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ESRF Newsletter

Inside catalysis

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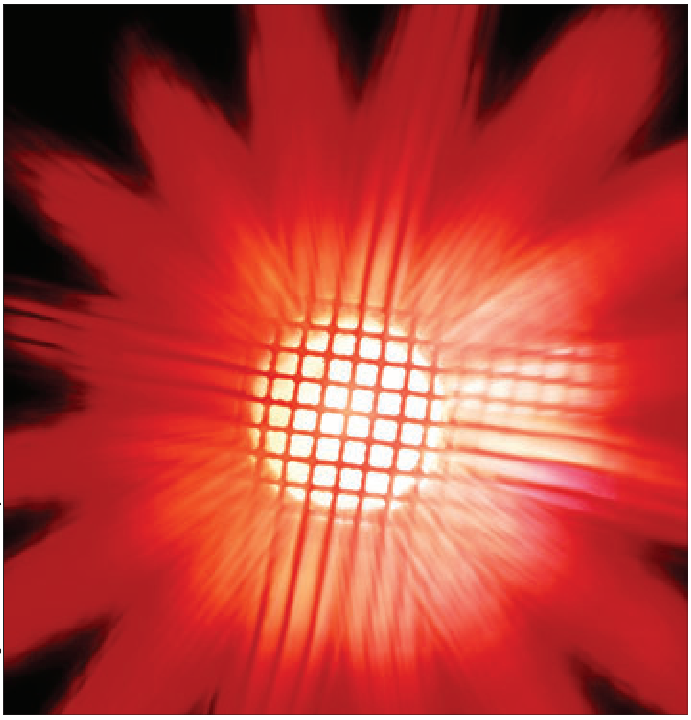

Cover image: Science Photo Library/A Pasieka

Exploring catalysis. Synchrotron sources are helping research into understanding catalysis. pp5–12.

On the cover: Abstract view of a catalytic converter inside a car exhaust.

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Editorial

Guest editors: Mark Newton and Roberto Felici

CURRENT SUCCESSES AND GREAT EXPECTATIONS FOR CATALYSIS AND SYNCHROTRON LIGHT

We may not often appreciate it, but the products of heterogeneous catalytic processes pervade our lives either directly (e.g. in the catalytic converters of cars) or indirectly (via products like fuel and plastics, which are derived from catalytic processes). Heterogeneous catalysts are one of the cornerstones of our global industrial economy, and full control of catalytic processes is one of the main goals of material science research today.

At the heart of the function of catalytic materials lies a plethora of nanoscale physics and chemistry, the full understanding of which has remained as elusive as it is highly desirable. Indeed the 2007 Nobel Prize for Chemistry awarded to Prof. Gerhard Ertl reflects this quest for such fundamental understanding. Rational, "bottom up" catalyst design is still, however, a largely unfulfilled dream.

While synchrotrons have been used to study catalytic materials for as long as synchrotrons have existed, a real revolution in the depth of understanding – at all levels of interrogation – that can be achieved has evolved rapidly over the last few years.

This has been the result of combining the enhanced properties that third-generation X-ray sources like the ESRF can offer with, for example, rapidly advancing detector technology. From the perspective of the world of catalysis research, the most important breakthrough has been the recent and rapid augmentation in the level of sophistication of the experiments being placed between the X-rays and their detectors. This is making it possible to span the material and pressure gaps that often divide academic and industrial research. This innovative synergy is resulting in an explosion of new experiments and possibilities that were quite unthinkable even a few years ago.

Making new discoveries

With respect to this field, the ESRF hosts several beamlines that have been, and continue to be, at the core of this new wave of research and development. ID03 has developed *in situ* surface-diffraction facilities that are world leading. ID24 and ID26 both have different capacities of X-ray absorption measurements that are currently unmatched anywhere in the world. ID11 and ID15 too are only just starting to be discovered by catalysis researchers, but already some entirely new and potentially paradigm-changing experiments have taken place there. Many of the collaborative research groups (CRGs), such as SNBL, DUBBLE and BM32, are also heavily involved and innovative amid the range of research types that the catalysis umbrella covers, and which their national communities are engaged in.

The articles featured in this issue of *ESRF Newsletter* highlight the research by Bert Weckuysen, Joost Frenken, Jan-Dierk Grunwaldt and Naoyuki Hara – all ESRF users. An insight into their work gives us a taste of just how central synchrotron-based research has become in tackling the diverse range of problems that this challenging, important and intriguing arena presents.

Goodbye ESRF Newsletter

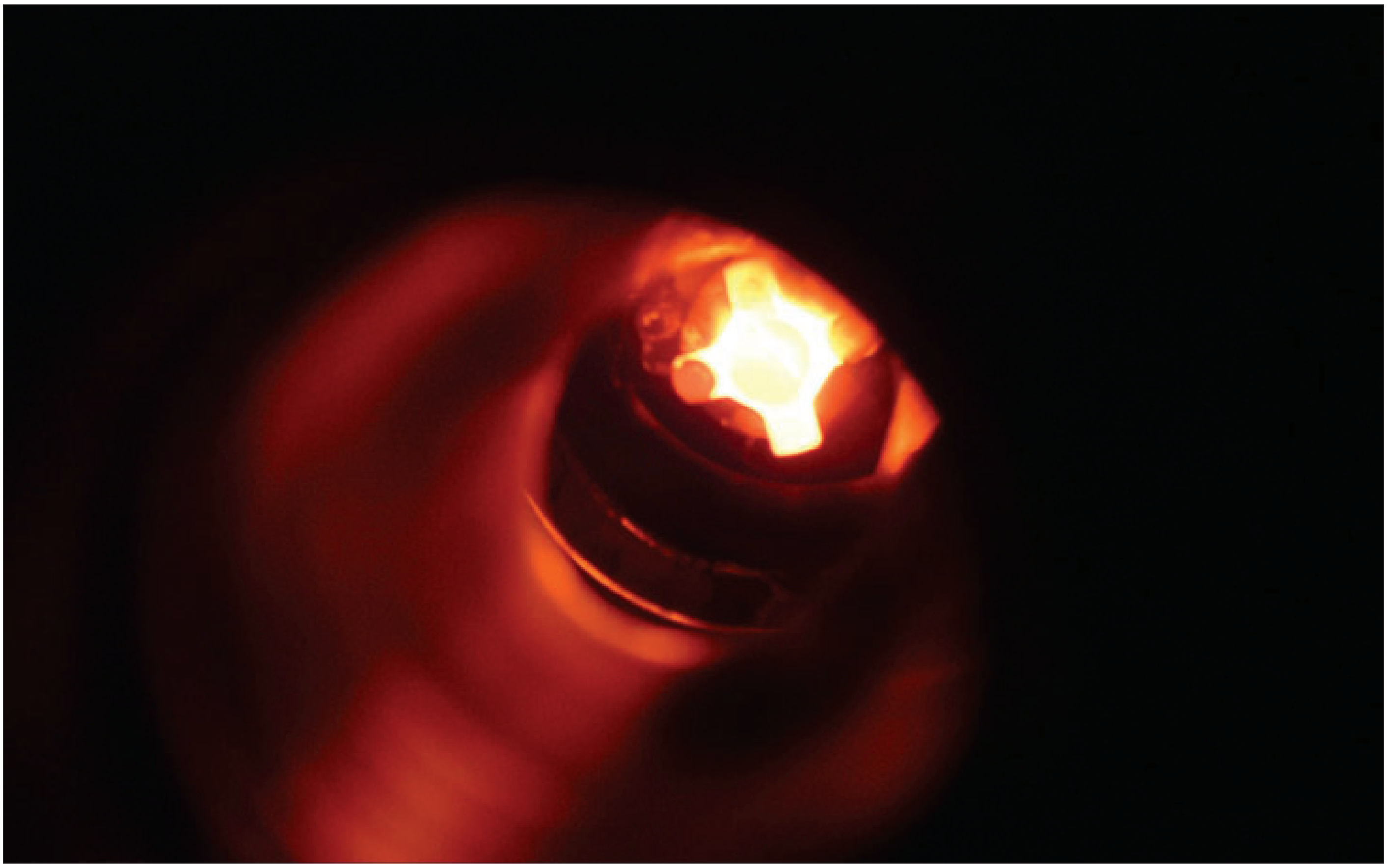
Hello ESRFnews

The *ESRF Newsletter* will become *ESRFnews*, the European Light Source magazine, from the next issue.

Coinciding with the 20th anniversary of the ESRF, the flagship magazine of the ESRF will feature a new design and sections – all to keep our readers abreast with what happens in the European synchrotron community.

ESRF

Feature news: catalysis



M Ackermann

Catalytic process. This sample was heated to 1000 °C in preparation for its use with operando X-ray diffraction experiment.

IT'S WHAT HAPPENS ON THE SURFACE THAT COUNTS

Recent surface studies using X-ray diffraction have revolutionised investigations into what happens on surfaces during catalysis, and several common assumptions have been overridden. Beamline ID03 at the ESRF is at the forefront of this research.

Since the 1960s, scientists have exposed samples, prepared under ultrahigh-vacuum (UHV) conditions, to small amounts of gases in an attempt to understand surface-chemical reactions. Electron-based techniques offer an insight into general reaction pathways, but these studies – often limited to single-crystal surfaces – don't take into account the high pressures often necessary for these reactions. Recently, technological advances have allowed the use of surface X-ray diffraction to determine the parameters of surfaces under ambient pressure. In some cases the outcome of this research has been spectacular.

A collaboration from the University of Leiden and the ESRF examined the elementary steps in the catalytic oxidation of carbon monoxide (CO) on platinum and palladium surfaces. This is one of the reactions that take place in the three-way catalyst in cars, which converts toxins in exhaust gases into less harmful substances. This catalyst consists of small particles of platinum, palladium and/or rhodium, which convert CO into carbon dioxide (CO_2) using oxygen (O_2). Being the only reaction path observed under UHV conditions, this led to the conclusion that CO and O_2 were absorbed onto the surface, and O_2 is split into two separate O atoms. Atomic oxygen (O) and CO then come together on the surface, resulting in the formation of CO_2.

However, under certain circumstances of high pressure (1 bar) and high temperature (>450 K), the catalytic process can run along a different path. In this case the surface first forms an atomically thin layer of platinum or palladium oxide. The CO molecules that come into contact with this layer immediately oxidise to CO_2. This process only occurs when the O_2 pressure is relatively high with respect to the corresponding pressure of the CO.

The formation of this thin layer of oxide on the catalyst was long considered to be detrimental to the catalytic process, but the University of Leiden—ID03 collaboration proved that the layer performs a vital role in the optimal functioning of the catalyst.

The formation of thin oxides on a model catalyst responsible for the CO oxidation also led scientists from University of Leiden to study the oxidation of a palladium (Pd(100)) surface. First, researchers used a scanning tunnelling microscope to study the effect with high temperature and noticed an increase in CO_2 production in conjunction with a roughening of the surface. Later they performed *ex situ* UHV experiments and realised that an intermediate oxide formed on the surface. It wasn't until they conducted experiments using diffraction that they could confirm the formation of a rough-bulk palladium oxide on the surface layer – and that this was the cause of the increase in CO_2 production.

The possibilities offered by ID03 also allowed scientists from University Giessen and the University of Lund to observe the surface structure of a rutherium-based model catalyst using a high-pressure chamber as well as X-ray diffraction. They were also able to monitor in real time how CO_2 formed during the reaction by simultaneously using mass spectrometry.

The team noticed that the activity of the catalyst increases in a transient way whenever it undergoes structural changes. It also discovered that the catalyst works stably at 400 K, but that above 500 K it starts to deactivate and stops completely at more than 650 K.

These are only some examples of how surface X-ray diffraction has become a valuable tool for studying the morphological and structural changes of single atomic layers at a surface during catalytic reaction. •

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CATALYSIS EXPERT TAKES A FRESH LOOK AT X-RAYS

Bert Weckhuysen is well known in the field of catalysis for his work on *in situ* spectroscopy but, since his 2001 appointment as a professor at Utrecht University, he has discovered the capabilities of synchrotron radiation.

Chemist Bert Weckhuysen admits that he is not a technique developer but that he tries instead to uncover methods to elucidate his scientific queries. "In our research we have questions, and we are looking for the best answers," he says. This approach adds to the legacy left by his predecessor, Diek Koningsberger, of using X-ray spectroscopy, which led him to the method of using synchrotron radiation in his regular research. His background in optical spectroscopy was also key.

Utrecht University

Bert Weckhuysen in his laboratory.

Weckhuysen combines different techniques to study mostly heterogeneous catalysts while the reaction takes place, if possible. The team at his laboratory in Utrecht mainly uses vibrational spectroscopy (i.e. Raman and infrared spectroscopy) and optical techniques (ultraviolet-visible-near-infrared and fluorescence spectroscopy), often in combination with a microscopic device.

Synchrotron radiation becomes important when studying catalytic solids that contain dispersed metal particles or metal oxides. "It provides unique opportunities for doing high-brow research. In most of the cases, synchrotron radiation enables characterisation studies, which are impossible to carry out at a regular university lab. On the other hand, it takes some time before the huge amount of data is worked out and fully understood," he explains.

Exploiting synchrotron techniques

Despite being relatively new to synchrotron radiation, Weckhuysen has already exploited different synchrotron techniques in his experiments. As well as using the ESRF, his team members are regular users of other light sources around the world. At the ESRF the Weckhuysen group uses diffraction, scattering, fluorescence and absorption techniques on beamlines such as ID24, ID26 and BM26. When his researchers travel to the ESRF, they carry their Raman and ultraviolet-visible spectrometers with them. "Each of the techniques helps us to find one bit of information about our experiment, like in a puzzle. By piecing them together we can obtain the full picture," he says. Weckhuysen and his team continue to explore new possibilities and they are currently focusing on using synchrotron infrared light in their research.

The potential of synchrotron light in catalysis research is no secret to Weckhuysen. He points out, however, that there are certain limitations to take into account. Back in 2004, his team made a surprising discovery: that some samples were damaged by the beam when the reaction was taking place. The scientists only found this out because they used different techniques on the sample, and, when synchrotron radiation was applied, the reaction occurred much faster. "This limitation is, luckily, manageable because we can change the parameters of the beam to avoid it," says Weckhuysen. He also acknowledges the difficulty of getting beamtime, "especially when you wish to apply special methods. It is a pity that some proposed experiments can never take place because of tough competition for beamtime."

Weckhuysen likes to innovate but, when asked about new possibilities for synchrotron radiation use in the next decade, he is cautious: "Predictions are risky; also in science. I have noticed that the moment you wish to predict something, someone else may have already been working on that specific topic. In any case, improved time and space resolutions are really necessary in the field of heterogeneous catalysis and I hope that in the years to come, synchrotrons will help us to push the current limitations." •

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ADVERTISEMENT FEATURE

SAES Getters CapaciTorr D400 Pump.

NEG module mounted within a Varian SIP.

SAES® Getters' advanced getter pump technology is adopted by the ALBA Synchrotron Light Laboratory and integrated within ion pumps at PETRA III, DESY.

saes getters

SAES Getters Group, the worldwide market leader in getter components for sealed-off devices and vacuum tubes, provides a wide range of Non Evaporable Getter (NEG) pumps and tailor made NEG pump devices for supporting UHV-XHV applications. Furthermore, a very large number of machines around the world encompassing electron and positron storage rings, synchrotrons, ion colliders and nuclear radiation facilities, now employ SAES Getters' NEG pumps in their vacuum chambers and systems. SAES Getters is therefore pleased to continue to serve the accelerator community with the implementation of its advanced getter technology to help meet the vacuum requirements of both the new ALBA Synchrotron Light Laboratory in Spain, and the PETRA III facility at DESY in Germany.

Non Evaporable Getter (NEG) materials are well known in vacuum technology to be an effective means of removing molecules through the process of chemisorption on the getters' active surface, and also by providing high sorption capacity through thermally driven diffusion of the sorbed gas into the getter bulk. The getter material is carefully prepared in the form of sintered bodies or laminated getter strip and assembled into specially designed cartridge pumps resulting in a high pumping speed for active gases. The two families of products developed by SAES Getters, the SORB-AC® and the CapaciTorr® getter pump series, have been widely adopted in UHV-XHV systems and optimized over the years for high energy machines. Following thermal activation the SAES Getters' NEG approach requires no electrical power when operated at room temperature and delivers extremely high gas capacity even in the presence of high electric and magnetic fields. Since there are no moving parts the SAES Getters' NEG pumps are also vibration free, very reliable and extremely easy to use.

The ALBA Synchrotron Light Laboratory (CELLS consortium) is a 3rd generation light source located in Cerdanyola del Vallès, a high technology zone just outside of Barcelona. This 3 GeV Synchrotron incorporates the SAES Getters CapaciTorr series of NEG pumps in combination with Ion Pumps to maintain an average dynamic pressure at 1×10^{-9} mbar to achieve a beam lifetime >15 hours at the designed current (400 mA).

The PETRA III Light Source at DESY Hamburg results from the rebuild of the existing 2.3-kilometer-long ring accelerator PETRAII. In addition to 1 km St707 getter strip mounted in the storage ring vacuum system it will utilize SAES Getter Wafer Modules (WP950) within Ion Pumps used in the beam-lines in order to increase in a very compact manner the local pumping speed and capacity for photo desorbed gases of photon masks and shutters.

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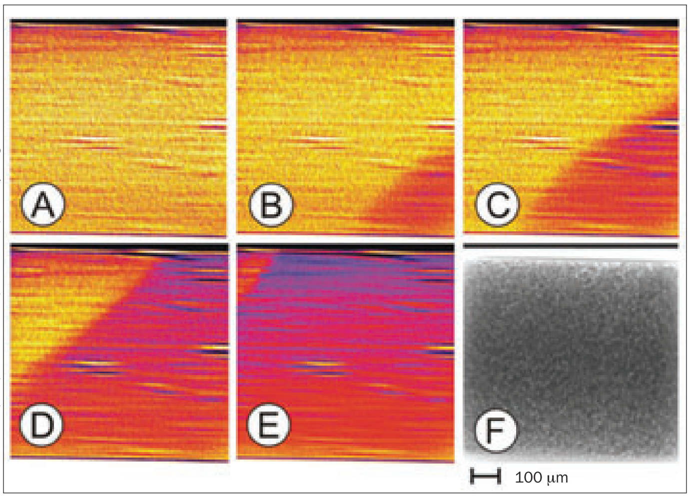
SCIENTISTS SHED LIGHT ON CATALYSIS MECHANISMS

Catalysis involves complex processes. Understanding catalysts on an atomic level should lead to an improvement in the performance of existing catalysts and help in the design of new ones. Thankfully, today's technology allows scientists to generate a much clearer picture of what happens during a catalytic reaction.

Synchrotron radiation makes it possible to carry out *in situ* experiments where scientists can monitor nanomaterials while the catalytic reaction takes place under realistic process conditions. It is not that simple, however, to recreate the real space and time conditions and at the same time monitor the structure and performance. This needs to combine physics, chemistry and reaction-engineering. Some teams, though, have taken on the challenge.

The partial oxidation of methane could be used for the production of hydrogen and synthesis gases (a mixture of CO and H_2). This is regarded as a first step in the so-called "gas-to-liquid technologies" to transform natural gas into liquid feedstocks, such as methanol. A change in a chemical reaction with time and space is often correlated to a variation in the state of the catalyst. Scientists therefore want to follow these changes in a spatiotemporal manner with a real catalyst to understand further what happens inside it.

Scientists from the Technical University of Denmark (DTU) and the Swiss Federal Institute of Technology in Zurich (ETH) specialise in monitoring reactions, such as the oxidation of methane. This is made possible by the X-rays of different synchrotron-radiation sources worldwide, the ESRF in particular.



J-D Grunwalt (DTU) / C Schroer (TU Dresden) and Spotlight on Science

X-ray absorption through a catalyst bed during partial oxidation of methane. These images show the whiteline energy of Pt (Pt L3-edge; 11586 eV) as a function of time: (A) t1; (B) t1 + 1 s; (C) t1+2 s; (D) t1 + 5 s; (E) t1 + 33 s; (F) transmission image; (A) to (E) were obtained by subtraction of the X-ray absorption image at time t from (F), which was collected at $t < t1$ (t1 is the time of the last image where no gradient was found).

2D monitoring of catalysts

"Only synchrotron sources provide the high intensity of X-rays needed for this kind of experiment. Hard X-rays have the excellent property of being able to penetrate materials, which allows us to engineer catalytic reactors that mimic the reactions under real conditions. This permits us to monitor the structure of the catalysts at the same time. That is also the reason why the excellent staff at the synchrotron sources make the effort to build up our catalytic reactors directly at the beamlines, although we could run them more easily directly in our own laboratory, but without looking into the reactor," explains Jan-Dierk Grunwaldt, the professor leading the team that has recently moved from ETH Zurich to the Department of Chemical and Biochemical Engineering at DTU.

One of the latest achievements by the group is the insight into the change in structure of palladium catalysts while the methane is oxidising. This was obtained using a flame-made catalyst that underwent a reduction and sintering of palladium particles during the oxidation of methane at more than 750 °C. Scientists noticed that this led to reduced catalytic activity by using X-ray absorption spectroscopy (XAS), X-ray diffraction (XRD) and online catalytic data at the Swiss-Norwegian beamline at the ESRF. This was the first time that the three techniques were used simultaneously at such high temperatures, and the results revealed a direct correlation between the structure of the catalyst and its performance.

The team, in collaboration with a group from the Technical University Dresden, Germany, also studied the structural changes of rhodium particles on ignition of the aforementioned catalytic partial oxidation of methane. On this occasion, the scientists used a spectroscopic cell with a gas supply and a CCD camera, and they combined X-ray absorption spectroscopy and online mass spectrometry at ID26. Thanks to these novel techniques, scientists were able to carry out spatially resolved 2D monitoring of the catalyst inside the catalyst bed (figure 1). They observed strong changes in the structure once the catalytic reaction was taking place along the catalyst bed. This became a Spotlight on Science (number 46) on the ESRF website.

These technical developments have also allowed the team to investigate the formation of nanomaterials together with Greta Patzke of the University of Zurich, such as tungsten/molybdenum oxides with a hexagonal tungsten bronze structure. These oxides belong to the family of transition metal oxides, which are interesting for selective oxidation reactions. The scientists could observe their transformation into other mixed nanostructures only at temperatures of more than 300 °C in atmospheres containing oxygen and hydrogen. The use of XAS and XRD at the Swiss-Norwegian beamline provided complementary information about the change in structure in short- and long-range order. By using additives during hydrothermal synthesis, the researchers could observe how the oxides formed and changed morphology.

The team now faces the challenge of combining XRD and XAS with Raman spectroscopy, which recently became possible at the Swiss–Norwegian beamline. "The first experiments seem very interesting. Apart from the possibility to study further important reactions, the spatiotemporal development of catalysts and tomographic studies are especially fascinating," explains Grunwaldt. •

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C Argoud

A cleaner future. Scientists at Toyota have been working with the ESRF to produce more environmentally friendly automobiles.

TOYOTA AND ESRF: WHEN GREAT MINDS THINK ALIKE

There are almost 10 000 km between Grenoble (France) and Aichi (Japan), but distance is not an issue when great minds think alike. A collaboration between the ESRF and Toyota R&D has led to new discoveries regarding car catalysts for greener cars. However, it is not only the Japanese who benefit from this collaboration.

Toyota's website shows artistic images of parts of a car integrated with nature, such as a flower growing from a rear-view mirror. The message behind these is Toyota's aim to make cars that produce zero emissions. The company is already commercialising the Toyota Prius, a car that consumes petrol in a more efficient way than the majority.

Behind the scenes, though, many years of research are needed before accomplishing a feat like the Toyota Prius or any other environmentally friendly automobile. In addition to carrying out research in their labs, Toyota scientists started using synchrotron sources to investigate catalyst noble-metal particles in static conditions at the Japanese light source SPring-8. "After the first experiments we wanted to analyse a catalyst while 'in action' and found out that we could only do it on ID24 at the ESRF," explains team leader Yasutaka Nagai.

Sakura Pascarelli, the scientist in charge of ID24, was not convinced at the beginning that the experiment was feasible. However, the collaboration between her group and the Toyota scientists soon proved successful. These days, Toyota puts together an international team of between six and eight people from Japan and Belgium who come to the ESRF twice a year as industrial users and exploit up to 500 hours of beamtime.

The experiment's success can be explained partly by the team behind the collaboration. "Gemma Guilera, the scientist in charge of the industrial activities on ID24, has done a terrific job through the years in adapting and further developing ID24 to make it more attractive for our industrial users," Pascarelli explains. "She also played a fundamental role in coordinating the activities between the beamline and the ESRF support groups, who were largely involved in our projects with industry."

When the Toyota team first arrived, the technique of turbo-XAS (X-ray absorption spectroscopy) was just being introduced on ID24. This acquisition method allowed the Japanese researchers to acquire X-ray absorption spectra using fluorescence detection with the required time resolution, on samples so dilute that they could not be investigated using the classical transmission geometry. Thanks to their interest in this technique, further developments were pursued in different scientific areas. Now Turbo-XAS is widely used, such as to study rocks and minerals of interest to geophysicists and geochemists.

The ESRF went even further to achieve a positive outcome to the experiments. The scientists needed a very fast detector, so the ESRF detector group implemented an ESRF-developed fast read-out detector on ID24 – the FRELON camera – which allowed acquisition of about one spectrum per millisecond. Today other researchers benefit from the use of this camera.

Results on board

However, the Toyota scientists' requirements didn't end there. The building of a cell where automotive catalysis could be reproduced was the next challenge of ID24 staff and the sample environment group of the ESRF. The cell needed to be compatible with the geometrical constraints of the fluorescence detection and also to reach very high temperatures and reproduce rapidly oscillating, reducing and oxidising atmospheres, "Just like in the catalytic converter of a car," says Pascarelli. At present, this cell can reach up to 1000 °C. However, because of the geometrical constraints, gases are fluxed on the surface (in the form of a powder pellet), not through, the sample.

These big efforts bore two particularly fruitful results. The first was the successful time-resolved observation that platinum redispersion takes place when this noble metal is embedded in a matrix with zirconium, ceria and yttrium. As a general rule, when catalysts age, the particle size of the noble metals increases. This makes the catalyst less efficient and ultimately unusable. In their research, Toyota scientists observed that the platinum particle size of an aged catalyst would grow to 7 nanometres, but it would decrease back to 5 nanometres after one minute, and then to 3 nanometres after 16 min.

The second result was the better understanding of the inhibition mechanism of platinum sintering, which will be disclosed at an international conference this year. This outcome has a direct impact on the catalyst development at Toyota. "The *in situ* XAS technique is very useful, and we believe that it will become more important in the future. The ESRF will continue to contribute to this kind of research," explains the Toyota team.

In 2007, with the aim of creating an even more realistic environment for research on catalysis (a technique called operando), the ID24 team widened the portfolio of techniques available to industrial users. At the same time Toyota moved its interest to samples that can be investigated using the more classical transmission geometry. "This change in geometry allows us to adopt a different concept, where the gases flux through the catalyst powder, simulating the exact situation inside a car catalyst," explains Pascarelli.

The scientists are now focusing on operando analysis, combining energy-dispersive XAS with infrared spectroscopy. XAS yields local and electronic structure information around the active metal site in the bulk of the sample, whereas infrared spectrocsopy tells us about the species on its surface. The ID24 staff developed and implemented a system where both techniques could be used simultaneously and in synchrony on the scale of milliseconds. "This set-up, coupled to the mass spectrometer, gives us a complete picture of what is going on in real time," asserts Pascarelli.

At present the Japanese researchers are building a specially designed beamline at SPring-8. This is another step forward in achieving the aim of having more environmentally friendly automobiles. "The construction of this new beamline, which will be very similar to ID24, proves that the research at the ESRF is very useful for them," comments Pascarelli, "and at the same time they have helped us to push our research capabilities forward." •

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Feature news

L Hardy

Making the earth move. The mechanical engineering group carried out vibration tests in January to determine the zones most prone to vibrations from planned construction works. A 1200 kg iron cylinder was dropped from 3 m, leaving a 5 cm indent.

UPGRADE: PURPLE BOOK TO BRICKS AND MORTAR

The ESRF recently announced a tender for architects to design the buildings of the Upgrade Programme.

Just before the close of 2007, the ESRF submitted the two-volume *Science and Technology Programme 2008–2017* report to its funding bodies for consideration and started a range of preparatory activities for its Upgrade Programme, thanks to a €5 m grant from the European Commission (EC).

The last issue of the *ESRF Newsletter* was largely devoted to the plans and proposals for the Upgrade Programme and its objective to preserve the world-class status of the European Light Source for another decade. This is a summary of the progress to date.

The autumn 2007 meeting of the ESRF Council unanimously endorsed the Upgrade Programme as the basis for further development of the ESRF. All member countries have since indicated on several occasions that they are attempting to identify funding possibilities for this programme over a period of 7–10 years. However, proposals for other European research infrastructures are currently in the final phases of funding discussions. Several ESRF member countries have put into operation first-class national light sources. That is why funding for the ESRF Upgrade Programme requires member countries to embed their decision into carefully balanced national planning for photon science infrastructures. To help with these efforts, the ESRF management has developed and presented member countries with a staggered scenario for the Upgrade Programme, with the second stage beginning some five years after the first, and each phase featuring core and optional investments.

The €5 m grant under the Framework 7 Programme of the EC has allowed important progress to be made with the design and planning of the Upgrade Programme, notably in the preparation of the experimental hall extensions and the designs of the new beamlines. The experimental hall extensions are a core element of the Upgrade Programme. An additional floor surface of 20 000 m^2 will provide much-needed breathing space, not only for up to 14 new long beamlines with nanofocusing capabilities but also for laboratories and infrastructures like computing. The timely construction of these buildings is a key factor in the overall planning of the Upgrade Programme.

In February 2008 a tender was issued to five preselected architectural teams. The successful proposal will be announced in July and the winner will assume the role of prime contractor for the construction of the new buildings. Construction is due to commence in 2010, after another year of further design and preparation.

The extensions present many technical challenges, in particular the strict vibration requirements of the floor and the need for perfect architectural integration into what has become a landmark building for the Grenoble area. The budget ceiling is low, yet many environmental and operational factors must be taken into consideration. Not least, construction-related shutdown periods must be as brief as possible to avoid interrupting users' work.

Once the building is complete in early 2011, existing beamlines can be relocated and new ones set up. To prepare the conceptual designs for the candidate beamlines, workshops will take place throughout 2008 to refine the plans outlined in the *Purple Book*. The remainder of 2008 will be a busy time for staff, users and the funding bodies, as the Upgrade Programme makes its first steps from paper to reality. ●

CLAUS HABFAST

BUILDING SANDCASTLES IS NOT JUST CHILD'S PLAY

The ESRF recently helped scientists to gain an insight into the complex interactions when a bond is formed by adding water to granules of sand. The results revealed the perfect recipe for making "sticky" sand.

The typical summer image of children building sandcastles on the beach seems far removed from international physics collaborations, yet a recent project undertaken by researchers from Germany, Australia and the ESRF has shown that these activities actually have something in common. Their findings confirm what any child already knows: that too much water will turn a sandcastle into nothing but a pile of mud. More important, however, is that the results reveal that the recipe for making perfect sandcastles is quite forgiving. Researchers were able to produce "sticky" sand with varying amounts of water.

ISTOCKPHOTO.COM/W Dunkin

Grains of sand bond with water to form polyhedral structures, or "sticky" sand.

Scientists from the Max Planck Institute for Dynamics and Self-Organisation in Göttingen, the University Erlangen, the Australian National University in Canberra and the ESRF studied the complex fluid structures of moist granules using X-ray microtomography on beamline ID15. First they measured the distribution of liquid inside a pile of glass beads. Later they analysed wet sand. As liquid was added to the mixture, they discovered that simple liquid bridges formed between the particles. These merged into polyhedral structures, such as trimers, tetrahedra and pentamers.

Surprisingly, the mechanical stiffness, or bond, of the wet sand remained more or less constant with moisture levels ranging from less than 1% to more than 10%, although the internal fluid structure changed enormously. However, beyond a certain point, the sand became saturated when too much liquid was added to the mixture, making the granules lose their "stickiness". By studying both glass beads and wet sand, the scientists were able to demonstrate that grain roughness is not an important parameter in forming a bond.

Understanding the complex interactions that govern the principles of sandcastle construction is a light-hearted side of a serious topic. It is precisely these structural relationships that will inform an understanding of certain natural catastrophes, such as landslides. "Wet granules are relevant to many fields and we now have a better understanding of their mechanical properties," says Stephan Herminghaus, head of the study at the Max Planck Institute.

MC

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DIFFRACTION AND TOMOGRAPHY PUSH BOUNDARIES

Researchers from the ESRF, the Institut NEEL and the Louvre Museum in Paris have recently developed a technique that combines X-ray powder diffraction and tomography to study microcrystalline samples.

Powder X-ray diffraction is a technique used to solve new crystalline structures, identify phases, analyse phase transformation and examine microstructural features. Thanks to recent technological developments, it can also provide high-lateral-resolution images. However, such two-dimensional mappings do not offer information about depth resolution, so this technique cannot be used to study materials with structured heterogeneities. To overcome this limitation and improve sensitivity at the same time, scientists from the ESRF, the Institut NEEL-CNRS/UJF and the Louvre Museum joined forces to use the synchrotron X-rays to reconstruct cross-sectional images of unidentified phases in nanomaterials and polycrystalline materials.

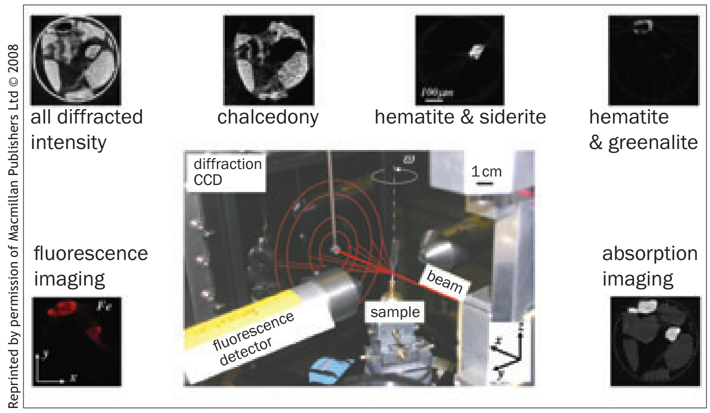
With this objective in mind, the researchers developed a technique on beamline ID22 that combines diffraction and microtomography. This applies simultaneous absorption, fluorescence and diffraction-data measurements while translating and rotating a sample that is illuminated with a focused or pencil beam. Thanks to the high flux combined with a fast read-out and low-noise (FRELON) camera, the scanning time for a 100 μm^3 sample is about six hours, resulting in a series of diffraction images, fluorescence spectra and absorption sinograms (images representing raw data). The sinograms represent a particular crystalline phase, elemental distribution and attenuation coefficient. The scientists then apply a mathematical inversion formula to the sinograms, resulting in cross-section images for each of the three measurements.

Although limited by the size and number of crystallites in the gauge volume of the sample, this method offers clear advantages: it can give contrast when other modalities cannot, provide multimodal images for a complete sample characterisation and allow the reconstruction of unknown crystalline phases without any previous information. It is also extremely sensitive because many diffraction images are recorded at different positions and angles.

The researchers first used a textbook powder and later a diamond to evaluate the new technique, identifying and locating five different phases. In the diamond the results showed a well crystallised cubic diamond in the central part of the sample, embedded within an amorphous carbon sp^3 phase matrix. In addition, crystallised ferrite grains were identified at the sample surface, as well as one single impurity grain of calcite. This was an indication of the method's sensitivity. Ferrite came from some contamination due to the razor blade that was used during sample extraction, while a trace of calcite was probably the result of a dust particle in contact with the pressure cell. For this weakly absorbing material, absorption and fluorescence tomography do not provide any contrast, while diffraction tomography reveals the distribution of phases (crystalline and amorphous) inside the sample.

Pencil-beam diffraction tomography links the quantitative structural global probe (like X-ray and neutron-diffraction methods) and the local compositional probe (such as X-ray fluorescence and absorption computed tomographies or electron diffraction).

PIERRE BLEUET



Solving new structures. Top: four reconstructed cross-sections corresponding to the entire diffracted intensity and three particular phases on a textbook powder. Bottom left and right: the fluorescence and absorption reconstructions.

Reference

P Bleuet *et al.* 2008 *Nature Materials*. 10.1038/nmat2168.

ESRF WELCOMES SOME NEW BIG GUNS

Several newcomers with international experience have filled some leading executive positions at the ESRF.

Whether you're walking along the corridors, cycling around the ring or simply deciding what to have for lunch in the canteen, you'll find that the ESRF is often full of familiar faces. The fact that the facility has more than 600 employees and 6000 users a year doesn't change the welcoming atmosphere and the feeling of community among its staff and visiting scientists. Some positions are more prominent and public than others, so it makes a difference when someone new takes over the helm.

Head of Users' Office: Joanne Mc Carthy

C Argoud

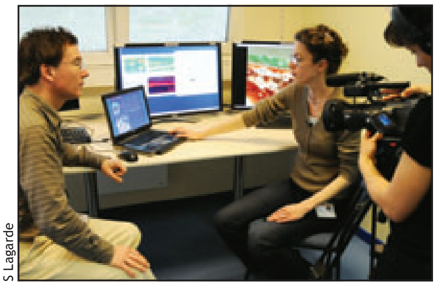
The main task of the head of the Users' Office is communicating with users. The former head, Roselyn Mason, held the job from when the position was first created up until her retirement in April. Joanne McCarthy (pictured on the right) is the new head of the group. She is a British physicist and was the previous beamline operations manager in the macromolecular crystallography group. Her experience as a scientist, as well as in dealing with the administration associated with the user programme of the MX beamlines, has given her good foundations on which to base her new role.

Head of theory group: Patrick Bruno

T Meinicke

After 10 years as director at the Max Planck Institute for Microstructure Physics in Halle, Germany, Patrick Bruno decided that he wanted to have more time for his research on magnetism in solid-state systems and it was a suitable time to go back to his home country, France. The 43-year-old theoretician now faces the challenge of building up the ESRF's theory group in terms of both staffing and fields of study. He predicts that magnetism, dynamics in nanostructures and condensed-matter physics will be three key lines of research in the future, stressing the importance of identifying the interests of users and beamline scientists.

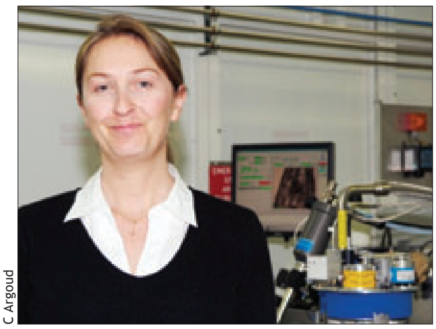
Head of communication: Claus Habfast



S Lagarde

Communication with both users and the general public is a key aspect of every research laboratory. Claus Habfast (pictured on the left) joined the ESRF last October as the new group leader of the communications unit. He has previously held positions as head of ESA TV at the European Space Agency and as a researcher at DESY and CERN. High on his list of things to accomplish are to inspire his new colleagues to organise European media visits and put more news on the ESRF website. In the medium term he wants to develop a centre for visitors together with the neighbouring Institut Laue-Langevin.

Business development manager: Diane Brau



C Argoud

Industrial users of the ESRF will soon meet Diane Brau, the new business development manager. Since March 2007 she has been the first point of contact for industrial users. She has a strong background in commercial and marketing activities, having gained experience from her previous job at the British detectors company Photonics Science.

MC

The machine

THE LATTICE EVOLVES AND IMPROVES BRILLIANCE

Brilliance (or the number of photons produced per second, per unit surface of the source, per unit solid angle and per unit energy bandwidth) is the figure of merit referred to when characterising the performance of a synchrotron radiation source. It is inversely proportional to the two transverse (horizontal and vertical) emittances of the stored beam, so the lattice design is an important factor in any improvement in brilliance.

Twenty-five years ago, during the era of the early designs of low-emittance lattices for third-generation light sources, there was great concern about the feasibility of such designs. To reach their goals of small emittance they needed to be pushed far beyond anything previously experienced with operational lattices. The commissioning of the ESRF in 1992, the first third-generation source, was straightforward using this design lattice. The original lattice has since undergone a series of modifications, all of which significantly improved performance levels well beyond the target goals of the original *Foundation Phase Report*.

At the design stage the ESRF storage-ring magnets were arranged with the intent of producing an expanded Chasman—Green lattice (i.e. a double-bend achromat lattice with zero dispersion in the straight sections, where the insertion devices (ID) are installed). The 32 straight sections had alternating high (32 m) and low (0.5 m) horizontal beta functions, which currently provide 5 m of space for IDs (figure 1).

The different stages of the lattice's evolution include the following important modifications:

- In 1995 the lattice settings were changed to allow for a finite dispersion in the straight sections by detuning the quadrupoles in the achromat. This new dispersion pattern, which is more balanced in the dipoles where the emittance is created, enabled the horizontal emittance to be decreased from 7 nanometres down to 3.7 nanometres. This, together with the coupling control below 1%, brought an increase in brilliance by a factor of 30.
- Also in 1995, one of the shortest zero-current bunch lengths ever achieved in a circular machine (4 ps) was obtained by running the lattice in a quasi-isochronous mode (with a momentum compaction of 1.5×10^{-5}, 10 times smaller than the usual mode). This remained an exotic tuning because the ultra-short bunch length could only be achieved at a very low beam current due to turbulent bunch lengthening.

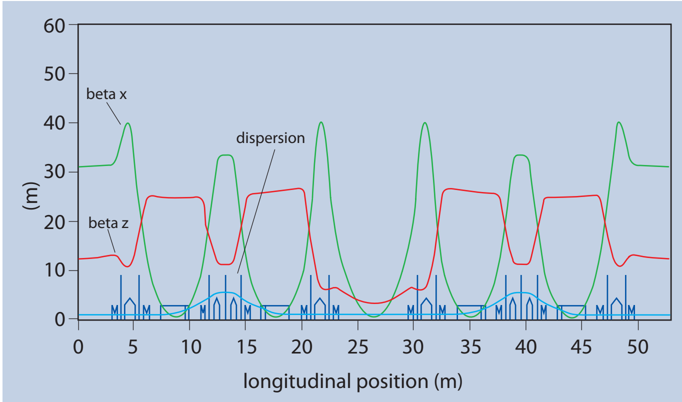


Figure 1. The lattice functions of the original design lattice.

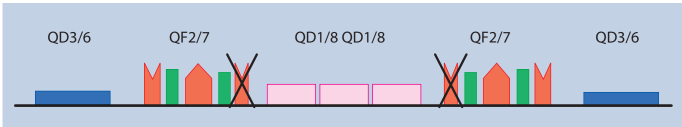


Figure 2. The layout of a straight section, with bending magnets (blue), quadrupoles (red) and sextupoles (green), and the three segments of an undulator (pink). QD1, QF2 and QD3 are the quadrupoles in a high-beta straight section, while QD6, QF7 and QD8 are in a low-beta straight section.

- In 1996 the vertical beta in the undulator straight sections was reduced from 13 to 2.5 m. This opened the way for low gap IDs because the modification of beta functions reduces the beam scraping in an ID vacuum vessel with a reduced beam vertical stay-clear, which consequently minimises the beam gas scattering lifetime reduction and the Bremsstrahlung emission on the beamlines.
- The horizontal focusing optics were successfully commissioned in 2000, providing virtual focusing of the electron beam downstream of the beamline and a reduction of the horizontal beam spot at the sample location, resulting in a significant increase in spectral flux per unit surface. This operation of the lattice with modified optical functions in the middle of a straight section opened up the field of detuned straight sections and breaking of the lattice symmetry.

Since 2006 the programme's aim of increasing the length of selected straight sections from 5 to 7 m has been tackled. The first stage of this programme was achieved in 2006 with the implementation in user service mode of a new lattice in which the two quadrupoles (QD1 and QD8) located on both sides of the IDs were no longer powered. These quadrupoles can be removed, allowing for longer (6 m) IDs to be installed.

The optical functions of the new lattice are shown in figure 3. Initially the lifetime of this lattice was shorter than what was achieved with the lattice used from 1996 to 2006. This was vastly improved in 2007, thanks to an increase in the vertical tune by one integer and the re-optimization of the harmonic sextupoles. The lifetime of the new lattice now surpasses the lifetime of the previous lattice in all filling modes (as illustrated in figure 4 for the multibunch fillings). As far as electron beam sizes and divergences are concerned, the only noticeable change is in the vertical plane, which is from the changing of the vertical beta from 2.5 to 3 m.

The final step in the increase in ID straight-section length from 6 to 7 m consists of shortening the long QF2/QF7 quadrupoles and moving the adjacent sextupole. This involves manufacturing new quadrupoles with a gradient of 25 T/m, but they can still be extrapolated from existing designs. This upgrade would provide greater brilliance and greater ID flexibility; longer IDs for a single beamline; sharing of a straight section between two experimental stations using the canted undulator approach; reshuffling the layout of the RF cavities; and freeing straight-section space for new beamlines (figure 5).

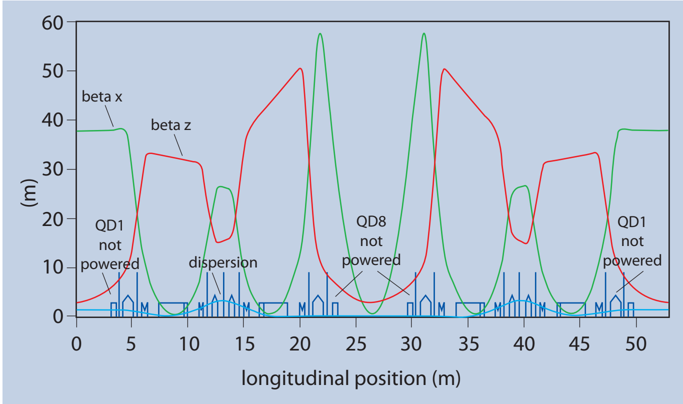


Figure 3. The optical functions of the new lattice design.

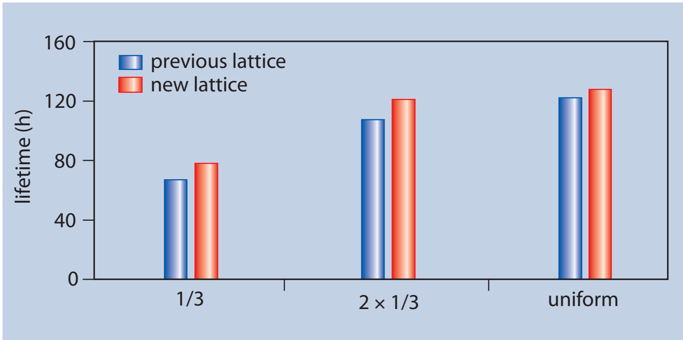


Figure 4. Modifications have increased lifetimes, illustrated here in multibunch modes with a ring current of 100 mA.

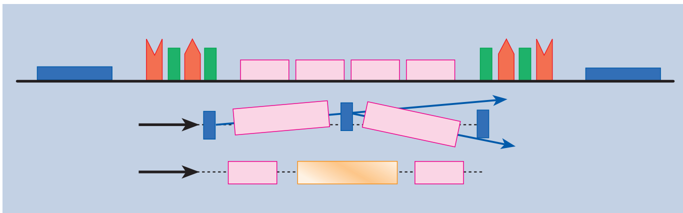


Figure 5. The flexibility of a 7 m straight section with four segments of insertion devices (pink); two longer canted undulators; and one RF cavity and two segments of undulators.

To minimize costs and shutdowns, 7 m straight sections will not be implemented on all straight sections, but one by one on a limited number of sectors. The drawback of this strategy is that it breaks the lattice symmetry and may have detrimental consequences for the dynamic aperture, resulting in a drastic reduction in injection efficiency and lifetime. Extensive computations and experiments are under way to simulate the symmetry breaking produced by a 7 m straight section. Obviously the behaviour of a 7 m straight section cannot be tested because magnets and coils have not yet been manufactured. On the existing lattice, however, it is possible to detune a single straight section and break the periodicity by powering each magnet of the straight section individually. Resonances are excited in a similar way as for a 7 m straight section, so experimental studies of detuned straight sections provide an efficient method of assessing the feasibility of the 7 m straight sections. This capability was implemented several years ago on three high-beta straight sections (ID4, ID6 and ID20) and one low-beta (ID11), which allowed the testing of different combinations of detuned straight sections. No impact on machine performance in terms of injection efficiency, lifetime and on-momentum horizontal aperture was observed for the different tested configurations. Preliminary drafting and design work of a 7 m straight section will start soon.

A ROPERT AND L FARVACQUE

User's view

C Argoud

Gerlind Sulzenbacher's research focuses on enzymes.

"We try to do as much as we can at our lab, but there are times when you need a synchrotron to continue the research."

GERLIND SULZENBACHER: A EUROPEAN BIOLOGIST

This scientist was born and raised in an Italian village that was part of Austria until the end of the Second World War. Her career has taken her to countries that include Denmark, Germany, the UK and France, where she currently lives and works. When asked where she is from, she replies with a smile: "I am European." Just like the ESRF.

Gerlind Sulzenbacher exhibits a persona that closely matches the European spirit of the ESRF. In addition to her pro-European outlook, she has built a career as a biologist and has been a user at the ESRF since 1999. She is currently a research engineer at the CNRS, University of Marseille, and her research is focused on studying the enzymes that are involved in sugar metabolism and understanding reaction mechanisms.

Despite having used other facilities, such as Daresbury, DESY and the Swiss Light Source, while holding previous positions, Sulzenbacher uses the ESRF most frequently. "It is very practical for me and the team," she says. "Because our measurements take from 10 minutes to an hour for each sample, we can do the experiments in 24 to 48 hour shifts," she explains.

Although the work at the ESRF is crucial for the team's success, the bulk of its experiments are conducted in the lab in Marseille. "We try to do as much as we can at our lab, but there are times when you need a synchrotron to continue the research," Sulzenbacher acknowledges. In this way, the team

TAKE NOTE

The Users' Organisation consists of representatives from each of the fields of research at the ESRF. These representatives are:

- **Gerlind Sulzenbacher (chair), macromolecular crystallography**
- **Stephan Roth, soft condensed matter**
- **Tullio Scopigno, high-resolution and resonance scattering**
- **Paolo Ghigna, X-ray absorption and magnetic scattering**
- **Chiara Maurizio, materials science**
- **Eric Maire, X-ray imaging**
- **Christian Kumpf, surface and interface science**

In addition, Thomas Buslaps is the ESRF's local liaison.

ONE BLOOD

The research on enzymes that can change certain blood groups is close to Gerlind Sulzenbacher's heart. In 2007 her team at the Architecture and Function of Biological Macromolecules Laboratory (CNRS–Université Aix–Marseille 1 and 2) published results explaining how an enzyme can convert group A and B blood into group O.

Blood group O is known as the "universal donor" because it can be transfused to all blood groups (A, B, AB and O) safely, but transfusing group A blood to a group B patient (and vice versa) causes an ABO accident, which can be lethal.

Labelling errors still cause deaths each year. Conversion of blood groups A, B and AB into group O (the universal donor) would avoid these risks. It would also have a great economic advantage, because transfusion centres currently have to carry – and constantly renew – stocks of four types of blood.

Sulzenbacher and her team discovered two families of enzymes with unique properties and took them to the ESRF to solve their 3D structure on beamline ID23. The enzymes proved so efficient that large-scale conversion of blood groups A, B and AB into universal donor group O are now feasible.

REFERENCE

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C Arguoud

Sulzenbacher hands Vincent Fernandez the Best Poster award at the Users' Meeting.

brings only selected samples that have previously been screened at home to the ESRF.

Sulzenbacher could be any other ESRF user if it weren't for the fact that she has been chair of the Users' Organisation since February 2007. Together with her colleagues, her principle duties in this role include promoting the research conducted at the ESRF by providing an organised framework for discussion within the users' community; establishing a direct link between the users and the ESRF management at the ESRF; and organising the annual Users' Meeting.

No real change

As daunting as these tasks may seem, Her life hasn't changed much from how it was before she became chair. She was already an active member of the Users' Organisation, representing the macromolecular crystallography group for two years. However, she points out that she visits Grenoble more often now to attend regular meetings in the role of a consultant.

Sulzenbacher still finds plenty of time for challenges. She wants to improve communication between the different communities that are represented by the Users' Organisation.

"Sometimes it is a shame that two communities, which may well work together, don't even know each other," she says. "For example, I am in the macromolecular crystallography group, but I believe that interaction with the soft condensed matter group could benefit my research. It is all a question of communication, and the ESRF Users' Organisation represents an ideal platform for that," she concludes. •

MC

Visiting a beamline

ID03 PROVES ATTRACTIVE FOR SURFACE SCIENCE

Since its upgrade in 2005 the ID03 beamline is one of the stars of surface science studies in catalysis at the ESRF. A combination of technical improvements and a competent, good-humoured team are the keys to its success.

To an outsider, the first impression of the ID03 beamline is that it is a dream place to work. There is a good level of understanding between the members of the team, who come from countries as culturally different as Italy and the Netherlands. Roberto Felici, who is in charge of the recently revamped beamline, has a constant smile. "Hard work is not incompatible with good humour," he says, while an equally smiling colleague, Didier Wermeille, shares this vision.


C Argoud

ID03 team. Most of the team enjoys the sun outside the beamline. Left to right: Roberto Felici, Didier Wermeille, Andrea Resta, Thomas Dufrane, Lucien Petit, Richard Van Rijn, Olivier Balmes and Helena Isern (inset).

Benefits of a smaller beam focus

It is precisely hard work that has made it possible to run the new beamline with such success since 2006. ID03 is an undulator beamline dedicated to surfaces and interfaces structural characterisations. It has a smaller beam focus than before the upgrade. This increases its flux density, together with energy regulation, which provides the opportunity for anomalous diffraction. The beamline is used to perform static-surface crystallography studies, as well as to look at processes at surfaces in real time. This is done either under ultrahigh-vacuum conditions, using a dedicated diffractometer coupled to a vacuum chamber, or in different experimental conditions on a second diffractometer, where it is possible to install specialised set-ups.

ID03's speciality is the structural and morphological characterisation of catalytic surfaces during reactions in real conditions. Recently the team has been focusing on studying the 3D structure of nano-objects, which can be prepared *in situ*, and the modification of their morphology induced by external conditions, such as temperature and atmosphere.

The beam reaches the experimental hutches without crossing any windows, so scientists can use the its coherence.

Roberto Felici was a beamline user at the time of the old ID03 and he acknowledges the progress of its research capabilities: "The stability of the beamline has improved a lot and we do not have to worry about the optical part of the experiment anymore. We can concentrate on the science itself and gain time."

The new possibilities that the ID03 offers have attracted significant interest from potential users, as well as many submissions of proposals for experiments. "It is difficult to measure how oversubscribed our beamline is because it fluctuates a lot between the March and September deadlines," explains Felici. Since the beamline opened, the volume of applications has varied a lot. "The numbers have still not stabilised," he says. "What is sure, though, is that we do attract more users than we can receive, so the beamline is attractive."

Another way of measuring the success of the beamline is by counting how many users celebrate their experiments with a glass of champagne at the end of their stay. It may not be an orthodox system, but it is an old tradition on the beamline that seems to work. Other traditions include a monthly lunch outside the ESRF and occasional skiing trips in winter. It might sound trivial, but these bonding activities create a wonderful working atmosphere among visiting groups.

MC

Scientific highlights

FIRST REPORT OF METAL–PEPTIDE FRAMEWORKS: 'BIOINSPIRED METAL–ORGANIC FRAMEWORKS'

Work done on the CRG BM1B Swiss–Norwegian beamline

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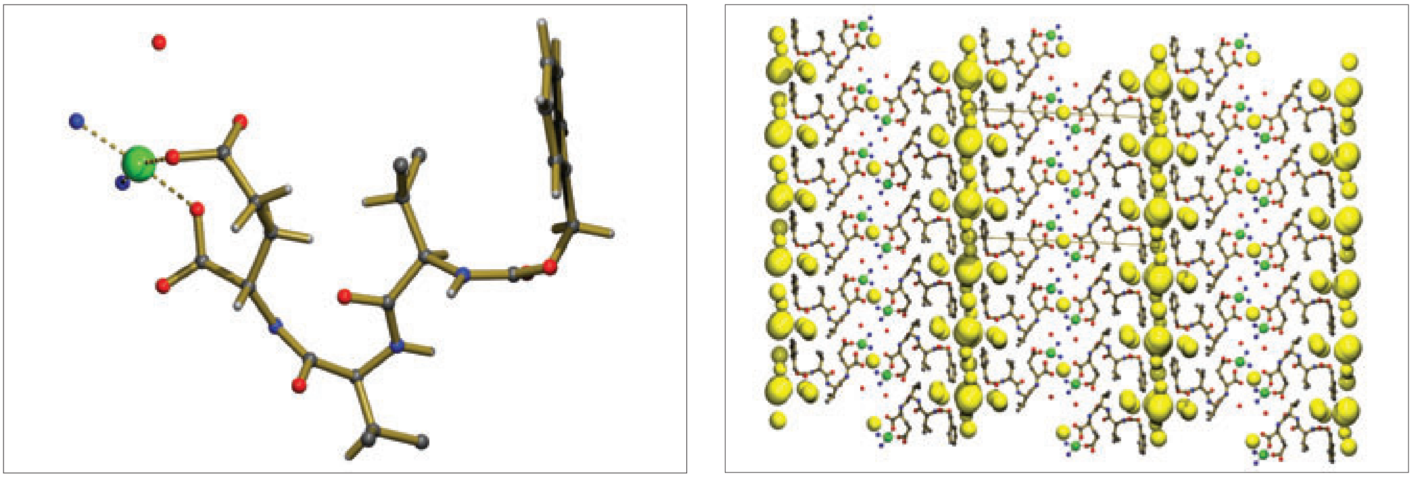

Refined model. The structure was determined with 'Free Objects for Crystallography' (FOX), a free, open-source program for *ab initio* structure determination from powder diffraction data. These images were generated by PLATON. The yellow balls are voids.

Metal-organic frameworks (MOFs) are crystalline compounds of metal ions and organic molecules. They form structures that are often porous and these pores can be used for the storage of gases such as H_2 and CO_2. Other possible applications of MOFs are gas purification, gas separation, catalysis and sensors. This work is the first report on metal peptide frameworks (MPFs), the "bioinspired" analogues to the well known MOFs. The MPFs were constructed from an oligovaline peptide family developed earlier by our group at the University of Basel (A Mantion *et al.* 2007). We have used a simple oligopeptide, Z-(L-Val)2-L-Glu(OH)-OH, to grow porous copper and calcium MPFs. The MPFs form thanks to the self-assembling properties of the peptide and specific metal-peptide and metal-ammonia interactions. They are chiral, are stable up to about 250 °C and have some internal porosity, which makes them a promising prototype for further developments.

Our work identified that metal-oxygen and metal-nitrogen bonding and various types of hydrogen bonding along with π-π-stacking are responsible for the formation of a porous network structure, which is stable up to about 250 °C. Despite the lack of single crystals, the crystal structure of the copper complex MPF-9 could be determined from powder diffraction data collected at BM01B on the Swiss-Norwegian beamline. The refined crystal structure is consistent with the spectroscopic information. The 1D channel architecture with two different pore sizes confirms the interest in MPF-9 for gas storage, gas or chiral separation or catalysis. Finally, the organisation of the Cu(II) ions in a 2D layer pattern yields a material with an interesting paramagnetic character. Here we therefore clearly show that small peptides are viable candidates for the preparation of complex metal-organic materials with defined topologies. MPFs thus further broaden the more general field of MOFs and entactic metal systems, which are interesting for a range of technical applications.

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FIRST RESULTS FOR SYNCHROTRON X-RAY POWDER DIFFRACTION IN PULSED HIGH MAGNETIC FIELDS

Research at the ESRF provides the first direct evidence for the modification of the co-operative Jahn–Teller effect in $TbVO_4$ by an external magnetic field

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Pulsed magnetic fields represent an economical way of accessing high magnetic fields beyond the superconducting limit (which currently stands at about 15 T). Recently this technique has been introduced at several synchrotron light sources. However, the short pulse lengths and long waiting times between pulses represent a significant challenge that can only be met through innovative measurement strategies, and adaptation of the coil and cryostat to the particular requirements of synchrotron experiments (figures 1 and 2). While the principle has been demonstrated at Spring-8 and the ESRF, experimental results remain rare. In a recent experiment on the CRG beamline BM26 (DUBBLE) at the ESRF, the scientists managed to overcome these difficulties and directly measured the effect that a 30 T magnetic field has on the Jahn–Teller (JT) effect in terbium orthovanadate ($TbVO_4$).

$TbVO_4$ is a textbook example of a material exhibiting the co-operative JT effect driven by phonon-mediated interactions between the terbium quadrupole moments. At high temperatures it crystallises in the tetragonal zircon structure, whereas on lowering the temperature to less than 33 K it undergoes a co-operative JT transition and the crystal spontaneously distorts along the [110] direction, lowering the symmetry to orthorhombic. The balance between $TbVO_4$'s magnetic and quadrupolar effects can be tuned by varying the strength of an applied field. The effect of large external magnetic fields on $TbVO_4$ has only recently been studied. It was predicted that the JT distortion would be suppressed

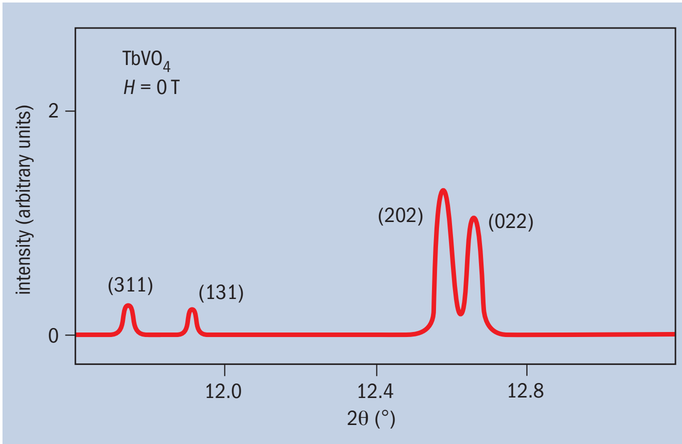

Figure 1. Part of the X-ray powder diffraction spectra of $TbVO_4$ taken at T = 7.5 K, showing the (311)/(131) and (202)/(022) pairs of reflections that are sensitive to the JT distortion.

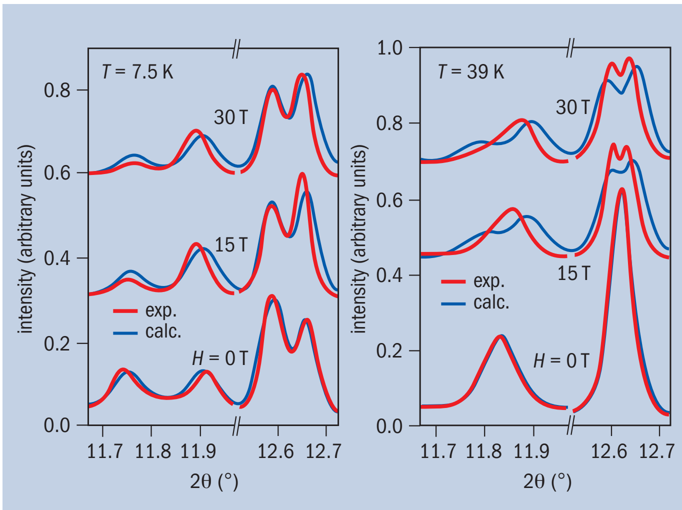

Figure 2. Comparison of calculated and measured spectra for various fields (0–30 T) and for temperatures 7.5 K (left) and 39 K (right). The red lines show the experimental data and the blue lines show theoretical calculations.

when fields of more than 29 T were applied along the c-axis of the sample (figure 2).

In this experiment the sample was placed in an environment that permits the application of high magnetic fields while monitoring changes using X-ray diffraction. The sample consisted of finely powdered crystals of $TbVO_4$ embedded in polyvinylpyridone to prevent movement of the powder grains due to magnetic forces. Data were collected by accumulating about 45 magnetic field pulses per powder diffraction spectrum. The JT transition manifests itself as a splitting of some of the powder lines in the spectrum due to distortion of the crystal lattice. Within the photon energy range studied, the (311)/(131) and (202)/(022) pairs of reflections are sensitive to the JT distortion (figure 1).

Spectra taken at different field strengths are presented in figure 2. Without an external magnetic field, the spectra taken at temperatures of 7.5 and 39 K closely follow theoretical predictions, and twin peaks in the former and single peaks in the latter can be seen. These temperatures are below and above the JT transition temperature, respectively. At 15 and 30 T, the spectra show splitting of the peaks at 39 K, thus providing evidence of the modification of the JT distortions of $TbVO_4$ by magnetic fields (figure 2). Although the observed splitting followed the predicted spectra, the degree of splitting was lower than expected. Various theories to explain the quantitative difference have been proposed, one being sample heating due to the magnetocaloric effect.

At the ESRF the technology for high magnetic fields will be developed further, with the aim of making this facility available at different beamlines.

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Gallery of events

ANNUAL USERS' MEETING PINPOINTS THE UPGRADE

The annual ESRF Users' Meeting and its satellite workshops brought together more than 400 users on 5–7 February at the European Light Source. The meeting focused on the latest developments and challenges of the Upgrade Programme. It also included two workshops on catalyst research with X-rays, and the structural biology of host–pathogen interactions.

Science at the nanometre scale was the main theme of the plenary session of this meeting. Ian Robinson from the London Centre for Nanotechnology gave a keynote lecture on the use of coherent X-ray diffraction for materials research in nanoscience. The ESRF management presented the latest news from the facility and its plans for the development of beamlines and accelerators in view of the Upgrade Programme. The meeting also included discussion sessions on the ESRF's future direction and a joint poster session.

The Brookhaven National Laboratory physicist Stuart Wilkins is the 2008 winner of the Young Scientist award. Before his appointment at Brookhaven, Wilkins was a beamline scientist on the ESRF beamline ID20. This prize recognises "his work on X-ray scattering studies of strongly correlated systems and in particular transition metal oxides". Since 2003, Wilkins has published 25 papers (some of the data were collected at the ESRF) and he was first or second author on more than half of these. •

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MEETING WORKSHOP FOCUSES ON CATALYSIS

We all use heterogeneous catalysts, directly in the catalytic converters of our cars and indirectly in products derived from processes mediated by a catalyst. Synchrotron-based research, especially at third generation sources, has recently started to open our eyes to how dynamic and complex the behaviour of these systems can be from atomic- to microscales. The ESRF organised a Users' Meeting workshop on the time-resolved study of heterogeneous catalysts and some of the processes that they mediate.

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Listening in. The catalysis workshop proved a great success.

The event attracted 118 participants, representing laboratories in 17 countries around the world. The diversity of the field and the relevance of X-ray-based study in this area were reflected by the participation of scientists based at 10 synchrotrons, with all of their differing capabilities.

A structured programme sought to assess the state of the art of *in situ* and time-resolved research by isolating technical areas of study: diffraction; small- and wide-angle scattering; surface X-ray diffraction and grazing incidence small-angle X-ray scattering; high-pressure photoemission; and X-ray absorption spectroscopies. A final session dealt with techniques that are emerging as potentially important methods for furthering our understanding of these problems: emission spectroscopy and resonant inelastic X-ray scattering; soft X-ray microscopy; pair distribution function methods in diffraction; tomography; and high-throughput methodologies for materials screening.

This varied structure highlighted that these materials require the coherent application of several techniques to gain the profound insight that is sought today. It is also clear that simply making an X-ray measurement is no longer satisfactory; the nature of the processes under study needs to be considered carefully.

The increasing importance and need for such synchrotron-based research, in the millisecond to minute range of time resolution considered in this workshop, was repeatedly demonstrated by the diverse, high-quality presentations. Prof. Bert Weckhuysen from the University of Utrecht, the Netherlands, gave a particularly adroit talk that indicated the importance of and rapid development in this area. Simply showing how the frequency of reports in this area has mushroomed in the last few years (using one of the world's leading general chemistry journals, *Angewandte Chemie*, as an example) spoke volumes for this increase in importance.

This growth in research on catalysis has produced an important need for detailed, time-resolved studies on a variety of lengthscales. The unique experimental possibilities provided by modern synchrotrons are therefore crucial for this research.

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STRUCTURAL BIOLOGY TACKLES PATHOGENS

The ESRF Users' Meeting international workshop titled Structural and Molecular Biology of Host–Pathogen Interactions attracted more than 150 participants. Organised by the Partnership for Structural Biology, it highlighted how structural biology studies have complemented cellular and molecular biology.

Half-day sessions were dedicated to three topics: bacterial adherence and invasion; virus and host cell factors; and immune response. The session on bacterial pathogens showed how various techniques, combined with macromolecular crystallography, have provided an insight into how bacteria assemble adhesion and secretion machinery to infect eukaryotic cells.

The session on virus entry and assembly provided a broader view, discussing mechanisms that viral pathogens, (e.g. HIV, influenza and rabies) use for assembly and replication. The contribution of structural biology, particularly synchrotron-based crystallography, has helped to define some of these. In the last session, presentations described how host cells respond to pathogens.

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