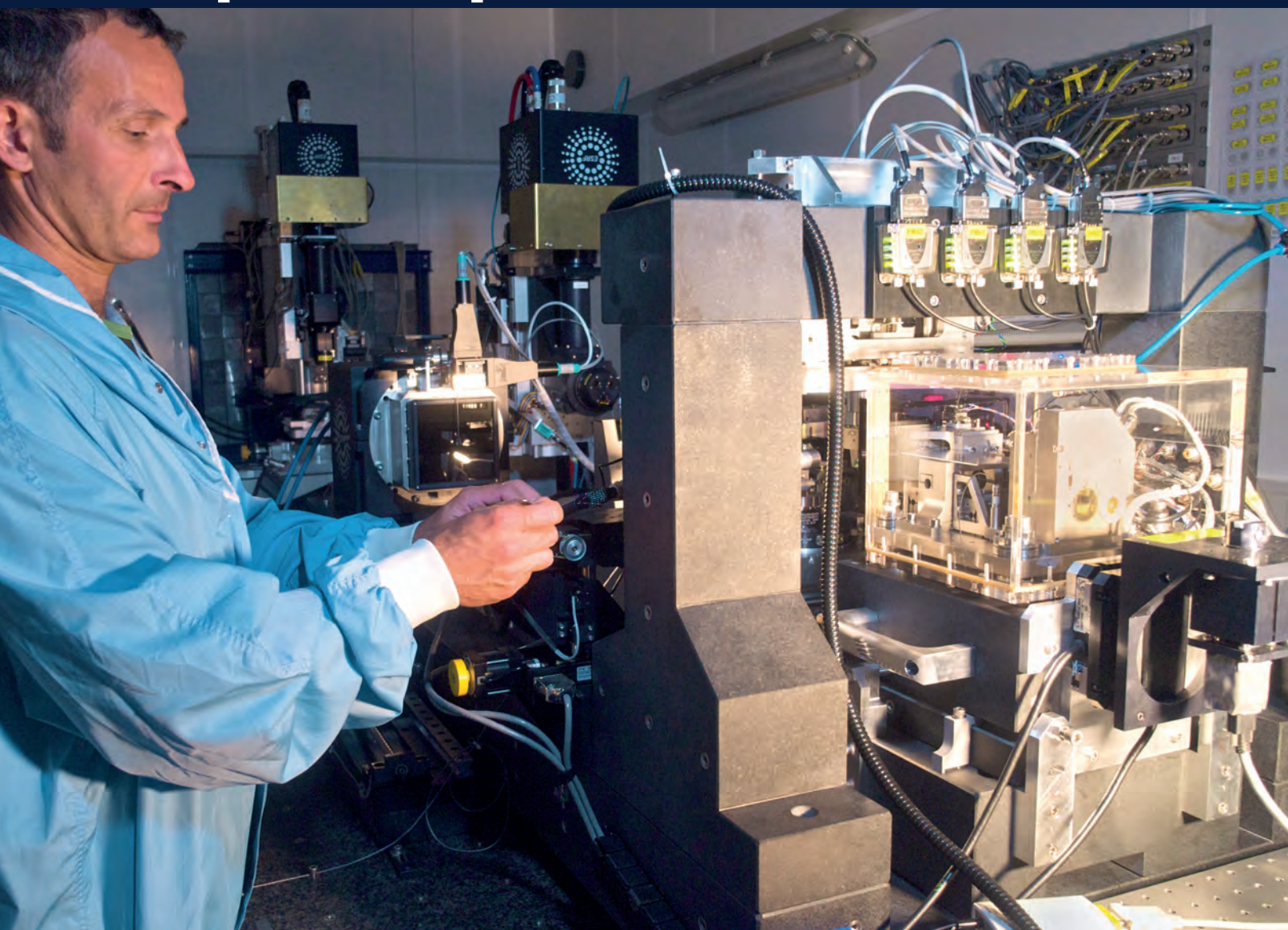


A LIGHT FOR SCIENCE

# ESRF news

Number 61 July 2012

## Special report: instrumentation

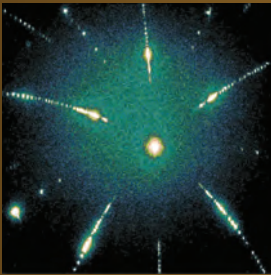


**The ESRF secures its future  
Turning technology into profit**

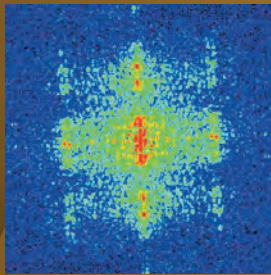


## X-ray Detectors Optimized for Your Applications

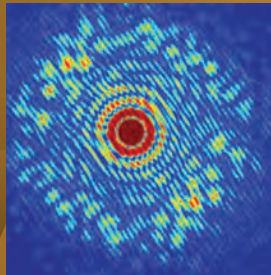
X-ray Diffraction



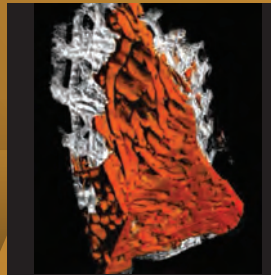
Soft X-ray Diffraction



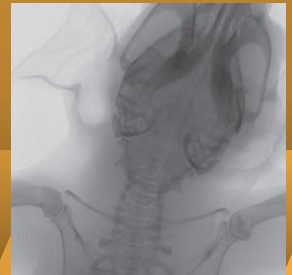
X-ray Holography



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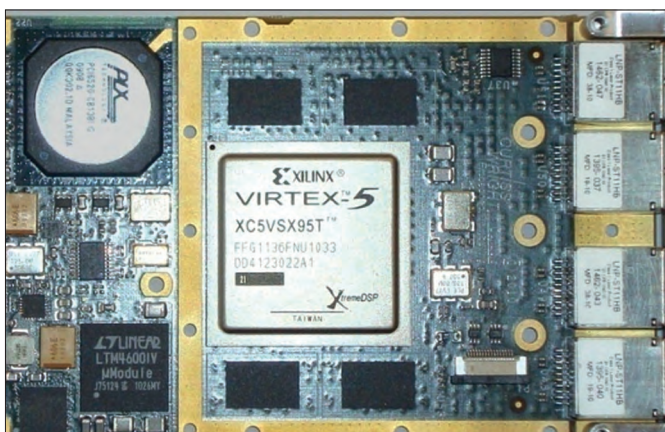


# A light for science



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The art of the undulator, p8.



Fast orbit corrections enhance beam stability, p11.



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ESRF/BLASCHA FAUST

**On the cover:**  
State-of-the-art: nanoprobe end station at beamline ID22 at the ESRF.

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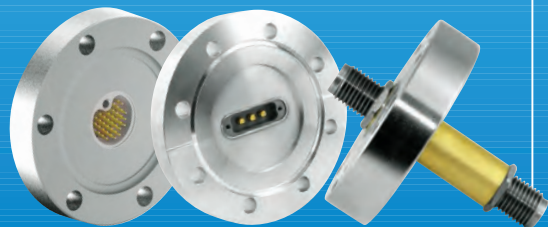
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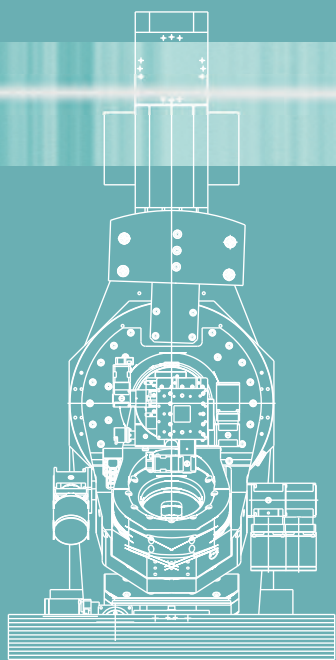
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## New: Modular Beam Conditioning Unit



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Instrumentation and associated enabling technologies are the backbone of every synchrotron facility, underpinning all scientific activities. In the early stages of the ESRF upgrade programme we recognised that the performance of future synchrotron beamlines would depend critically on innovative and successful instrumentation programmes. Furthermore, we reasoned that we must take an integrated approach to the specification, design and implementation of beamlines in order to fully exploit the opportunities offered by the enhanced ESRF source and new experimental hall layout.

Realising that our organisational structure was not fully suited for tackling the many challenges inherent to such a major modernisation of our beamline portfolio, we established a new multidisciplinary strategy that led, in 2009, to the creation of the Instrumentation Services and Development Division (ISDD). The division brings together all in-house experts from modelling to data analysis, including X-ray optics, detectors and electronics, mechanical engineering and instrument control, and now has a “critical mass” of more than 130 experts fully dedicated to instrumentation development and support. In addition, the division is able to promote synergies between different engineering areas for integrated instrumentation.

Since significant human and financial resources are required to develop new instrumentation, we have to make strategic choices in order to focus investment in the most relevant technologies in a cost-effective manner. Standardisation, which is a key challenge when dealing with instrumentation on a large scale, is one example. Although standardisation is often perceived as being contradictory to high-performance, at the ESRF we have adopted a “Lego” strategy whereby generic solutions are developed to offer a high level of versatility in the design of large instruments. In other words the “Lego” pieces can be combined and adapted for a wide range of technologies, each constituting a unique high-performance instrument in their own right. This approach has been proven to be highly economical without compromising performance, especially concerning X-ray mirrors, 2D detectors and software.

Another strategic decision we made was to invest in fabrication capabilities for components, which tend to be a niche market for industry but critical for ESRF applications. Dedicated laboratories and workshops for high-precision mechanics, X-ray multi-layer deposition, light converters for 2D detectors or crystal monochromators and analysers provide secured procurement and the capacity for new R&D programmes. In the most critical areas of instrumentation, these activities are framed by roadmaps that define our long-term strategies.

Cutting-edge instrumentation requires a substantial level of investment, often far beyond the capabilities of a single facility or medium-sized industries, which means that the most innovative developments will increasingly rely on partnerships between sources, institutes and industry. Several examples of such collaboration in Europe that aim to define standards and unify practice, particularly concerning software, are a positive sign of how our community is evolving.

**The ESRF co-hosts SRI2012**

The success of the Synchrotron Radiation Instrumentation (SRI) conference series is itself a recognition of the importance of instrumentation at synchrotron and free-electron laser facilities. SRI is the largest international forum for exchange and collaboration among scientists involving new concepts, technologies and instruments for synchrotron radiation research. Together with the members of the Scientific Programme Committee, we want to highlight not only the new trends in innovation but also the intimacy between instrumentation and science.

This year’s SRI programme reflects this ambition. We are delighted to note that long before the registration deadline the number of registered participants had exceeded 700, and that our technical and commercial exhibition will include more than 70 high-profile companies. These figures indicate the continuous growth in our community and the mutual importance of industry partnerships. The ESRF, together with SOLEIL is proud to contribute to our community by organising SRI2012, which we hope will be a memorable event for all.

**Jean Susini**, head of the ESRF Instrumentation Services and Development Division and co-chair of the SRI2012 Scientific Programme Committee



## Promiscuous plants

Hormones are not limited to the animal kingdom: they are central to the growth, development and defence response of plants. By understanding the molecular basis for plant-hormone modifications controlled by enzymes in the “GH3” family of proteins, processes such as fruiting and seed development could be targeted to help achieve the goal of sustainable development and food production. But the way in which an enzyme is able to selectively bind its substrates and catalyse different reactions has eluded researchers.

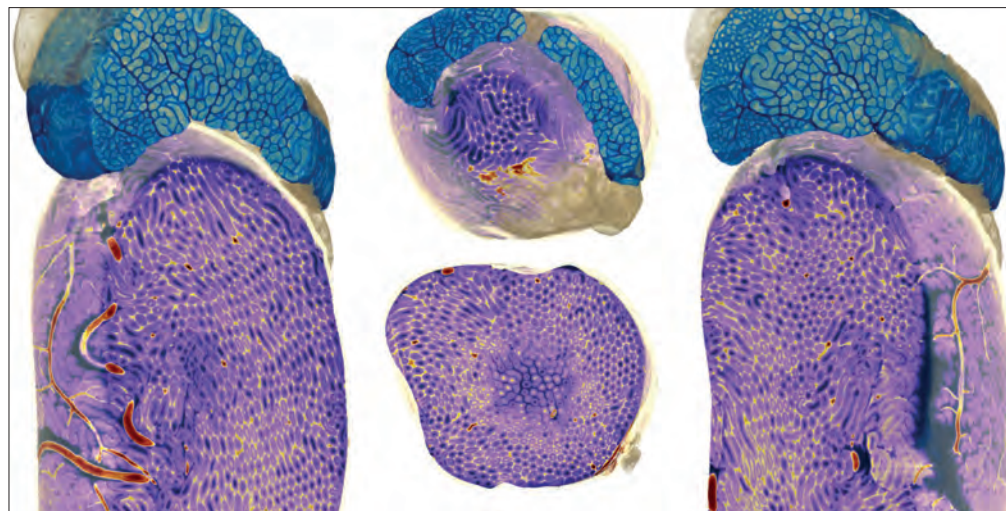
A team from Washington University in the US, the ESRF and the EMBL in Grenoble has now gained structural insight into the catalytic activity of the GH3 proteins. Crystal structures of two different GH3 proteins in complex with substrates and products, combined with extensive mutagenesis studies, allowed the team to elucidate the molecular basis for hormone selectivity, chemical activation and amino acid conjugation. The results demonstrate how a highly adaptable 3D scaffold allows the evolution of promiscuous activity across the crucial enzyme family.

Thousands of different crystallisation conditions were screened using the high-throughput platform of the Grenoble Partnership for Structural Biology (PSB). “The success of the project relied on a multinational collaboration and took advantage of the expertise available at the ESRF, PSB and EMBL, in particular the state of the art beamlines at the ESRF and the biophysical platform of the PSB,” said team member Chloe Zubieta of the ESRF’s structural biology group.

### Reference

C Westfall *et al.* Structural Basis for Pre-receptor Modulation of Plant Hormones by GH3 Proteins, *Science*; DOI: 10.1126/science.1221863.

# Clinical promise for interferometry



**Testes tomography** – 3D renderings of a rat testicle show the epididymis (blue), adipose tissue (yellowish) and vessels (red), in addition to concentric seminiferous tubules.

A team at the ESRF’s ID19 beamline has demonstrated an X-ray imaging method that could drastically improve computed tomography (CT) scans while reducing their radiation dose. The technique, which is also compatible with clinical CT apparatus, combines the high contrast obtained by X-ray grating interferometry with the 3D capabilities of CT.

Irene Zanette of the ESRF and Technical University of Munich and co-workers demonstrated the exceptional resolution of the new technique by imaging various soft tissue from a rat. Within the rat testes, rendered in 3D, minute details such as individual seminiferous tubules are visible. “These structures are simply invisible in standard CT, even in high-resolution set-ups – not only because of their tiny size, but even more so because they hardly give

any contrast”, explains Zanette.

Conventional biological X-ray imaging is limited because it relies purely on differences in the absorption of X-rays by different parts of an object – for instance, cancerous and healthy soft tissue may not show enough contrast to be distinguished clearly. In addition to intensity information, grating interferometry uses information from phase changes to produce “differential phase contrast” images, allowing density differences as low as  $0.5 \text{ mg cm}^{-3}$  to be discerned.

The technique can also yield so-called “dark-field” tomography images, which show the presence of sub-pixel sized fibres, cracks or pores in materials. Zanette and co-workers demonstrated the use of this dark-field signal to image wasp wings that have been fossilised in amber, revealing

features that were mostly invisible in previous X-ray investigations.

The ID19 study marks an important step towards the clinical implementation of grating interferometry – a measurement protocol that the team calls the “sliding window” technique. “We wanted to shorten the gap between the potential offered by this extremely powerful technique and its application in the biomedical field,” said team member Timm Weitkamp of Synchrotron SOLEIL. “Our sliding window method reduces the dose and acquisition time and makes grating interferometry compatible with the continuous rotation of the gantry used in clinical CT.”

### Reference

I Zanette *et al.* Trimodal low-dose X-ray tomography, *Proc. Natl. Acad. Sci. US*; DOI: 10.1073/pnas.1117861109.

## Preserving iconic design

**Sunlight, heat and mechanical stress may cause plastics to become brittle, change colour and degrade. This is of particular concern for the owners of iconic 1960s Italian lamps made from polyvinylacetate – a polymeric material sprayed over a metal frame to create innovative designs with subtle optical properties. Original lamps can**

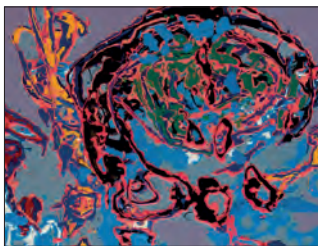
**be worth thousands of euros and are found in international collections and museums.**

In May, a team led by chemists Austin Nevin and Lucia Toniolo from Italy’s Consiglio Nazionale delle Ricerche and the Politecnico di Milano used infrared microscopy at the ESRF’s ID21 beamline to investigate the molecular and chemical changes that occur during the degradation of this polymer coating. Infrared light offers key information about how

**chemical bonds between components may break, where degradation products build up, and how additives evaporate in the composite microscopic structure.**

The team plans to compare microscopic samples from lamps in the Triennale Museum of Milan and from a private collection with polymers that have been artificially aged under controlled conditions, potentially leading to preventative chemical treatments.





## Inspiration-X

What happens when you let four artists loose at a third-generation light source? This oil painting – an interpretation of microtomography X-ray fossil scans at the ESRF – is one possibility. Created by Nina Grúňova from the Czech Republic, it represents a 100 m-year-old fossil wasp generated by superimposing layers of different coloured acrylic paint.

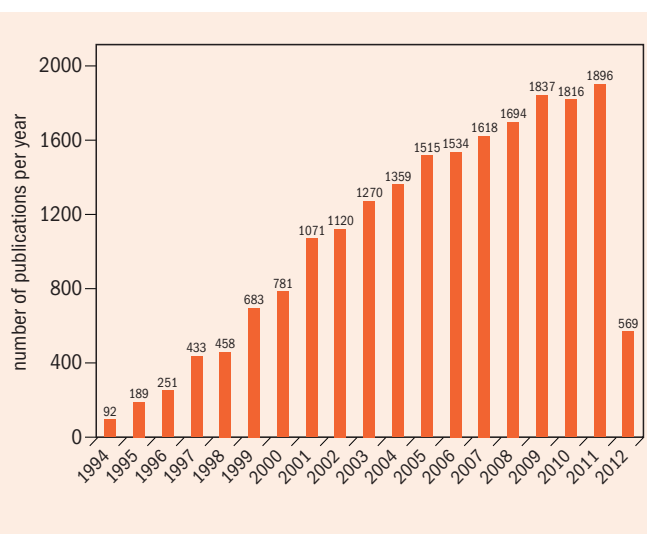
The painting was one of several ESRF-inspired works on display at an exhibition in Berlin inaugurated on 7 February by Helmut Krech of the ESRF and Matthias Petschke of the European Commission (EC). Grúňova and three other European artists spent five days at the ESRF last year observing how scientists go about their jobs, as part of an EC-funded project *Immersion in the Science Worlds through Arts*. For more, see [www.iswaproject.eu](http://www.iswaproject.eu)

# Milestone for ESRF science

The number of scientific publications produced by the ESRF has passed the 20,000 mark, with 20,196 papers having been published in peer-reviewed journals since 1994.

“This impressive figure testifies the importance and impact of the science carried out at the ESRF by a growing community of users coming from many disciplines, from both the countries of Europe and the rest of the world,” said Chairman of the ESRF’s Scientific Advisory Committee, Keijo Hämmäläinen. “No other synchrotron laboratory has achieved such high figures and I expect this growth to continue thanks to the big impact that the upgrade programme will have.”

Each ESRF experiment generates a world-class publication, with regular breakthroughs in materials research, nanotechnology and pharmaceutical science plus exceptional results in the new synchrotron fields of archaeology and cultural-heritage studies. During 2011, one publication every two weeks appeared on average in high



**On the rise – the number of peer-reviewed publications per year produced by ESRF experiments, with the 2012 total pertaining to publications registered before 24 May.**

impact factor journals such as *Science* and *Nature*.

In addition to the quality of the research carried out by users, these numbers reflect the close involvement of the user community with the continual development of the ESRF. Users typically have only a few

experimental sessions per year at the ESRF and limited time to complete their research before returning to their home institutes to interpret the data. Their success therefore depends on the careful planning of experiments, support from beamline staff and the reliability of the X-ray source.

## Users' corner

### Proposal deadline

For the second year running, a record number of proposals (1073) were received by the 1 March 2012 deadline. The next deadline for standard proposal submissions is 1 September 2012 for beam time in March–July 2013.

### New beam time allocation panels (BTAPs)

The BTAPs will be restructured to be based on groups of similar beamlines rather than on particular scientific areas. Each of the 10 new BTAPs will be entirely responsible for the review of proposals and allocation of beam time on its subset of beamlines. A new proposal form will be introduced for the September submission round and users must take care to request the correct beamlines in the electronic part of the form.

The new proposal form will

also ask users to choose the most appropriate scientific area and societal theme for their proposal. Although these choices do not influence which BTAP will review the proposal, they will help the ESRF to monitor its scientific activity.

### News from the beamlines

● **BM05** Two new stages for microtomography have been installed and commissioned, in addition to some new optics, improving the capability and accessibility of tomography experiments for industry clients who use both pink and monochromatic beam configurations. The stages and the software controls used to operate them duplicate the system at ID19, allowing easier operation for users working across both beamlines.

● **ID08** September 2012 will be the last round for proposals for

ID08 before its closure as part of the upgrade programme. The beamline will close during the summer of 2013 but will be fully available for proposals in the coming round. Partial user operation of the new soft X-ray beamline on ID32 is expected in the first half of 2014. A new 9T/4T fast sweeping UHV superconducting magnet will be available during the next round. Please contact ID08 staff to discuss experiments before submitting proposals.

● **ID21** A new X-ray absorption near-edge spectroscopy (XANES) full-field imaging station has been installed. The set-up operates in the 2–9 keV energy range and allows the simultaneous acquisition of up to  $4 \times 10^6$  XANES spectra over large sample areas with sub-micron spatial resolution.

● **ID29** The highly successful Pilatus 6M detector will be

upgraded to Pilatus 6M-F at the end of July, allowing faster frame rates (25 fps) and data collection.

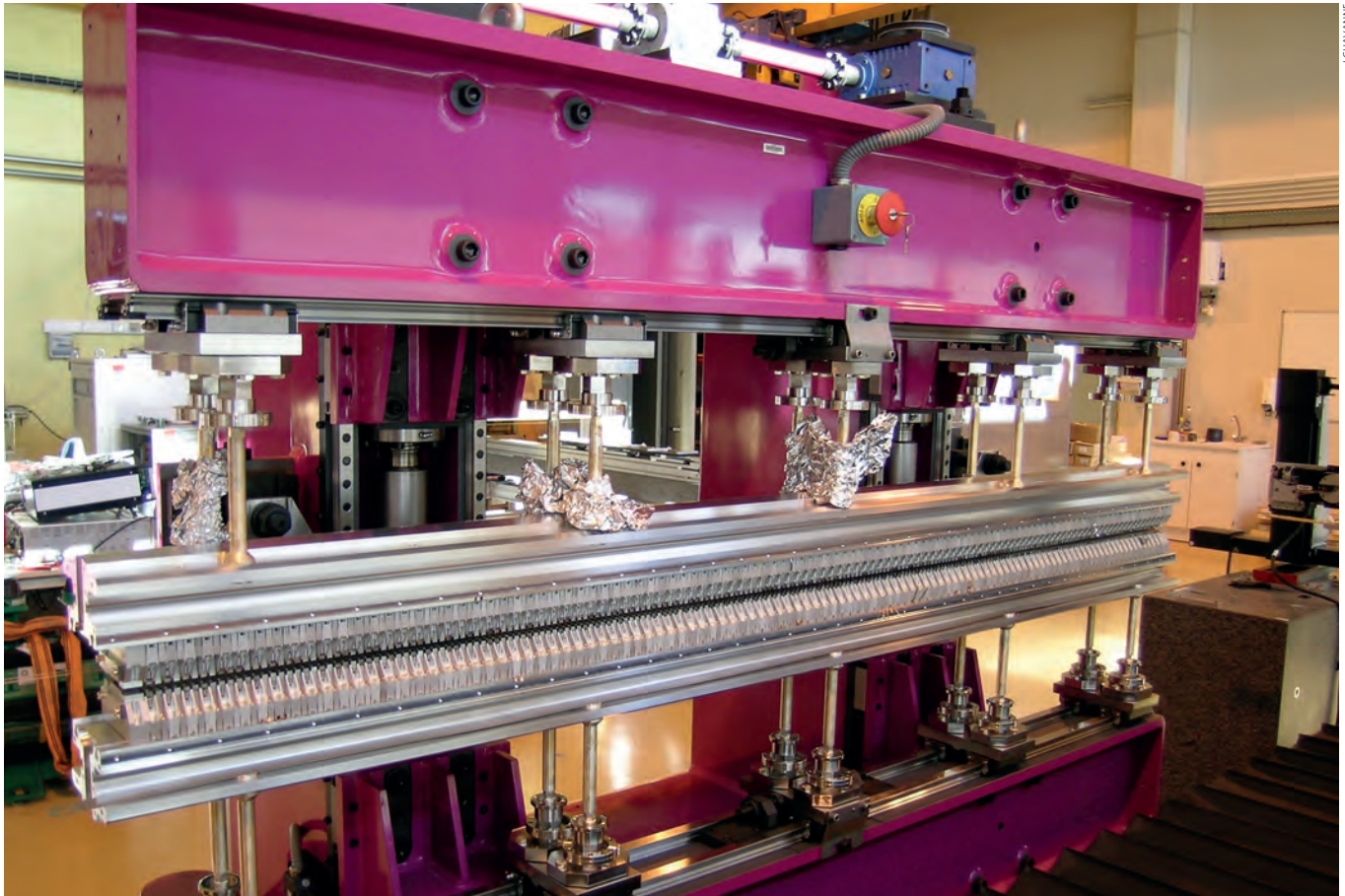
### Courses on site

● A Biostruct-X course “Introduction to Complementary Optical Spectroscopic methods in Macromolecular Crystallography (COSMX)” will be held at the ESRF from 19–21 September, addressing young scientists and students who intend to apply *in crystallo* techniques to macromolecular crystallography.

● A Hercules Specialised Course “HSC14: Neutrons and Synchrotron Radiation in Materials for Energy” will take place on the ESRF/ILL site from 17–21 September, addressing young scientists who wish to learn about the capabilities of neutrons and synchrotron radiation for the production, storage and economy of clean, renewable energy.

# The art of the undulator

Insertion devices are the defining feature of third-generation light sources. *Joël Chavanne* describes how the ESRF continues to push for the brightest X-ray beams possible.



J. CHAVANNE

**A cryogenic permanent magnet undulator undergoing tests in the ESRF Insertion Device laboratory. The 2 m long device contains 110 alternating magnetic periods and when cooled to 145 K can provide a peak field of 1 T for a gap of 6 mm.**

It's hard to imagine that synchrotron radiation, which today underpins thousands of peer-reviewed papers published each year in fields ranging from catalysis to palaeontology, was once considered a nuisance to science. For physicists in the 1950s who were trying to peer deeper into the structure of matter, the emission of photons by charged particles travelling on a curved trajectory reduced the scientific reach of particle accelerators. Researchers therefore did everything that they could to minimise this troublesome X-ray emission.

It did not take long for the potential of synchrotron radiation to be realised, however. First-generation light sources tended to be "parasitic" facilities, whereby beamlines based on dipoles were installed on experiments that were originally designed for particle physics. These facilities were followed by second-generation sources that

were dedicated to synchrotron light, based on radiation from dipoles and more sophisticated "wiggler" magnetic structures. By the end of the 1980s, considerable effort was under way to build third-generation light sources that produced much brighter and therefore scientifically useful X-ray beams.

## Into the light

Spectral brightness or brilliance is a key parameter when evaluating the performance of a light source. For a given wavelength, the brilliance quantifies the smallness of the source together with its capacity to emit a high photon flux in a narrow cone. High brilliance requires electron beams with a very small cross section and divergence, the product of these variables being defined as the emittance.

While second-generation sources had electron beam emittances of around 100 nm

rad or more, third-generation sources aimed at emittances less than 5 nm-rad. In addition to their small emittance, these new machines were to achieve their high brilliance through the use of long undulators and wigglers – otherwise known as insertion devices (see box on p9) – installed in dedicated straight sections. Owing to their capacity to produce narrow, intense and coherent X-ray beams, undulators have become the dominant X-ray sources used in third-generation facilities.

The most efficient and economical way to build the periodic magnetic structures of undulators is to use high-performance permanent magnet arrays placed on either side of the electron beam, which allow the strength of the magnetic field to be tuned by altering the gap between the two arrays. At the time when the ESRF was being built, however, nobody had ever built, installed and operated such devices at a large facility. The technology



therefore had to be entirely invented.

At the ESRF, more than 140 m of free space was available for the installation of various types of insertion devices around the storage ring. Ideally, the magnetic field of an insertion device should be transparent to the electron beam to minimise the impact on the closed orbit, beam size and angular divergence. This is not possible, however, due to various magnetic and mechanical imperfections, so magnetic measurement systems and field correction methods had to be developed.

In 1988, just as construction of the first buildings of the accelerator complex began, the ESRF Insertion Device laboratory was established. Its first task was to construct new types of magnetic measurement benches suitable for the detailed analysis of the magnetic field of insertion devices. By 1993, when insertion devices were installed around the ESRF storage ring, we had established the basis of correction techniques known as multipole and phase shimming. Multipole shimming corrects magnetic errors that disturb the position and divergence, and hence the stability, of the electron beam, for example when the field of the insertion device is changed. Phase shimming corrects a certain class of magnetic errors responsible for degrading the X-ray spectral quality of an undulator.

By the early 2000s our magnetic measurement and correction techniques had found their way to other light sources, including the Swiss Light Source, Diamond Light Source in the UK, Synchrotron SOLEIL in France and ALBA in Spain.

### In-vacuum

A milestone in the ESRF's development of insertion devices took place in December 1999, when the first in-vacuum undulator was installed. With this type of device the permanent magnet arrays are located inside the ultra-high vacuum of the storage ring with a much smaller gap between the magnetic arrays – as low as 4 mm – compared with traditional “in-air” devices, whereby the magnetic arrays are installed around the vacuum chamber in which the electron beam is circulated. In-vacuum undulators therefore allow us to achieve a shorter period and higher peak field, which extends the usable photon energy range of the undulator to higher photon energies. The first test of an in-vacuum undulator on the ESRF ring was carried out in 1996 thanks to fruitful collaboration between the ESRF and researchers at the Japanese SPring-8 facility.

In-vacuum undulator technology at the ESRF has progressively evolved since then, with improvements in both the magnetic assemblies and their support structures, and our work has served many other European synchrotrons. Since early 2000, several medium-energy storage rings have been constructed in Europe all of which rely heavily on in-vacuum undulators. The various developments carried out in the ESRF insertion device group have

also been very attractive to the accelerator magnet industry. Indeed, the ESRF has technology transfer agreements with Danfysik in Denmark and Bruker in Germany that have produced insertion devices now installed at several other sources.

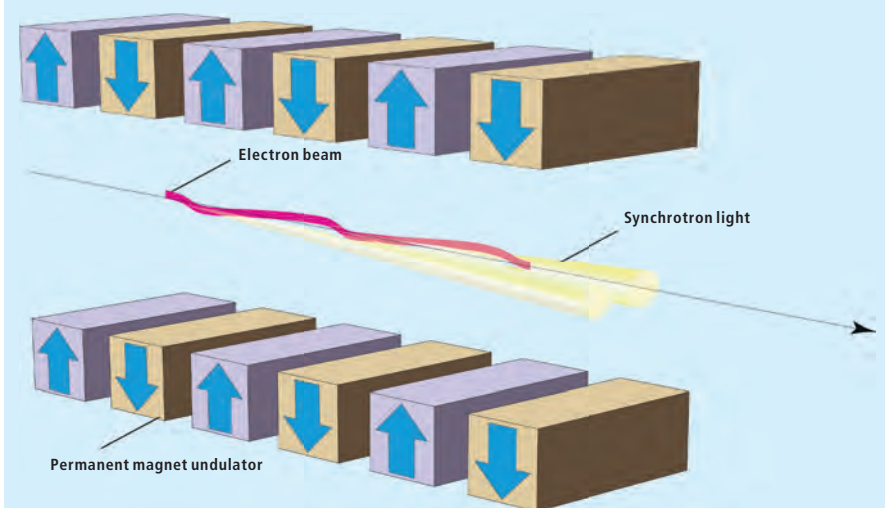
Recently, the ESRF took the next big step in insertion device technology: cryogenic permanent magnet undulators (CPMUs). The concept, which was initially proposed by staff at SPring-8, has been fully developed at the ESRF and in January 2008 the first CPMU was installed. The technology is a logical evolution of conventional in-vacuum undulators. In a CPMU, the in-vacuum magnet array of the undulator is cooled to cryogenic temperatures (around 145 K), which dramatically increases the stability of the permanent material against demagnetisation and increases the field strength for a given period and gap by more than a third compared with classical in-vacuum undulators. Other cryo technologies based on superconducting undulators are being developed at the ANKA source in Germany and the APS in the US. Superconducting undulators offer peak fields comparable to those of CPMUs, although the technology needs to be developed further to reach a mature state.

### Fundamental limit

The future of insertion devices will likely involve further development of cryogenic devices, either with permanent or superconducting magnets. Meanwhile, conventional (non in-vacuum) technology will become more flexible with the use of “revolver-type” devices that can accommodate several different undulators on the same support structure.

However, a fundamental limiting factor in the performance of insertion devices involves the electron beam itself: namely reducing the emittance, and therefore increasing X-ray brilliance, to benefit more from the coherence of the light produced by undulators. Future significant steps forward in light source technology therefore mean reconsidering the technology of accelerator magnets as a whole – with new, stronger magnets being installed on storage rings, for instance. Until that time, undulators will continue to provide synchrotron science with the brightest and most coherent X-rays possible, and the ESRF will continue to pack even more of them into the storage ring in longer straight sections for new and upgraded beamlines.

## In step: the origin of synchrotron light



Undulators and wigglers are specialist magnetic structures known as insertion devices, which apply a periodic transverse magnetic field to an electron beam. This causes individual electrons to oscillate and therefore emit light, or X-rays in the case of relativistic electrons. The properties of the emitted radiation depend both on the magnetic field of the insertion device and the characteristics of the electron beam itself. Wigglers produce an intense but incoherent X-ray beam, while undulators create much narrower and therefore much more intense beams with a high degree of coherence at discrete but tuneable wavelengths.

Coherence is a key feature of third-

generation sources: the practical energy range of photons produced by an undulator is governed by constructive interference, which gives rise to the existence of high brilliance light at discrete energies that are harmonically related. Compared with undulators, wigglers have much higher fields and periodicities, reducing interference and resulting in a smooth spectrum of radiation similar to that produced by bending magnets.

The first undulator was built in 1952 by Austrian engineer Hans Motz and co-workers at Stanford University in the US, building on the original idea from Russian Nobel prizewinning theoretical physicist Vitaly Ginzburg in 1947.

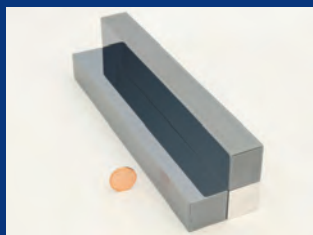
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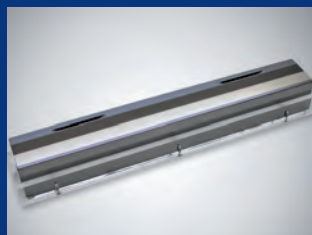
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# Electrons on track

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The ESRF electron beam in the transverse plane following reduction of the vertical emittance to as low as 4 pm-rad.

A new beam orbit correction system at the ESRF kicks electrons into line at a rate of 10,000 corrections per second, taking beam stability to record levels.

One of the defining features of a third-generation light source is an electron beam with an extremely low emittance. In other words, one in which the electrons are confined to a small distance and have nearly the same transverse momentum. But experiments can only take advantage of the small spot size on the sample if the orbit of the electrons in the storage ring is stable within a fraction of the photon beam size, which for some ESRF upgrade beamlines is as low as 10  $\mu\text{m}$ . To achieve such stability, beam position monitors (BPMs) located around the storage ring calculate an orbit correction that is applied by a set of corrector magnets.

The present version of the ESRF's beam orbit correction, implemented in 2009, uses 224 BPMs equipped with Libera Brilliance electronics and 96 corrector magnets. This set-up provides a position resolution of a fraction of micrometre compared with several micrometres using the electronics implemented in 1992 when our storage ring started up.

But today's experiments demand even higher beam stability. In particular, a major cause of orbit distortion is now the frequent changes of parameters in insertion devices, such as their gap and phase, due to increasingly creative ways of operating the beamlines. Given the obsolescence of some parts of the old system, and the availability

inside the Libera Brilliance units for the main components of the new system, we decided to go for another major upgrade.

#### Fast sampling

In May 2012, the ESRF orbit correction system underwent a radical improvement that increased the rate of orbit corrections from one every few seconds, as it was previously, to 10,000 per second. This required three key additions to our orbit correction system.

- The first is new set of wide-band power supplies able to drive the corrector magnets with a bandwidth of 500 Hz.
- Second, we built a fast data network that is able to broadcast the position data measured by the Libera modules (and the correction kick data) over the full system at a rate of 10 kHz via a network of high-rate optical fibres linking the Libera crates and all the power-supply controllers. The protocol managing this data flow (the "communication controller") was developed at the Diamond Light Source.
- Third, we installed eight power-supply controllers connected to the communication controller and to the data ports of the power supplies. These units are equipped with powerful FPGA (field programmable gate array) digital signal processors that compute every 100  $\mu\text{s}$  a new set of correction kicks derived from the position data.

The ESRF's orbit distortion now will be damped with a bandwidth going up to 150 Hz, as before, but with an accurate correction at the location of every BPM, which could not always be the case in the past. Crucially, it will make the effect of the transient parasitic kicks that occur during the change of the parameters of some insertion devices unnoticeable for the users working on the rest of the ring. The latter has been one of the biggest sources of instability in the source over the last few years of operation.

Before the beam orbit correction upgrade, vertical steps of more than 5  $\mu\text{m}$  could be observed over the two cells surrounding some badly corrected insertion devices. But recent tests have demonstrated sub-micrometre stability of the beam position over the 0–1 kHz range at all points around the storage ring – even during the severe cycle of undulator gap changes during and after storage ring refills.

Thanks to this upgrade the stability of the ESRF beam will reach that of the best third-generation sources in operation today. In addition, we expect that this new system will be a powerful diagnostic tool, allowing us to measure and correct imperfections of the ring lattice and to tune the storage ring optics in order to deliver beams with new record low emittance in the near future.

*Eric Plouviez*

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Change ahead: a movable detector for small-angle scattering studies on the ESRF's ID02 beamline.

# Channelled detector vision

While insertion devices, optics and sample-conditioning instrumentation are crucial for making possible the most demanding synchrotron applications, X-ray detectors have the responsibility for performing the actual measurements at the heart of the scientific problems under study. This is particularly challenging in facilities such as the ESRF, where the energy of the photons and the high flux of the beam make hard X-ray detection a complex instrumental problem. Pushing the efficiency and dynamic range of the measurements has been the main driving force for all detector developments at the ESRF so far. Very high spatial-resolution scintillators, high-performance CCD cameras equipped with optimised optics, fast-counting avalanche photodiode detectors or very high frame-rate hybrid pixel detectors are excellent examples of these efforts.

The know-how acquired at the ESRF during the last 20 years is being implemented during Phase I of the upgrade programme, by building state-of-the-art detectors specifically designed for the new beamlines. In addition, the ESRF continues to invest in detector technologies such as high-efficiency sensors for direct and indirect X-ray detection, advanced optics or new hardware and software for fast data acquisition. Extensive in-house expertise in these key technologies is fundamental for the ESRF because they will guarantee the continuous enhancement of current X-ray detectors.

But the ESRF developments should not be limited to incremental improvements of existing instruments. Phase II of the ESRF upgrade, beginning in 2015, represents an extraordinary opportunity to develop new

Decisions taken now about which advanced detector technologies to pursue will set the scene for synchrotron science in 2020 and beyond, argues *Pablo Fajardo*.

kinds of detectors that will push performance well beyond the limits of existing devices. 2D detectors able to produce the same data quality but operating at higher frame rates one or two orders of magnitude faster than current ones, and large-format diffraction sensors designed to provide simultaneously single-photon sensitivity and high quantum efficiency for experiments at very high photon energies, are examples of the type of instruments that will open the path to scientific opportunities that are not in reach today.

#### Detector roadmap

Work carried out within ESRFUP, the European Union FP7 project that has supported the preparation of the ESRF upgrade, has allowed us to identify and evaluate new development lines that are the basis of the proposed ESRF detector roadmap. The foreseen projects include advanced area detectors based on integrating active pixels (whereby every pixel includes some dedicated processing electronics) and implementing technology already validated for applications at X-ray free electron lasers and used in other scientific

areas but which is so far unexplored at storage rings. Two of the projects are quite specific for detection of high-energy photons for fast X-ray imaging and high-energy scattering experiments, while another one aims high-sensitivity X-ray detection with small pixels (less than 30 micrometres) that will push the instrument resolution in coherent imaging experiments as well as in energy dispersive inelastic scattering set-ups.

It is also vital that the ESRF maintains and develops its expertise in photon-counting hybrid pixel detectors, but in a way that complements rather than competes with the already numerous and overlapping initiatives that are being carried out by other laboratories and commercial companies. An unexplored direction that could have a tremendous impact on future synchrotron science is the construction of pixel detectors with optimised read-out for time-resolved measurements in the micro and sub-microsecond region – a timescale where third-generation storage rings are not defied by the upcoming fleet of X-ray free electron lasers.

Designing, building and integrating X-ray detectors are challenging projects that require substantial investment in resources, as well as high doses of innovative technological know-how. Moreover, since developing new state-of-the-art detectors involves development times that are of the order of a decade, initiatives and decisions taken today by the ESRF and by other synchrotron radiation laboratories will have a fundamental impact on the X-ray detection scenario and capabilities necessary for advanced scientific research at storage rings in the period 2020–2030.

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# Scintillating science

The ESRF's liquid-phase-epitaxy lab cooks up high-spec X-ray converter screens.

Scintillators are synchrotron users' windows onto the source, transposing X-rays to the realm of the visible so that they can be detected with CCD or CMOS cameras. At the ESRF, scintillator converter screens made from advanced materials sit behind the sample stages of most beamlines, soaking up roughly 50% of the energy of the X-ray beam and re-emitting it in the optical region to offer volumetric data from samples across fields ranging from biology to material sciences.

The sub-micrometre resolution and high image contrast demanded by today's experiments require scintillator films with a thickness between 1 and 20  $\mu\text{m}$  as well as providing high optical quality and uniformity. Thin scintillators minimise degradations due to focusing defects, diffraction and spherical aberrations, therefore boosting the image contrast and resolution. But the sophisticated techniques required to grow such crystals and their high specificity means that very thin single crystal film scintillators are not commercially available.

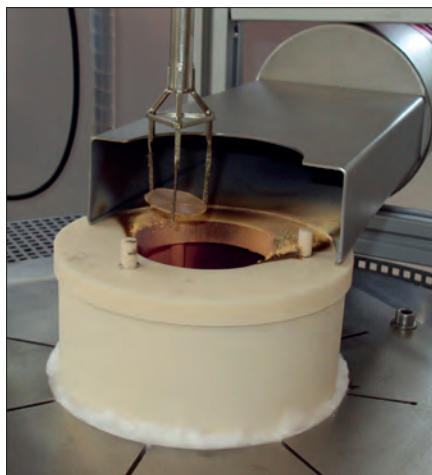
## One of its kind

In April 2010, the ESRF established a unique liquid phase epitaxy facility for single crystal film scintillators. Liquid phase epitaxy is a relatively simple technique whereby semiconductor crystal layers are grown on a solid substrate immersed in a melt. Its key appeal for synchrotrons is that it produces a homogeneous distribution of dopant activator ions within the film (the host material absorbs X-rays and the dopants emit light).

"We are the only people doing this – no other synchrotron does it," says Thierry Martin of the ESRF's Detector Unit. "For us the facility is crucial because there is over 10 years of experience at the ESRF in this area."

The smart 40 m<sup>2</sup> laboratory is divided into three sectors: a chemistry area for powder and substrate preparation, a separate "lead oxide" room in which toxic powders are handled, and a furnace area. The pleasant view of Grenoble's mountainsides is soon to be blocked out by the Bellefontaine experimental hall extension.

The first step in the scintillator fabrication process is to mix the ingredients for the



A single crystal film scintillator after growth and melt characterisation above the furnace.

**"It's like being a chef and adjusting the seasoning of a dish."**

melt, the bulk of which comprise powders of lead oxide and boron oxide as solvent plus gadolinium oxide, gallium oxide and roughly 2% of rare earth dopants such as terbium oxide or europium oxide. "It's a bit like being a chef adjusting the seasoning of a dish," says Martin when describing the task of tweaking the precise proportions of each element to achieve the best light emission.

The powder mixture is then transferred to a rubber sock and compressed at pressures of 2000 bar until it turns into a solid, maximising the amount of material that can be loaded into the platinum crucible of the furnace.

On being heated to a temperature of 1000 °C, the solid mixture turns into a liquid and the crystal growth procedure can begin. A circular gadolinium gallium garnet

substrate 170  $\mu\text{m}$  thick and 2.5 cm in diameter is positioned at the base of a three-pronged stick and dipped into the orange-coloured melt. This "seed" is rotated until, after 10 minutes or so, the film has grown on the top and bottom, and a scintillator layer 1–50  $\mu\text{m}$  thick is born. To make it as transparent as possible to visible light, the layer must have the same lattice structure as the substrate.

The crystal is then cleaned and polished at an external facility before finally being adapted for the specific optics and cameras of beamlines and cut into 8 × 8 mm squares. The growth process takes just one afternoon, in addition to about a week of preparation, and the same furnace melt is used for a month or so – producing around 50 screens. During fabrication, samples are extracted from the melt every now and again and taken upstairs to an X-ray test lab to check the optical properties. "The whole process is simple in terms of a few steps, but lots of care and safety precautions are required at each step, in addition to quality control," says Martin.

## Micrometre resolution

To achieve spatial resolution in the micrometre range (the highest resolution obtained is 400 nm, which corresponds to the diffraction limited system with visible light), the ESRF is now developing more efficient scintillators doped with exotic materials with higher density and higher atomic number, such as perovskite, hafnate and lutetium. Although such materials promise higher absorption efficiency than today's garnet- and orthosilicate-based materials, they are much less well understood. The team plays with these dopants to get good emission of light while making sure that it is compatible with the camera.

"We have been doing this for 15 years and learned a lot," says Martin. "Most of our products are used in-house because there is lots of imaging activity at the ESRF. There is no spin-out company but our products are available to buy, and collaborations would be welcomed concerning substrates and the growth of exotic materials." *Matthew Chalmers*

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# X-rays direct

The ESRF's in-house pixel detectors provide synchrotron users with faster image sampling at higher energies.

The majority of synchrotron experiments detect X-rays using the same technology found in digital cameras: semiconductor CCDs (charge-coupled devices), which must be coupled to an X-ray–optical converter. CCD detectors can be configured to provide a range of field sizes, spatial and temporal resolutions, but read-out noise and scattering during optical conversion limit their dynamic range and therefore applicability to techniques such as SAXS or GISAXS.

Although these limitations can be partly overcome by direct-detection CCDs that receive X-rays directly and integrate the resulting electric charge, as used in X-ray Photon Correlation Spectroscopy, this comes at the cost of slower read-out and a shorter sensor lifetime.

This situation prompted staff at the ESRF to develop a new technology based on pixel detectors, which offer faster read-out and higher dynamic range by converting all the X-rays directly into electronic signals. "Still many beamlines work with CCD detectors but for some applications you need a noise-free signal," explains Cyril Ponchut of the ESRF's Detector Unit. "We anticipated the needs of beamline scientists – we knew what the limitations of their detectors were, so that was the trigger for the MAXIPIX project."

The ESRF's MAXIPIX detectors, a semi-commercial product based on high-quality semiconductor sensors connected to the MEDIPIX-2 photon-counting read-out chip developed at CERN, are able to capture X-rays at a rate of 1400 frames per second. Combined with their small pixel size of 55  $\mu\text{m}$ , MAXIPIX detectors complement existing large area photon-counting systems such as PILATUS or XPAD by providing higher spatial and temporal resolutions but for smaller detection areas.

## Higher efficiency

Ponchut and colleagues are now pushing the detection efficiency of pixel detectors further by turning to less common semiconductors. MAXIPIX uses silicon, which is transparent to X-rays above 25 keV, but the higher atomic number of cadmium telluride (a group "II–VI" semiconductor) gives good absorption efficiency up to 100 keV. This opens the door to new applications of pixel detectors, in



## Going commercial: pixel detectors at the ESRF

The MAXIPIX project started out in 2005 and the detectors are now produced by the ESRF in a limited commercial capacity. The main advantage of MAXIPIX compared with other pixel detectors is its small pixel size (making it well suited for XPCS and inelastic scattering experiments) and its high frame rate (which extends the capabilities of time-resolved experiments). MAXIPIX detectors are also compact, and the devices have made an impact on the following ESRF beamlines:

- ID01: allows single photon detection for coherent diffraction experiments at small pixel sizes.
- ID03: installed on diffractometer arm to

collect surface diffraction data, avoiding time-consuming scanning sequences in reciprocal space.

- ID10: replaced direct-detection CCD cameras to extend time resolution to the millisecond range.
- ID13: enables micro-SAXS experiments that require accurate frame triggering, noiseless detection and small pixel size.
- ID16: allows detection of very low X-ray flux at significantly increased energy resolution.
- BM05: used for characterisation of multilayer mirrors at grazing incidence.
- BM32: used for SAXS and surface diffraction experiments.

particular for materials science and possibly medical imaging. "Today experiments in that energy range use integrating detectors and suffer from noise," explains head of the ESRF's detector and electronics group, Pablo Fajardo. "Moving to photon counting would produce noise-free detectors that would be a tremendous improvement for basically all applications."

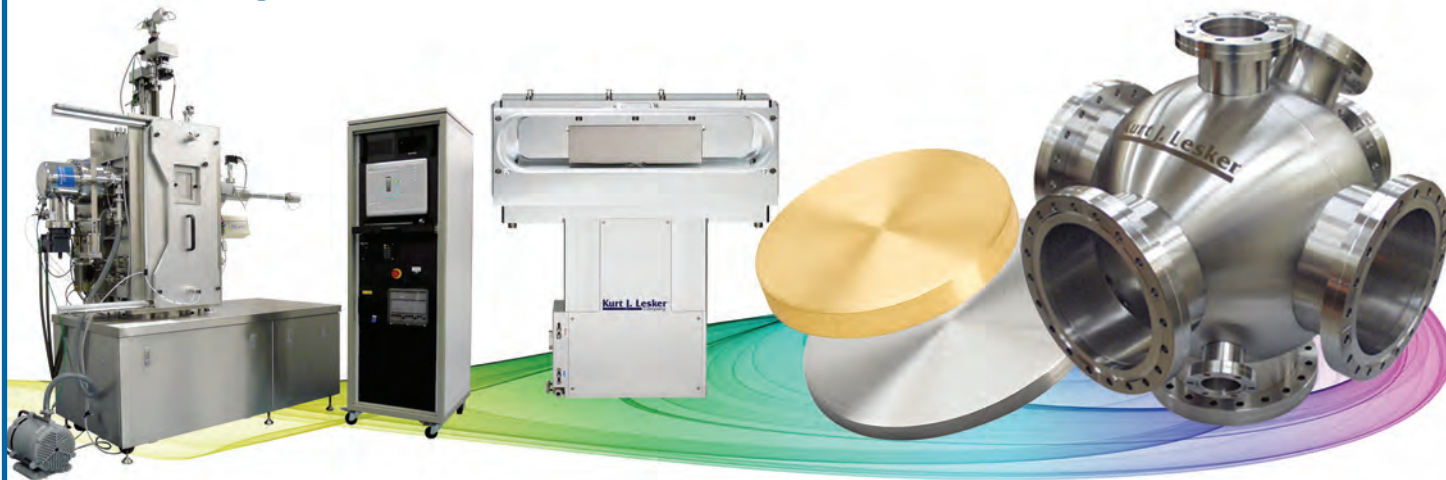
In August 2011, a European study called HIZPAD (high-Z pixel array detectors) coordinated by the ESRF concluded that cadmium-telluride (CdTe) and cadmium-zinc-telluride (CZT) are the most promising active materials to extend the energy range of pixel detectors. The higher absorption of these semiconductors will also improve radiation hardness and thus the read-out chip lifetime at all energies.

The limitation in the frame rate of pixel detectors is determined by the read-out speed of the pixel read-out chip. "MEDIPIX2 was the fastest chip available when MAXIPIX project was started, but with new read-out chips such as MEDIPIX3 now becoming available we can

gain one order of magnitude in frame rate," explains Ponchut. "It will take one or two years to turn these new chips into fully functional beamline detectors, but speed is not the only point: new chips also bring new features such as the possibility of energy-resolved imaging, and there is also a significant effort towards improving the integration of the pixel sensor to the read-out chip in order to be able to build large area detectors from smaller module units with the smallest possible dead gap between modules edges." Regarding sensors, he says, the key challenge now is to improve the quality and the fabrication yield of CdTe or CZT high-energy pixel sensors in order to make them a viable alternative to silicon.

MAXIPIX detectors have already been exported to the UK's Diamond source, DESY and, soon, SOLEIL. "MAXIPIX raises a great interest in the synchrotron community but at the moment it is difficult to foresee what the demand will be in the long term," says Ponchut. "This makes it risky to invest in higher production volumes just yet." *Matthew Chalmers*





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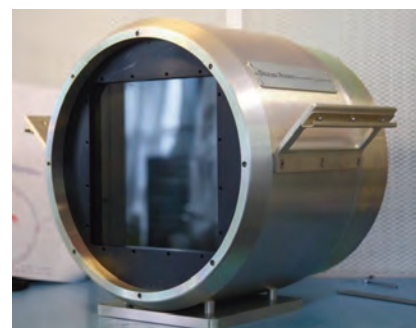
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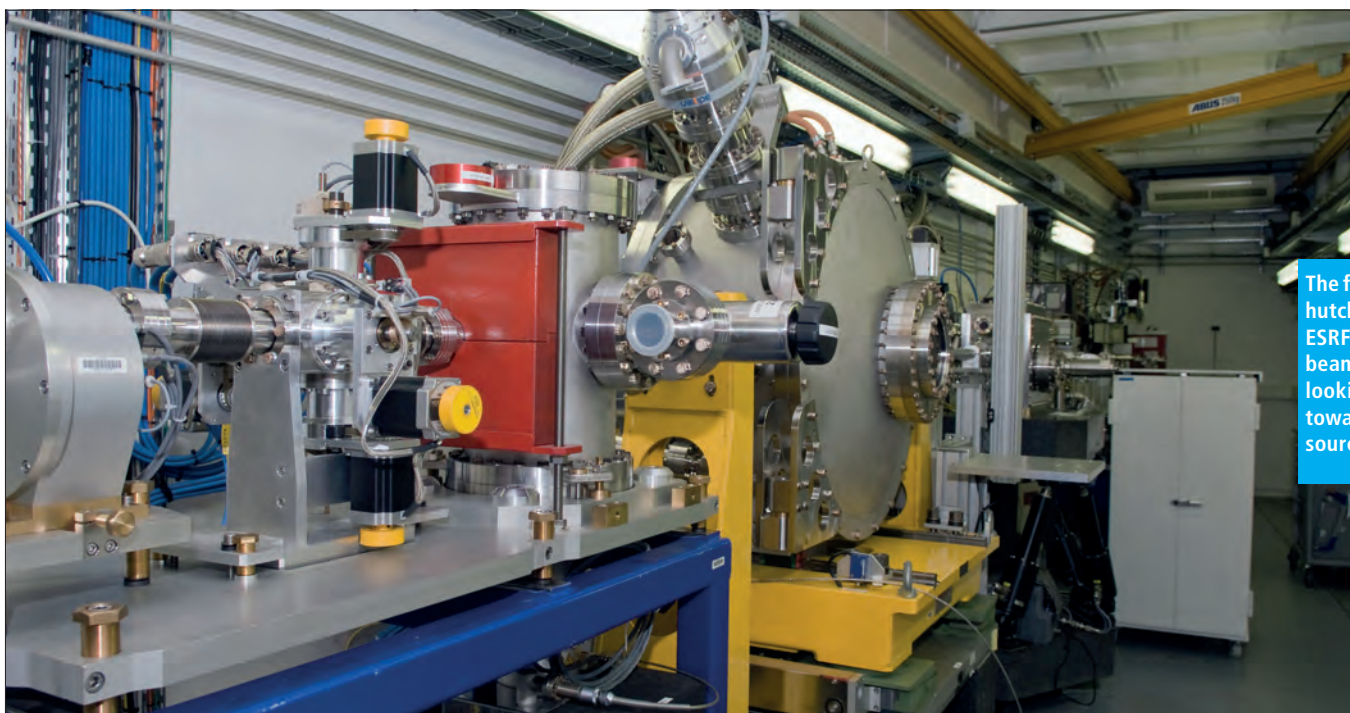


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# Evolution by optical

Precise modelling of the transit of X-rays from the storage ring to the sample is vital to maximise the source, explains the ESRF's *Ray Barrett*, and enhanced optical design is critical for the beamlines of the ESRF



CARGO/ESRF

The first optics hutch of the ESRF's ID06 beamline, looking towards the source.

In the 1987 ESRF foundation report, more commonly called the "Red Book", the proposed beamlines typically aimed for a minimum beam size of 10  $\mu\text{m}$ . A quarter of a century later, the target beam sizes for the ESRF upgrade and refurbished beamlines are typically 10–100 times smaller thanks to improvements in the source and optics, with some beamlines aiming for routine operation with beam sizes approaching 10 nm. Beamlines also demand that the beam characteristics be tailored to match the requirements of experiments, which entails a delicate compromise between factors such as flux, beam dimensions and energy band pass.

Since the cost of constructing or refurbishing a beamline is significant, it is vital to be able to model the beamline performance in order to evaluate different optical configurations and predict the effects of imperfectly aligned optics.

One of the primary tools for modelling beamline performance is ray tracing. In this "incoherent" modelling technique, which does not take into account photon interference effects, the trajectory of individual rays is

tracked from the source, through the optical system and on to the sample plane. Each ray is independent, and by following the paths of many rays emitted with a random distribution of angle and position, a map of the ray trajectories can be constructed at any point along the simulated beamline.

At the ESRF we use the Shadow code to optimise beamline designs, the latest version of which can handle low transmission calculations that require in excess of a million incident rays. A new code structure and improved ancillary software tools have also extended Shadow's ability to optimise the optical systems and to model freeform optical surfaces.

## The coherence challenge

Impressive reductions in the source emittance since the ESRF started up have led to an increase in the coherence lengths of the X-ray wavefront. Meanwhile, experimental

techniques that exploit these coherence properties, including holotomography and photon correlation spectroscopy, have flourished. The quality of optical components is therefore increasingly judged by their ability to interact with X-rays with minimal

impact on the coherence properties. Modelling the influence of optical components in this coherent regime requires us to consider interference phenomena for which standard ray-tracing methods are ill-adapted. Although simple geometric models allow a first approach for determining the required optical quality, more complete calculations of intensity distributions require a wave-optical treatment of the X-ray propagation. In particular, diffraction effects and other interference phenomena must be taken into account.

Recently, specific software has been developed at the ESRF that allows these



# Optical design

scientific potential of synchrotrons, upgrade.

effects to be modelled for devices such as multilayer-coated reflective focusing optics – modern optical components that are increasingly used to overcome some of the intrinsic limitations of mirror and/or crystal optics. These approaches are computationally intensive and, for routine use, are currently limited to optical systems with rather few components – typically just one. The modelling can become even more complex in the intermediate situation when the

X-ray wavefield is partially coherent, as is the case for ESRF beamlines. Recent developments of Shadow may provide an attractive approach to solving this problem by combining the relative simplicity of ray-tracing methods with the possibility of considering interference effects in a unified software framework.

## Source code

Critical to beamline design is the modelling of the X-ray source. Specific software codes originating from the ESRF permit detailed calculations of the characteristics of the source radiation – for example its energy range, tunability and polarisation – which are the starting point for simulating the optical system. The significant heat loads produced by modern undulator sources, a problem that is exacerbated by improvements in the storage ring such as longer straight sections and reduced emittance, are a key consideration when designing such upstream beamline components.

The power absorbed in the upstream optics exposed to the “white” beam must be dissipated efficiently to avoid thermal

deformations that modify the optical performance. Usually this is done using water- or liquid-nitrogen cooled heat exchangers in contact with the components – a process that must be optimised to ensure that the thermal deformations are less than or comparable to the intrinsic manufacturing quality. In the most complex cases, such as the ESRF upgrade inelastic scattering beamline UPBL6, this requires an iterative process whereby finite element analysis is used to model the deformations and the results are then fed into the ray-tracing code to simulate the effect on the whole beamline. The cooling scheme can then be modified until the configuration with the best optical performance is found.

As with the source radiation, the specific optical devices used in a beamline design are chosen according to a beamline’s operational requirements. To satisfy these diverse requirements, which include energy range and tunability, focusing, ease of use, and mechanical and thermal stability, the modern X-ray beamline designer has access to a richer toolbox of higher-quality optical devices than was conceivable when the ESRF started up.

For example, in recent beamline designs – such as the upgrade beamline “MASSIF” – it is possible to find combinations of compound refractive lenses, multilayer-coated bent mirrors and semi-transparent beam-splitting diamond monochromators all working together. Compound refractive lenses, the use of which was pioneered at the ESRF, have provided a powerful complementary means of focusing the beams that is particularly attractive in the hard X-ray range.

## Design at length

Long beamlines are a key feature of the ESRF upgrade because they allow extraordinarily small beam sizes. But to exploit their full potential, these beamlines require an extremely stable X-ray beam to prevent misalignments at the start of the beamline from “blowing up” at the end stations. Much effort has therefore gone into optimising

the thermal and mechanical stability of these systems, particularly for deflecting optics such as mirrors and monochromators. Due to the rather large asymmetry of the X-ray source size, which is larger horizontally than it is vertically, horizontal deflecting optics (often with a double reflection geometry) and the use of secondary sources by pre-focusing optics can help considerably in mitigating the effects of residual instabilities and imperfect optical devices. These strategies have been applied extensively on the UPBL4 and UPBL7 beamlines.

At the other end of the beamline, the challenge is to provide the final focusing elements for micro- and nano-probe beamlines. These devices, which include zone plates, nano-focusing refractive lenses and other focusing mirrors, are usually positioned close to or inside the end-station and many beamlines alternately employ different devices to optimise a particular experiment.

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**“The modern X-ray beamline designer has access to a rich toolbox of high-quality optical devices.”**

The ESRF has a significant optics development programme to provide the best quality devices for current and future upgrade beamlines. In addition to purely in-house developments, such as dynamically focusing Kirkpatrick–Baez mirror systems that allow the mirror curvature profile to be optimised for different focusing distances and deflection angles, we also collaborate with external groups and manufacturers. Optimal and routine use of these components for user experiments can be achieved only by effectively integrating them with stable and precise alignment mechanics. Even for externally sourced optics, this aspect of beamline design is largely dealt with in-house.

Beamline design is a continually evolving field, since it must respond to the requirements of new experimental techniques, source developments and improved optical device technologies. To meet these new challenges, computational processing capabilities promise new possibilities for optical design that will allow us to maximise the scientific potential of future ESRF beamlines.

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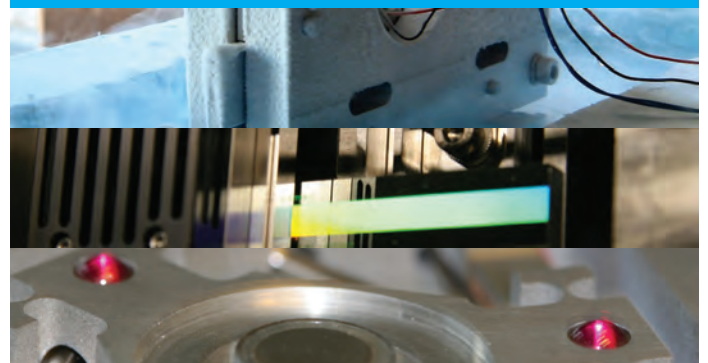
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# Keeping cool

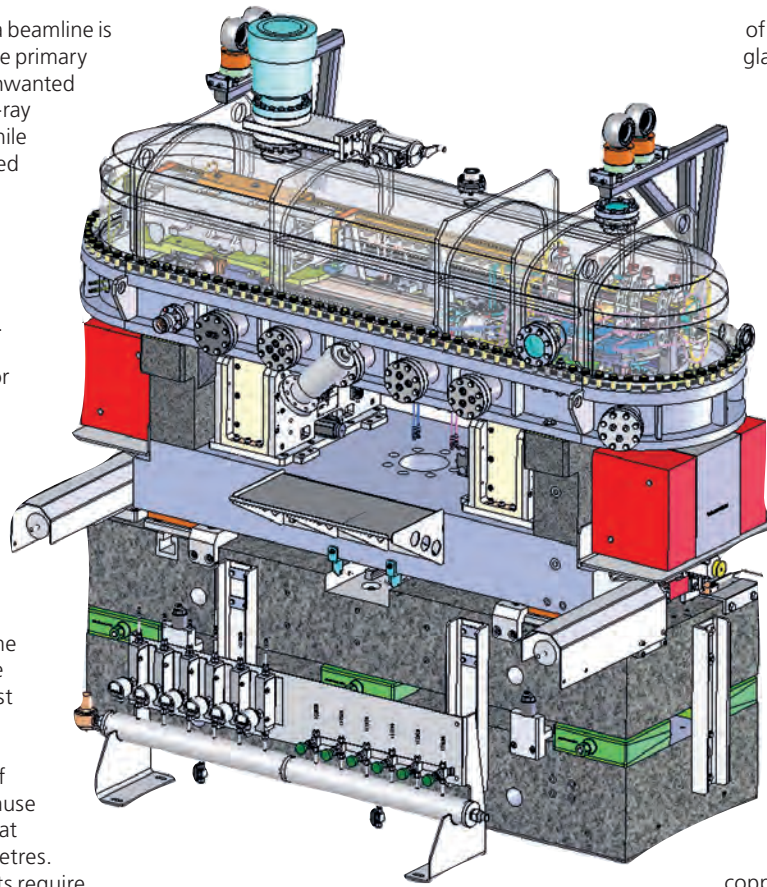
With some ESRF upgrade beamlines aiming for nanometre precision, the thermal stability of instruments and hutches is vital, explains *Robert Baker*.

The first optical element on a beamline is generally a long mirror whose primary function is to filter out the unwanted part of the raw or “white” X-ray spectrum from the beam, while directing photons with desired energies down the beamline towards the sample. This filtered portion of the beam is absorbed in the mirror and surrounding mechanical elements in the form of heat. Although heat load is spread along the length of the mirror due to the grazing beam incidence angle (typically less than 1 degree), without adequate cooling and thermal stability severe deformation and even permanent damage to beamline components could occur.

If we imagine that for some ESRF upgrade beamlines the sample may be placed almost 200 m from this first mirror (pictured right), it is clear that a fraction of a degree of angular displacement will cause a shift of the beam position at the sample by several millimetres. Given that some experiments require nanometre stability, we are therefore striving for angular mirror stabilities of some millionths of a degree!

## Stable options

There are a number of ways to deal with thermal instability. By careful choice of materials and design we can compensate for the thermal expansion of one part of a system by ensuring that another part gives the opposite effect. Applying such “athermal” techniques to complex mechanical assemblies is far from trivial, however, because they comprise many different materials with different coefficients of thermal expansion (CTE). The “thermal inertia” of each component may also differ: following a change in temperature, the length of one part of an assembly may change in a time of, say, 30 minutes while another part gives an equal but opposite change but in 10 minutes, causing a cyclic drift in position. Furthermore, athermal design imposes constraints in other domains such as optimising stiffness or reducing so-called Abbe errors,



**Solidworks 3D model of complete white beam mirror assembly, due to be installed on ESRF upgrade beamline UPBL4 in 2013.**

whereby small angular inaccuracies are amplified and cause larger linear errors.

Another approach to thermal stabilisation is closed loop compensation. Here, the position of an element, or of the X-ray beam itself, is read by a sensor and the information is fed back to the actuators that define and correct the element’s position. Closed loop compensation has proven successful in some applications, but it becomes somewhat complex when the absolute position reference is the photon beam itself (while we can measure the position of the beam on some form of detector, it is difficult, when movement is detected, to know whether it is the beam or the supporting mechanics of the detector that is moving).

Perhaps the most obvious approach to thermal instability is a careful choice of engineering materials at the design phase

of critical instruments. At first glance, it appears obvious to use low CTE materials such as Invar alloys or Zerodur. Practically speaking however, other factors must also be considered, such as engineering and radiation properties, vacuum compatibility, material matching and, of course, cost.

The main challenge is to reduce the distortion or bending of instruments due to non-homogeneous temperature distributions through a given part or assembly. This phenomenon can be quantified by choosing materials not only for their CTE but also for their *ratio* of CTE to thermal conductivity.

In the case of “punctual” heat sources, such as those in high heat-load white-beam mirror systems, thermal inertia also becomes extremely important. Sorting materials according to these two parameters reveals that more common engineering materials such as aluminium,

copper and some steel alloys tend to be more useful than materials such as Zerodur or Invar, which are expensive and difficult to machine.

## Cool by design

Design principles such as these have led to a radically different approach to thermal stability at the ESRF. First results obtained on the new high heat-load double mirror on upgrade beamline ID24, for instance, show higher thermal stability, faster settling time and lower drift than on previous mirror systems designed in the 1990s. In those days we tended to avoid aluminium in thermally critical applications because of its high CTE, preferring stainless steel, and would deal with heat loads via cooling and by using Invar in extreme cases.

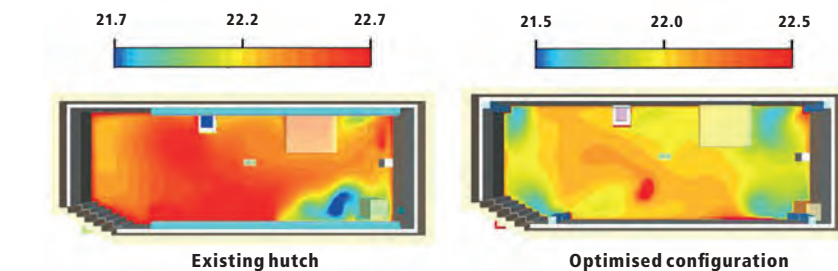
Now we “design in” thermal performance, with emphasis on fast settling time and minimal distortion. This is being done on all upgrade white-beam mirror systems. ID24 is the first installed system based on these principles, but several others are due for installation over the next 12 months as part of the ESRF upgrade programme. ▶▶

### Stabilising the environment

It's not just individual components that have to be kept cool. 24-hour cyclic variations in the ambient temperature along a beamline cause bending and distortion in all supports, structural elements and even the experimental hall floor, leading to unwanted low frequency movements of components. It is therefore extremely important to minimise thermal fluctuations in the beamline hutch environments.

In 2005, the ESRF modified the ID22 nano-imaging experimental hutch to produce the most thermally stable environment to date. Porous entrance ducts were installed along both sides of the hutch below the roof panels, with extraction ensured through perforated tiles in a raised floor. An entrance porch also limited thermal disturbance from the experimental hall. However, spatial temperature gradients remain significant and thermal stability is still insufficient for the most precise nano-imaging applications (left image above).

More recently, we modelled the thermal behaviour of the ID22 hutch thanks to an extensive data collection and measurement campaign run over several months. Parameters monitored included temperatures inside and outside the hutch, air speed,



building materials and even the shape of equipment and the effect of human presence. A 48-hour cyclic temperature variation was applied to the outside faces of the hutch model, and static and transient studies run to observe thermal performance. Once the model was considered "true to reality", several drawbacks were identified:

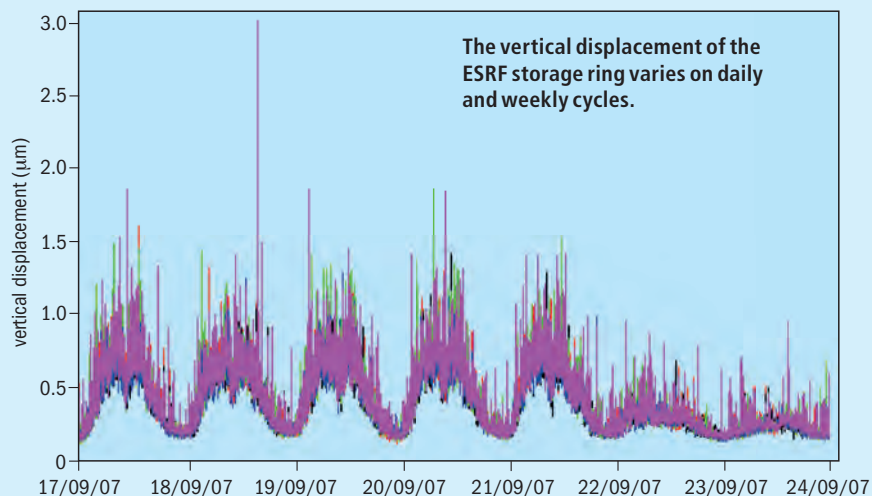
- 35% of the total airflow is "lost" through leaks, for example in the "chicaines" that allow cables, pipes and other services to be fed into the hutches from the outside.
- Large variations in airflow and uncontrolled areas cause temperature gradients.
- Heat generated from electronic devices close to the sample degrade stability.
- Sensitivity to outdoor weather conditions is too high.

By varying input parameters in the model, we obtained an improved configuration in which controlled air is injected through a porous sleeve running the length of the hutch placed above a textile membrane that covers the entire roof. Adjustable ducts ensure extraction at the four lower corners and directly above the electronics rack, and all walls (except the hutch entrance door) and the roof are insulated. Results (right image above) show a significant improvement in thermal stability ( $\pm 0.045^\circ\text{C}$  over 24 hours at the sample position) and much lower spatial gradients than before. Theoretically, stability down to  $0.01^\circ\text{C}$  seems possible. The first hutch to be built according to these specifications is the upgrade beamline end station at ID16, due to be operational in 2013.

## Good vibes

Experiments on the nanoscale place unique demands on the vibrational stability of synchrotrons. The ESRF upgrade beamline NINA, for example, requires samples to be positioned to within 5 nm for imaging purposes. In addition to minimising thermal drift and ensuring that infrastructure such as water pumps is placed away from the end stations, significant efforts are therefore being made at every stage of design to increase the stiffness of optical elements and sample stages, and to limit resonant vibrations.

With the ESRF being surrounded by rivers and roads, the floor vibration in the frequency range 1–10 Hz is typically  $1\text{--}10\ \mu\text{m}$  peak-to-peak during the day and  $0.2\ \mu\text{m}$  during night (the 2011 Japan earthquake caused displacements of  $10\ \mu\text{m}$ ). "In most cases we don't try to reduce this, but we do try to avoid differential vibrations between critical elements," says Philippe Marion, head of the ESRF's mechanical engineering group. "If we are not careful there is the possibility to amplify the vibration because of the resonance of support structures." The largest floor vibrations are below 20 Hz, so support structures with higher natural frequencies must be used. By attaching instruments to low-thermal expansion granite blocks bonded to the hall floor, the aim is to allow everything on the slab to vibrate in the same way without amplifying the floor vibrations.



The vertical displacement of the ESRF storage ring varies on daily and weekly cycles.

### Golden slab

In March 2012, engineers constructed a test slab for the  $4000\text{ m}^2$ , 1 m thick "golden slab" that will be the foundation of the ESRF's experimental hall extensions. The test slab is 15 m long and 3 m wide, and is undergoing extensive monitoring to check vertical movement, shrinkage, vibrational stability and lateral strain. The main goal, explains building engineer Paul Mackrill, is to prove the procedures and quality control for each layer: first, a layer of dry cement is laid down like a road, followed by levelling

concrete, a bitumen layer, and finally 35 cm of reinforced concrete with a very low water content to minimise cracking. "We need it to be as good as the existing slab and we aim for it to be a lot better," says Mackrill. "For vibration levels we cannot easily reduce the background noise, but we need to make sure that we don't amplify it in any way." The floor of the NINA beamline will be poured in September this year, with construction of the main slab expected to begin in March 2013.  
*Matthew Chalmers*



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# Setting the stage

*Matthew Chalmers* takes a tour of the ESRF's sample environment laboratory.



Should you ever need to get hold of a molybdenum screw at three o'clock on a Friday afternoon, the ESRF's sample environment laboratory is a good place to start. "We stock tantalum screws too," says group head Peter van der Linden when describing his group's inventory of specialised equipment. Keeping the ESRF experiments running 24 hours a day is one of three main tasks of van der Linden's group. The others are to operate the ESRF's instrument loan pool and to develop bespoke sample environments for the beamlines.

The ESRF loan pool allows users and beamline staff to tap into a wide range of specialised X-ray equipment without worrying about procurements or maintenance. It offers everything from pyrometers – expensive pieces of kit that measure the temperature of an object from a distance by its glow with a precision of a tenth of a per cent – to custom-built cryostats, high-pressure diamond anvil cells and furnaces.

Advanced sample environments are pushing ESRF science into new ground. Take catalysis furnaces, one of which developed in conjunction with Toyota sits on a window sill in the sample environment lab. In order to allow users to flow gases over a sample to study quick changes in its reactivity, the cell has to have a small volume so that the gas can be refreshed at a high rate. "Very often users use dirty or dangerous gases like hydrogen in the vicinity of a heater with its electrical contacts – that's not always a good idea," says van der Linden. "So you need to keep the volume small and separate the electrical from

**"It's the most complicated cryostat that I have created."**

the gas-filled part."

When the sample environment group started in 2000, its setups were defined around classic thermodynamic parameters such as temperature and pressure, perhaps also combining magnetic fields, whereas today's users want to be able to vary several parameters at once. A materials scientist, for instance, might want to do a stretch test on a sample, rotate it and be able to do it all at 250 °C to study material fatigue, crystal deformation and crack growth.

#### **Dream environments**

"Perhaps 80–90% of our work is responding to the needs of scientists and beamlines, but the rest we do ourselves because we can see that there will be a demand for something in the future," explains van der Linden as he proudly displays a cryostat that can cool a high-pressure diamond anvil cell to 4 K at a sample pressure of 1 Mb while rotating it around the sample and providing high micrometre stability. The demand for this particular device, he explains, came from staff at the ID27 beamline, but once available

it was also demanded by other beamlines. The group is currently working with the ESRF engineers building the upgrade beamline NINA, where samples have to be cryo-cooled on a stage that offers high mechanical stability and allows resolutions in the region of 20 nm.

"My most complicated sample environment is a cryostat for pulsed high magnetic fields that could finally include a high-pressure cell, which is technically difficult because there are constraints that are difficult to resolve," says van der Linden as we admire an intricate configuration of wires, tubes and machined steel parts located inside a copper coil designed to carry liquid helium. "It's the most complicated cryostat that I've created. The pulsed high magnetic field, low temperature, high-pressure environment makes possible unique fundamental experiments looking at the magnetic interactions between atoms."

Although the group's adjacent high-pressure laboratory was demolished in the works for the Chartreuse extension of the ESRF upgrade, the future looks bright according to van der Linden. "Sample environments will have to become more diverse to answer tomorrow's needs," he explains. "Smaller focal spots and smaller samples combined with bigger and faster detectors create a need for larger optical opening angles, better positioning and faster sample changing. Different thermodynamical parameters will be applied simultaneously, but they are also combined with other techniques such as optical spectroscopies. We will be busy for the years to come!"





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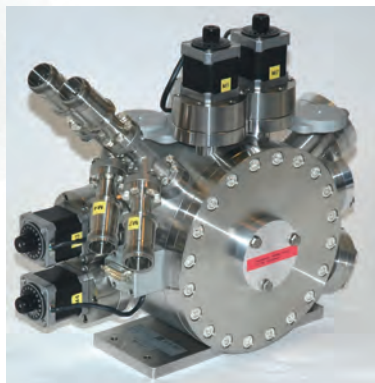


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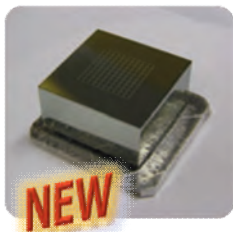
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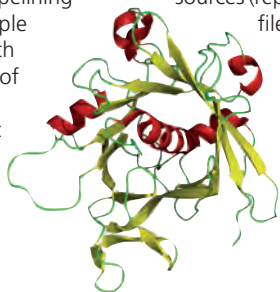
# Go with the flow

A new “workflow” tool at the ESRF will help users keep pace with the increased automation of online data analysis, say *Olof Svensson* and *Andy Götz*.

The time it takes to carry out experiments at the ESRF has decreased significantly in the past few years thanks to advances in the X-ray source, optics and detectors. As a result, beamlines are able to handle a much higher scientific throughput. The flipside, however, is that it has become more difficult for synchrotron users to keep up with data analysis during their allotted beam time, often meaning that users return to their home institutes without knowing whether their experiment has succeeded or not.

Automated online data analysis (ODA) is therefore becoming increasingly important in optimising users’ beam time. Since raw data alone does not always tell the user if the data is good, ODA allows researchers to interrupt an experiment if the desired data quality after preliminary data processing is not achieved, for example due to a faulty or misaligned sample. Alternatively, in the event where the desired signal is accurately measured, ODA allows researchers to put remaining beam time to use for different samples or improved studies.

The implementation of ODA can be divided into two categories. The first concerns specialist software packages for basic data processing such as image analysis and peak fitting. The second concerns “pipelining” – the stringing together of multiple data processing steps where each step may depend on the output of the previous one. Examples of pipelining include the automatic data reduction of images from SAXS and protein crystallography experiments.



**The increasing complexity of proteins targeted by macromolecular crystallography experiments calls for advanced online data analysis.**

## In the pipeline

Traditionally the “pipelining” aspect of ODA has been implemented in scripts written in Bash, Python or in high-level frameworks such as EDNA. These software developments tend to be of such complexity, however, that beamline scientists must rely on programmers to achieve the desired goals. This works well if the desired ODA is relatively simple and there are not too many steps involved between the detector output and the online display of the results. But if the ODA involves several steps, possibly combined with alternative analysis paths depending on intermediate results, it becomes increasingly more difficult for the scientist to specify and communicate his or her goals to the programmers. This is especially true when the process is not well defined, for example if users wish to experiment with different data analysis steps.

At the ESRF, not only is ODA becoming increasingly important but also increasingly complex. We are therefore exploring the feasibility of using a workflow tool for bridging the gap between beamline scientists and programmers. The idea of a workflow tool is that the pipelining is performed graphically by connecting different “actors”, which can be sources (representing detectors or reading files from disk), transformers

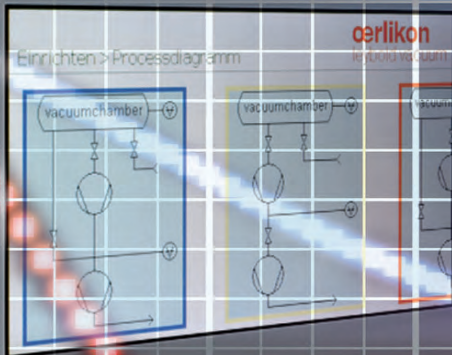
(representing data analysis or logical choices) and sinks (outputs to a file or a graphical user interface). The result is that beamline scientists should be able to develop their own tailored workflows, allowing them to modify analyses “on-the-fly”, while the programmers can devote their efforts to make the underlying infrastructure as robust as possible.

## A new dawn

The development of the workflow tool *Passerelle*, based on the open source *Ptolemy* workflow framework, is a part of the *DAWN* collaboration with the *Diamond Light Source* and *EMBL/Grenoble*. Tests have so far been performed on the ESRF’s macromolecular crystallography beamlines and on the upgrade beamline *ID24 (“TEXAS”)*, and feedback from scientists has been positive. Future developments will allow seamless use of the workflow tool from the ESRF standard beamline control system such that users benefit from ODA workflows developed and maintained by the beamline scientists without having to learn how to use the workflow tool itself. This is possible because the workflow engine can run independently of its graphical user interface.

Having already demonstrated the concept of the workflow tool and tested it on ESRF beamlines, we have recently integrated it with the beamline control to make it available on a routine basis. This will enable workflows to be used for a wider range of scientific problems, including offline data analysis, and allow users to take *DAWN* home to continue or repeat their analyses.

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# The ESRF considers its future

Two decades after producing its first X-rays, the ESRF is preparing the ground for 2030 and beyond.

As the ESRF's beamline portfolio expands into brand new experimental halls, as part of Phase I of a major upgrade, the ESRF has established a "Working Group for the Scientific Mission of the ESRF" to shape the facility's longer-term future.

The report, which is due to be delivered to the ESRF Council by the end of 2012, will address whether a major accelerator upgrade is viable, which areas of science should be developed, and how relations with industry should evolve. It will also determine how the ESRF should position itself with respect to the many other third-generation synchrotrons



P. GINTER/ESRF

that have sprung up since the first X-rays made their way down the ESRF's ID06 beamline in the spring of 1992.

"The working group is going to see what avenues may be open for ESRF in the future and hopefully stimulate members to favour the analysis of these possibilities," says working group chair Michel van der Rest, vice-chairman of the ESRF Council and former director general of Synchrotron SOLEIL. "But it will be the responsibility of management to make proposals and of the Scientific Advisory Committee to evaluate them for decisions by the Council."

## Next generation

The report was prompted by consideration of Phase II of the ESRF upgrade, which is expected to take place from 2015. "We asked: are the ESRF's partners going to invest in the facility after Phase II or will they be talking about dismantlement, because the facility will be over 30 years old by then," says van der Rest. "The coming years will also see a generation turnover at the ESRF, with many of the founders retiring, which is both an opportunity and a challenge because the ESRF is what it is today because of the commitment and the outstanding abilities of its staff."

With the ESRF now joined by some 20 other "third-generation" sources, is there a risk that it will lose its leading edge? "It's a bit of a free market, but at the same time there is increased co-ordination between sources, especially in Europe," he says. "There may seem to be a beamline glut coming up in Europe but when you look at the numbers, the scientific demand is still there."

Lower beam emittance is a key goal of light sources, since it allows smaller beams, and the ESRF is fast approaching the limit of what is possible without increasing the size of the storage ring or overhauling its components. By contrast, states van der Rest, the new MAX-IV source in Sweden will have an emittance 10 times smaller than the ESRF, and Japan's SPring-8 is aiming for a similarly low emittance by reducing the beam energy from 8 to 6 GeV – which also reduces the energy budget.

"There is a tremendous amount of information missing in the nanometre range, and there are a lot of technologies developing in this region, but how crucial are nano-beams for top science?" he asks. "The future cannot be linked solely to one 'trendy' aspect like the ultimate beam size or brilliance – other aspects, such as time-resolved experiments, will have to feature in the ESRF roadmap."

"It is important that you stay at a high level of performance, as the ESRF has demonstrated with its upgrade programme," he continues. "The support of member countries, especially those that have national sources, can only continue if the ESRF is clearly adding new dimensions." *Matthew Chalmers*

## Memory lane: the first X-rays at the ESRF

"At the beginning of the ESRF there were a lot of unknowns: would it work at all? I was responsible for the ID13 microfocussing beamline, which was one of three test beamlines, and we demonstrated liquid-nitrogen cooled monochromators – which had existed only as a concept. Everything back then was very rudimentary but I managed to set up a scientific programme with the help of dedicated staff and friendly users. History repeats itself because for the upgrade, ID13 was again one of three test beamlines selected – this time for nano-beam R&D. I would never have foreseen the science that we can do now and expect that the upgrade will also reveal numerous uncharted territories."

**Christian Riekkel, emeritus scientist**

"The first thing was to check the stability of the concrete slab, so we focused the beam on a slit using a bent silicon crystal and then had a forklift truck drive by or ran the overhead crane while recording the intensity variation. It was a simple experiment – we had an office chair as a support for some equipment and an LED showing the modest beam current! But it had a major impact because it showed that the floor could be stabilised by injecting concrete into drill holes."

**Michael Krisch, scientist in charge at ID28**



M. KRISCH

## Synchrotron or cycle track? The empty ESRF hall circa 1992.

"It was a very exciting time. The first synchrotron beam was taken in the evening until the middle of the night and the experimental hall was completely empty. I was the radiation protection officer and was cycling around the hall checking radiation levels. Those pioneering days remain a very good memory for a lot of people because the group was very small and you knew everybody."

**Elke Brauer-Krisch, beamline engineer on ID17**

"My only memory of that early period is seeing Elke Brauer bravely measuring the radiation coming through the doors to the optics hutch on ID11, and my deciding it was probably safer to shelter behind the nearest electronics cabinet (probably offering no protection whatsoever) before returning to the relative safety of my office!"

**Andy Fitch, scientist in charge at ID31**

# Two cultures mix at the Met

Synchrotrons are making a big impact on the museum world but there are still cultural barriers to break down. *Matthew Chalmers* reports from the SR2A symposium in New York.

Master paintings, medieval silverware, entire violins and gold bracelets worth millions of dollars – is no museum piece exempt from the intense X-ray scrutiny of synchrotrons to help scholars understand and preserve our cultural heritage? These and many more precious objects were the subject of the 2012 Synchrotron Radiation in Art and Archaeology (SR2A) symposium held at the Metropolitan Museum of Art, New York, between 6–8 June – the fifth such meeting since its inauguration at the ESRF in 2005.

Synchrotrons are clearly making a big impression on the museum scene. Techniques such as X-ray fluorescence and X-ray absorption near edge structure (XANES) can reveal why certain paint pigments become discoloured, and at the ESRF's ID21 beamline are providing insights in how to preserve vibrant works by artists such as Van Gogh and Matisse. The manufacturing methods employed in early ceramics and glass were another main topic of discussion at the New York event – a three-day feast of histograms and beautiful visuals spanning thousands of years of human history.

We were treated, occasionally accompanied by the sounds of excited school children visiting nearby galleries, to stunning tomographic images of sub-mm workmanship in a 260-year-old violin; diffraction experiments that can authenticate ancient astronomical computers; chemical maps from tree rings that may help pinpoint volcanic eruptions; and XANES spectra of fossilised feathers that reveal the colourful new world of paleobiology. Maisoon Al-Jawad from Queen Mary University in the UK described how she is using the ESRF to develop models of archaeological enamel that could lead to bioactive dental repairs.

## X-ray fears

It was also clear that there remain barriers between the art and science worlds. “The use of synchrotron-based X-ray techniques remains marginal, mostly because cultural-heritage specialists rarely interact with synchrotron specialists,” said physicist Volker Rose of the Argonne National Laboratory,



**Grand setting:** a reception at the New York Institute of Fine Arts just opposite the Metropolitan Museum of Art, where 137 scientists, conservators and art historians spent three days discussing the merits of synchrotrons in art and archaeology.

## “Connoisseurship will always play a central role.”

who presented new opportunities arising in nanoscale cultural heritage research. Ironically, Rose summed up the situation using analogies that only a physicist could love: the tendency of a system to minimise its potential energy and the notion of overlapping quantum wavefunctions.

The “extreme heterogeneity” of historic materials is one reason why the cultural heritage community is yet to take full advantage of synchrotrons, argues chemist Matija Strlic of University College London. “Scientific characterisation of material behaviour is carried out on a variety of scales, but not all measurable change on the micro scale is necessarily viewed as damage by curators or the public,” he stated.

Getting straight to the point, conservator David Thurrowgod of the National Gallery of Victoria, who has used the Australian Synchrotron to reveal lost paintings behind works by Degas and Streeon, said that curators want to know whether it's safe to put a \$50m painting in a beam, or if it's going to burn a hole, adding that he has tried but been unable to detect any damage in paintings that have been irradiated in synchrotrons. Franco Zanini of Italy's Elettra also faced difficulties in convincing the owners of classic violins (which can be worth upwards of €20m) to put their instruments on his beamline. “Now that we have shown that it is safe, more people are interested in using the technique,” said Zanini.

## Cultivating research

Art historians who spoke to *ESRFnews* – two of whom, interestingly, made their primary living from jobs as hospital radiographers – see no conflict, at least in principle, between the two cultures. They are enthusiastic about the use of synchrotrons, especially for helping to determine the provenance and authenticity of paintings and artefacts.

But connoisseurship will always play a central role according to the Met's curator for European paintings Walter Liedtke, who likens the situation to identifying a good wine. “If you want to know that a particular red wine is from the north of Italy, a chemist can tell you that,” he told *ESRFnews* from his bookshelf-flanked office tucked behind locked doors in the Met's Rubens and Rembrandt galleries. “But if you want to know that it's a 1982 Barolo worth 300 bucks a bottle, then you need to talk to someone who has had a lot of it.” Liedtke added that he would be keen for synchrotrons to help him decipher text that appears to be hidden beneath a painting in his gallery by Flemish artist Anthony van Dyck.

Connecting with curators is key to carrying out new and relevant cultural heritage research, and half of the attendees at this year's SR2A event said that they had made new contacts for research projects in this high-profile and rapidly growing field of synchrotron science. “Attendees have unanimously remarked upon the very significant progress in the field over the past years,” concluded Loïc Bertrand, director of the IPANEMA European ancient materials research platform at Synchrotron SOLEIL. “We therefore think that more public and private support could be brought to consolidate existing interface projects and infrastructures, and to foster the creation of novel tools using synchrotron radiation.”



# A man for all sources

SOLEIL beamline manager **Paul Dumas** has a rare command of the synchrotron scene.

His friendly, easygoing manner and broad synchrotron experience make it little wonder that physicist Paul Dumas is sought out by facilities across the world. In 2003 he was invited to become a member of the Scientific Advisory Committee of the UK's Diamond Light Source, followed by the same honour at Brookhaven's NSLS, then the Canadian Light Source and, more recently, Thailand's Synchrotron Light Research Institute and the SESAME source in Jordan.

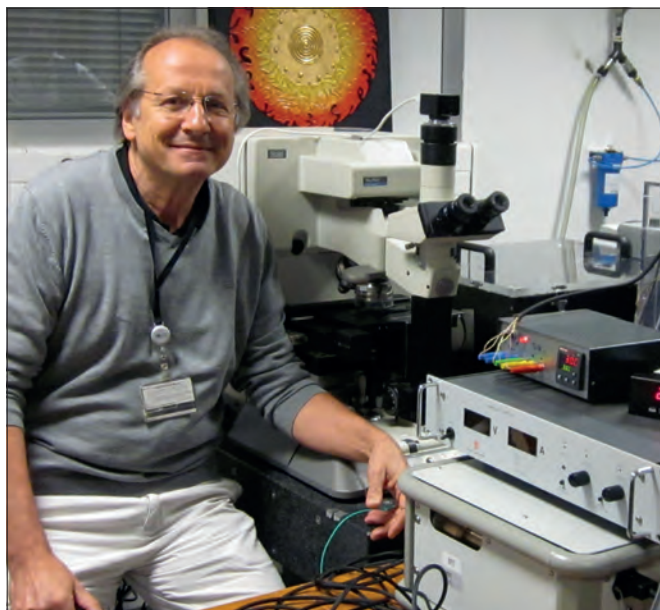
"After a while people start to ask for your views," he explains modestly over the phone from Synchrotron SOLEIL, where he is beamline manager responsible for infrared microspectroscopy on leave from the CNRS where he is director of research.

## Beneath the surface

Dumas began his career in surface science, diffraction and vibrational spectroscopy. But in order to understand more deeply how molecules bind to a surface, he wanted to study samples in the far-infrared region of the spectrum where traditional techniques fall short. In 1985, while working at the University of California, Berkeley, Dumas met an infrared specialist from Bell Labs who, a few years later, introduced him to the synchrotron at Brookhaven – an event that changed his professional life.

"We were dreaming of using infrared beams at synchrotrons, but nobody at the time thought that it would work," he says. What followed was one of the best times of his career: a period of intense activity with Brookhaven scientists that resulted in the world's first successful infrared beamline for surface science. "The synchrotron was really noisy at the time, so we had to fight to get the beam as stable as possible," he recalls. "The best results therefore often came between two and five o'clock in the morning, and we had to interact closely with the machine people."

While at Brookhaven Dumas



Feet on the ground: Paul Dumas at SOLEIL's IR spectroscopy beamline.

## Paul Dumas in brief

### Born

Algeria, 1948.

### Family

Three daughters.

### Education

Physics, Dijon (1981).

### Career

University of California, Berkeley (1984–86); LASIR, Thiais (1981–84; 1987–90); LURE (1990–2003); SOLEIL (2003–).

### Interests

Movies, culture, socialising.

met Yves Petrof, later to become director-general of the ESRF, who invited him to join the French source LURE in Paris. After his success at Brookhaven, Dumas had wanted to bring infrared synchrotron surface science to Europe, but since few groups were working in the area at the time, he was advised by his "synchrotron mentor" Gwyn Williams at Brookhaven to propose an infrared microscopy beamline instead. "It was a difficult period because I did not know if I would succeed and therefore whether or not I would

stay on the synchrotron," he recalls. "But finally, thanks to the director at LURE, we managed to do it at low cost."

Around that time, Dumas met the ESRF's Jean Susini at a conference in Denmark where, over a walk after a beer, the pair got talking about the potential for infrared microspectroscopy at the ESRF. Fast-forward and the ESRF's ID21 beamline is now one of the world's leading facilities for Fourier transform infrared microspectroscopy. Having learned a great deal about sophisticated beamline design, he has gone on

to help other synchrotrons get the beamlines that they want.

This year Dumas is co-chair of the Scientific Programme Committee for the Synchrotron Radiation and Instrumentation (SRI) conference – a triennial event that he says is "crucial" because instrumentation is the basis for scientific productivity.

## The big picture

Involvement with several facilities has given Dumas a broad knowledge of the light source landscape. He views synchrotrons as a "highway for science and ideas": bringing scientists together, exposing them to different fields and techniques, and providing them with the best equipment available.

So how will the synchrotron scene evolve? "Sure we have lots of sources in Europe, plus in the US, China and Japan, but they are springing up elsewhere too," he says, citing examples: Poland is currently building a 1.5 GeV storage ring; Iran and Turkey are both planning one, while also being partners of the SESAME source; and Argentina is eyeing up a third-generation source. Meanwhile, both Brazil and India are upgrading their existing storage rings and Thailand, reckons Dumas, will soon start thinking of upgrading its facility too. "These countries are expressing the need to have a center of excellence to promote their science," he says. "It shows that synchrotrons are not just for the most developed countries." Free-electron lasers, he says, will be complementary to, rather than competition for, storage rings.

Dumas is often asked why he has not wound up in the top-level management of facilities. "I have committed to myself that the best way to be inspired is to remain as close to the experiments as possible," he explains. "I have been extremely fortunate to have 'touched' the management of facilities without leaving the field where I am more expert: my beamlines and my science."

*Matthew Chalmers*

**"Synchrotrons are highways for science and ideas."**

# Leveraging the ESRF's intellectual property

The ESRF is preparing to capitalise further on its instrumentation know-how.

The ESRF is more than a suite of world-class X-ray beamlines: it is a unique hub for synchrotron instrumentation, with some 150 engineers and scientists working across specialist laboratories developing everything from undulators to detectors.

Much of the ESRF's technology, which is often developed in collaboration with other institutes, is sold through licence agreements with established companies. Over 20 agreements are in place, including: permanent magnet technology for insertion devices (Danfysik & Bruker Advanced Supercon GmbH), K-B systems for high performance focusing (IRELEC), and high-precision miniature X-ray slits (JJ X-Ray). ESRF instrumentation may also be sold directly.

## From prototypes to products

The goal of the ESRF now is to extend its product range and to streamline the process of turning expertise and new technologies into profit and jobs. "Many other synchrotrons do not have such a strong instrumentation infrastructure, so it's one of the missions of the ESRF to share our expertise with other sources," explains Ed Mitchell, head of the



ESRF's Business Development Office (BDO). "This year we want to develop our offer of unique technology and expertise, exploiting the ESRF's 20-year-long experience."

Some ESRF instrumentation is already close to market: for example high-performance scintillator screens, pixel detectors (particularly MAXIPIX), high-temperature furnaces, cryostats and advanced crystal optics. Until now, however, instrumentation has not been advertised and, when not licensed, is sold in limited volumes.

In addition to well defined X-ray instruments, the ESRF wants to integrate its expertise in specialised software and advanced analysis/modelling techniques. "We cannot say we are going to sell just mechanics, optics, electronics or software

because the high performance of an instrument comes from pooling these things together," says Muriel Mattenet of the BDO.

Intricate instruments such as K-B mirrors, particularly those designed for nanobeams, plus multilayer or crystal monochromators and white-beam mirror systems are a promising ESRF expertise given the trend towards nanoscale beams at synchrotrons worldwide. The ESRF currently has different versions of "transfocator" optics assemblies based on compound-refractive lens technology, but is converging on a standardised and therefore more commercially viable set up. Similarly, more general ESRF-designed hardware such as ion chambers and high-vacuum compatible beam viewers would benefit from economies of scale.

## Preparing for the future

Improvements in third-generation sources, better optics, enhanced thermal and mechanical stability, optimised control, and faster data acquisition have enabled the evolution from micro- to nano-probe synchrotron experiments. Extrapolating this trend, explains Mattenet, access to picoscale X-ray probes, faster data acquisition and more advanced control may become possible – with a move towards "ultimate storage rings" with improved emittance.

By leveraging the ESRF's intellectual property, the BDO aims to ensure that instrumentation meets the future demands of science and technology. "In terms of the science, we expect to see greater demand for *in situ* and *in operando* studies, in combination with shorter timescales and the ability to probe deeper into the structure of matter at higher resolution," says Jean Susini, head of the ESRF's Instrumentation Service and Development Division. "This will oblige instrumentation to be one step ahead of the science drivers, and to draw upon new industrial technologies." *Matthew Chalmers*

## Movers and shakers

### ID18 user award



Physicist and regular ESRF user Sebastien Couet, of the Katholieke Universiteit

Leuven in Belgium, has received the "Prof. Roger E Van Geen SCK-CEN" prize, for a significant contribution to science involving nuclear physics. The prize, worth €12,500 and awarded every two years for work by researchers at a Belgian research institution, was won by Couet's studies of the magnetic and dynamic properties of nanoscale thin films by nuclear resonant scattering, all of which were carried out at the ESRF's ID18 beamline.

### New group heads



The ESRF Experiments Division has appointed two new group heads: Marine Cotte (top) for X-ray Imaging and Roberto Felici (bottom) for the Structure



of Materials. Former head of the X-ray imaging group, José Baruchel, will become emeritus scientist on 31 July. In the Instrumentation Services and Development Division, Ray Barrett has been appointed as group head of X-Ray optics.

### All change in admin



Manuel Rodríguez-Castellano, a lawyer who holds a masters in business administration

and who has been ESRF director of administration since 2010, has been appointed head of the Administrative Division at Institut Laue-Langevin (ILL). Helmut Krech, his predecessor, returned on 16 April to the ESRF as acting director for the period until a new director of administration takes up service. On 1 September, Thierry Baudin will join the ESRF as new head of the personnel service.

### Physics olympics

In May, the ESRF and ILL welcomed the winners of the annual French Olympiades de Physique for a prize tour of the facilities. The four secondary school pupils and their physics teacher visited the high-pressure beamline ID27 and the French CRG beamline for protein crystallography, BM30A.



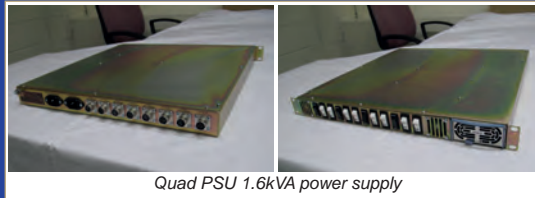




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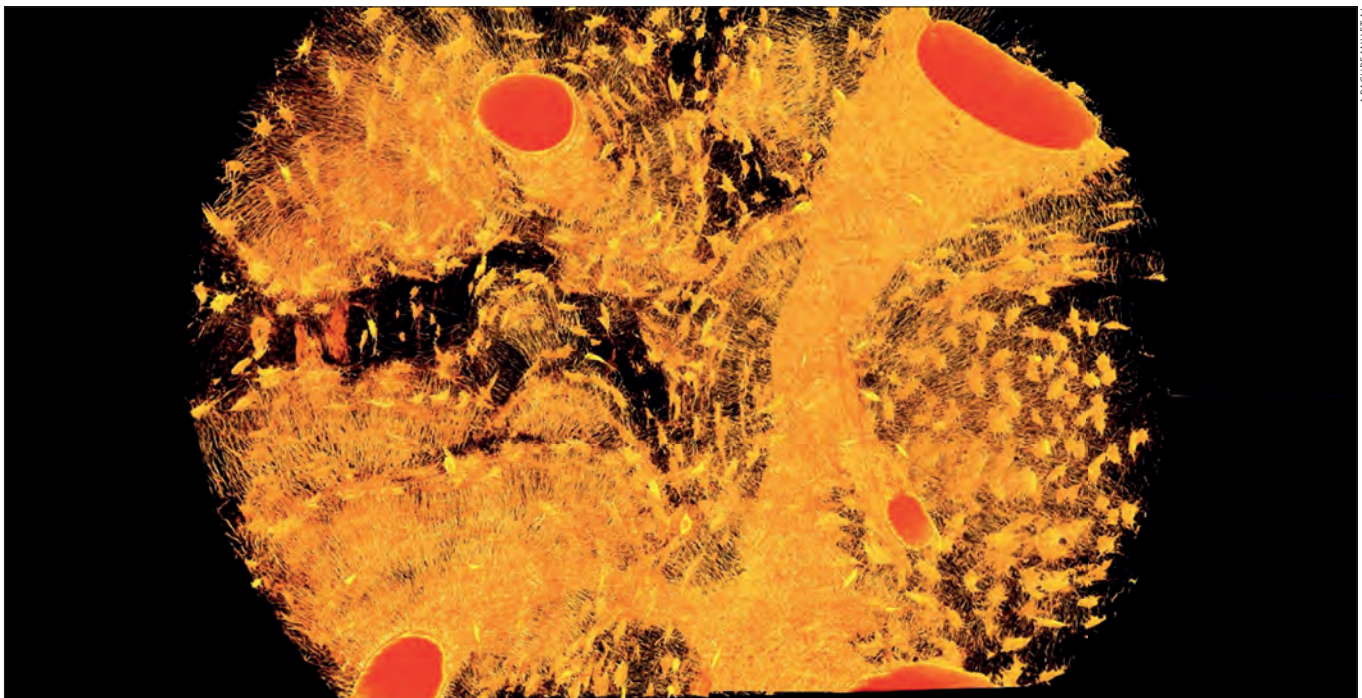
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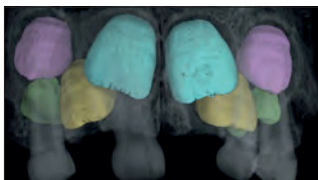


A. PACUREANU ET AL.

**Bone gives up its secrets:** This image, produced at the ESRF's ID19 beamline by X ray micro-tomography, reveals the microstructure of human bone in unprecedented detail. Based on a sample from the femur of a 92-year-old female, it shows for the first time over a large field of view the 3D "osteocyte lacuno-canalicular network" – a complex mesh of holes and channels embedded in mineralised bone. By allowing the transport of signals, nutrients and waste, this cell network is what gives bone tissue the ability to locally alter its mass and structure in response to damage or mechanical stress. Until now, however, its 3D organisation and its implications for bone remodelling have remained out of reach. In this image, which represents a volume of around 0.02 mm<sup>3</sup>, several osteons (the primary functional units of compact bone) can be seen with a large number of cell dendrites emerging radially from the central canal (red). In addition to answering fundamental questions in biology, the technique is likely to be of interest for developing strategies to deal with bone diseases and provides new input for biomechanical modelling. The work was carried out by Alexandra Pacureanu *et al.* of Creatis INSA Lyon & ESRF and co-workers at the UPMC in Paris (submitted for publication).

## In the corridors

### Are dentists bad for the brain?



PIA FOR EA/ESRF

The owner of this fine set of teeth, imaged last year at the ESRF's ID19 beamline, need not worry about the damaging biological effects of X-rays: he or she was an infant Neanderthal who lived tens of thousands of years ago. But according to a study published in April in *Cancer*, the rest of us might want to think twice about undergoing a dental X-ray. Elizabeth Claus of Yale University's School of Medicine and co-workers claim that exposure to some dental X-rays appears to be associated with an increased risk of intracranial meningioma – a type of brain tumour. Based on a case-control study of 1433 people diagnosed with intracranial

meningioma, the team found that individuals were more than twice as likely as those in a control group to report having ever had a "bitewing" dental X-ray. An even higher risk of meningioma was associated with those who reported having had panoramic X-rays taken of their entire mouths. The researchers point out that the X-rays were taken when radiation exposure was greater than it is now, but conclude that "considered use of this modifiable risk factor may be of benefit to patients".

### Edible electrons



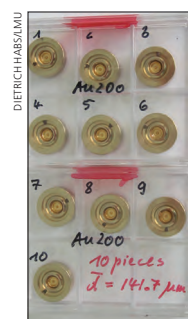
WWW.ELEMENTS-SCIENCE.CO.UK

Being circular in shape, storage rings are often described to the wider public in terms of doughnuts. But a team from Elements in the UK – "an offbeat destination for science news, features and comment"

– has taken the analogy a sweet step further. Drawing inspiration from the Diamond Light Source, the team used stop-start animation and a selection of sugary treats to introduce viewers to synchrotrons. The 2.5-min-long film has so far attracted over 40,000 views on YouTube.

### Gamma-ray optics

Scientists at the Institut Laue-Langevin (ILL) and Ludwigs-Maximilians University of Munich have demonstrated that gamma rays can be bent – a feat thought to be unrealistic given that refraction decreases as the energy of electromagnetic radiation rises. According to an ILL press release, the discovery overturns decades of theoretical predictions and opens the door to a new field called nuclear photonics. The gamma rays produced using ILL's PN-3 facility were preselected by a crystal spectrometer and half of them were funnelled towards a silicon prism. The output was then



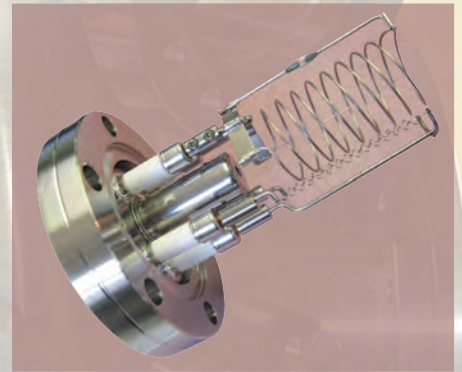
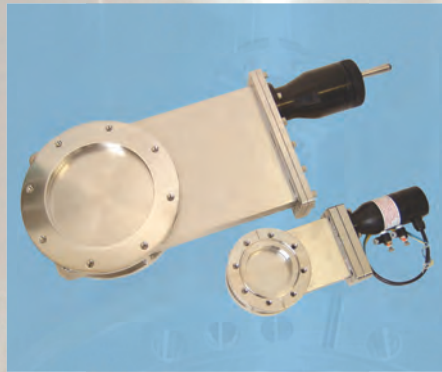
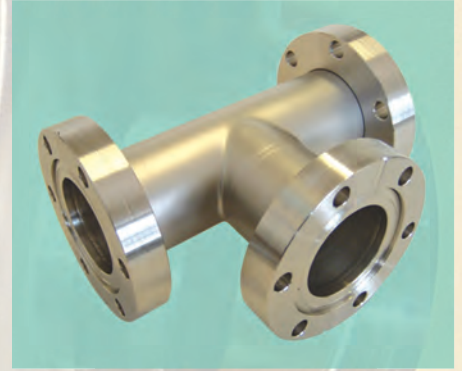
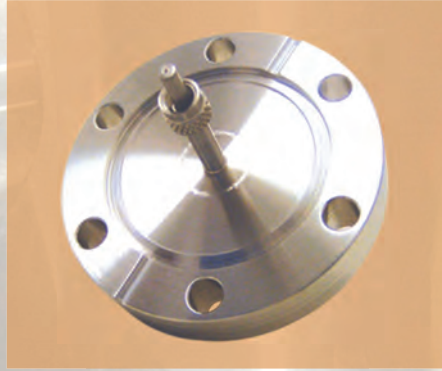
DIETRICH HABAS/ILL

compared with the unimpeded half of the beam. As the energy of the gamma rays was increased, the falling refractive indices, which had decreased into small negative numbers, suddenly flipped sign and started to increase. By replacing the silicon prisms with higher refracting materials like gold (pictured), it might therefore be possible to increase refraction to a level where it can be manipulated for optical techniques. ILL scientist Michael Jentschel said: "Twenty years ago many people doubted that you could do optics with X-rays – no one even considered that it might be possible for gamma-rays too. This is a remarkable and completely unexpected discovery." (*Phys. Rev. Lett.* **108** 184802).



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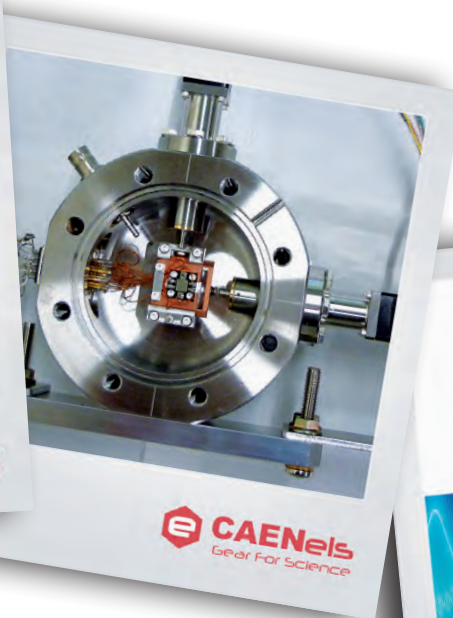
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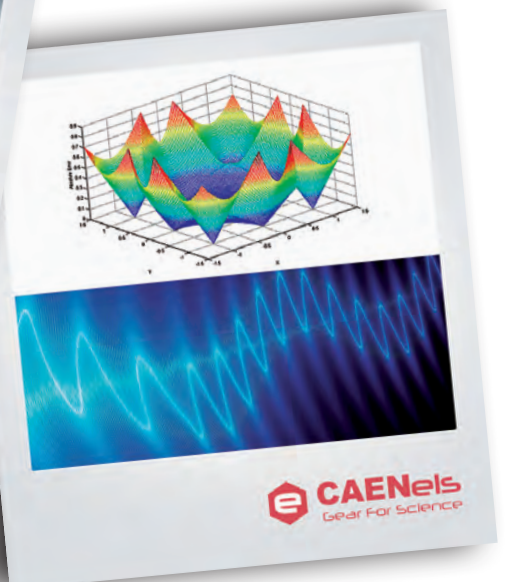
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