A Light for Science

ESRF news

Number 54 June 2010

Nature and energy

Unravelling the way human metabolism harvests energy

Closed European airspace tests the ESRF's adaptability

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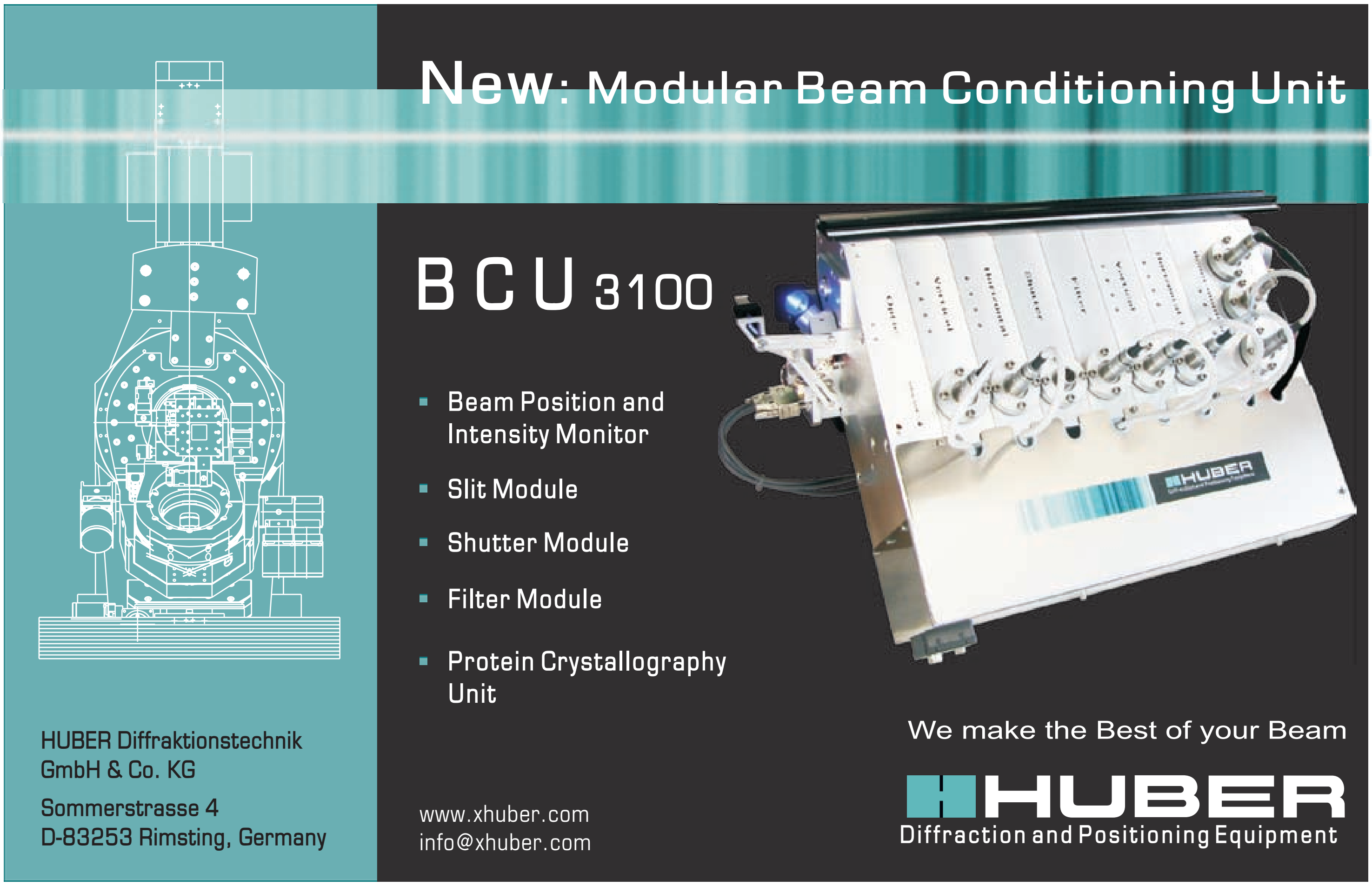
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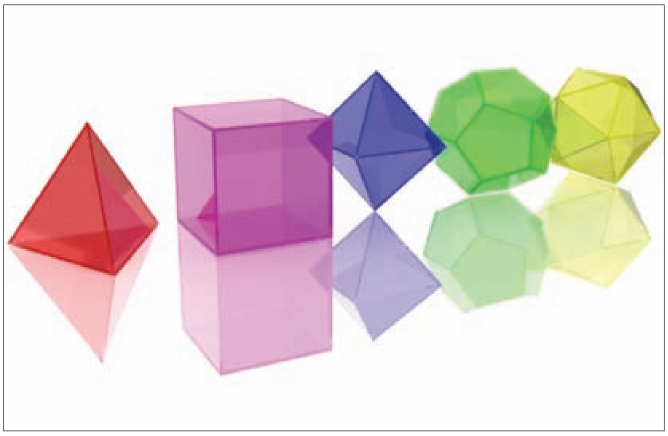
A light for science

Fischer-Tropsch fuels are becoming increasingly popular, p12



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Hassan Belrhali (left) and Amit Sharma in an experiment on BM14, p20

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On the cover: Nature is the greatest source of energy. In this picture, a sunflower thrives in the sun.

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ESRFnews

Learning from energy in nature

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Less than 300 years of modern industry and consumerism will have exhausted fossil resources that accumulated over 150–200 million years. Up to the start of the 20th century, renewable agricultural carbohydrates constituted the raw materials for fuel, chemical and material production. They were gradually replaced by petroleum-based derivatives. The depletion of petroleum resources, along with concerns about the warming of the planet by human activities, make it urgent to shift dependence from fossil resources to renewable biomass.

Sustainable alternatives should be developed commercially in the near future, both in terms of energy production and commodity products. As for future energy generation, only a multifaceted approach is likely to provide a viable solution; it will include nuclear and solar energy, hydroelectricity, hydrogen, wind and biofuel. Currently, biofuel may be expected to cover a small percentage of the total power that could be generated. The dual goal of producing biopower and biomaterials, while enhancing the management of greenhouse emissions, should be pursued. The concept of biorefinery has been proposed, which parallels the petroleum refinery in the imbalance between transportation fuels and chemical needs. The biorefinery operates on abundant renewable raw materials, which are fractionated and converted into commodity chemical molecules and transportation fuels.

The many scientific and technological questions to be addressed are related to an understanding of the photosynthetic process, the mechanism involved in the production of energy from this complex bioresource, as well as in the ways of mastering their conversion with limited pollution for large-scale deployment of biomass to biofuel technologies.

Photosynthetic organisms, such as plants, algae and some bacteria, produce more than 180 billion tonnes of organic matter each year from the fixation of carbon dioxide. Understanding the macromolecular architecture of the photosystems that are involved in the conversion of light to chemical energy during photosynthesis is of paramount importance. The elucidation of one of the most complete plant photosystem 3D structures obtained so far has been made using the structural biology beamlines at the ESRF and Swiss Light Source. Complementary to this finding, the application of a time-resolved Laue diffraction method to the study of a bacterial photosynthetic centre at the ESRF has revealed the structural changes occurring within the membrane protein complex a few milliseconds after irradiation (p10).

The breakdown of biomass involves the release of long-chain polysaccharides into small components, which can be further processed for energy production. A variety of organisms have evolved to take advantage of this nutrient source and increasing our knowledge of the biochemical machinery involved in such events offers new avenues to understand these processes. When it comes to humans, the production of energy through the breakdown of glucose, leading to the production of the high-energy compound adenosine triphosphate, is the result of a complex metabolic pathway called glycolysis. One step towards a complete understanding of such a mechanism has been made through the elucidation of one of the key steps catalysed by the phosphoglycerate kinase. The combined use of high-resolution NMR spectroscopy, macromolecular crystallography and the bio-SAXS beamlines at the ESRF unravelled such a complex mechanism (p9).

The conversion of biomass to clean-burning fuel or hydrocarbons can be performed through the Fisher-Tropsch process. This process is a key component of gas-to-liquid technology; it produces a petroleum substitute from coal, natural gas or biomass, for use as synthetic lubrication oil and synthetic fuel. A variety of catalysts can be used for the Fischer-Tropsch process, but the most common are cobalt, iron and ruthenium. Catalysts have been investigated using synchrotron methods allowing *in situ* and *in operando* characterisation of their structure and their state of oxidation, in environments reproducing the industrial conditions (p12). Such a unique scientific environment is key to attract industrial users and set the scene for developing a promising catalyst for biomass conversion.

These examples show the essential role that techniques at synchrotron radiation facilities are playing in the development of alternatives to fossil fuels. The challenge is to weave this research into integrated strategies to address key needs for sustainable energy development.

Serge Pérez, director of research

C ARGOUD

A new model of the ESRF is the first stop in the more complete group visits.

New exhibits to boost group visits

Nothing can replace a visit to learn more about an institute like the ESRF. Since spring, visitors have been taken around a new visitor's circuit that combines interactive scale models, instructive videos and glass display cabinets. The objective is to increase the number of visitors, currently 2000 people per year, by 50% while maintaining a high standard for each visit, notably for groups of students sent by the users, who make up a big part of the visits.

The general layout and functioning of the machine and beamlines are explained using two brand new, interactive scale models on the mezzanine of the ESRF central building.

On the freeway of the experimental hall, three designated areas are used to present specific aspects of ESRF beamlines: optics, sample environment, detectors and data acquisition. In each area, two glass cabinets contain objects of interest like X-ray monochromators and detectors, and a video screen shows how some of these devices are produced or perfected in the ESRF labs and how they are used on the beamlines.

Some 20 scientists working at the ESRF are now being specially trained to immerse the visitors during a two-hour tour in science at the ESRF.

The new visitor circuit is a precursor of the future visitors' centre of the ESRF and ILL, due to open in 2013.

If you wish to visit the ESRF with a group of students – or any other group – please contact our organiser at visits@esrf.fr.

Tracking down arsenic in contaminated waters

A major cause of water contamination worldwide is the so-called Acid Mine Drainage (AMD), an outflow of acidic water originating from metal or coal mines. This phenomenon is generated by sulfide oxidative dissolution. Arsenic is a common constituent of mine waters, but its concentrations in AMD polluted areas are naturally attenuated by newly formed iron precipitates (schwertmannite, which transforms into goethite and jarosite). The quantification of the arsenic linked to these iron phases in natural sediments, as well as their temporal and spatial distribution, is of major importance to understand the arsenic evolution in aquatic systems.

Although a large number of techniques have been previously used to quantify the percentage of those iron minerals and the arsenic associated to the different mineral phases (sequential extractions, X-ray diffraction, classical analytical approaches), most of them present limitations.

A team from the University of Zaragoza, Consejo Superior de Investigaciones Científicas, University of Girona (Spain) and University of Manchester (UK)

M P ASTA

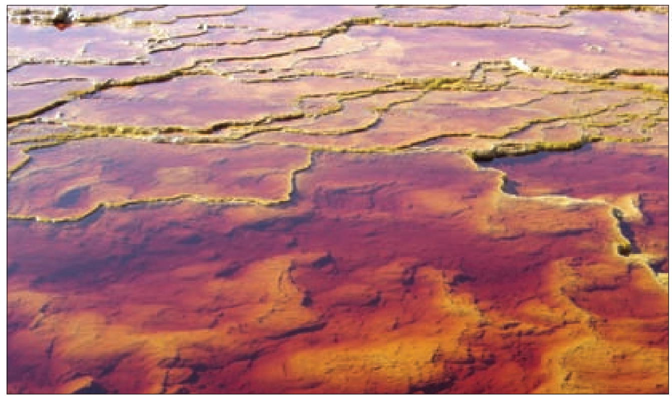


Image of the stream Aguas Agrias, in the basin of the river Odiel in Huelva (Spain), affected by the leachates of the mine Tharsis.

decided to use X-ray absorption spectroscopy (XAS) on BM8 and on two beamlines at Daresbury Laboratory (UK). XAS allowed scientists to study the amount of arsenic associated to the different precipitates individually, which is not possible with other techniques.

They identified and quantified schwertmannite, jarosite and goethite in natural heterogeneous samples and the percentage of arsenic associated to them. The results show that the highest arsenic concentrations trapped in solids are associated with sediments made up mainly of schwertmannite, corresponding to the upper stream solids. On the other hand, when the precipitates transform into goethite and/or jarosite, the arsenic concentration in the sediment is lower.

The formation and spatial distribution of the precipitates play a key role in the trapping and immobilisation of arsenic in acid mine drainage.

These studies are also very useful for the improvement of water-treatment systems.

Reference

M P Asta *et al.* 2010 *Chemical Geology* **271(1–2)** 1–12.

Three structural states of transporter protein revealed

A team from Imperial College London and the universities of Leeds and Oxford (UK) has captured the 3D atomic models of a single transporter protein in each of its three main structural states.

Data were collected on the macromolecular crystallography beamline ID29 at the ESRF, and further experiments were carried out at Diamond Light Source (UK).

The paper that the team published last April in the journal *Science* reports the mapping of the inward-facing structure of the bacterial Mhp1 transporter protein, the third structural state that they have determined for this protein.

Biologists have surmised that transporter proteins of this type, which sit in the cell membrane, carry molecules through the otherwise impermeable membrane by shifting between at least three distinct structural states, controlled by ion gradients.

The discovery offers remarkable insight into the movement of essential chemicals into cells of the body and creates an opportunity to develop brand new drugs.

Reference

T Shimamura *et al.* 2010 *Science* **328(5977)** 470–473.

ESRF-ILL-EMBL site is now EPN campus

The Institut Laue Langevin (ILL), the Grenoble outstation of the European Molecular Biology Laboratory (EMBL) and the ESRF, already physically close to each other, are now even closer. The site where they are all based is now called the European Photon and Neutron (EPN) science campus. A recently created website is the first tangible result of this new union.

The website (www.epn-campus.eu) targets users of the ESRF and ILL. It contains practical information such as the canteen and guesthouse opening times, details on how to get to the site and proposals for any spare time during an experiment. It also has online access to the site library.

Hot off the press: energy-converting enzyme solved

Scientists from the Medical Research Council (UK) have unveiled the structure of Complex I, one of the energy-converting enzyme complexes in the membranes of mitochondria and bacteria. The results show an unusual mechanism for controlling proton movement across the membranes that convert the energy from respiration into a form the body can use.

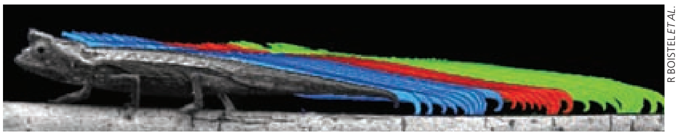
The team used ID23-2, ID29 and SLS. This complex, 18 nm at its largest, is also membrane bound, which makes data collection from crystals problematic. Scientists performed helical data collection, which allows the translation of a crystal between spots as it turns to minimise radiation damage.

Reference
R G Efremov *et al.* 2010 *Nature* **465** 441–445.

Chameleon receives 'fifth foot'

The Malagasy dwarf chameleon (genus *Brookesia*) is probably one of the most stable chameleons around. Scientists from the University of Poitiers, the National Museum of Natural History in Paris and the ESRF have unveiled the reason for this by studying the reptile's morphology using 3D X-ray phase contrast imaging on ID19. The team investigated the internal structure of the tail and inner ear of this species in order to identify precisely how they contribute to stable locomotion on a wide array of substrates.

Malagasy dwarf chameleons have adopted a more terrestrial way of life than most chameleons and, consequently, encounter broader surfaces when on the move. Unlike other chameleons, which use their long tails during arboreal acrobatic manoeuvres, this species' short tail is placed down on the substrate when walking on broad surfaces. This is proposed to reduce the instability created by grasping with hands ill-adapted for walking on broad surfaces.


R BOISTEL *ET AL.*

A series of video images illustrating how the Malagasy dwarf chameleon's tail curls and contacts the substrate during locomotion.

3D reconstructions of the vertebrae inside the tail have revealed morphological specialisations that explain this ability. The tip of this chameleon's tail is relatively mobile, owing to fewer and finer vertebrae as well as stronger ventral tendons. The tail is therefore able to bend and provide the chameleon with a fifth point of contact with the ground.

The inner ear morphology of the *Brookesia* chameleon also plays an important role in the balance of these lizards. Scientists compared it with several other species and discovered a highly specialised structure in *Brookesia*, in which the ear could be adapted to detecting weak accelerations and consequently help to correct posture more effectively.

The high-resolution images obtained at the ESRF were crucial in order to visualise the tail tendons, something that conventional X-ray sources do not allow. The detailed reconstruction of the inner ears enabled precise comparison of the subtle structures within each species' ear.

Reference
R Boistel *et al.* 2010 *Biol. Lett.* **doi: 10.1098/rsbl.2010.0322**.

Users' corner

At the last proposal submission deadline on 1 March, 1043 new proposals were received. These were reviewed in somewhat unprecedented fashion by the beamtime allocation panels (BTAPs) on 22 and 23 April via telephone conference as the ash cloud from the Icelandic volcano eruption caused major disruption to air traffic over Europe. More can be read about the consequences of the ash cloud in the article on p16. The next deadline for submission of standard proposals is 1 September, for beamtime from March to July 2011.

News from the beamlines

- **Two-tower Atomic Force Microscope (AFM) at ID01.** A new *in situ* AFM with two towers of piezo stages was commissioned at ID01 in April and is now available to all users at the beamline. This AFM is compatible with the Fresnel zone plate as focusing optics so that a beam size of 250 x 350 nm^2 can be reached on the sample in the AFM. Two piezo towers allow alignment of the AFM tip and the sample independently with respect to the focused X-ray beam. In addition, the sample stage contains a piezo rotation stage facilitating the measurement of asymmetric Bragg reflections. Moreover, all piezo stages are implemented in the SPEC software making alignment procedures, as well as mappings, easier.
- **DUBBLE (BM26)** has received funding for a 1M Pilatus detector for SAXS measurements.
- **The macromolecular crystallography beamline ID14-2** will be closed from 3 May to commission the pilot robotics for the MASSIF upgrade beamlines. It will reopen later to users to further develop the MASSIF sample flow and data-collection model. Reconstruction work on the MASSIF upgrade beamlines ID30 and BM29 begins this summer. Commissioning of optics for BM29 is expected to start in about a year's time while hutch construction for ID30 should be completed for radiation tests in March 2011.
- **The feasibility of performing new experiments on beamline BM16** has been successfully demonstrated during 2010. In particular, a permanent He recovery line was installed and the first cryogenic temperature measurements, using the ILL mini orange cryostat down to 32 K, were carried out for structural transition studies of acetone. Other sample environment equipment tested was the high-pressure diamond anvil cell. The pressure was increased only up until no further diffraction changes were observed in the metal-organic carboxylate complexes used, corresponding to 16 GPa. However, it is expected that the experimental set-up will allow pressures of at least 25 GPa to be reached.
- **In 2009 at SpLine (BM25)**, several major upgrades were implemented. At the SpLine Branch A, a state-of-the-art solid-state energy dispersive X-ray 13-element Si(Li) fluorescence detector was incorporated into the XAS station, enabling the measurement of chemical species in extremely low concentrations. Different sample environments such as LHe cryostats (10–300 K), a liquid sample container, a liquid-solid sample holder, high-pressure (3 bars) and low/high temperature (70–1200 K) "baby" chambers, all especially adapted to the XAS and SXRD stations, were designed, developed and installed at the beamline. In Branch B, a novel 2D detector is now on the HAXPES station. This detector will reduce acquisition time typically by a factor of up to 300. Use of a post monochromator will therefore be possible, improving the hard X-ray energy resolution and photoemission resolution.
- **A further important milestone achieved in 2009 on BM25** is the experimental station dedicated to the simultaneous combination of surface/interface X-ray diffraction and hard X-ray photoelectron spectroscopy (up to an electron kinetic energy of 15 keV). This novel and exceptional station has been opened to the scientific community and gives a unique opportunity to obtain crystallographic, chemical and electronic properties of bulk, surfaces and buried interfaces on the same sample region and under identical experimental conditions.

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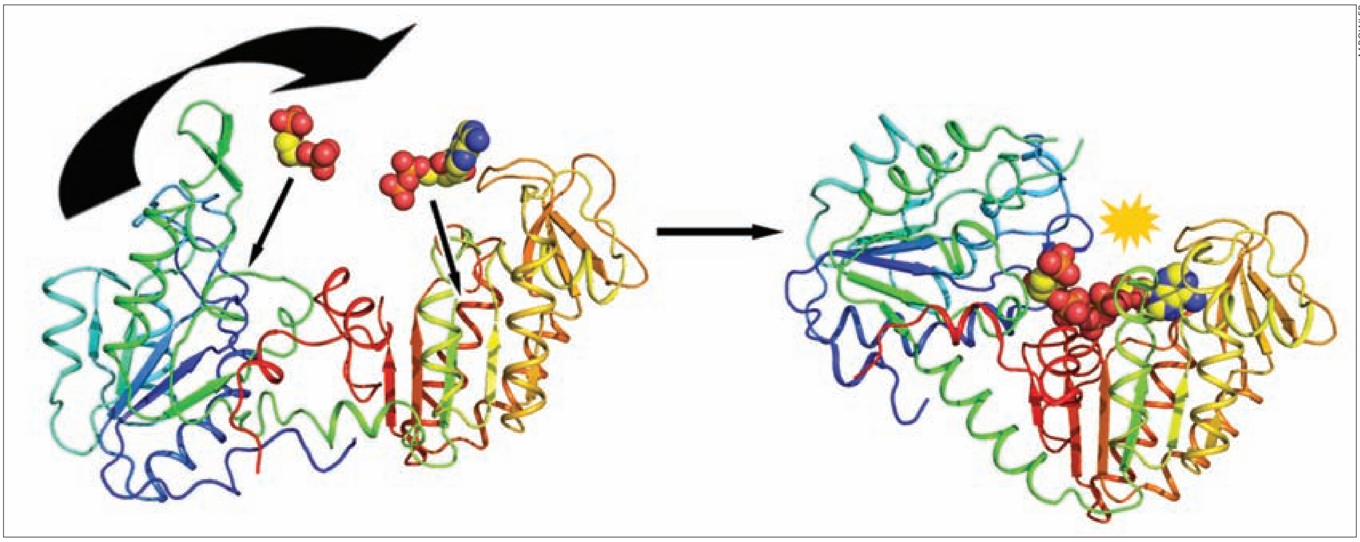

The first energy-generating step in glycolysis. PGK (shown as a cartoon coloured as a rainbow) lies open ready to receive the breakdown product of glucose 1,3BPG and ADP (left). Once bound, the enzyme swings together (a 40° rotation) closing around the molecules and forcing them together (right). When together, the phosphate "energy unit" is transferred to ADP to make ATP – the universal energy currency in all forms of life.

Unravelling the way human metabolism harvests energy

Harvesting energy is one of the most primitive and fundamental requirements of life. Glycolysis is the pathway that breaks down sugar into energy in almost all forms of life. It consists of 10 different reactions and phosphoglycerate kinase (PGK) is the first enzyme that produces energy in this process. The ESRF has given scientists new insights into PGK.

Glycolysis is the metabolic pathway that breaks down glucose into two molecules of pyruvate with the concomitant production of a high-energy compound, called adenosine triphosphate (ATP). The process requires no oxygen and is probably one of the most ancient pathways because it evolved before the accumulation of oxygen in the Earth's atmosphere. Glucose is an important fuel for all organisms and in mammals is the only source of energy used by the brain under normal conditions. It is also the only fuel that red blood cells can use.

The pathway consists of 10 separate reactions that comprise three main stages. The first stage traps glucose in a form that remains in cells and can be broken apart. The second stage is the cleavage of the six carbon ring into three carbon units. In the third stage, energy is harvested by the production of ATP, a molecule that is the universal "energy currency" in cells that can be used to do work such as muscle contraction. Phosphoglycerate kinase (PGK) catalyses the seventh step of the pathway and is arguably the most important because it is the first reaction that produces, rather than requires, energy. "It is quite a long and complicated process," explains Matthew Bowler, scientist at the ESRF who studies PGK.

PGK harvests the energy produced in glycolysis by transferring a phosphate "energy unit" from one half of a glucose molecule and adds it to adenosine diphosphate (ADP – ATP with one missing phosphate) to form ATP. Bowler teamed up with the University of Sheffield (UK), the Institute of Enzymology (Hungary) and the University of Manchester (UK) to unveil the structure of PGK.

PGK is formed from two lobes that bind the molecules separately. The protein then swings between fully open and a half-open conformation that binds the molecules. The protein closes completely around the molecules. One of the major problems is that phosphate groups are highly negatively charged and therefore repel each other when they get too close. PGK neutralises the negative charges of both ADP and phosphate allowing them to get close enough to react. It then pulls the phosphate off the three carbon unit and creates ATP. When it opens again there is a new three carbon unit that goes into the next step of the pathway and ATP is released to be used by the body.

The team just published the structure of the protein in its fully closed conformation just at the moment that ATP is formed. They looked at the chemistry of the phosphate "energy unit" as it moves from one molecule to the other. Combining the techniques of fluorine nuclear magnetic resonance with X-ray crystallography has revealed in unprecedented detail the chemistry of the reaction (M J Cliff *et al.* 2010).

They also recently studied the large domain movements needed by PGK to bind and release molecules using the new ID14-3 beamline at the ESRF. By combining small angle scattering and macromolecular crystallography, a full picture of both the domain movements in physiological solutions and atomic details of the molecules needed to generate energy has been developed. The results are currently being submitted for publication.

"It is important to know how proteins catalyse this reaction because it is so fundamental and universal, and nobody really knows what sort of transition states take place when it happens," explains Bowler.

M Capellas

Reference

M J Cliff *et al.* 2010 *J. Am. Chem. Soc.* **132** 6507–6516.

Scientists disclose some secrets of photosynthesis

Plants, algae and photosynthetic bacteria produce more than 180 billion tonnes of organic matter each year from the fixation of carbon dioxide. Despite the apparent simplicity of this process, it consists of different steps and many proteins take part in the chemical reactions.

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Plants go through different steps in photosynthesis that are fuelled by water and light.

At the end of April when the greyness of the winter was fading away and longer and sunnier days gradually began, a greener landscape spread through France. At the same moment, a timely paper from a team of the University of Gothenburg (Sweden) and the ESRF shed new light on how photosynthetic bacteria are responsible for the transformation of light into chemical energy, a mechanism that is also shared by plants.

The focus of the study was a photosynthetic reaction centre where the primary energy conversion reactions of photosynthesis take place, and is therefore central to the conversion of light to chemical energy during photosynthesis. Although this membrane protein complex was isolated from a photosynthetic bacteria called *Blastochloris viridis*, it is closely related to Photosystems I and II, which perform the same task in plants. Scientists wanted to study the structural changes that happened within the protein only milliseconds after inducing them with light. In order to follow the structural changes caused by light-induced electron movements, they used time-resolved Laue diffraction crystallography at ID9B. Their results showed that the membrane protein is able to stabilise the light-induced electron movements using subtle structural changes within the protein involving atomic movements of only a fraction of a nanometre (A B Wöhri *et al.* 2010).

Richard Neutze, leader of the team, explains that "this is the first time that the method of time-resolved Laue diffraction has been successfully used to observe structural changes within a membrane protein complex. Because our structural results provide new insight into how the charge separation reactions of photosynthesis are stabilised by protein structural changes, some key ideas could help guide the design of future synthetic systems for artificial photosynthesis."

Photosystems I and II

In plants, Photosystems I and II govern the first steps in the photosynthesis process (but, curiously, they were the last to be discovered). The way that photosynthesis works in plants is that Photosystem II harnesses light energy to split two water molecules (H_2O) into O_2, protons and electrons. It drives one of the most oxidising reactions known to occur in nature and is responsible for the production of atmospheric oxygen. Photosystem I also captures sunlight and takes the electrons released by Photosystem II through an antenna system, consisting of a pigment network, to the centre of the molecule, where it is used in the transmembrane electron transfer reaction.

A team from Tel Aviv University (Israel) used the structural biology beamlines at the ESRF and Swiss Light Source to uncover the most complete plant Photosystem I structure obtained so far, revealing the locations of and interactions among 17 protein subunits and 193 non-covalently bound photochemical cofactors. The new structure allowed the scientists to study the contacts among protein subunits, which can elucidate questions about its function and organisation (A Amunts *et al.* 2010).

Complementary techniques

Different X-ray techniques from X-ray crystallography can also be used to study photosynthesis.

Five years ago, Michael Haumann and Holger Dau, from the Freie Universität Berlin, used X-ray fluorescence on ID26 to investigate the kinetics of the photosynthesis process in Photosystem II. They confirmed the existence of a fifth step in the catalysis process of water into oxygen (Haumann *et al.* 2005). This step is particularly important because it is directly involved in the formation of molecular oxygen. In 2008, they used X-ray absorption near-edge spectroscopy to study the photosynthesis cycle with an additional intermediate and proposed a new reaction mechanism on a molecular basis for the release of dioxygen. Dioxygen is formed as a product of the water oxidation chemistry in Photosystem II (M Haumann *et al.* 2008).
M Capellas

References

A Amunts *et al.* 2010 *J Biol Chem.* **285(5)** 3478–86.
M Haumann *et al.* 2005 *Science* **310** 1019–1021.
M Haumann *et al.* 2008 *PNAS* **105** 17384.
A B Wöhri *et al.* 2010 *Science* **328(5978)** 630–633.

Fuelling nature to pr

Transportation fuels that reduce emissions are a promising answer to our polluted world. Despite its discovery in the 1920s, Fischer-Tropsch catalysis may well be a good candidate for a cleaner future. Colourless, odourless and low in toxicity, Fischer-Tropsch-based diesel fuel reduces sulphur, nitrogen oxide and carbon dioxide emissions compared with regular diesel, and it does not require engine modifications in vehicles for its use.

Image of the gas to liquids (GTL) plant of Shell in Malaysia. GTL is a refinery process to convert natural gas or ot

Hydrocarbons, such as crude oils, bitumen and natural gas, are still available in nature, so Fischer-Tropsch (FT) synthesis has only been partially popular since its discovery back in 1925. In certain moments in history it became relevant when there was a lack of easily available crude oil: during World War II its use was mostly military, later, the oil crisis in the 1970s led to some recovery of interest in FT. Today, the fact that natural liquid hydrocarbons are becoming less abundant, not easily accessible and containing more impurities, such as sulphur, nitrogen and metals, and that there is an increasing pressure for a more sustainable society, has put FT back in the limelight.

FT technology converts natural gas, coal or biomass to clean-burning fuel or hydrocarbons. In a first step, these resources are converted to synthesis gas, in short syngas (mixtures of carbon monoxide and hydrogen). Then the syngas reacts on a cobalt or iron catalyst to yield hydrocarbons in a kind of polymerisation process, leading to mainly long-chain hydrocarbons or waxes. These are often broken up again in diesel fuel molecules.

Cobalt catalysts are considered the most effective for the synthesis of long-chain hydrocarbons. These catalysts are optimised for conversion of stochiometric syngas mixtures, mainly coming from natural gas. On the other hand, iron-based FT catalysts are normally used for the conversion of synthesis gas from coal and they are promising catalysts for the conversion of biomass because they possess water–gas shift properties (i.e. altering the hydrogen to carbon monoxide ratio in synthesis gas). At the ESRF, users study both types of catalysts.

Scientists are trying to find out what happens during the FT reaction and with this knowledge they want to design more active and stable catalyst materials. The deactivation of a FT catalyst results in a gradual decrease in the FT reaction rate. There are many possible reasons for FT deactivation, such as reoxidation of active sites, sintering, catalyst poisoning from impurities in the syngas feed, polymeric surface carbon formation or surface metal-support compound formation (Tsakoumis *et al.* 2010). FT catalysts operate in multiphase medium and at higher pressures and temperatures. Not so long ago, the only way to study these catalyst systems was through *ex situ* methods of catalyst characterisation. These are not reliable due to the changes that the structure can undergo when withdrawn from the reactor. For example, cobalt metal particles can be reoxidised when exposed to air.

Using synchrotron methods and with a proper sample environment where the reaction conditions can be reproduced (i.e. high temperature, syngas reaction environment and preferably high pressures), scientists can now perform a direct *in situ* or *in operando* characterisation of a catalyst in FT reactions. The wide array of techniques allows scientists to get information on different aspects of catalysis structure, such as the nature of crystalline phases, sizes of metal and oxide nanoparticles (X-ray diffraction) or the oxidation state of the atoms surrounding the catalyst (X-ray absorption fine structure spectroscopy or XAFS). Where possible, these synchrotron-based techniques are directly combined with more conventional methods, such as Raman spectroscopy.

At the Swiss Norwegian Beamline (SNBL – BM1), scientists have tested cobalt catalysts over several years. The team at SNBL has built

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ther gaseous hydrocarbons into liquid synthetic fuels, using a syngas as an intermediate in FT.

a set-up with an *in situ* cell in collaboration with the Norwegian University of Science and Technology (NTNU) in Trondheim. In the cell, they include a small catalyst and a reaction mixture and observe the state of the catalyst under industrial conditions. According to Olga Safonova, scientist at BM1: "This sample environment allows the reproduction of experimental conditions (high pressure up to 20 bar, high temperature, space velocity of syngas and FT reaction rates) close to the ones used in industry. The set-up is suitable to perform structural studies on working FT catalysts using X-ray diffraction, X-ray absorption spectroscopy and Raman spectroscopy."

A team from the University of Lille recently carried out experiments on an alumina-supported cobalt catalyst using X-ray diffraction. The results showed that at a pressure of 20 bar and a temperature of 220°C, i.e. industrially relevant conditions, cobalt sintering took place during three to five hours of reaction. After eight hours of experiment, the cobalt in some catalysts would, to a smaller extent, transform into cobalt carbide. These two processes contributed to the deactivation of FT catalysts (Karaca *et al.* 2010).

In similar conditions, the team from the Norwegian University of Science and Technology carried out experiments using X-ray diffraction and X-ray absorption near edge structure (XANES) to reveal information about cobalt-based FT catalyst promoted with rhenium. Small amounts of noble metals like rhenium are added to FT synthesis in order to improve it. In the case of rhenium, it increases dispersion of the catalyst. This time the scientists studied the changes in the cobalt crystallites during FT synthesis in two different conditions: first, at FT synthesis conditions (483 K, 18 bar and low gas hourly space velocity) no significant changes in the cobalt crystallites were observed during the first hours of reaction. Cobalt carbide was not formed in these conditions. Running the reaction at higher temperatures and predominantly methanation conditions led to significant sintering of the cobalt particles and a further reduction of a partially reduced catalyst. This demonstrates how sensitive this complex reaction is to the reaction conditions (Rønning *et al.* 2009).

Iron-based catalysts have similar mechanisms to the cobalt ones. They consist of a precipitated iron oxide phase to which elements are added to improve the catalytic process. Scientists from the University of Utrecht (the Netherlands) went to the DUBBLE beamline (BM26) to measure the reaction process in different iron-based FT systems, including different amounts of additives. Typical so-called promoters are copper, potassium and silicon dioxide. The latter is added to disperse iron phases and to prevent sintering and attrition of the active catalyst, copper improves the reducibility of the catalyst and potassium is used to improve the selectivity of the catalyst towards longer hydrocarbon chains. The team combined XAFS and wide-angle X-ray scattering (WAXS) techniques to get complementary information. WAXS showed crystalline phases to be present after activation and during FT synthesis, and XAFS analysis suggested that, for some catalysts, the majority of iron was present in amorphous phases, which were harder to detect by WAXS (de Smit *et al.* 2009).

Industrial use

This type of research might seem very fundamental, but FT technology is already being used in several pilot and large commercial refineries all over the world (South Africa, Malaysia, Qatar, China) and most of the work done at the ESRF is somehow linked to industrial uses: companies like Shell, Total or Statoil often appear in the scientific results. The probable worldwide leader in FT research, though, is Sasol, a South African company that has been producing fuel this way since 1950. Sasol has made the most of the coal mines in South Africa and it extracts nearly 50 million tons of coal annually.

Researchers from SASOL have already visited ID31 twice trying to find the structure of a carbide generally considered responsible for the FT activity in iron-based FT systems. The second time, they brought with them a reaction chamber at 20 bar that could reach up to 300°C, in order to reproduce the exact conditions in industry.

M Capellas

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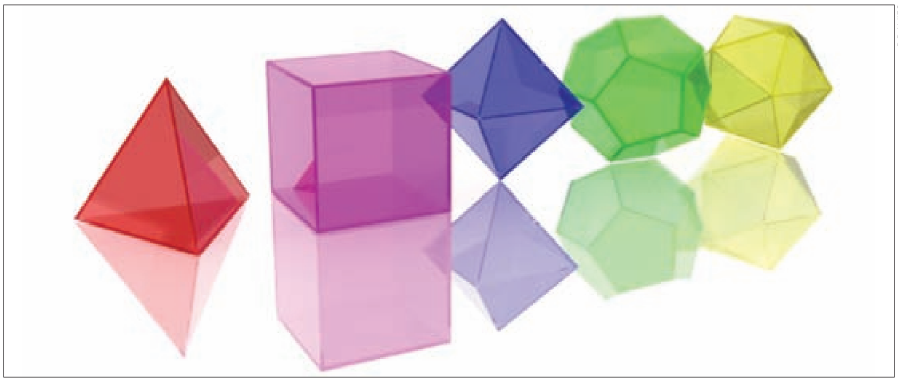

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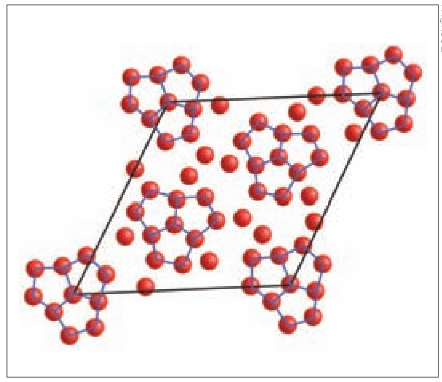

T SCHÜLLI

Left: the five Platonic solids: tetrahedron, hexahedron (cube), octahedron, dodecahedron and icosahedron. The first three structures are found as atomic arrangements in crystals while the latter two cannot form space-filling periodic structures due to their five-fold symmetry. The supercooled liquid droplets of this study were produced on a surface containing a large fraction of atoms arranged in pentagons (right).

Throwing light on the supercooling puzzle

Deep supercooling, a state where liquids do not solidify even far below their normal freezing point, is still subject to discussion.

A good example of this phenomenon is found every day in meteorology: clouds in high altitude are an accumulation of supercooled droplets of water below their freezing point. Scientists from the Commissariat à l'Energie Atomique et aux Energies Alternatives (CEA), the Centre National de la Recherche Scientifique (CNRS) and the ESRF Collaborating Research Group BM32 have found experimental insight into supercooling of liquids in contact with a substrate.

Well below their freezing point, supercooled liquids are trapped in a metastable state. This can only be achieved in liquids that do not contain seeds that may trigger crystallisation. In everyday life, though, there is usually some crystalline impurity in contact with the liquid that will trigger the crystallisation process, and therefore freezing. Controlling the solidification behaviour is important for applications ranging from hail prevention up to technological processes such as welding and casting, or even the growth of semiconductor nanostructures.

Supercooling was discovered in 1724 by Fahrenheit while observing that water droplets stay liquid below 0 °C. In the 1930s, the suppression or reduction of supercooling by the presence of crystalline solids in contact with the liquid was discovered. Experimenting with pure liquid metal droplets on glass (non-crystalline) substrates, David Turnbull confirmed in the 1950s the presence of deep supercooling in almost any elemental liquid where the solid state has 12-fold coordination (this means that in the solid state every atom is surrounded by 12 equidistant neighbours). This led to the speculation that the atoms in the liquid could locally arrange in icosahedra, a highly symmetric platonic solid with 12-fold coordination and five-fold symmetry. To form a crystal, however, one needs a structure that can be repeated periodically, filling the entire space. This is not possible with five-fold coordinated clusters. In the two-dimensional analogue, a plane cannot be filled by pentagons only, whereas triangles, rectangles or hexagons can fill a plane perfectly. In this example, pentagons are an obstacle to crystallisation.

Until today there was no experimental proof that these five-fold symmetric structures are at the origin of supercooling. The researchers from the CEA, CNRS and the ESRF studied the structure of a particular liquid, a gold–silicon alloy, in contact with a specially decorated silicon (111) surface, where the outermost layer of the solid featured pentagonal atomic arrangements. Their findings confirmed that a strong supercooling effect took place. "We studied what happened to the liquid in contact with a five-fold coordinated surface," explains Tobias Schülli, first author of the paper. The team performed the control experiment with the same liquid exposed to three-fold and four-fold coordinated surfaces, which reduced the supercooling effect dramatically. "This constitutes the first experimental proof that pentagonal order is at the origin of supercooling," explains Tobias Schülli. In his *Nature* "News and views" article, A Lindsay Greer, of the department of materials science and metallurgy at the University of Cambridge (UK), explains that "the results have wide implications not only for fundamental studies of freezing, but also for the practical control of the phase transition".

It was during their studies, originally focusing on the growth of semiconducting nanowires, that the scientists discovered the unusual properties of these liquids. As they were observing the first stage of growth of nanowires, they could see that the metal-semiconductor alloy that they used remained liquid at a much lower temperature than its crystallisation point and so they decided to investigate this phenomenon.

These liquid alloys enable the growth of sophisticated semiconductor nanostructures at low growth temperatures. Most of these nanowire structures are grown on silicon (111), the same surface used by the team. Semiconducting nanowires are promising candidates for future electronic devices. Prominent examples are solar cells, where scientists are working on the integration of silicon nanowires in order to increase their performance.

In this framework, a new instrument allowing chemical vapour deposition growth is currently being commissioned on beamline BM32, funded by the Fondation Nanosciences in Grenoble in order to boost the characterisation tools available for R&D in nanoelectronics, where nanowire-based devices are expected to play a key role.

M Capellas

Reference

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Observation of the collective Lamb shift for the first time

An ensemble of identical atoms submitted to irradiation by light behaves differently to a single atom. Jointly, the atoms emit light at a lower frequency than a single atom would. This effect, the so-called collective Lamb shift, was recently observed by scientists from the Deutsches Elektronen-Synchrotron (DESY), the University of Leuven (Belgium) and the ESRF.

M CAPELLAS

Part of the team in a recent experiment on ID22N. Left to right: Kai Schlage (DESY), Rudolf Rüffer (ESRF), Ralf Röhlsberger and Balaram Sahoo (DESY).

Research carried out by ESRF users has provided evidence of an effect that theorists had predicted more than 35 years ago but that could not be experimentally proven. And it made the cover of the journal *Science*.

The Lamb shift is a small difference of the oscillation frequency of electrons in the atom. It becomes visible when light causes atoms to radiate. The frequency shift occurs when the excited atom emits and re-absorbs its light several times before returning to its ground state. The discovery of the Lamb shift in hydrogen in 1947 laid the foundation for the development of quantum electrodynamics (QED) as a unified theory of interaction of light and matter. For this discovery, the physicist Willis Lamb received the Nobel prize in 1955.

When an ensemble of identical atoms is excited to radiate, it is possible that the emitted light of an atom is not only absorbed and re-emitted by the single atom but also by other atoms of the ensemble. As a result, the light emitted by these atoms exhibits a distinct red shift compared with the light that a single isolated atom would emit.

For the experiments, the team of researchers developed a new measurement method on ID22N. They placed an ensemble of ^{57}Fe atoms between two platinum mirrors separated by only a few nanometres and irradiated this array with X-ray radiation. In fact, the predicted collective frequency shift could be measured in this way, even though it was believed for a long time that the atoms must not be separated by more than a wavelength. The researchers took advantage of the fact that the radiation of ^{57}Fe atoms is enormously intensified, making the collective Lamb shift clearly visible. With the help of Mössbauer spectroscopy, the shift could be determined very precisely. The measured values are in excellent agreement with theoretical predictions.

"Theorists had predicted this effect more than 35 years ago."

This experimental method also offers new possibilities to study collective effects in the interaction of light and matter. For example, the researchers observed that the light from the ensemble of atoms was emitted almost 100 times faster than from a single isolated atom. This phenomenon is called superradiance. Superradiance enables a very efficient energy transfer between light and matter and it may play an important role for designing more efficient solar cells and in fast optical information processing.

T Zoufal

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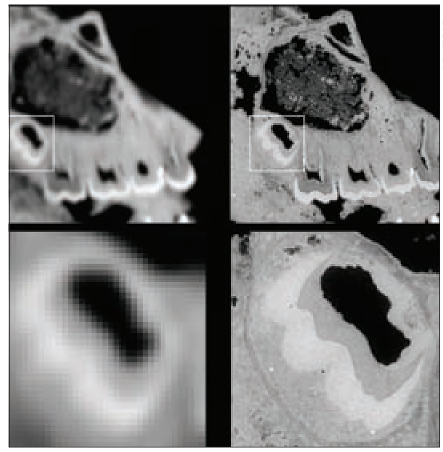
Well preserved new hominid fossil finds its way to the ESRF

The fossil of *Australopithecus sediba*, a 1.9 million-year-old hominid discovered last year, spent two weeks being scanned at the ESRF before its discovery was announced. The data will take the team years to analyse, but first results already unveil some clues.


I MONTERO


P TAFFOREAU


P TAFFOREAU

Left: Lee Berger and Paul Tafforeau discuss the data at ID17. Centre: rendering of the 3D scan of the skull of an *Australopithecus sediba* child. Right: comparison of a classical computer tomography scan (left) with a scan using synchrotron microtomography (right). The lower images are of a tooth. To study the development of teeth, a resolution of between 10 and 100 times higher than that used on this image is required.

On 9 April the cover of *Science* featured a skull of a 1.9 million-year-old fossil. In two articles, scientists described a new species of fossil hominid, *Australopithecus sediba* (*sediba* meaning natural spring or wellspring in Sotho, one of the many languages in South Africa). The species is thought to be a good candidate for being a transitional form between the southern African apeman *Australopithecus africanus* and either *Homo habilis* or even a potential direct ancestor of *Homo erectus*. The species has long arms, like an ape, short powerful hands, a very advanced pelvis (hip bone) and long legs capable of striding and possibly running similar to modern man.

Lee Berger, a scientist from the University of the Witwatersrand (Johannesburg, South Africa) who discovered the fossils in 2008, and Paul Tafforeau, palaeoanthropologist at the ESRF, after a joint examination of the fossils in Johannesburg, decided to use X-ray synchrotron microtomography at the ESRF to study these precious samples. Berger arrived at the ESRF on a snowy morning in February 2010, escorted by police and with a "diplomatic bag" attached to him.

The bag contained the uniquely preserved fossils, which belonged to a juvenile aged between 10 and 13, based on modern human standards. They included the skull, vertebrae, several pieces of bones and teeth. The team's original aim was to analyse in detail the teeth of the fossil. Studying their internal growth lines and structure to the daily level could provide the age at death of the individual. By comparing his real age and his developmental level, scientists would gain insights about his life history 1.9 million years ago. The skull was first completely scanned with a resolution of 46 µm on ID17 for four days, then all of the dental remains and most of the bones were studied on ID19 up to the sub-micron resolution level over seven days. The total amount of material studied represents nearly 40% of the bones of a complete skeleton.

Berger and the international team pushed the investigation further by using the ESRF to look at possible remnants of soft parts of the body that normally do not fossilise, such as the brain. The preliminary visualisation of the complete skull shows three potentially fossilised insect eggs. The eggs could belong to a wasp and a fly. The researchers are still not sure if these insects are contemporary to the remains of the body, but careful analysis of their densities should help to verify that aspect. If they are contemporary, they may have fed on the flesh of the hominid after death. The team also noticed an extended low density area that could be a remnant of the brain after its bacterial decay.

They noticed that the skull was broken. The explanation for this lies in the hypothesis of how the hominids died. The bodies appear to have fallen, along with other animals, into a deep cave, lying on the floor for a few days or even weeks. At that stage, a natural mummification appears to have occurred. The bodies were later washed into an underground lake or pool, probably as the result of a rainstorm. They did not travel far, maybe a few metres, where they were solidified, as if thrown into quick-setting concrete. Over the past 1.9 million years the land has eroded to expose the fossil-bearing sediments.

The team found a strange cavity in the brain case during the scans on ID17. Tafforeau explains the reason for this: "Before fossilisation took place, the brain would have lost most of its water during the mummification event, causing it to shrink to probably less than a 20th of its original size. Once the body was immersed in water, sediment filled the brain cavity very rapidly. This would have created a natural cast of the shrunken brain. Then, bacteria would have finished the natural decay of the rest of the brain, resulting in a cavity surrounded by the sediment." The rock that the fossils are preserved in is called calcified clastic sediment.

The preliminary hypotheses that the team has come up with need to be verified. Results need to confirm or contradict them and it may take years until the results are published.

It is only the second time that a complete hominid skull has been examined using powerful synchrotron radiation. The first one was of *Toumaï*, which was about 7 million years old. This kind of high-quality analysis on complete hominid skulls is currently only possible at the ESRF.
M Capellas

Closed European airspace tests the ESRF's adaptability

The ashes from a volcano in Iceland put the air traffic in Europe on hold for a whole week in April. The ESRF relies on flights for its user operation, so this event unleashed the creativity of the management and the travel office to find new ways of functioning.

J CUSACK

J CUSACK

Left: the Swedish team of users ponders on what non-air route will take them home the fastest. Right: German users waiting for rental car keys join the long queues in front of the travel office.

Three Swedish researchers, Glareh Askarieh, Karin Valegard and Li Zhang, arrived at the ESRF on 15 April for a one-day experiment on ID14-4. On the day of their experiment, the ash of the volcano Eyjafjallajokull started covering the European skies and grounded flights to the north of Europe. Their flights, due on Saturday 17 April, were cancelled, and so were the new flights that they were assigned to on 19 April. Askarieh even got two reservations for flights on the 21 April (one in the morning, one in the evening) to double the chances of finally leaving France. There was no luck. Finally, they left on 22 April in a convoluted trip that included a train to Paris, another one to Amsterdam (without a seat, though) and the hope that they could find other means of transport from Amsterdam to their destination, Stockholm.

The case of these three Swedes is probably an extreme of the disturbances that the volcano caused. Other cases include a German team from the Technical University of Dortmund that was forced into a nine-hour drive home instead of taking a one-hour flight. The scientists, Christian Sternemann, Christoph Sahle and Alexander Nyrow, stayed one day more than expected at the ESRF after their experiment on ID16, but seemed relatively happy when they got the key of their rental car to go back home. "The three of us drive, so it's not too bad," explained Sternemann.

For Karl Buch, from the University of Mainz, his five-day experiment on ID17 ended with a printed boarding pass but no flight to board: "I printed the pass in the evening and woke up in the morning to see that my flight had been cancelled." His alternative route was by train and that would probably last all day.

These users are only some examples of the "victims" of the volcano, but there were more. From the beginning of the worst air crisis in Europe the telephones in the users' office and at travel and reception didn't stop ringing. Joanne McCarthy, head of the users' office, explains that: "It was a completely unexpected situation and we were as much in the dark as the users as there was no way to tell how long this situation would last. The travel and reception and the users' office worked hard to ensure that users stranded at the ESRF could stay on at the guesthouse and to try to find alternative methods of transport for those wanting to leave and for those trying to get here. The situation was worsened by the ongoing French railway strike."

Despite the very complicated situation, only eight experiments were cancelled in all and six of those were one-day macromolecular crystallography (MX) experiments that were scheduled mainly over that weekend. There were 24 MX experiments scheduled over the period 16–22 April and many of those were able to continue thanks to the success of Remote Access data collection. Many users who couldn't travel had either sent their samples in advance or managed to send them by road. The samples were loaded into the robotic sample changers by local staff and users were able to run their experiments as planned from their home laboratories. Without this, the effect on the MX beamlines would have been much more dramatic.

The six MX experiments that were cancelled will be rescheduled later in the 2010/I period while the two non-MX experiments will be carried over into the 2010/II scheduling period. Some users who remained stranded at the ESRF had their patience rewarded with some extra beamtime on beamlines where users had not been able to come. This was the case of the Swedish researchers, who could still do some scans after their beamtime was finished: "That was a good opportunity, but we couldn't make the most of it because we didn't have enough crystals to scan for the five extra days," explains Askarieh.

Proposals reviewed by conference calls

Less than a week after the planes were grounded, the ESRF was due to hold the beamtime allocation panels i.e. the biannual meetings of the 11 committees that select the scientific proposals that are awarded beamtime in the next round. Having realised that the air chaos was going to last a while, the head of the users' office and directors of research took the decision to cancel the face-to-face meetings and put their shoulder to the wheel to organise, together with the so-called ESRF liaisons (scientists from the ESRF who act as secretaries to the committees) a series of telephone conferences with the members of each committee. The outcome of this "emergency scheme" was very positive: "Thanks to the immediate actions and superb co-ordination between the users' office, the liaisons and the chairpersons of the 11 committees, all the relevant proposals could be discussed in the telephone conferences. A list of selected proposals was drawn up with the usual care and full consensus between the panel members. We had almost all reviewers contributing to the allocation process, which is rarely the case in face-to-face meetings," explains Harald Reichert, one of the directors of research.

Despite the success of this format, it will not be applied in the next round of panel meetings: "We realised that especially the younger panel members and newcomers favour physical meetings at the ESRF. It allows them to get familiar with the process and profit from the direct exchange with their colleagues," explains Reichert.

M Capellas

Transporting radioactive samples from the laboratory to the ESRF

If Superman worked at the ESRF, he wouldn't have to worry about kryptonite. The radioprotection group makes sure that any potentially dangerous element enters the ESRF following strict safety rules. Together with the CEA and Forschungszentrum Dresden-Rossendorf, they have developed a new protocol for transporting radioactive samples.

Kryptonite is a fictitious material, but counterparts of it exist in real life. In fact, some of the materials that come to the ESRF could do much more harm than the imaginary material. Nuclear energy, a domain that is increasingly being studied by scientists worldwide, involves research in actinides – the radioactive and heaviest elements of the periodic table. The X-ray absorption spectroscopy (XAS) station of the Rossendorf beamline (ROBL, owned by Forschungszentrum Dresden-Rossendorf and part of the Actinet-I3 network programme) at the ESRF is dedicated to this research and is, as such, the first of its kind at a synchrotron in Europe. At ROBL, scientists study low concentrations of radionucleotides in a large variety of media, including aqueous and non-aqueous solutions, rock minerals, clay minerals, micro-organisms, plants and soil sediments, but also aspects of nuclear waste management and the development of nuclear fuel.

According to the safety regulations at the ESRF, all radioactive samples have to be controlled by gammametry before leaving the home laboratory and on arrival at the ESRF. These regulations are required by French and European legislations to control the radioisotope identity and mass before and after experiment. If the activity is lower or higher than estimated, the levels of radiation protection will be inappropriate, resulting in an overestimated or underestimated impact of a possible incident. Equally, the ESRF has to meet nuclear non-proliferation requirements by respecting a maximum quantity of nuclear matter, e.g. a cumulated limit of 1 g of plutonium in transit per year. A difference of 2 or 10% between the estimated activity and the effective activity will have important consequences on the possible number of experiments to be carried out.

Improving the system

The ESRF radioprotection group joined efforts with the nuclear division of the CEA Atalante (Marcoule) and the ROBL beamline to define a common procedure for sample radioactivity measurements between the home institutes and the ESRF.

They developed dedicated cells where the samples would be inserted. These sample cells must meet specific safety regulations and technical requirements allowing XAS measurements. It is a difficult task due to the various sources of errors from the sample itself but also from geometric variations. The variety of radionucleotides that may be present at the same time in a cell brings another source of difficulty into the measurement.

P AVAVIAN/CEA

A scientist fills the inner cell with a liquid sample at the Atalante facility. Protection equipment is mandatory, including hot lab hoods and a respiratory mask.

Each sample cell can hold a maximum of 12 samples (solid or liquid), it is composed of a modular arrangement of four inner cells. The inner cell can hold a maximum of three samples of 200 µl. It is composed of a Teflon core, two Kapton windows and two stainless-steel plates, all parts being held together by screws and Viton o-ring sealing. The combination of Teflon and Viton o-rings has proven a safe enclosure of all solution media tested to date, including mineral acids and organic solvents. Solutions are introduced into each compartment through a small inlet with a needle. Inlets are closed with tap screws and are glued shut. After completion, a first radiological control of non-contamination is performed on the outside of the cell. Inner cells are then positioned in the sample cell with a second confinement barrier of Kapton windows with flat o-rings and outside frame to form a double-confined device.

The final radiological control is performed on the outside windows of the sample cell before the gamma spectrum is collected. The gamma spectrum is a fingerprint of the contents of the cell. It is a quantitative identification of the radioisotopes and impurities present in the cell by estimating the radioisotope concentration. Gamma spectra are recorded at the home facility before transportation of the cell to the ESRF and at the ROBL beamline on arrival at the ESRF.

To rationalise the procedure between the Atalante facility and ROBL at the ESRF, several gammametric tests have been performed with radioactive standards. Quantitative gamma measurements highly depend on the sample geometry, mass or volume, positioning of the cell with respect to the detector and detector calibration. These parameters are difficult to establish with precision and must be defined with radioactive standards in fixed geometries. This is the purpose of defining a reference cell in accordance with four parameters: the theoretical activity of the sample itself, intrinsic errors (volume, mass, etc), extrinsic errors (positioning of the cell on the detector) and calibration uncertainties.

Working with "real samples" entails several additional challenges. First, the exact amount of radioelement inside the sample and consequently inside the cell is difficult to define with precision, given the processing of the sample and uncertainties in volumes or masses. In that respect, solutions (they are more homogeneous) are less subject to errors than solids are. Second, mixing several radioelements (and potentially several radioisotopes) may lead to interferences between the gamma emission lines, resulting in misleading integration values. Finally, the geometry of the positioning of the sample cell on the detector is difficult to adjust with a sufficient reproducibility.

The collaboration among the three labs has already achieved a standard of handling of radioactive samples that is being implemented by other synchrotron sources. The team is now focusing on producing a cell for calibration in the home laboratory and at the synchrotron prior to the sample-cell measurement. This procedure would ensure a better transferability of the data.

L Venault, C Den Auwer, P Moisy (CEA), P Colomp (ESRF), A C Scheinost and C Hennig (FZD)

Building the site of the future

It only takes a stroll in the experimental hall to notice that the ESRF Upgrade is already a reality. Simultaneously with some of the Upgrade beamline (UPBL) projects underway, the details of the EX2, the extension of the experimental hall, are being defined.

MASTER

NICKL & PARTNER

Top: a view of the new entrance from above. Bottom: an artist's impression of the science building's entrance. The building will be located next to the ESRF/ILL stores.

A very important aspect of the first phase of the Upgrade Programme is the beamlines that will be upgraded and those that will change position in the floor plan. The floor plan foreseen at the beginning of the Upgrade has evolved into a new one where fewer shuffling of beamlines means better performance as well as notable financial and time savings.

One of the beamlines that will benefit the most from the new floor plan is ID31. It is relatively easy to move it to another location thanks to the simplicity of its optics and diffractometer. The preferred position for the beamline is at one of the high-β sectors, which are found at even-numbered straight-sector beamlines. Due to the smaller source divergence in a high-β sector, the refurbished beamline will be able to achieve higher resolution.

This is the reason why it will occupy the space free on ID22, after NINA will be built on the ID16 slot. The space left by ID31 will be taken by UPBL2 (former ID15). UPBL2 requires a low-β section, which translates into an odd-numbered beamline slot like ID31. The space left by ID15A will be occupied by the Large Volume Press. The high-pressure branch of ID09 will go to ID15 B. Within the refined floor plan the moves of beamline ID10A/C and ID03 to new positions have been cancelled.

The reorganisation of the long beamlines will start taking place as soon as the design report for EX2 is ready. Its preliminary design report, in preparation, will provide detailed 3D plans of the buildings both inside and outside, and technical proposals for the building-specific problems of the project. However, there are UPBLs that do not need EX2 to become a reality. This is the case of UPBL09B, the time-resolved beamline. It will be kept in its current position and has already started its upgrade: a new monochromator and a laser have been delivered and will be commissioned by the end of the year. By 2011, the beamline will increase its intensity by a factor of 50 for many applications. An important added value of the revision of the floor plan is the integrated saving of three-year shutdown of a beamline. Other UPBLs in construction are UPBL10 and 11.

The works for the long beamlines will start at the end of next year. This will be followed by a three-month shutdown from December 2011. The final commissioning of the new facilities will take place in April 2013.

A new way of welcoming visitors

Visitors to the ESRF will feel the difference when entering the site in three years' time. Thanks to an €8 m investment by the French government and the local authorities, the new site entrance complex will include the site entrance and badge delivery, a visitors' centre, a logistics platform/stores for the whole campus, new offices for the ESRF works committee, a major restructuring of roads and parking space as well as an upgrade and extension of the current restaurant. The contract was awarded to the Spanish company MASTER Ingeniería Arquitectura in June 2010 and the new buildings are foreseen to become a reality between mid-2012 and early 2013.

Not far from the entrance, the so-called ESRF–ILL science building will be built at the same time. The building will be a hive of future projects for the large-scale instruments, housing new joint laboratories, the science library and scientific partnerships, notably the Partnership for Soft Condensed Matter. The construction of this building was awarded to the German architects Nickl & Partner in spring. This 5000 m^2 building will be ready for use in September 2012. Its construction with an associated cost of €10.5 m has the same funding source as the new site entrance.

M Capellas

H Belrhali: India meets Europe on BM14

This is the story of a scientific collaboration that became a friendship, a community of crystallographers urging for synchrotron access and a beamline looking for new staff. When the three merged, it was the beginning of the new BM14.

"Five years ago, it was clear to me that BM14, the macromolecular crystallography (MX) beamline where I have worked since 2001, was probably going to close down at the end of 2009," says Hassan Belrhali, scientist in charge of the beamline.

The end station was owned and operated by the UK Medical Research Council (MRC) in partnership with the European Molecular Biology Laboratory (EMBL), Belrhali's employer, since 2001. MRC informed EMBL and the ESRF that they would concentrate their funding to the newly built national facility, Diamond, when an equivalent beamline would be available.

Convinced that BM14 was still worth operating, Belrhali started thinking of possible partners to "rescue" BM14. Because of the privileged situation of Europe in respect to the number of third-generation synchrotrons and because of his Moroccan origins, Belrhali thought to make BM14 available to a wider community. Indeed, several emerging countries possess strong protein crystallographer communities but do not have secured access to synchrotrons. Before initiating any approach, Belrhali received strong support from EMBL management (Stephen Cusack and Ian Mattaj), the ESRF director-general (Bill Stirling) as well as MRC BM14 head (Dave Stuart).

The idea became more concrete when Belrhali met Amit Sharma, an Indian scientist working at the International Centre for Genetic Engineering and Biotechnology in New Delhi. Both scientists started to collaborate on key enzyme structures of *Plasmodium falciparum* (the malaria causative agent) and they became friends. Sharma told Belrhali about the situation of biocrystallography in India. The strong community, consisting of about 40 groups spread all over India, was urging their authorities to secure access to a modern synchrotron facility

M CAPELLAS

C ARGOUD

Left: Hassan Belrhali and Amit Sharma during an experiment on BM14 in December 2005. Right: BM14 staff working on the beamline.

to carry out structural studies. Indeed, despite being constituted by first-class scientists, the Indian MX community had very limited access to synchrotrons worldwide – mainly via collaborations – and their unique national synchrotron, a second-generation machine called Indus-1, had modest performances in the MX field.

The idea of getting India involved in the running of BM14 started to brew in Sharma and Belrhali's heads. The "availability" of BM14 and the concomitant access needs of the Indian MX community led them to propose to the Indian MX delegates to consider BM14 as a potential platform that they could share with the EMBL and the ESRF.

BM14 represented fast access, staff training and a capacity building opportunity.

Talks with the Indian MX delegates and authorities were very constructive, and soon it appeared to be a strong candidate to join EMBL and the ESRF. "The Indian MX community certainly had different options on different synchrotrons worldwide. However, besides the reliability and performance of the machine and the MX beamlines, what is also unique at the ESRF is its international atmosphere, where everybody comes from different places," says Belrhali. "The ESRF is the only 'international' synchrotron in the world, where many communities feel at home."

Lengthy negotiations driven by the ESRF director-general, Francesco Sette, in the context of the joint structural biology group, led to a scheme where the Indian Institute of Immunology would be associated to the EMBL in a consortium for five years (from 2010 to 2014) on BM14, with the mandate to operate the beamline. Both partners would form a local team – all EMBL staff – and would share the beamline and related costs equally. The ESRF strongly favoured the partnership, bought the beamline and offered to refurbish the 15-year-old optics hutch. As compensation, the ESRF would get 43% of beamtime.

Several meetings and a lot of calculations later, the new BM14 took off. In 2009 the beamline was shared by MRC, India and EMBL, but from 2010 it is entirely run by the consortium. In addition to Belrhali, another scientist (Babu Manjasetty), a postdoctoral researcher (Silvia Russi) and a technician (Sylvian Thuaudet) have been recruited.

Malaria becomes global

Malaria affects more than 1.5 million people in India, where the disease is endemic. Because it affects such a large number of the population, scientists in India study it actively. Sharma is one of them. "Amit taught me that malaria starts becoming a global concern, and not only in developing countries. Climate change is modifying the world temperature patterns, and with the huge circulation of people around the world, the disease is spreading," Belrhali says.

From his first experiments on BM14 assisted by Belrhali, Sharma and collaborators determined the structure of a key protein that allows the parasite to invade human red blood cells, before proliferation and the destruction of host cells. This work featured in *Nature* in 2006.

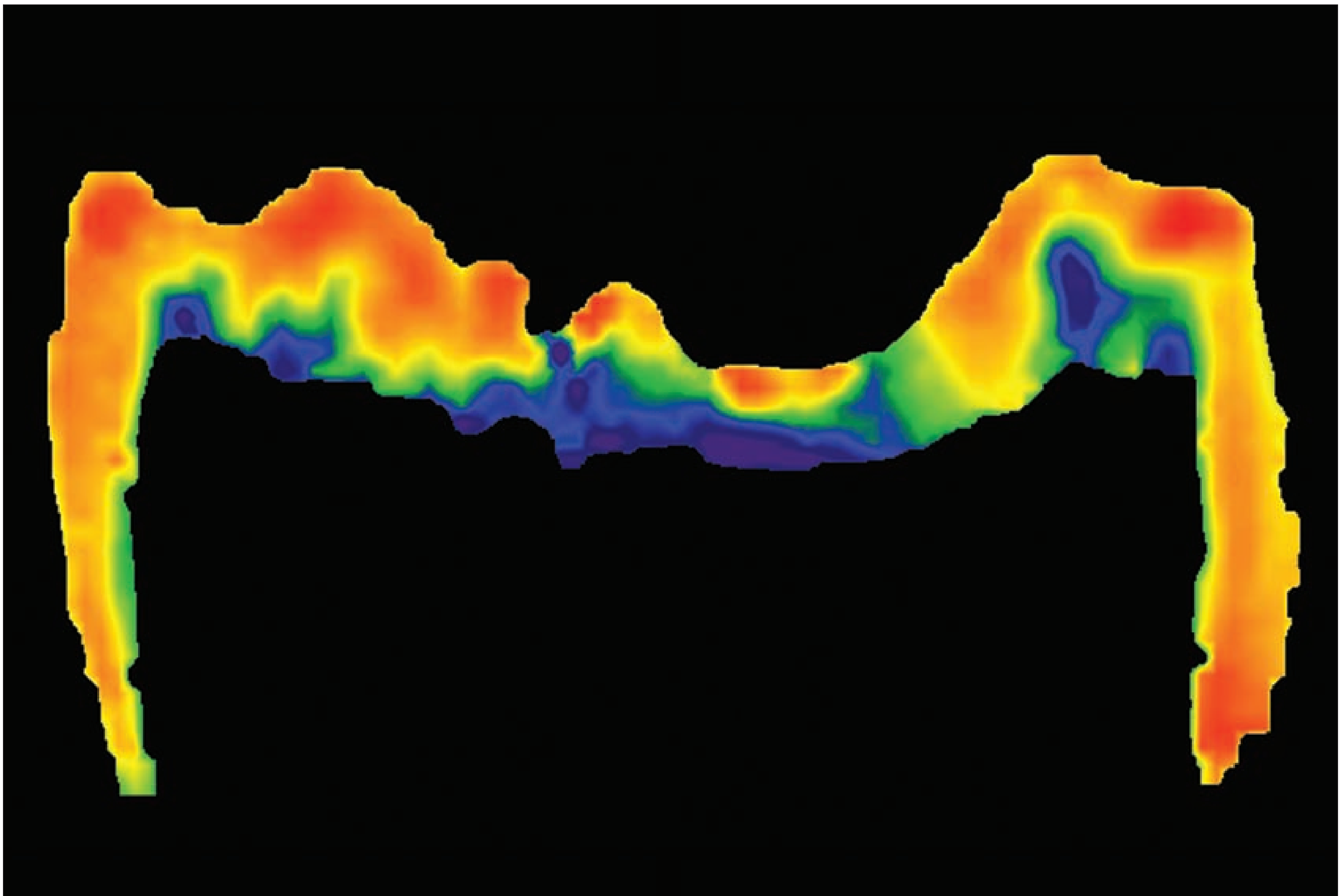
Another parasite that Belrhali is studying in collaboration with Mohamed Ali Hakimi, from the University Joseph Fourier (Grenoble), is *Toxoplasma gondii*, the parasite that produces toxoplasmosis. Despite being different from *Plasmodium falciparum*, Hakimi recently found an inhibitor of *Toxoplasma* that appeared to be efficient against *Plasmodium*. The three scientists (Hakimi, Belrhali and Sharma) have just been awarded a grant to investigate the new inhibitor properties structurally on both parasites.

Training and development

BM14 is the perfect place to accurately measure low anomalous signals that will lead scientists to solve biological structures. It is also the ideal place to train scientists and students in macromolecular crystallography phasing. In the framework of the ESRF Upgrade Programme, it also represents another MX beamline in the ESRF MX portfolio.

The contract with India lasts until 2014. "What will happen next is not clear, India might extend it and I believe that the beamline could serve other emerging communities. A synchrotron is a fantastic place to socialise, meet other cultures and carry out good science," Belrhali acknowledges.

M Capellas

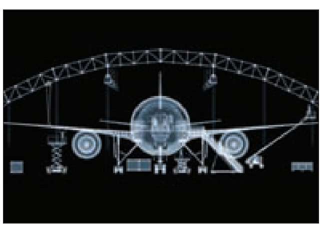


M AL-JAWAD

Texture map of archaeological dental enamel showing the distribution of crystallite orientation across a section of a partially mature tooth. It is generated from the analysis of 2D X-ray diffraction patterns collected on the XMaS beamline by an interdisciplinary team led by Maisoon Al-Jawad (Queen Mary, University of London, UK) and it will be used to model the 3D crystallisation processes occurring during enamel maturation.

In the corridors

Photographer X-rays the world



N VEASY

A Boeing 777 captured in X-rays.

The British photographer Nick Veasey is famous for his technique of taking pictures: he uses X-rays to capture nature in films that are subsequently scanned into a digital file. Some of his photos include an iPod, a Mini, a bat, shoes and pieces of clothes. Probably his most well known artwork is a picture of a Boeing 777, which is made up of 500 separate X-ray images.

His work is compiled in the recently published book *X-treme X-ray*, produced in collaboration with the Science Museum in London (UK). It is published for children and serves as an introduction to the power of X-ray radiation.

The book can be bought at www.carltonbooks.co.uk.

No more bikes in the ESRF experimental hall

C ARGOUD

The well known ESRF red bikes will start to disappear.

The famous red bikes of the ESRF experimental hall are being progressively phased out seven years from when they were first introduced (before that there were bikes of all colours). Currently this mode of transport still takes scientists to the beamlines faster. Due to the high use of the bikes, they have rapidly deteriorated and it has become impossible for technical services to keep them in good condition. For now, walking is the best way to get to your experiment.

Sony says goodbye to the floppy disk

Sony has announced that it will stop producing 3.5 inch floppy disks in March 2011. The reason for this decision is "lack of demand and competition from other storage formats", as stated on the *BBCnews* website.

R DAVIES

The end of the floppy disk.

The death of floppy disks started in 1998 when Apple decided not to include a floppy drive in its G3 iMac computer.

The first floppy disk was invented by IBM in 1971 and was 8 inches in size. In 1981 Sony shrank it to 3.5 inches. The standard disk holds 1.44 MB of data, equivalent to a three-minute song.

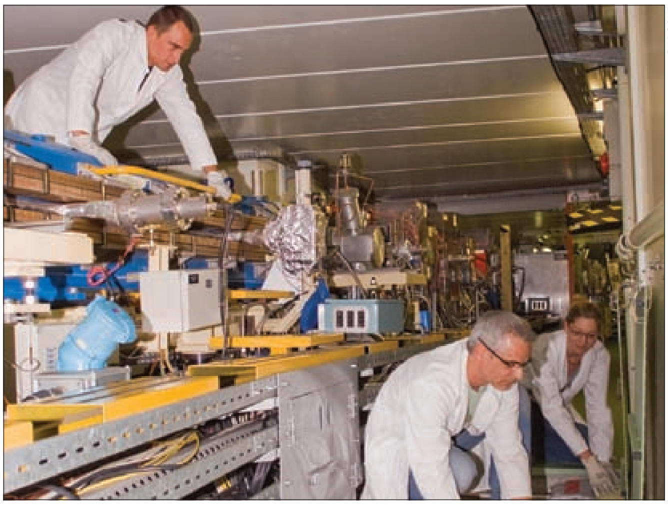
Making the ESRF a radiation-free place

The radioprotection unit applies rules set by the French authorities to keep radiation at bay.

Several years ago, the ESRF decided to make the experimental hall a radiation-free place for workers. This meant that people could move around the facility without having to carry a dosimeter with them to calculate their exposure to radiation. Also, it involved monitors that sense the amount of radiation in the air being placed around the ring and on the optical hutches on the beamlines. Today, working in a synchrotron without wearing a dosimeter is still a privilege, so ESRF staff and users can consider themselves lucky.

Checking the radiation levels is the responsibility of the radioprotection unit. With Patrick Colomp as its head, this unit inside the safety group makes sure that every bit of the ESRF is safe, starting with the experimental hall, passing by the beamlines and ending in the machine, where radiation is produced. The team includes two radioprotection technicians.

The rules on radioprotection are set by the French government. They include the thorough inspection of each beamline twice a year internally plus once a year by an external company. "We are checking them constantly," explains Colomp. "Every time there is an intervention we check before and after to make sure that everything is OK. Having this job, you don't really spend much time in the office, you are always around the ring," he adds.



C ARGOUD

The radioprotection unit in action in the storage ring. From left to right: Yann Pira, Patrick Colomp and Anne-Cécile Rocheville.

Being present on the beamlines is a "must" for the radioprotection team. Colomp insists that if they are around and talk to the scientists then they will get to know them and will be more willing to discuss any safety worries that they may have. Being present pays off and avoids problems, such as those that existed in the early days of the ESRF, when to find holes drilled in the lead blocks of the experimental hutches was not out of the ordinary.

Radioactive samples

Another important task of the radioprotection unit is the control of radioactive samples at the ESRF. The amount of these samples studied at the ESRF has fluctuated over the years, depending on the scientific interests of the users. Today, most of the radioactive samples go to BM20 or ROBL, the CRG beamline owned by the Forschungszentrum Dresden-Rossendorf, where radiochemistry studies are carried out. An article on new protocols on transporting radioactive samples is on p18 of this issue.

With the Upgrade Programme, the team will be even busier as it will need to make sure that all of the new buildings in the experimental hall are as safe as the ESRF is today.

M Capellas

Movers and shakers

Head of Technical Infrastructure Division

Rudolf Dimper

This summer a new division at the ESRF comes to life: the Technical Infrastructure Division. Rudolf Dimper has just been appointed head of this new division, which will have around 85 people.

It will merge the current groups of the Technical Services Division and Computing Services Division into five groups and one project team, notably the Systems and Communication group, Management Information Systems group, Vacuum group, Geodesy and Alignment group, Buildings and Infrastructure group, and the project team for the Upgrade Programme buildings works on the ESRF site (EX2/CPER). Its main aim is to provide the best possible technical infrastructure for the other divisions and our scientific visitors. It is a service division, although it will include some minor internal research and development activity.

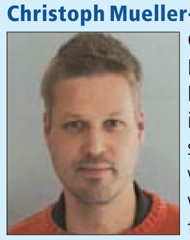
The forthcoming years will be particularly challenging with many ambitious projects ranging from a new data centre in the ESRF central building, the modernisation and extension of our various database tools, to the extension of the experimental hall and the new site entrance. Synergies between the groups will allow streamlining internal work flows that today require complicated individual actions.

From 2003 until his new appointment, Dimper has been head of the Computing Services Division.

New permanent scientists at the ESRF

Christoph Mueller-Dieckmann, biologist on ID29 and Paul Tafforeau, paleontologist on ID19, have both recently changed their five-year contracts to permanent ones.

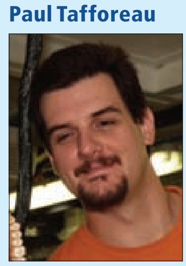
Christoph Mueller-Dieckmann



Christoph Mueller-Dieckmann is a German scientist who started working at the ESRF in 2006 as a beamline scientist on ID29. He is currently also project manager of the ESRF macromolecular crystallography upgrade.

Before working at the ESRF, he had a postdoctoral fellowship at the European Molecular Biology Laboratory (EMBL) in Hamburg. He is a chemist by training although he moved to the field of macromolecular crystallography during his PhD.

Paul Tafforeau



Paul Tafforeau is a French scientist who first used the ESRF during his PhD studies in paleontology.

His PhD work on fossil tooth enamel using X-ray microtomography was the first study of fossils using synchrotron radiation. He later became a postdoctoral researcher on ID17 and ID19, then a CNRS researcher based at the ESRF. He is currently scientist on ID19, focused mainly on experiments in paleontology.