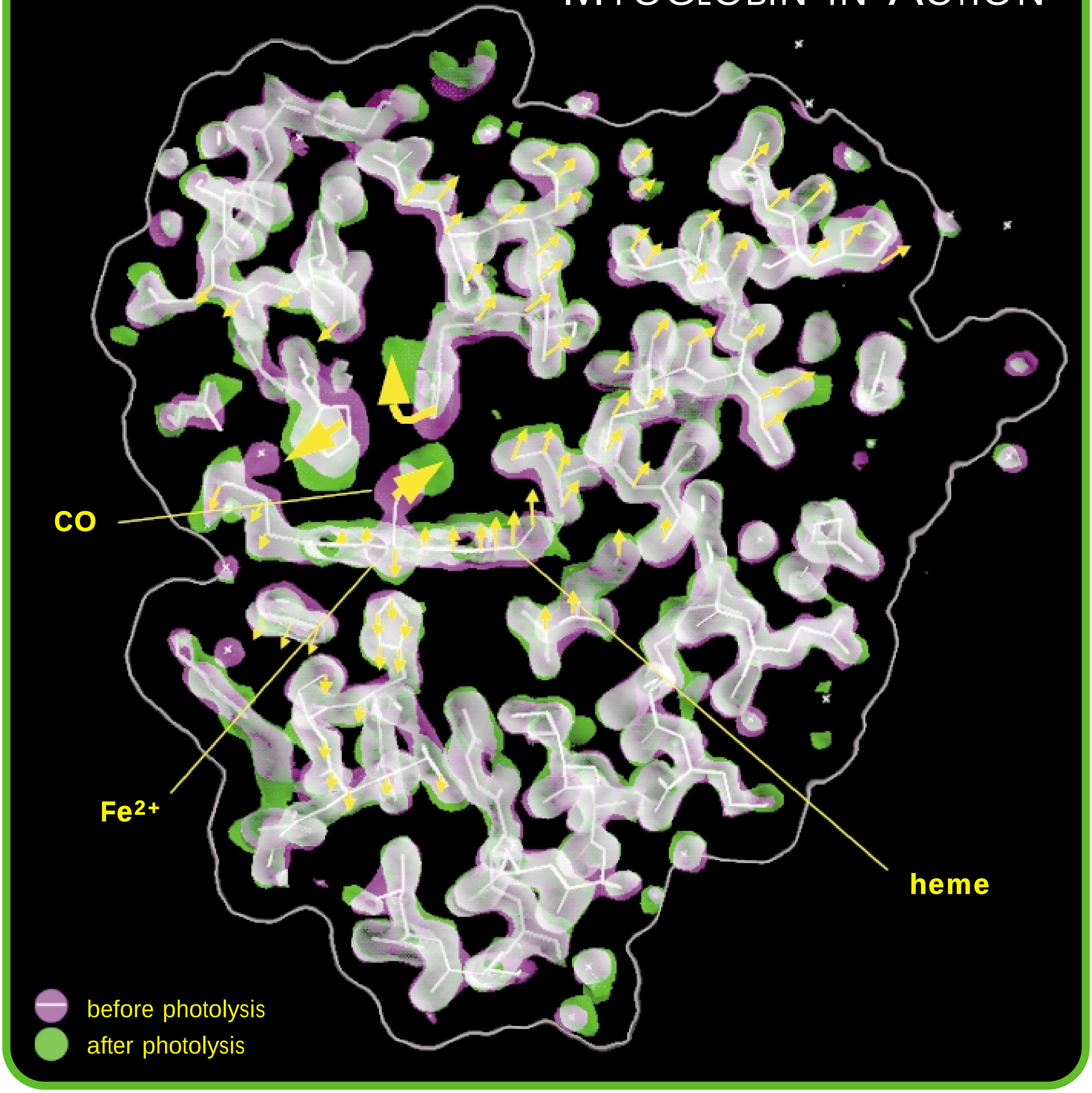
ESRF
# NEWSLETTER
EUROPEAN SYNCHROTRON RADIATION FACILITY

July 2003 · N° 37

## Myoglobin in Action

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# Contents




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# Spotlight on...

## Watching an ultra-fast biological reaction essential to life

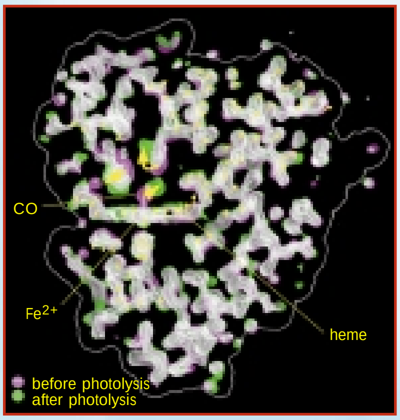
***A team of scientists from the USA in collaboration with staff at the ESRF have succeeded in filming a protein at work in unprecedented detail. The protein is the oxygen-storing molecule myoglobin, which plays a central role in the production of energy in muscles. The motion of the protein was recorded using ultra-short flashes of X-rays. The new insight to the functionality of myoglobin has led to a deeper understanding of the molecular processes associated with breathing. This study has just been published in Science (20 June 2003).***

*Every time we contract a muscle, myoglobin releases oxygen which is used by all mammals for the production of energy. Muscle cells use myoglobin as a peak-load buffer when blood cannot supply oxygen fast enough, for example when the circulation is blocked during muscle contraction. The oxygen molecule is initially confined in a cavity called the heme-pocket, where it is chemically bound to an iron atom.*

*The three-dimensional pictures taken on ID09A (the White Beam Station) locate all the 1432 atoms in the protein. They also pinpoint how the carbon monoxide (CO) molecule – used here as a replacement for oxygen ($O_2$) for technical reasons - literally finds its way out of the very dense atomic structure near the iron atom. Scientists have discovered that the CO molecule does not move out smoothly; in fact it spends most of its time captured in five tiny cavities inside the protein. In the first cavity near the iron atom, the CO molecule makes an extremely brief visit lasting only 100 picoseconds. Iron naturally tends to try to rebind CO, but distal chains, which are parts of the molecule, block the passage so that the CO molecule cannot go back to the iron. The film has shown that the motion between the five cavities is very fast. The CO molecule reaches the fifth cavity after 30 nanoseconds and then it disappears into the solvent surrounding the protein. The interesting thing is that eventually another CO molecule, released from a myoglobin molecule nearby, will diffuse back towards the iron, most likely through another route. The iron accepts the incoming CO due to the fact that the structure of the protein has changed to allow for the rebinding.*

*Watching myoglobin as it functions is more complicated than it may seem. First of all, scientists have to control the start of the experiment extremely precisely. The experiment begins by the injection of a flash of laser light to perturb the molecules and release the CO molecule inside the protein. Very shortly afterwards, they expose the protein crystal to an intense flash of X-rays. The X-rays are scattered by the protein into Laue diffraction patterns.*

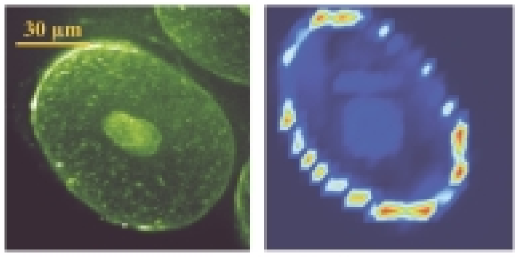

*Schotte et al, Watching a Protein as it Functions with 150-ps Time-Resolved X-ray Crystallography, Science vol. 300, 1944-1947 (2003).*


***Experimentally determined electron densities within a 6.5-Å-thick slice through the myoglobin molecule before (magenta)and 100 picoseconds after (green) photolysis. Where these densities overlap, they blend to white. The white-stick model corresponds to the unphotolysed structure and is included to guide the eye. The direction of molecular motion follows the magenta-to-green color gradient. Three large-scale displacements near the CO-binding site (large arrows) are accompanied by more subtle correlated rearrangements throughout the entire protein (small arrows; not drawn to scale).***

# Editorial

## Future Directions for the ESRF?



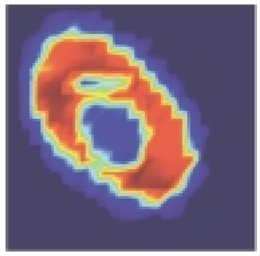



**Fig. 1: The ESRF infrared station will complement X-ray microscopy and fluorescence investigations carried out on ESRF beamlines such as ID21 and ID22. This example shows how the information obtained by calcium mapping of a human hair (left: ID21, courtesy of J. Susini) is complemented by infrared mapping of lipids (top-right) and proteins (bottom-right) (Mirage-LURE, courtesy of P. Dumas).**

*The ESRF is a very busy place. More than forty beamlines cover a wide range of scientific applications and attract about 5000 researchers each year, from some 600 institutes in over thirty countries. This intense scientific activity results annually in at least 1000 articles in refereed scientific journals, written by external users and the ESRF's staff. Work on the beamlines is underpinned by the very reliable operation of the ESRF's X-ray source – the storage ring and accompanying accelerator complex. To a scientist working on an X-ray beamline reliability is of paramount importance. Here, the ESRF's Machine more than fulfils the expectations of our user community. Averaged over 2002, X-ray beams were available for 96.8 % of the scheduled time (98%, if the refill time is included). "Front-end-open" injection, the routine mode of operation since February 2003, should lead to a further increase of the beam time made available for experiments at ESRF. The mean time between failures is also important to our users. A vigorous programme of fault analysis has increased this crucial indicator from 38 hours in 2000, to almost 58 hours in 2002. So currently beamline and X-ray source operations at ESRF are highly efficient and productive.*

*But that is the situation now – what about the future? We have no intention of standing still. The ESRF's scientific strategy for the next five years is laid out in the Medium-term Scientific Programme (MTSP), which is updated annually in discussion with the Science Advisory Committee (SAC) and the ESRF Council. The MTSP contains ambitious programmes involving the development of new research areas (the infrared microscopy station [see **Figure 1**], intense continuous and pulsed magnetic fields, high resolution/high energy photoelectron spectroscopy), and the enhancement of existing fields (the Partnership for Structural Biology, engineering and materials, nanoscience, new numerical methods for theory). In addition, as a leading international research centre, the ESRF must help train today the scientists, engineers and teachers of tomorrow. Both the SAC and Council support the idea of a station dedicated to the training of young people in synchrotron radiation techniques. We consider this to be an essential investment in the future.*

*Where the longer term is concerned, ESRF staff have begun to consider strategic choices for a period of, say, the next ten years. Crystal ball gazing is a notoriously inexact science, but nevertheless we must attempt to identify the critical issues facing the ESRF over the next decade. The ESRF's mission (so far) has been to construct, develop, and operate the X-ray source and the beamlines, all at the highest possible level of scientific effectiveness. Will this mission remain the same over the long-term? Should the ESRF continue much as before or should we focus on new scientific areas? If so, what are these new areas? There are many such questions to be asked in a discussion involving all groups and services of the ESRF, in cooperation with the external scientific community.*

*These discussions take place in an increasingly complex international scientific environment. At a time when many new third-generation synchrotron radiation sources are coming on line or are under construction, there is a strong pressure on science budgets across Europe. This makes it incumbent on us to demonstrate clearly the unique capabilities of the ESRF and our complementarity with national sources. Similar considerations apply with respect to the various free-electron laser projects at different stages of development.*

*The ESRF is funded directly by the seventeen partner nations. Nevertheless, we see a modest but increasing involvement of the European Union – currently some 16 FP6 projects involving ESRF staff are under consideration in Brussels. With moves to create a European Research Council, "Europe" is likely to assume an increasingly important role in the funding of science in the wider scientific community and of large scale scientific infrastructures such as the ESRF.*

*The Convention between the "Contracting Parties" (the governments of our member countries) was concluded for an initial twenty year period that ends on 31 December 2007. At that time changes to the membership or the conditions of ESRF membership will become possible. Clearly it is essential that current and potential new members recognise unequivocally the scientific, technical and personnel benefits of being involved in the ESRF. To this end we must demonstrate the potential of the ESRF, with exciting (and demanding) new projects for the future development of the X-ray source and beamlines, to allow innovative and imaginative science. Over the next year, in cooperation with the SAC, the Users' Organisation, the Delegations and the wider user community, we will develop this long-term strategy for the ESRF. This is not an easy task but it is essential if we wish to ensure the long-term future of our unique multinational facility.*

**W.G. Stirling**
***Director General***

# 38TH AND 39TH MEETINGS OF THE ESRF COUNCIL

**held on 25-26 November 2002 and 16-17 June 2003**

## New Members to the ESRF Company

The Council took note of the changes of Membership (DESY replacing the Forschungszentrum Jülich from 1 January 2003 and CCLRC replacing EPSRC from 1 April 2003), and approved the corresponding amendments to the Statutes of the ESRF.

## Scientific Programme and PSB

The Council noted a document presented by Management on **emerging scientific opportunities** at the ESRF. The scientific trends described in this document referred specifically to the convergence of different methods in X-ray science and to the application of synchrotron radiation to new areas.

In addition the Council noted a preliminary **Medium-term Scientific Programme for the period 2004 – 2008** showing perspectives for the further development of the radiation source and the scientific infrastructure, including an indication of the costs involved.

The Council supported Management's intention to establish an **Integrated Structure for Synchrotron Radiation Training** at the ESRF, under the condition that the operation of this structure will be self-financing.

The Council agreed that the maximum number of **permanent scientific appointments** be raised from 35 to 40 during the next two years and appointed new members to the Standing Committee on Permanent Scientific Appointments.

Concerning the **Partnership for Structural Biology (PSB)**, the Council requested, in consideration of the long term issues involved with the PSB,

• more detailed information on the Institut de Virologie Moléculaire et Structurale (IVMS) and its relationship with the PSB,

• that the contractual arrangements concerning the construction, property and conditions of the later use and share of costs of the common PSB building be submitted to the Council before their implementation.

## Beam Time Allocation and Scientific "Juste Retour" (Fair Return)

The Council

• noted Management's report on **beam time allocation**, including a new proposal of how to deal in the future with Management Contingency beam time,

• set as medium-term goal that approximately half of the current machine operating time (*i.e.* ~ 425 shifts per year) multiplied by the number of beamlines in operation (including public use of CRG beamlines) be made available as **peer-reviewed user time**,

• noted the current **scientific** *juste retour* situation, which continued showing an over-proportional use by the Nordsync community and, for the first time, by the UK community,

• noted that, therefore, the Nordsync consortium and the UK will make **additional contributions** to the 2004 budget over and above their pro rata share.

## Scientific Partners and Collaborating Research Groups

The Council

• approved that a new arrangement on the long-term use of synchrotron radiation is made with the Government of **Portugal** (this was signed on 10 January 2003),

• noted the conclusion of a new arrangement on the medium-term use of synchrotron radiation with the Institute of Physics of the Academy of Science of the **Czech Republic**,

• authorised the Director General to undertake negotiations with the **Israel** Academy of Sciences and Humanities with a view to a renewal of the current arrangement for a further period of five years.

The Council approved

• the contracts on the **sale of**

- beamline **BM14** to the British Medical Research Council (MCR) and

- beamline **BM16** to the Spanish Ministry of Science and Technology (MCT),

as well as the corresponding contracts on the operation of these new CRG beamlines for five years,

*From left to right: Jochen Schneider, Bill Stirling, Christoph Kratky and Helmut Dosch.*

• the extension of the contract concerning the construction of the **Spanish CRG beamline BM25** by another two years,
• the extension of the contracts with the Collaborating Research Groups **XMaS** and **ROBL**, concerning the operation of the beamlines BM28 and BM20 respectively, by another five years.
The Council authorised the Director General to enter into negotiations with the INFN with a view to an extension of the contract concerning the operation of the **GRAAL** experiment at the ESRF.

### Financial Matters

The Council
• approved the final accounts for the financial year 2001 and discharged the Director General with respect to the implementation of the **budget for 2001**;
• approved the award of the new audit contract, for a period of five years, to the company CONSTANTIN ASSOCIES;
• approved the **budget for 2003** as presented by the Management, providing for expenditure of 73 391 k€ in payments and requiring Members' contributions of 64 402 k€;
• took note of the Medium-Term Financial Estimates (2004-2008) and noted the comments and reservations made by the various Heads of Delegation concerning the likely availability of funds to support the planned ESRF **budget of 2004**.

### Appointment of Directors, Chairpersons and Committee Members

The Council
• decided on the appointment of a new **Director of Research**,
• re-elected R. Comès as **Council Chairman** and R. Feidenhans'l **as Vice-Chairman** for the period from 1 January 2004 to 31 December 2005,
• noted the direct nomination of thirteen scientists by the Members and Scientific Associates for **membership of the Science Advisory Committee** for the period from 1 January 2003 to 31 December 2004, and appointed a further ten scientists to achieve a satisfactory scientific coverage of the scientific themes for the ESRF.

**Karl Witte**

## Sine Larsen, new Director of Research

***Professor Sine Larsen of the University of Copenhagen, Denmark, has joined the ESRF as Director of Research on 1 June 2003. Professor Larsen is a distinguished crystallographer whose work encompasses both structural biology and structural chemistry. She has specialised in the areas of chiral chemistry, charge density studies, and structural biology. After appointments at M.I.T. and the Danish Technical University, in 1974 Professor Larsen joined the University of Copenhagen where she became Director of the Centre of Crystallographic Studies (in 1994), and Professor of Structural Chemistry (in 1997). She has served on many international committees and is General Secretary and Treasurer of the International Union of Crystallography.***

### Members of the Science Advisory Committee for the years 2003/2004

R. Abela, (Paul Scherrer Institut, Villingen),
J. Bordas (Laboratori de Llum de Sincrotró, Barcelona) until June 2003,
L.Braicovich (Politecnico di Milano),
B. Capelle (Laboratoire de Minéralogie-Cristallographie, Paris),
J.L. Carrascosa (Centro Nacional de Biotecnología, Madrid) from July 2003
J. Daillant (LURE, Orsay)
G. Fiquet (Laboratoire de Minéralogie-Cristallographie, Paris),
J.P. Gaspard (Université de Liège),
J. Hastings (Stanford Linear Accelerator Center),
D. Juul Jensen (Risø National Laboratory, Roskilde),
A. Korsunsky (University of Oxford),
G.H. Lander (Institute for Transuranium Elements, Karlsruhe),
P. Monceau (CRTBT - CRNS, Grenoble)
U. Pietsch (University of Postdam),
H. Reynaers (Katholieke Universiteit Leuven),
G. Rossi (Laboratorio Nazionale TASC – INFM, Trieste),
G. Ruocco (Universita di Roma),
G. Schmahl (Universität Göttingen),
G. Schneider (Karolinska Institutet, Stockholm),
H.P. Steinrück (Universität Erlangen-Nürnberg),
J.L. Sussmann (Weizmann Institute of Science, Rehovot),
T. Ryan (University of Sheffield),
E. Weckert (HASYLAB, Hamburg),
K. Wilson (University of York).

# Jean-Louis Laclare

It is with regret that we learnt of the passing away on 17 April 2003 of Dr Jean-Louis Laclare, Project Leader then Machine Director of the ESRF from 1986 to 1996.

Jean-Louis will be remembered by many at the ESRF as a remarkable leader. His career began in 1969 in the Corpuscular Optics Section of "Saturne", a former laboratory of the French Atomic Energy Commission in Saclay, near Paris. Following his thesis on the design of a stretcher ring (ALIS) for the Saclay electron Linear Accelerator, he headed for Canada and worked on two similar projects. Back at Saturne, he worked on a 100 MeV mini electron ring project to be used for metrology. In 1972, as head of the Optics Section, he coordinated the Machine Physics aspects of the renovation of Saturne. This consisted of replacing the old 3 GeV proton synchrotron in use since 1958 by a new strong focusing synchrotron which was considered at that time as highly innovative. In 1974, he became head of the Machine Physics Group and commissioned the new machine in 1978. Full energy performance was reached in 1979 once he had solved the problem of longitudinal instabilities. Over this period he acquired an international reputation by publishing a large number of papers on the subject of coherent instabilities, phenomena which limit the performance of most particle accelerators. He then further developed the Saturne injector system with the design and commissioning of an innovative Radio Frequency Quadrupole in 1984 and a new injector of the synchrotron type MIMAS.

*From left to right: N. Lawrence, J.L. Laclare, R. Haensel, A. Miller and G. Mülhaupt, at the start of the ESRF in 1987.*

In 1986, Jean-Louis Laclare was appointed Project Director of the ESRF, *i.e.* the person charged by the Director General with the organisation of all work related to the construction of the synchrotron radiation source (accelerator complex, buildings and technical infrastructure). Two major challenges faced him:

• to build - and make work - the world's first third-generation synchrotron radiation source within the scheduled time and allocated budget,

• to deal with all the surrounding administrative and infrastructure aspects involved in creating a world-class institute from scratch.

He did both to perfection. Rapidly putting together a team, made up mostly of young beginners who under his leadership soon overcame their lack of experience to make a valuable contribution to the project, he inspired all his colleagues to attain the above goals, an "obsession" to which he constantly strived. The construction started in 1988, the target specifications of 100 mA, 7nm horizontal emittance, 10 h lifetime and $10^{18}$ photons/s/0.1%BW/mm$^2$/mrad$^2$ brilliance from an undulator being reached in mid 1992, 6 months ahead of schedule and (proudly) within budget. The ESRF light source was born! These specifications may appear modest by today's standards but at that time they were considered as quite speculative by a number of accelerator experts. But Jean-Louis Laclare did not stop there. He immediately perceived that much higher brilliance, lifetime and current as well as much lower emittances were attainable. Within a few years of development and by the time he left the institute, the machine's parameters of performance had improved by from two to three orders of magnitude: A magnificent achievement.

In 1996, his vocation of "accelerator builder" moved him to return to Paris to head the SOLEIL project. With his customary drive, the project was ready to go ahead in 1998 with the site specifications, building design, accelerator components on the critical path ready for tenders, and 13 out of the 24 beamlines of the programme selected and pre-designed. Unfortunately the project was held up by the Government, so he moved on to work on neutron facilities. From 1999 to 2001, he was project leader of CONCERT, a joint undertaking of the CEA and the European Spallation Source, to carry out the study of a multipurpose facility driven by a High Power Accelerator of the 25 MW class and capable of serving simultaneously a spallation neutron source, radioactive beam source for nuclear physics, a test bunch for muon-neutrons production, and a demonstrator for transmutation of nuclear wastes. In 2001, the CONCERT and ESS teams were merged with Jean-Louis Laclare as the new ESS Project Director. When the CEA left the ESS project late 2002, he joined the project team for the Spiral2 extension to the Ganil Facility in Caen. He was still actively concentrating on this last project on the day of his departure.

Jean-Louis Laclare will be remembered as an outstanding accelerator builder and project manager and as a true and dedicated fighter. Despite his heavy workload, he liked to pass on his scientific expertise by teaching in accelerator schools and training new young physicists. He was always available to explain with simple words complex topics like beam instabilities. He will be missed by all the colleagues who had the chance of working with him and learning from him. Through his major accomplishments, the synchrotron scientific community is greatly indebted to him.

**Pascal Elleaume**

# THE 13TH SESSION OF THE HERCULES COURSE

## 2 March to 11 April 2003

The HERCULES Courses (Higher European Research Course for Users of Large Experimental Systems) aim to train young European researchers (PhD students, post-doctoral scientists, …) to optimise the present and future use of "neutron and synchrotron radiation" by taking advantage of state-of-the-art instruments for their research work. From its inception in 1991 the main emphasis of the HERCULES annual sessions has resided on the coupling of practicals carried out at four Large Instrument partners (ESRF, ILL, LURE, LLB) with lectures and tutorials. At the end of the 2003 session more than 900 young researchers (about 800 from the E.U.) will have attended the sessions, out of about 1500 applicants.

The specificity of the HERCULES programme is to offer a basic theoretical and experimental training to a multidisciplinary audience composed of young biologists, chemists, physicists, geoscientists, industrial scientists, at the scientific site in Europe where two European Large Instruments (ESRF and ILL) are located. The participants can therefore know and use the exceptional infrastructure, as well as its associated high-level human potential. HERCULES also helped to build a European network of young researchers using neutrons and synchrotron radiation for condensed matter studies (HERCULES web site).

The HERCULES programme is supported by the European Commission, the French Ministry of Education and Research, the CNRS and the CEA, the four partners (ESRF, ILL, LURE, LLB) and the synchrotron radiation and neutron Round Tables.

This year, during the six week period from 2 March to 11 April 2003, Grenoble hosted the 13th session of this HERCULES course. The school continues to attract a large number of scientists: 65 full-time and 6 part-time participants took part in the 13th session. This year the selected PhD students and post-doctoral scientists came mainly from the European Community, but also from eastern Europe (Poland, Czech republic, Russia,..), from America (US, Cuba) and from Asia (Taiwan).

*Attendees of the HERCULES 2003 course on neutron and synchrotron radiation for "physics and chemistry of condensed matter" & "biomolecular structure and dynamics"*

The students were divided into two sessions:

• Session A for Physics and Chemistry of Condensed Matter, with 44 full time participants,

• Session B for Biomolecular Structure and Dynamics, with 21 full time participants.

The HERCULES Course includes lectures, practicals and tutorials, visits to laboratories and a poster session. The integrated practical training represents about 40% of the training time of these sessions. Tutorials and specific practicals using beam were carried out at the ESRF and LURE for X-ray studies, and at the ILL and LLB for neutron experiments.

For hands-on training, the students were divided into 17 experimental groups (4 students per group) and carried out experiments and data analysis set up by more than 100 local instructors. In this way, the students could establish contact with the scientists in charge in the large-scale facilities, and therefore become familiar with the potential there.

The schedule was quite dense. However, there was time for the participants to enjoy some of Grenoble's sounds and Alpine sights. The school gave the students other opportunities for mingling with each other at the poster session, the skiing outing and the dinner party, plus the "classic" Wine and Cheese final party.

**New! For the 2004 session, the HERCULES Course will also include, over the six week sessions, a special one-week programme at ELETTRA (Trieste), dedicated to soft X-rays, ultraviolet and infrared radiation and their new fields of applications namely to chemistry, soft matter and biology...**

## HERCULES 2004

Higher European Research Course for Users of Large Experimental Systems

**Grenoble, 22 February - 2 April 2004**
**Trieste or Saclay: 14-20 March**

**Session A: «Neutron and synchrotron radiation for physics and chemistry of condensed matter»**
**Session B: «Neutron and synchrotron radiation for biomolecular structure and dynamics»**

**Information:**
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**Deadline for application: 24 October 2003**

# FP6: Making the future of European science brighter

## 11-13 November 2002

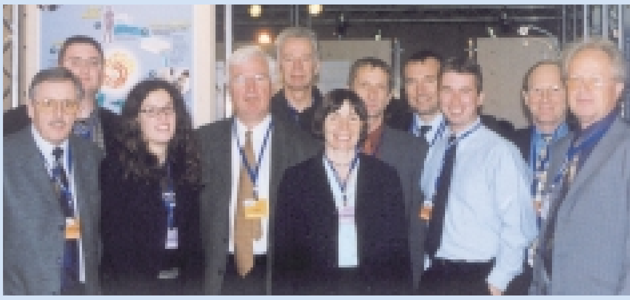
Europe has a long-standing tradition of excellence in research and innovation, and European teams continue to lead progress in many fields of science and technology. However, its centres of excellence are scattered across the Member States and all too often their efforts fail to add up in the absence of adequate networking and co-operation.

In order to strengthen European research, the 6th Framework Programme encourages the development of a true European scientific community equipped with the best skills and know-how, and supports scientific and technical work of the highest quality conducted through transnational projects.

The 6th Framework Programme will also greatly contribute to the creation of the European Research Area (ERA), a genuine European internal market for research and knowledge, where EU and national R&D efforts are better integrated.

A large conference was organised in Brussels in November 2002 to launch the 6th Framework Programme and the European Research Area. The 3-day event attracted more than 8000 participants from 61 countries and 200 speakers, including a line-up of 20 ministers, 3 Nobel laureates and scores of business leaders.

A group of thirteen ESRF staff members attended this event with the objective to investigate how the ESRF could become more involved in the EU programmes and make the institute more visible to the scientific community, industry and worldwide. Another goal was to make contacts with scientists, possible industrial partners and anyone interested in the laboratory. The ESRF is already very active, being involved in a total of 16 proposals to FP6, on nanotechnologies, biology and information technology. But its participation in the integrated projects and networks of excellence can certainly be improved. An additional benefit of being present in Brussels was the attendance at the conferences that took place during these three days. They went from subjects related specifically to FP6 and how to contribute to the programme, to talks about purely scientific matters (nanotechnologies, genomics and biotechnology, etc.), mobility of researchers or science and society.



*Part of the ESRF group that attended the FP6 Conference in Brussels.*

*The representatives of each of the EIROforum organisations, together with commissioner Philippe Busquin, at the signature ceremony.*

# EIROforum is officially born

The FP6 Conference also constituted the framework for the official signing of the EIROforum Charter by the Directors General of each of the seven leading European research organisations that constitute this coordination and cooperation group*.

A primary goal of EIROforum is to play an active and constructive role in promoting the quality and impact of European Research. It will mobilise its substantial combined expertise in basic research and in the management of large international projects for the benefit of European research and development. This will be possible by exploiting the existing intimate links between the member organisations and their respective European research communities. There are already five working groups set up, focused on different specific aims: Outreach and Education, Instrumentation, Scientific Workshops, Human Resources and Grid Technologies.

** EIROforum members are the European Organization for Nuclear Research (CERN), European Molecular Biology Laboratory (EMBL), European Fusion Development Agreement (EFDA), European Space Agency (ESA), European Southern Observatory (ESO), European Synchrotron Radiation Facility (ESRF) and Institut Laue-Langevin (ILL),*

*Ed Mitchell shows a visitor a 3D protein structure.*

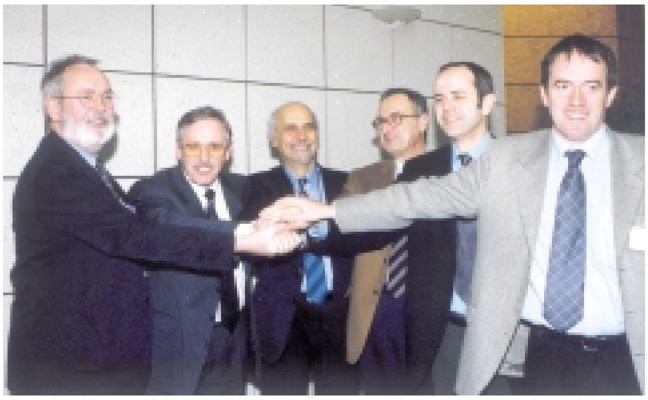
# Launch of the Partnership for Structural Biology

## 15 November 2002

An important step forward in Structural Biology was made at the ESRF on Friday 15 November 2002. The Directors of the ESRF, the ILL, the EMBL and, for the IBS (which is sponsored by the CEA, CNRS and UJF), the Director of the Département Sciences du Vivant of the CEA, the Director of the Département Sciences de la Vie of the CNRS and the President of the University Joseph Fourier, signed a Memorandum of Understanding on the Partnership for Structural Biology (PSB), an integrated programme and resource pool in structural biology, unique in the world.

The ceremony took place in the auditorium, and was attended by representatives of the four partners, as well as many personalities such as Bernard Bigot, Director of the Cabinet of the Minister for Research and New Technologies; Geneviève Fioraso, Deputy to the Mayor of Grenoble; Christine Crifo, Conseillère Générale and Jean Caune, Vice-President of the Métro.

Like much of today's research in biology, the PSB is directed towards a better understanding and treatment of human diseases. The Human Genome Project has produced an enormous body of information about the sequence of the human genome. However, more detailed information on the structure, function and interaction of the tens of thousands of proteins encoded by this genome is required in order to fully exploit this new panoply of data for novel disease treatments. In particular, knowledge of the three-dimensional structures of key human proteins or human pathogens is vital to speed up the difficult task of discovering new antibiotics or anti-cancer drugs.



*The Directors of the institutions constituting the PSB join forces for a prosperous future.*

The idea of setting up a partnership in this field was born two years ago. According to Bill Stirling, "here in Grenoble we have the infrastructure, the equipment and, indeed, the responsibility to contribute to research in structural biology". Indeed, X-ray crystallography at third-generation synchrotron sources, such as the ESRF, is one of the key techniques that will build the database of three-dimensional information on protein structure. The expertise of EMBL, coupled with nuclear magnetic resonance techniques and neutron scattering (specialities of IBS and ILL) constitute further important and complementary structure determination tools.

Individual companies, both established pharmaceutical enterprises and promising start-up biotechnology companies such as Astex Technologies, GlaxoSmithKline, Pharmacia Italia, Ribotargets and Aventis have already joined the Partnership as Associate Members and contribute to its activities and developments. Close interaction between academic and commercial concerns is essential for the rapid development of fundamental discoveries into new medicines.

# FaME38 opening ceremony

## 26 November 2002

The opening ceremony for FaME38 took place on 26 November 2002. This new Facility for Materials Engineering has been set up jointly by a consortium of seven UK universities headed by Salford University, the ILL and the ESRF. The ceremony was attended by engineers and scientists representing member countries from across Europe. The guests were welcomed in speeches given on behalf of the ILL by Colin Carlile and for the ESRF by Bill Stirling. Mme Fioraso, Deputy to the Mayor of Grenoble, represented the City and the Département de l'Isère, from which FaME38 takes the '38' part of its logo. The basic philosophy and resources of FaME38 were described by the project manager, Peter Webster from Salford University, the institution that is responsible for the administration of the project. The Vice-Chancellor of the University, Michael Harloe, spoke of the close connections that Salford has with the ESRF and ILL. He referred to some famous Salford-born individuals including James Prescott Joule, whose name is now used as the SI unit of energy, and the industrial landscape painter L. S. Lowry, some of whose prints can be seen on the walls of FaME38. He then 'opened' the laboratory by cutting a chain using a large pair of industrial shears. The opening was followed by a demonstration of equipment, a display of posters from materials engineering users of ESRF and ILL and concluded by a champagne reception. In the evening a dinner was held at the Château de la Commanderie.

The following day a FaME38 technical Forum was held for engineers and scientists involved in the project. The facility is now open to users, in particular for all academic and industrial engineers with interests in residual stress measurements, who are invited to contact FaME38 at FaME38@esrf.fr or to visit the web-site www.ill.fr/FaME38.

# 13th Users' Meeting and satellite workshops

## 10-14 February 2003

The 13th Users' Meeting and associated Workshops attracted 366 participants from Europe, the USA and Japan. The Users' Meeting day, which took place in the Grenoble World Trade Centre, included scientific talks, the Young Scientist Award, posters sessions and discussion groups about the main scientific areas at the ESRF.

The workshops also provide a good opportunity to learn about various fields of research. This year, they concentrated on Inelastic Scattering, Membrane Structure and Function, and Detectors for Beamlines (see reports hereafter).

On the Users' Meeting day, there were four talks highlighting some very recent advances made employing synchrotron radiation. W. Eberhardt, from Bessy, Germany, described the "Study of magnetism and magnetic materials using synchrotron radiation". Anton Plech, from the University of Konstanz, Germany, focused on "Resolving structural kinetics on the picosecond time scale". The following talk, "The hard X-ray microprobe: elucidating composition/ structure relations in real-life materials", was given by K. Janssens, from the University of Antwerp, Belgium. Finally, Miquel Coll, from the CSIC, Spain, spoke about "DNA transfer machinery".

The Users' Meeting was not only about scientific plenary talks. It was also about knowing what the scientist next door (or next beamline) is doing. Therefore, an exhibition of 68 posters illustrating different experiments decorated the hall of the conference centre. The scientists working on each of them were happy to satisfy the curiosity of anyone stopping by. At the end of the day, a prize for the best poster went to Olga Kurapova for her poster entitled "Beryllium parabolic refractive lenses".

And still more prizes! The Young Scientist Award offered by the ESRF Users' Organisation went this year to

*Executive Committee of the Users' Organisation.*

Claudia Dallera, from the Politecnico di Milano for her work on high-energy X-ray emission and high-energy photoemission at the ESRF (see below).

This meeting was also full of news. The Users' Organisation announced the change of their chairman for the next two years and also of some of their nine members. Marco Grioni, from the Institut de Physique des Nanostructures in Lausanne takes over the duties from Keijo Hämäläinen. Bill Stirling drew up a balance of the improvements achieved in 2002. He also highlighted some news from the facility and major projects underway and stated that "the ESRF is growing healthily".

## The Young Scientist Award goes to Claudia Dallera

Claudia Dallera, an ESRF User from the University Politecnico di Milano, received this year's Young Scientist Award. The prize recognises her outstanding contribution to instrumentation, spectroscopy and high-energy photoemission. For this young scientist, the first woman to get this prize, it was a real surprise: "I didn't expect it and it certainly has a special meaning to me, since I started my research career at the ESRF".
At present, Claudia works at the Department of Physics of the Politecnico di Milano, where she studies the electronic properties of novel materials by means of X-ray spectroscopies with synchrotron radiation as well as with laser sources.

## Membrane Structure and Function

### 10-11 February 2003

On 10 and 11 February a workshop on the subject "Membrane Structure and Function" was held in conjuction with the ESRF Users' Meeting. This was the first workshop held under the auspices of the recently created Partnership for Structural Biology (http://psb.esrf.fr) and was attended by approximately 100 participants.
Following a brief introduction to the PSB by Stephen Cusack, the scientific sessions covered investigations into membrane transport process, membrane dynamics, electron transport, light harvesting as well as membrane protein characterisation.
Audience participation was enthusiatic throughout, with many excellent discussions following (and sometimes during!) the presentations.

# INELASTIC X-RAY SCATTERING IN DISORDERED SYSTEMS AND SOFT CONDENSED MATTER

**10-11 February 2003**

The satellite workshop on "Inelastic X-ray scattering in disordered systems and soft condensed matter" attracted 80 participants from Europe, USA and Japan. It featured four sessions with invited speakers. A poster session, together with two discussion panels, provided the forum for intensive discussions.

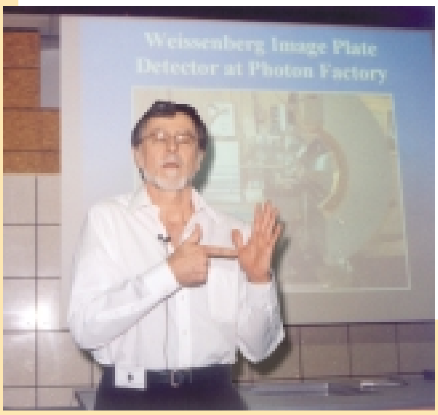
The workshop brought together experimenters and theorists interested in the study of collective dynamics and relaxation phenomena in disordered condensed matter to discuss the contribution to the field of the inelastic X-ray scattering technique as a complement to other experimental methods and theoretical approaches. The workshop also provided a good opportunity to discuss challenging perspectives for future studies on increasingly interesting areas such as materials in extreme conditions, and materials with mesoscopic organisation: polymers, macromolecules, soft matter and biologically interesting compounds.

# X-RAY DETECTORS: "THE WAY TO GET MORE OUT OF YOUR BEAMLINE"

**13-14 February 2003**

The goal of the workshop was to give the ESRF user community a view of the state of the art in X-ray detection world-wide, as well as of future perspectives. The workshop also served as a platform for discussions between detector developers and synchrotron scientists.

An excellent speakers field from Europe, the United States and Japan attracted a total of 120 participants.




*R. Hamelin from Area Detectors System Corporation.*

The workshop was organised in 5 sessions: user perspectives, spectroscopy detectors, gas-filled detectors, CCD-based systems, and "other systems", plus an introduction talk by Jules Hendrix who pioneered large scale imaging detectors. Every session provided both an introduction to the specific technology and an overview of ongoing developments, with ample time for discussions between the presentations.

From the presentations and the discussions it became very clear that the field of X-ray detection is extremely lively and there are many new opportunities. This is to a large part due to new developments and possibilities in micro-electronics which allow for custom design of integrated circuits. Besides this, there are various efforts world-wide in the area of materials and devices for more efficient and higher spatial and energy resolution X-ray conversion. Therefore, from a technological point of view, the field looks very exciting and promising.

A much more delicate issue that became clear during the final discussion is the difficulty for collaborations between institutes and facilities, either within Europe or between Europe and the United States. Due to the absence of appropriate mechanisms it remains difficult to export detectors or even technology to other facilities, and collaborations remain based on person-to-person relationships.

In conclusion all participants agreed that the field of X-ray detectors remains very promising, and efforts should be made in order to benefit from the new technological opportunities.

# Open Days

## Saturday 22 and Sunday 23 March 2003

Almost 2000 people from Grenoble and the surrounding area had access to the secrets of the science carried out at the ESRF during the Open Days (March 2003).

"Interesting", "instructive", "fascinating", "impressive"… these are some of the adjectives the visitors used to describe their stay at the ESRF. During a couple of hours on average, the "grenoblois" found out first about the structure of the synchrotron using the model in the Central Building, then moved on to the real thing inside the ring. A fantastic view from the roof of the tunnel let visitors see the most precious tool of the ESRF: the magnets and insertion devices that produce the beam of light. The scientific experiments carried out using this light constituted the next step of the visit. Three beamlines from three different domains (biology, X-ray imaging and microscopy) were open to the public. An optics stand attracted lots of visitors due to its hands-on tools.

But visitors are not self-taught, so the 76 volunteers who collaborated in the success of this event provided them with accurate explanations about the science done at the synchrotron. According to a visitor, "scientists were very clear and educational, and they definitely set a good example for teachers". From the other side, scientists were also delighted with their new "job": "It is tiring to spend some hours talking without stopping, but it is also very satisfying to see eyes shining with interest!", explains a biologist.

On Sunday afternoon the ESRF got a new look. Loud voices, laughter and running steps in the corridors filled the facility with a breeze of life and energy. The responsibles for this relaxed atmosphere were the children of the ESRF staff, who exceptionally were allowed on the site.


# Modernisation of the ESRF web site

Four staff members from different ESRF departments were brought together to form a team called the Web Task Force with the mission of overhauling and modernising the web site. There was a need to present a web site with a homogeneous appearance and coherent navigation to the outside world, and additionally to provide page editing and management facilities for our many web authors.

The quantity of information available on the ESRF's internet and intranet sites represents several tens of thousands of pages. It was therefore paramount to select appropriate tools for the management of such a large number of pages, in particular for the collaborative editing, updating, and reviewing. The new system is based on Zope, an open-source web application server. It is a content management system that permits the separation of the content from the presentation. The main advantages for our web authors are through-the-web page editing, template-based page creation, and collaborative editing and management of pages and sections.

The new site went on-line in December 2002. Good progress has been made in the conversion of old pages. This task will continue throughout 2003, and already new needs are emerging. These future developments will increasingly tend towards providing dynamic web contents.

Gary Admans, Rudolf Dimper, Francoise König, Marie Robichon

*The new ESRF home page*

# New video: Showing life at the ESRF

A new video on the ESRF has just seen the light. The lenses of a camera captured the atmosphere surrounding a user's experiment. Conceived by the Communication Unit, the film is the final-year project of three Communication graduates from Grenoble. These students dedicated six months to the creation of the film, intended to provide insight into the ESRF.

The making of the film consisted in following three Portuguese users throughout the five days of their experiment, from the moment they arrived at the ESRF Guesthouse up to when they left with their suitcases full of data to be analysed back home. Scientific research aside, there is a whole human dimension to the users' trips to the ESRF. And this is exactly what the film wants to show: intense hours of work, enthusiasm and a host of cooperation between scientists and staff at the ESRF.

The video will be played to groups of visitors onsite and will be sent to schools and universities. The idea is to spread the message that science goes beyond incomprehensible formulae and complicated machines and that it has another side, much more human and motivating.

**If you are interested in obtaining a copy of the new videotape (8 minutes, in English or French), available soon in DVD, please contact the Communication Unit at information@esrf.fr**

*Ondina Figueiredo, Head of the Portuguese group of users, talking to Jean Susini, Head of ID21, the beamline where the experiment took place.*

# Physics on Stage Festival

## 8-15 November 2003

**About 400 science teachers from 30 European countries will meet at ESTEC in the Netherlands from 8 to 15 November to exchange the best practices in science education.**
**The Physics on Stage Festival will be part of the European Science Week, an effort of the European Commission to bring science closer to the public. It is organised by EIROforum (see page 8) with the objective to improve the way science is taught in schools and to make it more attractive to young people.**
**The theme of this year will be "Physics and Life". The ESRF will present examples of experiments carried out in this field.**

**More information**
**on the festival can be found at**
www.physicsonstage.net

# Operation with Users in 2002

During the year 2002, the full complement of 30 ESRF beamlines, together with 8 Collaborating Research Group beamlines (CRGs) were open for user experiments, the CRGs making 1/3 of their beam time available for general ESRF users. For the purposes of scheduling experiments on beamlines, 2002 was divided into two periods, February to July, and August to the User Meeting in February 2003, with run periods of seven to eight weeks, broken by shutdown periods for installations, commissioning and maintenance.

Scientists requesting beam time submit applications for two deadlines – 1 March and 1 September - each year. For the proposal rounds in 2002, all 1489 applications for beam time were submitted electronically. Although the main beamline construction effort was complete by 1999, requests for beam time continued to rise in 2000 and 2001, and remained at a high level, totalling 24 573 shifts in 2002 (see Figure 1). In the past, requests for beam time have outweighed the beam time available for allocation by roughly a factor of two. This pattern continued in 2002, when 11 759 or 48% of the shifts were allocated beam time.

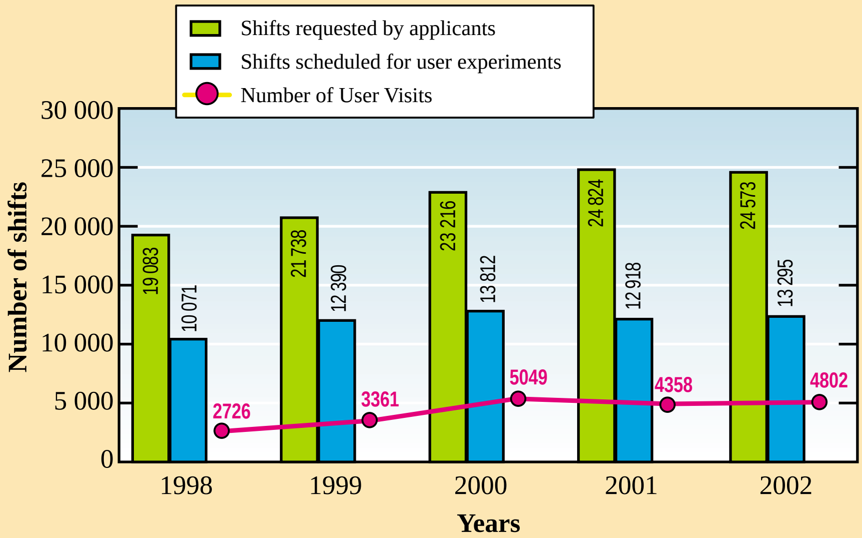



*Fig. 1: Details of shifts requested, shifts scheduled for user experiments and user visits, per year, 1998 to 2002.*

## Experimental programme

Figure 1 also shows the increase in the number of shifts delivered for experiments over the scheduling periods since 1998. In parallel, the number of visits by experimentalists to the ESRF has risen markedly, reaching 5049 user visits in 2000, the peak in 2000 corresponding to a longer scheduling period.

The breakdown of shifts scheduled in 2002, per scientific area, is shown in Figure 2. A feature of this period has been the increasing number of projects concerned with applied research in materials science, engineering and environmental matters. As seen in Figure 2, experiments in these areas accounted for 10 % of the total number of experiments carried out in 2002. In addition, ESRF is attracting increasing numbers of projects relating to medical research, which amounted to 5% of the beam time scheduled. The macromolecular crystallography Block Allocation Group (BAG) scheme continues to successfully cater to the needs for regular access and high throughput for the 33 consortia working in this area.

To cater to the experimental needs of a wide diversity of research projects, beam deliveries to the stations in 2002 were made in a variety of modes:

- multibunch (2 * 1/3 and uniform filling) - 68%
- hybrid mode - 9%
- 16-bunch fill - 19%
- single-bunch fill - 4%.

## Next deadline for proposals: 1 September 2003

Finally, interested readers are reminded that deadlines for proposals for beam time are 1 March and 1 September each year. Further details are available on the Web at www.esrf.fr.

**Roselyn Mason**

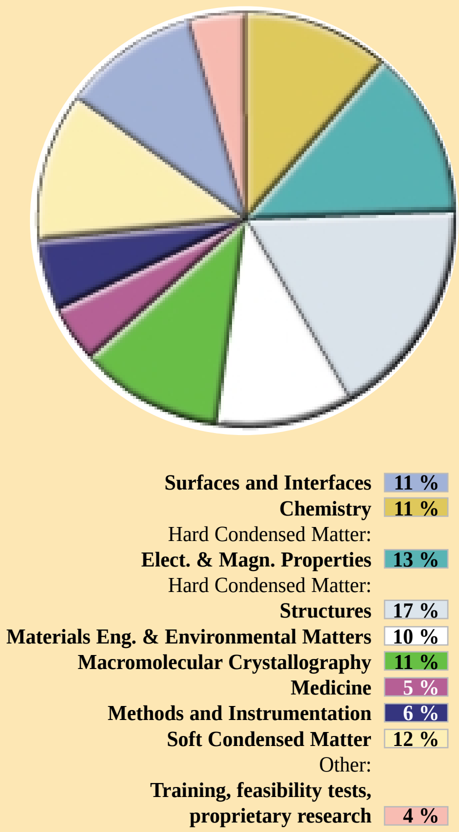



*Fig. 2: Shifts scheduled for experiments in 2002, by scientific area.*

# SELECTED SCIENTIFIC HIGHLIGHTS

**Surface and Interface Science Group**

### ID1 - Direct determination of strain and composition profiles in SiGe islands by anomalous X-ray diffraction at high momentum transfer

*T. U. Schülli[1,2], J. Stangl[1], Z. Zhong[1], R. T. Lechner[1], M. Sztucki[2], T. H. Metzger[2], and G. Bauer[1]*
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*2 ESRF*

Anomalous X-ray scattering is employed for quantitative measurements of the Ge composition profile in islands on Si(001). The anomalous effect in SiGe is enhanced exploiting the dependence of the complex atomic form factors on the momentum transfer. Comparing the intensity ratios for X-ray energies below and close to the K edge of Ge at various Bragg reflections in grazing incidence diffraction setup, the sensitivity for the Ge profile is considerably enhanced. The method is demonstrated for SiGe dome-shaped islands grown on Si(001). It is found that the composition inside the island changes rather abruptly, whereas the lattice parameter relaxes continuously (see **Figure 1**).

Publication:
*Phys. Rev. Lett. 2003, 90, 66105*

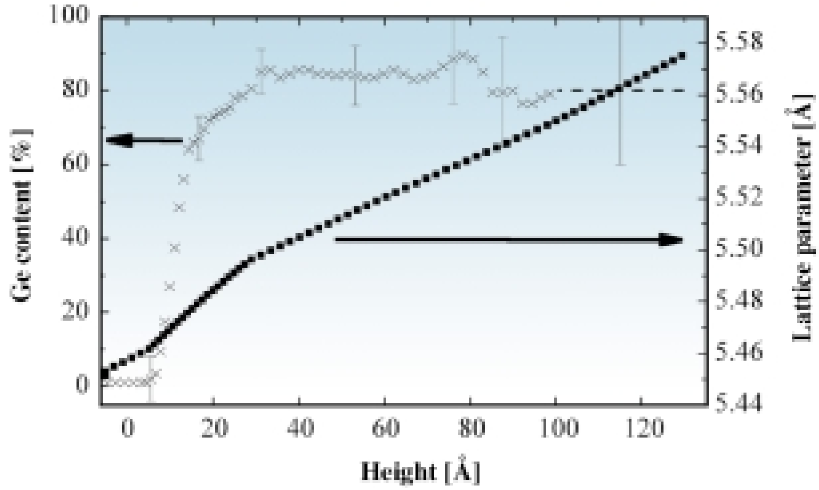


*Fig. 1: Anomalous diffraction result for the lattice parameter profile and the evolution of the Ge concentration in SiGe-islands as a function of height.*

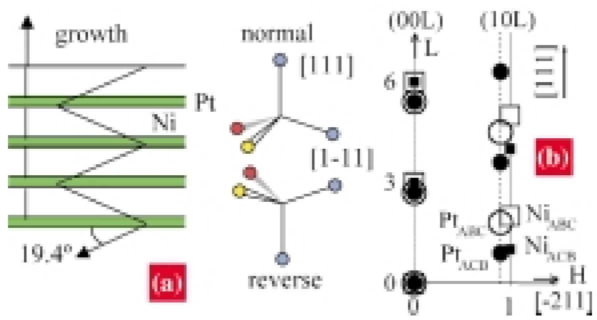


*Fig. 2: (a): schematic structural model of a Pt-Ni multilayer, used to explain the origin of PMA. The stacking reversal (ABCABC... => ACBACB ...) of the (111) planes, observed for Pt on Ni, but not for Ni on Pt, leads to successive Ni layers having reversed stacking sequences. Among the four <111> easy magnetisation axes of Ni, only the one perpendicular to the surface is continuous through the whole multilayer. The other three, which are close (within 19°) to parallel to the surface, rotate at each new Ni layer.*
*(b): side view of the reciprocal lattice for Pt on Ni (111), in the (H 0 L) plane. L and H are respectively the Miller indexes in directions perpendicular and parallel to the surface (Ni lattice units). Empty (solid) squares: Ni Bragg peaks with ABC (ACB) stacking, empty (solid) circles: Pt Bragg peaks with ABC (ACB) stacking. The different locations in the Pt rod of the two stacking sequences allows us to identify the one that is actually occurring.*

### ID3 - Stacking reversal as a source of perpendicular magnetic anisotropy in Ni-Pt multilayers

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*3 Hewlett Packard, 1000 NE Circle Blvd, Corvallis, OR 97330-4239, USA*

Based on surface X-ray diffraction and resonant magnetic surface X-ray diffraction measurements on ultra-thin films of Pt on Ni(111) and Ni on Pt(111), we propose a simple explanation for the perpendicular magnetic anisotropy (PMA) observed at room temperature in Ni-Pt multilayers. The stacking sequence of Ni grown on Pt(111) is the same as that of the Pt substrate (normal stacking: ABCABC) whereas that of Pt grown on Ni(111) is reversed (ACBACB). As a consequence the Ni layers in Ni-Pt multilayers alternate between normal and reversed stacking. The [111] direction, which is normal to the interface planes, is invariant under the two types of stacking whereas the other equivalent crystallographic directions are not. This specific symmetry selects the [111] direction as the preferred magnetic easy axis over all the multilayer stack, causing PMA.
The influence of the Pt layer thickness on the PMA is also discussed (see **Figure 2**).

Publication:
*Phys. Rev. B 67, 220405 (2003).*

## ERRATUM

***Some contributions to the previous Newsletter (N° 36) were not fully acknowledged. We apologise to the authors. The correct contributions and acknowledgements are listed below:***

**I)** ID19 - Extension of "holotomography" to new scientific areas; authors are P. Cloetens, ESRF, and R. Mache, Génétique Moleculaire des Plantes-UMR 5575, Université Joseph Fourier, Grenoble.

**II)** ID21 – Zernike phase contrast X-ray microscopy at 4 keV photon energy with sub-100 nm spatial resolution. Please refer to "Phase Contrast X-Ray Microscopy at 4 keV Photon Energy with 60 nm Spatial Resolution" (ESRF Highlights 2002); authors are U. Neuhäusler (ESRF), G. Schneider (Lawrence Berkeley National Laboratory, Berkeley, US), W. Ludwig (ESRF and INSA Lyon, Villeurbanne, France), M. A. Meyer and E. Zschech (AMD Saxony LLC & Co KG, Dresden, Germany) and D. Hambach (Institut für Röntgenphysik, Universität Göttingen, Germany). Acknowledgements: E. Anderson and B. Harteneck from Lawrence Berkeley National Laboratory, Berkeley, USA for fabricating the phase rings used in the experiments.

**III)** ID1 - *in situ* WAXS investigation of tensile stress and structure in wood slices. The authors are Jozef Keckes[1], Ingo Burgert [1-2], Klaus Frühmann [2], Martin Müller [3], Klaas Kölln[3], Myles Hamilton[4], Manfred Burghammer[4], Stephan V. Roth[4], Stefanie Stanzl-Tschegg[2] & Peter Fratzl[1]
1 Erich Schmid Institute for Materials Science, Austrian Academy of Sciences and Institute of Metal Physics, University of Leoben, Austria
2 Institute of Meteorology and Physics, BOKU - University of Natural Resources and Applied Life Sciences, Vienna, Austria
3 Institute for Experimental and Applied Physics, University Kiel, Germany
4 European Synchrotron Radiation Facility, Grenoble, France.

**IV)** ID10B - Nucleation and growth of inorganic crystals of polymer monolayers at the air/solution interface; A. Berman, Y. Golan, Y. Lifshitz (Ben-Gurion University, Beer-Sheva, Israel) and O. Konovalov (ESRF).

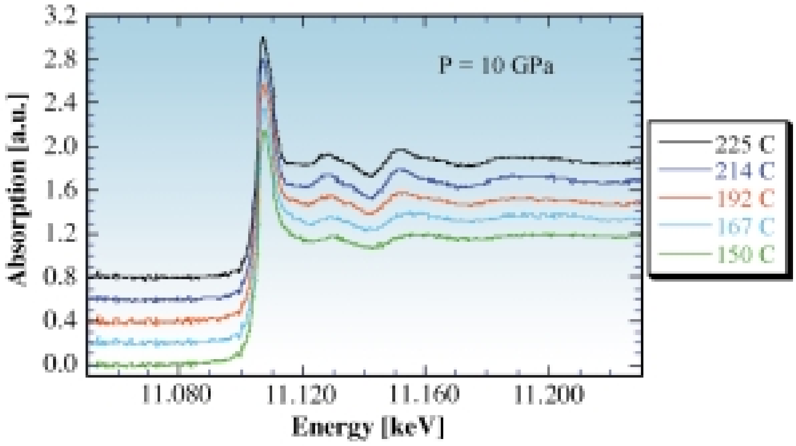


***Fig. 4: Ge K-edge XAS on $Ge_4SbTe_5$ at 10 GPa and temperature from 150 C to 225 C. 300 spectra were recorded in this temperature range at a rate of ~ 1 spectrum per second.***

## X-ray Absorption and Magnetic Scattering Group

### *ID8 - Integrated resonant Raman scattering*

*L. Braicovich[1], A. Tagliaferri[2], G. van der Laan[3], G. Ghiringhelli[1] and N. B. Brookes[2]*
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*2 ESRF*
*3 Daresbury Laboratory, UK*

Recently, we have introduced a new method for the study of magnetic 3d-systems: Integrated Resonant Raman Scattering (IRRS) [1]. The integral of the signal in the scattering channel $2p^63d^n \rightarrow 2p^53d^{n+1} \rightarrow 2p^63d^{n+1}3s^1$ (n ground state 3d occupation) is measured directly with a special movable detector in the geometry shown in **Figure 3** (the detector is the black dot). In this geometry no absorption dichroism is possible, whereas a dichroism in scattering is seen due to the symmetry breaking when measurements are made at an angle. The measurements are made along the cone shown in the figure (angle β) with left and right incident light. Both the difference (dichroism) and the sum of the signals have a characteristic angular dependence related to the anisotropy of the ground state. This has allowed, for the first time, the ground state multipoles up to order 4 to be measured. The IRRS signal integrated over the $L_3$ excitation is given vs. β in the figure. The difference in the anisotropy of Co in the ferrite and in the metal is clearly visible.

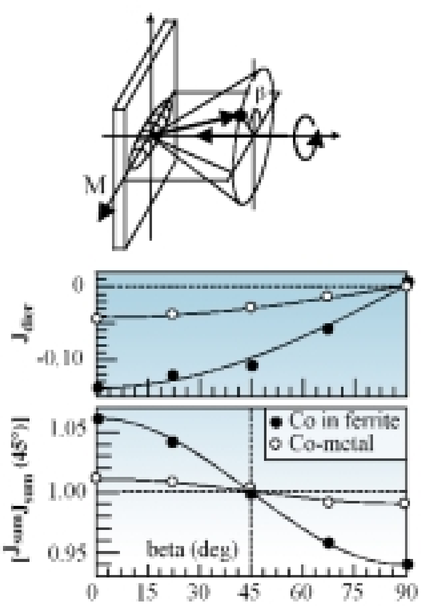


***Fig. 3: Difference of anisotropy of Co in the ferrite and in the metal.***

Reference:
[1] *Phys. Rev. Lett. 90, 117401 (2003)*

### *ID24 - Feasibility studies of crystallisation kinetics at high pressure and temperature*

*M. Wuttig[1], H. Dieker[1], R. Detemple[1], D. Wamwangi[1], M. Delheusy[2], J.-P. Gaspard[2], G. Aquilanti[3], S. Pascarelli[3],*
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*2 Lab. de Physique de la Matière Condensée, Univ. de Liège*
*3 ESRF*

We have performed time-resolved studies of crystallisation kinetics at high temperature and pressure on chalcogenide alloys of interest for high density optical data storage. Understanding the structural transformation from the amorphous to the crystalline state is essential because it represents the time limiting step of rewritable optical data storage processes. Since crystallisation is accompanied by a large density increase, the possibility to study this kinetics at different pressures helps disentangle the contribution of nucleation and growth to the crystallisation process (see **Figure 4**).

## High-Resolution and Resonance Scattering Group

### ID16 - Electronic interactions in the expanded metal compound Li-$NH_3$

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M. Tuel-Benckendorf[3]; E.D. Isaacs[4]
and P.M. Platzman[4]
1 Western Michigan University, Kalamazoo, USA
2 ESRF
3 APS, Argonne, USA
4 Bell Labs, Murray Hill, USA

Inelastic X-ray scattering was used to measure the plasmon as a function of electron density in liquid lithium ammonia as well as the low temperature solid phase. As the electronic density is lowered, electronic correlation effects cause the random phase approximation (RPA) to break down, requiring more advanced theoretical treatments. The deviation from RPA becomes greatest at the lowest electronic densities. We also see evidence for decreased electronic screening as shown by an increase in the strength of the pseudopotential at lower concentrations.

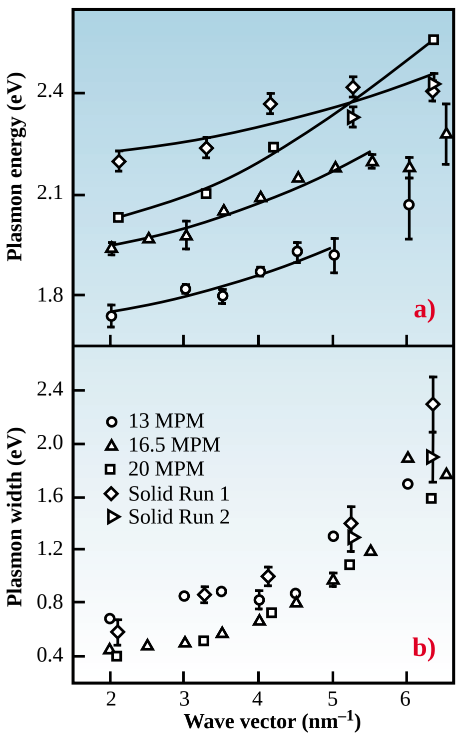


*Fig. 5: a) Plasmon energies plus fits to the expected parabolic dependence on the wave vector. b) Dispersion of plasmon widths. Open symbols refer to liquid solutions with different mole % of metal (MPM).*

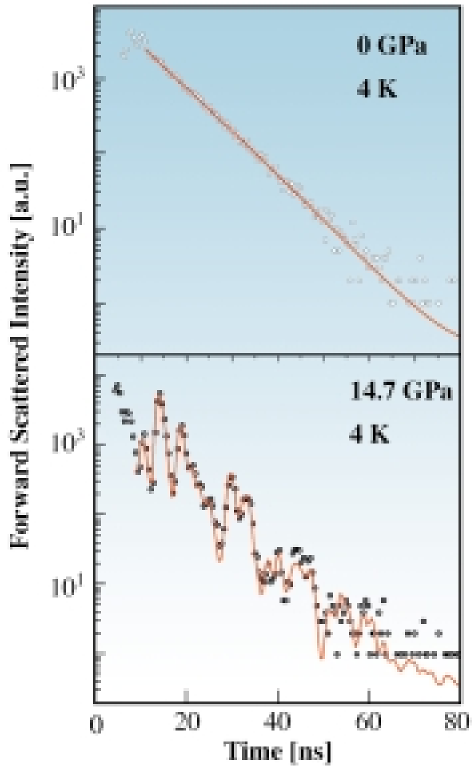


*Fig. 6: NFS spectra of SmS, measured at 4 K and different pressures (circles) and corresponding fits (lines). The spectrum at 0 GPa clearly shows a non-magnetic state whereas that at 14.7 GPa is typical for magnetic behaviour.*

Plasmon behaviour in the solid is similar to that of the heavier alkali metals, but surprisingly different than in the liquid (see **Figure 5**).

Publication:
*PRL 89, 236404 (2002)*

### ID18/22N - Pressure induced magnetic order in the non-magnetic SmS

A. Barla[1], J.P. Sanchez[2], Y. Haga[2],
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R. Rüffer[1], M.M. Abd-Elmeguid[3], R. Lengsdorf[3], J. Flouquet[2],
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2 CEA Grenoble, France
3 II. Physikalisches Institut, Universität zu Köln, Germany

SmS, which was intensively studied for more than three decades for its valence and insulator – metal transitions, remains still puzzling [1]. At ambient pressure, SmS is a non-magnetic ($Sm^{2+}$) semiconductor (black phase) which undergoes a pressure-induced first-order isostructural (NaCl-type) transition at 0.65 GPa (room temperature) towards a metallic phase (gold phase) where the Sm ions are in an intermediate valent (IV) state (v ~ 2.8). Furthermore, in the gold phase the ground state is an insulator up to $p_\Delta$ ~ 2 GPa. An interesting question concerns the behaviour of the IV non-magnetic ground state of SmS as a function of pressure and its possible crossover into the trivalent magnetic state. So far all attempts to find any sign of magnetic ordering in this system gave no positive results although band structure calculations always predict a magnetic ground state for the high pressure phase of SmS [2]. Here we present the first clear evidence that above $p_\Delta$ the collapse of the insulating gap coincides with the appearance of magnetic ordering. This result was obtained by performing $^{149}$Sm high pressure nuclear forward scattering (NFS) of synchrotron radiation at beamline ID22N. From these measurements we obtain information about the magnetic hyperfine field at the $^{149}$Sm nuclei as a function of p and T. Our data (see **Figure 6**) show that at about 2 GPa a first order phase transition occurs from the nonmagnetic IV state into a magnetically ordered state with a large moment of ~ 0.6 $\mu_B$, stable up to at least 19 GPa. The magnetic ordering temperature increases continuously with T and its value (~ 20 K) at low pressure is in the range expected from the comparison with other monosulphides with trivalent rare earth ions. This strongly supports the idea that already at $p_\Delta$ the Sm ions are trivalent.

References:
[1] P. Wachter, *Handbook of the Physics and Chemistry of Rare Earths*, edited by K.A. Gschneidner, Jr., L. Eyring, G.H. Lander and G.R. Choppin (North Holland, Amsterdam, 1994) vol 19, p. 177
[2] V.N. Antonov, B.N. Harmon and A.N. Yaresko, *Phys. Rev.* B 66 (2002) 165208

Soft Condensed Matter Group

### ID2 - in situ observation of the transformation of colloidal amorphous calcium carbonate into microcrystals

D. Pontoni[1,2], J. Bolze[2], N. Dingenouts[2], T. Narayanan[1], and M. Ballauff[2]
1 ESRF
2 Polymer-Institut, Universität Karlsruhe, Germany

The formation of calcium carbonate from supersaturated solutions has been studied for more than a century and it has received renewed interest, especially in the field of biomineralisation. In particular amorphous calcium carbonate has emerged as a precursor to the formation of more stable crystalline forms. The detailed mechanism of phase transformation leading from the initial amorphous material to the final thermodynamically stable crystalline form (calcite) is currently a topic of intense research activity. In order to probe the underlying mechanism, we have performed time-resolved combined small- and wide-angle X-ray scattering (SAXS/WAXS) following the rapid mixing of the reactants using a stopped-flow device. The time-resolved SAXS/WAXS directly demonstrated that the particles generated by the reaction of $Ca^{2+}$ and $CO_3^{2-}$ are amorphous. The transformation of these amorphous $CaCO_3$ particles proceeds by dissolution and subsequent heterogeneous nucleation of the crystalline modification in the reacting mixture [1]. The crystalline modifications thus generated were identified (calcite, vaterite, or aragonite, depending on the initial reagent concentration) and no solid-solid transition was observed. Stopped-flow SAXS/WAXS is therefore well-suited to follow the mineralisation from aqueous solution in great detail (see Figure 7).

Reference:
[1] D. Pontoni, J. Bolze, N. Dingenouts, T. Narayanan, and M. Ballauff, *J. Phys. Chem.* B (Lett.), **107**, 5123 (2003).

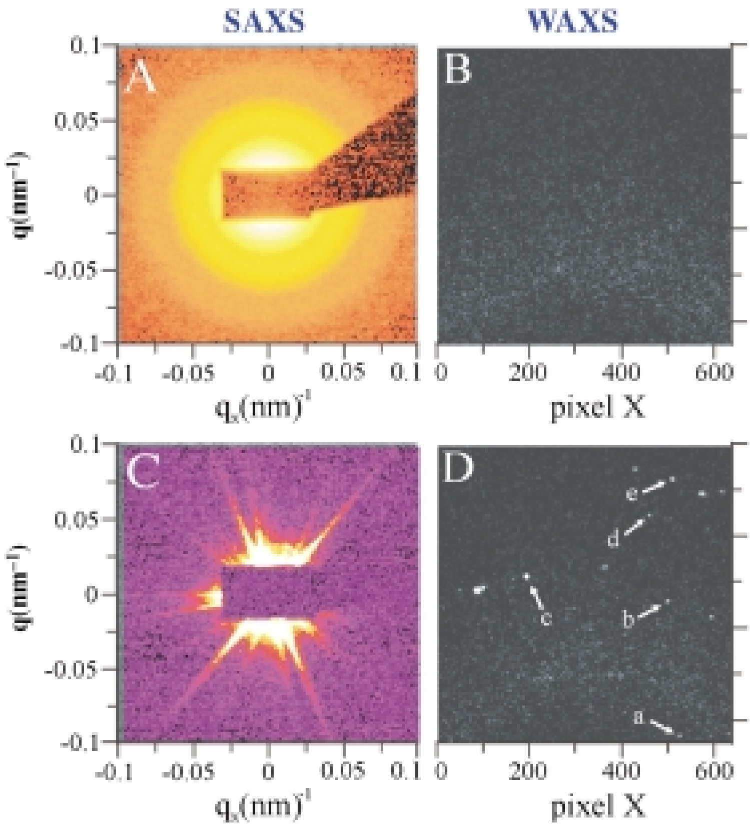


*Fig. 7: Simultaneous SAXS (A) and WAXS (B) 2d images acquired 30 seconds after mixing 9 mM solutions of $Na_2CO_3$ and $CaCl_2$•$2H_2$0. The typical modulation of the isotropic SAXS intensity indicates the formation of ~ 200 nm sized colloidal spheres. The absence of Bragg diffraction spots in the WAXS data confirms that these spheres are made of amorphous calcium carbonate. Simultaneous SAXS (C) and WAXS (D) 2d images acquired 1 hour after mixing 50 mM reagent solutions. The highly anisotropic SAXS signal suggests the presence of microcrystallites. The corresponding WAXS image shows diffraction spots which can be assigned to the various crystalline modifications of calcium carbonate. The spots indicated by arrows correspond to the following Bragg reflections: [a] calcite (012); [b] vaterite (004); [c] aragonite (032); [d] aragonite (310); [e] aragonite (302).*

### ID10B - Two-stage surface freezing of a single top layer in a smectic-A membrane

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The crystallisation of a single liquid top layer of smectic membranes of the compound 4O.8 has been studied with grazing-incidence X-ray diffraction. As this process takes place in two steps, involving an intermediate hexatic smectic-B layer before the final crystalline-B surface structure is reached, it provides a model for melting in two dimensions. The positional order has been investigated quantitatively by measuring the scattering profiles and the associated correlation lengths. The surface liquid-hexatic phase transition is found to be continuous, while the hexatic-crystal transition is weakly first-order with an abrupt change of the in-plane positional correlations. The surface phase transitions do not modify the liquid in-plane structure of the interior layers.

## Macromolecular Crystallography Group

### ID29 - DNA damage repair and breast cancer

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*Hutchinson/MRCResearch Centre, Cambridge, UK*

The repair of DNA double strand breaks in cells is an ongoing process in all organisms. Without such repair of DNA cells become unviable and, in higher organisms, this can lead to cancer. Consequently, sophisticated pathways for chromosomal DNA repair have evolved which are continually battling against DNA damage. One method of double strand break repair is homologous recombination. This pathway utilises the advantageous position of cells having homologous chromosomes and when one chromosome becomes damaged the partner chromosome acts as a template for repair. In higher organisms the class of proteins known as Rad51 promotes strand exchange between homologous chromosomes, assembling as oligomers at sites of double strand breaks. Additional components of recombinatorial DNA repair systems have been discovered, for instance BRCA2 which has been postulated to mediate oligomerisation of Rad51 at sites of DNA damage. Using single crystal X-ray diffraction data recorded at ID29, the crystal structure of a Rad51-BRCA2 complex (see **Figure 8**) was solved. This illustrates that Rad51 binds to the so-called BRC repeat of BRCA2 which can thus mediate interactions between individual Rad51 monomers and control the assembly of the Rad51 nucleoprotein filament essential for strand-exchange reactions. Mutations in the highly conserved BRC sequences of BRCA2 are associated with a predisposition for breast cancer and the structure also implies that mutations in these BRC repeats would prevent Rad51 binding to BRCA2. The formation of Rad51 foci at DNA damage points would thus not occur and the structure thus provides a glimpse of the molecular origins of breast cancer (see **Figure 9**).

***Fig. 9: The nucleoprotein filament of RecA (Rad51 equivalent from E. coli). This filament binds to DNA at sites of DNA double strand break damage.***

Publication:
*Nature, 420 (2002) 287-293*

***Fig. 8: The protein structure of a Rad51-BRCA2 complex. Rad51 is shown in blue and purple and the BRC repeat ligand is shown in green.***

## X-ray Imaging Group

### ID17 - In-vivo observation of damage and repair in blood vessels from exposure to multiple microbeams of X-rays of hundreds of Grays

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*3 ESRF*
*4 Inst. of Pathology, Luzern, CH*
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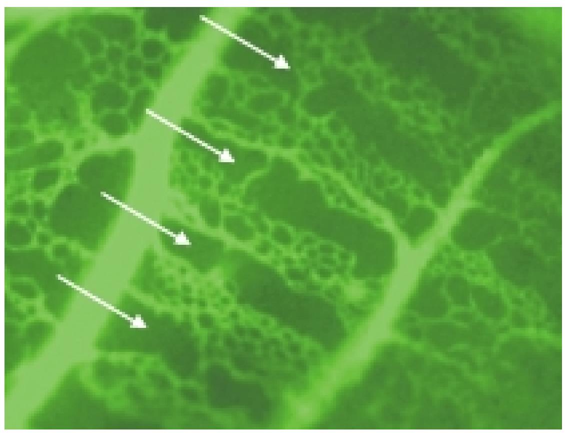
The Chorio-Allantoic Membrane (CAM) of chickens allows direct observation with time of the radiation effects on rapidly developing blood vessels. CAMs in a Petri dish have been exposed to arrays of 51 parallel, thin (about 25 micron wide) closely spaced (5/mm) vertical (10 mm-high) microplanar beams of synchrotron radiation X-rays along the surface. The entrance doses ranged from 300 Gy to 1200 Gy. All CAMs survived the observation period of 24h. Massive damage, which was not repaired within 24h, was seen after application of entrance doses of 600 or 1200 Gy. For the 300 Gy exposures, the microvasculature between the microbeams was minimally affected and was repaired within 24h. Vascular bridges were observed across areas situated directly in the path of a microbeam. This experiment demonstrates that the microbeam effect can also be observed in-vivo in the CAM model. At entrance doses of 200 to 300 Gy, the microvascular damage between the beams is absent or minimal. Larger blood vessels survive multiple exposures in their course with 300 Gy microbeams. The damaged microvasculature situated directly in the path of the beams can be repaired, at least partially (see **Figure 10**).

This experiment gives important information on the damage-repair mechanism of vasculature and microvasculature in radiotherapy with microbeams.

***Fig. 10: Chorio-Allantoic Membrane of the chicken egg, irradiated with micro-beams (arrows) of 300 Gy, 28 µm wide and 200 µm center-to-center distance, 24 h after irradiation. While larger vessels have survived multiple hits by micro-beams, the capillaries have been de-stroyed. The width of the destroyed areas is larger than a micro-beam, but in some areas new capillaries bridge the gaps.***



### ID22 - In situ XAFS study of water-saturated haplorhyolitic melt at 1.2 GPa / 800°C: Nickel speciation and melt-density measurements

*M. Muñoz[1], V. Malavergne[1], F. Farges[1], H. Bureau[2], B. Ménez[3], A.Simionovici[4], A.Somogyi[5], C. Schmidt[6] and M. Wilke[7]*
*1 Lab. Géomatériaux, Univ. de Marne-La-Vallée, France*
*2 Lab. Pierre-Süe, CEA Saclay, France*
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*4 Lab. des Sciences de la Terre, ENS Lyon, France,*
*5 ESRF*
*6 GeoForschungsZentrum, Potsdam, Germany*
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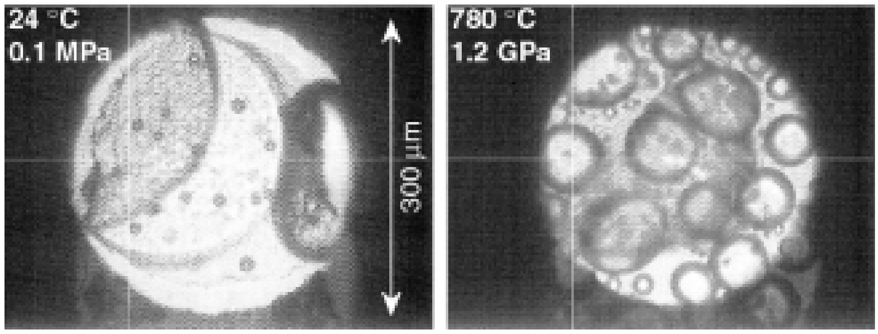


*Fig. 11: Water saturated haplorhyolitic glass in an aqueous $NiCl_2$-solution (0.35 M) under ambient and in situ conditions.*

Trace elements play an important structural role in volcanic magmas, and the presence of water can highly affect their transport properties. Furthermore, water is one of the most important parameters controlling volcanic eruptions, and models for magmatic ascension before the exsolution of water are essentially based on approximations of the density values.

In order to investigate the speciation and density of the hydrated melt, *in situ* high pressure and temperature X-ray Absorption Fine Structure (XAFS) experiments were performed at the Ni K-edge, at ID22. The X-ray beam was focused to V x H = 3 x 5 $\mu m^2$ with the ID22 KB-mirror.

The experimental setup consisted of an externally heated Bassett-modified Hydrothermal Diamond Anvil Cell (HDAC). The sample chamber was loaded with a water-saturated haplorhyolitic glass together with an aqueous $NiCl_2$-solution (0.35 M) and an air bubble. The XAFS data were collected in fluorescence mode using a Si(Li)-detector. The experiment was carried out at temperatures up to 800 °C, and the corresponding hydrostatic pressure was calculated to be approximately 1.2 GPa. Micro-XANES spectra were collected *in situ* in the silicate melt and in the aqueous phase. Spectroscopic data are first interpreted in terms of speciation. At the experimental P-T conditions, nickel is essentially four-coordinated in the hydrous melt, whereas its coordination is 5 in the glass. Moreover, based on the theoretical description of the absorption edge, the XANES spectra are used to derive the "fluid/melt" partition coefficient for nickel (0.22 ± 0.05), as well as the density of the hydrated melt (0.9 ± 0.2 g/cm$^3$) (see **Figure 11**).

## Optics Group

### Microfocusing by curved mirrors and multilayers

*O. Hignette, G. Rostaing, P. Cloetens, Y. Dabin, A. Rommeveaux, Ch. Morawe, A.K. Freund, ESRF*

Focusing hard X rays using grazing incidence reflecting surfaces is probably the method mostly employed on synchrotron beamlines. The architecture of choice universally adopted is the so-called Kirkpatrick-Baez system (KB), which consists of two orthogonal crossed mirrors. One focuses the source image in the horizontal plane while the second focuses it in the vertical plane. The ESRF Optics Group has opted for a dynamic system with actuators bending the mirrors into the elliptic shapes required for a given experiment. The rather complex alignment is accomplished using automated sequences with the help of CCD position sensors. So far, the Optics Group has produced almost 40 bent mirrors, most of them in KB pairs, and there are more to follow. The most outstanding result has been obtained recently at ID19, where a spot size less than 100 x 100 nm$^2$ was measured at 20.5 keV (**Figure 12**). The total gain in flux density compared to a pinhole was estimated to 5 x 10$^6$.

*Summarised by Christian Morawe*

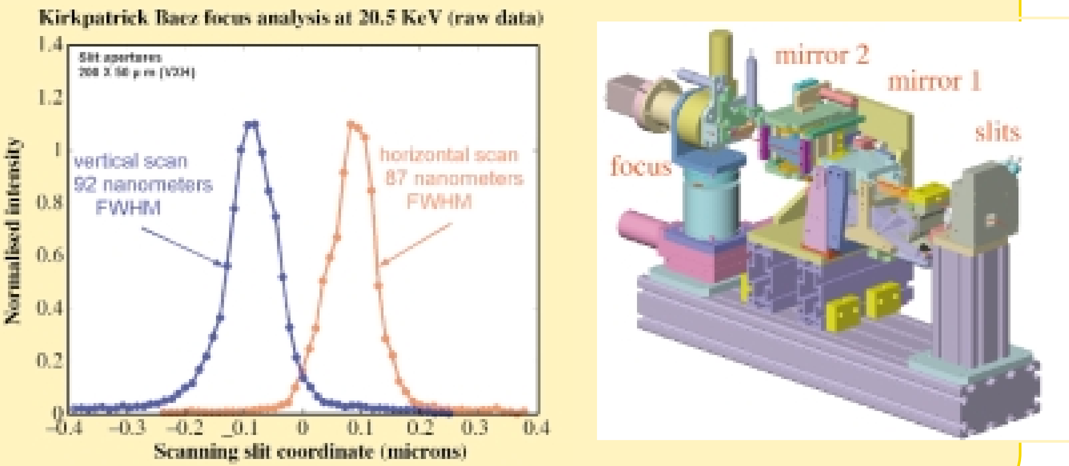


*Fig. 12*

# NEWS FROM THE CRGS

**BM01**: The Swiss Norwegian Beamline (SNBL) has continued to support four different experimental techniques, including high-resolution powder diffraction, EXAFS, X-ray diffractometry and a large area image plate detector. In recent months, several user groups, in collaboration with in-house staff, have been developing methods for investigating the *in situ* behaviour of a variety of materials. The samples have varied in crystal quality from almost perfect single crystals through to heterogeneous catalysts. For some projects, access to the combination of experimental tools available on SNBL has been particularly useful.

In a study of Rochelle salt ($NaKC_4H_4O_6 \cdot 4H_2O$), the group of F. Mo (Trondheim, Norway) established that both temperature and relative humidity are crucial factors in determining the onset of the transition between paraelectric and ferroelectric phases. In addition, an external DC field is required in order to pole the crystal domains. A sample cell has been developed in Trondheim and successfully tested in the KM6 multi-axis diffractometer on **BM01A**. This cell permits very high quality single crystal data to be collected under precisely controlled conditions of temperature and humidity, with application of an external field for poling the crystal. It can be noted that relative humidity can be a critical parameter for many crystal systems, particularly macromolecular crystals, but until now little effort has been put into the use of humidity as an experimental variable.

$LaNi_5$-based intermetallic hydrides enjoy a broad range of applications in hydrogen storage, with nickel-metal hydride batteries being currently the most important one. A specially designed cell for *in situ* studies in $H_2$ atmosphere was attached to a metal hydride hydrogen storage unit developed by V. Yartys *et al.* (IFE, Kjeller, Norway) providing hydrogen gas at convenient pressures in a range 2-3 bar $H_2$. Heating-cooling cycles in the temperature range 20 – 100 °C were accompanied by hydrogen absorption or desorption. Measurements with the high-resolution powder diffractometer on **BM01B** revealed the structural phase transitions in a tin-doped $LaNi_5$ alloy during absorption. A continuation of the project using the rapid data collection possible on the MAR345 area detector on **BM01A** should further reveal the kinetics of the process.

On **BM1B**, a group led by J.-D. Grünwaldt from the ETH Zürich has been using *in situ* extended X-ray absorption fine structure (EXAFS) spectroscopy, combined with on-line gas analysis, to gain insight into the structure of heterogeneous catalysts, *e.g.* $Pd/ZrO_2$ for methane combustion, Cu/ZnO for methanol synthesis and $AgAu/MeO_x$ for selective oxidation. Catalytic reactions were performed in an *in situ* EXAFS cell directly at the beamline and combined with on-line gas analysis using a mobile mass spectrometer. Temperature control of the reaction cell allowed progress in the catalytic reactions to be followed up to 500 ºC.

**BM02**: Due to some specific developments, the D2AM French CRG Beamline is mostly known as a DAFS Materials Science Beamline since this technique is now widely used and allows the characterisation of manufactured organised surfaces (ESRF Newsletter n°36, June 2001, page 19). This of course should not mask the activity of the SAXS community which is well established and allows 2D SAXS experiments to be performed with very high resolution: indeed some recent experiments allowed us to reach very small Q values (see details on **Figure 1**).

Moreover, as the presently available detectors do not fulfill all experimental needs, a Silicon Active Pixel detector is under development. First results have shown that a very high dynamical range can be reached on 2D images using this technique (J. Appl. Cryst. 35 471-476, 2002). An improved chip (XPAD-2) allowed us to reach count rates as high as $2 \cdot 10^6$ photons/pixel/second (see **Figure 2**).

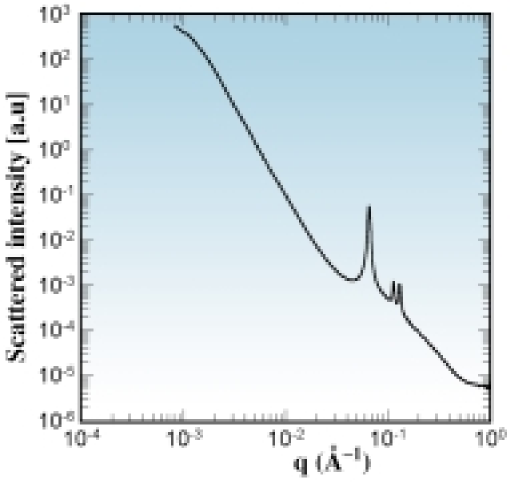


*Fig. 1: combined USAXS and SAXS on ordered hexagonal mesoporous silica SBA-15 by F. Ehrburger-Dolle, E. Geissler , F. Bley, F. Livet, A. Rigacci and C. Vix-Guterl.*

**BM16** has now become a CRG beamline since the sales contract between the ESRF and the Spanish Ministerio de Ciencia y Tecnología was signed in April. This new CRG will carry out both protein crystallography (PX) experiments, with full MAD capabilities, and SAXS.

The commissioning of the beamline began in January 2003. The radiation test for the newly constructed experimental hutch was successfully passed on 7 April. So far, the X-ray beam has been focused at the PX sample position. The newly installed mirrors work within specifications, the focal spot being < 0.3(h) x 0.7(v) $mm^2$ at the Se edge. The photon flux measured at the sample is within the expected order of magnitude (*i.e.* $10^{12}$ photons/sec).

The PX sample environment is available since the beginning of June. The first data collection test is expected to be performed by summer 2003. Provided everything continues to follow the schedule, the beamline will be open to the first Spanish users by September and to public ESRF users from January 2004 on. The installation of the equipment for the small angle scattering (SAXS) end-station is scheduled to begin in the October shutdown.

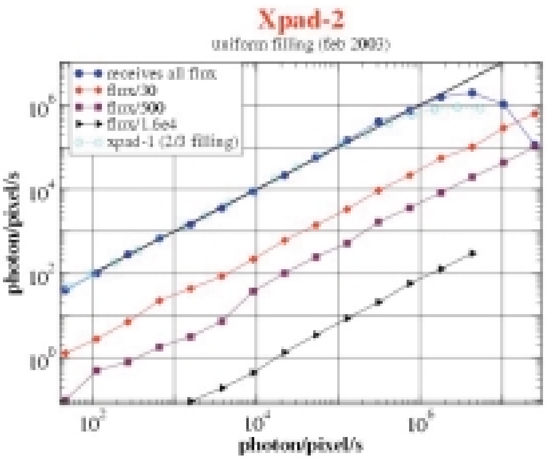


*Fig. 2: Count rates of a new Silicon Active Pixel detector with an XPAD chip.*

# Resonant Magnetic X-ray Scattering under High Magnetic Fields

L. Paolasini, F. Yakhou, C. Detlefs, J.-P. Valade, A. Bombardi, F. De Bergevin, P. P. Deen, S. Wilkins, P. Bernard and G. Peppelin
ESRF

We present the ongoing project to extend the capability of the Magnetic Scattering Beamline ID20, to make available a continuous magnetic field of up to 10 Tesla for low-energy resonant X-ray diffraction experiments in the low temperature range.

The control of thermodynamical variables such as temperature, magnetic field, electric field and pressure is essential for the determination of the complex relationship between the correlated microscopic properties in materials in a variety of research disciplines. Experiments under extreme sample environment conditions have often led to unexpected and exciting science, or have provided input to the understanding of debated physical issues. Investigations on the properties of matter at low temperatures by Heike Kamerlingh Onnes led to the discovery of superconductivity and, *inter alia*, to the production of liquid helium (Nobel Prize 1913). The invention by Percy W. Bridgman of an apparatus to produce extremely high pressures allowed important discoveries in various fields (Nobel Prize 1946). High magnetic fields have more recently been the key factor for the discovery of the quantum Hall effect (Klaus von Klitzing, Nobel Prize 1985) and of a new form of quantum fluid with fractionally charged excitations (Robert B. Laughlin, Horst L. Störmer and Daniel C. Tsui, Nobel Prize 1998). Indeed, the need for high magnetic fields as a probe of physical phenomena was recognised several decades ago, and a number of dedicated national facilities have been established all over the world.

For a long time, neutron scattering has been the technique of choice for the investigation of microscopic interactions in solids. Due to the high penetrating properties of this probe, various sample environment equipments have been developed in the last fifty years, reaching a well established status. Similar sample environments for X-ray magnetic scattering represent a major challenge for this technique. The possibility to carry out experiments under such extreme conditions would provide the scientific community with unique research opportunities for the study of a broad field of phenomena in condensed matter. The combination of high magnetic field and of the Resonant X-ray Scattering (RXS) technique is expected to be substantial relevance to problems in the physics of strongly correlated electron systems such as high temperature superconductors, non-Fermi liquid and heavy-Fermion systems, colossal magnetoresistance compounds, structural, magnetic and quantum phase transitions, frustrated and low-dimensional systems.

We present here the project to extend the capabilities of the ID20 beamline by making a continuous magnetic field environment of up to 10 Tesla available for low-energy resonant X-ray diffraction experiments in the temperature range from 1.6 to 300 K. This project, discussed already in the ESRF Medium-term Scientific Plan and endorsed by the ESRF Scientific Advisory Committee, includes the development of the cryomagnet itself, a high-load diffractometer built from non-magnetic materials, and the construction of a new experimental hutch to accommodate these instruments. The project is well on its way: the construction of the new hutch was undertaken in autumn 2002 (see **Figure 1**). During the shutdown of operations the entire beamline was recabled, and a new control hutch was rebuilt. Works are now completed, and ID20 is again fully operational for experiments in the first experimental hutch. The diffractometer is currently under construction at HUBER GmbH, and will be installed in October 2003.

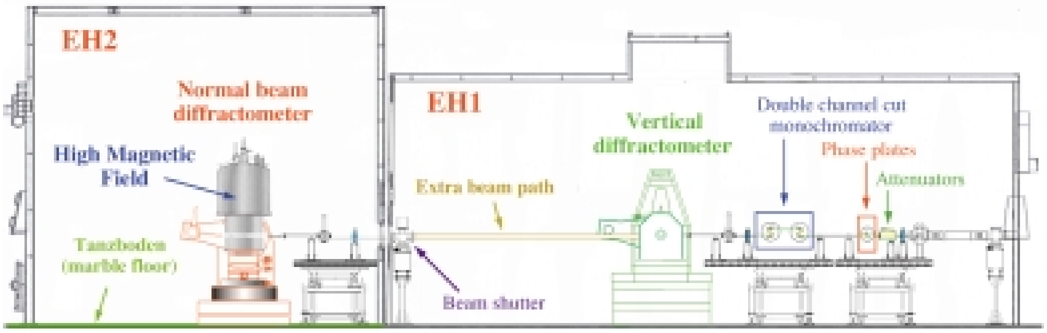


*Fig. 1: Layout of the experimental hutches EH1 and EH2 and future experimental setup of the ID20 beamline. The new experimental hutch features an extended height to allow sample changes without removing the cryomagnet from the diffractometer, and to reduce the magnetic stray field effect on the roof. The MICROCONTROLE diffractometer in EH1 will be adapted for the vertical scattering geometry, whereas the "normal beam" HUBER diffractometer in EH2 will be dedicated to the horizontal scattering geometry. An extra in-vacuum beam path will assure the link between the two hutches. The up-stream part of EH1 will contain all the devices for manipulating the incident monochromatic beam characteristics.*

## Split-Coil Superconducting Cryomagnets for X-ray Diffraction

Superconductors are the key components of magnets, and almost all high-field research magnets used today are based on superconducting coils, cooled down by liquid Helium [1].

In the case of scattering experiments, the required access to the sample region is realised by the use of a split pair magnet geometry. The coils have to be split in two parts, separated by a strong mechanical structure (wedge type spacer for X-ray or aluminium rings for neutrons) to compensate the strong attractive forces between the coil halves. The upper limit of the magnetic field is then defined both by the split dimensions and the type of superconducting materials used for the wires. It is also obvious that the dimensions of the Split-Coil Superconducting Cryomagnet (SCSCM) have to be compatible with the load capacity of the diffractometer on which it is installed. The latter has to be built with non-magnetic materials, because the SCSCM creates a stray magnetic field in the environment (proportional to the magnetic field strength and to the internal coil geometry), which decays with the distance, and can influence the steel-made materials as well as the electronic devices and detectors. Moreover, the presence of steel in close proximity to the magnet can cause excess force on the cryostat components, leading to damage to the internal magnet support system, or poor cryogenic performances.

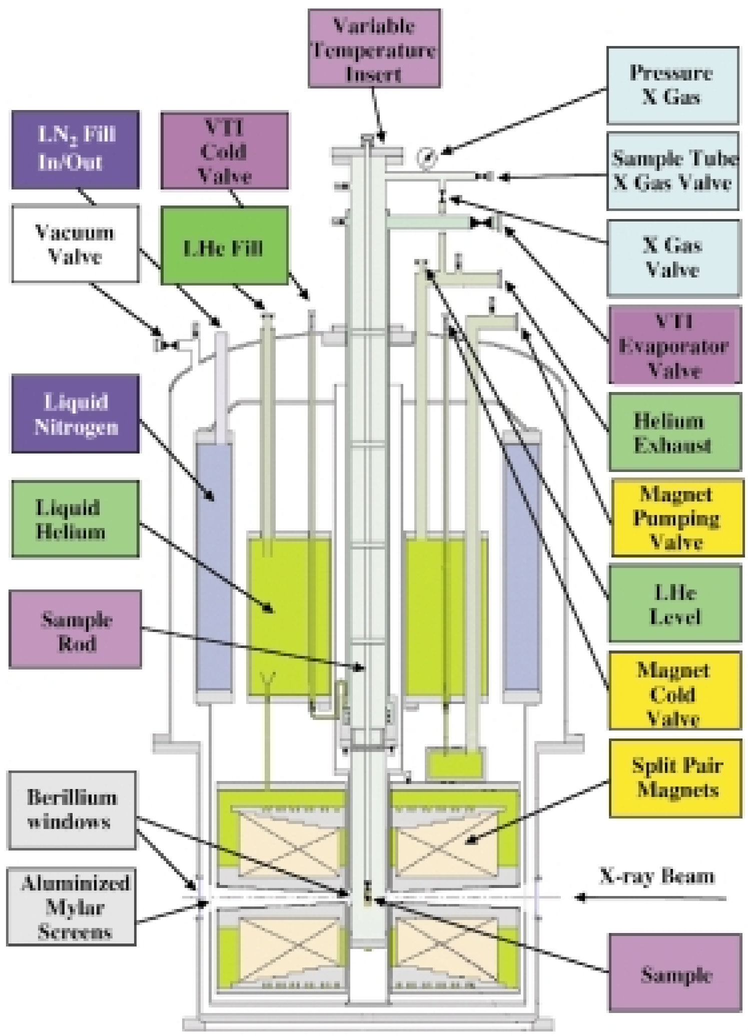


*Fig. 2: Cross-section of the future 10 Tesla split-pair cryomagnet that will be installed on the "normal beam" HUBER diffractometer, accommodated in the new experimental hutch EH2.*

An important technical point, which is specific to the RXS method, is defined by the working photon energies, which fix two main physical limitations for the construction of a SCSCM for X-ray diffraction: wide opening angles to perform magnetic diffraction, and low absorption windows. These specifications have a direct consequence on the internal custom design of the magnet and on the maximum magnetic field strength obtainable. Finally, the possibility to have a large sample space is another remarkable specification, which increases the versatility of the SCSCM to perform experiments using devices as a uniaxial pressure stick, a low temperature devices ($^3$He or dilution cryostat), or an additional stick with an extra rotation stage (to rotate and tilt the sample around the scattering vector).

Figure 2 shows the cross-section of the future 10 Tesla split-pair cryomagnet that will be installed at ID20 in the new experimental hutch. The cryomagnet will be constructed by Oxford Instruments and delivered at the ESRF in 2004. The upper part of the SCSCM is a standard design, supplied for many superconducting magnet systems for neutron scattering experiments. A variable temperature insert (VTI) able to reach 1.8 K at the sample position could be replaced in future with a high cooling power (> 40 mW) $^3$He insert to reach temperatures below 1 K. The bottom part of the SCSCM is a completely new custom design, with a very large angular access to the photon beam and low absorbing beryllium windows and aluminised Mylar thermal screens. Due to the asymmetric distribution of the wedge type spacers, the maximum magnetic field obtainable with such internal geometry cannot exceed 10 T at 4.2 K. This limit is also fixed by the close proximity of the iron wall of the new experimental hutch and the load capabilities of the new diffractometer. This latter will be adapted for the horizontal geometry ("normal beam" diffractometer).

The future capability to implement a high magnetic field on the new diffractometer will allow the application of the RXS technique to some of the most exciting fields of condensed matter physics. This project, expected to be fully developed by the end of 2004, will overcome present limitations, and will allow to fully exploit the potential of the ID20 beamline.

**Reference:**


[1] S.W. Van Sciver and K.R. Marken, Physics Today, August 2002, p. 37

# Injection with Front-End Open

P. Berkvens[1], J.-L. Revol[2], P. Elleaume[2]

1 ESRF: Safety Group, 2 ESRF: Machine Division

On 5 February 2003, the operation of the ESRF beamlines was dramatically changed, allowing a truly continuous operation. Previously, the front-end shutters of all beamlines were closed for a few minutes during injection, resulting in large temperature variations on the monochromators and / or mirrors which caused interruptions of data acquisition ranging from several minutes up to an hour following each injection.

This problem has now been solved by leaving these shutters open during injection. This type of injection is similar in many ways to the "topping-up" in use at APS and SLS, also currently under development for Diamond and Soleil. Thanks to the naturally long lifetime of the electron beam at the ESRF, however, top-up can be carried out at a very low repetition rate (typically once every 12 hours in the most demanded multi-bunch modes). This is beneficial for the most sensitive and advanced beamlines, since it reduces the necessity of gating the data acquisition at frequent injection times.

Before implementing the injection with front-end (FE) open mode of operation, a feasibility study was carried out to verify that this mode of operation was compatible with the present ESRF radiation protection policy. This policy stipulates that all staff working at the ESRF are considered as non-exposed workers. This implies that the dose limits for the public, as defined in the Euratom/96-29 decree, do not exceed an effective dose of 1 mSv per year. Taking into account a working time of 40 hours per week over a maximum of 52 weeks per year, this corresponds to a maximum dose of 2 µSv over 4 hours. Radiation monitors interlocking the beam in the linac pre-injector are used whenever it is potentially possible to exceed this dose limit.

*Fig. 1: Radiation monitor close to the hutch of a beamline.*

The accidental steering of a single 1 µs electron beam pulse injected from the booster into a beamline with its front-end open would result in an integrated dose, largely exceeding the 2 µSv limit. No interlocked radiation monitor could therefore be fast enough to protect against such an accidental situation. Such a scenario could take place with a very particular and highly improbable setting of the beam in the booster and of the ring quadrupole and dipole magnets. Nevertheless, a detailed study has demonstrated the impossibility of steering a beam from the booster into a beamline, if a beam is already stored in the storage ring. In other words, to guarantee the safety of the beamline with front-end open, it is mandatory to verify that some beam is already stored in the ring. In order to guarantee the presence of a stored beam in the ring, a dedicated and redundant beam current monitor has been developed and integrated in the machine personnel safety system. Injection with FE open is only allowed if this current monitor measures a stored beam greater than 5 mA.

Measurements were carried out to assess dose values outside beamlines during injection with front-end open for different machine tunings and filling modes. These measurements have shown that, under normal operation conditions, the injection losses, as well as the losses due to bunch cleaning[(1)], in all filling modes produce very low dose rates compatible with the above-mentioned limit. Significant dose values were only measured with seriously degraded storage ring optics, resulting in a very poor injection efficiency. Under extreme conditions, a maximum dose of 0.5 µSv per extracted booster pulse was measured. This would exceed the dose limit over a 50 mA top-up operation. It was therefore decided to install radiation monitors outside the optics hutches on all beamlines, fast enough to interlock the corresponding front-end if dose limits exceeded the authorised level during injection. The monitors chosen were 50-litre ionisation chambers from the company PTW (**Figure 1**).

**Figure 2** shows the dose measured over 4 hours around the optics hutch of the ID14 beamline, during the first one and a half month of operation during which uniform, 16 bunch, single bunch and 2 x 1/3 filling modes were delivered. Clearly, the measured dose is dominated by the background level in the experimental hall (which varies between 80 and 100 nSv.h$^{-1}$), with a small contribution from scattered bremsstrahlung, which depends on the type of filling mode. The pattern shown in **Figure 2** is similar on all beamlines and no significant dose values were integrated during the injections.

Concerning the machine, the implementation of the injection with FE open as a standard mode of operation was rather straightforward. The only modification concerned the logical unit of machine protection system which had to be updated. For this purpose, a new condition has been implemented which deactivates the injection permit with front-end open in case there is less than 5 mA stored in the ring. A further modification was the implementation of a software counter to inform the beamlines

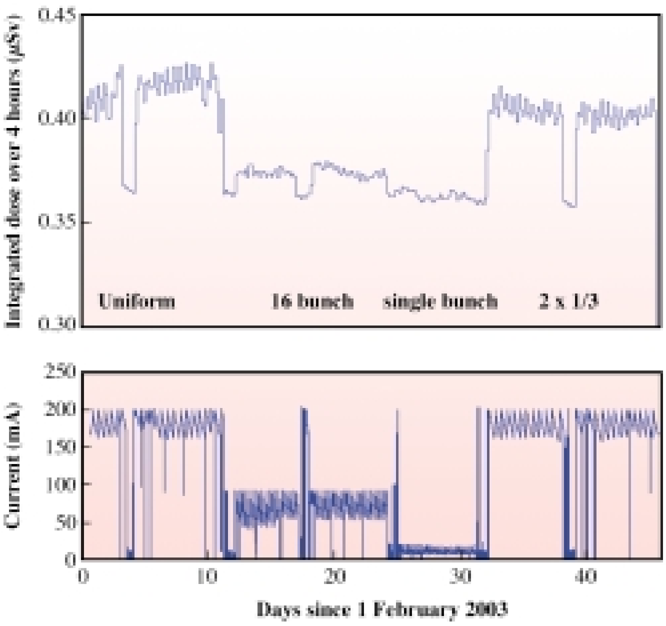


***Fig. 2: Typical dose measurement from one radiation monitor during the first one and a half months of operation.***

of the injection schedule. The value of the counter indicating the remaining time until the next injection is easily readable and can be used to synchronise the data acquisition. In order to fully benefit from the injection with front-end open for the beamline, it is essential to maintain the magnetic gap of undulators and wigglers unchanged during injection since any variation would induce a heatload change in the optics. In this respect, one should recall that permanent magnet material can be demagnetised when exposed to high-energy electrons. In-vacuum undulators which are operated with a small gap (5-6 mm) are the most susceptible to such radiation damage. Following a mis-steering of the beam injected from the booster, such demagnetisation was observed at the ESRF in 1993 on two undulator segments installed on ID6 and ID10 which were made with NdFeB type permanent magnet material. A detailed study showed that $Sm_2Co_{17}$ material, while more brittle and with a less remanent field than NdFeB, is much more resistant to radiation damage and less temperature-sensitive, and was therefore selected for the in-vacuum undulators. Nowadays, a much closer diagnostic and control of the losses during injection, by means of the network of neutron and bremsstrahlung detectors located both inside and outside the ring tunnel, has made such accidents very unlikely for undulators with in-air magnets (magnetic gap > 11 mm). Since then, no further radiation damage to undulators has been observed. As a result, the magnetic gap of such undulators is left unchanged during injection. In-vacuum undulators are built with more resistant material ($Sm_2Co_{17}$) but when closed to a gap of 5 or 6 mm, even though the injection efficiency should stay close to 90-100%, it is expected that significant and repetitive electron beam losses will take place in some of them. At present there is insufficient information on the long-term effect on the magnets subjected to such losses. For this reason, it has been decided to limit the minimum gap to 8 mm (instead of 6 mm) for all in-vacuum undulators during injection. For the corresponding beamlines, this generates a maximum 30 % heatload variation over a few minutes. This situation, although not ideal, is still much better than the situation prior to 5 February where the gaps of all in-vacuum undulators were fully open during injection. In the long term, it is planned to reduce the 8 mm gap limit as low as possible following a detailed quantitative study performed on the ID6 machine beamline.

During 2002, performance of the X-ray source was excellent, with records for both availability and Mean Time Between Failures (MTBF). The machine availability reached 98% with a MTBF of 57.8 hours. These figures include the 95 beam interruptions which occurred during the beam delivery. All beam losses which took place during the refill were not included in these statistics, however, they will now be detrimental to the beam's quality with the injection FE open mode of operation. Dedicated work has been performed on the injection elements on both hardware and software in order to reduce the number of failures.

A direct consequence of the improvement made on the control of the injection elements and on the insertion devices was a reduction of the refilling time, which is also beneficial.

The reactions of the users have been extremely positive, despite the remaining constraints imposed on those who are using in-vacuum undulators. The thermal variations induced by the refilling (from 160 to 200 mA, twice per day), have almost disappeared in multibunch mode. This improvement, associated with no realignment after injection and the possibility to use the beam during injection, now results in an increase of beam availability on most of the beamlines. Some users are now using beam during injection, especially those who perform long scans. The horizontal and vertical beam stability is affected during injection. The perturbation which occurs for a few ms every second is perceived differently, depending on the experiments and the beamlines.

In multibunch modes (uniform, 2 x 1/3 or 2/3) where the lifetime is long (50-90 hours), a twice daily injection with front-end open is certainly close to ideal for the beamlines. The situation is not as favourable in 16 and single bunch modes where the reduced lifetime imposes more frequent injections in order to limit the current variation. In addition, these filling modes, which must be delivered with a very high degree of purity, require a few minutes for the cleaning process[(1)] after each injection. However, work has been initiated to improve this situation.

To conclude, it is fair to say that whatever the filling mode of the ring, and despite the diversity of experiments performed on the various beamlines, the injection FE open mode of operation has provided a significant added value to the ESRF beamlines.

*(1) The users of exotic time structure modes (such as single bunch, 16 equally spaced bunches) require a large contrast of current between the main bunch(es) and the adjacent "unfilled bunches". This purity (typically $10^{-9}$) is obtained by the application in the storage ring of a cleaning process based on the difference in betatron tunes between the main bunch and the low populated bunches. Due to the low purity after injection and the transverse excitation of the beam during the cleaning process, the beam cannot be used by the most demanding beamlines during the injection period.*

# RESTART OF THE HQPS AFTER BREAKDOWN

J.-F. Bouteille
ESRF Machine Division

After 7 years of excellent service, the ten machines constituting the High Quality Power Supply (HQPS) suffered from a serious damage and had to be stopped for investigation. Three months of intense repair work, commissioning and tests were necessary before the HQPS could go back to operation.

## Why is HQPS needed at the ESRF?

Grenoble, known as the capital of the French Alps, is situated in a mountain region that has always been a high producer of electricity. As a result, a dense network of very high voltage aerial lines surrounds Grenoble. Such aerial lines are sensitive to lightening. The consequence for the ESRF site is a much larger number of voltage or frequency drops of the main compared to any other major city. Such drops when higher than 10 % inevitably trip either a power supply or a pump of the water-cooling system resulting in a beam trip. These voltage drops frequently result from arcing to the earth from lightning on the 225 kV or 400 kV lines. During the time it takes for the circuit breakers to isolate the faulty line and extinguish the arc and before the line is available again, drops occur lasting from less than 100 ms to one second, with a voltage loss from 5% to 80%. The observed average is a 20% voltage drop lasting 200 ms. Proper functioning of the HQPS is partly responsible for the high availability and reliability of the beam to the users. It also avoids hidden damages, which generate subsequent equipment failures.

## How does it work?

At the ESRF, an auxiliary supply called HQPS is able to clean the 50 Hz mains. It is based on 10 units of 1 MVA each, connected in parallel to the 20 kV input line. The stand-alone peak power is 7.2 MW with a redundancy of 0.8 MW.

Each unit is based on rotating equipment: an alternator, an accumulator and a diesel engine. It is not always easy to realise that the HQPS is able to replace the 20 kV mains without any consequence to the user. The mechanical components tend to be slow compared to the speed at which the lightning strikes the high-voltage distribution lines. Nevertheless the arrangement of the different power components is such that this system reacts fast enough to overcome the consequences of the thunderstorm. The system is equipped with different elements shown in **Figure 1**.

During the normal mode of operation the circuit breaker is closed, the accumulator is charged at 2400 rpm, and the alternator runs at 1500 rpm. In this mode, the alternator alone is able to compensate for any drops below 20%.

When an event occurs on the input mains the alternator compensates to produce a wave within the +/- 5% voltage tolerance. When the drop is such that the energy to compensate is too large (*i.e.* greater than a 10% drop on three phases, 20% on one phase) then the circuit breaker is opened and the accumulator feeds the load for a maximum of five seconds. In the meantime the diesel engine starts up and takes over the load. It takes one second to reach 1500 rpm and then the clutch is closed. The transfer of power between the accumulator and the motor is launched and after three seconds it delivers the nominal power. The diesel engine then recharge the accumulator before reconnecting to the mains, thereby avoiding any vulnerability in the case of a second drop occurring straight after the first one. When the mains is within the voltage and frequency tolerances, the resynchronisation procedure starts in order to return to a normal situation.

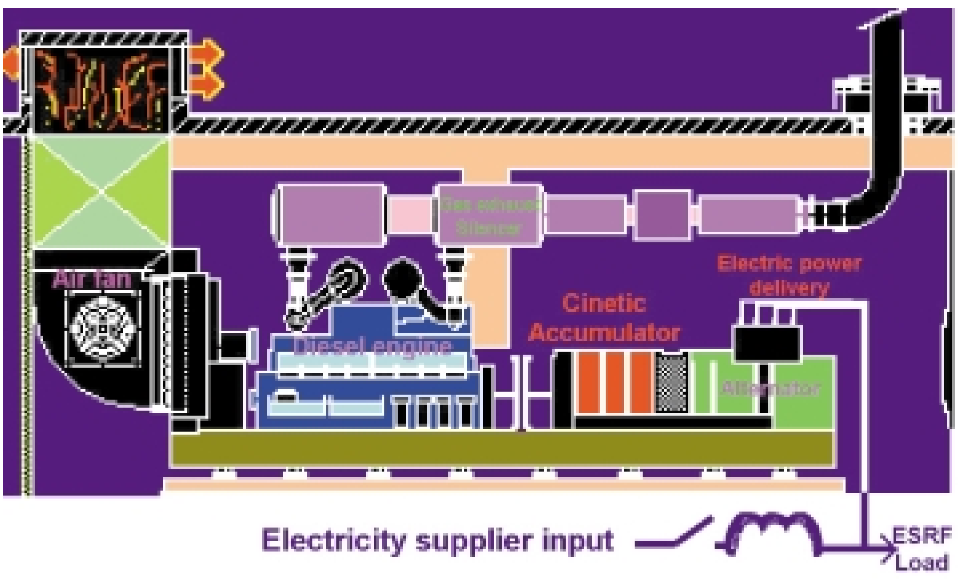


*Fig. 1: Main components of the HQPS.*

### Serious damage

In October 2002, one of the ten diesel engines was found to be seriously damaged after seven years of duty and almost 600 diesel start-ups to compensate for drops. All ten machines were then stopped for investigation. It was clear that all machines were suffering from the same problem, which had developed to various degrees of gravity depending on the engine. In order to determine the exact reason for the damage and hence the responsibility for it, the ESRF has sought legal advice. Upon the ESRF's initiative, the President of the Tribunal de Grande Instance de Grenoble appointed an expert to evaluate the damage and determine the responsibilities. In parallel, authorisation was given to repair eight out of ten diesed engines - two machines remained unchanged for the purposes of investigation. For economy reasons, six were changed and two rebuilt.

The complete beam support was extracted from the building each time and the new motor unit reassembled.

**Figure 2** shows the "flying" 20 tons of equipment during the move.

After three months of intense repair work, commissioning and tests, eight units of the system were put back in operation to protect the storage ring. The two remaining machines will later be needed to shield the injector and recover the initial protection. During the work only 13 hours of beam were lost, thanks to the sunny spring with very few thunderstorms.

*Fig. 2: Extraction of a beam support from the HQPS building.*

## Workshop on Superconducting Wigglers and Undulators

**30 June and 1 July 2003**

The object of this workshop, which was attended by 44 colleagues from 17 different external laboratories, was to review the recent technological developments in the field. The first part dealt with superconducting wigglers. Results were presented concerning very high field wigglers (Spring-8, 10 T and BESSY II 7T) with a semi-cold bore (20 deg K). The performance of the Max-Lab wiggler, which is a short period multipole wiggler with a cold bore, was presented. The second part of the workshop dealt with superconducting undulators. Accel is currently manufacturing two very innovative undulators for the Singapore Light Source and for Anka, which make use of cryocoolers and do not use any liquid nitrogen or helium. A large scale R&D effort is being initiated in the US involving engineers from APS, LBNL, NSLS and SLAC, which consists in engineering studies, the development of various prototypes and the necessary instrumentation for magnetic field measurements. A number of presentations were dedicated to the very important issue of the power deposited at 4 deg K by the electron beam.

The transparencies of the talks are available at "http://www.esrf.fr/Accelerators/Conferences/ID_Workshop/proceedings"

**Pascal Elleaume**

# Events

## X-RAY ABSORPTION SPECTROSCOPY WORKSHOP

### 19-20 June 2003

Coffee break and poster session.

This workshop, held at the ESRF auditorium, was organised by N. Brookes, S. De Panfilis, S. Diaz-Moreno, J. Goulon, T. Neisius, S. Pascarelli and A. Rogalev (Science) and S. Rhoufari (Administration). The aim of the workshop was to present the most recent highlights and to discuss the future perspectives of the XAS technique at third generation sources. A list of 19 invited speakers attracted more than 90 registered scientists from 11 countries, including researchers coming from the main synchrotron radiation facilities in Europe, USA, Brazil and Japan. A few selected abstracts from young scientists and students were included as short oral presentations in the final programme. A total of 23 contributions was divided in six sessions, which animated dynamic and interesting discussions. The diversity of topics ranged from ultra-fast chemical reactions, to catalysis in "operando" conditions, from industrial applications, to environmental science and matter under extreme conditions.

## MEETING OF THE "BIG THREE"

### 2-3 June 2003

On 2 and 3 June 2003 a tripartite meeting between the three high-energy synchrotron facilities APS, ESRF and Spring-8 took place at Argonne National Laboratory attracting more than 70 participants. These 3-way meetings are traditionally held every 18 months on one of the three sites. The aims are to discuss matters of common interest, to exchange information and to identify and conduct collaborative projects. The subjects discussed this time included recent source and beamline developments as well as the latest scientific results. Particular attention was devoted to optics in a satellite workshop on 29 and 30 May and to communication and public relation issues in a parallel session. Detailed information can be found on the APS website: http://www.aps.anl.gov/aps/frame_science.html.

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