

# ESRF Newsletter

**Researchers study how ice  
melts in contact with soil**



European Synchrotron Radiation Facility

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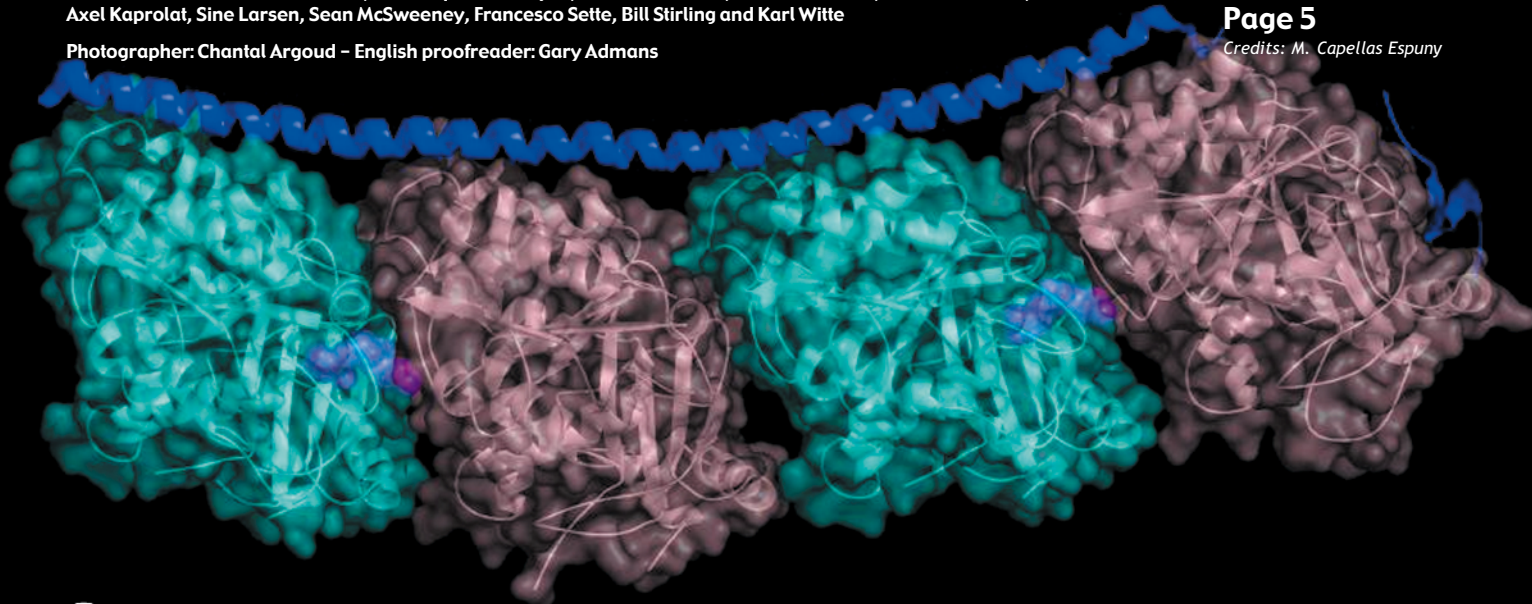
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Page 5**

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The tubulin RB3 complex. The complex includes two tubulin  $\alpha$ - $\beta$  heterodimers with colchicine bound to  $\beta$  subunits at the interface with  $\alpha$ . Page 16

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# Editorial

W.G. Stirling - Director General

## THE ESRF'S LONG TERM STRATEGY

Since the earliest days of the ESRF, forward planning has always been a high priority for the facility. The Foundation Phase Report of 1987 (the "Red Book") laid out a detailed plan for the development of the world's first insertion-device based synchrotron radiation source. This road map resulted in the first electron beam in the storage ring in 1992, the inauguration of the ESRF and the start of regular operation for Users from 1994 with initially ten operational beamlines, and, from 1999, operation with the full complement of thirty beamlines.

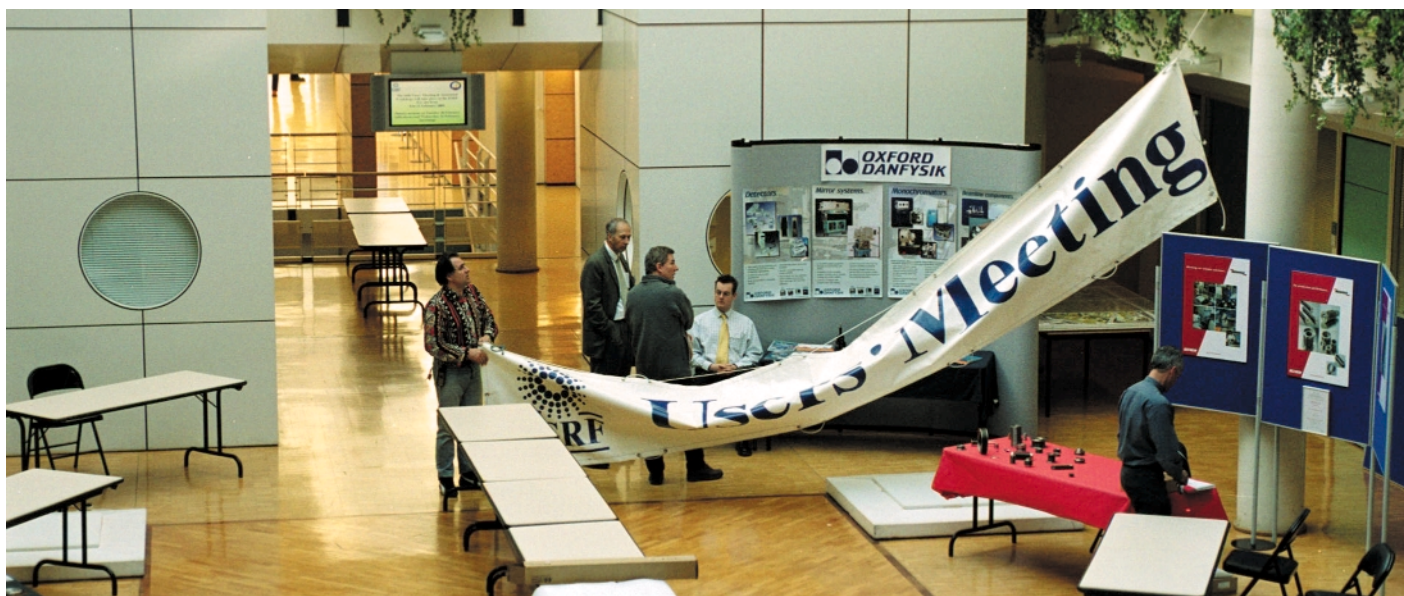
In the mid-nineties, well before the end of the construction period, a new long-term strategy for the period 1998

to 2007 was worked out and adopted in 1996. The objectives set at this time included a programme to maintain the X-ray source at its optimum capabilities and the refurbishment and enhancement of the beamlines. The importance of a lively internal scientific life at ESRF was also recognised.

Since then, within the framework of the long-term strategy, five-year Medium Term Scientific Plans (MTSP) have been updated each year, supported by a corresponding financial plan, the Medium Term Financial Estimates (MTFE). The MTSP and MTFE have been the framework within which source and beamline developments have been carried out over the last twelve years.

It is now time to consider the development of the ESRF on a time scale longer than that provided by the MTSP, taking account of the continuation or renewal of the ESRF's Convention after 2007. A draft Long Term Strategy (LTS) paper has been widely discussed - between ESRF Management and the Science Advisory Committee (SAC), and by the European scientific community - and was presented to the users during the Users' Meeting of 10 and 11 February 2004.

The photo shows the preparation of the Users' Meeting, which represented the start of the community-wide discussion of the LTS. The LTS considers future directions for the ESRF on the time scale of 10 to 20 years. An exciting



The preparation of the Users' Meeting.

and ambitious programme of new scientific themes - and the corresponding equipment requirements for X-ray source, beamlines and scientific infrastructure - is under consideration and will continue to be discussed with the Users, SAC and Council.

**Why do we need the LTS?** The ESRF has been extremely successful in terms of the quality (and quantity) of the science produced, the performance and reliability of the X-ray source, and the technical innovations of the ESRF's beamlines and instrumentation. Why not simply continue as we are, without major changes or interruption of the experimental programme? To me the answer is very clear. If we do not

experimental programme. In all of these the crucial element is the quality of the resulting science.

New infrastructures should be created to exploit the possibilities of our source and beamlines to their full potential. We include in this category, advanced instrumentation (and, in particular, refinements of current detectors and qualitatively new detector concepts), scientific collaborations exemplified by the Partnership for Structural Biology, and training facilities in SR science and techniques.

While the ESRF's Machine works extremely well (and is still the only one of the three "big" machines to operate routinely at 200 mA) future

exploited. One obvious area is the study of ever-smaller structures, moving from the micro to the nano-domain. Today the ESRF can routinely provide X-ray beams with dimensions of the order of a few microns: the record is below 100 by 100 nanometres. The challenge for tomorrow is to approach 10 nanometres or less.

These very ambitious ideas are currently being further considered, including the determination of the financial and manpower costs, with the help of the ESRF SAC and the wider SR community. To put even a part of these ideas into practice will require a major effort by the ESRF's staff, the SR community, our guiding committees (the Council, Administration and

*If we do not continue to improve and innovate,  
the ESRF will stagnate*

continue to improve and innovate, the ESRF will stagnate - and the new Synchrotron Radiation (SR) sources around Europe will overtake the ESRF in terms of performance and scientific output (not to mention the various FEL projects under consideration). So we need to think about how we can do even better than in the past, how we can retain a leading position in the production and exploitation of hard X-rays, working in cooperation with our European partner SR centres.

To achieve this, we can identify three areas of our activities where major development and refinement will result in significant scientific advance, namely scientific infrastructure, the ESRF X-ray source, and the

improvements are possible. With a suitable upgrade to the radio-frequency system, operation at 500 mA (or even 1A) may be envisaged; this would provide immediate benefits in terms of X-ray beam intensity, but problems associated with the increased heat-load will need to be solved. Even more exciting is the possibility of significantly decreasing the horizontal emittance, by up to a factor of 8. This would require a radical new design of the magnet lattice, but would open up completely new scientific areas.

The future experimental programme should focus on scientific fields where the unique capabilities of the ESRF's X-ray beams (brilliance, coherence, time structure, stability ...) can be

Finance Committee and Science Advisory Committee), and not least by the national Partners of the ESRF. Is it worth it? My reply is a most definite Yes! The return in terms of new science will more than balance the investment to keep the ESRF at the cutting edge of scientific research. ●



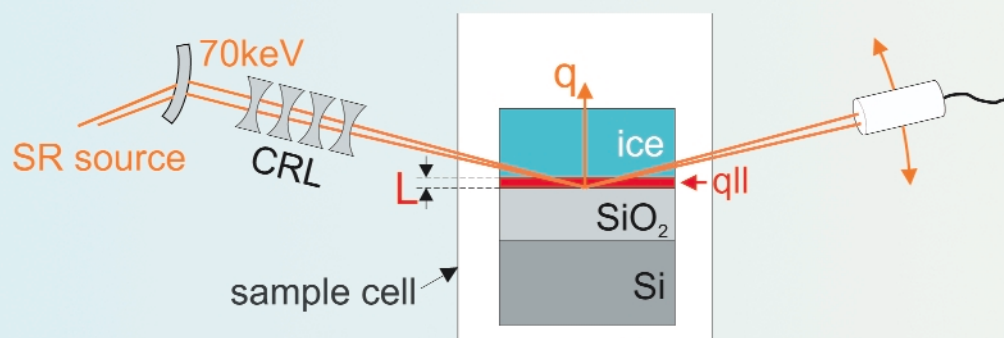
## Feature news

# RESEARCHERS STUDY HOW ICE MELTS

**A** team of scientists from the Max Planck Institute for Metals Research in Stuttgart (Germany) and the ESRF has studied how ice starts to melt at temperatures as low as  $-17^{\circ}\text{C}$ . This can occur when ice is in contact with  $\text{SiO}_2$ , a material commonly found in soil. Below the melting temperature of ice, a layer much denser than 'regular' water forms between the ice and the  $\text{SiO}_2$ . The researchers were able to observe such changes occurring thanks to the powerful X-rays at the ESRF. These results may help to explain natural phenomena such as how glaciers slide or the stability of permafrost.

Water is well known for its strange properties such as its expansion during the transition from liquid to solid. At school, students learn that the density of water is  $1 \text{ g/cm}^3$ . This is somewhat of an over simplification, however, due to the complex behaviour and relationships between water molecules. In reality water is thought to exhibit density fluctuations between a high-density liquid (HDL) and a low-density liquid (LDL) on very short time scales.

The team of scientists working at the ESRF discovered an odd water layer thanks to the high-energy X-ray microbeams of the synchrotron. They started by attaching crystalline ice onto silicon dioxide. The 24 mm long sample was kept in a specially-designed chamber in which the temperature of the sample could be stabilised and accurately controlled. As the X-rays penetrated the sample to the interface between water and silicon dioxide, the researchers started heating it from  $-25^{\circ}\text{C}$  to  $0^{\circ}\text{C}$ . By the time the structure reached the melting point, the sample already contained a 5 nm layer of water. This water was found to be 20% more compact than normal water, having a density of  $1.2 \text{ g/cm}^3$ .



Sketch of the experimental setup. A monochromatic high energy X-ray beam is focused by a compound refractive lens (CRL) to a size of  $5\ \mu\text{m}$  (vertical) by  $15\ \mu\text{m}$  (horizontal) at the sample position. The ice-SiO<sub>2</sub> interface is kept in a specially designed chamber in which the temperature of the sample is stabilised. The X-ray beam penetrates the sample at almost normal incidence from the side, thus illuminating only the ice-SiO<sub>2</sub> interface.

These results represent a step forward in understanding the behaviour of ice. They may help explain natural processes such as the movement of glaciers. The motion of glaciers can mainly be explained by the internal deformation induced by gravity, (being relatively slow at around only 10 m/year). Another process that is thought to contribute to this movement is basal sliding. Basal sliding can occur ten times faster when the base of the ice is near the melting point and some water is present to enhance glacier movement. Nevertheless, observing the results of the experiment performed at the ESRF, this pre-melting phase at a lower temperature than the melting point could support a basal sliding theory.

The ramifications of this study are not only confined to glaciers. Permafrost is another example where a lower

melting temperature could further our understanding. Permafrost describes rock or soil composite structures that remain below 0°C for two or more years and often contain more than 30% ice. Permafrost areas cover large inhabited regions, yet the interfacial-melting phenomenon is not well understood. The results confirmed by the team of researchers at the ESRF could be important for civil engineering projects within these regions.

The results of this experiment open the way for new research: “we will study how the ice behaves in contact with different solids instead of silicon dioxide”, explains Veijo Honkimäki, one of the authors of the paper. ●

M. C.

*S. Engemann et al. Interfacial melting of ice in contact with SiO<sub>2</sub>, Physical Review Letters, 92, 205701 (2004).*

## NANOPARTICLES GROWING IN A FLAME

**F**ire has fascinated mankind from prehistoric time. Man exploitation of fire is incredibly ancient, perhaps dating back to 1.6 million years ago (the time of homo erectus)! Since then, our lives have changed radically: fire would have helped them at night to keep other large carnivores at bay, in cooking and preserving meat, for hardening wooden tools and eventually to move around the world. Unknown to early man, the campfires probably produced a rich mixture of carbon nanoparticles and these pyrolytic pigments were used to paint prehistoric cave walls. Indeed, pigments used in prehistoric ceramic porcelains from China, Egypt and Greece were produced using a flame. The previous issue of the ESRF Newsletter already offered a glimpse into how Renaissance artisans made use of nanoparticles. Now scientists explore the growth of nanoparticles in a flame by exploiting the high brilliance of synchrotron radiation.

As in the past, flame synthesis is still one of the most versatile and promising technologies for large-scale production of nano-scale materials. Recently, pyrolysis was shown to be an ideal route for the production of single-walled nanotubes, quantum dots and a wide

variety of nano-structured ceramic oxides. Nanomaterials have distinctly different properties to bulk materials because the number of atoms or molecules on their surface can become comparable to that inside the particles. However, the key limitation is

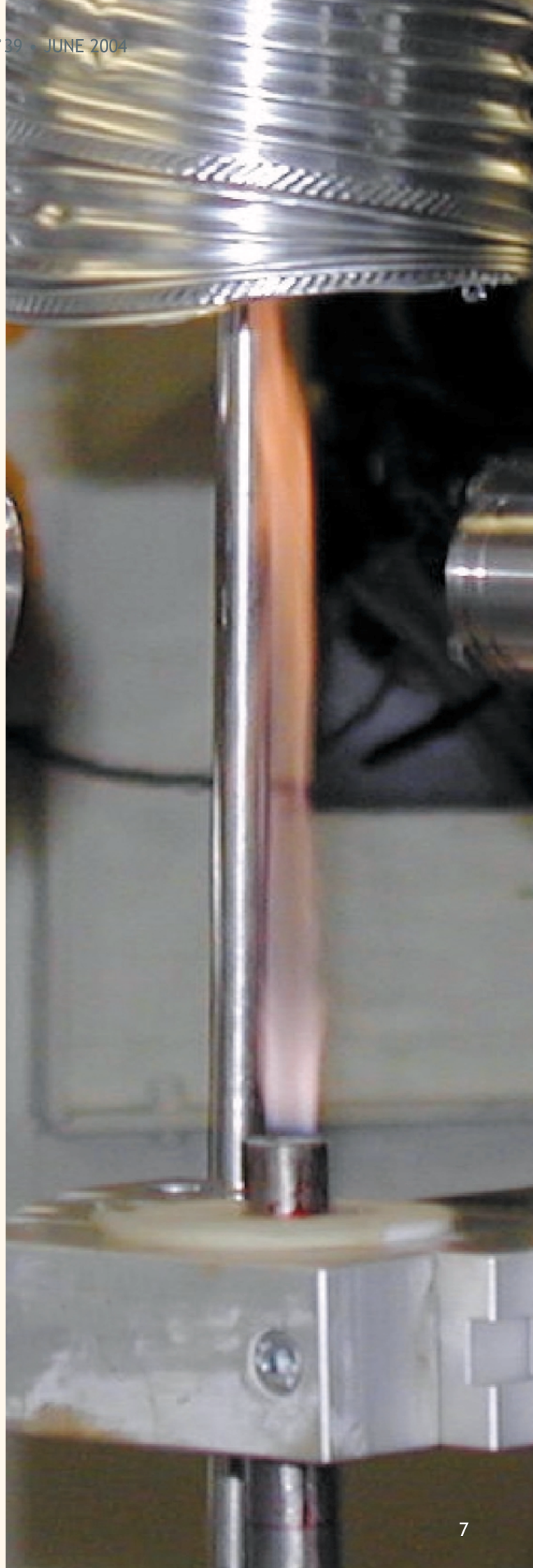
the lack of large quantities of nanosize particles with closely controlled properties at a cost that will allow scientists and engineers to comfortably explore new practical applications. Therefore, understanding the mechanism of growth is primordial in engineering these materials. So far, the *in situ* observation of particle growth in a flame has been hampered by the high temperatures (~ 2000 K, e.g. the boiling point of lead or melting point of platinum), rapid kinetics (sub-millisecond scale), dilute growth conditions (parts per million by volume), and optical emission of synthetic flames.

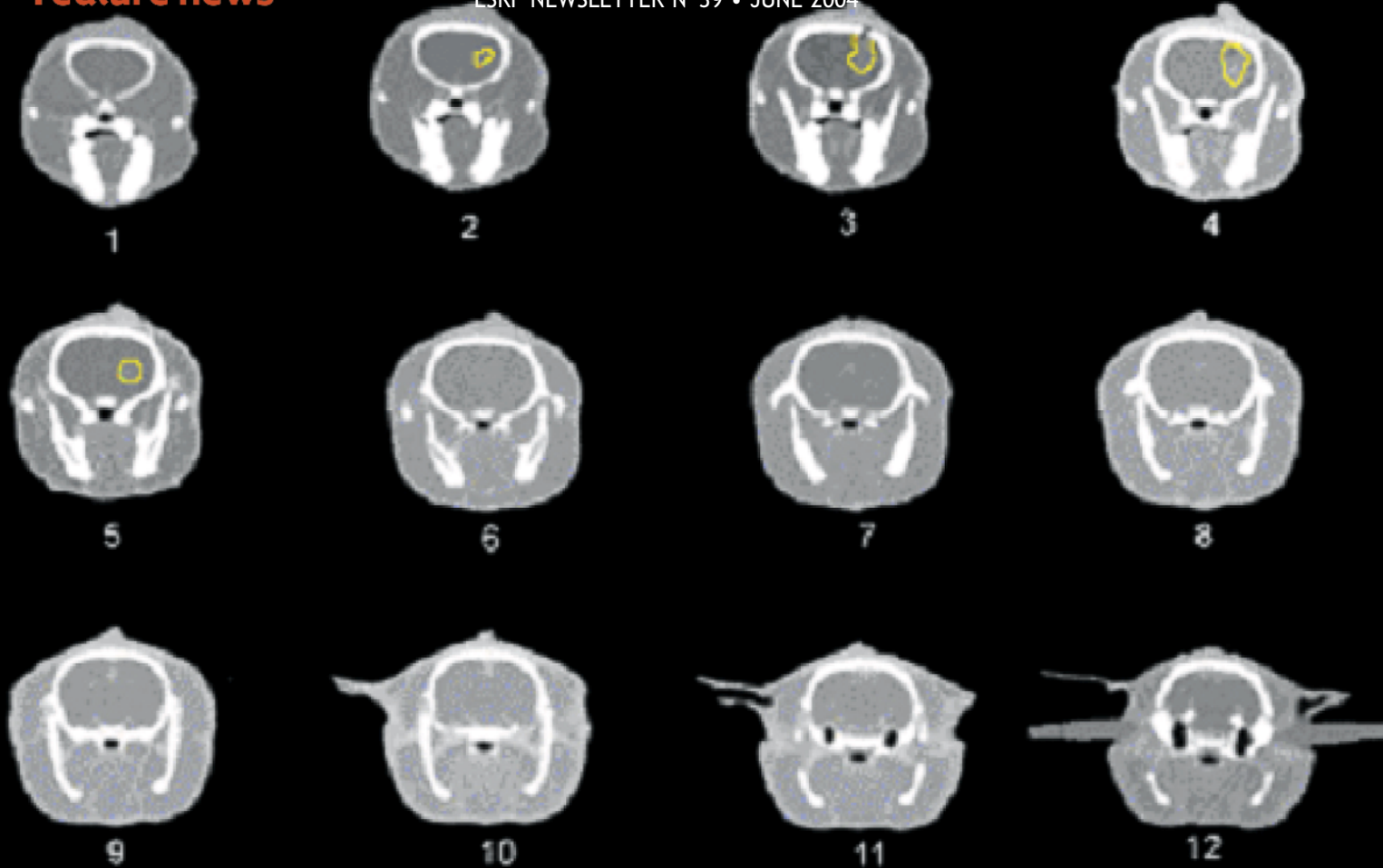
A team of researchers from the United States (University of Cincinnati), Switzerland (ETH Zürich) and the ESRF has investigated how the nanoparticles grow in the flame with millisecond resolution. They used the Small-Angle X-ray Scattering (SAXS) technique at beamline ID02 to probe the dynamics of nanoparticle growth. For the first time, direct two dimensional mapping of nanoparticle (silica) nucleation and growth were made on size-scales from 0.5 to 500 nanometres and on time-scales as short as 100 microseconds. The results support a simple diffusion-controlled growth law model. The data encourage current attempts at simulation and modelling of nanoparticle growth, while offering a technique for direct verification of their predictions. Furthermore, the results indicate the feasibility for studies of nanometre-scale aerosols of toxicological and environmental concern (e.g. diesel engine exhaust, power plants, etc.). ●

NARAYANAN THEYENCHERI

*G. Beaucage et al., Probing the dynamics of nanoparticle growth in a flame using synchrotron radiation, Nature Materials (May, 2004).*

The flame was produced with a methane and oxygen mixture to which the precursor material was fed by the carrier gas (nitrogen). A highly-collimated and intense monochromatic X-ray beam (size 300 micrometres) passed through the flame. The scattered intensity in the forward direction is recorded by a two-dimensional detector housed inside the 10 metre evacuated flight tube.





After each irradiation, rats were injected with iodinated contrast agent. Thereafter, tumor computed tomography imaging was performed by taking 12 images. Slice thickness was 1mm. The tumor is visible on slices 2 to 5. Ears bars are clearly visible in slice 12.

## NEW CLUES FOR BRAIN CANCER TREATMENT FOUND AT THE ESRF

**A** team of researchers from the University Hospital of Grenoble (CHU - Inserm U647) and the ESRF has found a new treatment that improves the survival of rats with high-grade gliomas. This research was carried out at the ESRF Medical Beamline. It showed that after a year of treatment, three rats out of 10 were considered cured, whereas without treatment, all would have died. The results have recently been published in the scientific journal *Cancer Research*. A glioma is one of the most frequently diagnosed brain tumours in adult humans, and it is not curable.

radiotherapy using hospital X-rays only has a palliative effect because gliomas are some of the most radio-resistant human tumours. Chemotherapy is ineffective most of the time. Several therapeutic techniques have been developed over the last few years using animal models, but none has had such successful results as this new treatment combining cis-platinum with monochromatic synchrotron X-rays. This new technique combines chemotherapy with radiotherapy in such a way that both techniques are effective when associated.

In this study, a drug called cis-platinum was injected into the brain of rats bearing F98 glioma. The substance entered the DNAs of the tumour and limited the tumoral proliferation. A day later, at the Medical Beamline, the tumour was irradiated with X-rays of a very precise energy (monochromatic). The difference between these X-rays and the conventional X-ray sources used in hospitals is the brilliance: the beam produced by the ESRF synchrotron is a hundred thousand times brighter than the beam produced by a hospital X-ray machine, allowing the beam to be tuned at a convenient wavelength.

Today, the median survival for patients with glioma is less than a year. Around five to ten adults out of 100.000 suffer from this brain tumour. Traditional



The team (from left to right): François Estève, Hélène Elleaume, Jean-François Adam, Jean-François Le Bas, Wendy Renier, Aurélie Joubert, Nicolas Foray, Caroline Boudou, Marie Claude Biston, Anne Marie Charvet. Absent in the picture : Sylvain Bohic and Jacques Balosso.



This *in vivo* experiment was preceded by *in vitro* experiments on cells using the same tumoral model (F98). This tumour is extremely radioresistant and it spreads very quickly. The mean survival time of untreated rats was 28 days. If cis-platinum was injected, they survived up to 39 days. If the rats were irradiated with X-rays at a certain wavelength, it could result in a maximum of 48 days of survival. The combination of both treatments, with a specific radiation dose and a specific X-ray wavelength appeared to be the most efficient treatment tested and offered a mean survival time of around 200 days. This means a 6-fold increase in the life span of treated rats compared to those which did not receive any treatment.

The success of the trials has led CHU and ESRF researchers to envisage the elaboration of a protocol in order to use these techniques on humans. "There is a lot of technological development to be carried out, but it is feasible", explains Doctor François Estève, one of the

authors of the paper. "The ESRF Medical Beamline is a unique place in the world where pre-clinical and clinical research in radiotherapy with synchrotron radiation is possible nowadays" says Doctor Alberto Bravin, scientist in charge of the Medical Beamline. The differences between rats and humans are great, so doctors and physicists are unable to say whether they would have the same outstanding results if humans were treated. But François Estève is convinced that "taking into account the impossibility nowadays of healing this brain tumour, it is essential that we try this method". ●

M. C.

*Biston et al., Cure of Fisher Rats Bearing Radioresistant F98 Glioma Treated with cis-Platinum and Irradiated with Monochromatic Synchrotron X-Rays, Cancer Res. 64: 2317-2323 (2004).*

*J-F. Adam et al., Synchrotron Radiation Therapy of Malignant Brain Glioma Loaded with an Iodinated Contrast Agent: First Trial on Rats Bearing F98 Gliomas, Int. J. Radiation Oncology Biol. Phys., Vol. 57, No. 5, pp. 1413-1426 (2003).*

## RECORD REQUESTS FOR BEAM-TIME

**F**ollowing the Users' Meeting in February, potential users submitted a record number of 923 applications for beam-time for the March deadline. This represents an increase of some 23% over the number of projects submitted in September 2003.

Just like in the September round, the scientific area that received the most proposals was Hard Condensed

Matter Structures, with 158 proposals, followed by Soft Condensed Matter, with 144.

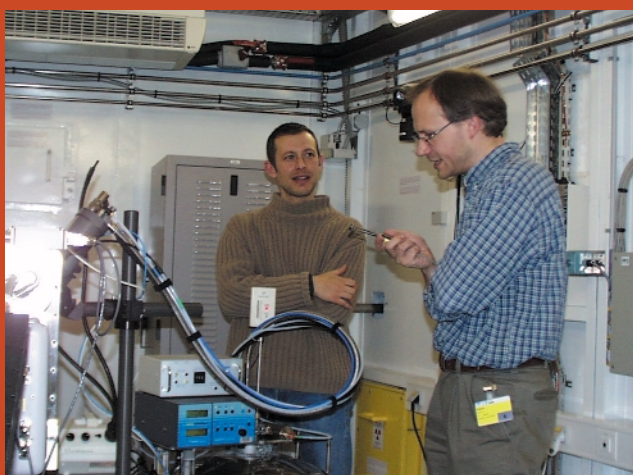
Nine Review Committees of experts in a range of scientific domains met in parallel at the ESRF in April to assess the applications for their scientific quality and recommend projects for scheduling on beamlines during the second half of 2004. ●

ROSELYN MASON

# THE PARTNERSHIP FOR STRUCTURAL BIOLOGY TAKES SHAPE

**A** year after its creation by four research institutes in Grenoble ESRF, EMBL, ILL and IBS, the Partnership for Structural Biology (PSB) is becoming a reality by uniting the activities of the four partners in a joint building. Ground breaking for the dedicated laboratory takes place in June this year. Scientific collaboration between the teams is also taking shape.

## Gemini: A new state-of-the-art beamline inaugurated at the ESRF



Didier Nurizzo (left), scientist in charge on ID23-1, discusses with Martin Noble (right), head of the first team of users on this beamline, from the University of Oxford.

The first end-station of the new ID23 beamline at the ESRF is operational. Associated with the PSB scientific programme, it is designed to achieve high-throughput measurements for macromolecular crystallography in an automatic, industrialised and user-friendly environment. Modern macromolecular crystallography experiments take under one hour to complete compared to an entire day or days a few years ago. This turnover is expected to increase with the automation of the beamlines being developed at the ESRF and EMBL. By the end of 2004, the first ID23 station will be equipped with a robotic sample changer allowing users to rapidly screen and assess their projects.

The PSB project advances. "Everything is set", says Stephen Cusack, Director of the EMBL Grenoble outstation. Indeed, the construction of a new laboratory complex on the international site in Grenoble starts during June and should be completed for the scientists 15 months later. The laboratory complex will also host the IVMS, the molecular virology institute of Grenoble's Joseph Fourier University.

The Partnership for Structural Biology is set to become a structural biology centre unique in the world. The PSB is already playing a leading role in the various large-scale structural biology initiatives now underway in Europe, notably the Structural Proteomics in Europe (SPINE) and BIOXHIT projects funded by the EU, and will be a centre for training young scientists in the field. Collaborations with other European institutes are encouraged.

"To obtain insight into fundamental biological processes or to speed up the development of new antibiotics or anti-cancer drugs - it is absolutely necessary to gain detailed knowledge about the structure of biological molecules and their complexes," says Stephen Cusack, head of EMBL-Grenoble and current chairman of PSB. "To do this, we have combined our resources to create a unique pool of modern scientific techniques and instrumentation which we will use to study proteins related to human health and disease, notably viral and human proteins."

In the context of the PSB, the ESRF has constructed a new, state-of-the-art, X-ray beamline for macromolecular crystallography, which is already partly operational, and the ILL has set up a unique laboratory for the deuteration of proteins, which is available to user groups. The Partnership will also include high-throughput protein expression and crystallisation facilities as well as facilities for protein characterisation. ●

M. C.

# THE ESRF IS PARTICIPATING IN THE EU 6th FRAMEWORK PROGRAMME

The Framework Programme (FP) is the EU's main instrument for research funding in Europe. It is proposed by the European Commission and adopted by Council and the European Parliament following a co-decision procedure. Each programme covers a 5-year period. The 6th Framework Programme covers the period from 2003 to 2006.

FP6 aims to contribute to the creation of a true "European Research Area" (ERA). ERA is a vision for the future of research in Europe, an internal market for science and

technology. It fosters scientific excellence, competitiveness and innovation through the promotion of better co-operation and co-ordination between relevant actors at all levels. Economic growth increasingly depends on research, and many of the present and foreseeable challenges for industry and society can no longer be solved at national level alone. The FP is the financial instrument that will help make the European Research Area a reality. The ESRF is involved in the following projects that have been accepted in the 6th Framework Programme:

## PROJECTS

## AIM

**FLASH**  
Understanding Fast  
Light-Actuated Structural Changes

To exploit recently developed ultrafast pulsed X-ray technology so as to open new windows on fundamental processes in photo-chemistry and photo-physics.

**IA-SFS**  
Integrating Activity on Synchrotron and Free  
Electron Laser Science

To support transnational users of national facilities in the domain of synchrotron and Free Electron Laser science and to support joint research activities to enhance the effectiveness of facilities and the development of novel sources.

**BIOXHIT**  
BIOcrystallography on a Highly Integrated  
Technology Platform for European Structural  
Genomics

To develop, assemble and provide an integrated platform for high-throughput structure determination using X-ray crystallography with Synchrotron Radiation.

**DYNASYNC**  
Dynamics in Nano-scale Materials Studied  
with Synchrotron Radiation

To study different dynamical aspects in nanostructures: diffusion, phonons and magnetization dynamics on carefully selected model-nanostructures, using different experimental methods, but mainly Nuclear Resonant Scattering.

**X-TIP**  
Nano-scale chemical mapping and surface  
structural modification by joined use of  
X-ray microbeams and tip assisted local  
detection

To deliver instrumentation and techniques that merge the ability of Synchrotron Radiation Spectroscopies in providing elemental composition, chemical status and structural information with the lateral resolution of Local Probe Microscopes.

**Ductilisation of Bulk Metallic Glasses (BMGs)  
by Length-scale Control in BMGs Composites  
and Applications**

Springs made of BMG have their elastic limit at 2%, and when the limit is surpassed, they fail by fracture. The ductile BMG-composites proposed by this project will maintain the superior 2% elastic limit and their near 10% room-temperature plasticity will avoid failure beyond the elastic limit until replaced at the end of a far longer operational life-time.

**NanO<sub>2</sub>**  
Oxidation of Nanomaterials

To understand the interaction of oxygen with metallic nanoparticles. Surface sensitive *in situ* techniques for high oxygen pressures and temperatures will be combined with *ab initio* thermodynamic calculations.

## OBITUARY

## CARL-IVAR BRÄNDÉN



Professor Carl-Ivar Brändén, Director of Research of the ESRF from April 1992 to March 1997, died on 3 May 2004 after a long illness.

When Carl-Ivar Brändén joined the ESRF in April 1992, the storage ring had just started operation, the first beamlines were being designed and doubts concerning the Experimental Hall floor had stirred up Management and scientific

community. During his term of office, the ESRF's beamline programme took shape and the Experiments Division grew by more than 100 scientists and technicians. Carl-Ivar Brändén was in particular responsible for the fields of biology, chemistry and medicine, with the aim of providing the corresponding communities with adequate facilities and infrastructure at the ESRF. Under his guidance the field of macromolecular crystallography developed into one of the major activities of the ESRF. Those who knew him during his activity at the ESRF will remember his friendliness and his subtle sense of humour. He will be sadly missed by his many friends and collaborators around the world. ●

W.G. STIRLING

## POLAND JOINS THE ESRF

**T**wo weeks before becoming part of the European Union, Poland joined the ESRF as a Scientific Associate, at a level of 0.6% as regards financial contributions and scientific use. Professor Jacek Kossut, Director of the Institute of Physics of the Polish Academy of Sciences, who signed the agreement, announced that he was "very enthusiastic" about this official relationship and hoped that it was "the beginning of a larger activity at the ESRF". From the ESRF side, the agreement was signed by Bill Stirling, the Director General, and Helmut Krech, the Director of the Administration.

"Mission accomplished!" Those were the words of Professor Jacek Kossut, Director of the Institute of Physics of the Polish Academy of Sciences, when asked for his impressions about Poland joining the ESRF. "This is the happy ending to ten years of discussions between Polish scientists and administrators and the ESRF." In fact, Poland was interested in being involved in the ESRF almost from the beginning of operations. At that time, however, the option of arrangements with Scientific Associates had not yet been developed; only scientists from countries represented by Members, contributing a minimum of 4% to the ESRF budget, had regular access to the facility. Nevertheless, even before Poland became part of the ESRF family, the country was already present at the facility with two Polish staff members and quite a number of studies carried out in collaboration with scientists from Member countries. Among the proposals for the second

half of 2004, 1.3% were received from Poland, which shows the remarkable activity of the Polish scientific community. Scientists from 15 different Polish institutions have applied for beam-time, not only on ESRF beamlines but also on Collaboration Research Group beamlines. "The synchrotron community in Poland is even bigger than I thought", explained Professor Kossut.



From left to right: The Director General of the ESRF, Bill Stirling; the two Polish scientists at the ESRF, Maciej Lorenc and Johanna Hoszowska; and Prof. Krystyna Jablonska-Lawniczak, Prof. Bogdan Kowalski and Prof. Jacek Kossut, from the Polish Academy of Sciences.

The ESRF's agreement with Poland will last for two years (July 2004 - June 2006). After that, "and depending on the demand", says Kossut, it is even envisaged that they join forces with the Czech Republic and Hungary to form the CENTRALSYNC consortium. In this way, the three countries would participate at more than 1% in the ESRF. This would allow them to have an observer on the Council, the body that makes decisions about important issues of company policy. ●

M. C.



## Interview

# MONIQUE NAVIZET

## The art of constructing

Technician Monique Navizet, who participated in the construction of various beamlines at the ESRF, has started a new career collecting data at the macromolecular crystallography beamlines. In her spare time, she creates beauty in the paintings she does.

**T**he technician Monique Navizet always wanted “to build things”. She participated in the construction of beamlines such as ID03 or ID32 when she started working at the ESRF in the 90’s. Later on, she was involved in building the MEDEA beamline, dedicated to microelectronics industry. This was a very specific beamline and the first one to have a clean room in all the ESRF. Her work when mounting a beamline consisted of doing the conception of the beamline, designing the plans and following the construction. MEDEA was dismantled at the end of 2003, six years after its construction. This closed a professional period in Monique Navizet’s career, but opened new opportunities for her.

Since the beginning of the year, she has been working part-time for the Macromolecular Crystallography

Group in the framework of MXpress, a service of full data collection of samples sent per post by companies. “My work has changed completely, but I find this new field very interesting. Moreover, it is healthy to change, and it is important to answer to the needs of the place where you work”. She admits to still being in a learning stage, since she’s never worked in the field of Macromolecular Crystallography before. She is even improving her English, since most of her new colleagues are British. The other 50% of her time, she is involved in other projects, namely the European X-TIP (*see page 11*).

Monique is not only enthusiastic about learning, but also about teaching. Therefore she has very regularly supervised trainees at the ESRF, an activity that pleases her a lot. Indeed, she greatly appreciates contact with people. That is what has led her to

### WOMEN TECHNICIANS

“It was very difficult to speak about mechanical construction in the 70’s for a woman”, she explains. The job of technician in mechanical engineering “wasn’t considered a job for women”, she adds. The first times she went to the workshops where they constructed the instruments, she was observed with surprise by the workers. Now, years later, she has a trustworthy and respectful relationship with them: “Now they are used to working with me. Moreover there are a few more women doing this job, which is positive”.

become an elected member of the village of Proveyzieux, of 500 inhabitants, where she has lived since she came from Brittany thirty years ago.

Her political activities leave her little time to dedicate to her hobbies of painting and pottery. Nevertheless, she has some exhibitions behind her and others foreseen in the near future. Taking a brush and letting it glide over a canvas represents a “time out” in her busy life: “When I paint, I forget about work and my political life”. “It’s my passion”, she says. ●

M. C.

## Users' views

### CLAUDIO NICOLINI:

“The ESRF has to be the voice for the synchrotron community in Europe”

**Claudio Nicolini moved from nuclear physics to biomedical research in the middle of his scientific career. Although his CV may include a brief period in politics, he now devotes himself to research in biophysics, halfway between physics and biology. He holds the chair of Biophysics at the Medical School of the University of Genoa and he is also Director of the Nanoworld Institute in Genoa. His research relationship with the ESRF started one year ago when he began coming to ID13 as a user. He defines the microfocus beamline as “a unique facility to characterise nanostructure”, and is convinced that synchrotrons will exist for many years to come.**

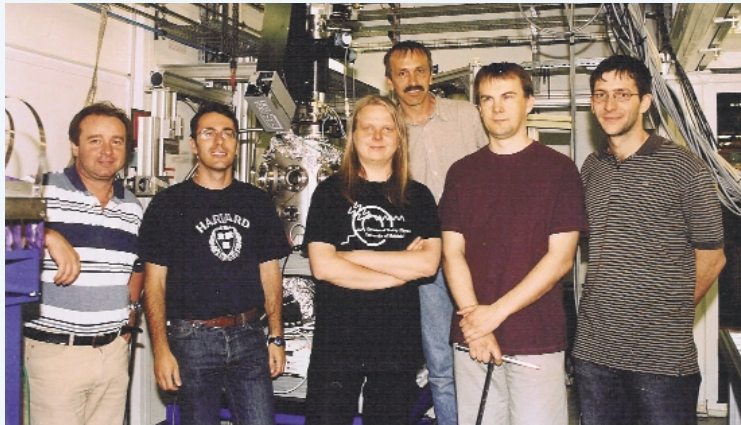
**H**e lived for almost 20 years in the United States and, although he has now returned to live in his home country of Italy, he still shares his time between the two sides of the Atlantic. This has allowed him to see two different ways of working in science and he is clearly more favourable to the American one. “In the USA scientists collaborate

readily, so that they can enrich each other, whereas in Europe there is not such a culture, and this is bad”, he says. In this sense he defines the science performed using synchrotrons in Europe as being like “many churches”, each of them working independently. He maintains a quite critical opinion towards the construction of national synchrotron sources all over Europe.

Since the cost of building and maintaining a synchrotron is enormous, he thinks that “there should be a concentration of resources, and all the X-rays scientists should join forces, instead of being scattered between many synchrotrons”. We expect to hear more of Professor Nicolini’s controversial views ! ●

M. C.

The ID16 team (from left to right): Roberto Verbeni, Giulio Monaco, Simo Huotari, Christian Henriquet, Gyorgy Vanko and Fabio Pasquali.



## Visiting a beamline

# ID16 CELEBRATES ITS TENTH ANNIVERSARY EXPLORING A NEW TECHNIQUE

**O**n ID16 everyone is very busy these days. A new instrument wrapped in aluminium in the shape of a big ball is taking all the attention. It is the instrument that will allow VOLPE (VOLume Sensitive PhotoEmission from Solids) experiments, a new spectroscopic tool to investigate electronic properties. "It is a very promising technique, and we are all investing a lot of time in this", explains Giulio Monaco, scientist in charge of the beamline. The team has already got some results of preliminary tests of the instrument and this month they will start doing some physics research with it.

This is not the technique mostly used at this beamline where most of the experiments are in the field of fundamental physics. On ID16 they essentially carry out scattering experiments both with very high energy resolution to study the acoustic properties in materials and with moderate energy resolution to find out the electronic structure of mainly crystals.

The techniques require a lot of changes in the setup, that's why "the Machine Day (the day in the week where there is no beam) is always the busiest for us in



Christian Henriquet and Roberto Verbeni in the Experimental Hutch.

the whole week, because that's when we can change it", explains Giulio Monaco.

This frantic activity doesn't leave the team much time for celebrations of the 10th anniversary of the beamline. Ten years of science on ID16, which have been a reality thanks to the present team as well as other staff, who are still at the ESRF, such as Francesco Sette, Michael Krisch and Bernard Gorges. Roberto Verbeni, the Beamline Operation Manager (BLOM), has worked on ID16 since the first days of the beamline. Giulio Monaco defines him as the "cornerstone of the beamline". Indeed, he acknowledges that the first two years were the most exciting ones since the work was focused on the construction of the beamline. "Work is never over", he says convinced. In fact, in the last five years, most of the instrumentation in the beamline has changed.

The dynamic team of ID16 includes two scientists, one BLOM, two post-docs and two PhD students, who do their research partly on this beamline. Their origins range from southern Italy to northern Finland, Hungary and France. Despite the different cultures, the team gets on very well. They often go for lunch together and some of them even for holidays! One can definitely feel the good vibrations in the team when talking to them. Christian Henriquet, the technician, irradiates energy: "I worked in the industry for 20 years, and it is only now that I really enjoy my work: I love my job and the people I work with are really excellent", he explains. ●

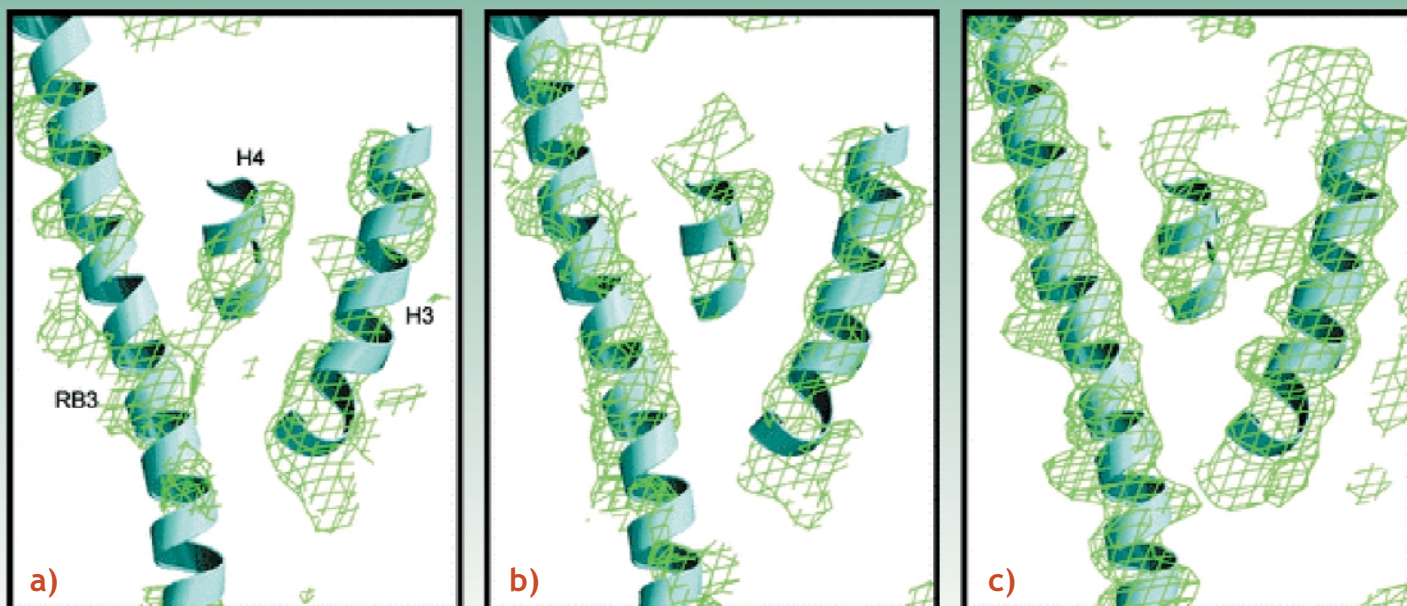


Fig. 1: Electron density maps from a) initial experimental MIRAS phases, b) zero-dose corrected data and c) after improvement using non-crystallographic symmetry methods.

## Selected scientific highlights

### MACROMOLECULAR CRYSTALLOGRAPHY GROUP

## Zero-Dose Correction Enlightens the Mechanism of a 2500 Year Old Drug

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P.A. Curmi, I. Jourdain, S. Lachkar, A. Sobel

*U440 INSERM/UPMC, Institut du Fer à Moulin, Paris, France*

Crystals of large macromolecular complexes are often particularly prone to X-ray radiation damage as they tend to diffract very weakly, so that the full intensity of our brightest sources is required in order to obtain the highest possible resolution diffraction data. The collection of complete data sets can be very cumbersome, as radiation damage will compromise diffraction data quality from the very beginning of the experiment.

It has been shown, however, that it is in principle possible to partially correct for this damage by extrapolating to obtain diffraction data as if they were collected at zero X-ray dose [1]. The usefulness of this technique has been demonstrated in the solution of the crystal structure of a

210 kDa complex between tubulin, colchicine, and the stathmin-like domain of a neural protein (RB3). Crystals of this complex typically diffract to not better than 4 Å resolution on our brightest MX undulator beamlines. Initial weak SeMet (4 Se atoms for 210 kDa) SAD phases did not allow the immediate elucidation of the structure (Figure 1a). However correcting the diffraction data for radiation damage improved the phases to such an extent (Figure 1b) that when non-crystallographic symmetry was applied (Figure 1c) a model of the complex could be built. The final model was determined based on experimental MIRAS phases, after screening hundreds of native and derivative crystals. The results of this very exciting work reveal i) the interaction between RB3 and tubulin and provides an explanation as to why this complex is not incorporated

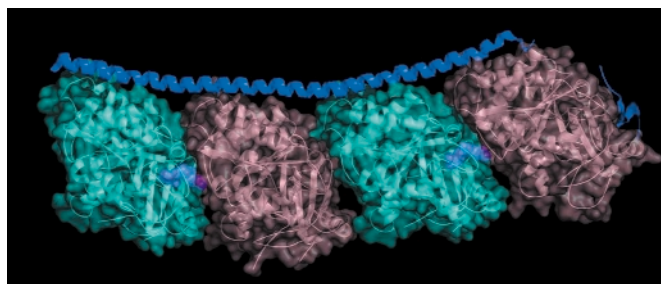


into microtubules,  
 ii) the interaction between tubulin and the anti-gout drug colchicine known for 2500 years, and  
 iii) the differences between curved, soluble tubulin heterodimers and tubulin heterodimers as found in protofilaments.

The latter aspect provides insights into microtubule dynamic instability.

*Nature*, **428**, 198 - 202 (2004); \* These authors contributed equally to this work.

[1] K. Diederichs, S. McSweeney, R.B.G. Ravelli, *Acta Cryst.* **D59**, 903-9 (2003).



*Fig. 2: The tubulin RB3 complex. The complex includes two tubulin  $\alpha$ - $\beta$  heterodimers with colchicine bound to  $\beta$  subunits at the interface with  $\alpha$ .*

## SURFACE AND INTERFACE SCIENCE GROUP

### The Structure of Ge(111): C<sub>60</sub> - (13<sup>1/2</sup> x 13<sup>1/2</sup>)

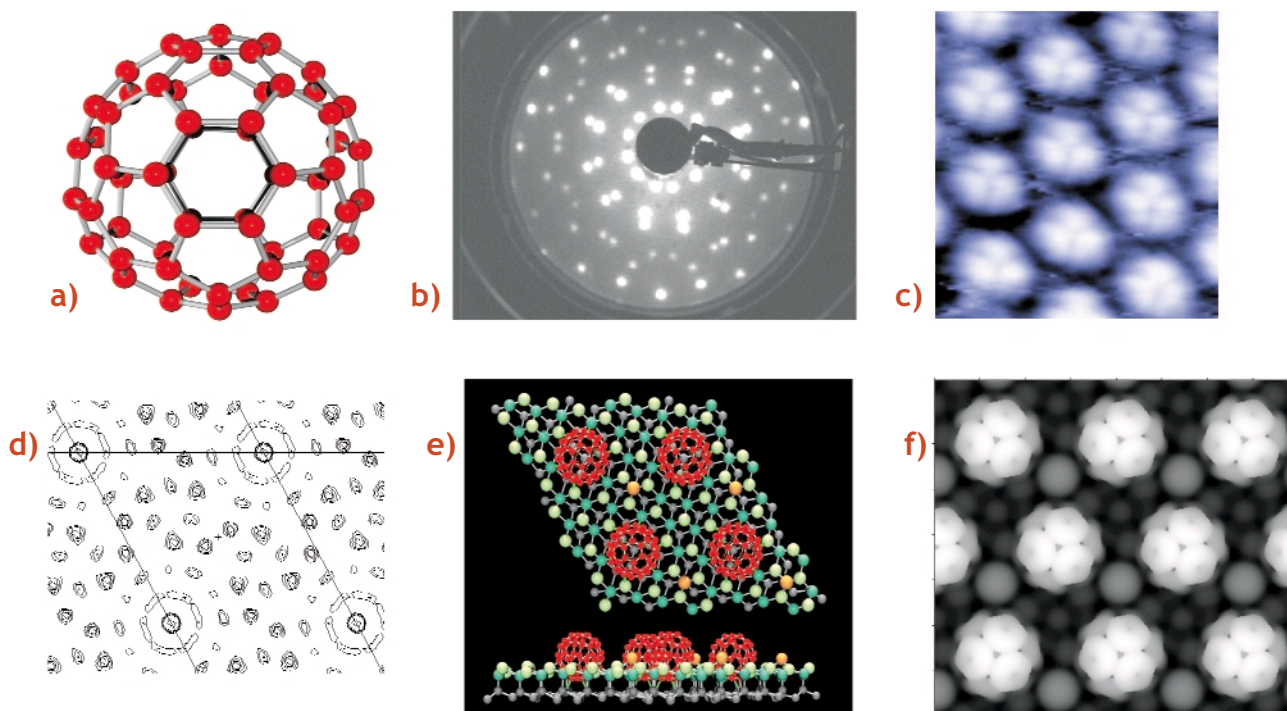
X. Torrelles, J. Rius, O. Bikondoa, P. Ordejon, E. Machado

ICMAB-CSIC, Barcelona, Spain

T.L. Lee, J. Zegenhagen *ESRF*

On the Ge(111) surface, the C<sub>60</sub> molecule, which had been discovered in 1985 by the 1996 Nobel prize laureate Sir Harold Kroto, gives rise to a (13<sup>1/2</sup> x 13<sup>1/2</sup>) reconstruction. The C<sub>60</sub> molecules are

resting immobilised on the surface and desorb at a temperature of above 630°C, which is astonishing considering the bulk-sublimation temperature of about 200°C.



*Fig. 3: Analysis of (13<sup>1/2</sup>) x (13<sup>1/2</sup>) reconstruction of C<sub>60</sub> on Ge(111) by Grazing Incidence Diffraction and Scanning Tunnel Microscopy. (a) C<sub>60</sub> molecule; (b) LEED image of (13<sup>1/2</sup>) x (13<sup>1/2</sup>) reconstruction of C<sub>60</sub> on Ge(111); (c) High resolution STM image; (d) result of analysis of more than 2000 recorded structure factors by direct methods; (e) resulting 3D structural model; (f) resulting theoretical STM "image" of the C<sub>60</sub> on Ge(111). The C<sub>60</sub> - C<sub>60</sub> distance is 1.4 nm.*

This surprising stability must be explained by the structure of the molecular adsorbate, the determination of which, with more than 100 participating atoms, is a terrific task. The Ge(111): C<sub>60</sub> - (13<sup>1/2</sup> × 13<sup>1/2</sup>) sample was prepared in the surface characterisation laboratory (SCL), characterised by LEED and STM, loaded into the ID32 UHV baby-chamber, which was mounted on the ID32 diffractometer. The Grazing Incidence Diffraction (GID) study was carried out at the ID32 beamline, recording at 11 keV close to

2000 in-equivalent structure factors. Application of direct methods and least square refinement gave a unique, three-dimensional structural model resolving also the C<sub>60</sub> orientation. The surface unit cell contains one Ge adatom and the C<sub>60</sub> molecule is sinking into the surface, because six Ge surface atoms are missing. Based on this model, theoretical calculations of the local density of states at different energies around the Fermi level are very well reproducing the STM images obtained at corresponding bias values.

## HIGH RESOLUTION AND RESONANCE SCATTERING GROUP Phonon Dispersion in Graphite

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S. Reich, P. Ordejon *Institut de Ciencia de Materials de Barcelona (CSIC), Spain*

H. Requardt *ESRF*

Despite the importance of graphite as building brick for a large number of technologically relevant materials such as carbon nanotubes or intercalated graphite, surprisingly little is known about its vibrational properties. Due to the lack of sufficiently large crystals, inelastic neutron scattering cannot be utilised, and calculations differ qualitatively and quantitatively from each other. Exploiting micro-focusing techniques, a recent inelastic X-ray scattering experiment at beamline

ID28 succeeded to precisely determine the dispersion of the in-plane optical phonons in a tiny single crystal of 4x10<sup>-4</sup> mm<sup>3</sup> volume. These results - in conjunction with *ab initio* calculations - have allowed us to establish the long-range nature of the in-plane force constants, and to obtain finally a coherent picture of the vibrational properties of this important elemental solid.

*J. Maultzsch et al., Physical Review Letters 92, 075501 (2004).*

## TECHNICAL DEVELOPMENTS ON THE BEAMLINES

### BM 26 (DUBBLE) – Industrial Polymer Processing

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E.L. Heeley *University of Sheffield, UK*

W. Bras *ESRF*

Polymers are processed in molten form using a variety of techniques such as extrusion and injection moulding. Extrusion forms the basis of most industrial polymer processes. Solid pellets are fed into an extruder, which consists of a heated screw that melts and processes polymer into a shaping device known as a die prior to being cooled. Recently, a scaled-down commercial

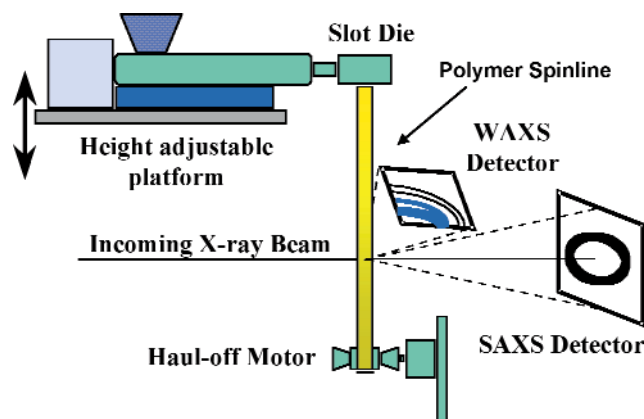


Fig. 4: Schematic of the Axon BX18 extruder.

extruder instrument (Axon BX18) has been installed on the Dubble CRG beamline BM26b, which has enabled real-time polymer processing experiments to be conducted. **Figure 4** shows a schematic picture of the extruder instrument on Dubble.

This instrument is currently being used to investigate the structure development during the processing of commercial grade polyolefin materials, such as polyethylene and polypropylene. Combining Small and Wide Angle X-ray Scattering (SAXS/WAXS) techniques

whilst the polymer tape is extruded on-line, allows detailed information of the semi-crystalline structure development of the polymer material to be studied. Understanding the mechanisms of such processing methods and the structure of the material produced allows industry to develop tailor-made polymers, which have the ideal properties for their specific end use as well as being relevant in fundamental research due to the information that can be gathered about the development of macro- and micro-structure.

## ID13 – An Ink-jet System for the Study of Hydration Processes in Biopolymers

M. Rössle *EMBL-DESY, Germany*

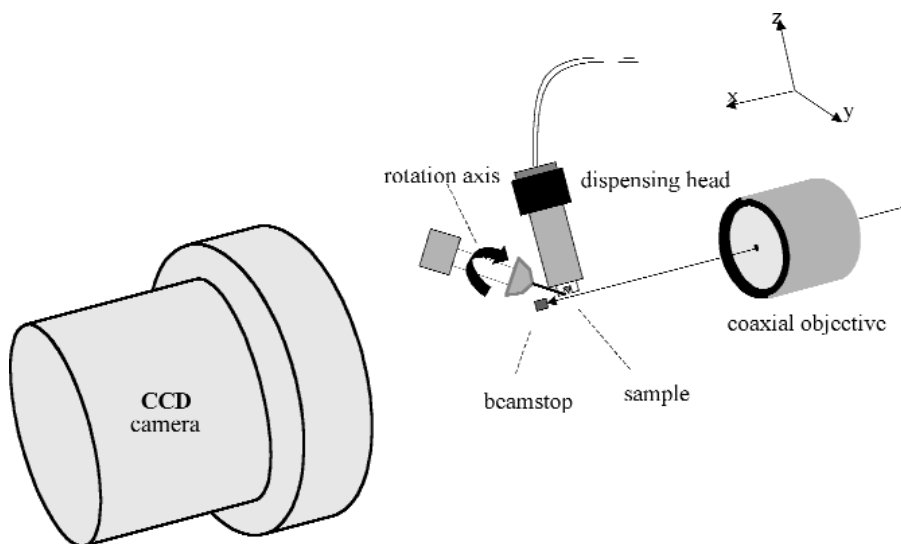
D. Flot, M. Burghammer, C. Riekkel *ESRF*

J. Engel *Fk Z-Karlsruhe, Germany*

H. Chanzy *CERMAV-Grenoble, France*

Fluid quantities down to the picolitre range can be dispensed with a high temporal and positional precision using the ink-jet technology. At flight velocities of about 2 m/s, it is possible to increase the liquid

occurring in Pogonophora tubes.[1] The setup on the ID13 micro-goniometer is shown in **Figure 5**. We have recently extended these experiments to other polysaccharide systems, like starch. In addition we



*Fig. 5: Schematic design of ink-jet system on the ID13 micro-goniometer.*

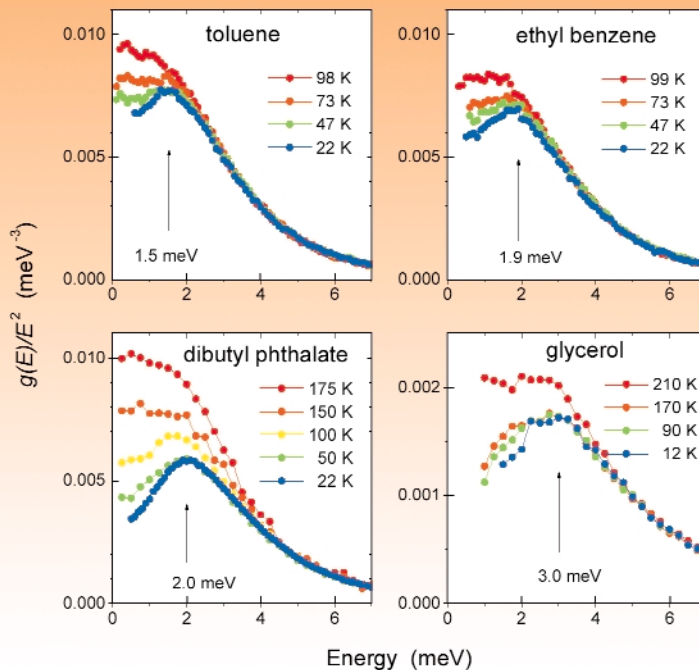
*The dispensing head is located about 200  $\mu\text{m}$  above the sample, which is fixed to the rotation stage. The co-axial objective allows an easy matching of beam position, sample position and path of micro-drops.*

concentration at a specific point of a solid on the subms time scale. For droplets of about 50  $\mu\text{m}$  diameter, the surface energy becomes larger than the kinetic energy, and the droplets merge with the solid without splashing. This allows initiating a hydration reaction on a biopolymer material spatially and temporally with a very high precision. We have used an ink-jet system to study the intracrystalline hydration of  $\beta$ -chitin

are exploring a crossed droplet setup in order to investigate fast mixing reactions.

[1] M. Rössle, D. Flot, J. Engel, M. Burghammer, C. Riekkel, H. Chanzy *Biomacromolecules*, **4**, 981-986 (2003).

Fig. 1: Reduced DOS of collective motions in toluene, ethylbenzene, dibutylphthalate, and glycerol glasses. Arrows indicate the energy of the boson peak estimated from the data at lowest temperature.



## Scientific article

# UNIVERSAL DYNAMICS OF GLASSES

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T. Asthalter *Physikalische Chemie II, Universität Stuttgart, Stuttgart, Germany*

The dynamics of glasses is often more difficult to understand than that of crystals. In some sense this is surprising, because order may have an infinite variety of forms whereas chaos should be universal. The universal trends of disorder, however, are often obscured by particular molecular properties. Disregarding such molecular modes and focusing exclusively on collective vibrations, we found a universal law for the high-frequency part of the spectrum of vibrations in glasses [1].

In order to measure exclusively the collective motions, we studied the glass dynamics using probe molecules. When the probe 'swims' in a glass matrix without coupling to the host molecules, it follows the collective motions of the glass but is not sensitive to the local modes of the host molecules.

In order to monitor the motion of the probes, we used the isotope-selective technique of nuclear inelastic scattering [2] and probe molecules with a resonant nucleus in the center of mass. With this approach, one monitors exclusively the motions of the central resonant

nucleus. Furthermore, in this way one selects pure translational motions of the probes: Rotation is disregarded because the spectator nucleus is in the center of mass; the few intra-molecular modes are separated in energy. The selected pure translational motions of the probe give the "density of states of collective motions" (CDOS) of the glass matrix with a correlation length (determined by the probe and host molecules sizes) of more than  $\sim 20 \text{ \AA}$  [1].

We investigated toluene, ethylbenzene, dibutylphthalate, and glycerol glasses. The probes were neutral ferrocene molecules with the central resonant  $^{57}\text{Fe}$  nucleus for the three first glasses and  $^{57}\text{Fe}^{2+}$  ions for glycerol. The insensitivity of the probes to local vibrations in the glass is confirmed by comparison to the total DOS available from neutron data.

The reduced CDOS  $g(E)/E^2$  is shown in Figure 1. For all studied glasses it clearly exhibits an excess of low-energy modes - the so called 'boson peak'. The positions of the peak are consistent with the boson peak energies in the total DOS from neutron and light scattering data [1]. The

temperature evolution of the boson peak shows the same features as observed with other methods: It is temperature independent at low temperatures, becomes less pronounced at higher temperatures and is completely hidden by low-energy modes when approaching the glass-liquid transition. This clear manifestation of the boson peak in the CDOS proves that the boson peak in the total DOS is largely composed of collective modes.

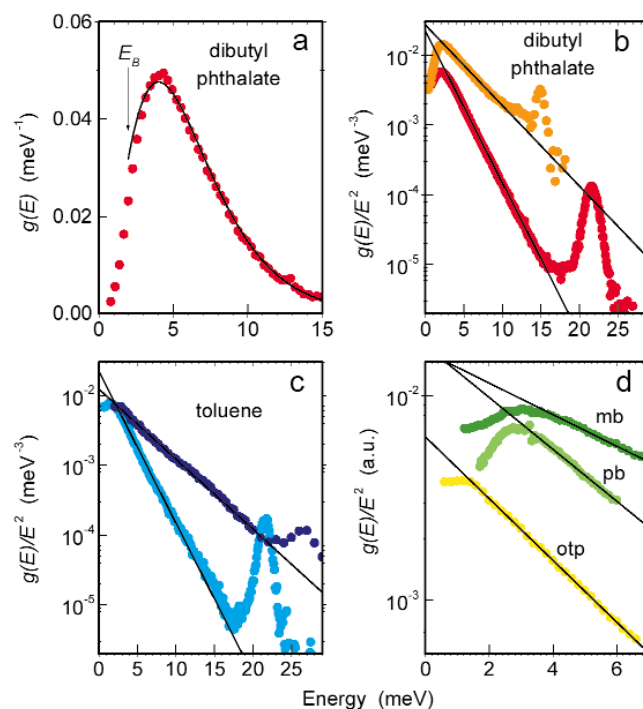
Beyond the boson peak, the reduced density of states of collective motions reveals for all studied glasses a *temperature-independent exponential* behavior:

$$g(E)/E^2 \sim \exp(-E/E_0), \quad \text{Eq. (1)}$$

which, to our best knowledge, was not reported earlier. Quite intriguing, the characteristic "decay" energies  $E_0$  correlate with the energies of the boson peak. **Figure 2a** shows that **Eq.(1)** describes the CDOS perfectly at high energies, still quite well near the maximum, and starts to fail approaching the energy of the boson peak. In a log scale,  $g(E)/E^2$  follows a straight line over three decades of the reduced CDOS and starts to deviate from **Eq.(1)** only when obscured by the eigen mode of ferrocene at 22 meV (**Figures 2b-c**). Such a large "dynamical range" allows for a clear identification of the CDOS behavior with **Eq.(1)**.

We also found this exponential behavior in the total DOS available from neutron data for all glasses studied here as well as for other molecular glasses. The most evident examples are shown in **Figures 2b-d**. In toluene, for instance, the total DOS follows **Eq.(1)** in the energy range up to 22 meV and over two decades of  $g(E)/E^2$  (**Figure 2c**). In comparison to the total DOS, the CDOS exhibits slightly lower energy of the boson peak and a considerably steeper exponential slope. This difference can be explained by different correlation lengths accessible with various techniques [1, 8].

In terms of fragility  $m$ , the studied glasses cover a substantial part of the Angell diagram as they represent very fragile (toluene,  $m = 105$ ), fragile (dibutylphthalate,  $m = 85$ ), and intermediate (glycerol,  $m = 53$ ) glass formers. Thus, they can be considered to be representative. The exponential behavior of the reduced DOS is seen both in nuclear inelastic and inelastic neutron scattering data; it holds for molecular glasses, polymers, and proteins. Moreover, the empirical findings are also corroborated by theoretical considerations [8]. These are



**Fig. 2:** (a) DOS of collective motions in dibutylphthalate at 22 K. Reduced DOS of collective motions in (b) dibutylphthalate and (c) toluene at 22 K (●, ●) in comparison to reduced total DOS (●, ●) from neutron data [3,4]. The neutron data are scaled to match our data at lowest energy. (d) Reduced total DOS from neutron data for orthoterphenyl (otp, ●) [5], polybutadiene (pb, ●) [6], and myoglobin (mb, ●) [7]. Solid lines show the fit according to **Eq.(1)**. The arrow indicates the energy of the boson peak  $E_B$ .

strong indications that the exponential behavior of the reduced DOS of collective motions is a universal feature for glasses.

- [1] A. I. Chumakov, I. Sergueev, U. van Bürck, W. Schirmacher, T. Asthalter, R. Ruffer, O. Leupold, and W. Petry, *Phys.Rev.Lett.* (2004).
- [2] see, e.g., recent review in A. I. Chumakov and W. Sturhahn, *Hyperfine Interactions* **123/124**, 781 (1999).
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- [7] W. Doster, S. Cusack, and W. Petry, *Phys. Rev. Lett.* **65**, 1080 (1990).
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## Machine highlights

ESRF front-end high power absorber. The radiation traversing the absorber in the upper part of the slit falls on a piece of glidcop at grazing angle, while the radiation traversing the lower part of the slit is transmitted through the absorber. The absorber is equipped with bellows on each side and the radiation is shut on and off by moving it up and down. The slit aperture is only 2 mm wide and allows a complete collection of the undulator central cone. The radiation outside of this aperture is absorbed upstream of the absorber by a fixed slit absorber. Such a movable absorber can take heat load from undulators larger than  $400 \text{ kWmm}^{-2}$  and, together with the fixed slit, it can stop a total power higher than 12 kW.

## ESRF HIGH POWER UNDULATOR FRONT-END

**D**uring the construction phase at the ESRF, there were two types of ID front-end: those designed for wigglers (large total power, medium power density), and those for undulators (medium total power, high power density). In both cases the maximum current was 100 mA and the minimum magnetic gap 20 mm. Consequently, the design specifications were 15 kW total power and  $140 \text{ kWmm}^{-2}$  of power density. Other than the few cases of UHV beamlines, nearly all front-ends were equipped with beryllium windows that needed a

carbon filter to limit stress and temperature rise in the beryllium. It was soon realised that surface roughness, impurities or porosities in the graphite were a source of small angle scattering (or decoherence) that prevented the beamlines from benefiting from the ultra low vertical emittance (30 nm in a routine operation, 9 nm at best). Nowadays, undulators installed in an ESRF five metre long straight section with a minimum magnetic gap of 11 mm can produce powerful synchrotron radiation with a power density of about  $300 \text{ kWmm}^{-2}$  and a

total power of 11 kW at 200 mA current. Two in-vacuum undulators recently installed on the ID27 straight section will reach a power density of 400 kWmm<sup>-2</sup>. This corresponds to a power density of 2 kWmm<sup>-2</sup> at normal incidence to the beam when located on the absorber. In order to operate the latest undulator designs while preserving the high coherence of the photon beam, a new front-end targeted for a high brilliance high energy undulator was designed,

vacuum joint and only one layer of Glidcop between the coolant and the photon beam.

The Carbon filters and Be window configuration has been replaced by a CVD (chemical vapor deposition) diamond window. CVD diamond presents many advantages. It has a higher thermal conductivity, which limits the peak temperature in the window allowing the window to be leak tight even at full ID power. In

$\alpha_{INC} (^{\circ})$	$P_{A0}(W.mm^{-2})$	$P_{TOT}(kW)$	$T_{MAX}(K)$	$TW_{MAX}(K)$	$S_{VM}(Mpa)$
1.5	49	2 x 6.4	557	366	412
1	33	2 x 6.4	472	342	280
4	52	2 x 3.3	599	359	456

ESRF old and new absorber thermal stress analysis maximum values.

The old absorber has an incidence angle of 4°.

$\alpha_{INC}$  : absorber incidence angle ;  $P_{A0}$ : power density at the centre of the beam projected on the absorber;

$P_{TOT}$  : total power;  $T_{MAX}$  : absorber maximum temperature;  $TW_{MAX}$ : cooling channel wall maximum temperature;

$S_{VM}$  : Von-Mises stress.

tested and implemented on many beamlines. The upgrade consists essentially of the replacement of the X-ray absorber and the filtering system.

The opening angle for the radiation produced by an undulator is small both horizontally and vertically. The total power produced by undulators can therefore be collimated through a pre-slit which intercepts part of the synchrotron radiation outside the central cone in order to minimise the horizontal aperture of the X-ray absorber and its absorbed power.

An upstream pre-slit (2 mm horizontal aperture at 14 metres from the ID centre) and a compact high heat load absorber then replaced the original X-ray absorber. Each of them can stop a maximum power of 12 kW. The X-ray absorber is designed to stop more than 400 kWmm<sup>-2</sup>. The absorber is made using round pieces of Glidcop AL15 copper (SCM metal products Inc.) which have been machined into the required shape by the wire cutting process, to intercept the photon beam at an incidence angle of 1.2° in the vertical plane to the photon beam. The water cooling channels are made outside of the vacuum. The advantages of this approach are that manufacturing is relatively simple; there is no water-

addition, it dramatically reduces the small-angle scattering and therefore preserves the high coherence of the photon beam. The window is composed of two parts, the diamond window and its water cooled copper chamber. The diamond part is made of a CVD foil 300 µm thick that is sealed to two molybdenum rings by a diffusion bonding technique.

The first high-power front-end configuration was installed in January 2000 for the ID10 beamline (Troika). A systematic refurbishment campaign was then established making use of all the short and long shutdowns. In August 2004, twenty beamlines will operate with this new design. ●

JEAN-CLAUDE BIASCI

LIN ZHANG



## LOOKING INTO THE FUTURE

**T**he ESRF Users' Meeting, on 10 and 11 February 2004, focused mainly on the future of the facility, discussing a Long Term Strategy for the next 10 to 20 years. The ESRF Management asked the Users to consider a number of propositions for the renewal of the ESRF's facilities, in order to preserve the ESRF's status as a world-leader in X-ray research. In addition to the plenary session, three workshops, the Young Scientist Award and the prize for the best poster were included in the programme.

### Workshops

#### Phonons in Crystalline Materials

The aim of the workshop was to discuss the contribution of inelastic X-ray scattering and nuclear inelastic scattering with respect to related experimental methods and theoretical approaches, and to provide a forum to discuss future possibilities and challenges. The workshop featured sessions on high-pressure studies, superconductors, phonons in confined geometries, advanced materials and new instruments. The similarities of X-ray and neutron-based experimental techniques and their associated theoretical approaches were emphasised at the meeting.

MICHAEL KRISCH

#### Resonant X-ray Scattering in Electrically-ordered Systems

The aim of the workshop was to discuss the present and future possibilities for RXS investigations of electronic order, including studies of charge, magnetic, and multipolar ordered states.

Starting at Brookhaven in 1988, the subject of RXS is growing, and work is now in progress at the ESRF on a number of beamlines (ID20, XmaS, ID08, ID03), as well as in many synchrotron facilities around the world.

It is clear that the experimental and theoretical activity around the RXS technique has matured and is in a position to contribute actively to the understanding of strongly correlated electronic and magnetic systems, in particular those where different microscopic processes

Gallery of events



compete in terms of ordered parameters.

The final session of the workshop was devoted to emerging areas such as soft-energy RXS, inelastic RXS and the sample environment for extreme conditions (high pressure and high magnetic fields). It highlighted the future for this technique, and how this field will benefit greatly by an increase in the brilliance of the machine.

LUIGI PAOLASINI

## Around the Cell Nucleus

The workshop covered all aspects of the cell nucleus, spanning topics from the dynamics of the entity as a whole right through to individual assemblies of macromolecules and their mechanisms in DNA storage and nuclear trafficking. The scientific results presented came from a wide palette of methods, including several

microscopic techniques (optical, fluorescence, electron and X-ray). It was clear that considerable advances have been made in the field recently although the nature of chromatin folding in the nucleus was still unsettled. Also, a number of impressive and novel X-ray crystal structures were reported. The information gleaned from these has shed light on how the proteins of the nucleus operate together. Other experimental techniques included fibre diffraction, small angle X-ray scattering and neutron scattering. One of the brightest highlights of the workshop was the crystal structure of Nuclear Core Particles, which are linked together by their DNA (T. Richmond *et al.*). These important structures bridge the gap between the chromatin fibre studies and the atomic resolution structures of chromatin proteins and DNA. ●

WILLIAM SHEPARD

# The Young Scientist Award 2004

## Anton Plech

Time-resolved X-ray scattering studies carried out by Anton Plech gained him this year's Young Scientist Award. "This prize encourages me to keep on doing science, it is a confirmation that what I am doing is the right thing", explained Plech. Anton Plech is a researcher at the University of Konstanz. He was awarded the prize for his "time-resolved X-ray scattering studies of nano-particle dynamics and visualization of chemical reactions in solutions with picosecond time resolution".

This scientist took the first steps in his career at the ESRF, when he had a post-doctoral position at the ID09

beamline. Michael Wulff, his former boss, proposed him as a candidate for this year's prize.

Anton Plech, who often comes to the ESRF as a user, is a good spokesman for synchrotron facilities: "The ESRF offers outstanding possibilities for research", he says. The prize for the best poster went to Thomas Roth and his colleagues for their poster "The Nuclear Lighthouse Effect". ●



Keijo Hämäläinen, previous Chairman of the Users' Organization gives the prize to Anton Plech.

M. C.

## Observing the formation of iodine molecules with X-rays

Anton Plech's latest research was recently published in the American journal "Physical Review Letters". Two teams of scientists led by Michael Wulff at the European Synchrotron Radiation Facility (ESRF) in Grenoble and by Savo Bratos from the University Pierre et Marie Curie in Paris, have measured how two atoms team up and form a diatomic molecule. In their new study, which uses short flashes of X-rays from the synchrotron, iodine molecules in liquid CCl<sub>4</sub> were split into atoms by a small explosion produced with a very short laser pulse. As the molecules are engaged within a liquid, the liquid prevents most of the emerging atoms from splitting apart. After a few collisions with the CCl<sub>4</sub> molecules in the cage, a new but strongly vibrating molecule is born. Very short X-ray pulses, only 100 ps long, *i.e.* a tenth of a billionth of a second, were used to measure the distance between the atoms as the newly-formed molecule cools down forming stable iodine.

A. Plech *et al.*, *Visualising Chemical Reactions in Solution by Picosecond X-ray Diffraction*, *Physical Review Letters*, **92**, no 12, 125505-1 to 125505-4, (2004).

## Latest workshops at the ESRF

The organisers of recent ESRF workshops give their impressions

### EUROPEAN WORKSHOP ON NUCLEAR RESONANT SCATTERING FOR THE STUDY OF NANOSCALE STRUCTURES

Rudolf Ruffer

*Head of the High Resolution and Resonance Scattering Group*

Ralf Röhlsberger *HASYLAB at DESY*

#### What was the context of this workshop?

The enormous brilliance of modern synchrotron radiation sources has opened new avenues for the characterisation of low-dimensional systems like thin films and nanoparticles. In this field, the technique of nuclear resonant scattering enjoys a growing interest with applications in many disciplines of natural sciences.

It is the virtue of this technique to probe magnetic order as well as dynamical properties in basically the same experimental setup. The use of isotopic probe layers, for example, allows one to determine these properties with very high spatial resolution; focusing techniques and X-ray interference effects lead to a very high sensitivity for smallest amounts of material. This rapid increase of applications together with technical developments made the workshop very timely.

#### What was the aim?

The scope of this workshop, jointly organised by the ESRF and DESY, was:

- To summarise recent results obtained via nuclear elastic, quasi-elastic and inelastic scattering from nanoscale structures
- To identify those fields of applications which will grow in the future
- To identify new/further fields of applications which may benefit from the nuclear resonant scattering technique.

Further workshops will follow in the context of the European programme Nanotechnologies and Nanosciences.

## BIOXHIT WORKSHOP ON AUTOMATED X-RAY PROVISION

Sean McSweeney *Head of the Macromolecular  
Crystallography Group*

Olof Svensson *Technical Beamline Support Group*

### What was the aim?

The main goal of the workshop was to create collaborations in order to develop integrated software that can be shared in general by synchrotron radiation facilities and in particular by BioXHIT (see page 11) participants.

### What was the outcome of the workshop?

The result of the workshop was the creation of projects that are suitable for collaborative efforts. The general feeling of the participants was that the workshop was timely since automation is a key part of the planning of macromolecular beamlines at Synchrotron Radiation facilities and the cooperation afforded the most economical means of achieving the Macromolecular Crystallography communities goals.

[www.esrf.fr/Conferences/BioXHIT/FinalProgramme](http://www.esrf.fr/Conferences/BioXHIT/FinalProgramme)

## SECOND WORKSHOP ON METROLOGY FOR X-RAY OPTICS

Olivier Hignette *Optics Group*

### What was the aim?

To share the experience of the metrology community specialised in synchrotron radiation X-ray optics with emphasis on present laboratory procedures and detailed technological developments and focus on the limits and achievements of instruments with respect to the needs of the end users.

### What were the main subjects of the workshop?

The long trace profiler (LTP), the basic instrument of the community, is still the subject of many developments in the US, Germany, France and India with precisions well below 100 nanoradians. But new architectures such as stitching interferometry or Schack Hartmann show promising results. These instruments are used to drive the processes used for figure finish of the mirrors used in X-ray optics. Ions are used in Germany and Emission machining (EEM) in Japan with current results at about one-nanometre residual errors and goals of micro focusing mirrors with 30 nanometres spot sizes at SPring8.

[www.esrf.fr/UsersAndScience/Experiments/Optics/MirrorAndMetrology/MetrologyFiles](http://www.esrf.fr/UsersAndScience/Experiments/Optics/MirrorAndMetrology/MetrologyFiles)

## IMPROVING TECHNOLOGY TRANSFER POLICIES

Manuel Rodríguez Castellano *Head of the Industrial and Commercial Unit*

### Why did the Technology Transfer workshop take place?

The workshop was organised to bring together people from different European research centres, to share experience in order to improve individual and collective capacities to implement appropriate policies on technology transfer in our organisations. The attendance was beyond expectations.

### Will there be other workshops like this one in the future?

The participants clearly expressed their wish to organise other similar events in the future, focused on more specific topics, such as patents, creation of start-ups or remuneration of inventors.

[www.esrf.fr/Industry/Seminars/TTpostWorkshop](http://www.esrf.fr/Industry/Seminars/TTpostWorkshop)

# Looking back



## TEN YEARS OF USERS AT THE ESRF

Ten years ago, the first users came to the ESRF. Here is an excerpt of an article written at that time by Roselyn Mason, Head of the Users' Office.

"The ESRF was proud to welcome its first users, as planned, on 1 September 1994. This marks the beginning of the official user programme. The first teams arrived to work on the Troika Beamline, Beamline 9, the biocrystallography station on Beamline 4 and the surface diffraction Beamline, Beamline 7.

Both the machine and the beamlines have run smoothly and efficiently. Users have been pleasantly surprised by the long lifetime of the beam and its positional stability, by its high brilliance and the high flux at the sample, as well as the reliability and smooth operation of the beamline equipment. Many users have commented that they have left the ESRF with far more and better experimental data than they had expected."

*Article from Roselyn Mason in the Newsletter of November 1994.*