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ESRF news

Number 53 March 2010



Materials for energy

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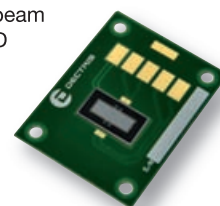
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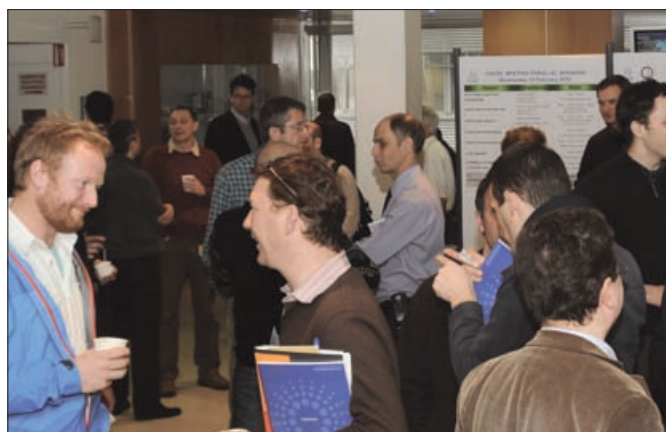
XBPM4 is a 4-quadrant x-ray beam position monitor based on CVD diamond technology, suitable for hard x-ray synchrotron beam lines.



A light for science



New 'greener' materials are already being used in transportation, p8



120 scientists gathered for the workshop on nanobeams, p16



Patrick Bruno, head of the theory group, received two prizes recently, p19



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Conceptual view of the use of hydrogen for a greener future.

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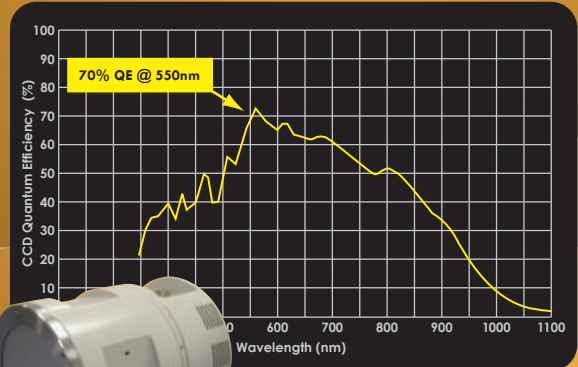
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Energy-related studies a high priority

The modern world runs on energy that, in the main, was captured millions of years ago from the Sun by photosynthesis in trees and other prehistoric flora. Today, by burning coal, oil and natural gas, we use this energy to fuel our transport systems, electricity grids, industry and agriculture, and to heat and cool our homes, work and leisure places. It is clear by now that this supply of fossil energy is limited, with some predicting that oil production will peak in the coming decade. Furthermore, returning large quantities of carbon dioxide to the atmosphere increases the amount of energy that it absorbs from the Sun with consequences for the balance of our planet's climate systems. There is a need to develop alternative sources of energy and to ensure that energy, irrespective of its source, is used as efficiently as possible, which then results in that energy having to be stored, delivered and converted into heat, light or motion with minimal losses.

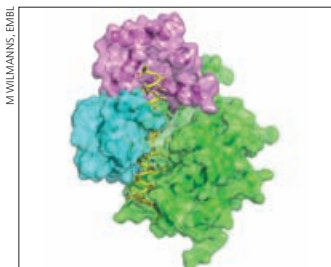
Materials research is at the heart of developments for modern energy systems. Photovoltaic panels convert sunlight into electricity that can be used locally or fed into the grid. The most common material currently used is bulk silicon, although there are worldwide efforts to produce more highly performing systems based on other semiconductors, thin films or dyes, etc. Fuel cells require a fuel, such as hydrogen, alcohols or hydrocarbons, that are oxidised electrochemically to produce electricity, which can then be used to power a vehicle with an overall improved efficiency as compared to burning the fuel in an internal combustion engine. The fuel is oxidised at the anode and is separated from the oxidant at the cathode by an electrolyte that allows ions to flow internally between the electrodes. The performance of the cell depends critically on these components and many new materials are being designed for optimum cost and efficiency. Similarly, new materials for rechargeable batteries, especially those based on lithium, are under development. If hydrogen is used as a fuel, a means to store it on a vehicle is needed. Hydrogen-storage materials, based on light elements that bind and release hydrogen under mild conditions are actively sought. Similarly, materials that can adsorb carbon dioxide or other pollutants can be used to help clean up the products of combustion from a fossil-fuel power plant, perhaps to be buried so that it does not enter the atmosphere.

Materials research is a main topic at synchrotron radiation sources and research on energy-related materials is being performed at many beamlines at the ESRF, most prominently at the beamlines of the Structure of Materials Group, that extend to high photon energies (ID11, ID15 and ID31 to name just a few). Studies by diffraction and spectroscopy reveal the composition, atomic structure and crystalline state of materials, and imaging allows defects and aggregates to be observed. *In situ* studies can be particularly effective to follow materials or even complete devices under operating conditions to watch the evolution of the components, e.g. the electrolyte in a working fuel cell or lithium-ion battery during the electrochemical cycle. From such observations, a better understanding of the performance can be obtained and thereby improvements and new designs envisaged.

In this issue of the *ESRFnews* you will find examples of research conducted at different ESRF beamlines on fuel cells (pp8–9), solar cells (pp10–11), hydrogen storage (pp12–13), and lithium-ion batteries (p14). Research on energy-related materials is one of the main drivers for the Upgrade Programme of the ESRF. The Upgrade Programme includes a significant increase in capacities with, in particular, an extension into the nanoworld, which becomes ever more important for the development of high-performance materials. Research on energy-related materials is a prime example of research performed at large-scale facilities with a high return for society. The Upgrade Programme of the ESRF enables us to boost our contribution for the development of a sustainable society in a resource-limited environment.

Andrew Fitch, group head of the Structure of Materials Group and responsible for ID31
Harald Reichert, director of research

“Materials research is a main topic at synchrotron radiation sources”



Science Signaling front cover image: 3D structure of DAPK (green and yellow) when bound to calmodulin (violet and blue), obtained by X-ray crystallography.

Communications system hacked

Cells rely on a range of signalling systems to communicate with each other and to control their own internal workings. Scientists from the European Molecular Biology Laboratory in Hamburg (Germany), have now found a way to hack into a vital communications system, raising the possibility of developing new drugs to tackle disorders like neurodegeneration, cancer and cardiovascular disease. Using the ESRF beamlines BM14 and ID29, as well as others at DESY, they have pieced together the first snapshot of what two of the system's components look like while interacting.

One way that these signalling systems work is by triggering a flood of calcium ions inside the cell. These get picked up by a receiver, a protein called calmodulin, which turns this calcium signal into action by switching various parts of the cell's machinery on or off. Calmodulin regulates a set of proteins called kinases, each controls the activity of specific parts of the cell, consequently altering the cell's behaviour.

The results revealed the molecular structure of one of these kinases, a protein called death-associated protein kinase (DAPK), when bound to calmodulin. The structure showed how calmodulin binds to a particular section of DAPK, switching the kinase on so that it can go and change the function of its targets. The team then worked out which of DAPK's building blocks, or amino acids, were crucial for calmodulin to bind.

Reference

I De Diego *et al.* 2010 *Science Signaling* **3**(106).

Partnership for Soft Condensed Matter moves full steam ahead



Richard Wagner (left), director of the ILL, and Francesco Sette (right), director general of the ESRF, sign the Memorandum of Understanding for the creation of the Partnership for Soft Condensed Matter.

For the Partnership for Soft Condensed Matter (PSCM), 2010 looks promising, after 2009 finished with the signature of the Memorandum of Understanding (MoU) between the ILL and the ESRF, and the first workshop dedicated to the collaboration took place.

It still does not have a physical place to be exclusively based, but the PSCM is taking shape fast. Until the science building becomes available, the PSCM facilities will function within the soft-matter laboratories available at both the ESRF and the ILL. But the full-scale operation requires a new building.

In November 2009, the ceremony for the signature of the MoU marked the institutional commitment to push the project forward. The PSCM will combine research with X-rays and neutrons, and will attract academic and industrial partners to fund and set up laboratory techniques and sample environments to strengthen what the ESRF and the ILL offer themselves. "We are confident of the success of this collaboration, after all, we are not first-timers regarding partnerships," explains

Serge Pérez, director of research at the ESRF. "The experience of the Partnership for Structural Biology (PSB), established in 2002, has shown us that this is a very good model for expanding the synchrotron's scientific capabilities," he adds.

The scientific programme of the partnership is already being defined. The first step started in December 2009 when the "Scattering and complementary techniques" workshop, co-organised by the ILL and the ESRF, brought together 90 participants. It aimed to gather potential collaborative partners from universities and other research institutions from within the member countries and associates of the ESRF and the ILL, as well as to discuss the scientific agenda of the partnership and to explore external funding.

The housing of the PSCM

By early 2013, the PSCM will be able to host 20–30 scientists and technicians working in 800 m² of laboratory and office space in the new science building of the joint ILL/ESRF campus. The recruitment of a scientist and a technician is

currently taking place.

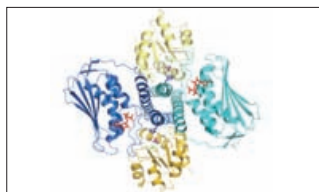
In these laboratories, scientists will prepare experiments with highly complex self-assembled and non-equilibrium soft-matter systems that cannot be easily transported to Grenoble. The planned facilities of the PSCM include instruments for on-site sample preparation, complementary techniques for pre-characterisation (e.g. microscopy, rheology, light scattering), complex sample environments (surface and interface techniques, systems in confinement, optical tweezers, etc), advanced data analysis and modelling, and a platform for networking and scientific exchange.

The PSCM will benefit the four ESRF soft condensed matter beamlines ID02, ID10A, ID10B and ID13, as well as beamlines such as ID9B, ID15 and ID16. The collaborating research groups at the ESRF, notably BM02, BM16, BM26 and BM32, may also take advantage of the new facility. Access will be through peer review and collaboration agreements when a partner contributes to the staff and equipment of the PSCM.

The senses of bacteria revealed

Bacteria, as well as other domains of life but not animals, sense their environment through protein couples called “two-component systems”: a sensor histidine kinase and an effector response regulator. The signal transduction process involves three steps: the sensor autophosphorylation, the phosphate transfer to the effector and, finally, the spontaneous or sensor-mediated dephosphorylation of the effector.

Scientists from the Instituto de Biomedicina de Valencia-CSIC (Spain) have unveiled the structure of the complex formed by a system of two components of the bacteria *Thermotoga maritima* in the process of signalling. The results clarify why each protein in the couple recognises specifically



Ribbon representation of the complex between the receptor dimer (blue) and effectors (yellow) viewed from the membrane.

the other protein. They also provide insight into how the phosphorylation state of each partner is recognised, as well as on the mechanism of the sensor-mediated dephosphorylation of the response regulator. In addition, the structure suggested a novel mechanism of sensor autophosphorylation that was confirmed experimentally.

The team carried out most of its research on beamline BM16, but also on BM30A and ID14-3.

Reference

P Casino *et al.* 2009 *Cell* **139**(2) 325–336.

Scientists research the sound of atoms

The sound that is produced by loudspeakers originates from vibrations induced when magnetic energy is transformed into mechanical energy. At an atomic level this occurs because some specific atoms (the so-called rare earths) strongly deform following the direction of the magnetic field and “move” the neighbouring atoms around.

Scientists from the ESRF, the INRIM in Torino (Italy) and the Institut Néel (France) have measured the tiny atomic movements responsible for the macroscopically observed giant magnetostriction effect. In a film of amorphous TbFe₂, researchers have managed for the first time to observe simultaneously the distortion of the electronic cloud in the Tb atoms and the tiny changes in neighbouring bond lengths (of the order of 60 femtometres). Their results have been selected as an editors’



Magnetostrictive materials are used in the stabilisation of suspension bridges.

suggestion for the journal *Physical Review B Rapid Communications*.

Being able to quantify the amplitude and sign of atomic displacements is important because, in general, these manifest the competition existing in matter between various energy terms. In magnetic systems, as is the case here, they provide a unique tool for analysing the coupling that exists between magnetic and elastic properties at the atomic scale.

Reference

S Pascarelli *et al.* 2010 *Phys. Rev. B* **81** 020406(R).

Users' corner

The most recent proposal deadlines were 15 January for long-term projects and 1 March for standard proposals for ESRF beamtime. The next beamtime allocation panel meetings to review proposals submitted for both deadlines will be 22–23 April. The next deadline for standard proposal submission is 1 September.

The 20th ESRF Users' Meeting and associated workshops took place on 8–12 February. More can be read about this in the dedicated article on p16.

The funding bodies regularly ask the ESRF for the most up-to-date information on publications resulting from experiments at the ESRF. This is why the scheme for uploading these into the database has been improved. After each proposal round, every user from the past three years will now receive a short e-mail with a link to a simple online form. This form allows the user to upload references to new publications (and also PhD theses, etc) with a single cut-and-paste operation. The ESRF/ILL library will check and add these into the SMIS

library database, ready for use in a later proposal round. The existing SMIS interface for adding publications to the database will continue to exist as an alternative to the online form.

News from the beamlines

● **Plenty of news from ID20.** One of the first beamlines to move into the new ESRF beamline floorplan will be ID20, which will move from its current location to ID06. Current plans foresee the closure of the beamline for part of the 2010/II and almost all of the 2011/I scheduling periods, resulting in a reduced amount of beamtime being available for allocation to user experiments during these two periods.

The Conceptual Design Report (CDR) for the new beamline on ID06 is available for consultation at www.esrf.eu/UsersAndScience/Experiments/ElectStructMagn/beamline-portfolio/CDR_future-ID06.pdf.

There is still some flexibility in the plans for the future of the beamline to adapt to users' wishes and suggestions: interested users are therefore invited to consult the CDR document and send any

comments or suggestions directly to Carsten Detlefs (detlefs@esrf.fr).

In the meantime ID20 boasts the following new features:

- Installation and commissioning of a new four-circle vertical diffractometer with an open chi-circle for azimuthal scans.
- A double phase plate in-vacuum set-up available for user experiments, enabling full polarisation control of the incident photon beam (circular and linear polarisations).
- Experiments under applied electric and magnetic fields combined with incident circular polarisation open up new possibilities to study the evolution of magnetoelectric domains in multiferroics.

● **The range of applications of the X-ray Magnetic Circular Dichroism (XMCD) studies at the ID12 beamline** is now greatly enhanced. A new experimental station, based on a 17 T superconducting solenoid magnet purchased from Cryogenic Ltd and equipped with a 2.3K cryostat, is now open to users. The newly developed instrument allows the recording of high-quality

XMCD spectra and element specific magnetisation curves using XMCD in the photon energy range 2–15 keV. A rather high sweep rate of the superconducting magnet allows one to record a hysteresis curve from –17 to 17 T in less than one hour. The purpose-built helium gas constant flow cryostat, developed in-house by the ESRF sample environment support service, allows users to have complete temperature control at the sample in the range 2.3–300 K. X-ray dichroism spectra are collected in total fluorescence yield detection mode using a silicon photodiode placed in the cold bore of the magnet, ensuring very high sensitivity and opening up new possibilities for the exploration of behaviour of spin and orbital magnetic moments under a high magnetic field in a variety of samples, such as crystals, thin films, multilayers and nanoparticles.

● **The ID14-3 bio-SAXS beamline** now has a new PILATUS 1M detector available for users that considerably decreases data-collection time and improves the accessible q-range to 0.05–5 nm⁻¹.

A look into polymers in fuel cells

Low emissions, quiet performance and high energy efficiency are the biggest assets of fuel cells, currently used mostly in transportation. These cells are becoming an optimum way to gear ourselves to a more environmentally friendly world.

Fifty years after their first application in aerospace, fuel cells are slowly getting closer to being part of our everyday life. Many buses use fuel cells and this is intrinsically coupled with the use of hydrogen as a source of energy. Unlike a battery, a fuel cell does not store energy, it transforms it.

There are several types of fuel cells and they are defined by the type of electrolyte that they use to convert the chemical energy into electrical energy, the kind of fuel involved and their operating temperature. The best known fuel cell is that based on a proton exchange membrane fuel cell (PEMFC), which can be used in portable and stationary applications, as well as in transportation. The core of PEMFC is a polymeric electrolyte membrane able to conduct the protons from the anode to the cathode. Its commercial variant is based on Nafion, by DuPont, as the polymer electrolyte.

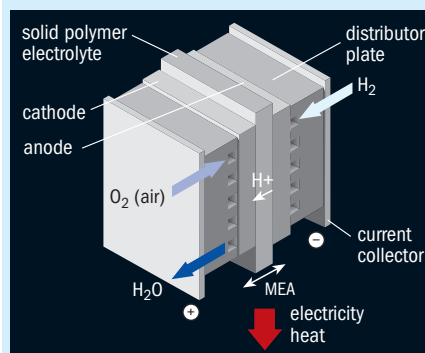
The current membranes are limited by different factors, such as the cost of the platinum-based catalyst, the cost of Nafion, the complex water management for the optimal ion conductivity and the permeability of these membranes to methanol, which could otherwise be used directly as fuel. The fuel for PEMFC is hydrogen obtained from methanol or a hydrocarbon, such as methane or propane, or from electrolysis of water, possibly conducted by using solar panels.

The electricity production in a fuel cell involves the reaction between the supplying gases (hydrogen and oxygen) flowing along the opposite faces of the membrane. Therefore, to meet oxygen, the hydrogen, in ionic form (protons), must migrate across the membrane from one surface to the other. The electrons produced on hydrogen oxidation are forced to pass outside the cell,



A Mercedes Benz Citaro London bus running on fuel cells. This kind of bus was first used in 2004 in the English capital. Several cities around the world already use fuel cells in their buses.

Take a look inside a fuel cell



The fuel cell uses hydrogen and oxygen to create electricity. The reaction occurs in a structure consisting of two electrodes (the anode and the cathode) separated by the electrolyte membrane, which lets the ions through. The electrodes activate the hydrogen oxidation as well as the oxygen reduction.

In the case of a proton-exchange membrane, the hydrogen at the anode is dissociated into protons and electrons. At the cathode, the oxygen, electrons and protons recombine to form water.

through an external circuit, and in this way provide the electric power. To effectively transport protons, the membrane needs to be humidified. However, an excess of water may produce cathode flooding and the consequent decrease of the cell performances.

Several groups are studying membranes like Nafion at the ESRF to monitor *in situ* the changes that it goes through during the oxidation and reduction processes, the aging of its nanostructure, or its hydration degree as a function of the operative electrochemical parameters. The scientists use beamlines such as ID02, ID13, ID15 and BM26.

Recently, a team from the Istituto di Struttura della Materia in Rome, University of Camerino (Italy), and the ESRF measured the water in a running fuel-cell membrane in real-life conditions. For this experiment, they used the high-energy beamline ID15B and determined the overall presence of water and the hydration degree in each layer of the membrane with the highest precision ever.

To observe the overall amount of water in the membrane, the team carried out the experiment by irradiating a Nafion 117 membrane, about 140 μm thick, with an X-ray beam with a cross-section equivalent to its

thickness. The researchers took a sequence of diffraction patterns that showed the water changes induced by changes of the working conditions. In this way, the variations in the degree of water could be correlated with the cell voltage.

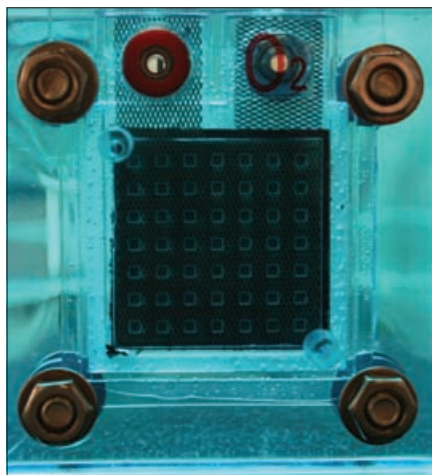
The team also carried out spatially resolved experiments to determine the water distribution along the membrane thickness. This helped the scientists to elucidate in detail the complex water dynamics occurring in the active component of a running fuel cell. Valerio Rossi Albertini, a member of the team, explains that: "the water dynamics in the membrane of a fuel cell is one of the main aspects in the use of such devices for locomotion. The variable working conditions, for instance because of the request of rapid increase of power supply during the acceleration of a vehicle, may produce dysfunctions and electrochemical instabilities due to water overproduction. Conversely, an insufficient hydration of the supplying gases or the heat release due to the proton current in the membrane may result in its drying. The method developed at ID15 can help in understanding and describing such complex water dynamics."

Hotter and drier

Hydration control is therefore a key aspect in membranes like Nafion and its temperature for optimal operation is limited to about 90°C. The current trend among scientists is to increase the operation temperature of fuel cells and, consequently, of the membranes, which will have to operate with less humidification.

To improve the mechanical stability of membranes like Nafion, which contain fluorine for proton conductivity, researchers usually increase their crystallinity or form composites. A team from the Max Planck Institute for Solid State Research, FuMATech (both in Germany), the CEA (France) and the ESRF has taken a different approach by developing a new kind of fluorine-free membrane. The membrane consists of a new synthesised poly(p-phenylene sulfone) ionomer containing merely sulfone units ($-SO_2-$) connecting the phenyl rings in which each ring is sulfonated ($-SO_3H$).

The team used the technique of X-ray scattering on the DUBBLE beamline to characterise their new membranes under controlled humidity conditions. They noticed that the high degree of sulfonation reached in these membranes translates into a microstructure characterised by very narrow hydrated, hydrophilic domains, which are well connected on longer scales. These unique microstructural features lead to the



A fuel cell on its oxygen side.

advantageous combination of high proton conductivity (especially at high temperature) and low electro-osmotic drag, i.e. low coupled transport of water and protonic charge carriers.

So far Nafion has been the most used polymer in PEM fuel cells, but its sensitivity to radical attack and its need for high humidification limits its operation temperature to about 90°C. This temperature is not high enough to minimise CO poisoning of platinum, used as the PEMFC anode catalyst, and to avoid

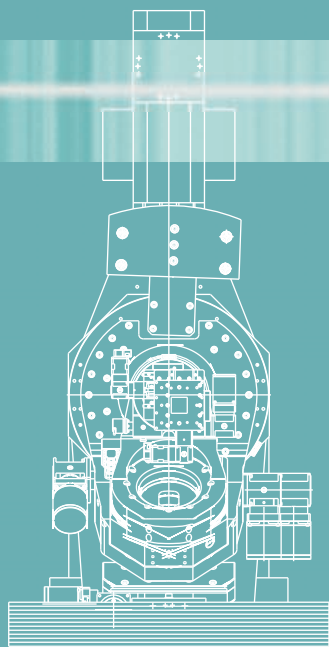
complex humidification and expensive cooling systems. The new fluorine-free poly(p-phenylene sulfone) developed by the Max Planck Institute in Stuttgart shows seven times higher proton conductivity than Nafion at 135°C. "All of this is thanks to its high ion-exchange capacity. The resulting unique membrane microstructure made of hydrophilic channels is much narrower and better connected than the ones found in Nafion, as revealed by X-ray scattering data of membranes under controlled humidity conditions. Poly(p-phenylene sulfone) or new similar ionomers, could replace Nafion in PEMFC in the future," explains Giuseppe Portale, from DUBBLE. There are some challenges on the way though. Klaus-Dieter Kreuer, from the Max Planck Institute, explains: "Unfortunately, the membrane mechanical properties are still not optimal for PEMFC applications. The next step is to develop poly(p-phenylene sulfone) membranes with better mechanical properties by forming composites, blends or even block-copolymers, but still preserving very high conductivity at high temperature." *M Capellas*

References

C C De Araujo 2009 *Phys. Chem. Chem. Phys.* **11** 3305–3312.

V Rossi Albertini 2009 *Advanced Materials* **21** 578–583.

New: Modular Beam Conditioning Unit



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The image shows an 85 kg, multicrystalline block of silicon with a base area of $42 \times 42 \text{ cm}^2$ and a height of 21 cm, corresponding to 2×2 columns, each with a finished vertical edge length of 156 mm. The columnar crystals that grew from the bottom to the top are clearly visible.

Scientists look closer at the breakdown of solar cells

With an efficiency of 20% of energy captured from the sun, silicon solar cells are the choice for commercial photovoltaic cells. Researchers from the Fraunhofer Institute (Germany) are working on improving them and have successfully used the ESRF to test their samples.

Silicon solar-cell technology has been around for more than 50 years, although it took two decades to start taking off. In recent years, new candidates for photovoltaic processes have emerged, such as polymer cells or thin films made of cadmium telluride, copper indium gallium selenide or amorphous silicon. Today, however, silicon solar cells are more effective and widely used in commercial applications, compared with polymers or thin films.

Currently, silicon solar panels can produce energy for at least 20 years, but a breakdown of the cells can shorten their life. A team from the Fraunhofer Institute for Solar Energy Systems in Freiburg (Germany) came

to the ESRF to carry out an experiment on silicon wafers less than a year ago. The focus of the research was to better understand the breakdown behaviour of solar cells by studying how metal precipitates occur in the wafer. This is the first step to avoid breakdown in production and to improve solar-cell processing by manipulating defects (e.g. through optimised temperature steps) to make the material better and enhance efficiency, even for cheap (and therefore not very clean) material. Despite the short time since their beamtime, the researchers have published three papers from their measurements on the nanoimaging station of ID22.

The researchers carried out two different experiments. They measured the breakdown location of finished solar cells, formed by a boron-doped substrate and a phosphorous diffusion at the front, by forming a pn-junction. The metals that they detected are located in the vicinity of this junction, so the scientists concluded that they might create high electric fields causing breakdown.

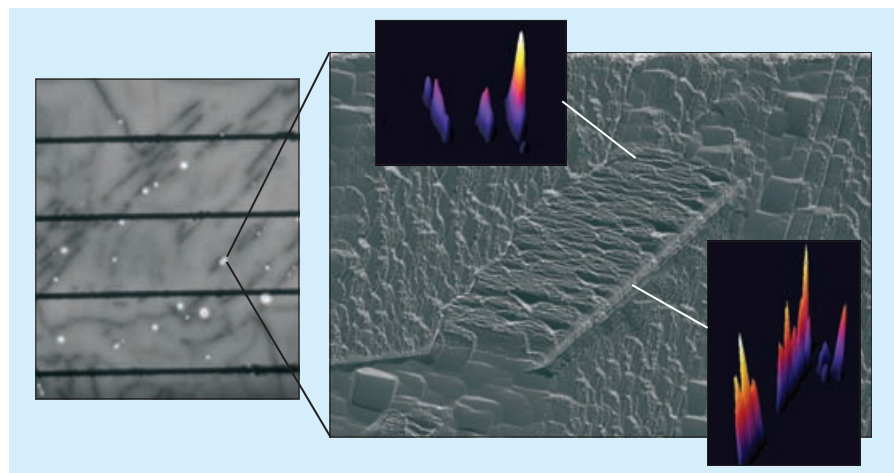
In another experiment, they used two boron-doped wafers that had been atomically bonded at the Max Planck Institute for Microstructure Physics in Halle (Germany) in a certain way to simulate a grain boundary (as present in multicrystalline silicon). A controlled

contamination with specific metals allowed them to study the precipitation behaviour of the metal atoms at the interface or grain boundary. Because the effect was isolated, scientists could study their samples far more precisely than they would on standard multicrystalline silicon.

Thanks to a beam size of only 100×100 nm and the high flux of ID22, the researchers could detect transition-metal precipitates with a diameter of the order of 10 nm. The team also noticed that grain boundaries house iron precipitate colonies at exactly the same position where breakdown sites have been detected. These groups of transition-metal clusters have been identified to be responsible for the local diode breakdown, which, in the worst case, destroys the solar panel.

In a previous study, the team observed that the breakdown behaviour of solar cells does not depend on the nature of the impurity but on its concentration. The latest results show that the breakdown seems to be induced by individual large clusters of more than 30 nm, rather than by smaller impurity particles finely distributed along the grain boundaries.

Investigations such as this one represent a small but important step towards a more generalised use of silicon solar cells. "Research in silicon solar cells is increasing, as new research facilities appear and industrial companies enhance their in-house research. We are all trying to bring down the cost, and this is the



Left: part of the solar cell that shows the dark recombination active features. Except for the straight black lines running from left to right, they belong to the solar cell and are situated at the surface of the silicon wafer. The large image on the right is an SEM image of the part of the solar cell that is indicated. The two 3D images are the metal precipitates.

goal of our research at the ESRF: to enable the use of cheap substrate material without losing efficiency," explains Martin Schubert, head of the "silicon material characterisation" team at the Fraunhofer Institute.

In an interview during the Solar Summit conference in Freiburg (Germany) in 2009, the director of the Fraunhofer ISE, Eicke R Weber, said that: "In 2050 it will be completely normal to everyone that solar energy is the way that

energy is produced." For some, 40 years may seem a lot, but it is a relatively short time for a future of, possibly, never-ending energy.

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Storing gases: a key

The quest to become more environmentally friendly has driven scientists to develop materials to store hydrogen or greenhouse gases. The ESRF has proven a useful tool in this rapidly evolving field.

Fossil fuels are becoming scarce and hydrogen is starting to be the next best thing for transport applications. Because hydrogen is the lightest element in the periodic table, it has the highest energy-to-mass ratio of any chemical, its main source is water and it does not pollute. Despite the great advantages, its use is still a challenge.

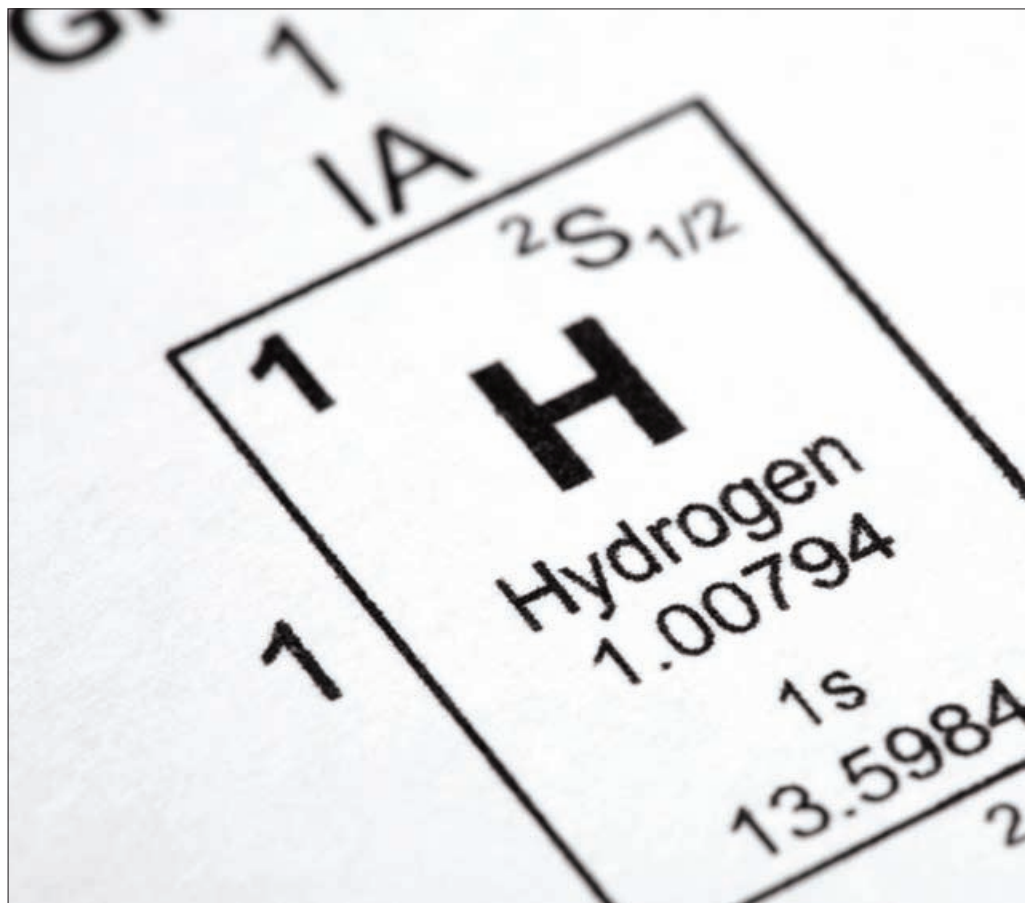
The mass production of hydrogen is the first obstacle that scientists face in the implementation of this element as a future fuel. Another drawback, and possibly the biggest, is that because hydrogen is a gas at ambient temperature, it takes up a lot of space. This is why the scientific community is trying to find the perfect formula to store it. Once stored, it could produce energy by combustion, or fuel cells could convert it into energy electrochemically.

There are many different approaches to the question of storing, and the subsequent release of, hydrogen. The fact that the future systems would have to store and deliver the gas to a fuel cell in mild conditions makes it more difficult for the community to find solutions.

Hydrogen-storage approaches can be divided into physical storage, where hydrogen molecules are captured by porous solids or pure hydrogen is compressed or liquefied, and chemical storage, where hydrogen atoms are chemically bound to other elements.

Hydrogen can be reversibly stored in certain solid materials. Today, scientists focus on compounds of light elements with hydrogen in order to discover the successor for petrol, which needs to have high hydrogen-storage capacity both by mass and volume. Three classes of the materials currently receive significant attention, namely the borohydrides, which contain boron and hydrogen, amides based on nitrogen and hydrogen, and alanates containing aluminium and hydrogen.

Several research groups come to the ESRF to study hydrogen-storage systems at different beamlines, such as BM1, ID31 and ID11. A team from BM1 has recently explored stable borohydrides, such as LiBH_4 and $\text{Ca}(\text{BH}_4)_2$, which have very high hydrogen contents of 18 and 11.5 wt%, but, unfortunately high



hydrogen-release temperatures as well.

More recently, together with a group at the University of Aarhus (Denmark), they prepared and characterised novel anion-substituted modifications of these materials.

The joint team also prepared novel materials by cation substitution, e.g. $\text{LiZn}_2(\text{BH}_4)_5$ by a reaction of LiBH_4 and ZnCl_2 . The idea was to introduce a less electropositive metal (Zn) in the structure of the borohydride. "We discovered a much unexpected structural chemistry of these new compounds, which store large amounts of hydrogen and release it at low temperatures of some 80–100°C," explains Yaroslav Filinchuk, of BM1. He continues: "We have already proven that the novel modified borohydrides show interesting structural, chemical and physical properties." Scientists aim for unstable materials, as they can release the hydrogen in mild conditions, whereas if they are too stable, they require a lot of heat to release it.

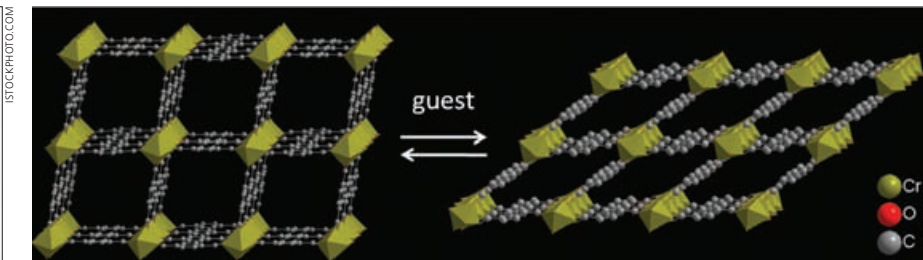
Hydrogen-fuelled buses are already a reality, for example in Japan and Germany, but general application of hydrogen as a new fuel in cars is still some time away, despite the fact that the automobile industry is starting to associate itself with academic research.

Metal-organic frameworks

Another promising way of storing hydrogen, as well as capturing gases such as CO_2 , is the so-called metal-organic frameworks (MOF). MOFs are extended crystalline networks made of metal/oxide groups held together by organic linkers, with large, open pores that make them ideal for storing gases. Their pore size and shape can be easily tuned by changing either the organic ligands or the metallic clusters. They can potentially have other applications, such as sensors in nanotechnology, and they are already used in catalysis and ion-exchange processes. The advantages of these materials in comparison with the hydrides are that they have a low density and the hydrogen storage is governed by a physisorption process and not a redox reaction. Scientists come to the ESRF to study crystalline structures of different MOFs using mostly X-ray powder diffraction on beamlines such as ID31 and BM1, although they have also used microdiffraction at ID13.

An active group in this domain, and regular user of the ESRF, is the team from the Institut Lavoisier in Versailles (France). As if it was a Meccano, they have managed

for a greener future



Left: hydrogen, a simple element that has given hope to scientists in the quest for a more environmentally friendly world. Above: The MIL-53 is a very flexible metal-organic framework. On the left, the dried form of the structure (large pore form); on the right, the structure (narrow pore form) after adsorption of various guests, such as carbon dioxide or water.

without breaking bonds and retaining the crystallinity of the materials. The reverse process was achieved by heating the solvated form, which ended in the material closing pores with almost no accessible porosity. The scientists came to the ESRF to study the structure before and after the “breathing” process, using X-ray powder diffraction.

Today the team is working on the use of MOFs for their separation properties (gases, liquids) as well as to develop biomedical applications using non-toxic biodegradable iron MOFs.

Industrial applications on hydrogen storage are already under way. Researchers from the company BASF showed recently that, compared with pressurising an empty container with hydrogen, if the MOFs are added they increasingly take up higher amounts of hydrogen with less pressure.

Sequestration of toxic gases

CO₂ and CH₄ are two types of gases that are currently damaging our planet, so their elimination would be another step towards a cleaner environment. CH₄ is not adsorbed by MOFs as well as CO₂, but, on the other hand, both of these gases are adsorbed at room temperature, unlike hydrogen.

A team led by the University of Aix-Marseille in collaboration with the team from the Institut Lavoisier, together with IFP, the University of Caen, the University of Montpellier and the ESRF (all in France), recently studied MIL-53 (Cr) for the separation of mixtures of CO₂ and CH₄ at ambient temperatures. MIL-53 (Cr) changes its pore size and shape in response to adsorption of molecules such as CO₂ and H₂O, going from a narrow-pore to a large-pore form. However, apolar molecules like CH₄ don't normally have any effect. The breathing behaviour of the MOF in the presence of gas mixtures is not yet clear to scientists, especially when they contain a component that provokes breathing and another one that doesn't, like CO₂ and CH₄.

It is necessary to separate the two gases as part of the capture, transportation and sequestration of CO₂. For this it is required to obtain a pure CO₂ (>95%) prior to its storage, either in former gas or oil reservoirs or other geological areas of interest. As CO₂ is produced by industry within a complex mixture of CO₂, CH₄, CO, H₂S, CH₄..., one has to capture CO₂ with a high selectivity versus the other components. MOFs, with their tunable pore size, large sorption capacities, good selectivity and easy regeneration, offer a nice alternative to zeolites or amines.

Experiments at the ESRF allowed the team to study the breathing of the solid upon adsorption. By combining diffraction with Raman spectroscopy and computer simulations, they evaluated the “breathing” pattern of the MILs. They found that the coadsorption of CO₂ and CH₄ leads to a similar breathing pattern of MIL-53 (Cr) as with pure CO₂.

For the future, scientists find potential in the flexibility of some MILs: “One could imagine benefiting from the flexibility by applying a mechanical pressure to make the MIL-53 solid close its pores and desorb gas mixtures, for an easier regeneration without the need for thermal or vacuum treatments,” explains Christian Serre of the Institut Lavoisier.
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Designing the lithium-ion batteries of the future

Synchrotron research is increasingly being used by scientists working on lithium-ion batteries.

In 1899 a Belgian electric car equipped with lead-acid batteries reached a speed of 108 km per hour. More than a century later, cars powered by batteries are just starting to become a reality but are still far from everyday use. Studies on batteries have not evolved as much as other fields of research. This is due to the lack of suitable electrode and electrolyte materials, in addition to the difficulties in mastering the interfaces between them (Armand *et al.* 2008).

Batteries have two electrodes connected by an electrolyte that conducts ions. When an external device is connected to the battery electrodes, electrons go from more negative to more positive potential and provide electrical energy to the external circuit. One of the most popular batteries today is the lithium-ion battery, which is widely used in electronics and is expected to be a candidate for hybrid-electric car applications. This battery was first commercialised in 1991 and it stores about five times more energy per unit mass than the old lead-acid batteries.

The drawbacks of this battery include the limited amount of lithium available worldwide if this was to be used, for example, in the automobile industry, as well as the risk of explosion due to the combination of combustible material and oxidising agent. The “rocking-chair” mechanism in lithium-ion batteries can involve large changes of the electrode’s volume. These generally deteriorate and age the electrical contact and scientists are investigating how the structure of the electrode changes over time.

Synchrotron X-rays offer scientists a way to obtain information about the battery electrode’s structure and beamlines such as BM1 or ID31 are often used in this field. A team from the National Institute of Advanced Industrial Science and Technology in Osaka (Japan), as well as the ESRF and SPring-8, studied *in situ* a single crystal system of a gold model anode for lithium-ion batteries (Renner *et al.* 2008). The researchers went to ID32, where they used X-ray diffraction.

They studied a gold-lithium alloy because gold has many electrons and this provides a very strong diffraction signal. Thanks to their experiments they could follow the gold system’s structural degradation and



Lithium batteries are widely used in mobile communication devices, such as PDAs or smartphones.

pulverisation under different electrochemical conditions. The team is now preparing new experiments at the ESRF: “We’ll still use gold, but we will progressively focus more on silver and silicon as possible candidates for the anodes. Silicon is indeed a very promising candidate as a single-element anode in lithium-ion batteries, as it can increase the storage capacity of battery anodes,” explains Frank Renner, leader of the team and head of the Interface Structures and High-Temperature Reactions Group at Max Planck Institute for Iron Research in Düsseldorf (Germany).

Phosphate-based compounds are also of great interest as electrodes for the community studying batteries due to their low cost. At the ESRF, several groups have studied the crystal structure of materials such as sodium pentamolybdate tetradiphosphate or different vanadium phosphates as cathodes for lithium batteries (Dubarri *et al.* 2008, Filinchuk *et al.*, 2008, Caignaert *et al.* 2007). Synchrotron radiation sources have also been used for the study of lithium iron phosphate.

Batteries for microelectronics applications

Thin-film solid-state batteries are the best candidates for use in microelectronic devices, such as medical implants or smart sensors. Today, negative electrodes of existing solid-state thin-film, lithium-ion batteries are usually made up of pure metallic lithium. This element has a low melting point and, consequently, it is not adapted for microelectronic integration. Scientists from the Eindhoven University of Technology (the Netherlands) are studying the possibility of using germanium electrodes in thin-film microbatteries. Although germanium has not

been studied thoroughly for batteries due to its high cost, this would not be an issue in thin-film batteries. In addition, it has higher electrical conductivity than silicon.

The team characterised the material’s structural changes using *in situ* electrochemical X-ray diffraction (Baggetto *et al.* 2009) and recently used the DUBBLE beamline at the ESRF to carry out *in situ* X-ray absorption spectroscopy. This technique proved to be an efficient tool to track the material reaction mechanism and allowed them to derive the evolution of the short-range ordering of germanium thin-film electrodes as a function of lithium content. Their results are currently being submitted for publication.

Research on batteries using synchrotron radiation is experiencing a period of rapid growth. “Batteries are becoming more important, especially in the framework of the new engines in the automobile industry. In Germany, at least, there is a real will by the government to fund this research intensively,” says Frank Renner.

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Egyptians made opaque glasses using synthesised nanocrystals

Research done at the ESRF has shown that craftsmen in ancient Egypt, contrary to belief, made opaque glass by adding synthesised calcium-antimonate crystals to a translucent glass. These compounds are nanocrystals and give the glass its non-transparent look.

The Egyptian civilisation is one of the most interesting of the ancient world, mainly thanks to the advanced skills of its craftsmen. However, little is known about the first real production of glass objects, which appeared during the 18th Egyptian dynasty (1570–1292 BC). For this reason, a team from the Centre de Recherche et de Restauration des Musées de France, the Institut de Minéralogie et de Physique des Milieux Condensés (IMPMC) and the ESRF, has studied ancient Egyptian opaque glasses from the Louvre Museum and the British Museum.

During the 18th dynasty, glass objects were opaque or translucent coloured glass and were dedicated to the upper classes, who used them as perfume or cosmetic containers. Opaque white, blue and turquoise glasses were opacified by calcium-antimonate crystals dispersed in a vitreous matrix. Among the different types of glass opacifiers, antimonate compounds have had a predominant role throughout history. They are found from the origins of glass technology in Mesopotamia until modern times. However, both their technology and provenance remain obscure.

To answer these questions, the researchers developed an original strategy focused on the investigation of the crystals and the vitreous matrices. For the first time they reproduced opacified glass by the addition of crystals in the laboratory under controlled conditions. They also compared these synthetic glasses to ancient Egyptian glasses using appropriate micro and nanoanalytical techniques never used before on this type of material: transmission electron microscopy at the IMPMC and micro X-ray absorption near-edge spectroscopy at ID21. The synchrotron-based measurements proved to be well suited to the selective measure of the antimony oxidation state in the vitreous matrix. Antimony, combined with the microstructure observations and the crystalline phases identification, is one of the key parameters used by the researchers as an indicator of the opacification process employed.

Until now, scientists thought that ancient Egyptian opaque glass was made from *in situ* crystallisation. This new study has refuted this assumption. Contrary to what was thought, the researchers demonstrated that Egyptian glassmakers were able to synthesise *ex situ* calcium-antimonate compounds, which do not exist in nature, and added them into the glass to opacify it. The results also show that



Figure 1 (top and bottom left and centre): opaque, coloured glass from the 18th Egyptian dynasty. a) Small amphorae (inventory number AF2622). b) Shards (inventory numbers AF12707 and AF13175). c) Blue and turquoise necklace (inventory number E2341). These objects come from the Egyptian Antiquities Department of the Louvre Museum. Figure 2 (bottom right): micro X-ray fluorescence (μ -XRF) analysis of a polished fragment of Egyptian glass (sample c). μ -XRF elemental maps of antimony (red), calcium (green) and silicon (blue). The map size is $72 \times 36 \mu\text{m}^2$, the pixel size is $0.5 \times 0.5 \mu\text{m}^2$ and the probe size is $1.1 \times 0.3 \mu\text{m}^2$.

these opacifiers are nanocrystals.

This outcome made the researchers want to investigate further and try to reproduce the conditions of the preparation of calcium-antimonate crystals. The compounds were fired between 700 and 1100 °C for 1–18 hours, depending on the case. Subsequently, they studied the nature, crystallographic structure and oxidation of antimony on the crystals obtained, and the results were very close to the analysis on the Egyptian samples.

“Until now, Egyptian blue and green pigments were the only high-temperature compounds known to have been synthesised in ancient Egypt. Our results show that

calcium-antimonate glass opacifiers were also synthetic compounds. These findings provide further evidence for the sophisticated chemistry and remarkable know-how of this civilisation. We believe that this work is the starting point for a complete reassessment not only of ancient Egyptian glass studies but more generally of high-temperature technologies used throughout antiquity,” says Sophia Lahlil, the main correspondent of the research.

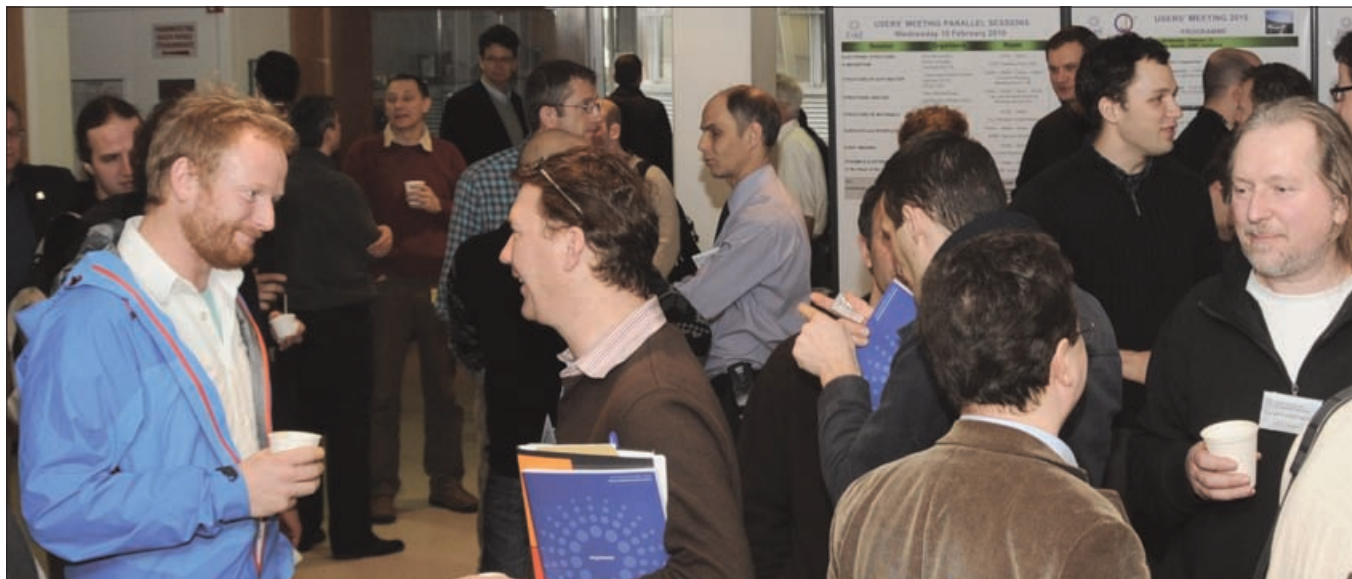
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Nano is the name of the game

The recent Users' Meeting, which took place from 8 to 12 February, hosted a workshop on nanobeams, which is one of the most important aspects of the Upgrade Programme.



Coffee break: scientists had time for informal chats with other researchers at the nanobeams workshop in the framework of the Users' Meeting.

Nanobeams are already here. In fact, the first nanobeam at the ESRF was achieved at ID13 in 1996, although it was only "nano" in one direction. However, nanobeams were not common back then. Today they are routinely used on several beamlines, such as ID01, ID11, ID13 and the nanoimaging station at ID22. The new Upgrade beamlines are also expected to use these tiny beams.

In the framework of the Users' Meeting, which took place on 9–11 February, the ESRF dedicated two and a half days to taking a snapshot of current research using nanobeams and attracting new communities of users in this domain. "This is still an unexplored territory and the work is done to a large extent in collaboration, so this workshop has been an opportunity to look for new collaborations and maybe to address the challenges that we are facing in this field," explains Manfred Burghammer, who is in charge of the ID13 beamline.

There is now a wide variety of experiments using nanobeams: from soft-matter research and biological applications to hard X-ray materials science. The implementation of nanobeam-compatible sample environments, which enable *in situ* techniques such as heating/cooling, humidity control and mechanical deformation, will be a big challenge for years to come. In addition, nanobeams often exhibit a high degree of coherence. This property can be exploited for coherent X-ray diffraction imaging (CXDI) experiments. The method has the potential to reveal structural details of the sample, which are much smaller than the size of the nanobeam. "There has been an increase in the

number of proposals that intend to perform such CXDI experiments using coherent nanobeams," explains Burghammer.

In the programme of the workshop, the organisers brought together specialists from different fields, including an expert in transmission electron microscopy, David Cooper, from CEA/LETI in Grenoble (France). Certain non-X-ray techniques such as this have capabilities in nanoscale characterisation and can be used to complement X-ray studies at the nanoscale.

Josef Keckes, professor at the University of Leoben (Austria) and one of the speakers at the workshop, uses the ID13 monochromatic nanobeam, 100–200 nm in diameter, for wide-angle X-ray scattering (WAXS) for the characterisation of graded nanocrystalline coatings. These are used as protective hard coatings on working tools or as biocompatible thin films deposited on implants. Keckes explains that: "until now, for such systems, there was no technique to characterise depth profiles of phases, residual stresses, microstrains or texture on a sub-

micron scale." For him, the nanobeams have opened up new avenues of research: "The synchrotron nanobeam matches the structure and mechanics in compositionally graded nanocrystalline thin films for the first time. In comparison with transmission electron microscopy, the synchrotron nanobeams may help to determine lattice parameter and strains with a resolution at sub-micron level. This is a new quality."

Nanobeams are also useful in medical applications. Boris Kysela, speaker at the workshop and senior lecturer in human molecular genetics at the University of Birmingham (UK), carries out experiments on the nanoimaging station of ID22. Together with his team, he tries to develop poly-functionalised nanovectors that can image and target tumour cells and deliver the therapeutic payloads to the cell nucleus of the targeted cell populations. To achieve the desired effects, very few nanoparticles are required and they have to be aimed at the right place. "At present, synchrotron X-ray fluorescence microscopy (SR-XFM) is the only technique available for quantitative elemental imaging of whole cells with very high sub-organellar spatial resolution in 2D and 3D," he explains.

There will certainly be more workshops dedicated to nanobeams in the future, as the use of nanobeams is rapidly growing in the scientific community. "In workshops, in five years' time, we will look back at the capabilities of today and we will be amazed about the rapid evolution of the field," says Burghammer.

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"The use of nanobeams is rapidly growing in the scientific community."

The Young Scientist Award recognises energy researcher Yaroslav Filinchuk

Yaroslav Filinchuk is the new recipient of the 2010 ESRF Young Scientist Award for “his outstanding work on the chemistry of solid-state hydrides”. This Ukrainian chemist has worked at the Swiss–Norwegian beamline (BM1) at the ESRF since 2006. He is currently a visiting professor at the University of Aarhus (Denmark). At 31, he already has more than 100 peer-reviewed papers, mostly focusing on energy themes, such as hydrogen storage. His work coincides with the theme of this issue of *ESRFnews*, which can be found on pp12–13. *ESRFnews* finds out more about this researcher.

What does this prize mean to you?

Mainly, it is having the research activity of my academic project publicly recognised. Reputation is difficult to build up – it’s not just about writing a large amount of papers for publication. I am very pleased that scientists from other areas recognise our work as an achievement in this rapidly developing field of research. It also gives me more confidence and helps me to move forward.

Did you always want to do research on energy or did you get into it by chance?

For a long time my aim has been to do high-quality research in a rapidly evolving field. This is an understandable aspiration – that way one can learn quickly and collaborate or compete with other researchers, i.e. enjoy the science in all of its different forms. I was very fortunate to take part in some research on hydrides at the University of Geneva in 2001. Later, I changed the direction of my research towards materials that were more suitable for studies at the ESRF and which turned out to be the most promising for hydrogen-storage applications.

How relevant do you think your research is today, or will it be more so in the future?

As I said above, the field is quickly emerging, and this often occurs in potential applications that have high expectations. Industry, mostly automotive companies, is carrying out active research in the same areas: their researchers attend the same conferences and collaborate with academics. For instance, we worked alongside scientists from General Motors and I visited them at the central research labs in Michigan. It is, however, up to us to find new applications and for industry to implement them. I hope that our research is helping to solve hydrogen-storage problems. The progress made in recent years leads me to believe that this is happening.

What do you like most about your job?

Research freedom, the highly qualified team of colleagues that I enjoy working with, and meeting many interesting people who come to the joint ESRF/ILL/EMBL site. In fact, we don’t even have to travel much as the top



Yaroslav Filinchuk putting a sample on the Swiss–Norwegian beamline (BM1) at the ESRF.

researchers come to Grenoble fairly regularly for their own needs, so we are all here in a stimulating environment.

What do you like the least?

The limits of what I like the most of my job. This translates into a lack of a dedicated research group, which is necessary for the freedom of the scientific activity at a certain stage of the academic career. The fact that the aim of the large facility where I work is to give service to users is inevitably pushing my research interests into the background. I am also missing university life, where people have more time to spend with their colleagues, where they meet not only busy researchers who visit for a quick experiment, but also the students.

Now that you are a visiting professor at the University of Aarhus, do you enjoy giving lectures?

As a teenager I actively participated in national and international contests in chemistry – known as “olympiads”. This experience helped me to learn how to explain my ideas clearly and in a simple way, but it also made me addicted to the “romantics of education”. I like to communicate with young people and giving lectures or less formal classes is one of the ways to do this. During my time at the University of Aarhus I try to fill the gap between university life and the environment of a large facility. Certainly, this stay allows me to spend more time carrying out research on hydrides and to enjoy the social life with the many interesting people here.

Do you prefer the environment of the university or the ESRF?

I definitely prefer combining the two. Large facilities play an important role in my scientific life: during my stay in Geneva I worked a lot with neutron diffraction using the Swiss Neutron Source at the Paul Scherrer Institut, where I have made a lot of good friends. I was also a user of the BM1 beamline, where I am working now. Some people are successfully combining the two environments while staying on either side, and it seems that this is what I am doing.

What have you gained from your experiences at the ESRF?

It has given me experience of teaching and helping different people from around the world, from undergraduate students to professors, to operate the diffraction instruments and to perform their experiments. Our beamline and the large facility as a whole provides an immense variety of options and I am happy to be among the people developing the lab and helping our visitors to use it. This is an important experience that will serve me well during my entire scientific career. It has also enabled me to start an independent research project, which I am succeeding in doing.

What are your plans for the future?

I am not making any definite plans as it is difficult to map out the future. Having collaboration with PhD students and postdoctoral researchers is a necessity in order to implement the scientific ideas that I currently have. This is a prerequisite for keeping the ongoing work in this dynamic and internationally competitive field and would allow us to move forward.

How do you see the world, in terms of the environment, in 20 years’ time?

I am very positive about how things have been developing (including environmental standards) during the last few years and over the centuries. Our homes, workplaces, the cities and industries have gradually become safer since the time of the industrial revolution. So I do not have any major concerns about the environment – the ongoing efforts are able to provide our sustainable development for well over 20 years. As for environmental issues, as in many other areas, making responsible decisions and consistently implementing them should suffice for future growth and development. In years to come I see us using less oil and gas as fossil fuel, and with bigger diversity in energy sources, which will make our environment cleaner, economies less vulnerable and, consequently, the quality of life higher. And I hope that cultural diversity will keep pace with the technological one.

The ESRF extension moves a step closer to construction

The signature for the contract of the ESRF extension marks the kick-off of the physical changes to the layout of the facility. The first phase is due to take until 2015 to complete.

Extending a place like the ESRF is not a small task. In the framework of the ESRF Upgrade Programme, many technical considerations have to be taken into account, especially if the new upgraded beamlines have to attain beamsizes of nanometres routinely. After several months of fine-tuning the project for the ESRF extension, the signature of the contract between the ESRF and the engineering company GINGER Séchaud Bossuyt took place on 27 November 2009.

The signature indicates the beginning of the study phase and the subsequent construction works to implement the ambitious Upgrade Programme. The extension of the experimental hall by 18 000 m² will allow the housing of up to 13 modernised existing beamlines, including seven new Upgrade beamlines (out of a total of eight Upgrade beamline projects).

Precision will be the name of the game in the new €30 m extension of the ESRF. An important aim of the Upgrade is to routinely deliver extremely small X-ray beams for new science in the nanoscale range. In this context, the new experimental halls will host long beamlines of up to 120 m, instead of the 60–80 m-long beamlines that feature currently at the ESRF. Even extra-long beamlines of up to 250 m, required for X-ray imaging and analysis, will be accommodated in new satellite premises.

In order to keep the X-ray beam stable with an extremely small size, potential vibrations coming from the neighbouring motorway, the river flow or traffic inside the buildings should not be amplified. The conception of the new infrastructure has taken all of these aspects into account, and it is foreseen that 7500 m² of slab (thick concrete floor) with an



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C. ARGOUË

Top: a view of the upgraded ESRF. The golden sectors represent new buildings. Bottom: Bernard Thiers (left), representative of the engineering company Séchaud Bossuyt, and Francesco Sette (right), director general of the ESRF, at the moment of the signature of the ESRF extension contract. In the background are the staff that were involved in the preparation of the contract.

“The extension will provide an additional 4000 m² of laboratory and office space.”

extremely low vibration level will be inserted in the experimental hall. The extension will also provide the ESRF with an additional 4000 m² of laboratory and office space.

The new buildings are not only designed for cutting-edge research, but the architects of the project, Sud Architectes (part of the team mandated by GINGER Séchaud Bossuyt), have also given their design an ecological slant. For example, the building roofs will be covered

with grass to increase the thermal inertia of the hall and to reduce the rain water flow that is taken up by the ground networks.

The construction of the new facilities takes place in the framework of the first phase of the Upgrade Programme, which runs from 2009 to 2015, covering a capital investment of €180 m. The civil construction works are scheduled for commissioning in 2012.

M Capellas

P Bruno: the theorist who cooks ideas

Patrick Bruno, head of the ESRF's theory group, joined the facility two years ago and declares that he is very happy with his new life. He has recently received two important prizes.

"The more results that you have, the more you realise that there is still a lot that you don't know," says Patrick Bruno, head of the theory group at the ESRF. Bruno pronounces these modest words a few weeks after becoming the recipient of the 2009 Prix Jean Ricard (of the French Physical Society) and the Ernst Mach Medal (of the Academy of Sciences of the Czech Republic).

In his office, scientific publications and papers with graphs and equations pile up on the table chaotically, while the whiteboard is full up with calculations. It feels as though there are ideas constantly being "cooked" in these few square metres. He is the chef, his office is his kitchen, and his career and accolades are the meal.

Bruno considers himself a vocational theoretician, despite having started his career as an experimentalist. Today, he says that he sometimes cannot stop thinking about work even in bed at night: "Frequently I have ideas while there are moments, conversely, when I have the 'blank page' phenomenon and I can read papers and study but the inspiration is low."

At work, he spends a lot of time studying (a paper in depth per day) either in his office or in the library and discusses it with his colleagues. He claims that the latter is his favourite part of the job. He also appreciates "the great freedom" that he has in his research activity: "For me, this is of the highest importance. I enjoy being trusted in my work."

His fondness to learn new things has allowed Bruno to make detours into different fields of research on several occasions. "Sometimes I learn about other subjects and I find that I can bring something new to these studies, to contribute into painting the overall picture of nature in a richer and more adequate way." When challenged to describe his job as he would to a child, he answers: "I try to understand why magnets are magnets and why they behave



Patrick Bruno gives a talk during a symposium in his honour in December at the ESRF. During this event he received the Jean Ricard prize from the president of the Société Française de Physique, Michèle Leduc.

Patrick Bruno in brief

- Born in 1964
- 1989: PhD thesis *Anisotropie magnétique et hystérésis du cobalt à l'échelle du plan atomique: théorie et expérience*, Université Paris-Sud
- 1989–1991: Postdoc, University of Regensburg, Germany
- 1991–1998: CNRS staff scientist at the Institut d'Électronique Fondamentale, Université Paris-Sud
- 1998–2007: director at the Max Planck Institute of Microstructure Physics in Halle, Germany
- 2007: receives the Leibniz Prize of the German Research Foundation
- 2007–present: head of the theory group at the ESRF
- 2008: elected member of the German Academy of Sciences, Leopoldina
- 2009: awarded the Ernst Mach Medal of the Academy of Sciences of the Czech Republic
- 2009: receives the Prix Jean Ricard of the French Physical Society

the way they do. That is, anyway, what I've told my kids, who are nine, seven and five, and they seem to have grasped the idea."

Bruno's latest research interests have been magnetic semiconductors, the Berry phase-related phenomena in

nanostructures, the diabolical points in molecular magnets, as well as spin-dependent transport phenomena.

Bruno's wife and children have graciously taken on the challenge of moving to different countries and settling down in new places

throughout his career. He has lived most of his life in France and Germany, but he has also spent some time in the US, Switzerland and Japan. Despite all of this travelling he still considers himself "deeply French", he appreciates being close to his relatives, but he acknowledges that he now looks at his country with a more analytical view.

He is no stranger to Grenoble, as he used to spend holidays in the Alps with his parents as a child. For now, for family reasons, he is taking a break from his trips – his most recent (although seldom) have mainly been within Europe.

Since he arrived at the ESRF, Bruno has set up a small team of four people (plus temporary visitors) that work very closely with the theory group of the Institut Laue Langevin. This new position is very different from his previous job as managing director of the Max Planck Institute of Microstructure Physics in Halle, Germany. During his nine years at the Max Planck Institute his role demanded more administrative duties than his current job.

"When you manage big groups you are like an orchestra conductor, while now it is like being a soloist." He adds that the process of producing ideas is something that is not easy to transfer to co-workers, so at the ESRF all members of his group have their own projects: "I find it intellectually more gratifying to be able to do the job myself, rather than having to explain the process to my colleagues." According to Bruno, the theory group as a whole has a duty to "make things easy" for beamline scientists by giving them the theoretical assistance.

Bruno has been at the ESRF for two years and he avows that he has "no regrets". He finds the diversity of the research at the facility, as well as the international atmosphere, "culturally enriching and very stimulating". Most of all, though, he says: "I am very grateful to the ESRF for letting me do my hobby as a job."
M Capellas

The BLOMs guard the ESRF beamlines

The beamline operation managers are key in the preparation of experiments at the ESRF.

The beamline operation managers, or BLOMs, are a group of scientists linked to different beamlines who are in charge of assisting in the preparation of experiments, providing user support as local contacts and carrying out technical development. They are also in charge of the maintenance of the beamline and are the interface between the scientists and the engineers (mechanics, electronics and software). In brief, they make sure that the beamline runs smoothly.

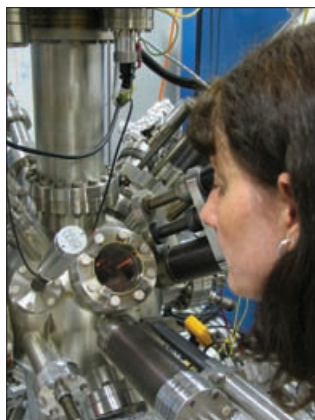
Some BLOMs are attached to one beamline, others have two. This is the case for Helena Isern, BLOM at ID3 and ID32: "My job requires being ready to help the scientists to be successful in their experiments and to solve problems continuously. I help the local contacts all of the time to prepare and find solutions for the experiments on both

beamlines. Sometimes I feel like a firefighter."

Despite not being trained for putting out real fires, most of them have enough experience under their wings to be able to extinguish figurative fires that may occur during the operation of the beamline. The majority have a physics PhD and were scientists at the ESRF before becoming BLOMs.

For BLOMs, science is still important, and most of them that were interviewed by *ESRFnews* acknowledge that they still carry out research in collaboration with other groups. Their own research projects are, on the other hand, put to one side. "If you do your job as a BLOM properly there is not much time left...", explains Thomas Buslaps, BLOM at ID15.

Building up a relationship with the user groups seems to be one of the most positive aspects of the work of a BLOM. They



Helena Isern, BLOM on ID03 and ID32, working on the beamline.

reckon that knowing the users is important to understand the needs of the beamline and that contributing to the success of an experiment is a great reward. Trying out new things is also gratifying: "It is a lot of fun when we try new experiments or we

commission new devices," says Federico Zontone, BLOM at ID10.

With the Upgrade Programme coming up, many of the BLOMs will be even busier than before. "The preparation of the upgrade will bring extra workload, because we will have to operate the existing beamline while constructing the new ones," explains Buslaps. A challenging task, but also satisfying, as Flora Yakhou-Harris, BLOM at ID8, explains: "We will add the design, building and commissioning of a new beamline to the operation of the present one! Twice the job in the same day. I am not sure how we are going to achieve that... The main thing, though, is that until now, I used beamlines (ID20, ID08) designed by other people. I will fully participate in the design of the new one, UPBL7, and this is very exciting."

M Capellas

Movers and shakers

Scientist in charge of ID01 Tobias Schulli



The anomalous scattering beamline ID01 is now led by Tobias Schulli, after

Till Metzger's departure on a sabbatical to the synchrotron source BESSY in Berlin (Germany).

Schulli is no stranger to ID01. He did his PhD *Anomalous X-ray Diffraction from Semiconductor Nanostructures* on the beamline back in 2003. After a postdoctoral position at the Commissariat à l'Énergie Atomique (CEA) in Grenoble, where he initiated the *in situ* growth of silicon-germanium nanostructures, he joined the Xenocs company to work on the development of X-ray optics.

In 2006 Schulli was appointed as beamline scientist on BM32 at the CEA, where he worked on the growth of metallic nanoparticles on silicon surfaces. During this

time he took part in defining and developing the scientific case and the refurbishment of the *in situ* nanoparticle growth station on BM32 in order to make it compatible with chemical vapour deposition and the growth of semiconductor nanowires.

His main objectives for the future of ID01 are to combine new X-ray tools emerging from the improvement of focusing methods with traditional X-ray techniques, as well as with versatile sample environments. His numerous local and international collaborations, and the knowledge of the local research and technology landscape, will help him to achieve his aims.

Leibniz Prize laureate Peter Fratzl



The director of the Max Planck Institute of Colloids and Interfaces in Potsdam

(Germany), Peter Fratzl, is one of 10 recipients of the Leibniz Prize 2010. This is the highest German research prize and consists of a research grant of €2.5 m that has to be used within seven years.

Fratzl's first paper as a user of the ESRF (beamlines ID13 and BM26) dates back to 1998. His work is focused on natural materials, such as bone or plant tissues and their mechanical properties. He analyses the relationship between the properties and structures of biological materials and develops new bioinspired materials. His work is mostly basic research but also yields findings that are significant to the treatment of diseased bone tissue and especially of osteoporosis.

He has had more than 300 articles published in various scientific journals.

First ESRF staff elected as fellow of the American Physical Society Jorg Zegenhagen

The scientist in charge of



ID32, Jorg Zegenhagen, has been appointed as a fellow of the American Physical

Society (APS). Zegenhagen was elected a fellow for "his innovative contributions to the study of surfaces and interfaces with synchrotron radiation and his support of international science".

The APS Fellowship Program was created to recognise APS members who have made advances in physics through original research and publication, or made significant innovative contributions in the application of physics to science and technology. They may also have made significant contributions to the teaching of physics or service and participation in the activities of the society.

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In this respect, the SLS Detector Group is seeking a

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Your tasks

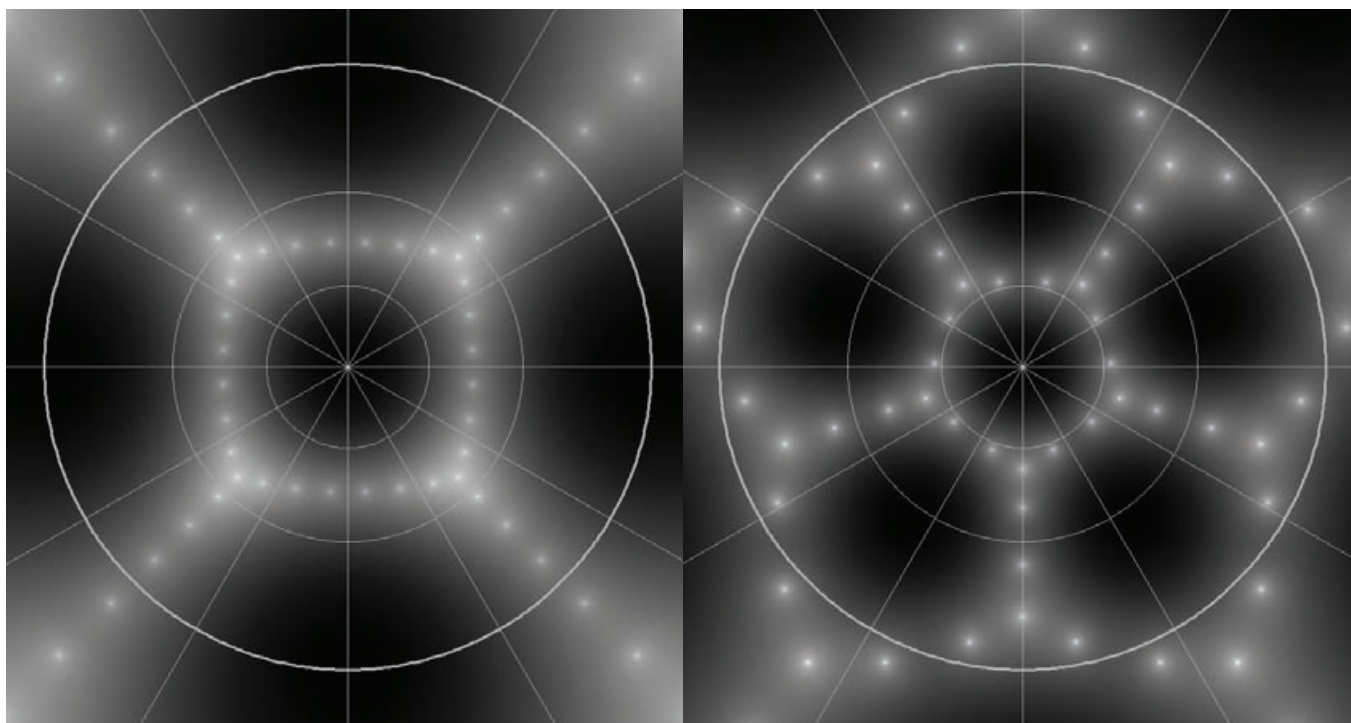
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Majorana stars representation of the ground state of spin systems (with $S=40$) of cubic and icosahedral anisotropies, respectively. The “stars” correspond to nodal points of the wavefunction and fully characterise the quantum state of the system. The quantum dynamic manifests as a “dance” of the “stars”. Images produced by Patrick Bruno, ESRF Theory Group (unpublished).

In the corridors

NASA sells bargain space shuttles



If you have a spare few million euros you can buy a space shuttle.

NASA is holding a sale. The agency wants to get rid of several space shuttles and they are due to go on sale this autumn, when they will stop flying. The original asking price of €29m has now been reduced to €20 m. NASA is hoping to sell the shuttles to museums or schools.

However, the main engine of these vehicles will not be sold – they are free. NASA had hoped to charge up to €58 000 but due to the lack of interest it is now giving them away. The only requirement is that the buyers pay for their transportation.

Celebrities talk science, but not always correctly

The British charity Sense about Science publishes a list of incorrect scientific claims that are made by celebrities every year and the charity asks scientists for the right answers.

A couple of examples below show that famous people could do with a bit of help to get the science of their statements right, according to Sense about Science.

● The actor Roger Moore stated: “There are even surveys suggesting that eating foie gras can lead to Alzheimer’s, diabetes and rheumatoid arthritis. In short, eating foie gras is a tasty way of getting terminally ill.”

Fact: there are many well characterised genetic and environmental factors that can directly increase a person’s risk of developing Alzheimer’s or diabetes, such as high blood pressure, high cholesterol, old age and having changes in the gene apolipoprotein E. There is no scientific evidence that eating foie gras will directly cause Alzheimer’s disease, diabetes or

rheumatoid arthritis.

● Atomic Kitten singer Natasha Hamilton said: “Like many women, I was unaware [of] the dangerous chemicals [that] antiperspirants contain, which have been linked to breast cancer.”

Fact: these claims are not substantiated by the evidence. Research has also shown that it is unlikely that these products would even enter the body, as they are too large to cross the skin and reach the bloodstream. The skin is generally impermeable to charged chemicals, such as aluminium, which is found naturally in our food and drinking water.

According to Ellen Raphael, director of the charity, “by correcting these mistakes, the scientists are not just helping celebrities but also giving the public the means to explain why these claims are wrong”.

Research into wooden bones looks promising

Turning wood into bone that is almost identical to the human tissue may sound like science-



This could be your next limb.

fiction, but it’s not. Scientists from the Istec laboratory of bioceramics in Faenza (Italy) have implanted treated wood into sheep’s legs. The type of woods used are rattan, red oak and sipo.

So far, the results are promising. Because of the sponginess of these kinds of wood, they enables blood, nerves and other compounds to travel through them. This means that it will eventually fuse with the real bone. In fact, some particles from the sheep’s own bones are migrating to the bone made from wood.

Time magazine included this research in its “50 best inventions of 2009” list – it made it to number 30.

Reference

A Tampieri *et al.* 2009 *J. Mater. Chem.* **19** 4973.

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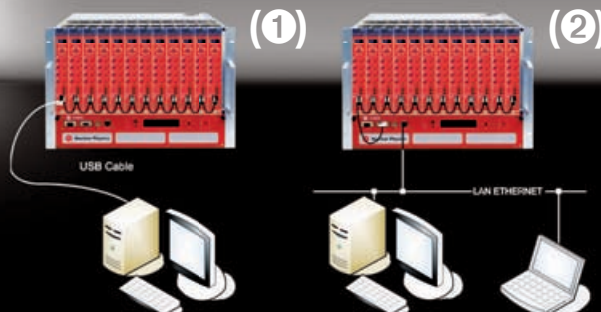
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