free electron laser, FLASH, enters operation
2012 NASA’s Curiosity rover uses X-ray diffraction to analyse Martian soil
2009 Yonath, Steitz and Ramakrishnan win Nobel for ribosome structure using synchrotron X-rays
2012 NASA’s Curiosity rover uses X-ray diffraction to analyse Martian soil
2009 Yonath, Steitz and Ramakrishnan win Nobel for ribosome structure using synchrotron X-rays
2012 NASA’s Curiosity rover uses X-ray diffraction to analyse Martian soil
2014 Protein Data Bank nears 100,000 deposits
2014 Protein Data Bank nears 100,000 deposits
2016 European XFEL enters operation
>2020 Synchrotrons strive towards X-ray diffraction limit
Focus on: Year of Crystallography

A structural revolution

ESRFnews visits the Cavendish Laboratory in Cambridge UK, where in 1912 William Lawrence Bragg derived the equation that made X-ray crystallography possible.

A walk along a quiet corridor flanked by glass and wooden cabinets in the Bragg building at the Cavendish Laboratory in Cambridge is a trip through some of physics’ greatest hits. There is Maxwell’s desk, J J Thomson’s discharge tube, Rutherford’s alpha-particle detectors, Chadwick’s neutron chamber, Cockcroft and Walton’s rectifier tube, and more. Next to a large metal model of the double helix built by Crick and Watson sits a primitive X-ray spectrometer and a box of crystals (bottom left) which, a century ago, enabled a young graduate student to visualise the atomic structure of matter for the first time.

William Lawrence Bragg (WLB) was just 22 when his famous formula linking the X-ray diffraction pattern from a crystal to its underlying structure was presented at a meeting of the Cambridge Philosophical Society on 11 November 1912. It was the subject of his first paper, and it won him the 1915 Nobel Prize for Physics. To date, he is the youngest recipient of the award and he is also the only person to have shared it solely with a parent: his father, William Henry Bragg (WHB). It is also thanks to WLB that the Cavendish museum exists.

“Bragg realised the historical importance of the apparatus, not just his own, and in 1948 while he was head of the department he collected together and put on display as much of the historical material as had survived,” explains Malcolm Longair, an astrophysicist and former head of the Cavendish Laboratory. “Despite having personally made the crucial breakthrough, WLB believed that his father tended to receive most of the credit for X-ray crystallography.”

History in the making

Born in Australia, and already armed with a physics degree, WLB arrived at Cambridge in 1909 to study natural sciences, the same year his father became a professor at the University of Leeds. A renowned scientist and fellow of the Royal Society, WHB had worked on X-rays since their discovery in 1895, and was studying radioactivity at the time. An experiment in Munich, Germany, in April 1912 was to change the Braggs’ lives. Theorist Max Laue of Munich University, having learned that crystals contain periodic arrays of atoms, directed his colleagues Paul Knipping and Walter Friedrich to test whether X-rays are diffracted by a crystal. It turned out that they were, proving the wave-like nature of X-rays and winning Laue, alone, the 1914 Nobel Prize for Physics. However, incorrect assumptions about the nature of X-rays and crystals had left him unable to account for the precise arrangement of the diffraction spots.

According to the 2008 biography William and Lawrence Bragg, Father and Son by John Jenkin of La Trobe University in Australia, news of X-ray diffraction reached WHB via a letter from Norwegian physicist Lars Vegard dated 26 June 1912. Bragg senior first discussed the contents of the letter with his son during a summer holiday, and the pair set about trying to explain Laue’s diffraction pattern. Much of the work was carried out at Leeds, which was much better equipped experimentally than Cambridge and where WHB had just built the first X-ray spectrometer – the forerunner of the modern diffractometer (see picture, left). WHB was of the opinion that X-rays are particle-like, and thought the spots were the result of particles travelling through channels in the crystal. However, while strolling through Cambridge one day, WLB had the distinctly wave-like insight that X-rays are reflected off successive atomic planes in the crystal. In this picture, a diffraction spot only appears if a certain “Bragg” condition is satisfied: $n\lambda = 2d\sin\theta$, where $n$ is an integer, $\lambda$ is the X-ray wavelength, $d$ is the interatomic spacing and $\theta$ is the angle of incidence. This neat, optics-inspired equation accounted for all of Laue’s diffraction spots, and in particular revealed the first crystal structure – zinc sulphide – to be face-centred cubic, not cubic as Laue’s interpretation had shown.

“It shows a particular sort of mind at work,” says Mike Glazer, a crystallographer at the University of Oxford who is writing the autobiography of WLB and his wife. “In Germany the education system was very formal and mathematical and I think Laue got bogged down with the details, whereas in the UK and Australia there was more focus on visual things. Given also that WLB was a gifted artist, all of this contributed to the simple picture that turned out to be the answer.” But we should not overlook WHB’s role, continues Glazer. “William’s contribution is enormous because he built the ionisation spectrometer which allowed you to

“These two people changed the world.”
measure the intensity of the reflected spots, whereas the Laue method just produced spots on a film."

From 1913 onwards father and son worked closely together, with WHB dispatching a second X-ray spectrometer to the Cavendish for WLB to work with. Their first joint publication, in which WLB's original formula was changed from a cosine to its modern sine form (probably, says Glazer, because it was a more natural convention when using the spectrometer), presented the structure of rock salt and subsequent papers would solve diamond and many other systems. The breakout of World War I, in which both Braggs played active technical roles, brought the work to a premature end and in 1919 WLB moved to Manchester University. For the next 15 years X-ray crystallography at Cambridge was taken up in the department of mineralogy under John Bernal, who later started to apply the technique to biological systems.

Targeting proteins
When WLB returned to Cambridge in 1938 as head of the Cavendish, protein crystallography was all the rage. Max Perutz had just started working on haemoglobin and, although wary of the huge challenges involved, WLB persuaded the UK Medical Research Council (MRC) to sponsor a biology unit within the Cavendish. It was here in 1953 that Crick and Watson uncovered the structure of DNA – work that WLB had at one stage ordered them to stop, although later changing his mind. In 1957 the unit moved into a dedicated building known as the MRC Hut. Then in 1962, the year Nobel prizes were awarded to four of the group's scientists (Crick and Watson – medicine; Kendrew and Perutz – chemistry), the MRC financed a separate Laboratory of Molecular Biology and protein crystallography finally broke away from the physics department (see left). "The support Bragg gave to the biological side was absolutely crucial," says Longair.

WLB left the Cavendish in 1953 to become director of the Royal Institution in London, where he devoted much of his time to outreach activities. Glazer was one of the many schoolchildren who witnessed his public lectures, and later met WLB professionally. He says there is a big difference between the person and the image of WLB. "He was very much an Edwardian character who called everyone by their surnames, and he was very different to his father who tended to keep his feelings bottled up whereas WLB was more emotional and prone to bouts of depression."

Although WLB’s lack of recognition haunted him for much of his life, in truth most people have not heard of either of the Braggs. "These two people have changed our world, yet they have been ignored," continues Glazer. "Laue was a great man but he stumbled. If we didn’t have Bragg's law we wouldn't have modern pharmaceuticals, genetics, electron diffraction, and so much more. And you wouldn’t have your synchrotron!"

Matthew Chalmers

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How did the IUCr come to be?
The biologist Paul Ewald was the driving force behind the IUCr in the mid-1940s, although it was Lawrence Bragg himself who became its first president. Before then, crystallography had just one journal based in Germany, which was shut down during the Second World War. Ewald, who by then had moved to the UK, recognised the need for a politically neutral journal, which demanded an independent organisation. So the IUCr was formed and Acta Crystallographica was launched.

How does the union operate?
We are still modelled around our journals business, and are unique among scientific unions in that we publish nine journals. We launched the ninth, called simply IUCrJ, in January at the opening ceremony of the International Year of Crystallography. It is an open-access journal designed to capture high-impact papers. Apart from around 25 journals and administrative staff based at our headquarters in Chester, UK, we work for free. I see it as an honour to serve the union because the IUCr is the continuing thread for the future of our subject. We don’t have members, we just have 51 countries who adhere to the union. It is directly and actively managed by scientists, which for me is the reason for its success.

What have been the IUCr’s successes?
Aside from our journals, International Tables are an extremely important part of our business. They are serious reference works, since crystallography employs a high level of mathematical and geometric definitions and involves an incredible amount of detail. Within a year of the discovery of quasicrystals in 1982, the IUCr had issued a new definition of the term “crystal”.

How did the International Year of Crystallography arise?
The IUCr mooted the idea with the United Nations, which is the only body that can declare an international year, in late 2011. Morocco was the UN member state that moved the motion, since proposals from smaller countries have more chance of success. The motion was voted in by UN members in July 2012, which was a surprise because it gave us just one year to prepare, and UNESCO was designated the official partner.

What are the goals of the event?
To reach out to grass roots everywhere, from the public to crystallographers in all corners of the globe. The whole business of this year, and indeed many of these international years, is to strengthen ties with countries that might be less prominent scientifically. This is where the discoveries of the future are going to come from, so it makes real sense.

What was the highlight of the opening ceremony?
We succeeded in the main objective: to make laypersons understand what the word “crystal” means – that you can have crystals everywhere, from salt factories to medical supplies. The focus on the BRICS countries was certainly the highlight of the meeting for me. These five states are uniquely poised because they’ve got 50% of the world’s population and, unlike the situation in the past, more people have access to quality education. If there is just one clever child in...
focus on: year of crystallography

19 March 2014

ESRFnews

... every 100 villages in India, it means there are an awful lot of clever people in India. Historically we didn’t have the means to catch them, but this is changing. Remember: you only need one von Laue or one Bragg to change the world.

How do you rate the importance of von Laue and the Braggs’ discovery?

The two discoveries were of the highest intellectual level, but compared to some fundamental discoveries (such as general relativity or quantum mechanics) the discovery of X-ray diffraction might not stand out particularly. In terms of its broader impact on science and society, however, it is unrivalled. It’s like an octopus: its tentacles get everywhere. Seeing where atoms are and what they are doing is incredibly valuable, and nowadays scientists don’t even need to know how the techniques work.

There’s an old joke about a chemist and a crystallographer entering a darkened room: the chemist walks around and when he hits a piece of furniture he knows its location; the crystallographer just switches the light on. We shouldn’t forget also that crystallography existed for many hundreds of years without X-ray diffraction. There are writings in India about the external properties of diamonds dating to 300 BC, and India was the first to develop methods for the crystallisation of salt, while the Chinese did the same for sugar. But it was X-ray diffraction that allowed us to look inside crystals.

How has the union’s role changed?

In the beginning, the crystallographers needed the union, whereas these days the union needs the crystallographers. Scientists are more individualistic and financially independent now, but the IUCr still has a lot to offer. Our triennial congresses are always a big success, and provided we keep our journals up to date and keep them in the public eye, the union will go on. A good scientific paper is always read by somebody, but some scientists these days seem more keen to chase the razzmatazz of a handful of journals and their associated media coverage. Scientists should have the confidence to publish proper full papers to be read by people who know what they are talking about.

How have synchrotrons impacted the activities of the IUCr?

This has been a very big thing for the union because synchrotrons cover all aspects of crystallography and structural science. They are of crucial importance. We are proud to have launched the Journal of Synchrotron Radiation in 1994, which continues to perform very well. It’s a growing market because synchrotrons keep getting bigger, better and more numerous, drastically cutting the time that you need to get a crystal structure. X-ray free electrons are similar in that respect, and both are targets for our new IUCr title.

What will X-ray crystallography look like 50 or 100 years from now?

No scientist worth his salt would make a prediction about the future. Science proceeds by nonlinear jumps. When X-rays were discovered, people thought that they might have medical, but not many other, uses. Nobody thought that they could diffract, yet within a year of that discovery we got Bragg’s law. What we can say is that X-ray crystallography will be used to examine all sorts of things that we don’t even call crystals today, such as matter with lesser degrees of order. And although complex crystals such as human proteins are now hot topics for structural scientists, there is still much to learn about the basics. We don’t even understand fully the chemistry and dynamics of a crystal of table salt!

Will there still be a role for the IUCr?

Science advances through our imagination, artistry and lack of fear. When people many centuries in the future look back upon today, they will see that the field of crystallography is just starting. And as long as there is crystallography, there will be an IUCr. Matthew Chalmers

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Copper clue to Parkinson’s

X-ray fluorescent microscopy at the ESRF suggests copper plays a critical role in the onset of Parkinson’s disease, raising the possibility of novel therapies.

Affecting some five million people worldwide, and more than 0.1% of people over the age of 40, Parkinson’s disease is the most common neurological motor disorder. It is marked by the malfunction or death of brain cells in a region of the mid-brain called the substantia nigra, leaving sufferers with progressive tremors, muscle stiffness and slowness of movement. Symptoms of the condition, which was first identified 200 years ago, can be treated by medication or surgery but there is no cure. Studies have linked the progressive brain-cell death in Parkinson’s disease to altered concentrations of metal ions, trace amounts of which are essential for normal brain function. The details of these neurodegenerative pathways remain unclear, but synchrotron X-ray techniques at the ESRF and elsewhere have recently allowed neuroscientist Kay Double of the University of Sydney, Australia and colleagues to track bio-metal concentrations in the brain with unprecedented sensitivity (Neurobiology Aging 35 858).

Unexpectedly, the team found that levels of copper are significantly reduced in the neurons located in the vulnerable brain regions of Parkinson’s sufferers, suggesting that copper plays an important role in protecting neurons. This, explains lead author Katherine Davies of the University of New South Wales, potentially opens up new possibilities for the development of novel protective therapies. “Why is there so much copper in the substantia nigra of a healthy person’s brain and so little in the brains of Parkinson’s sufferers? There must be a reason,” says Davies.

Single neuron mapping

Parkinson’s disease primarily affects neurons that produce the chemical messenger dopamine. As the condition progresses, the amount of dopamine produced by the surviving brain cells is not sufficient to allow the patient to control motor functions normally. Until now, researchers have been limited to studying whole tissue samples. But the micrometre beam sizes available at the ESRF, combined with elemental mapping via X-ray fluorescent microscopy, allowed the team to measure bio-metal levels inside single neurons. Analysing substantia nigra samples during two visits to the ESRF’s ID22 beamline in 2011 and one visit to the I18 beamline at Diamond Light Source in the UK in 2012, the team found that copper levels in this region of a healthy brain are around two times higher than elsewhere in the brain, suggesting that there is a particular need for copper in this region. “If we can correct this copper deficit we could potentially slow down brain-cell death and thus slow the progression of the disease,” says Double, adding that up to 80% of neurons can have degenerated by the time most patients are diagnosed with Parkinson’s disease.

Therapeutic potential

The researchers spent two years carefully checking their conclusions before they published. “It is well known that substantia nigra iron levels are elevated in Parkinson’s disease but we were claiming that copper is severely reduced, so we had to be really certain of that,” says Davies, who carried out the experiments at ID22 along with Kay Double and physicist Sylvain Bohic of the Grenoble Neurosciences Institute and the ESRF. “We had to find out what could be causing this depletion and whether it has a functional consequence for brain-cell health.” To investigate, the team studied an antioxidant protein in the brain called SOD-1. This key protective enzyme requires small amounts of copper to function normally, but in Parkinson’s disease the activity of SOD-1 was found to be altered in concert with the pattern of cell death. These data, says the team, provided evidence that the change in copper in the brains of Parkinson’s patients enhances the vulnerability of the degenerating brain regions.

“Human brain tissue is extremely precious, and it takes time to measure samples, so we carried out the experiments at multiple sites with a range of samples,” explains Davies. Synchrotrons are very highly regarded for their accuracy and sensitivity and, indeed, were the only way we were able to measure metal levels within individual brain cells.”

Biochemist David Brown of the University of Bath in the UK, who was not involved in the work, says that the results fit with previous studies of bio-metal concentrations in the brain. “This is a valuable technical study demonstrating that you can work at the level of single cells, and it confirms that the surviving tissue in Parkinson’s disease handles copper differently,” Brown told ESRFnews. “It would also be interesting to apply the technique to other metals, especially iron, and to obtain a deeper understanding of the role of the copper transporting protein CTR-1 in the Parkinson’s-disease brain.”

Restoring brain copper levels has been successfully used to treat other neurological disorders where copper levels are reduced, such as Alzheimer’s disease. The crucial next step is therefore to test whether restoring brain copper levels to normal levels will increase survival of brain cells. “We will again be relying on the power of the synchrotron to investigate,” says Double.

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From neutrons to X-rays and back

Bill Stirling, former ESRF Director General, became Director of the Institut Laue—Langevin (ILL) in January. He aims to launch an ambitious ILL upgrade and to strengthen ties with the ESRF.

The ILL is a special facility, says Bill Stirling. Founded by France and Germany in 1967, it was the most powerful neutron source in the world when it entered operation. “The fact that it retains that position more than 40 years later is a testament to the work of those who built it and to the policy of maintaining it through regular upgrades,” says Stirling. “On a personal level the ILL is where I learned the trade and began to understand what it is to be a scientist, so when I was invited to become director I saw it as an opportunity to give something back.”

Stirling arrived at the ILL in 1973, just after the UK had joined the facility, and was a staff scientist there until 1987. He worked on instrumentation, building triple-axis spectrometers that are still in use today, while researching quantum fluids, lattice dynamics and magnetism. It was not until he moved to the University of Keele in the UK that he discovered the value of X-rays. “We were next door to Daresbury and so some colleagues and I developed a particular way of working where we used neutron beams to develop ideas about the magnetic structure and then used finer, element-specific X-ray experiments to see where the magnetism was coming from.” In 1995 he moved to Liverpool University in the UK.

Complementary science
During the 1990s Stirling became a regular user of the ILL and the UK’s ISIS neutron source as well as the ESRF and the NSLS at Brookhaven National Laboratory in the US. Along with his colleague Malcolm Cooper of Warwick University, Stirling also proposed and led the development of the ESRF’s UK-operated XMaS beamline at BM28. Then, in 2001, he became Director General of the ESRF where, during the next few years, he worked with his colleagues to set in motion the ESRF Upgrade Programme.

Pursuing Phase II of the ESRF Upgrade is essential.

“I realised that the ESRF was a wonderful facility, but that if it didn’t do something dramatic it would be caught up by other labs,” he says. “My successor, Francesco Sette, has ensured that we now have a beautiful new experimental hall and new beamlines that will help maintain the ESRF’s position as the only international facility and the best in terms of scientific output.” Pursuing Phase II of the ESRF Upgrade is essential, he says. “Synchrotrons are wonderful tools and it’s absolutely clear that Europe needs to keep up with the best.”

Stirling’s goals as director of the ILL are foremost to complete the facility’s Millennium programme – a series of instrument and infrastructure upgrades that has boosted the average brightness by a factor of almost 25 over the past few years. He is also hoping to start another ambitious upgrade phase next year called Endurance, which will prepare the ILL for the 2020s. The other main task, he says, is to continue with the reinforcements that all reactor operators have had to make as a result of the Fukushima accident in Japan.

“There is an issue with the image of nuclear and we are under continuous scrutiny from authorities,” he says. “But research reactors are vital to so many areas, such as the effects on materials under radiation for civil nuclear power programmes including ITER and the production of medically important isotopes, for which the ILL plans to launch a test programme. People talk about 2030 as an end date for the ILL, but technically there is no reason why it would stop then.”

Good relationship
Stirling is also keen to strengthen the ILL’s links with other institutes, ranging from its neighbours on the Grenoble EPN Science Campus to neutron sources internationally, including ISIS and the proposed European Spallation Source. Since 2009, Stirling has been an advisor to the French technological research organisation CEA working on the GIANT Innovation Campus, which includes eight research and higher-education institutions in Grenoble. Although the relationship between the ILL and the ESRF is good, he says, there are opportunities for much closer collaboration. “We already share lots of things, and this is becoming stronger with the Science Building and joint summer schools, but each institution has its own priorities and we need to see how these can best be met for all.”

Stirling’s decision to enter science was made at an early age, his parents having instilled in him a keen interest in nature. His father was also a physics teacher, and as a child Stirling spent a lot of time “mucking around with electricity and radios”. He counts his gaining a first-class degree in physics, and later winning the Glazebrook medal and becoming a Fellow of the Royal Society of Edinburgh, as career high points. “I’ve always thought of myself as a physicist first and a manager second, so recognition for that aspect was always important,” he says. Matthew Chalmers
Industry and academia double up

The ESRF’s Dutch—Belgian beamline DUBBLE is helping companies carry out R&D in the automotive, catalyst, polymer, food and many other industrial sectors.

The DUBBLE facility at BM26 has two branches specialising in materials science research. A SAXS/WAXS branch caters primarily for soft condensed-matter research, although metallurgists are regular visitors, and an EXAFS branch specialises in online catalysis experiments. Since the beamlines entered operation in 2000 there has been a strong emphasis on building sample environments and infrastructures that allow realistic processing conditions to be mimicked. This reflects the importance of the materials- and chemistry-based industries to the Dutch and Belgian economies, and makes DUBBLE an attractive destination for dozens of scientists in the region who work in these areas.

A natural way for industry to benefit from the beamline’s advanced X-ray techniques is via collaborations with universities and/or public–private networks. This research is non-proprietary and results are published via the conventional scientific channels, but some companies such as Janssen Pharmaceuticals in Belgium and DSM in the Netherlands are also using DUBBLE directly. “One issue for direct industrial involvement in our potential user base is the cost of synchrotron beam time, which is worth paying if one has a pressing problem in a production plant but can be more difficult to justify to obtain background knowledge that might only become relevant 5–10 years down the line,” explains DUBBLE project leader Wim Bras.

Unique opportunities

The Dutch Polymer Institute (DPI) is a major public–private agency in the Netherlands that funds projects ranging from polymer processing to the development of materials for organic photovoltaic cells. Since 2006, the consortium has funded a post-doc based at the ESRF whose task, besides in-house research, is to develop new online techniques and to support DPI’s research groups when they visit the ESRF. Typically between three and five DUBBLE post-docs and students are working on industrially relevant subjects at any one time. “Data generated by DUBBLE have been highly relevant in the research carried out in the boundary domain between academia and industry, and offer unique possibilities for the polymer community,” says Jacques Joosten, who is chairman of the DPI executive board.

A group of DPI industrial partners has recently supported a three-year programme that will see a postdoc and PhD student based at the ESRF. “The SAXS/WAXD facilities at DUBBLE enable us to characterise the organisation of molecules and understand how this process is affected by external stimuli at length scales ranging from Ångstroms to hundreds of nanometres,” says Luigi Balzano of DSM research. “This gives material scientists unique opportunities to see molecules at work and to design new materials tailored for applications based on a bottom-up approach.”

We can design new materials tailored for applications based on a bottom-up approach.”

Cross fertilisation

An interesting cross fertilisation between pure and applied research, continues Bras, is the development of confocal X-ray spectroscopy, which allows 3D chemical mapping of samples. The technique was initially used to study metal concentrations in water organisms around chemically polluted sites, as well as for ageing effects in the glass of church windows. “The group from Utrecht is using the same methodology now looking at real catalysts but also the glass-manufacturing company Corning is carrying out collaborative experiments to improve their understanding of ion-exchange processes, which underpin the very strong glasses used for computer screens and mobile phones,” says Bras.

As many as 30 companies are linked in some way to DUBBLE, and Bras estimates that around half of all Dutch—Belgian beam time has industrially relevant projects in the background. “Since many of the results from experiments carried out in proprietary beam time are also published in the open literature, we have a win-win situation whereby our users benefit from their interactions with industries while the funding agencies can point to excellent research output in the form of PhD theses and publications.”

Matthew Chalmers

A state-of-the-art gas system allows catalysts to be studied under controlled flow, pressure and temperature.

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Matthew Chalmers
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In the corridors

Mobile X-ray lines

UK firm SGX Sensortech has released a free app that delivers X-ray transition energies direct to your smartphone or tablet. The app replaces the firm’s “slide-rule” device, which measured around 20 x 10 cm, and allows users to select any element and display its X-ray transition energies as well as more general properties. Unlike the physical slide-rule version, it also offers a reverse energy lookup service that allows quick checks of contamination levels during an analysis, for example. If a user sees a line that they don’t recognise, they can input the transition energy and the app will automatically display the elements and lines that are closest to this value.

Flight centenary

While Max Laue and the Braggs were busy founding the new science of crystallography a century ago, something else remarkable was taking place on the other side of the Atlantic: the first commercial flight. To mark this milestone, which took place in Tampa Bay, Florida in January 1914, The Guardian newspaper has produced an interactive map that uses live data to track every commercial plane in the air. Among many aviation facts detailed on the site are the number of airline tickets sold in 1914 (roughly 1200) compared to more than three billion per year today. The pioneering Tampa “boatplane” carried one passenger and travelled just a few metres above the water at a speed of 100 km/h (www.theguardian.com/world/ng-interactive/2014/aviation-100-years).

A storage ring too far?

If in the distant future CERN was to follow the path of other particle physics labs and become an X-ray facility, synchrotron users could find themselves with a 100 km-circumference storage ring at their disposal. Although the Geneva lab will be busy operating its 27 km-circumference Large Hadron Collider (blue circle above) for the next 20 years in the search for exotic new elementary particles, it announced in February that it is exploring the feasibility of a 80–100 km-circumference machine that smashes particles together at much higher energies. That’s a lot of potential synchrotron beamlines. But even though the brightness of electron storage rings scales roughly as the cube of the radius, such a giant ring would be overkill for X-ray science. Accelerator physicists in the US are, however, exploring the feasibility of converting the 6.3 km-circumference Tevatron collider at Fermilab into an ultimate (diffraction-limited) X-ray source.

Way to go

The ESRF is the 19th most photographed place in Grenoble, according to the new Google map showing hot places to take photos (www.sightsmap.com).

Toothpick tomography: This tomographic slice of a commercial bamboo-based toothpick, measuring 2 mm across, was imaged at the ESRF’s ID19 beamline using X-ray microtomography in combination with hard X-ray phase-contrast imaging. The high sensitivity of the technique reveals different filling states of the cells, allowing researchers to track water transport in plants. Alexander Rack of the ESRF’s X-ray imaging group produced the image as a feasibility test to compare laboratory phase-contrast computed tomography with synchrotron-based images. “In lab CT you can see the same morphology but it would be more noisy and offer less contrast,” he explains. “Synchrotron-based phase-sensitive X-ray imaging techniques also require a lower radiation dose, which is crucial for studying plants in vivo in order to investigate processes such as embolism repair.” ID19 users have recently applied the imaging technique to young living trees.

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PILATUS 12M-DLS

Customized single-photon-counting X-ray detectors
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PILATUS 12M detector for the Diamond Light Source, UK. With more than 12 million pixels on an active area of 0.34 m², the detector covers a 2θ range of ±100° and will operate in vacuum at 10⁻⁶ mbar. This specific solution, built by DECTRIS in close collaboration with the I23 team at Diamond Light Source, represents a breakthrough in long-wavelength macromolecular crystallography.

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