A LIGHT FOR SCIENCE

ESRF news

Number 63 March 2013

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ESRF

A light for science

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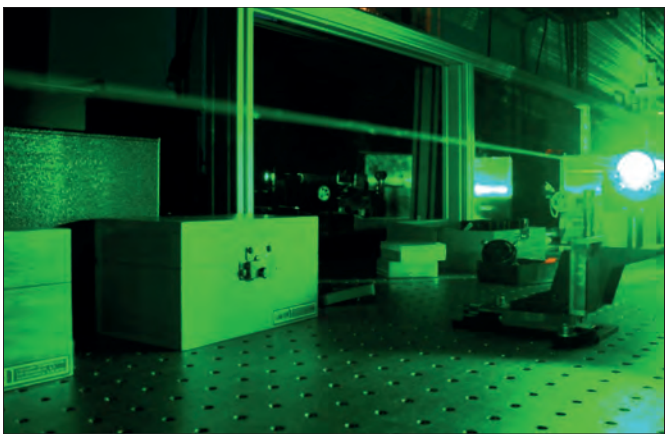

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Three steps to the future

2012 was perhaps the ESRF's most important year in its two decades of operation. When the first X-ray beams were extracted from the storage ring in May 1992 they brought to life the world's first third-generation source for hard X-rays. Since then, the users and the staff of the ESRF have turned an engineering achievement into a scientific success story, with over 20,000 peer-reviewed publications and the award of four Nobel prizes for work that has involved the ESRF: Roderick MacKinnon (2003), Ada Yonath and Venki Ramakrishnan (2009) and Brian Kobilka (2012).

Three important developments were initiated last year, shaping a new long-term perspective for the ESRF and demonstrating how we can maintain our leading role in synchrotron science both in Europe and worldwide until 2030 or beyond.

The first is a renewed long-term strategic mission for the ESRF. Elaborated by a Council working group chaired by Michel van der Rest, this policy document (which has been published on the ESRF's website) includes far-reaching recommendations on the scientific and technological evolution of the ESRF in a highly competitive European and international context. In short, the ESRF is not yet at the limit of its capabilities and therefore has the potential to remain a world leader for the foreseeable future. The required investments are minimal given the expertise that we have already accumulated. Furthermore, by continuing to play a central role in synchrotron science and developing new schemes of collaboration, the ESRF will serve best its wide user community and the interests of its member and associate countries.

"Last year we laid out the path towards the ESRF of the next 20 years"

The second achievement in 2012 was the preparatory work for Phase II of the Upgrade Programme, which allowed scientists in the Accelerator and Source Division to identify a major upgrade of the storage ring lattice. The new design reduces the horizontal emittance of the beam from the present 4 nm to around 0.1 nm, considerably increasing the brilliance of the source, and it is also fully compatible with the existing storage ring tunnel and beamline layout. The ESRF Council has authorised a detailed technical design study to be undertaken, with a final decision on implementation and construction expected in June 2014. Preliminary planning would see the ESRF operate with its present storage ring until the third quarter of 2018, when it would be shut down for approximately one year. Operations would restart towards the end of 2019, with the new storage ring becoming a cornerstone of the ESRF's global leadership in synchrotron science for the following decade. A white paper on Phase II of the Upgrade Programme is available via the ESRF website, and details of workshops held during the 2013 users' meeting to establish the science case for the Phase II upgrade can be found on pp10–12 in this issue of *ESRFnews*.

The third issue shaping the ESRF's future concerns our members and scientific associates. The attraction of the ESRF continues to grow, with South Africa joining the ESRF through a contractual arrangement and several countries in emerging large economies keen to join the ESRF as either full members or associates. The ESRF Council and management are working hard to develop these relations. We are also reviewing the level of participation of our existing partners so that we can bring the financial contribution of each partner into line with its long-term scientific use. This will reinforce the principle that beam-time allocation is based on scientific excellence, which is a prerequisite for the long-term success of the ESRF.

The year 2012 also saw an exceptionally long accelerator shutdown, the longest in 20 years, to allow major construction works. Yet the ESRF was successfully restarted and user operation resumed according to plan despite many unknowns linked to disruptive construction works, excavations and ground subsidence.

Last year was significant in that it laid out the path towards the ESRF of the next 20 years. In addition to all the actions under way to shape our facility, perhaps the most important is to make sure that the younger generations of scientists, engineers, technicians and administrators know about the ESRF and its opportunities. We wish to remain a pioneering field of science that attracts bright minds – a challenge that I hope to take on with new efforts and initiatives outlined in a forthcoming issue of *ESRFnews*.

Francesco Sette, *ESRF Director General*

Microsamples from masterpieces.

LEDs and Van Gogh

The increasing use of LED lighting in museums and art galleries could be accelerating the demise of famous works of art, a new study carried out by an international team at the ESRF and DESY has shown. It is already known that some pigments, especially the chrome yellows used by Vincent Van Gogh, turn brown under normal lightning conditions. This stems from the chemical reduction of chromium and is enhanced by the presence of sulphur in the pigment.

Different types of chrome yellow were used by Van Gogh, and these react differently to light, depending on their chemical composition. Regular chrome yellow maintains its colour after exposure to green-blue light and also when irradiated with more damaging UV light, the team found. But laboratory tests with flecks of less stable varieties of chrome yellow paint turned a brownish-green colour after just a few days of exposure to green-blue light (*Analytical Chemistry* **85** 851–867).

Brazil to break ground for Sirius

Sirius will supersede Brazil's exisiting second-generation light source.

Scientists in Brazil are about to prepare the site of a new third-generation synchrotron called Sirius, the first high-brightness photon source in Latin America. Located at the Brazilian National Synchrotron Light Laboratory (LNLS) in the state of São Paulo, the $330 m project is expected to be open for user operation in 2017, LNLS director José Roque told *ESRFnews*. "The building executive project should be finished by June 2013, and ground breaking should start by the end of April with the terrain cleaning, followed by the earth-levelling and drainage work."

The LNLS has been operating a soft X-ray source since 1997, hosting some 1500 users per year. Sirius, a 518 m-circumference machine with an energy of 3 GeV and a horizontal emittance of 280 pm rad, will provide 20 hard X-ray dipole-based beamlines with critical photon energies of 12 keV. It is based on a lattice similar to that proposed for Phase II of the ESRF Upgrade and to that being adopted at the MAX-IV Laboratory in Sweden.

Initially, says Roque, Sirius will have 13 beamlines. Five will be based on in-vacuum undulators for studies including micro-spectroscopy, coherent scattering, nano-diffraction and micro-focusing structural biology. A further five are to be based on bending-magnets for EXAFS, SAXS, powder diffraction, microtomography and infrared studies. The remaining beamlines will employ wigglers and elliptically polarising undulators.

Former ESRF director-general Yves Petroff, who has been LNLS scientific director since December 2009, says that one of the biggest challenges has been to stabilise the project's finances. Another challenge, he says, has been hiring people from abroad. "It's not so quick to get a visa, and there is a lot of paperwork involved in the buying and delivery of components, so there is some bureaucracy here that needs to be improved. This is changing now though."

There are a lot of scientific opportunities in areas such as health, biology and catalysis, says Petroff. "Industry is investing in the project too, and with the kind of emittance we expect from Sirius I expect we will have a lot of international users."

Bill Stirling, also a former ESRF director general, is also a member of LNLS scientific committee, while the head of ESRF's Instrumentation Services and Development Division, Jean Susini, is a member of the X-ray spectroscopy Beamline Advisory Team for Sirius. Although there is no formal agreement between the ESRF and LNLS, says Petroff, the ESRF provides a template. "The scientific model of the ESRF is among the best in the world."

Users' corner

The next Beam Time Allocation Panel meeting to review proposals submitted for the 15 January 2013 (Long Term Projects) and 1 March (standard proposals) deadlines will be 25 and 26 April.

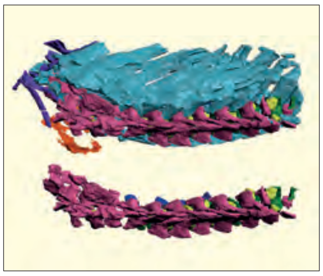
The next deadline for standard proposal submission is 1 September. Proposers must use the most recent experiment methods template available on the user guide web pages and respect the two-page length limit for this document. They should also ensure that experiment reports are submitted for all relevant previous proposals.

The 23rd ESRF Users' Meeting & Associated Phase II Workshops took place on 4–6 February. More can be read about this in the dedicated articles on pp8–12.

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

News from the beamlines

- **ID01** has increased its device and sample environment portfolio. A QuicK-MAPping procedure ("K-map") using a piezo positioning system supplying a 2D scanning probe in real space with a sub-micronic beam at a sampling rate of up to 100 points per second has been introduced, which accelerates scanning diffraction microscopy and allows a quicker and easier navigation on the sample. A lightweight He-flow cryostat providing temperatures in the range 2–300 K has been installed. Made by the ESRF sample environment unit, the cryostat can be mounted on the Hexapod of the ID01 diffractometer with a special holder and is compatible with scanning X-ray diffraction microscopy. In the next proposal round, ID01 will be available for only half of its usual beam time. The beamline will be closed in December in order to install the new optics and to move the end station into the new EX2 building. User operation will resume in November 2014.
- A new monochromator has been installed on **BM26A** (DUBBLE). Equipped with both Si-111 and Si-311 crystals, the energy range of the beamline is now 4.5–45 keV.
- An upgraded Laue micro-diffraction setup has been implemented on **BM32** (CRG-IF). Laue micro-diffraction uses a broadband white micro-beam (5–22 keV) combined with sample-scanning and a 2D detector for mapping strain and orientation in polycrystals. The new setup uses fixed-curvature ion-beam-polished KB mirrors mounted on hexapods for micro-focusing, which provide larger acceptance and reduced beam size at the sample. Precise translation stages for the microscope and a piezoelectric shutter were also added. The LaueTools data analysis software suite enables the automatic treatment of Laue maps and the display of 2D orientation and deviatoric strain fields.



Early tetrapod vertebrae imaged at ID19 using high-energy X-rays.

Retracing our first steps on land

Around 400 million years ago, four-limbed creatures began to make short excursions into shallower waters. How these early "tetrapods" transferred to land, eventually evolving into amphibians, reptiles, birds and mammals, is a subject of intense debate among palaeontologists and evolutionary biologists.

ESRF users are shedding light on this crucial evolutionary step. Stephanie Pierce of the Royal Veterinary College in London and colleagues at the University of Cambridge, Uppsala University and the ESRF used high-energy X-rays at ID19 to reconstruct the backbones of 360 million-year-old tetrapods in exceptional detail – with unexpected results.

All tetrapods have a backbone formed by a series of vertebrae. In modern tetrapods a vertebra is composed of only one bone, but early tetrapod vertebrae had multiple parts. What was previously thought to be the first bone in the series is actually the last, found the team, which has ramifications for the functional evolution of the tetrapod backbone (*Nature* **494** 226–229). "By understanding how each of the bones fits together we can begin to explore the mobility of the spine and test how it may have transferred forces between the limbs during the early stages of land movement," says Pierce.

One of the fossilised creatures also had a string of bones extending down the middle of its chest. This would have strengthened the ribcage and allowed the animal to support its body weight on its chest during tentative steps onto land. "The results of this study force us to re-write the textbook on backbone evolution in the earliest limbed animals," says Pierce.

Study fuels nanotoxicity debate

Metals contained in nanoparticles can potentially enter into the food chain, a new study at the ESRF shows. The finding, published in February, emerged from detailed chemical maps of soya plants grown in soil mixed with zinc and cerium nanoparticles, and adds to the debate surrounding nanoparticle toxicity.

Humans have been producing nanoparticles for millennia, mainly by burning wood. But the past decade has seen nanoparticles specifically engineered for applications. The very different behaviour of materials at the nanoscale make nanoparticles key components of numerous consumer products ranging from sunscreen to car tyres. Eventually, these particles end up in soil, water or air.

J L GARDEA-TORRESDEY

Soya bean plants during their maturation in greenhouse conditions.

Jorge Gardea-Torresdey from the University of Texas in El Paso and colleagues at the University of California in Santa Barbara, SLAC and the ESRF grew soya bean plants to maturity in soil mixed with zinc oxide and cerium dioxide nanoparticles, which are commonly used in industry, and studied the distribution of zinc and cerium throughout the plants using X-ray micro-beams at the ESRF and SLAC (DOI: 10.1021/nn305196q).

Cerium was found to be present in the root nodules and also reached the plant pods in the same chemical state as it was in the nanoparticles. Zinc was also found throughout the plants, although in a different form. Since zinc is already present in most plants, explains team member Hiram Castillo of the ESRF, it is not surprising that zinc from the soil can enter into the plant tissue. "There is a lot of controversy surrounding this research topic, so we must be responsible in what we publish and take care not to over-interpret data," he told *ESRFnews*.

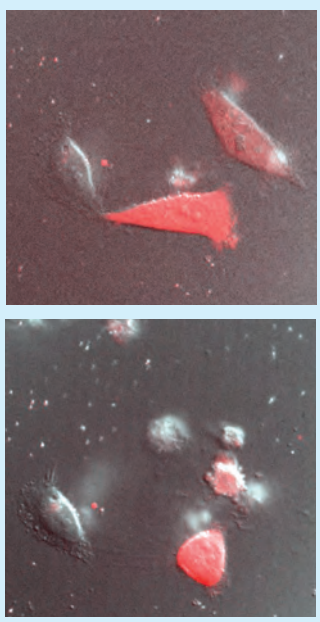
Castillo and colleagues have obtained similar results for other species of plant and nanoparticles (see p26). "Our results obtained at ID21 show that foliar exposure to silver and titanium oxide nanoparticles leads to an internalisation of the particles inside lettuce leaves without visible toxicity symptoms," says Camille Larue at the Université Joseph Fourier in Grenoble. "The next step is to go towards more environmentally relevant conditions: exposure in soils, low concentrations and longer exposure periods."

Scientists are concerned that irrigation with waste water and the use of sewage sludge as fertilizer might allow potentially dangerous particles to enter the food chain. Whilst it is not possible to directly attribute nanoparticle ingestion to any particular disease or symptoms, explains Arturo Keller of the University of California in Santa Barbara, who was not involved in the research, "the fact that these potentially dangerous particles are being taken up by such a common crop suggests a need to review what materials are used in agriculture around the world," he said.

Killer red brings imaging into focus

Over-illuminated fluorescent proteins undergo significant transformations that result in photobleaching and thus a loss of fluorescence. This phenomenon is usually a nuisance for common fluorescence-based imaging applications, although it can be put to advantage for some techniques, such as in situ photo-activation of photosensitising fluorescent proteins.

KillerRed is a red fluorescent protein that generates highly cytotoxic reactive oxygen species when illuminated with strong light. Now, X-ray crystallography combined with spectroscopy has allowed ESRF users to elucidate the molecular mechanism of the laser-induced photo-conversion that results in a photobleached state of the molecule. The image shows light-induced killing of HeLa cells using KillerRed, visualised by fluorescence microscopy before illumination (top) and after (bottom).



J P KLEMAN

The results, obtained last year by Eve de Rosny of the Université Joseph Fourier in Grenoble and Philippe Carpentier of the ESRF, will help in the design of new improved photosensitive or photoresistant fluorescent proteins for chomophore-assisted light inactivation and may even provide the basis for enhanced photodynamic cancer therapy (*J. Am. Chem. Soc.* 134 18015).

Users span the sciences

The 23rd ESRF users' meeting held on 5–6 February took delegates from our prehistoric origins to the long-term future of the ESRF, reports *Matthew Chalmers*.

"You're looking at the most complete map of an early vertebrate ever achieved," Per Ahlberg from Uppsala University in Sweden told delegates of the 2013 users' meeting, standing before a 3D image of the skull of an ancient creature called Romundina. "It's really quite beautiful work," he said, pausing himself to admire the nerve canals and blood vessels that could be seen in the half a billion year old sample.

Vertebrates come in many different forms – reptiles, fish, mammals and birds, for instance – but they all have a common ancestor. To understand the transitions between species, such as how the first land creatures emerged from the oceans and how the jaw evolved, researchers have to extract as much information as possible from what look to the untrained eye like lumps of rock. Fossils from this period are often as flat as a pancake and therefore difficult to interpret, explained Ahlberg, but some contain delicate internal anatomy that once housed the brain and sensory organs.

The fossilised bones in some samples contain perfectly preserved cellular structures (image right). However, these structures are difficult to image using conventional grinding or slicing methods. Propagation phase contrast microtomography, a technique pioneered for fossils at the ESRF, can image the internal and external features of a fossil at resolutions of less than a micron, revealing immense anatomical and histological detail. The ability to model hard-tissue histology in 3D casts light on tissue composition, organisation and growth, and also allows researchers to identify muscle attachments on the bones. "Data such as these are coming thick and fast," says Ahlberg. "If it weren't for Paul Tafforeau and the staff at ID19 we would not have the world's finest palaeontological imaging facility here."


P AHLBERG/S SANCHEZ

Cellular structure revealed in the fossilised bone of a 380 million-year-old fish called *Compagopiscis croucheri*, which is among the most primitive jawed vertebrates known.

Raising the temperature

Bernhard Keimer of the Max Planck Institute for Solid State Research in Stuttgart, Germany, has his feet firmly in the present. He is grappling with one of the biggest problems in physics – the origin of high-temperature superconductivity – which is a distinctly quantum phenomenon. Specifically, it is a consequence of Bose-Einstein condensation: bosons are particles with integer values of spin angular momentum and therefore are able to occupy the same quantum state, constituting a macroscopic wavefunction that can cause a bulk material to lose its electrical resistance. Low-temperature superconductivity, discovered a century ago, is the result of electrons pairing up into bosonic "Cooper" pairs as a result of vibrations of the crystal lattice. But high-temperature superconductivity, discovered in 1985, is proving harder to tame.

"The science coming out of the ESRF is truly wonderful."

Researchers at MPI are making progress thanks to X-ray studies at the ESRF and elsewhere. Recently, Keimer and his colleagues used resonant inelastic X-ray scattering to produce single magnetic excitations ("magnons") in copper-oxide based superconductors, or cuprates. "We know that magnons are intimately involved in superconductivity, but we need a quantitative understanding by detecting magnons through magnetic excitations," he said. "This wougive us the value of transition temperature." Keimer's team is also studying new classes of high-temperature superconductors in other metal oxide materials, and he expects within the next decade or so that researchers will have a complete understanding of high-temperature superconductors, which have potentially revolutionary applications.

Science for all seasons

Other lectures at the 2013 users' meeting spanned novel phases of lipids, G-protein coupled receptors (see box, p9), nanostructures, and materials for spintronics applications. "The science that is coming out of the ESRF is truly wonderful. It is really a pleasure to witness," remarked director general Francesco Sette. The overall number of published papers since the ESRF started up has now passed 21,500 – a record for a synchrotron facility. Also, despite there being 30% less beam time in 2012 due to building works, the number of proposals went down by just 5%.

All attention now is on completing Phase I of the upgrade while preparing the ground for Phase II, due to begin in 2015. "2012 has been a cornerstone between the past and the future," said Sette. "We have the opportunity to open a new chapter in synchrotron science."

Young Scientist Award goes to Simon Kimber

Chemist Simon Kimber has won the 2013 ESRF Young Scientist Award for his outstanding work on X-ray pair distribution function techniques and their application to nanostructured materials. Simon completed his PhD on spin and orbital ordering in ternary transition metal oxides at the University of Edinburgh, UK, in 2007 and spent two years at the Helmholtz-Zentrum in Berlin before taking up a post-doctoral position at the ESRF's ID15 beamline in May 2010.

Why did you win this award?
For discovering something fundamental about the structure of nanoparticles, and for having developed techniques not commonly used at synchrotrons.

Why does it matter?
For me, the award closes the door on 10 years of research. Nanoparticles are important because they are all around us in products such as paint, sunscreens and catalytic converters. Optimising the performance of these things requires an understanding of their structure.

How did you go about your research?
Standard crystallography techniques are not applicable to nanomaterials because the concept of the unit cell falls apart, so I use pair distribution function analysis, which involves a pair-wise sum over atomic displacements. ID15 is vital for these experiments because you need high energies.


C ARGOUD

What's the best thing about your research?
The best thing about the ESRF is the exposure to so many different types of science and the fact that nobody tells you what to do. For me it was a real advantage. Meeting users is also nice.

What's the worst thing about your research?
It's unbelievably hard work. You spend periods of weeks manically talking to yourself and reading hundreds of journal articles. It's 1% inspiration and 99% perspiration. Research is purely about having the right mindset, and stubbornness.

Where will you be in five years?
Probably not doing research. I believe that the biggest contribution in science is to go into organising it better because the questions are so difficult now that you need bigger research groups, yet funding and professional structures haven't caught up with this. I find it difficult to recommend science as a career for young scientists. It gives you unrivalled job satisfaction and work-life balance, but academic job prospects are poor. If I were to remain in research it would be at a facility such as the ESRF rather than at a university.

What would you be if you weren't a scientist?
I would have loved to have done some serious skiing and climbed more mountains, but as you get older you realise that there is more to life than your career.

• The winner of this year's poster competition is Giovanna Boumis from the University of Rome "Sapienza" for her poster "Crystal structure of *Plasmodium falciparum* Thioredoxin Reductase, a validated drug target."

Molecular doorbells


M CHALMERS

Gebhard Schertler of the Paul Scherrer Institute in Switzerland digests G-protein coupled receptors (GPCRs).

"GPCRs are the link between the inside and outside of the cell. It's like a doorbell that you ring and see whether someone answers. We now have about 50 structures from 15 different GPCRs, mostly obtained during the past 5–10 years. Beta-blockers were the first drug to exploit them, and now some 30% of drugs target various GPCRs. But understanding the signalling pathway is much tougher, and that's what we do today. Signalling inside the cell is a highly nonlinear process and we are only at the beginning of understanding this. No other synchrotron has contributed to my career more than the ESRF, and it was the constant support of the ESRF that really enabled the first GPCR structures. The ESRF upgrade is a very nice thing to do because we'd really like more dynamical information, and time-resolved WAXS at higher flux would allow us to do really new stuff. X-ray free-electron lasers (XFELs) will change the way we work and I think there will be a very fruitful collaboration between synchrotrons and XFELs."

P GINTER/ESRF

Synchrotron science enters a new phase

An upgraded storage ring would boost the brilliance of the ESRF's X-rays by a factor of at least 30. The science on offer from such a machine was the focus of discussions at this year's users' meeting.

The ESRF is already more than half way through a major upgrade. When complete in the next 18–24 months, the facility will boast an enhanced source, 18 new and upgraded beamlines and 21,000 m^2 of additional space. The €150 m project will give users brighter and smaller X-ray beams, improved beamline facilities and instrumentation, and allow a higher throughput of experiments.

To cement the ESRF's role as the leading provider of hard X-rays from 2020 onwards, management has proposed a radical reconfiguration of the storage ring that will increase the brilliance and coherence of its X-rays much further (see box below). The new storage ring will form the centrepiece of Phase II of the ESRF upgrade, due to take effect from January 2015, and opens up new avenues for synchrotron science that are now being explored by users and management.

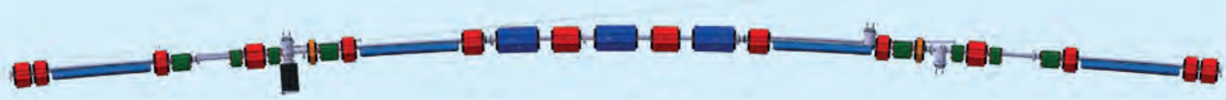
The thrust of Phase II is to make extensive use of highly brilliant nanobeams and multiple techniques in order to extract maximal information about matter and materials across all relevant timescales. Increased brightness allows higher experiment throughput and increased levels of coherence offers users new ways to determine micro- and nanoscale structural properties in both hard condensed matter and biological materials.

Get real

The five core science drivers of the ESRF upgrade are: nanoscience and nanotechnology; pump-probe experiments and time-resolved diffraction; science at extreme conditions; structural biology and soft matter; and X-ray imaging.

Phase II will open a new dimension to X-ray imaging: time. A higher coherent photon flux density reduces acquisition times, taking users from static pictures towards time-resolved imaging at all length and timescales.

Supercharging the source



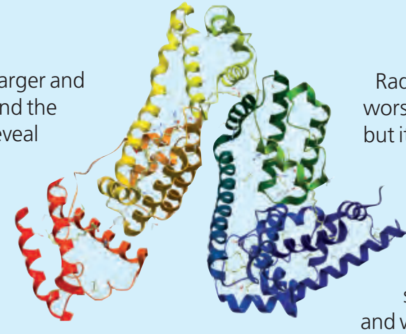
The X-rays produced by today's synchrotrons are much larger in the horizontal plane than they are in the vertical, which can severely limit the resolution of experiments.

Last year, staff in the ESRF's Accelerator and Source division devised a way to reduce the horizontal emittance by a factor 40 (to around 100 pm – lower than any other light source in the world) without increasing the size of the storage ring or moving a single beamline. The new lattice would increase the brightness by at least a factor 10, reuse 90% of existing infrastructure and require no more than two six-month long shutdowns.

The basic idea is to replace two dipoles in each of the ESRF's 32 arcs with seven weaker dipoles (pictured), which bend the electron beam in a more continuous trajectory. Despite the increased brightness, the integrated power load on the first optical element is no more than that on many existing beamlines.

The new machine is not expected to be any more difficult to run than the existing one, and will be just as stable. Phase II of the upgrade would also see the Vercors building reinstated to provide space to assemble the new storage ring, later housing new long beamlines.

Do we need all of these photons?

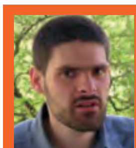


James Holton of the University of California at San Francisco surveys the implications of the upgrade for structural biologists:
"With current equipment there is no great advantage for structural biologists in having higher flux density – crystals already burn up so fast that most of the world's macromolecular crystallography (MX) beamlines use absorbers. Coherence also has no real impact on diffraction, so at first blush many in the MX community might say: No! We don't need this upgrade – please don't shut down the ESRF for a year!

But the purpose of the ESRF is to look beyond current technology and support new experiments that no single nation can support on its own. The upgraded storage ring will have beams capable of collecting an entire MX data set in a few milliseconds, which opens up new regimes in how experiments could be done. The heavy-hitting projects of the future will be the structures of larger and more dynamic systems and the functional studies that reveal how they work, not just how they look. This means a return to room-temperature experiments, and lots of them. Crystals of molecules that function by undergoing large conformational changes tend to not diffract very well. The trick is finding conditions that can get a whole crystal full of molecules to sit still long enough to get their picture taken. Functional studies involve mechanisms, mechanisms are tested with kinetics and kinetics are done with time-resolved experiments. Many of the problems encountered with time-resolved structural studies may go away with micron-sized crystals, traded off against problems of radiation damage.

Radiation damage is a lot worse at room temperature, but it can be overcome if you can combine data from enough crystals. There is a vast chasm of unexplored dose-rates between what current synchrotrons can deliver and what X-ray free electron lasers do with their femtosecond-class pulses. The new ESRF will reach across that chasm.

Provided fast enough detectors become available and that we can introduce fresh samples into the beam rapidly enough, the femtolitre-class assay volume of the future ESRF MX beams could be the 'search engine' the MX community needs to learn the biological significance of each step in the dance performed by nature's nano-machines."



Julian Stangl
Johannes Kepler University, Austria

"More brilliance will certainly help us, as will longer beam-lines and more coherence."

Single-bunch phase contrast imaging, for instance, could capture crack propagation in solids or fuel injection through a steel nozzle on a scale of 100 ps. Combining real-time imaging with nano-focusing capabilities, such as those of the Phase I upgrade beamline NINA, would allow *in operando* characterisation of electronics devices such as packaged MEMS and of slower processes – for instance, copper electro-migration inside chips or the development of a living organism. "Operating devices are getting smaller and smaller so we need to study the dynamics of nanofeatures on all timescales," said the ESRF's Alexander Rack.

In general, the new source will allow studies of more complex, real-life systems under conditions much closer to those that materials experience. Catalysts, for example, could be investigated under near-industrial conditions such as high pressures, allowing their performance to be optimised. In metallurgy, very fast processes and the changes involved will be observable, such as what really happens during welding and how that affects subsequent weld performance.

For users who study matter under the most extreme conditions, which is relevant in chemistry, planetary science and hard condensed-matter, smaller and brighter beams offer unprecedented opportunities. Smaller beams mean smaller samples, higher pressures and fields, and new techniques – chemical mapping with sub-micron selectivity will be feasible, for example. "We want to extend the phase diagram of the elements up to and beyond the 500 GPa range, to discover new structures induced by the hybridization of core electrons," says Claudio Mazzoli of the Politecnico di Milano University, Italy. "We want to measure the structural changes in the dense fluid state, to extend the melting curves to map order parameters in ferromagnetic superconductors and to understand matter in the proximity of quantum critical points."

Complementary science

The upgraded source will allow experiments that complement those on offer from X-ray free-electron lasers (XFELs). "There are lots of things that the new storage ring can do that compare favourably with other light sources, including XFELs," says Jochen Schneider of DESY. "Due to the large increase in coherent fraction the new ESRF reduces the coherence gap between storage rings and XFELs strongly, and will open unique opportunities."

Structural biology is an example of an area

John Seddon
Imperial College London, UK

"It's very good to be participating at this crucial moment for the ESRF's future."

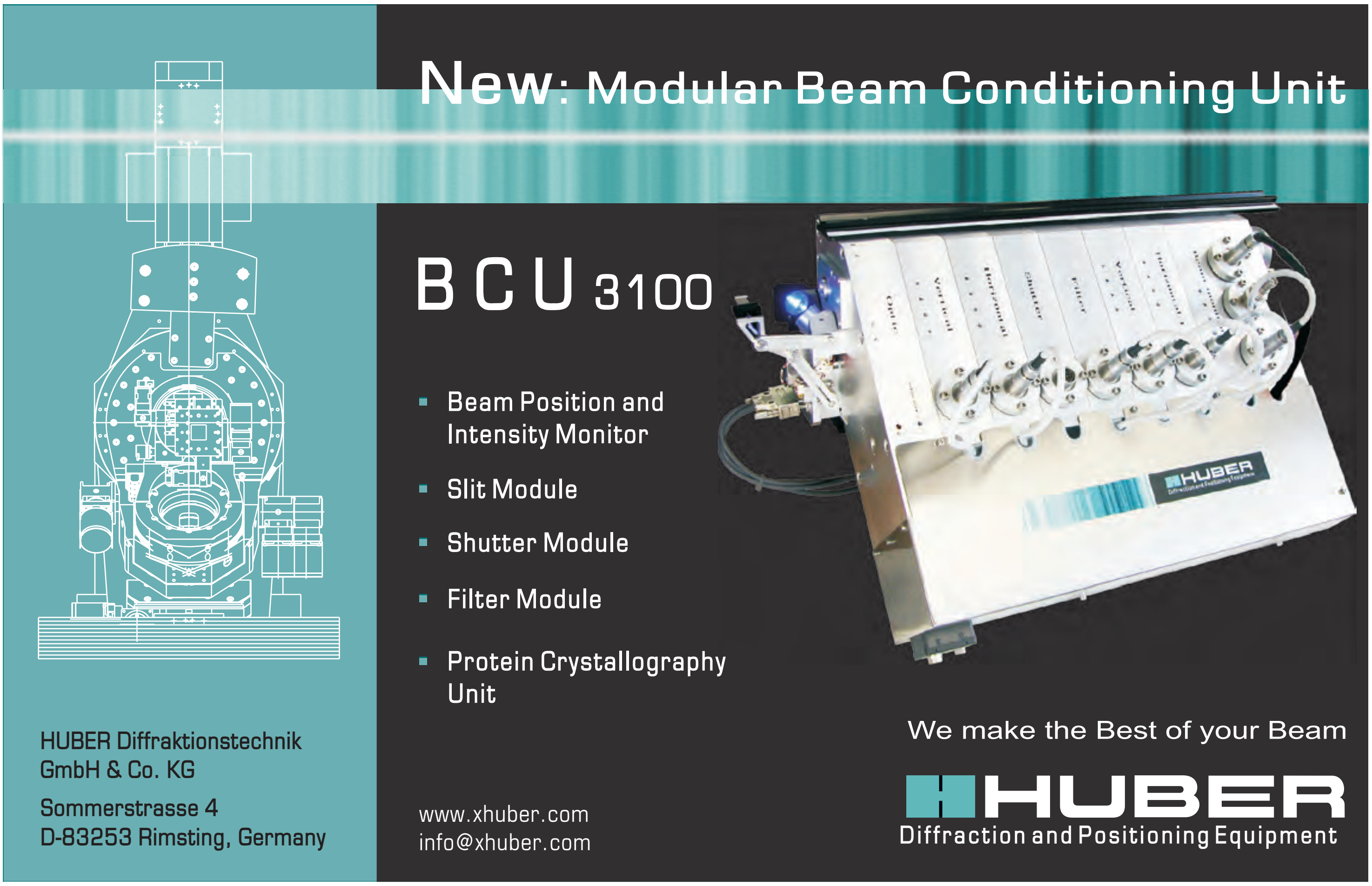
Martina Müller
Research Centre Jülich, Germany

"The lateral resolution would improve considerably if the brilliance goes up."

where XFELs and "ultimate" storage rings could complement one another. The ESRF's biologist users should be reassured that their experiments won't be unduly affected by the increased brightness of the source (see box above). A major leap in brilliance and coherence would enhance the performance of all scattering and diffraction techniques, enabling structural and dynamical investigations of functional biological and other soft-matter systems in unprecedented detail.

In order to handle the much faster and more voluminous data that will stream from the upgraded source, a major part of the Phase II upgrade is to be put towards "enabling technologies", which encompasses software and new detectors. "I appreciate that we are still in Phase I and users don't want to change the brand new beamlines that they have just built," said research director Harald Reichert. "But remember that Phase II beamlines will not be operational until 2020."

The conclusions from five scientific workshops held on 4 February will be fed into a report to be submitted to ESRF Council this spring, and further workshops devoted to the Phase II science case are planned during the coming months.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The central science

Chemistry experiments at the ESRF cover everything from catalytic converters and cement to fundamental studies of molecular bonds as they form and break.

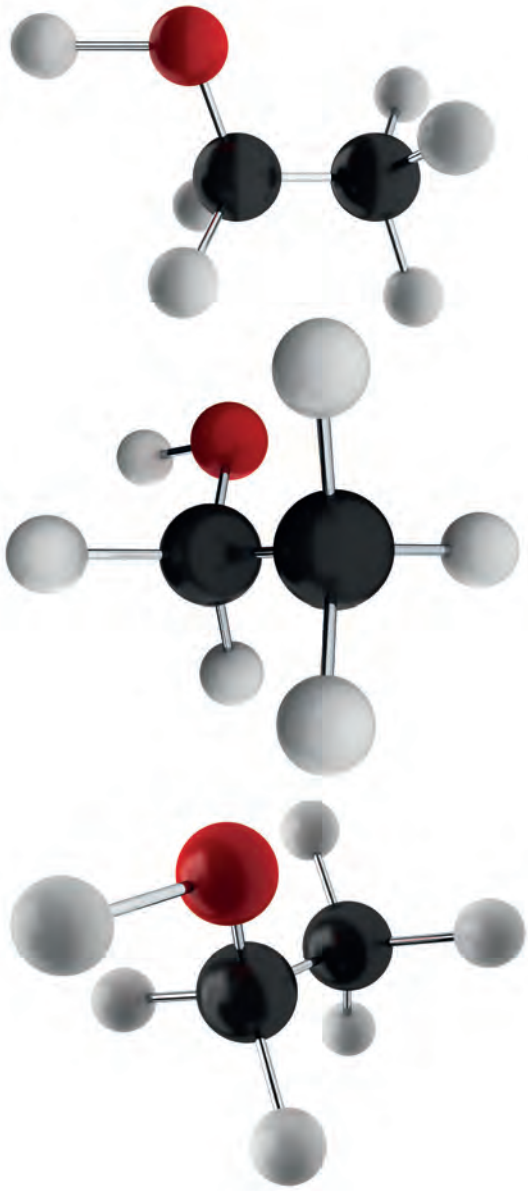
Andy Fitch is a relatively rare beast at the ESRF: a chemist. "There are a lot of physicists here, but we chemists are few and far between," he says. That is not to say that chemistry is rare, however: roughly 12% of the ESRF's beamline proposals are classed as chemistry although areas such as soft matter may also be considered branches of chemistry, says Fitch, who is scientist in charge of the high-resolution powder diffraction beamline ID31. "Chemistry goes everywhere, it's a central science."

Whether it is superconductors or organic molecules, the ESRF's small X-ray beams allow researchers to look at the structures of materials in order to better understand their chemical and material properties. Indeed, since organic molecules are the basis for the pharmaceutical industry, medical research is a major thread of chemistry. Developing hydrogen storage materials is another active area of structural chemistry at the ESRF, whereby users seek to understand the absorption of hydrogen's chemical bonds in different materials.

Science *in situ*

Where the ESRF has come into its own, though, is real-time studies of chemical processes. The very high flux of the source enables users to undertake detailed *in situ* studies of real-life systems as they evolve with time, such as finding out what happens to the materials that make up the anode or cathode of a battery as ions diffuse from one to the other. Similar electrochemical processes are relevant for fuel cells.

In situ catalysis is a major strand of ESRF's chemistry activities. The palladium and cerium nanoparticles that clear up toxic gases from your car exhaust, for instance, can be optimised using XRD-XAS to reveal what's happening in those materials as the reaction takes place. Major car manufacturers including Toyota have improved their catalytic converters thanks to the ESRF. "This is a new field," says ID15 user Marcos Fernández-García of the Institute of Catalysis and Petrochemistry in Madrid. "We can use X-ray diffraction and absorption to detect changes in the size and shape of palladium nanoparticles during operation, and elucidate its chemical origin and consequences using complementary techniques such as infrared and mass spectrometry."



"Not all chemistry research is directly geared to applications."

Industrial relations

There is a strong industrial flavour across ESRF's chemistry research. Visiting the Dutch-Belgian DUBBLE beamline in December, *ESRFnews* met users funded by energy firm Chevron who are working on heterogeneous catalysis, by using the beamline's dedicated system to flush gases over their samples and looking at structural changes under realistic *in operando* conditions. Reactions relating to the famous Fischer–Tropsch process (see p19) and to oil-refinery are also performed here, as are numerous industry linked experiments spanning batteries, ceramics, semiconductors and pharmaceuticals, explains DUBBLE project leader Wim Bras. "This is the kind of background information companies require to think about products 10 years from now"

The chemical industry itself is constantly trying to improve its efficiency and reduce its environmental impact, such as designing paints that are water- rather than oil-based, and companies such as Unilever are frequent users. The reactions that take place inside a cement mixture are another example of an apparently simple substance that is giving up its chemical secrets at the ESRF, for example allowing additives to be designed that cause cement to work better at lower temperatures.

Elemental research

Not all chemistry research is so directly geared to applications. Time-resolved studies at ID09B, for instance, allow users to watch changes in chemical bonds on time scales as short as 10 ps, allowing individual reactions to be "filmed" in real time (see p21). Discovering and fabricating new molecules is another example. Porous materials called metal organic frameworks are promising storage materials being studied at the ESRF, where the hope is to find specific applications by joining the right metal with the right linker to generate the desired micro-porous architecture. "Synchrotrons radiation is a way of characterising these things," says Fitch.

So why the dearth of chemists at the ESRF? "It's not a traditional chemistry laboratory," says physicist Michael Wulff, scientist in charge of ID09B. "Some chemists might be a little frightened of this big machine with its physics-based tools, but chemists are extremely useful because they know the relevant systems to study."
Matthew Chalmers

The ESRF lights up the

Environmentally friendly washing detergents, better performing fuels and healthier foods. Indust

Procter & Gamble: The science of laundry

We have all seen the television adverts: people dressed as scientists promoting the latest brand of washing detergent. What is less well known is that synchrotrons are playing a vital role in bringing these and numerous other consumer products to market. "There really is hard science behind our products," says chemist Eric Robles, who is a research fellow at Procter & Gamble (P&G). "We develop our products based on strong scientific foundation to ensure that they make a significant difference in terms of performance."

P&G scientists have used the ESRF since 2004, working across several beamlines including ID01, ID02, ID13 and ID19 to study the microstructure of products including hair conditioners, dishwasher liquids, fabric conditioners, shampoos and facial creams. Better knowledge of the microstructure of the colloidal formulations allows researchers to tune the performance and stability of products and meet the company's strict environmental sustainability targets.

As much as 90% of the energy used to wash clothes goes towards heating the water in washing machines, so one of the main goals is to develop detergents that work at lower temperatures. P&G's latest innovation, Ariel Excel Gel, boasts cleaning at 15 °C thanks to specially designed enzymes and polymers, offering considerable energy savings. It is also highly concentrated, requiring less water to manufacture and reducing transport and storage costs.

The majority of liquid detergent formulations, explains Robles, exist as micellar solutions to ensure easy dosing and fast dissolution, but this requires the formula to have a high water content. If the water content is reduced, liquid crystals will start to form unless organic solvent is added, which adds more to the cost of a product and dents its environmental credentials. Another approach is to formulate it as liquid crystal, but the presence of other ions typically destabilises the product and causes "phase splits". With the help of SAXS, Robles and colleagues mapped the phase diagram of Ariel Excel Gel to determine the regions that are physically stable. As a result, Ariel Excel Gel became the first liquid detergent with a liquid-crystal microstructure with the lowest water content possible without the use of organic solvent, says the team.

It is also vital to test how a product behaves when in use. The performance of washing machines varies widely, so P&G uses a research laboratory instead to assess a product's washing ability. The team also tests how formulations behave during storage under different conditions, since they have to withstand both the scorching heat of Saudi Arabia and subzero temperatures in Russia.

"There are only very few capabilities that the ESRF doesn't have," says Robles, who is P&G's global co-ordinator for synchrotron research. "The appeal of the ESRF is its industry office and the relationships that we've built with the beamlines over the years." Synchrotrons in the US are not so well geared up for industry access, he says, and it can be hard to get access to other synchrotron facilities around the world.

"The appeal of the ESRF is its industry office."

P&G uses other sophisticated techniques including NMR and neutron experiments to improve the stability and environmental credentials of its products. The science behind a product may not always be visible to a consumer, but investing in science is critical to innovation, says Robles.

Matthew Chalmers

Unilever: Food for thought

When a tub of ice cream is transported, stored and even taken out of your freezer, temperature changes may destroy the microstructure of the ice crystals and cause recrystallisation that spoils the sensory experience. Food giant Unilever has used high-resolution tomography at the ESRF to get round the problems of temperature abuse. Results obtained at the ID19 beamline in 2009 revealed the variations in the microstructure of ice cream samples in a series of 3D images with a voxel size of just 0.56 μm, leading to improved products.

chemistry in your life

ry users from Procter & Gamble, Infineum and Unilever explain the science behind their products

Infineum: **Imaging boosts injection**

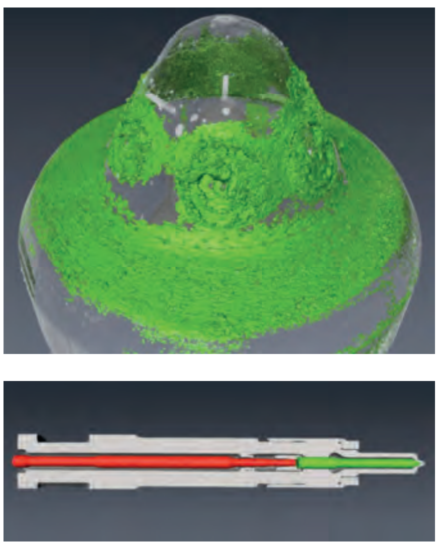
Ever tightening global emissions standards have demanded significant changes to diesel engines in the past decade. Specifically, manufacturers have had to gain very precise control over the fuel injection process. Diesel engines require a fine mist of fuel to be injected into the combustion chamber to facilitate good mixing of fuel and air, which in turn means more efficient burning of the fuel. To aid this process, injection systems today commonly operate at pressures of around 2000 bar and temperatures above 100 °C. They are also machined to extremely high precision: injection spray holes are commonly between 80–150 μm in diameter.

During the operation of such finely engineered systems, however, carbonaceous deposits can alter the operation of the injector or flow of fuel and thus affect the combustion process, making it difficult to control emissions and fuel consumption. For this reason, diesel sold throughout Europe contains additives designed to clean and maintain the fuel system.

Analysis of the deposits is vital if we are to understand their impact on injector operation and develop better chemical additives to reduce them. This is commonly done by cutting the injector open or disassembling it, and then analysing the deposits with typical laboratory techniques. Cutting destroys the apparatus and disassembly modifies its performance when the components are put back together. Therefore, since injectors are often part of on-going test programmes, a non-destructive means of analysis is desirable.

The ESRF's ID19 station offers unique possibilities for characterising the deposit phase, providing phase-contrast imaging and high spatial resolution while maintaining high-enough X-ray energies to penetrate up to 1.5 cm of steel. These factors were crucial to a two-year long study performed by Infineum with Paul Tafforeau of the ESRF and Ali Chirazi of the Institute for Condensed Matter Chemistry of Bordeaux. Using such methods we were able to determine the volumetric deposit map for the injector and compare this to the parameters measured during engine operation. The experiment also revealed that the deposits inside the injector spray holes contained metal, showing that the chemistry of the deposit phase inside the injector is different to that seen on the tip in the combustion chamber.

In addition to spray-hole experiments, scanning the internal parts of the injector was of interest to see if deposition had occurred on the fast-moving parts responsible for fuel flow. Deposition on these parts can affect the synchronisation of components because the engine's electronic control unit can no longer maintain the same precision control. However, since the deposition can occur in several places it was necessary to scan the majority of the body of the injector (which measures 12 cm in length), at a resolution of 6 μm – generating a 900 GB dataset. An even higher resolution scan would be desirable, but would render the data volume too large to realistically process.



Tip of a 12 cm long diesel injector (full unit also pictured) showing carbonaceous deposits.

In collaboration with Lyle Pickett of Sandia National Laboratories at Livermore and Alan Kastengren of Argonne National Laboratory, both in the US, we have also employed the techniques available at ID19 to scan the spray hole zone at 0.65 μm resolution in order to generate detailed internal surface geometry models. The science of machining injector spray holes is an area of intense interest due to its importance in controlling the flow, spray and thus combustion behaviour, and modelling is an integral part of such studies.

Computational fluid dynamics allows us to understand the effects of flow and cavitation on the structure of the spray, and one of the challenges is to obtain detailed structural geometry of the spray holes inside the injector at a resolution high enough to generate accurate surface models. It is hoped that the ESRF will help improve this work by allowing much more accurate inclusion of the effects of internal surfaces on flow behaviour. Meanwhile, Infineum continues to work with the ESRF to utilise and evaluate the techniques in other areas relevant to our business aims.

Peter Hutchins, Infineum

More recently, a Unilever team used the ESRF to tackle the saturated fatty acids in foods. A common way to provide texture to oily food products such as margarine and chocolate is to introduce a network of small triglyceride crystallites, which are rich in saturated fatty acids that raise blood cholesterol levels and contribute to the risk of heart disease and strokes. Researchers at Unilever discovered that a particular mixture of cholesterol-lowering plant sterols, γ-oryzanol with β-sitosterol, could provide a healthier alternative. The molecules in the mixture assemble very differently compared to molecules in a normal crystal with 3D long-range order, and the company chose to come to the ESRF to investigate its structure. "The ESRF covers all of the length scales involved in this mixture," explains Unilever research scientist Ruud den Adel. Small- and wide-angle X-ray scattering at the ID02 beamline revealed a network of tubules in samples of sunflower oil that form rapidly when cooled below the melting point of the gel.

Unilever spends almost €1 bn per year on research and development, and is using the ESRF for several applications, says research scientist Gerard Van Dalen. "The objective of our latest study is to understand and improve the quality of freeze-dried vegetables after rehydration by assessing the multi-length scale pore structure and modelling its impact on the rehydration behaviour," he says.

Matthew Chalmers

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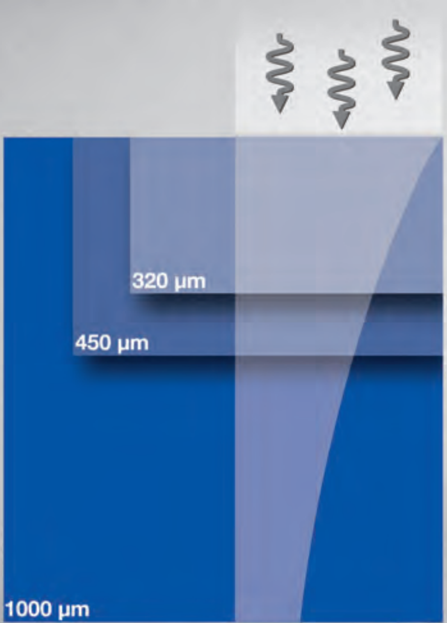

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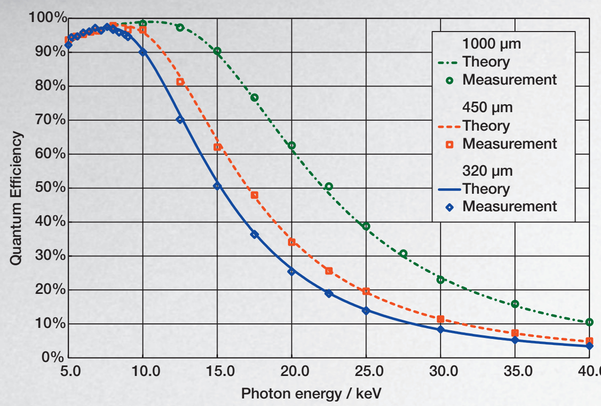

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Mechanical reactions go green

A recent experiment at the ESRF reveals how the mechanical action of steel balls drives chemical reactions without the use of potentially dangerous solvents

Despite continuous use ranging from extractive metallurgy to pharmaceuticals, mechano-chemical reactions remain mysterious.

It is one of the first experiments that we encounter during school chemistry lessons: mixing up ingredients with a solvent to produce a solution. In the classroom the solvent might be water, but industrial solvents tend to be more hazardous. Some are flammable, such as the turpentine used to thin paints, while others can be toxic or carcinogenic, like benzene. Bulk solvents used across the chemical industry are a major environmental and safety concern, but there are other routes to produce solutions.

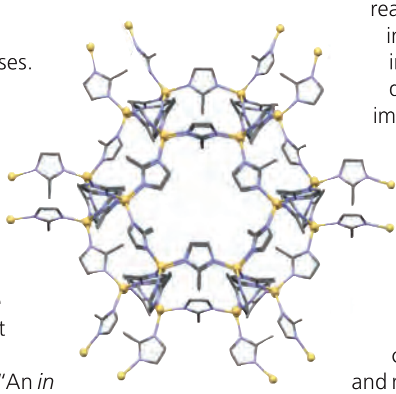
Mechano-chemistry offers an energy-efficient alternative to solvents. Mechanical action clearly breaks chemical bonds – witness what happens to your clothes after years of being tumbled around in washing machines. What is less widely known is that mechanical forces can also synthesise new chemical compounds and materials. In "ball milling", reactions are driven by the intense impact of steel balls roughly 1 cm in diameter in a rapidly vibrating jar that also contains the reactants and catalysts. Chemical transformations take place at the sites of ball collisions where the impact generates localised extremes of heat and pressure.

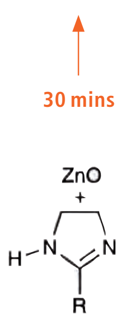
Ball milling has become a popular way to produce highly complex chemical structures such as porous metal-organic frameworks. Mechano-chemistry is already widely used in the pharmaceutical, chemical and metallurgical industries and is now emerging as an environmentally friendly alternative to solution-based syntheses. But the process is difficult to model. Conventional experiments do not provide any kinetic information because they rely on stopping the mill and examining the products at convenient intervals, explains the ESRF's Simon Kimber. "An *in situ* experiment like ours gives directly the time dependence of the reaction."

High impact

Now Kimber and an international team have used the ESRF's ID15B beamline to perform the first real-time study of a milling reaction, using X-rays to track rapid transformations as the mill mixes, grinds and transforms simple ingredients into a complex product (*Nature Chemistry* **5** 66–73). "When we set out to study these reactions, the challenge was to observe the entire reaction without disturbing it, in particular the short-lived intermediates that appear and disappear under continuous impact in less than a minute", says team leader Tomislav Friščić of McGill University in Montreal, Canada.

The researchers studied the mechano-chemical production of a commercially available metal-organic framework called ZIF-8 from the simplest and non-toxic components (see inset). Such materials are promising candidates for capturing large volumes of gas and could, if manufactured cheaply and sustainably, become widely used for carbon capture, catalysis and hydrogen storage. The team, explains Kimber, came to the ESRF because of the machine's high-energy X-rays capable of penetrating 3 mm thick walls of a rapidly moving reaction jar made of steel, aluminium or plastic. "The X-ray beam must get inside the jar to probe the mechanochemical formation of ZIF-8, and then out again to detect the changes as they happened."





Simple components are transformed into the ZIF-8 metal-organic framework after 30 minutes of mechanochemical action.

The unprecedented methodology at ID15B allowed the team to observe the reaction kinetics, reaction intermediates and the development of their respective nanoparticles in real time. The results reveal mechanochemistry to be highly dynamic, with reaction rates comparable to or greater than those in solution. The technique also enabled Friščić and co-workers to probe directly how catalytic additives such as organic liquids or ionic species change the reactivity pathways and kinetics.

The study opens the door to other types of mechano-chemical reactions in ball mills, which can now be studied and optimised for industrial processing. "These results hold promise for improving the fundamental understanding of processes central to pharmaceutical, metallurgical, cement and mineral industries and should enable a more efficient use of energy, reduction in solvent and optimise the use of often expensive catalysts," says Friščić. "This translates into good news for the environment, the industry and the consumers who will have to pay less."

Matthew Chalmers

Synthesising fuels of the future

The ESRF is helping chemists optimise a 90-year-old reaction that turns ordinary solids, liquids and gases into valuable hydrocarbons.

In the not too distant future the Earth will run dry of the stuff that shapes our lives more than most: oil. Energy companies are already turning to more arduous methods to extract oil than simply drilling into reservoirs – tar sands, for instance, represent a vast source of hydrocarbons but at an economic and environmental cost. Chemists have a vital role to play in finding alternative ways to turn the basic elements into fuels of the future.

In the 1920s, German chemists Franz Fischer and Hans Tropsch discovered a reaction that synthesises oil and other long-chain hydrocarbons from the basic building blocks carbon monoxide (CO) and hydrogen. The input can be biomass, coal or pretty much any source of carbon, while the output is fuel, free of sulphur and other pollutants, that can be used in existing car technologies. The Fischer-Tropsch process was optimised during World War II when Germany needed a domestic source of oil. Some countries, notably South Africa, have stuck with it and today the nation's state oil company SASOL converts more than 40 million tons of coal per year into 150,000 barrels of liquid fuels and other useful hydrocarbons. Shell produces a similar volume via Fischer-Tropsch synthesis at a new facility in Qatar, which turns natural gas into liquid hydrocarbons including its "V-power" fuel sold widely in Europe.

Reaction mechanics

The Fischer-Tropsch process may be a recognised industrial process, but it is not fully understood. Chemists want to know the intermediate steps of the reaction so that they can make it run at lower temperatures and pressures with higher yields. "At the ESRF we try to understand the mechanism of the reaction so that we can optimise it," says Roberto Felici, scientist in charge of the ESRF's ID03 beamline. "We take a more physics approach, whereas users such as SASOL are more empirical – they modify the catalysts while monitoring the reaction rate and choose which works the best."

"The gap between the ESRF and industrial needs is getting smaller."

Staff at ID03 collaborate with a consortium based at Leiden University in the Netherlands called NIMIC (Nano-IMaging under Industrial Conditions). Recently, for example, investigations into the pre-stages of the reaction during which CO is oxidised showed that the catalyst, palladium, is in a metastable oxide state that is very likely to be the driver of the reaction. "It was always thought that the metal was inert," says Felici. It is just one example of progress being made towards optimising the 90-year-old Fischer-Tropsch reaction.

The NIMIC and ID03 teams study each stage of the process. They start with a single crystal of catalyst, which is usually iron or cobalt, and study the process in different crystallographic planes. Once they understand the single crystal, the scientists distribute nanoparticles on a surface to see if the catalyst behaves in the same way in bulk form. The third phase, usually performed either on the ID31 or ID15 beamlines, involves *in situ* studies of nanoclusters in a supporting oxide environment, which corresponds to the pellets used in a real environment.

The goal is to optimise the reaction, which means avoiding degradation of the catalyst. In an industrial plant, explains Felici, the nanoparticles have to be replaced regularly. "They have tanks 20 m high filled with pellets and catalyser, so if you can find a way to make the reaction to occur at lower temperatures or make the catalyser live longer, all these things would have a huge impact."

Industry links

The renewed attention being paid to the Fischer-Tropsch process involves close collaboration between the ESRF and industry. Researchers from SASOL have recently used powder diffraction at the ID31 beamline to study the reaction catalysts, specifically to understand the structure of carbides thought responsible for driving iron-based systems. The ESRF's Belgian-Dutch DUBBLE beamline and its users collaborate with Shell and other companies on Fischer-Tropsch catalysis, while staff at the Swiss-Norwegian SNBL beamline are working with Norwegian oil company Statoil. "It's a very hot topic because if you could improve the efficiency by a few tens of percent it would be more economically viable," says scientist in charge of ID31, Andy Fitch.

In the past we have obtained energy from very few sources, explains Felici, but in the future we will have to diversify, and the Fischer-Tropsch process is one of the ways to get hydrocarbons from cheap sources that have a lower impact on the environment. "We cannot say if it is the way but it is definitely one of the ways forward," says Felici. The studies taking place at the ESRF are quite recent, and are driven mainly by the facts that the price of oil is going up and fundamental science is heading more towards applied research, he explains. "The gap between our facility and industrial needs is getting smaller."

Matthew Chalmers

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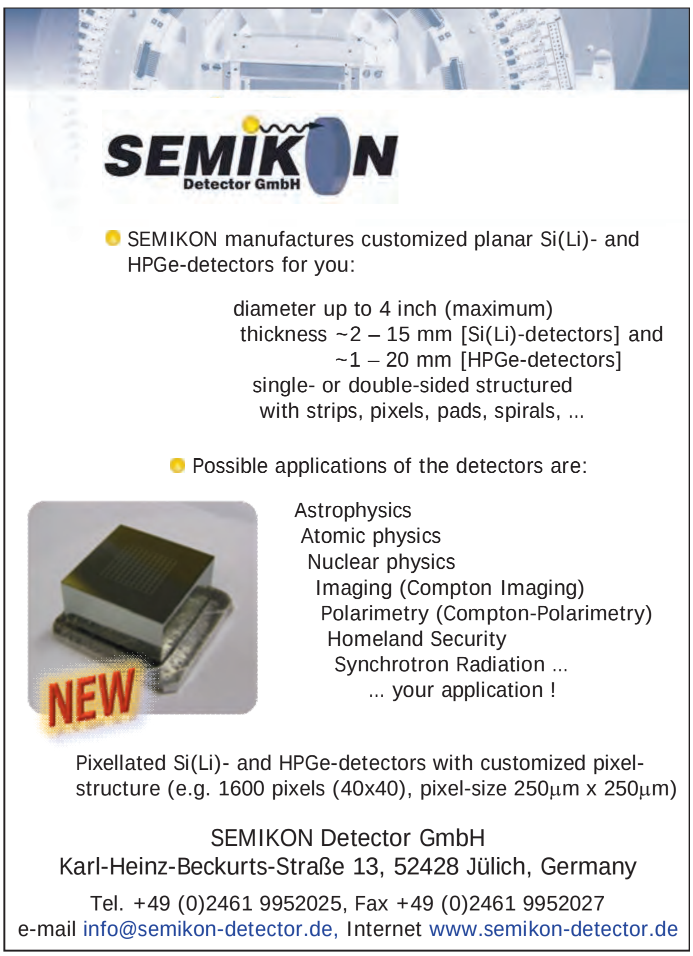

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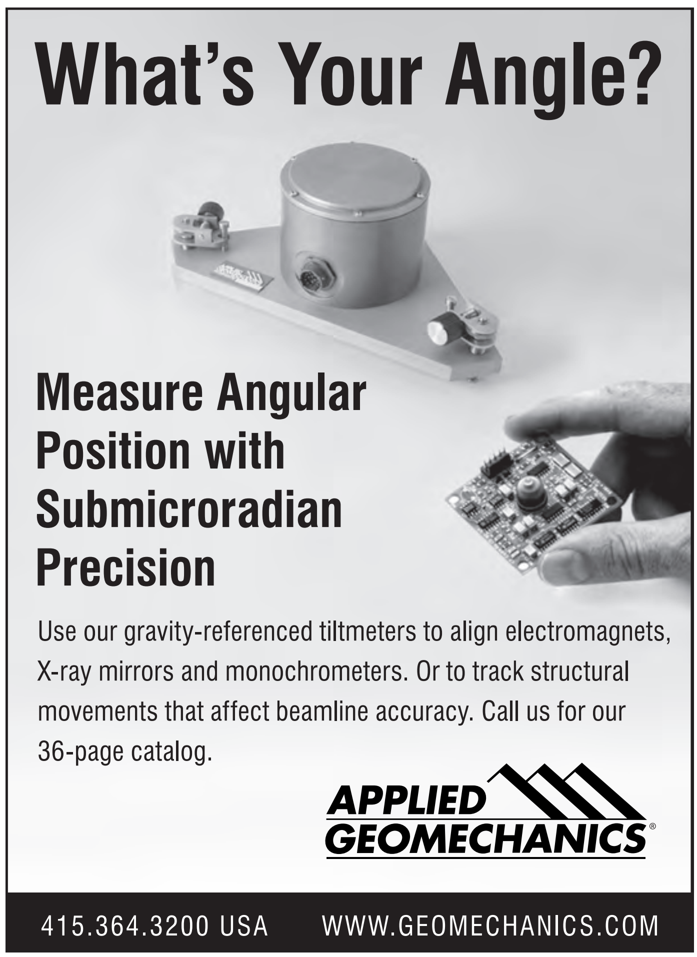

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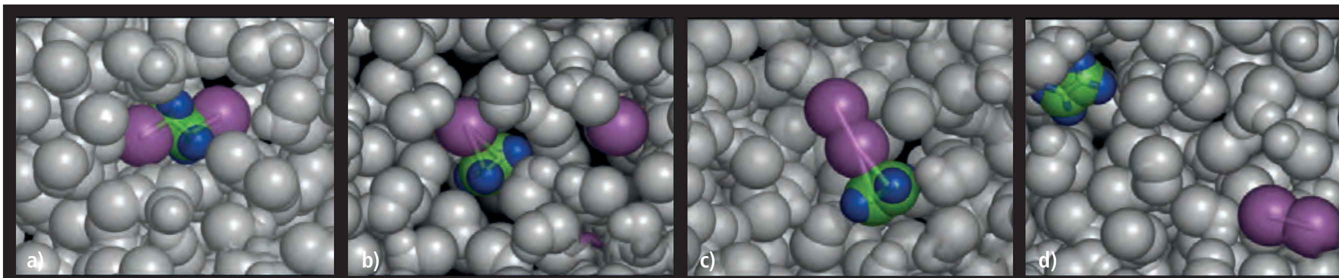

The molecule diiodoethane has two iodine atoms (purple) separated by a cluster of carbon (green) and hydrogen atoms (blue). A flash of light breaks the carbon-iodine bond producing C_2H_2I with an exotic triangular C-I-C bond and a free iodine atom (b). A linear isomer is formed 500 ps later (c) that prefers to liberate itself after 1 μs while ejecting the iodine molecule into the methanol solvent (grey), closing a long standing debate.

Extreme chemistry

Michael Wulff, scientist in charge of the time-resolved beamline ID09B, takes ESRFnews behind the scenes of a chemical reaction.

Do you make films of chemical reactions?
Yes, although they are more like cartoons because we stick together stills taken at controlled intervals. The stills are maps of interatomic distances and require a fair amount of interpretation aided by theory, so you can't simply play it back like a movie and see what all the actors do.

What is chemistry's fundamental timescale?
All chemical bonds vibrate with a characteristic period of 10–100 fs, depending on the types of atoms involved. So this is the fundamental chemical scale because it is the time during which new bonds between atoms form. Binding is triggered by electronic transitions that take place on much shorter timescale of attoseconds, but the atoms need time to fit into the new orbital configuration and this is about 1000 times slower.

What is the shortest snapshot you can take?
About 50 ps, although we have gone as short as 10 ps using tricks to squeeze more out of the data. So we cannot probe the fundamental formation and breaking of bonds but we can understand how the kinetics of the molecular reactions influence the rate and outcome of a reaction. Static studies only give you the initial and final products, not the reaction pathways.

What is the theoretical minimum?
It's governed by the electron bunch length, which in a circular machine cannot be compressed much further due to the dispersive nature of synchrotron radiation. With Phase II of the ESRF upgrade we could gain a factor two in time resolution, but the real revolution will be coherent X-ray scattering and therefore better spatial resolution. X-ray free electron lasers (XFELs) currently offer a resolution of 100 fs and should be able to reach 10 fs.

How does the technique work?
Chemical reactions rarely take place in the solid state because molecules need space to move and find partners. So we start with a solution containing trace amounts of a light-sensitive molecule and send it through a nozzle that produces a thin ribbon of liquid. Then we send a short laser pulse into the centre of the ribbon, which is tuned to a wavelength that dissociates the molecule, and start firing flashes of X-rays at the same spot. We might repeat this 1000 times while recording the scattering X-rays on a detector. The X-ray pulse itself is delivered by two rotating "choppers" that allow X-rays to pass through for a period of 250 ns.

How did ID09B come about?
It was initially built for Laue diffraction on proteins. In 1994 we built a prototype chopper that could select single pulses and a nanosecond pulsed laser, which allowed us to take snapshots of proteins such as myoglobin. To probe chemical reactions, a fully synchronised femtosecond laser and chopper system was built in 1998. We were the first beamline to do this in the world, but there were many technological challenges in synchronising the instruments.

What's the most spectacular result so far?
It was a 3D movie of myoglobin made in 2003, which showed how the protein interacts with a gas such as oxygen. It provided the first view of a protein in action with atomic resolution and the paper has received over 400 citations.

Are chemists the main users?
It's around 50:50 chemists and biologists, with a few physicists too. We cannot address fundamental physics because of the very fast timescales involved. Physics has driven this field, but on these very short timescales physics, chemistry and biology converge into something that you might call femtoscience. For example, in protein reactions the fundamental act is an electronic transition on the attosecond timescale, which is physics, whereas the way that the electron clouds are calculated and new bonds are formed involve a lot of chemistry. This then triggers biological reactions that take place much later.

Are users motivated by particular applications?
They are mainly driven by fundamental interest. For instance, you can take a few atoms and calculate possible molecular geometries and predict many structures, and chemists are interested in comparing this "quantum chemistry" to reality.

What is the Holy Grail in this field?
Watching how hydrogen bonds form and break in water, which is extremely difficult because there is only one electron in a hydrogen atom so it produces virtually no X-ray scattering. Hydrogen bonds are vital in understanding the physics and chemical properties of water, which is essential for life because it is the linking medium in all protein reactions.

Will XFELs put you out of business?
No, for several reasons. Firstly, larger molecules that are important for drug design move on slower timescales even though the primary events are fast. Second, we bridge the gap between synchrotrons and XFELs. We have a beam with perfect stability whereas XFEL beams, at least so far, jump around and make it much more difficult to interpret data. Also, XFELs can presently run one experiment at a time, compared to almost 50 on a synchrotron.

The ESRF expands

Four years after architects were invited to submit their plans, the ESRF upgrade buildings are nearing completion. Major works impacting on the accelerators and beamlines were completed last year as scheduled, and the ID16 satellite building was inaugurated in October.

"Core" and "shell" works on the Chartreuse and Belledonne hall extensions (pictured) are complete, and air-treatment units are now being put into service before the concrete floor is poured. Coordinating a project of this scope is no mean feat, says project manager Emmanuel Bruas. "We had to strive continuously to strike a fine balance between technical compromises and financial and timescale constraints."

C ARGOUD

C ARGOUD

C ARGOUD

The Belledonne experimental hall extension pictured in June (top) and August (middle) last year, and how it looked in January 2013 (bottom).

K MARTEL

The 3 m wide, 3.2 m high granite support for ID20's Raman spectrometer.

ID20 shapes up for summer

The ESRF upgrade beamline ID20, formerly located at ID16, is a new project dedicated to high-resolution inelastic X-ray scattering. It has two end stations each equipped with a state-of-the-art spectrometer: one dedicated to high-resolution resonant inelastic X-ray scattering (RIXS), and the other dedicated to X-ray Raman spectroscopy.

Together these tools offer unprecedented bulk information about samples that will benefit a broad group of users, such as those interested in low-Z materials for applications in lithium batteries and hydrogen storage.

ID20's optics is almost complete: many of the mirrors and monochromators have already been installed and commissioned, while the rest will be installed during the next two or three months. "For the time being the beamline has been commissioned up to the secondary sources that will be refocused at the sample position," says scientist in charge Giulio Monaco. "The last focusing stage requires special KB mirror setups."

The RIXS spectrometer, which will be mainly used to study low-energy electronic excitations in 3d and 5d systems, is ready for operation and is expected to collect its first X-ray spectra before the end of March. The ID20 team will then start to install the Raman spectrometer, which is positioned in a 9 ton granite structure (pictured above) and will be used mainly to measure absorption edges at energies below about 1 keV.

The project has suffered sizeable delays from the manufacturers of the mirrors, says Monaco. "Despite that, we aim to welcome the first users in June this year."

Machine straightens out

The first new 7 m straight section of the ESRF storage ring was installed in January, as part of an ongoing upgrade of the accelerator complex. Longer straight sections allow "canted undulator" configurations, whereby additional X-ray beams are tapped off the storage ring. The storage ring has 32 straight sections that originally were 5 m long. Since 2006, many of these have been increased to 6 m by removing quadrupole magnets at each end of the sections, which were employed in previous lattice configurations.

The point of the 7 m sections is to distribute the radio-frequency (RF) cavities better, freeing up more space for additional beamlines, explains operation manager Jean-Luc Revol. "If we want an extra beamline at ID07, say, we have to remove one cavity from there and put it somewhere else, such as ID23," he says. "We can also put another quadrupole in the middle of a 7 m section, which means we can close the gap in the in-vacuum undulators and increase the photon flux." Changing the local optics breaks the symmetry of the storage ring, but detailed studies undertaken by staff in the Accelerator and Source Division showed that the asymmetry does not adversely affect the beam lifetime.

New vacuum chambers and chicane magnets were also mounted during the shutdown.

Two identical 1.6 m-long canted undulators have been re-installed in the ID23 section and three single-cell RF cavities are to be installed in the space between them during this year's summer shutdown. Replacement quadrupoles with much higher gradients have also been installed on either side of the straight section.

The extensive engineering project was also excellent preparation for future work towards a new storage ring lattice, says Revol, although the arrangement of components will be completely different. The Phase II machine will have 5.8 m straight sections and will be symmetric.

A chemist at heart

Bauke Dijkstra, the ESRF's new director of research for chemistry and the life sciences, finds elegance and beauty in the fundamental reactions between biological entities.

Faced with the choice between studying either chemistry or biology at high school, Bauke Dijkstra did not take long to decide. He wanted to do something with his hands, something practical. Chemistry had a good image at the time, he recalls, having proved important in increasing agricultural productivity and in combating the spread of insect-borne tropical diseases. "I chose chemistry not because of the chemistry teacher, who was not very inspiring, but as a subject in which you can make things, in which you do something new."

He went on to study for a degree in biochemistry at Groningen University in The Netherlands, where experiments with chickens would set him on course to become a structural biologist. His supervisor had found that male chickens injected with a female sex hormone produce certain proteins that in hens would end up in egg yolks. Bauke was tasked with isolating a protein that imports the hormone into the cell and transfers it to the nucleus.

He failed miserably, he recalls, probably because proteolytic enzymes had already degraded the protein before he could even show it existed. "At that time nothing was known about the structure of DNA–protein interactions so I decided then that it would be interesting to do a PhD in structural biology." It was a relatively new field and synchrotrons were yet to make an impact, but in 1977 Bauke visited the DORIS storage ring at DESY in Hamburg where, as a 'parasitic' user he studied an enzyme that degrades membrane phospholipids. "It was obvious from then onwards that synchrotrons would become important in my life."

Bauke Dijkstra in brief

Born: 5 October 1948.

Education: PhD, University of Groningen (1980).

Career: University of Utrecht (1980–1982); University of California at Los Angeles (1983–1985); Associate (1985–1993) followed by full professor (1993–2012) of Biophysical Chemistry, University of Groningen.

Family: Married.

Interests: Reading fiction, listening to classical music.

"I decided to do a PhD in structural biology."

Change of scene

By the time Bauke had swapped the low-lying plains of The Netherlands for the mountains of Grenoble last year to take up his new position as ESRF research director, he led a 15-strong group that have become regular ESRF users over the past 20 years. The group's research focused on enzyme structure with the aim of understanding catalysis. He was motivated by the atomic-scale interplay of reactive groups in the active sites of enzymes, which often results in "very subtle, intriguing and elegant chemistry". In short, it is the chemical reactions between biological entities that drive his research interests. "If you understand a reaction and the high-energy transition states that occur during a reaction, you can use this information for designing potent inhibitors, so you move into the area of drug design."

A seminal result from his group came in the early 1990s when a PhD student was working on an enzyme called dehalogenase, which cleaves carbon–chlorine bonds and is therefore important for decontamination. He realised that you can modulate the activity of the enzyme by changing the pH of the environment and you can even block the enzyme reaction half way through. "When we had a look at the underlying electron density we saw for the first time the covalent intermediary, as predicted – it was very exciting."

Bauke has mixed memories of a period much earlier in his career when he worked briefly at Utrecht University using NMR to determine the conformation of sugars on the surface of proteins. By the time he left Utrecht he had a new definition of NMR: nachtelijke metingen van ruis, which translates as "nightly measurements of noise".

The field of structural biology has changed completely since those early days, he says. "When I was a student we were developing methods all the time, especially computer codes, but today's PhD students don't need to program any more," he says. "They can just jump straight into the science, which is good, but by doing the programming itself you learn a lot about the methodology."

Holistic view

Bauke is still getting to grips with his new role, not to mention the ESRF's extensive beamline portfolio. A typical day is spent attending meetings, but he is keen to continue to find time to do research. "What I like so far is that we as management get a lot of input from the Science Advisory Committee and Council, which is different to a university and stimulating because you get fresh ideas from people outside here."

Although synchrotrons have become vital tools in structural biology, the "real" science still occurs in the labs, he says, where people work hard to produce and purify complexes and get the crystals they need to characterise the interactions between molecules. "It's not enough to publish just the structure of a protein; to advance knowledge you need to complement your research with studies about its function. Indeed, today's publications in top journals are usually based on a combination of techniques."

So, after 40 years of working in structural biology, have his views on the nature of life changed in any way? "No," he says flatly. "You know, life is fantastic but I already had an interest in it. Perhaps in the future people will look at things more holistically."

Matthew Chalmers

Tango takes on industry

Since its creation in 1998 the ESRF's control system, Tango, has helped revolutionise synchrotron performance. A new project aims to make Tango the first choice for industry, too.

"What the web is to CERN, Tango is to the ESRF," says Ed Mitchell of the ESRF's Business Development Office. Tango might not be as famous as the web, but it has become the de facto control system for European synchrotrons. At the ESRF, which has tens of thousands of devices, Tango's software regulates everything from the radio frequency transmitter in the storage ring to the synchronisation of beams with sample stages. Without this unified way to access and control the menagerie of equipment there would be less automation and less feedback, which translates to less stable beams.

Tango has also been widely adopted in other research institutes. It was chosen to control diagnostics at the Laser Mégajoule (LMJ) facility under construction near Bordeaux, which uses lasers to study inertial confinement fusion, and is being used increasingly by other laser projects in France, most recently the ThomX laser-based compact synchrotron light source being built at the Linear Accelerator Laboratory in Orsay. But Tango is general-purpose enough to find applications further afield. A bigger user community will ensure more resources to provide support, bug reports and fixes and improvements, explains the ESRF's Andy Götz, spokesperson of the Tango industrialisation project. "This is the way open source works."

Technology transfer

In order to take Tango outside academia it is vital to involve industry, which provides the majority of control systems currently in use. In 2012 the ESRF applied to GRAVIT, which is a technology transfer unit based in Grenoble financed at a regional, national and European level. GRAVIT's experience has been essential in preparing Tango for industry, and its sponsorship will help finance an industrial demonstrator, says Mitchell. "This project shows the economic value of the work done at the ESRF – especially with the potential beyond synchrotrons to large-scale facilities and to control industrial facilities or factories beyond even this."

It is not in the interests of the community for Tango to be sold commercially, says Götz. Instead, the goal is for Tango to remain open source and to make it freely available, with only additional services such as maintenance being sold. This is a common business model in open source software, with examples including Linux, Android and Apache. By following this route, Tango should enable industry to solve problems at lower cost while simultaneously benefitting from long-term support. In terms of value, Götz estimates that a multimillion euro market per year is achievable.

As well as being open source, Tango offers many benefits for industry including the ability to deal with high data rates, documentation and support for different languages and tools. Tango is one of the few open source control systems available with so many features and such a large number of research institutes using it. "The other control system of this kind is EPICS originating from the Advanced Photon Source in Chicago, but it is less modern than Tango and the European origin of Tango makes it good for European business," says Götz. The aim is to create a foundation to manage and protect Tango, which would be partly financed by industry and initially partly by the European Union. The foundation would hire staff to keep improving the software.

Tango specifications

The basic concept of Tango is a distributed object that wraps heterogeneous hardware and/or software in an efficient manner. Even though this concept has been in use for over 20 years at the ESRF, it is still well adapted to today's needs. New industrial solutions are moving to this concept only now, but they are not open source. Tango's wrappers are implemented in C++, Python or Java and run inside device servers, which provide network services based on underlying protocols in CORBA and ZMQ. This frees the programmer from the network layer so that he or she can concentrate on the code specific to his or her hardware or software. This simple concept is very powerful and can be extended from one device to tens and even hundreds of thousands of devices.

Good timing

The ESRF will continue to act as custodian of Tango on behalf of the collaboration. "The point of taking Tango to industry is not so much about making money for the ESRF but more about fulfilling the ESRF's role in transferring technology to and hopefully creating jobs in industry," says Götz. The ultimate goal is to make Tango an industrial standard. "We think Tango is technically up to it."

The GRAVIT project, which will run until September 2013, is a first step in this direction – and the timing is good, says Jean-Michel Chaize, head of the ESRF's core Tango development team.

"Governments across Europe are actively promoting the use of open source software in public institution or private companies with a view to enhance the competitiveness and the sovereignty of industry," he explains. "Our initiative with Tango is in line with the recommendations of France's Prime Minister and the UK, Germany and Italy are following the same strategy. I am convinced that our development has great potential use for controlling factories or machines in industries of all shapes and sizes who wish to break their dependence from big proprietary software."

Matthew Chalmers

"It is not so much about making money for the ESRF"

SGX SENSORTECH

Multi-element X-ray detectors for beam-line applications

SGX Sensortech have a distinguished heritage in the manufacture of Silicon Drift (SDD) and Si(Li) detectors. Previously known as e2v scientific and Gresham Scientific, SGX specialises in producing detectors from standard designs through customised assemblies to complex multi-element detectors. All detectors are designed to meet the highest specifications, including the largest single active area (100mm^2) available of its kind.

Applications include:

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- Focused or planar sensor arrangements
- Custom collimation
- Application specific designs
- High solid angle
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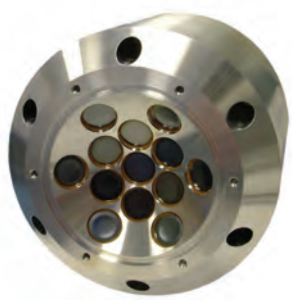
- Slide options: manual / adapted for translation tables



Also available is a choice of close packed 3 x 100mm^2 or 6 x 100mm^2 sensor arrays

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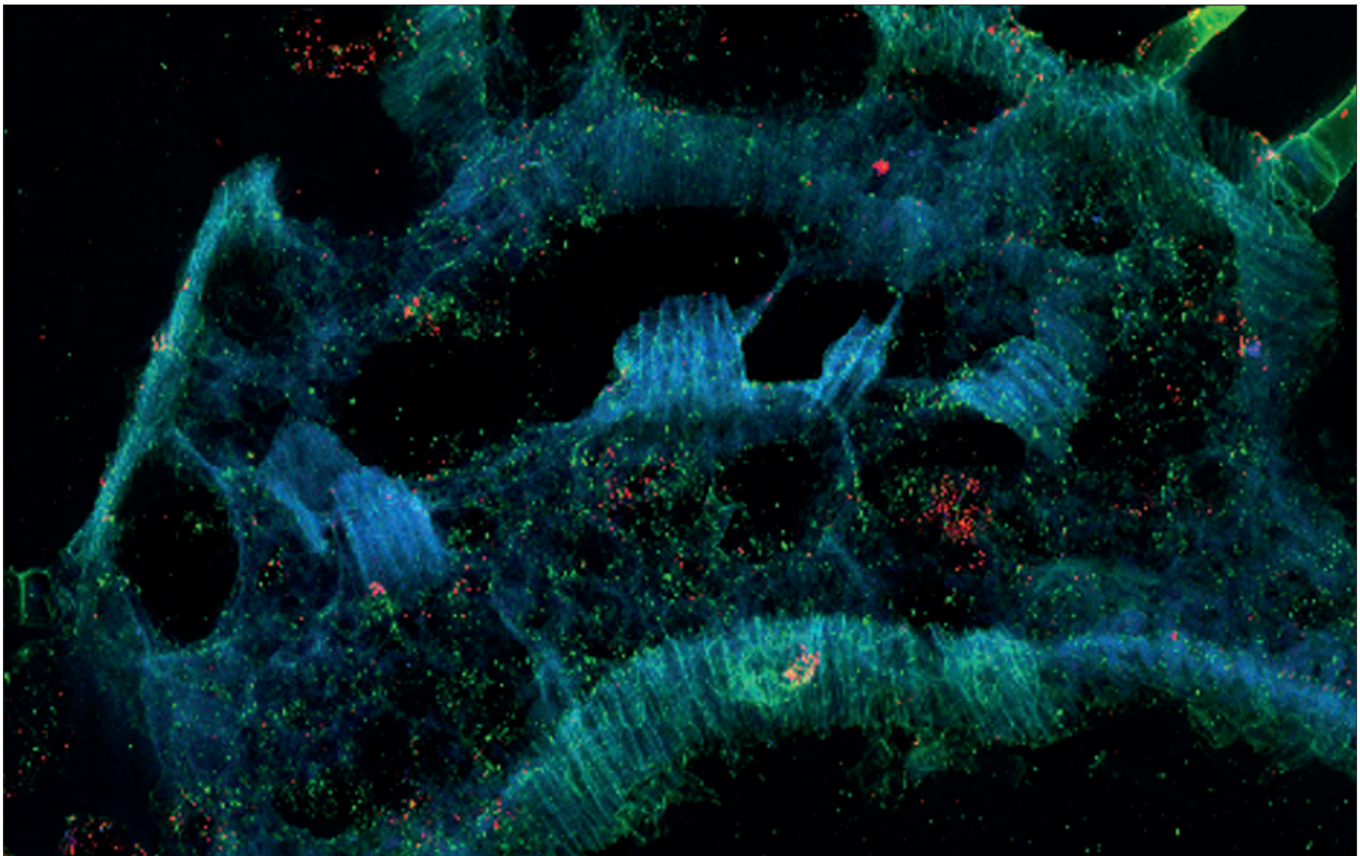


Cucumbers quick on the uptake: This X-ray image (1.1 x 1.5 mm) of a cross section of the leaf of a cucumber plant shows the uptake of titanium dioxide nanoparticles (TiO_2, red). These nanoparticles are widely used in sunscreens, disinfectants, gas sensors and solar cells, so it is vital to ensure that exposure to them is safe. By studying the speciation and distribution of titanium in cucumber plants exposed to TiO_2 nanoparticles using X-ray fluorescence mapping and X-ray absorption spectroscopy at the ID21 beamline, ESRF users from the University of Texas at El Paso found that these nanoparticles can potentially enter the food chain via agricultural products. However, the plants did not show significant signs of toxicity within the exposure time used (*Environmental Science and Technology* **46** 7637).

In the corridors

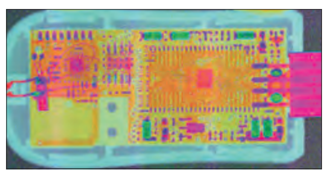
Graphene hits the jackpot

The European Commission has awarded €1 bn over the next 10 years for research into graphene and the same amount for research into the human brain. Graphene – sheets of carbon one-atom thick – has amazed researchers with its range of physical and chemical properties, and project "Graphene" will exploit these in institutes across Europe. Meanwhile, the "Human Brain Project" will create the world's largest facility for modelling the brain, leading to treatments for neurological diseases. "This multi-billion Euro competition rewards home-grown scientific breakthroughs," said EC vice-president Neelie Kroes.

Student summer programme

The ESRF and the ILL have teamed up with Université Joseph Fourier in Grenoble to host a Summer Bachelor Programme from 3 June to 13 July 2013. The programme is open to English-speaking science undergraduate students in their 2nd, 3rd or 4th years at university. Tuition fees amount to €500 and the ESRF and the ILL are offering grants to four students to help meet costs. Students will also benefit from a two-week research placement at the ESRF or ILL. The deadline for applications is 22nd March 2013. (www.esrf.eu/events/esrf-ill-summer-bachelor-programme).

3D X-ray camera



A team at Manchester University, UK, has developed a camera that produces 3D colour X-ray images in near real-time "without the need for a synchrotron X-ray source". The researchers imaged a USB dongle (pictured) in which they identified the elements bromine, barium, silver, tin and zirconium from energy sensitive radiographs and fluorescence patterns (represented by different colours). The device could improve security screening, medical imaging and industrial inspection, claims the team (*Analyst* **138** 755). They are now seeking industrial partners to refine the technology for specific applications.

Tabletop neutrons

Normally, you need a large reactor or accelerator. Now, physicists in Germany and the US have designed a neutron source that would fit into a university laboratory. Short laser pulses ionise a solid target and create a strong electric field that accelerates the ions, driving nuclear reactions in a second target that produce neutrons. Until now, the flux from laser-based sources has been relatively low, but by exploiting "relativistic transparency" the team attained a record yield of 10^{10} neutrons per steradian (*Phys. Rev. Lett.* **110** 044802).