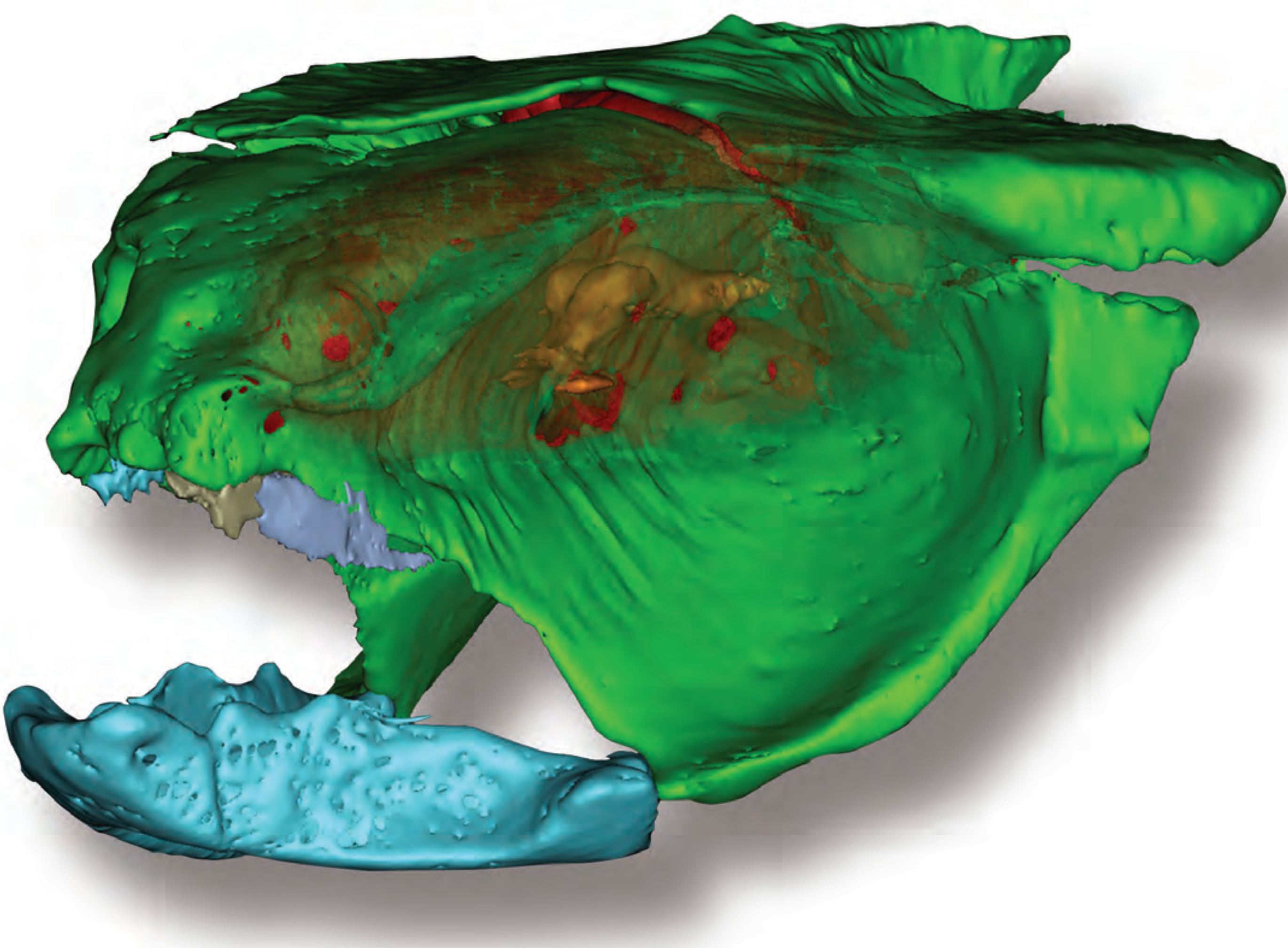
A LIGHT FOR SCIENCE

# ESRF news

Number 58 June 2011



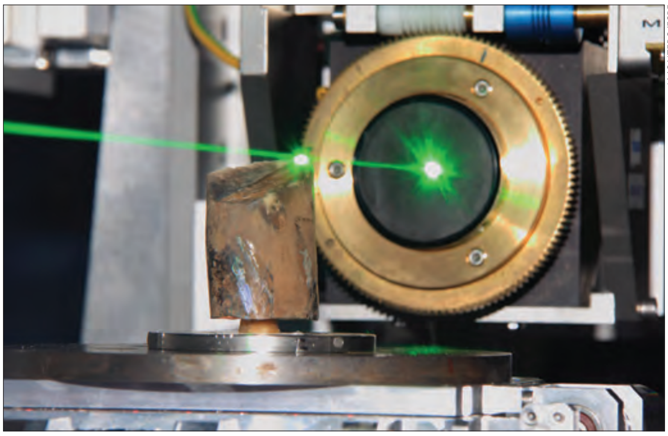
## Synchrotron culture: fossils to fine art

**Mimicking the moves of the Chinese silkworm**

**Pascal Elleaume: 8 January 1956 – 19 March 2011**

ESRF

# A light for science



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W BRAS

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A PRADEL (CNRS)

**On the cover:**
Oldest fossil brain revealed in a 300 million-year-old fish.

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ESRFnews

# Penetrating a new world



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Understanding the anatomy of ancient species has always been a dream of palaeontologists. Unravelling the ingredients and manufacturing methods of ancient materials requires non-destructive techniques to preserve our cultural artefacts. In both cases, synchrotrons have proved powerful tools to study precious samples that are testimony to nearly 600 million years of evolution on Earth.

**"Synchrotrons have proved vital tools to study precious samples that are testimony to nearly 600 million years of evolution on Earth"**

In the past decade, palaeontological research at synchrotrons has produced breakthrough results and has become a major topic at the ESRF. Our success stems from the unique imaging capabilities of third-generation X-ray beams (high-flux, coherence and high-energy monochromaticity) combined with in-house expertise in palaeontology. The first ESRF palaeontology publication, a 3D analysis of enamel in primate teeth, opened the door to numerous other studies, including insects in amber, charophytes, dental development and bone histology. Subsequent developments using high-energy local phase contrast micro-tomography paved the way to the complete scan of the 7.5 million-year-old Toumaï skull at a resolution of 45 μm, and to the recent mapping of the entire skull of a new *Australopithecus* species that lived 1.9 million years ago in what is now South Africa. New techniques, such as grating interferometry, long propagation phase contrast imaging and improvements in large-fossil imaging possibilities are producing extremely promising results. Diffraction contrast tomography, for instance, is allowing the study of biomineralisation patterns with large crystalline grains, which occur in trilobite eyes or echinoderm endoskeletons. Whole organisms can now be swept plane by plane at sub-micron resolution to produce virtual images in 3D. Together with sophisticated data processing, X-rays are driving a revolution in the field of palaeontology.

X-ray microanalyses can also be applied to nearly all kinds of materials found in museums, from hard materials such as metals to soft ones such as paper, and also hybrid materials, such as paintings and bones. As a result, synchrotrons are impacting the fields of art, archaeometry and conservation science. By identifying the ingredients and chemical reactions involved in tiny, heterogeneous samples of cave paintings or ancient cosmetic and pharmaceutical products, ESRF users can both unveil our cultural origins and understand the dynamic changes that cause artefacts to degrade. Techniques including tomography, fluorescence, absorption spectroscopy and diffraction can be combined for 2D or 3D microanalysis of the same sample, while synchrotron-based Fourier transform infrared micro-spectroscopy is a powerful emerging technique for cultural-heritage science. Algorithms are key factors when interpreting experimental data both at synchrotron sources and on portable X-ray instruments, which have been used to analyse the recipe used by da Vinci to paint shadows on faces in his quest towards making his art look alive.

This issue of *ESRFnews* illustrates the great impact and potential of cultural-heritage and palaeontology studies at the ESRF. It shows how the most recent successes come not only from unique ways to provide high-quality images collected from non-destructive investigations, but also from the know-how of beamline scientists and the complex processing tools that they have developed. The gap between large-scale synchrotron facilities and the cultural-heritage community is being bridged, with specialists from both sides transferring technologies. Studying priceless collections of cultural and fossil artefacts in depth while preserving them for investigation by future generations has become a reality that illustrates the impact of the ESRF to a large audience.

**Serge Pérez**, *director of research, ESRF*

**X-rays on the rise: 33-day record.**

## ESRF smashes previous record

On 16 March, the ESRF set a new record of 795.5 hours for continuous storage ring operation – breaking its previous record of non-stop delivery dating from more than two years ago. The delivery was interrupted only by the dedicated machine time, which occurs one day per week, and the 33-day period may have gone on longer had it not been for the start of the scheduled March shutdown. "This record illustrates the continuous efforts of the ESRF to maintain and develop the accelerator complex," says Operation Group manager Jean-Luc Revol. "It was also achieved during a period when the ESRF upgrade programme was being practically implemented." Uninterrupted X-ray delivery is important not only in terms of availably to users but also for the stability and quality of the experiments. During the last two weeks of the record, the machine was operated in 16-bunch mode, which produces a stroboscopic beam specifically for time-resolved experiments.

# The state of things to come

An important step in the ESRF upgrade took place on 20 April with the arrival of the first solid-state radio-frequency amplifier – one of 14 such units due to be installed over the next year to help guarantee the ESRF's long-term performance. Radio-frequency (RF) amplifiers feed the cavities that accelerate electrons, and traditionally take the form of high-power klystrons. High-power solid-state amplifiers combine hundreds of transistor amplifier modules, and the units are extremely failsafe because they can function even if some of the individual modules fail.

Due to losses in cavities and, of course, the emission of synchrotron radiation, RF power must continually be supplied to the ESRF's beam. The Linac and booster are operated up to six times per day to top up the electron beam current, and the solid-state amplifier is expected to reduce electrical power consumption in the booster RF system from about 1200 kW to 350 kW when operated in nominal pulsed mode.

Within 24 hours of its delivery at the ESRF, the 75 kW solid-state tower was hooked up to a test load and demonstrated a DC to RF conversion efficiency of nearly 60% – above the specified 55%. A further 13 such towers will be delivered over the next year and combined in pairs: the first eight will be connected to the booster cavities during the coming long winter shutdown, while the rest will help evaluate solid-state technology on the storage ring. The ESRF's new amplifiers are delivered by ELTA/AREVA and the design comes from SOLEIL, where its elementary building blocks were qualified.

"For applications similar to the ESRF, the future very likely belongs to solid-state technology," says Jörn Jacob, head of the ESRF's RF group.

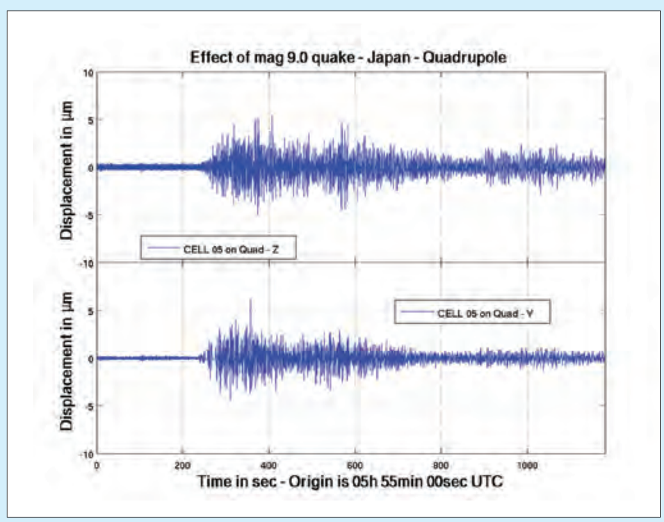

J.JACOB, ESRF

**The ESRF's first solid-state radio-frequency amplifier.**

## ESRF senses earthquake in Japan

Roughly 15 minutes after the devastating earthquake off the coast of Japan took place on 11 March, the ESRF recorded significant vibrations on the floor in the technical gallery due to the arrival of high-velocity "P waves". Vertical (top) and horizontal (bottom) displacement levels measured on CELL 05 quadrupoles in the storage ring revealed the presence of large low-frequency amplitudes during the earthquake. The main event lasted about 500 seconds and peak-to-peak vibrations reached 10 µm, compared with a background of 1 µm typically observed in the morning at the ESRF. The magnitude 9.0 earthquake took place at a depth of 32 km near the east coast of Honshu on 11 March at 06:46:23 local time in Grenoble.

Japan's Photon Factory at the KEK laboratory in Tsukuba suffered extensive damage during the earthquake despite being located 300 km away from the epicentre. Some RF cavities at the facility moved by up to 10 cm in the beam direction, several vacuum bellows were broken and many control cabinets and other instruments fell out of their normal positions. Differences in the floor levels were also observed. Fortunately, user operation had finished on the morning of 11 March and nobody at the facility was injured. "We are hoping to be able to accept general users in this fall at the latest," said director of the Photon Factory Soichi Wakatsuki in a statement on 28 April. The UK's Diamond source also registered the Japan quake.



# Cerium enters a rare and spectacular phase

As a solid is subjected to higher pressures, its atomic arrangement changes. For most elements the transition is accompanied by a decrease in volume, which means that a single crystal crushes into a poly-crystal or a powder. But cerium, the most abundant rare-earth element with applications ranging from catalysts to fluorescent lamps, does not conform to this picture. At a temperature of 300 K and pressure of 0.75 GPa, the volume of cerium decreases without any accompanying change in its structure.

ESRF users have now shed light on the mechanism behind this counter-intuitive transition. Frédéric Decremps of the Université Pierre et Marie Curie (IMPMC) and co-workers from the IMPMC, Lawrence Livermore National Laboratory and the CEA took the world's only existing single crystal of cerium to the ESRF's ID09 beamline, where X-ray diffraction experiments revealed its non-equilibrium phase diagram.


T GRAY/PERIODICTABLE.COM

**Rare-earth element: cerium.**

When the sample was subjected to high pressures in a diamond anvil cell, it underwent a transition that has strong analogies with the liquid-gas transition of classical systems – a phenomenon that has never been observed in any solid. The initial phase of cerium transforms to a new phase with about 15% less volume. Yet the team observed that the single-crystal quality remains and, more surprisingly, two single crystals with different volumes coexist. The researchers also determined the high-pressure variation of volume in a single crystal of cerium along different isotherms. "This a major first in crystallography," Decremps told *ESRFnews*.

In 2009, Decremps and co-workers showed that the variation of volume during the phase transition is provoked by a mechanical anomaly: the more solid cerium is compressed, the more it becomes compressible – behaviour that had never been observed in a pure element. The new work sheds light on transformations generated by the delocalisation of f-electrons, which could be the cause of the exceptional mechanical properties of cerium-based metallic glasses. These have promising technological applications because they deform plastically at almost ambient temperatures.

"A bright and small X-ray beam was vital to obtain high-quality data on cerium's equation of state at high pressure and high temperature," says Decremps. "This experiment is feasible only at the ESRF on beamline ID09, which is partly devoted to high-pressure high-temperature experiments and has enough flux to collect excellent data on a small area of the sample."

**Reference**

F Decremps *et al.* 2011 *Phys. Rev. Lett.* **106** 65701.

## Users' corner

At the last proposal submission deadline on 1 March, 1088 new proposals were received. Fewer Long Term Project applications were received than usual, which is likely to be the result of disruption to available beam time on numerous beamlines linked with on-going and imminent upgrade programme work, although a record number of standard and structural biology proposals were received.

This high number of proposals coincides with a record low amount of available beam time since the long shutdown of ESRF, from 5 December until 3 May 2012, means that only 60–70% of the usual beam time will be available for allocation in the scheduling periods 2011/II and 2012/I. Hence competition for beam time will be even fiercer than usual.

The next deadline for submission of standard proposals is 1 September, for beam time during the period May–July 2012.

### News from the beamlines

• The new **ID24** sector will host two complementary energy dispersive XAS spectrometers (EDXAS_S and EDXAS_L), which will use beam on a time-shared basis. EDXAS_S will feature a small focal spot, of the order of 3 microns FWHM in both directions, optimised for studies of matter at extreme conditions. An "in-situ" laser heating facility will be available for studies of matter at very high pressures (P > 100 GPa) and temperatures (T > 3000 K) using diamond anvil cells. EDXAS_L will offer a focal spot of variable size, from 10–200 microns FWHM.

The experimental station will be equipped for studies of magnetism under very high-pulsed magnetic fields and for time-resolved XAS investigations in materials science, chemistry and catalysis. Synchronous EXAFS and IR spectroscopy coupled to mass spectrometry on the millisecond timescale will be available, providing the ideal platform for understanding the structure–function relationship around the active catalytic site.

• A detector upgrade on the Swiss–Norwegian beamline **BM01A** was carried out at the end of March. The existing CCD detector of type Onyx from Oxford Diffraction Ltd (now part of Agilent Technologies) has been replaced by a Titan detector supplied by the same company. The new detector has the same physical dimensions (i.e. 165 mm diameter), but has a read-out cycle that is about four times faster. Typical data-collection times will be reduced by at least a factor of two. In addition, this latest generation of CCD detectors has a much improved dynamic range.

The new detector was operational within a few hours of installation and has already been used by several external groups, including scientists from the Department of Materials of the ETH Zurich and from the Laboratory for Neutron Scattering at the Paul-Scherrer Institute. The enhanced performance of the new CCD detector will provide opportunities to improve both the quantity and quality of the diffraction data from BM01A.

• The **ID14-3** bio-SAXS beamline, which is a part of the ESRF upgrade programme, has now been confirmed as operational for users until December, at which point bio-SAXS will move to **BM29** with first experiments scheduled for June 2012 (just after the long ESRF winter shutdown). In related news, the increasing level of automation of the beamline has enabled the first successful service experiments for industrial customers (carried out on 18 April).

• The French **CRG D2AM (BM02)** is currently being upgraded to improve its potential for anomalous material studies. The beamline was closed to users from March to June for an experimental hutch extension (now 3 m longer), which will allow it to house the new 6-circle diffractometer in kappa geometry (from Newport, with installation scheduled for April 2012), and an enlarged SAXS table. A new monochromator, designed for spectroscopy, is also under construction and will be installed on the beamline during the winter shutdown. The programme will be completed with new mirrors.

• The German CRG Rossendorf beamline **ROBL** on **BM20** will be closed for a major optics upgrade from the end of July until November. This is immediately followed by the long ESRF shutdown. During this period, the beamline will not be available for user experiments.

# Illuminating the past

Fossils and artefacts offer rare glimpses of our evolutionary and cultural origins. The ESRF brings them to life at the micro-scale and is tooling up for a second decade of success.

## Open-access fossils: byte-sized information

Micro-computed tomography scans allow internal structures and 3D morphologies to be viewed at increasingly finer resolutions, and they are the reason why palaeontology consumes over half of the ESRF's computing resources. In 2009, the ESRF launched a free online database of fossil scans that have been carried out there, limiting repetitive use of fragile specimens and promoting scholarly exchange.

According to the ESRF's Paul Tafforeau, who developed the open-access database, such digital data may represent the primary evidence of new species, given the ESRF discovery of new fossil insects in opaque amber. Select scans of these weird creatures can be viewed, as can prehistoric embryos and in April this year a new paleoanthropology section was launched containing scans of hominin dental remains.

Negotiations are currently under way to add scans performed at the ESRF in collaboration with other scholars and repositories.

Check out your ancestors' teeth at http://paleo.esrf.eu.

P TAFFOREAU/ESRF

**Microtomographic data of *Homo sapiens* fossil remains, now available to view online.**

Paul Tafforeau opens a black, foam-padded flight case and retrieves a heavy stone cylinder about 30 cm long. It looks like a log of solid rock, but it's probably the only fossil of its kind in the world. On screen in the experimental hutch at the ESRF's ID19 beamline, Tafforeau pulls up a preliminary 3D image of what's inside: a stunning picture of two complete, interwoven skeletons from different creatures. What happened 250 million years ago that caused this pair, one terrestrial and one aquatic, to die in each other's limbs? He can't say too much yet.

Ancient objects have long been subjected to laboratory analysis by scientists and historians striving to identify our ancestors through fossils or uncover ancient trade routes through artefacts. The value of synchrotrons to archeometry wasn't noticed until 1986 but they, and the ESRF in particular, are now routinely producing scientific papers in top journals (pp12–13). Traditionally, fossils were studied by slicing up them up with a diamond saw. But whole fossils can now be placed directly in the ID19 or ID17 beamlines and subjected to non-invasive techniques that yield remarkable 3D images. Similar micro-scale techniques reveal the chemical composition of cultural artefacts, how they were made and how we can preserve them.

Cultural-heritage research is good for the ESRF's public profile – and for Europe, where annual income from heritage is estimated to generate over €300 bn in trade and services.

### Spinning out

Mathematical and experimental tricks that allow fossils up to 20 cm across to be imaged with a relatively low-energy beam have benefited materials science and recently boosted industry use of the ID19 beamline. The ageing of cultural artefacts, meanwhile, gives access to timescales vastly longer than any laboratory can offer. Studying the corrosion of very old metal-based artefacts, for instance, may help researchers to design containers that can store waste safely for thousands of years. Some leading glass manufacturers are interested in the methods used at the ESRF to study Egyptian and Roman glasses, the properties of which are governed by a sophisticated nanotechnology.

It wasn't envisaged that palaeontology or cultural heritage would become such a major part of the ESRF's portfolio, but a powerful model has been built by matching state-of-the-art beamlines with high-level, in-house expertise, and other facilities are following suit. "SOLEIL has gone as far as building a dedicated institute for cultural heritage and palaeontology, although the ESRF currently has more activity in this area than anyone else," says ESRF's head of instrumentation and services Jean Susini.

ID19 is to be refurbished with even better optical components to improve beam quality and manipulation, while the biomedical beamline ID17 where large fossils such as hominid skulls have been imaged will also be upgraded with a new sample stage and detector dedicated to high-quality microtomography. There are plans for a dedicated palaeontology suite in the ESRF's main building plus a new computing facility to speed up tomography reconstruction, and this year hominid fossil images were added to the ESRF's open-access fossil-image database (see above).

Several beamlines are already well equipped for cultural-heritage studies, offering complimentary techniques from imaging to high-energy diffraction and X-ray spectroscopy. Because cultural heritage specialists rarely interact with synchrotron scientists, it can be difficult for less familiar methods such as X-ray absorption spectroscopy to be adopted (p10). "Existing resolutions of 1 micron – 100 nm is likely the right scale because it targets individual layers in paintings or crystals in glass," says Susini. "But specific areas will be helped by the development of nanobeams at the ESRF."

As *ESRFnews* went to press, ceramics from the Louvre and da Vinci's artistic techniques were among proposals lining up for ESRF beam time. These and the mysteries of Tafforeau's tandem fossil will no doubt soon be on a news stand near you.

*Matthew Chalmers*

NRICP, TOKYO

**The ESRF shows Bamiyan cave paintings made of oil, hundreds of years before the technique was supposedly invented in Europe.**

# Synchrotron culture

Studies of cultural artefacts are not just about pretty pictures and press releases, explains *Marine Cotte*, scientist in charge at ID21.

KRÖLLER MÜLLER MUSEUM

**In 2009, researchers used the ESRF to study cadmium sulphide yellow pigment from James Ensor's 1921 *Still Life With Cabbage*, which fades over time as small white globules appear on the paint surface (see bottom right).**

Most of us are familiar with the first X-ray image in medicine: Wilhelm Röntgen's wife's hand, complete with wedding ring, in 1895. However, few people know that when Röntgen first communicated the result, he also reported the first X-ray radiography of a painting – a piece of wood painted with lead white pigments. Three months later, a friend of Röntgen described the results: "In a wooden box the tablets containing metallic pigments, such as cinnabar, chromium yellow and Berlin blue can be separated from those tablets that do not contain metallic pigments, for example carmine and gamboges, with the cover closed." Subjecting paintings and other works of art to X-ray radiography caught on fast in the cultural-heritage community, and today synchrotrons are at the forefront of this powerful technique.

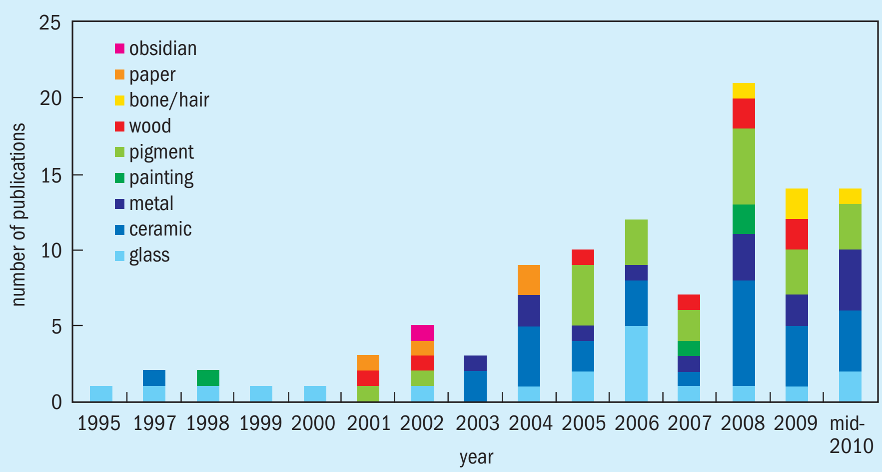
**Toxic revelation**

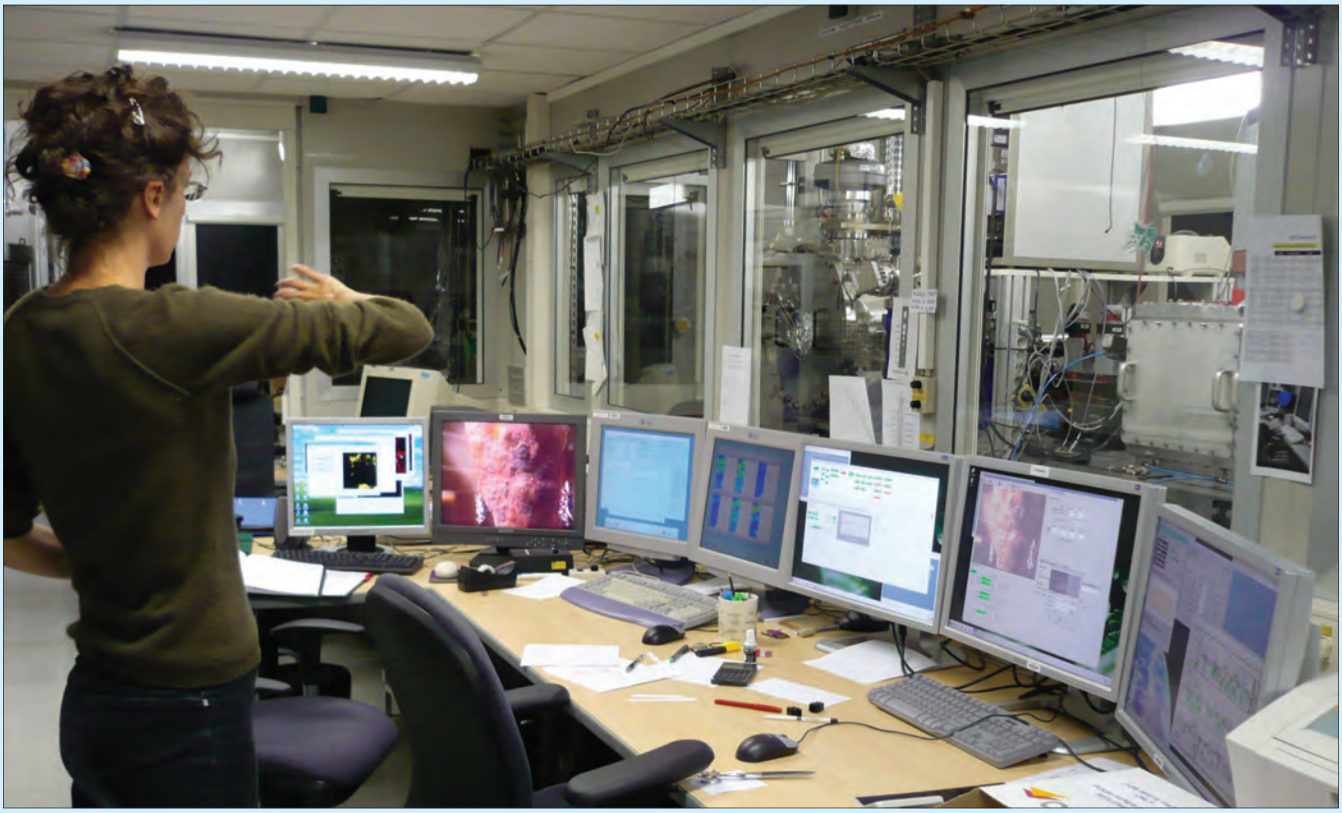
One criticism commonly associated with such studies, which recently include chemical analysis of Van Gogh paint samples at the ESRF, is that their impact is due to the beauty of objects rather than the scientific value of the work. Research on fascinating artefacts certainly appeals to a wide audience, but working on a famous work of art is not sufficient in itself to get beam time on the ESRF. The ESRF's Environmental and Cultural Heritage Science (EC) review panel is vigilant to the scientific value of the proposals. One can't underestimate the importance of such experiments for historical knowledge or for preserving artefacts for future generations. Besides its scientific relevance, the media interest increases the visibility of synchrotron facilities in general.

I encountered synchrotrons at the ESRF's ID22 beamline in 2001, while I was training as a chemist at the Centre of Research and Restoration of French Museums beneath the Louvre Museum in Paris. Our team studied mummy skins and also modern skins that had been treated with chemicals mimicking ancient cosmetics, which contained lead, mercury and arsenic – all of which are now known to be highly dangerous. Focusing on lead, we assessed the toxicity and studied the chemical reactions in these ancient practices by combining infrared micro-spectroscopy with X-ray micro-fluorescence to follow drug penetration within the skin. The former was done at the LURE facility, while the latter was at ID21, but since 2004 it has been possible to combine both techniques at the ESRF.

## XAS on the rise

Most cultural-heritage studies employ X-ray fluorescence or diffraction, natural extensions of laboratory techniques to deliver information about local elemental composition and crystallographic phases. But X-ray absorption spectroscopy (XAS) is an underused tool that reveals the chemical state of a sample, allowing researchers to address its mode of manufacture or ways in which to preserve an artefact. XAS publications are on the rise, addressing a widening range of materials (right) but the technique is yet to be widely adopted by the cultural-heritage community. [Source: *Acc. Chem. Res.* **43** 706].





JAVIER CHILLIDA

# State of the art: Marine Cotte analyses degraded pigment at the ID21 beamline

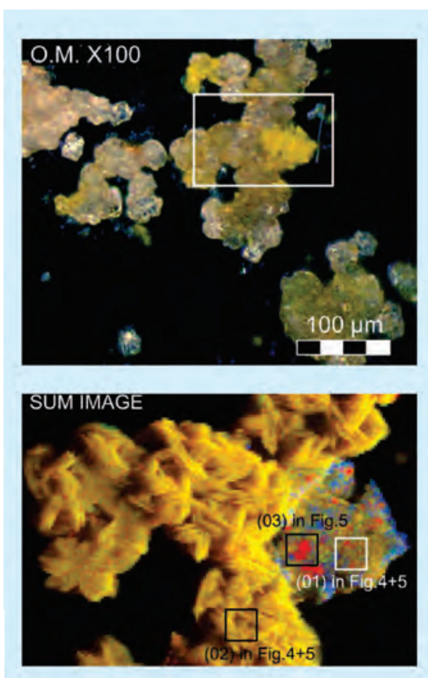
**A decade ago, studying art or ancient objects with synchrotrons was perceived as rather exotic, but in the past five years the ESRF has been at the forefront of a surge in such research. An important step was the establishment of the Environmental and Cultural Heritage Science (EC) review panel committee in 2005. More recently, France's SOLEIL source has established a cultural-heritage institute (IPANEMA) with a dedicated beamline (PUMA), which is currently under construction, demonstrating a reciprocal interest between synchrotron and cultural-heritage communities. Dedicated international conferences, such as Synchrotron Radiation for Art and Archaeology, which started at Grenoble in 2005, are also driving momentum. Synchrotrons including the ESRF, SOLEIL, ELETTRA, PETRAIII, Spring8, DIAMOND and the Australian Synchrotron advertise their cultural-heritage science on their web pages, seeing it as an ideal way to showcase the capabilities of new instruments.**

At that time there had already been some studies at ID21 into the degradation mechanisms in wooden warships or in metallic artefacts. But since then, particularly during the last five years, cultural-heritage studies have taken off at the ESRF and elsewhere (see above). My research has since shifted from cosmetics to paintings, but both fields are based on similar mixtures of pigments and binders, for which the mechanical properties are important.

**Broad impact**

The complex mixture of inorganic, organic and even hybrid materials in artistic materials drives innovative techniques that benefit the entire synchrotron community. An example is the fitting programme PyMca, which is not only used daily on many ESRF beamlines for X-ray fluorescence but also in the wider scientific community. The software was initially developed to get rid of complications caused by the interference of emission lines from light and heavy elements in X-ray fluorescence, which can determine the elemental composition of the complex mixtures and proved crucial in the analysis of the *sfumato* technique that da Vinci used for his *Mona Lisa*. More recently, ID21 staff have developed a wavelength-dispersive spectrometer that is ideal for such line separation. It already has several applications for analysis of ancient materials, and is another example of a technique driven by cultural heritage research that will benefit other fields.

A decade ago, the cultural heritage community viewed synchrotrons as inaccessible, even fearsome, entities. Step by step, researchers have realised that the techniques on offer, be they infrared imaging or X-ray diffraction, are natural extensions of familiar laboratory techniques. The next challenge is to make techniques that are more specific to synchrotrons (refer to graph), such as X-ray absorption spectroscopy, more accessible to the cultural-heritage community.




ANAL. CHEM. 81 2600

**Left: the composition of whitish/transparent globules that form on the surface of Ensor's painting (top) was revealed at the ESRF. µ-XRF was used to map the cadmium (red), sulphur (green) and lead (blue) in the white rectangle.**

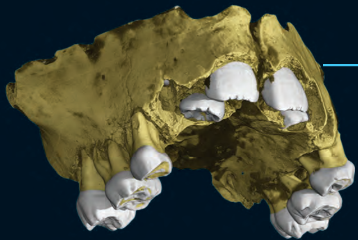
# Shedding light on the past

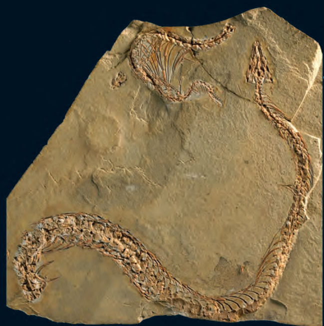
A selection of the ESRF's fossil and cultural hits

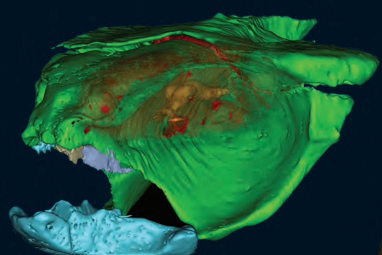
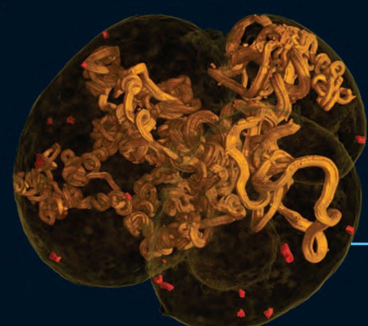
**TODAY**

| | |
|---|---|
| **1887** | Micro X-ray beams reveal subtle chemical processes that cause yellow pigment to turn brown (2011 *Anal. Chem.* **83** 1214) |
| **1884** | Pigment analysis of lost portrait beneath Van Gogh's *Patch of Grass* (2008 *Anal. Chem.* **80** 6436) |
| **1545** | Accumulation of sulphur compounds oxidising to sulphuric acid in wood from the *Mary Rose* (2005 *Proc. Natl. Acad. Sci.* **102** 14165) |
| **1450** | X-ray fluorescence reveals traces of mercury in the hair of Agnes Sorel, famous mistress of King Charles VII of France (2005) |
| **800** | Micro-diffraction shows degradation of Chinese silk fibres is due to loss of protein chains (2006 *Biomacromolecules* **7** 777) |
| **750** | Infrared spectroscopy reveals use of oil in paintings found in Bamiyan caves in Afghanistan (2008 *J. Anal. At. Spectrom.* **23** 820) |
| **500** | Diffraction reveals secrets of longevity of Maya blue pigment (2006 *Journals of Materials Science* **44** 5524) |
| **0** | Sulphur and chlorine analysis from Pompeii frescos explains why red pigment turns to black (2006 *Anal. Chem.* **78** 7484) |
| **150 BC** | Studies of fibres and pigments in Dead Sea Scrolls helps date sacred texts (2010) |
| **1500 BC** | XANES studies of Egyptian glass reveal secrets of opacification (2009 *Appl. Phys. A* **98** 1) |
| **2000 BC** | Egyptian make-up reveals wet chemistry has been practised for thousands of years (2001 *Nucl. Instrum. Methods Phys. Res., Sect. B* **181** 744) |

## MILLION YEARS AGO





**0.1 m** — Microtomography shows Neanderthal teeth grew faster than human ones (2010 *Proc. Natl. Acad. Sci.* **107** 20923)

**1.9 m** — 3D imaging of rare hominid fossil *Australopithecus sediba* reveals new human ancestor (2011)

**7 m** — Complete imaging of our earliest pre-human ancestor – the Toumaï skull – the first synchrotron image of whole fossil hominid skull (2006)

**12 m** — First published picture of a fossil using synchrotron microtomography: lower molar of a close relative to orangutan (2003 *Nature* **422** 61)

**65 m** — Microtomography resolves jaws of last ammonites, bringing clues about their extinction (2011 *Science* **331** 7)

**95 m** — Laminography of snake with legs offers clues as to whether snakes evolved from land or sea creatures (2011 *J. Vert. Paleontol.* **31** 1)

**100 m** — 3D structure of 356 animal inclusions revealed in 2 kg of opaque amber (2008 *Microsc. Microanal.* **14** 251)

Holotomography reveals oldest evidence of reproduction using sperm in microscopic bivalve crustaceans (2009 *Science* **324** 1535)

**300 m** — Oldest and only fossil brain revealed in an ancient shark-like creature by holotomography (2009 *Proc. Natl. Acad. Sci.* **106** 5224)

**400 m** — Internal structures of fossil algae revealed with 3D phase-contrast microtomography (2005 *Am. J. Bot.* **92** 1152)

**590 m** — Sub-micron images of microfossils at early developmental stages show complex embryonic development (2006 *Science* **312** 1644)

Image credits (top to bottom): Van Gogh Museum, Amsterdam; Mary Rose Trust; Royal Museum of Fine Arts, Antwerp; American Chemical Society; C Reyes-Valerio; D Bagault C2RMF; P Tafforeau, ESRF; P Tafforeau, ESRF; A Houssaye; P Tafforeau, ESRF; A Pradel/CNRS; P Tafforeau, ESRF.

# How a scientist cut his teeth


Paul Tafforeau focuses in on a fragment of an uncoiled ammonite's body chamber.

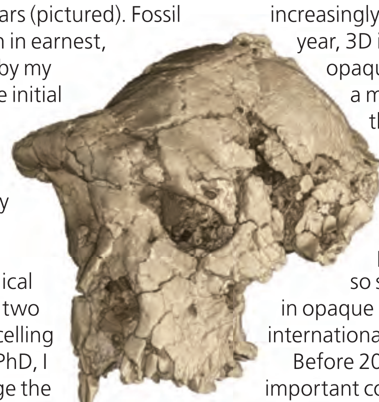
*Paul Tafforeau*, beamline scientist on ID19, reveals how within a decade the ESRF became the world's finest fossil photobooth. It began with a broken jaw at the turn of the millennium…

I was studying the dental structures of primates at the University of Montpellier II at the time. One day my PhD supervisor gave me a box containing beautiful primate jaws and told me that I could cut and polish whatever I wished to carry out my research. Having done so, I was then asked to return the fossils to the collection responsible. To my horror, and due to a misunderstanding between my supervisor and the collector, it turned out that they weren't meant to be cut. I had destroyed the teeth in 10 almost complete jaws that had been gathered over a period of decades, and vowed to never again use destructive techniques to investigate an important fossil.

After trying other techniques, I heard about the ESRF from a regular ID19 user at INSA in Lyon. At the end of 2000, he agreed to perform a test for me during one of his own experiments to see whether microtomography could resolve the microstructure of dental enamel. Afterwards, José Baruchel, head of the ESRF imaging group, helped me to prepare what was probably the first-ever proposal to scan fossils at a synchrotron, and the subsequent study into the 3D structures of modern and primate teeth was so successful that others followed rapidly. The ID19 team started to give me the beam time that nobody wanted, the isolated nights during weekends and vacations, and it soon became clear that synchrotron imaging held huge potential for palaeontology. I invited colleagues to collaborate or loan samples, and almost every time results exceeded expectations.

**Going large**

In 2003, a need to image larger fossils brought the lower jaws of fossil apes (hominoid primates) to ID17. Soon afterwards came a proposal to perform the complete imaging of the oldest known hominid skull, *Sahelanthropus tchadensis* (Toumaï) from Chad aged 7 million years (pictured). Fossil imaging on ID17 began in earnest, although as predicted by my physicist colleagues the initial experiment on the skull failed due to the lack of penetration of X-rays through this very dense fossil.




After three years of regular tests and technical advances on ID19, and two years since almost cancelling the original aim of my PhD, I was finally able to image the microstructure of primate dental enamel without resorting to cutting tools. By that time, at the end of 2003, we had performed 13 experiments on ID19 and ID17 plus a few on BM5, including primate teeth, fossil algae and the first imaging of fossil insects in amber.

It was then that the ESRF's management decided that synchrotron studies in palaeontology were of major importance, and in 2004 I began a one-year position as junior scientist that was opened in the imaging group. Constant exchanges about tomography development soon revealed the machine's potential, and new beamline equipment and processing tools installed for material sciences, biology and industry were increasingly tested on fossils. During that year, 3D imaging of fossil inclusions in opaque amber also took off, with a masters student attached to the project followed by a PhD and later a postdoc position. Although the vast majority of amber is studied using photonic devices, the ESRF's phase contrast technique was so successful in revealing inclusions in opaque amber that it became an international hub for this activity.

Before 2004 was over, a long and important collaborative project was established to study the dental development and dental structures of fossil humans and apes – some images of which have recently been added to the ESRF's open-access database of fossil scans. At that time, each scan took several hours followed by at least three weeks of manual processing, and the failure level was around 80%. Nowadays, scans still take still several hours but for an imaged volume eight times larger, while processing takes around a week and the failure level has halved.

**"I started to invite colleagues to collaborate or loan samples, and each time the results exceeded expectations"**

**Going public**

In 2005, palaeontology was officially represented at the ESRF panels with the creation of the Environment and Cultural Heritage committee, and activity shifted up a gear. The next phase was driven by imaging large fossils at ID17, where the failure of the first experiment on Toumaï had prompted intensive research to allow high-quality imaging of large fossils. Experimental and mathematical tricks saved the day, allowing the best pictures ever produced of fossils 10–20 cm in diameter with just 0.025% of the beam being transmitted through the sample versus a theoretical requirement of 10%.

The Poitiers palaeoanthropology laboratory then opened a two-year position to allow the complete scanning of the Toumaï skull, during which I was permanently attached to the ESRF. The result was the highest-quality scan of a complete fossil hominid skull and the work also drove new processing tools adapted to meet the high quality control of fossil imaging. Since that time, palaeontologists have developed their own specific methods, many of which have found use for other ID19 and ID17 users.

After the final Toumaï skull data had been reconstructed in late 2007, the ESRF opened a five-year scientific position for palaeontology within the imaging group. That position was made permanent in 2010, and a major refurbishment project of ID19 and ID17, strongly driven by palaeontological activities, was officially started. The construction phase of that project will start in a few months and will push much further the possibilities of non-destructive imaging on fossils, as well as benefiting many other user communities.

In just a decade, palaeontology at the ESRF grew from being a basic user study to a regular producer of high-impact publications that today takes up a quarter of ID19's activities and half of the entire laboratory's computing resources. There is currently only a single official palaeontologist at the ESRF, but through many collaborative projects with external institutes we now have two postdocs, one PhD researcher and one visiting scientist on ID19, with further positions planned for the near future. Thanks to the audacious decisions of management, along with the support of José Baruchel and many others, the ESRF became the world's leading institute for high-quality 3D imaging of fossils – all without breaking a single bone.

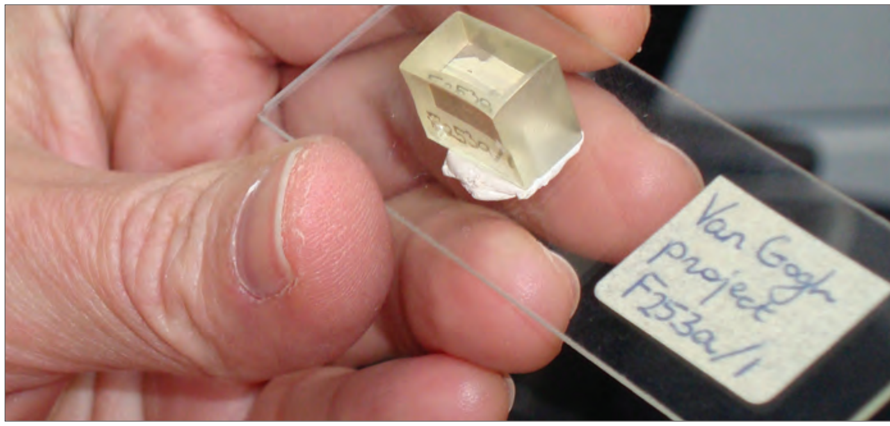

**X-ray microtomography reveals the jaws and teeth of 65 million-year-old ammonites (see left).**

# Future-proofing the past

Behind the scenes at the Van Gogh Museum in Amsterdam, *Matthew Chalmers* finds conservators juggling cutting-edge science and ethics to preserve our cultural heritage.

What would happen to a famous painting if curators were to suddenly withdraw all care and attention? How many generations would come and go before, say, Van Gogh's *Sunflowers* merged into a dirty smudge – and would that necessarily be a bad thing? These are among questions I put to Ella Hendriks at the Van Gogh Museum (VGM) in Amsterdam, where she is head of conservation. "I don't think that we can know how long a painting would last," she explains softly from behind her desk in a smart, carpeted office. "When do you decide that it's gone?"

Paint on a canvas may seem rock-hard and static, but in fact it is a highly mobile system even many decades after the paint has dried. Back in the 19th century, people were more interested in restoring paintings to their original condition, but today such intervention is deemed futile because curators know that paintings change from the moment that they leave the easel. "There's a lot going on in the paint layers," says Hendriks. "Today we try to preserve the equilibrium."




**Micro-art: a fleck of old paint from a Van Gogh canvas holds clues to conservation.**

### Synchrotron synergy

Synchrotrons are helping to elucidate these microscopic processes, which take place in layers too thin to be resolved easily by standard laboratory microscopes. The way that paint, particularly lake red, fades is one actively researched area. Last year, a microscopic fragment of paint from Van Gogh's *View of Arles with Irises* was subjected to X-ray diffraction at the ESRF, revealing the reaction in lead chromate responsible for the discolouration of yellow pigment under UV illumination – reinforcing the need to keep sunlight at bay. X-ray imaging at DESY in Hamburg had previously revealed a portrait beneath Van Gogh's *Patch of Grass*, property of the Kröller-Müller Museum in Otterlo, but this was the first collaboration between the VGM and the synchrotron community.

A deeper understanding of ageing processes raises questions about the extent that the knowledge should be used to preserve a painting. Take the climate cases used in the VGM to keep masterpieces in stable ambient conditions. Curators could remove all of the oxygen from the cases so that certain undesirable reactions in the paint stopped taking place, but the long-term consequences of this aren't known.

In fact, Hendriks says some of the problems that conservators are addressing now are due to the fact that paintings have been treated in the past. "People act with the best of intentions, but there is always a danger of over-reacting by applying new techniques that may have a detrimental long-term effect."

In the 19th century, one restorer thought he had found a way to get round the task of revitalising old varnish by holding a painting over a bathtub of alcoholic vapour. The varnish regenerated and the microcracks in it disappeared, as planned, but the vapours also softened up the paint layers and ultimately ruined the work. In the 1960s, scientists came

## Naked art's hidden layers

The work going on in museum research departments worldwide goes against the stereotype that art and science are opposites. Conservators have science as part of their training and act as a bridge between the art and other worlds. The fact that their work is now turning to synchrotrons is a natural step for conservators developing new ways to keep paintings intact.

Surveying visitor opinion, staff at the VGM found that the public want to know more about the conservation work and to get a peek behind the scenes. Having got up close and personal with an 1890 Van Gogh oil painting in a studio (pictured), I can strongly recommend it. As my eyes scanned the green and blue paint laid bare on a table without protective glass or frame, the peaks and troughs carved by the artist's individual strokes brought the canvas alive. A splatter of red blobs from a distance captures the essence of poppies swaying in the breeze.


**Palpable genius: Van Gogh's *Wheatfield under a Cloudy Sky* reveals its depth.**

"The difference between art and science is more about different languages, organisation and traditions, rather than the interest and curiosity of people involved," says Joris Dik of the Technical University of Delft in the Netherlands. "Art history has always been part of the humanities, and so is more aligned with literary techniques than archaeological ones, unlike palaeontology, which has traditionally been part of the sciences."

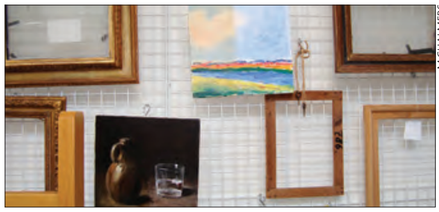
When big museums opened up research departments early last century, conservation science was born and in the late 1920s it proved its worth in the Netherlands during a court case concerning the famous "Wacker" series of Van Gogh forgeries. This was the first time that chemical analysis of paint samples and X-rays were used as evidence in a court trial, which had brought the art world and connoisseurship into disrepute.

up with synthetic varnishes that they claimed did not yellow over time, but it turned out that these new products were oblivious to some of the solvents that had been developed to remove varnishes. Even something as apparently innocuous as strapping batons to the back of a painted wooden panel to strengthen it can, given enough time, introduce forces that cause the panel to split.

As a conservator, Hendriks says that her job is to intervene in the artworks as little as possible, although she admits that the consequences of this minimalist approach are also unknown. Even handling an unvarnished canvas without gloves could leave her DNA on the surface, making it harder to identify that of the artist.

Besides, Van Gogh paintings were subjected to a rigorous campaign of treatment in 1926–1933. Most were varnished at this time, whereas the artist favoured a matt surface, and some were retouched by adding new paint. Many of the canvases have also suffered more serious structural effects due to wax-lining – the process of ironing a second canvas onto the back of a painting to make it more sturdy. Favoured until just a few decades ago, over time the wax becomes a permanent part of the structure that causes the painting to darken. Yet, Hendriks points out, nobody knows what condition the paintings would be in had they not been wax-lined.

A major appeal of an old painting, however, stems from the very things that mark its demise: networks of microcracks in the varnish, subtle browning and softening of colours, and even bulk damage that tells the story behind the work. Distinctive cracks in some Van Gogh paintings formed when he used to roll up a canvas and send it to his art-dealer brother in Paris, and Hendriks occasionally comes across one of the artist's hairs or a fingernail imprint while studying a specimen. Ageing is also an important mark of provenance and authenticity.



M CHALMERS

Tests show the dulling effect of paint varnish.

**Modern culture**

Conservators don't know what to expect from more modern works of art because there is no reference of ageing. "Freedom of expression has taken the place of craftmanship in some areas," says ESRF user Joris Dik of the Technical University of Delft in the Netherlands. "There is an artist who spreads peanut butter over the floor: how do you preserve that?"

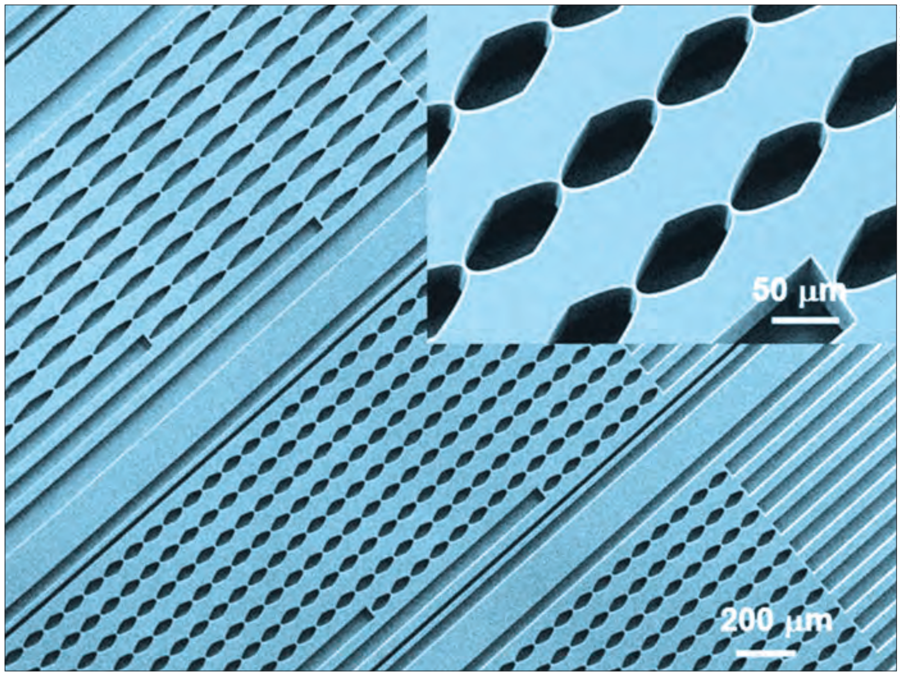
Dik, who's work scanning Van Gogh paintings with a mobile X-ray source helped lead to the recent collaboration between the VGM and the ESRF, envisages that paintings will one day be protected indefinitely by coating them with self-healing materials. "First we need to understand the degradation mechanisms, then the reaction kinetics, and finally what can be done to halt the deterioration."

Meanwhile, the conservation department at the VGM is pondering the effects of rock music on its 300 or so masterpieces due to open-air concerts on the nearby Museumplein. Hendriks is also working out how to deal with tiny white blobs produced when zinc white and lead white pigment particles turn into metal soaps over time. "There's still a gap between identifying the mechanisms and knowing how we should use that knowledge," says Hendriks. "There is a lot of constructive discussion between conservators, and approaches may vary in different countries."

As to why we should be preserving these splashes of pigment on pieces of cloth in the first place, Hendriks directs me to the 5–10 thousand people who visit the VGM every day. "I think Van Gogh has a special significance to many people," says Hendriks after a pause. "To each person it's different, but if you imagine it wasn't there, the world would be a much poorer place."

# The ESRF has the X factor

Demonstrated at the ESRF 15 years ago, refractive lenses are now operating at seven synchrotrons in five countries.

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**Rare refraction: unlike visible light, X-rays have a refractive index close to and slightly below unity, meaning that a large number of concave lenses is needed to focus an X-ray beam.**

Rainbows are the products of refraction, when the many wavelengths of sunlight are deflected at slightly different angles as a ray enters and exits a water droplet. A pair of refractive lenses positioned in front of the eyes corrects for impaired vision, and the refraction of radio waves by the upper atmosphere allows a short-wave radio transmission in Brazil to be picked up in China. This desirable property of electromagnetic radiation is virtually non-existent for X-rays, though. X-rays barely undergo reflection either, unless at very small angles. It's no wonder that Röentgen used an "X" to name the rays that he had stumbled across.

A century later, in 1996, X-ray refraction was demonstrated at the ESRF by Antatoly and Irina Snigirev, Victor Kohn of the Kurchatov Institute in Russia and Bruno Lengeler of the Institute of Physics at RWTH Aachen University. Refractive lenses for X-rays work like glass lenses for visible light, but have much larger curvatures and are made from low-Z materials, such as beryllium, carbon, aluminium and silicon to keep absorption to a minimum. Because the deflection of hard X-rays is very small, tens or hundreds of lenses have to be placed in series to obtain useful focal lengths, which can be adjusted by simply adding or removing individual lenses. Such a compound-tunable X-ray lens was installed at the ESRF's ID11 beamline in 2009.

**Focal point**

The original ESRF refractive lens in 1996 comprised 30 closely spaced, 0.6 mm diameter holes drilled into an aluminium block and was able to focus a 14 keV X-ray beam to a spot size of 8 microns. The following year, high heat load beryllium and aluminium lenses were installed at five beamlines to focus and collimate beams. Today, refractive optics are in place on half of the ESRF's beamlines and are in wide use at other synchrotrons, providing focal points ranging from a few millimetres to tens of metres and spot sizes ranging from tens of nanometres to tens of microns.

Without refractive lenses, the diffraction, fluorescence and imaging capabilities of the ESRF would not be as impressive as they are. More traditional diffractive optics, such as aperiodic gratings called Fresnel zone plates, are well developed for soft X-rays. However, adapting them for more energetic beams is difficult. "Refractive and diffractive optics are competing in microbeam focusing and imaging," says Anatoly Snigirev. "But Fresnel zone plates are limited to energies of 15 keV, whereas refractive lenses have an energy range from a few keV to hundreds of keV."

Since 2000 it has been possible to make composite refractive lenses from silicon. Microelectronics-fabrication technology allows deep vertical structures to be etched perfectly in a smooth surface, and is now being transferred to materials such as diamond that have low X-ray absorption and low thermal expansion. "Diamond lenses are a dream and the future, but still require some technology development," says Snigirev.

In 2009, Snigirev and co-workers from France and Russia proposed a new type of hard X-ray interferometer based on two parallel arrays of compound refractive lenses etched in silicon, allowing new ways to study advanced nanoscale materials, such as photonic crystals (Snigirev *et al.* 2009).

**"Today, refractive optics are in place on half of the ESRF's beamlines"**

Faults in self-assembled colloidal crystals that can seriously affect the optical properties of photonic crystals have already been revealed by the microfocusing capabilities of compound refractive lenses (Hilhorst *et al.* 2009), and more recently the Snigirevs, in collaboration with researchers at Moscow State University, demonstrated a high-resolution transmission X-ray microsope based on parabolic refractive lenses that is ideal for studying periodic mesoscopic structures (Bosak *et al.* 2010).

This year, parabolic refractive aluminium lenses were installed close to the source inside the ESRF storage ring to measure the vertical emittance, which is now routinely of the order of 3–4 pm and therefore approaching the diffraction limit of conventional optics. "The imaging resolution of refractive lenses is in theory better than that of pinhole cameras, and having two different emittance diagnostics systems allows a cross-check of the results," says Friederike Ewald of the Accelerator and Source Division.

Bruno Lengeler says that most textbooks in optics claim even now that there are no refractive lenses for X-rays. "The development in the past 15 years has clearly shown that this statement is far too pessimistic."

*Matthew Chalmers*

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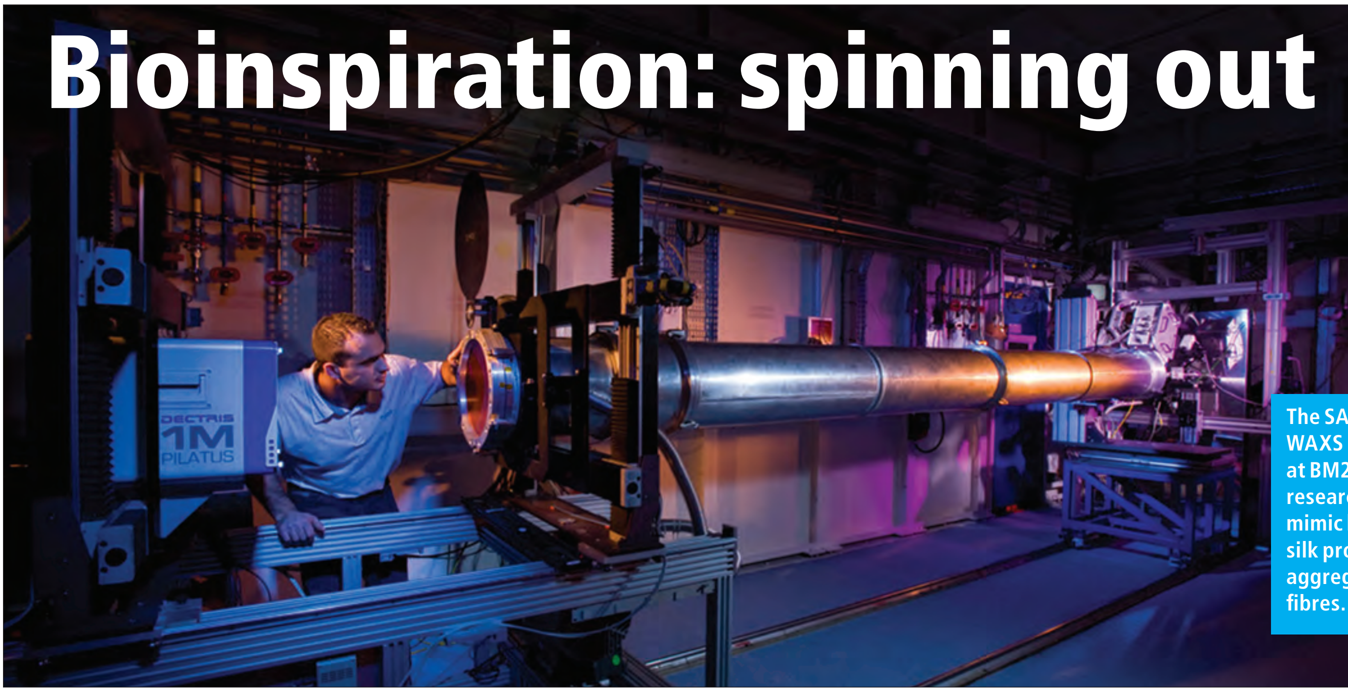
# Bioinspiration: spinning out




ARTECHNIQUE

The SAXS/WAXS set-up at BM26 helps researchers mimic how silk proteins aggregate into fibres.

**At the ESRF's DUBBLE beamline, researchers are investigating how silkworms transform a soft protein gel into a high-performance fibre, finds *Matthew Chalmers*.**

A few choreographed wiggles is all it takes for the Chinese silkworm *Bombyx mori* to produce one of nature's toughest materials: silk. To these small ivory caterpillars, it is the raw material for thumb-sized cocoons that they build for protection while undergoing metamorphosis into moths. To humans, who have been harvesting the cocoons for millennia, silk is the source of fine clothes and scientific wonder.

The high-performance properties of Dyneema and Kevlar, materials used in bulletproof vests, rely on the same kind of chemical and molecular tricks employed by silkworms or spiders. But nature manages to pull off its elegant manufacturing feat at room temperature with only water as a solvent, whereas industrial polymer processing requires high temperatures, pressures and polluting chemicals. By carefully reprocessing silk into forms other than fibres, it can even be used to develop artificial bone and tendon. Neural grafts made from pig veins filled with spider silk were recently shown to enhance the regeneration of nerves over distances of up to 6 cm (Radtke *et al.* 2011).

"We don't know exactly how the worms process their silk to make such high-performance fibres, although presumably they have to align the molecules to make them line up into a semi-crystalline structure," explains Wim Bras, project leader of the Dutch–Belgian DUBBLE beamlines at BM26, where the experiments were carried out. "If we could mimic what these little guys do, then not only do we understand more about biology but we can apply this knowledge to develop better materials and manufacturing processes,"

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**A silkworm visits the ESRF.**

says Bras from inside the DUBBLE experiment hutches where we met, his voice periodically drowned by a lathe machining sample environments for the next round of users.

### Dispatching caterpillars

In March, a team led by Chris Holland of the University of Oxford's Silk Group took silkworms to DUBBLE to study the mechanical forces necessary to produce this unique natural fibre. Three quarters of a silkworm's body is filled with a concentrated protein gel produced by its salivary glands, which is pulled through a specially shaped duct during which it aligns, dehydrates and aggregates into a fibre. Polymer processing uses the same principle: because polymers have a natural tendency to align given appropriate processing techniques, molten polymer can be moulded into cables stronger than steel by extruding it through a narrow die.

Microscopy tells researchers a lot about spider and silkworm spinning ducts, which are nature's equivalent of a polymer extrusion die. But it is the shear forces during spinning that hold the key to how silk molecules are coaxed into alignment and aggregation, giving silk its valuable mechanical properties. The molecules are so small that X-rays are required to resolve these structural changes, giving scientists clues that allow them to understand and perhaps replicate the spider and silkworm's genius.

The Oxford team placed drops of unspun silk gel in a shearing apparatus that can apply small forces in different relative directions, and determined the orientation of the molecules using X-ray fibre diffraction. Although nobody knows for sure what's going on *in vitro*, the gel can be sheared at different rates until orientation starts to appear. Combining SAXS and WAXS, the team was able to study the aggregation process at all relevant length scales, 1–100 nm, before returning to the UK to analyse the data.

Ten years ago, experiments at the ESRF revealed how the speed at which spiders spin silk determines the orientation and alignment of the silk proteins in the fibre (Riekel *et al.* 1999). The DUBBLE experiments take this research further upstream, quite literally, by understanding how the gel is spun inside the creature. "This is really the beginning of this research into natural silk processing," says Bras, who says that the idea for the collaboration emerged over a beer at a polymer conference in north-west England. "It's the sort of thing that people win an Ig Nobel prize for!"

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# Lighting the way

## The worldwide leadership of the ESRF owes much to the distinguished service of accelerator physicist **Pascal Elleaume**.

Pascal Elleaume was one of the rare people whose contributions have changed the face of synchrotron science. When he was hired as one of the ESRF's first young scientists in 1986, before the project was officially founded, Pascal had already worked six years at the French synchrotron radiation centre LURE in Orsay. He would go on to help demonstrate the qualitative difference between second- and third-generation synchrotron sources, which have become ubiquitous in modern science. For the past 10 years he was director of the ESRF's accelerator and source division, during which the performance of the machine – its brightness, reliability and availability – increased significantly.

A physicist and mathematician of rare value, Pascal devoted his scientific knowledge to the benefit of the ESRF and, through it, to the wider accelerator community. Some 20 synchrotrons have been designed since the ESRF started up, and almost all have used his advice and expertise. Yet he was not one to boast about his achievements.


C JARNIAS/C ARGOUD

**Pascal Elleaume: a physicist and mathematician of rare value.**

### Free-electron laser

Pascal Elleaume was born in 1956 and went on to become a brilliant student. At the age of 18 he entered one of the most prestigious French "grandes écoles" – the Ecole Normale Supérieure, rue d'Ulm in Paris. After graduating when he was 22, he began research into condensed-matter physics at the Paris VI University followed by a masters degree in statistics and probability at the University of California in Berkeley.

In 1980, Pascal was hired as an accelerator-and-laser physicist at LURE in Orsay. At the time, word was out that a new type of "free-electron laser" had been developed at Stanford University, and LURE researchers had decided to build such a device themselves. Initial calculations showed that it was possible but difficult, and Pascal was put to work on the problem. Although risky, Pascal decided to start a PhD on this subject, stating that the challenge was what made it interesting. His deep grasp of electrodynamics allowed him to redo the calculation quickly, and the outcome suggested that a new magnetic structure – an optical klystron – was required if lasing were to be achieved. Pascal played a leading role in bringing the project to fruition. Experiments were conducted two or three nights per week until 4.00 a.m., after which he would take a nap before presenting the results the following afternoon, complete with the theory to interpret them. Lasing was observed in 1983, and the following year he wrote a brilliant thesis presenting the pioneering observation and interpretation of free-electron laser radiation at the LURE. Pascal went on to characterise the first optical klystron using undulators at the Orsay laboratory. It was he, more than his supervisors, who directed himself to the work.

Two years after arriving in Grenoble, Pascal created the ESRF Insertion Devices Group, which has been responsible for the world's most successful design, construction and commissioning of undulators for synchrotron radiation. More than 100 beamline-tailored insertion devices, which can be independently set with minimal manual interference, are now operational on the ESRF's storage ring.

Pascal's mathematical prowess enabled him to devise many different types of undulators and wigglers that have augmented the development of synchrotrons. His inventiveness allowed him to develop devices whenever the need arose – such as the "pin-hole" cameras that you can find on most of today's storage rings to measure beam emittance. Pascal was also able to communicate his expertise. He initiated several freeware computer programs that model either the magnetic field, the radiation produced or the behaviour of the beam in insertions devices. His book on wigglers, undulators and their applications has become the standard text in the field, and he was invited on several occasions to give courses at the CERN accelerator school. He has supervised several PhD students and was always, though with characteristic modesty, willing to share his internationally recognised expertise with colleagues and other accelerator centres.

### No equals

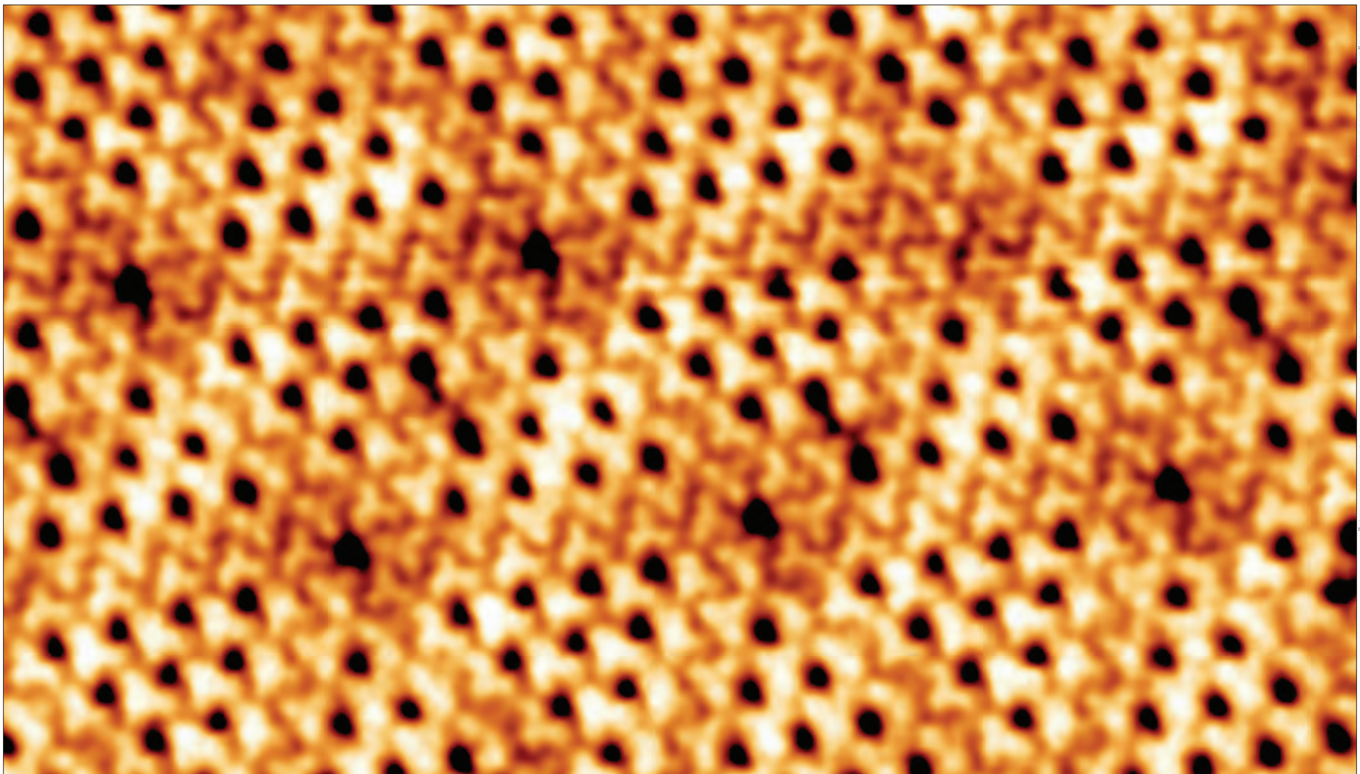
The ESRF upgrade programme is now in full swing, and the accelerator division is again setting the standards for the next 20 years in the field of synchrotron radiation. Major new technical developments are planned or already accomplished, such as new orbit feedback systems, sharply decreased vertical emittance, and new RF-powering systems. All of this stems from the many groups that have worked under Pascal's leadership, which are now able to guarantee even higher brightness, reliability and availability of the ESRF source.

Pascal wasn't one to put himself out in a crowd. He was more reserved and was careful not to encroach on the space of others at conferences, which gained him the respect of all. He was rigorous and demanding of himself, and when he agreed to be part of an international committee he took a great personal interest in carrying matters through. Those who have worked closely with Pascal during his 25 years at the ESRF also know that he always talked about his family with enthusiasm and pride. More recently he had rediscovered the pleasure of mountain trekking with family and friends.

Pascal has seen to it that our machine has no equals. His untimely death in an avalanche in the Alps is a tremendous loss for the ESRF, but we continue to follow the path that he laid out.

Pascal Elleaume (8 January 1956 – 19 March 2011) is survived by his wife Hélène and his children Nicolas, Olivier and Camille.

*Francesco Sette, Jean-Marc Filhol* and *Yves Petroff*



KRÜGER, HERMANNS, BERNIEN AND UMBACH, INSTITUT FUER EXPERIMENTALPHYSIK AT FREIE UNIVERSITÄT BERLIN

**Magnetic nano-triangles:** This raw scanning tunnelling microscopy (STM) image reveals a periodic pattern of triangular domains (side length 9 nm) in a 2D metal-organic network of iron atoms and organic nickel-based linkers, which was prepared by evaporation on a gold surface. The ability to image a sample at a beamline gives users vital information about its properties before X-ray studies are carried out. After being imaged, this sample was transferred under ultra-high vacuum from the STM to the ultra-high vacuum, high-field magnet at ID08, where X-ray magnetic circular dichroism revealed that the iron atoms retain their magnetic moments. Such magnetic molecular monolayers open the possibility of tuning the magnetic properties of metallic surfaces – one of the key issues for future device technology.

## In the corridors

### First-generation X

Researchers at Maastricht University Medical Centre in the Netherlands have dug out a 115-year-old apparatus used by X-ray pioneers Jozef Hoffmans and Lambertus van Kleef, and compared its imaging capabilities with those of a contemporary device. The pictures weren't bad but an image of the hand of a cadaver required a radiation dose 1500 times higher than modern equipment and an exposure time of 90 minutes versus 21 ms today. It's no wonder that the pioneers of X-ray imaging suffered burns, hair loss and vision problems. The results constitute the first ever diagnostics of a first-generation X-ray system, revealing a large focus, the emission of radiation with a soft component, and a low output (*Radiology* **259** 534).

RSNA

NASA

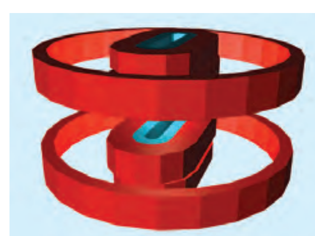
**Space Shuttle *Endeavour*.**

### Go for launch

When Space Shuttle *Endeavour* launched for the last time on 16 May, it was carrying a 7-tonne experiment that will search for the missing matter in the universe. The alpha magnetic spectrometer (AMS) was dropped off at the International Space Station, where it will track charged particles that constantly bombard our planet from outer space. Precise measurements of the cosmic-ray flux will allow a single antinucleus to be identified among a billion other particles, signalling the existence of large amounts of antimatter elsewhere in the universe. The detector, which is the work of a 600-strong international collaboration at the CERN laboratory near Geneva, may even help physicists track dark matter. "Never in the history of science have we been so aware of our ignorance," says AMS deputy spokesperson Roberto Battiston. "Today we know that we do not know anything about what makes up 95% of our universe." *Endeavour* also took the first cephalopods into space – baby squid that may tell biologists how bacteria behave in microgravity conditions.

### Super centenary

In April 1911, Dutch physicist Heike Kamerlingh Onnes discovered that mercury loses its electrical resistance when cooled to 4.2 K. Today, superconductors lie behind levitating trains, medical scanners and attempts to harness fusion energy. They can also be found in the wigglers and undulators of the ESRF. Despite its applications, though, superconductivity has eluded the best minds in science. It was 1957 when theorists finally accounted for Onnes' fortuitous breakthrough, but the discovery of high-temperature superconductivity in copper oxides 30 years later, with superconducting transition temperatures up to 93 K, shattered any illusion that superconductivity had been cracked. If history is anything to go by then the dream of a room-temperature superconductor may lie in good old experimental luck.



ESRF

**A superconducting 5.2 T wiggler.**

# So you want to communicate science?

## The ESRF's communication group is at your disposal, says *Matthew Chalmers*.

C ARGOUD, ESRF

**Claus Habfast explains the ESRF to members of *lightsources.org*.**

**Meet your audience**
- Explain to people why they should care about your research: the most important thing scientifically is not necessarily the most interesting thing to them.
- Recognise your knowledge: is someone, however educated, expected to know what words like "coherent" and "diffraction" mean?
- Occasionally escape the comfort zone of established knowledge: audiences want your vision of the future.
- Add colour: twists and turns within the research, surprises, mishaps, bruising night shifts…
- Keep peers out of mind: the better communicator you become, the more envious your colleagues will be.

Communication is the lifeblood of science, since claims to new knowledge are published in refereed journals. A century ago, anyone literate could pick up a scientific paper and understand roughly what it was about, but most scientific papers today are comprehensible to other specialists only. Engaging the people who pay for science and exist with its outcomes, the public, presents a unique challenge in the 21st century.

So what should scientists do if they think their research merits broader attention? Outreach activities range from giving public talks to writing books and presenting your own television series, and can add a rewarding dimension to a research career. Many funding agencies expect researchers to devote some of their time to it, and may offer training in how to deal with the media. Some scientists cross over entirely to communication roles – an ideal way to stay up close to science without having to specialise in a discipline, for instance. The bravest science communicators may become school teachers.

Head of the ESRF communication group, Claus Habfast, completed a PhD in accelerator physics at CERN. Today, he and others in a team of five give nearly 500 journalists advanced notice of important results from the ESRF beamlines, and prepare text, quotes and visuals to make it easier for journalists to write the story. Press releases are how most science stories get into the media these days, often under embargo, although journalists receive so many that most never see the light of day – especially when their competitors are probably on the same mailing lists.

"Synchrotrons struggle to communicate because it can be difficult to get hold of the information and distribute it when users are from so many different countries," says Habfast. The ESRF also covers a wide range of science: a press release about a fundamental physics or materials study is less likely to attract a news editor than analyses of an important fossil or discovery of a drug target, so special efforts are required. "A study into the phase diagram of lithium at high pressures can be made more relevant simply by stating that lithium is inside mobile-phone batteries. Images are also vital," says press officer Montserrat Capellas, "especially at the ESRF where experiments produce pictures that resemble identifiable objects." By letting the communication group know about an experiment in advance, it allows time to produce media material. If a journalist then calls you up to find out more about your research, it helps to have prepared an answer to the "so what?" question (see left).

The internet is changing the way that science is communicated at all levels, and blogs are an example of how scientists can bypass traditional media and go direct to the public. Science blogs don't have the audience of a mainstream newspaper or magazine website, but they can road-test your communication skills and some have been turned into successful books. If there's a strong visual aspect to your research, and cameras don't fill you with fear, video clips can make a big impact when targeting online media. The communication group handles an increasing amount of video – recent hits including live silkworms (p19).

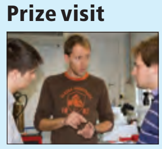
"The internet allows you to segment the communication more finely by target group," says Habfast, mentioning the ESRF's *Facebook* wall and *Twitter* feed for structural biology (tag @MXESRF). "With *lightsources.org*, all of the major synchrotrons work together to make the most of this – so when a publication is based on data from several synchrotrons, we try to issue identical releases rather than compete against each other."

# Movers and shakers

### Italy at the ESRF

On 8 April the ESRF hosted a delegation from Italy headed by Luciano Maiani, president of the funding agency Consiglio Nazionale delle Ricerche (CNR) and former director-general of CERN. In addition to being shown microscopic samples of Roman wall paintings that have been studied at the ID21 beamline, the delegation discussed ways in which to widen the benefits of the ESRF for Italy's academic and industry researchers – particularly in the geosciences, new materials and sustainable-energy research. Italian scientists make up nearly 13% of all ESRF users with an important community in solid-state and nuclear physics, and Maiani was pleased to learn that Italy contributes a third of the ESRF's PhD students. "Working together with Sincrotrone Elettra, its national light source, there is a bright future for synchrotron science in Italy," concludes Francesco Sette.

### Prize visit



The ESRF has received two winners of the 2010 European Union Contest for Young Scientists, who each won a one-week stay at the ESRF and ILL. The final stage of the contest, which took place last year in Lisbon, is a showcase of the best of European student scientific achievements. Radko Kotev from Bulgaria (left) and Sebastian Cincelli from Italy (right) had a full visit of the ESRF in March, during which they heard Jeroen Jacobs (centre) explain how high-pressure cells work. A winner of the 2008 EU contest, Gunnar Peng from Sweden, took up his prize at the ESRF at the end of March.