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ESRF Newsletter

The ESRF celebrates ten years of User Operation

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Cover:
view of the ESRF
in winter.

One of the very first comparisons of 'absorption' and 'phase contrast' images of a human vertebra, recorded on BM05, at 19 keV, in 1994 (page 9).

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Call for proposals

The ESRF is currently carrying out a design study for x-ray diffraction under pulsed magnetic fields up to 60 T. Such a facility would require the construction of a new beamline, and the development of novel experimental equipment, techniques and data collection protocols — namely, the duration of the 'plateau' region of the magnetic field pulse is on the order of 15msec, and the repetition rate is about 1-3 pulsed per hour.

In order to conceive an experimental configuration that best suits the needs of our future user community the ESRF is carrying out a survey of the potential scientific applications.

Contributions from all areas of hard and soft condensed matter physics, chemistry and biology are welcome. Your contributions will serve as the basis for more advanced discussions in a workshop of the ESRF User Meeting in 2005.

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Editorial

W.G. Stirling - Director General

TEN YEARS OF USER OPERATION AT THE ESRF

In this special edition of the Newsletter we celebrate ten years of User Operation since the inauguration of the ESRF on 30 September 1994. At the inauguration ceremony all twelve Contracting Party countries were represented by senior representatives of science ministries or research councils. Indeed, France, as host country, was represented by M. François Fillon, then Minister of Higher Education and Research who emphasised the important role played by scientific collaboration at the European level and noted the 'extraordinary concentration' of high-level scientific facilities and infrastructure in Grenoble.

In September 1994, the ESRF User Office dealt with 312 scientific proposals requesting beam time on the 11 public and 4 CRG beamlines available from January 1995. At the last proposal round in October 2004 our User Office received 901 applications for the 30 public beamlines and 10 CRGs now operating. In 1994, at this early stage of the ESRF's development, beam time requests were increasing with extreme rapidity – an increase of 36% was registered between the first round (March 1994) and that of September 1994. It is gratifying to note that the ESRF is still very attractive to the European scientific community, with a growth in proposals from 1622 for 2003 to a record of 1675 in 2004.

At the time of the inauguration, 4000 hours of User Service Mode (USM) were programmed for the Machine – this was delivered with an average availability of about 87%. Currently, the Machine delivers 5400 hours in USM with a remarkable availability of close to 98%. The end of 1994 was marked by an important evolution in the ESRF's X-ray source. At that time, plans were finalised for a step-by-step increase in the Machine current from 125 mA in September 1994 to finally achieve 200 mA in November 1995, which has been the routine operating current of the ESRF ever since.

It is instructive to consider those issues that were preoccupying the Management at the time of the inauguration. The first user experiments which started on 1 September had gone reasonably well – the users were generally 'happy' with the beamlines and their equipment, and the lifetime, brilliance, flux and stability of the X-ray beams. But it had already become clear to the beamline staff that there were insufficient scientific staff, resulting in an 'intolerable workload'. In the intervening years, Management has tried to rectify this problem by increasing the number of postdoctoral scientists on each beamline from one to two, and by introducing the Beamline Operation Managers (BLOM). Another on-going issue is that of computing at the ESRF, both technically and organisationally. The rapid evolution during 1994 of the requirements of the different divisions and services using information technology, and the even more rapid advances in computer technology, were creating needs for major upgrades of the system and changes in its architecture. This evolution to satisfy changing demands still continues.

Looking back at the minutes of the ESRF Council Meetings of 1994, and the 'Inauguration' Newsletter of November 1994, I am struck by the many issues that continue to be of importance today. However, the ESRF is much 'bigger' now – in terms of beamlines, experiments, and users – and has matured significantly over the last ten years. As we develop the Long-Term Strategy we are now looking forward to the ESRF of ten to twenty years from now, an ESRF with even more brilliant beams opening up new scientific fields. This special issue of the Newsletter celebrates ten years of user activity. As well as articles on scientific and technical developments over this period, the history of the ESRF, of the Users' Organisation and of the CRG beamlines is dealt with. On the lighter side, photos and anecdotes describe the early 'heroic' days of User Operation. I look forward to the day in 2014 when together we celebrate twenty years of successful User Operation at the ESRF! ●

A shot that has travelled around the world. This picture has been used in posters, magazines and other kind of publications worldwide to illustrate synchrotron radiation. The photo was taken on the 2nd of November 1992, the first day that white beam arrived to the beamline ID11.

Ten years of science at the ESRF

THE MATERIALS SCIENCE GROUP

The Materials Science group provides facilities for a large range of diffraction and imaging experiments. The group comprises seven experimental stations and, after an exciting past, looks ahead to a challenging future.

The Materials Science group started out as the Diffraction group and was in the beginning entirely using diffraction techniques. The first beamlines were ID09 White beam, ID11 Materials Science and BM16 Powder Diffraction, and these beamlines were among the front-runners of beamline construction. In fact ID11 was the first experimental beamline to take beam from a third generation high-energy insertion device on 2 November 1992. It was realised early that two major fields of synchrotron radiation research were essentially missing in the original plans for beamlines at the ESRF. Macromolecular diffraction was only covered by a Laue station ID09 and high-pressure diffraction was completely missing. This was recognised by the group and ID30 High-Pressure and ID14 Macromolecular Crystallography were designed and approved. ID14 was eventually removed from the group but ID15 High Energy was incorporated into a new structure called the Materials Science group, which now comprises seven experimental stations.

The early plans for experimental stations for Materials Science called for access to high-energy radiation and the ID09 and ID11 beamlines were initially powered by wigglers. The rapid development of insertion devices during the last few years made these wigglers obsolete and in-vacuum undulators are now almost entirely used. In fact the prototype in-vacuum undulator was installed at ID11. The success of the undulators also paved the way for an upgrade of the Powder Diffraction beamline BM16 (bending magnet based) to an undulator beamline ID31 and an upgrade of the High-Pressure beamline ID30 to a new station ID27 with better focusing capability. During the same period ID09 was divided into two parts; one for time-resolved studies and one for high-pressure diffraction. High-pressure diffraction at the ESRF started at ID09.

The undulator development was one of three major developments during the first three years. The other two were the emergence of area detectors and the capability for micro focusing.

The first area detectors employed were imaging plates (IP), however the duty cycle proved to be too slow for most experiments and the IP were rapidly replaced by the ESRF developed FRELON CCD camera, which has been the workhorse during the period. In addition to the ESRF detectors several commercial detectors also entered the scene and the approach to diffraction at synchrotron facilities changed completely from the use of 0-dimensional detectors and four-circle diffractometers to fast area detectors with precision motion simpler goniometers.

The third important step was the development of micro focusing of particular interest to the 3D microscope development on ID11 and to the High-Pressure beamlines. The focusing capability has steadily gone from about 0.1 mm to the present limit of about a micrometre without appreciable loss of flux in the resulting radiation spot.

During the period the beamlines of the group have become world leading in their fields.

ID09B is the premier time-resolved beamline in the world with a time-resolution of about 50 picoseconds, ID11 is the pioneer for the 3D microscope for *in situ* bulk characterisation and also for studies of irreversible reactions, BM16/ID31 has allowed high precision powder data to be used for solving very large structures (even proteins), ID09A and ID30/ID27 are the world's premier high-pressure beamlines and ID15 completely dominates the high-energy spectrum above 100 keV and pioneers high-energy imaging. A truly impressive collection of beamlines.

The past has been exciting and the future promises to be even more challenging. ID11 will lead the way with an extension of the beamline to allow nano focusing down to 50 nm opening the field for nano science. All beamlines will work towards a combination of techniques such as imaging and diffraction. Automation both in beamline operation and data analysis will continue. The further expansion into time-resolved and *in situ* studies is foreseen. ●

ÅKE KVICK

FROM IDEALISED SURFACES TO IMAGES OF THE 'REAL' WORLD

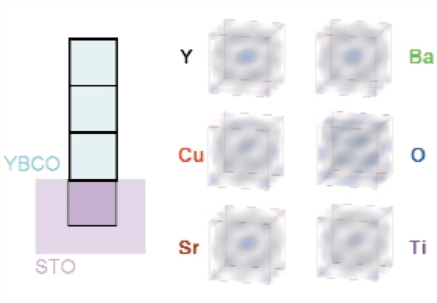
A decade ago, surface physics was mostly dealing with in-vacuum prepared, idealised surfaces, which do not exist in nature. Synchrotron radiation already played an important role and the in-plane structure of clean surfaces and adsorbates was solved by grazing-incidence X-ray diffraction (GIXRD). However, the marvellous beam properties of the ESRF immediately allowed new experiments, which were previously deemed impossible.

From 1994 on, the 3D structures of increasingly complex surfaces and interfaces have been solved routinely at ID03 using GIXRD and crystal truncation rod scattering. Soon experiments moved to more challenging, realistic, and technologically relevant problems: studying epitaxial growth in real time on length scales from pm to µm; unravelling the relationship between structure and magnetism of surfaces by employing resonant magnetic X-ray scattering; revealing the secrets of catalysis by monitoring gas reactions on crystalline surfaces at high temperatures and gas pressure. All of these experiments require an extremely brilliant beam of hard X-rays, which is provided by the ESRF.

In the first user experiment in 1995 at the beamline ID32, which is dedicated to surface X-ray diffraction (SXRD), X-ray standing waves and spectroscopy, we (Jorg Zegenhagen and his group from the MPI-FKF) analysed the $SrTiO_3$(001) (STO) surfaces by grazing-exit X-ray fluorescence. Excitingly, despite the tiny solid angle

Some of the first users of ID03 celebrating with ESRF staff the success of the first experiments on the beamline.

(10^{-4} sr), the detector was running at maximum count rate. Our successful experiment was rewarded with an excellent Italian dinner at the home of the beamline scientist in charge, who was Fabio Comin at that time. Most experiments, then spectacular, are now ordinary and, unfortunately, no longer rewarded this way. We now solve structures of surfaces in contact with UHV, gas, liquid, solids with large unit cells (> 100 atoms) under static and dynamic conditions, aiding the understanding of problems such as the corrosion of metals and the functioning of novel gate oxides. Images of the elemental structure of complex adsorbates (see Figure) can be reconstructed by combining X-ray Photoelectron Spectroscopy (XPS) with X-ray Standing Waves (XSW).



Model (left) and images (right) of the elements of STO and $YBa_2Cu_3O_7$ on STO obtained by XSW/XPS at ID32.

ID01 became operational in 2000. It was designed in the nineties for X-ray small and wide-angle scattering (SAXS and WAXS) near absorption edges. Partial structure factors from amorphous materials, liquids and solutions were determined. In pioneering experiments, using SAXS at grazing incidence (GISAXS), nanometre-sized aggregates and interface roughness correlations in thin solid films, and highly-oriented, stacked lipid membranes on solid surfaces were investigated. Now, work at ID01 focuses on solving morphology, structure, strain, composition, and correlation of low-dimensional structures *e.g.* semiconductor quantum wires and quantum dots on or close to the surface. The small dimensions and the corresponding small scattering signal require the highly brilliant, tuneable synchrotron radiation offered by ESRF. The results help us understand and improve self-organised growth to increase the performance of novel electronic and/or opto-electronic devices based on quantum confinement effects.

Outstanding X-ray properties and experimental facilities allow unique studies in Surface and Interface science, but there is still room for improvement. Owing to the small source size, focusing to sub-μm beamsize will allow the study of single quantum dots, single grains, or whisker surfaces. The brilliant beam will not only allow the coherent imaging of nano-sized objects but will also permit XPS with some 10 meV resolution and XSW imaging with chemical sensitivity and unprecedented resolution. ●

TILL METZGER
JORG ZEGENHAGEN
SALVADOR FERRER

OPTICS FOR THE ESRF

When the first ESRF beamlines opened for User Operation, X-ray optical elements had to be ready to condition the ultrabright beams of a 3rd generation synchrotron source without degrading their quality. The early decision to set up an Optics Group and a dedicated test beamline as part of an ESRF internal long-term development strategy enabled to meet this goal and to achieve substantial improvements and innovations.

At that time, beams were typically a millimetre wide. Total reflection mirrors and perfect single crystals were the most widely used optical elements. Focusing setups obtaining focal spots of about 10mm were the state of the art, and the best optical surfaces had slope errors of about 1mrad. What was considered to be a major challenge only one decade ago nowadays appears to be modest. The performances of all types of optical elements have been improved by at least one order of magnitude. Spots of 1mm can be provided routinely and special setups achieving 100nm spots with a flux gain exceeding 106 are available. Surface characterisation techniques reach the 100nrad level. On top of the general progress in X-ray optics performance, many innovative developments were made at the ESRF.

Single crystal preparation and cryogenic cooling

The crystal preparation laboratory has developed cutting edge techniques to fabricate single crystal monochromators for extreme requirements like nuclear resonance scattering. The heat load on the optics of 3rd generation light sources is extremely high. Here the ESRF pioneered the theoretical and the experimental work on cryogenic cooling of silicon crystals.

Diamond optics

The exceptional thermal properties of diamond make it an excellent material as high heat load monochromator. It took years of careful studies at ESRF in collaboration with industry to produce diamond crystals of the required size and quality that are also used as polarisers on many beamlines.

Multilayer (ML) coatings

The design and fabrication of dedicated ML interference coatings for hard X-rays has bridged the gap between high-resolution crystal optics on the one and total reflection mirrors on the other extreme. MLs are the element of choice when beams with high flux and medium energy resolution are required. Tailored lateral and in-depth thickness gradients make MLs very flexible optical elements that can be adapted to the given experimental needs.

Kirkpatrick–Baez (KB) focusing devices

The arrangement of two crossed and curved mirrors is an excellent focusing method, down to spots of 100nm. The optical alignment and surface figuring is done by automatic alignment routines so that KB systems have become user-friendly. Merging KB devices with graded ML coatings improved the throughput significantly.

Benders and metrology

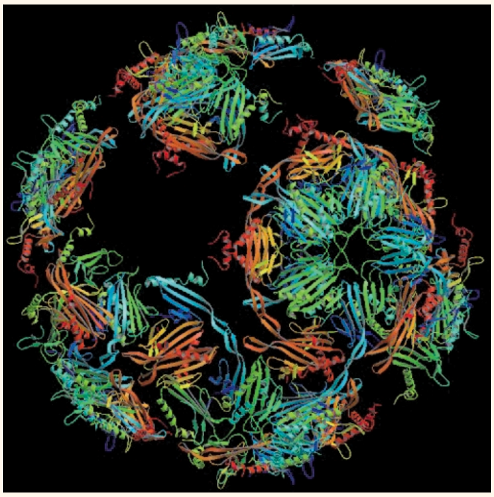
The precise characterisation of flat and curved optical surfaces is mandatory to improve their quality. The metrology laboratory has made a crucial contribution to their successful operation on various beamlines. In collaboration with Technical Services, dynamical bending devices were developed that are the backbone of focusing optics based on crystals, mirrors and multilayers.

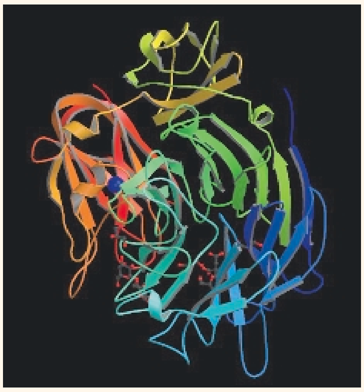
Compound Refractive Lenses (CRL) and derivatives

Lenses based on refraction are perfectly matched to focusing undulator beams of 3rd generation light sources. Pioneering work was done at ESRF in collaboration with external institutes to develop and test CRLs as novel optical elements for hard X-rays. At present, a variety of circular and planar CRLs is available for use or under development. These devices can focus to below 1 μm. They are simple to install and to align and are very stable.

X-ray optics development is ongoing. Ambitious projects to improve the brilliance of the X-ray source and ever tighter experimental requirements on beamlines call for still better optics. Increasing scientific efforts towards the exploitation of nano-technologies will push optics towards 10nm resolution. The strength of the ESRF Optics Group is the variety of optical elements it can produce, the experimental facilities for fabrication and evaluation, and in particular the expertise of the staff. At this point we want to express our sincere thanks to many external collaborators. ●

CHRISTIAN MORAWE
ANDREAS FREUND





Images of the earliest and latest structures resolved at the ESRF beamlines.

EVOLUTION OF MACROMOLECULAR CRYSTALLOGRAPHY

The number of Macromolecular Crystallography (MX) experimental facilities at the ESRF has evolved enormously in the ten years of ESRF public operations. In 1994, one shared beamline (ID02B) was available to the community. By 2004 this number had risen to seven ESRF plus three CRG beamlines dedicated to MX.

The initial impact of ID02 was nothing less than revolutionary. The combination of high X-ray intensity, small focal spot-size and low beam divergence led to a quantum leap in the imagination of what was actually possible for MX with synchrotron radiation beamlines. However it was clear that the demand for synchrotron access would not be met by a half share of ID02B (and the soon-to-appear BM14), thus the construction of the four end-station beamline ID14 began around this time. Beamline BM14 came online during 1995 and rapidly became 'the place' in Europe for performing Multi-wavelength Anomalous Dispersion (MAD) experiments. Many 'impossible' protein structures were elucidated, and the bounds of the imaginable were further extended. Construction of the four end-stations of ID14 continued until the year 2000. During the construction period the operational beamlines of ID14 rapidly established a strong reputation, and subsequent to completion has become one of the highest performing beamlines in the world in terms of structures deposited to the Protein Data Bank (PDB).

Meanwhile, and despite its excellent performance, it was decided to move BM14 to an insertion device (ID29). This decision was taken as a result of the clear advantages of undulator-based MAD beamlines over bending magnet-based instruments, as demonstrated at ID14-4, the APS and other facilities. The final portion of the ESRF's portfolio (ID23) is still in development. When completed, this beamline will comprise two end-stations for macromolecular crystallography: a tunable MAD beamline (ID23-1 already operational) and a fully dedicated microfocus facility, ID23-2.

The construction and operation of such a large number of beamlines during a period of intense activity at the ESRF would not have been possible without the efforts of both the committed and professional MX team, and the enthusiastic advice and backing of the ESRF's support group infrastructure. Instrumentation specific to MX has been developed by the ESRF MX group and the EMBL-Grenoble's instrumentation group (a close and enduring collaboration that was forged during the conception of ID02B). This collaboration has resulted in many instruments for MX, in particular the microdiffractometer and the sample changer currently in construction for all ESRF beamlines.

Today the ESRF runs a successful MX programme with more than 2000 visitors arriving for peer reviewed experiments each year. In addition, a significant percentage of the beam time access goes to support proprietary research. The future of MX research at the ESRF is secure and is being extended through the development of the Partnership for Structural Biology (see Newsletter 39). ●

SEAN MCSWEENEY

TEN YEARS OF X-RAY IMAGING AT THE ESRF

The X-ray Imaging field has experienced a lot of improvements since the first days of beam. Today, the demand for beam time in the X-ray Imaging beamlines is five times higher than the beam time available.

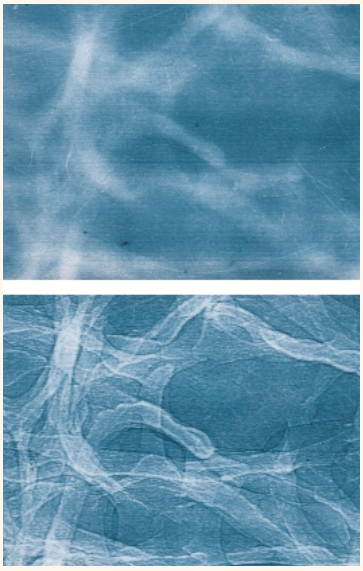
How it all began

Preliminary X-ray imaging experiments were performed as soon as beams were available on the first ESRF beamlines: this included the first topography images on ID11 and scanning imaging experiments on ID13. Imaging and microanalysis were an important part of the second beamline construction phase, with the long (150 m) Medical and Topography beamlines, as well as the Microscopy ID21 and Microfluorescence ID22 beamlines.

The scientists realised rapidly that the association of the beam's features with the new detectors (for instance the FreLoN camera, developed at the ESRF) and computers allowed easy use of phase contrast techniques, and the implementation of high-spatial resolution microtomography. These partly unexpected developments were first tested on the Optics beamline BM05 and then developed in the hard X-ray range on ID19 and ID22. A reliable and robust X-ray microscope was built on ID21, and allowed a combination of imaging and spectroscopy techniques. The coronary angiography programme was implemented at ID17. It was based on the K-edge subtraction method and the programme required specific optical components and complex instrumentation such as the 'medical chair'.

Successful techniques

The current names of the beamlines of the X-ray Imaging group show their evolution: ID17 is now called Biomedical, ID19 Tomography & Topography, ID21 X-ray Microscopy & Microanalysis and ID22 Micro-Fluorescence-Imaging and Diffraction. The work performed on these beamlines can be characterised by a few key words such as 'three-dimensional', 'high-spatial / temporal resolution', 'coherent beams', '*in situ*', 'adapted sample environment' and 'combination of techniques'. The enhancement of the possibilities of the imaging/microanalysis techniques, and their applications to a range of topics (which include physical, medical, materials science and engineering subjects, but also new areas like geophysical, environmental, archaeological and biological studies) characterise the present period.

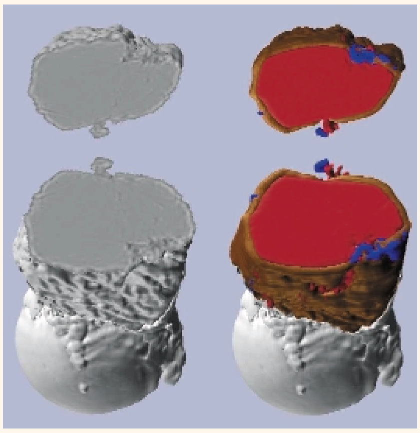


One of the very first comparisons of 'absorption' (sample to detector distance = 6 mm) and 'phase contrast' (same distance = 1 m) images of a human vertebra, recorded on BM05, at 19 keV, in 1994 (P. Cloetens, M. Salomé *et al*, Highlights 1994/1995).

Today, ID17 covers many aspects of the synchrotron radiation-based biomedical research. The parallel and sometimes coupled development of diagnostic, functional imaging and radiation therapy constitutes the main of this work. For instance, very encouraging survival curves have been obtained on rats bearing a glioma type brain tumour. These programmes are based on a strong collaboration with local and European hospital teams, and the efficient use of the ESRF Biomedical facility.

The neighbouring microscopy and microanalysis beamlines ID21 (low energy 2-7 keV), ID22 (high energy 7-60 keV) and ID18F end station (6-28 keV) are complementary. They allow various techniques that include tomography, fluorescence, absorption spectroscopy and diffraction, to be combined for two or three-dimensional microanalysis on the same sample. An infrared microscopy end-station is currently under development to extend the range of microanalysis techniques.

Synchrotron radiation wide field imaging (topography, microtomography) is now applied to a large variety of topics, with an intensive use of phase contrast and an



Microtomography of a fly-ash particle: on the left a 3D rendering of the 'absorption' microtomographic image is shown; on the right additional chemical information -distribution of Rubidium (red), Manganese (brown) and Iron (blue) - is obtained through the combination of techniques (Compton and fluorescence microtomography). Voxel size 3x3x3 µm^3 (B. Golosio *et al.*, Highlights 2003).

increasingly quantitative analysis of the results. Microtomography is now available on a series of beamlines (BM05, ID15, ID17, ID19, ID22) to cover the academic (and industrial) needs, the requested time being recurrently a factor 5 above the available time.

The perspectives

The techniques implemented at the ESRF also develop outside of the synchrotron radiation world. The common wish of the ESRF staff and of our users is to take full advantage of the specificities of third generation synchrotron radiation facilities, which are not available elsewhere. The biomedical topics that have an actual fundamental or clinical relevance and where synchrotron radiation is the best tool are now well identified. The ESRF will concentrate on these topics, which in the future will include clinical radiotherapy. On the other hand, to be able to answer the scientific and technical questions associated with modern science and technique, synchrotron radiation microtomography is moving towards higher spatial and temporal resolutions. The temporal resolution (now in the 10 seconds range) is important for a wide range of evolving phenomena, and should go towards the sub-second range. The spatial resolution, today in the 1 µm range, should be improved up to the 50 nm range (nanotomography project). To succeed in this project we will need to develop more precise focusing optics and more sensitive detectors. ●

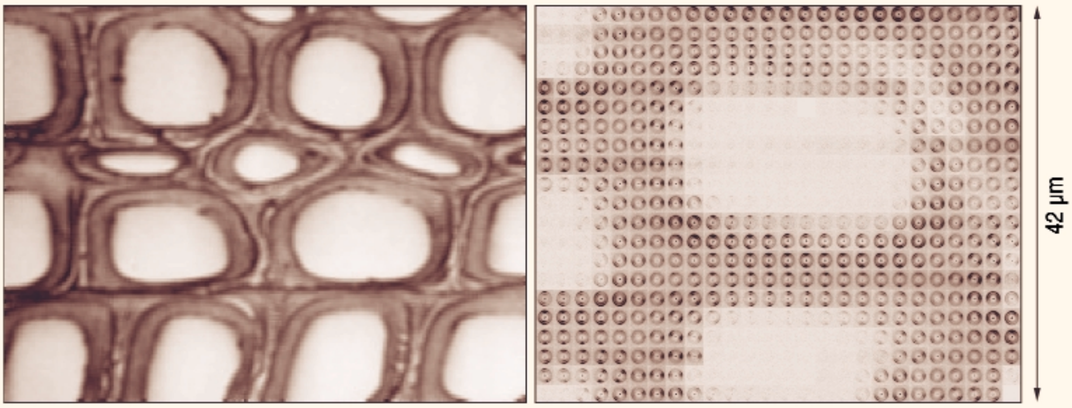
JOSÉ BARUCHEL

SOFT CONDENSED MATTER GROUP

The soft condensed matter group is currently composed of beamlines ID02, ID10A/B and ID13. Experimental techniques like small- and wide-angle scattering (SAXS/WAXS/GISAXS) or dynamic X-ray scattering (XPCS) address questions on microstructure, kinetics, dynamics and rheology of complex and nanostructured materials.

The High Brilliance beamline, ID02, is a state-of-the art SAXS/WAXS/USAXS instrument optimised for high flux, resolution, and stability. In a way, it is a culmination of several generations of SAXS/WAXS instruments developed at 2nd generation sources such as Hasylab. As a result, time-resolved experiments in the millisecond range can be performed rather routinely even with low scattering samples. For instance, the high resolution permitted to resolve the interference fine structure of the axial repeat of myosin molecules in a contracted muscle sarcomere and to use it as a tool to monitor Angstrom scale molecular motions during the 'working stroke'. For USAXS applications, a second branch with a Bonse-Hart camera has been developed, which complements the USAXS range covered by the pinhole SAXS/WAXS camera. Last but not least, these techniques are combined with a variety of advanced sample environments specifically suited for soft matter research.

At the origin of the Troika beamline, ID10, was the idea of constructing a modular 'open undulator beamline' for rapid testing of new scientific ideas and instrumentation. The novel concept of diamond beam splitters allowed the separation of ID10 into two independently operating beamlines. The pioneering work on diamond crystal monochromators in collaboration with the ESRF optics group is still of considerable interest in view of future increases in machine current and source brilliance. Photon correlation spectroscopy with coherent X-rays (XPCS) was a topic co-invented on the ID10A beamline and has proven in particular to be useful for studies of soft matter. In the beginning, mostly model systems were investigated yielding the proof-of-principle, and nowadays XPCS is used in various scattering geometries



Left: optical microscopy of spruce wood cells. Right: composite image of a spruce wood cell obtained by scanning diffractometry using a CCD-camera (3 micron beam,13 keV). The composite image shows the cell outline and allows determining the cellulose nanofibril orientation in the cell wall S2-layer. Lichtenegger *et al.*,J.Appl.Cryst.(1999) 32,1127.

to measure surface and bulk dynamics. The ID10B beamline has evolved in the area of surface scattering techniques and has in particular developed a Langmuir trough for *in situ* studies of two dimensionally organised molecules and macromolecules. The possibility of scattering experiments on liquid surfaces is also unique. Many surface scattering experiments such as GISAXS on quantum dots have been pioneered on this beamline.

The Microfocus beamline, ID13, has attracted a range of X-ray optical systems allowing the generation of micrometre or sub-micrometre X-ray beams. Thus a 0.7 micrometre beam from a Bragg-Fresnel lens was already demonstrated in 1994. Systems like capillaries, waveguides, Fresnel lenses, refractive lenses or KB-mirrors were mostly tested in collaboration with external groups. Scanning SAXS/WAXS experiments have been particularly useful for the study of hierarchical materials such as the wood ultrastructure. (see Figure) Early on, single crystal microdiffraction techniques were applied to protein structures such as the Nucleosome core particle. The current success of microdiffraction techniques in protein crystallography owes very much to the development of a user-friendly microgoniometer developed in collaboration with the EMBL. Routine use of nanobeams down to currently about 100 nm is gaining more and more importance.

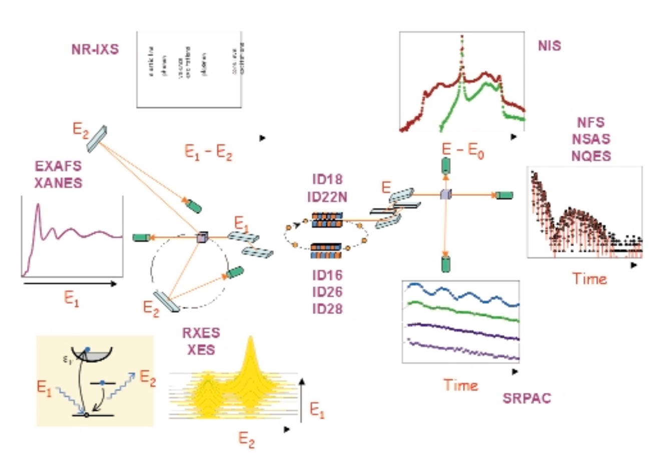
What are the instrumental and scientific challenges for the soft condensed matter group? New generations of area detectors with high sensitivity (single photon counting!), small pixels size (<100 microns) and rapid readout (extending into sub-millisecond-range) will open new areas of science. Rapidly developing beamline automatisation and on-line data reduction techniques will facilitate the work of beamline scientists. Novel optical systems, such as refractive lenses, will provide the possibility to simplify instrumentation. Developments in micro- and nanoscience will present new instrumental challenges. The complexity of systems, in particular in-vitro or in-vivo studies on biological systems, calls for a much closer interaction between beamline scientists and external users. Bridges to neighbouring disciplines have to be strengthened. ●

CHRISTIAN RIEKEL

HIGH RESOLUTION AND RESONANCE SCATTERING GROUP: FROM VISION TO REALITY

The spectroscopic tools gathered in the HRRS group encompass a variety of technical and scientific fields. During the foundation phase of the ESRF these techniques were merely dreams. Today they have become reality and combine tools suitable for the investigation of electronic and magnetic properties as well as dynamics in a very general aspect.

The study of inelastic processes with milli electron volts (meV) and sub-meV energy resolution opened up new fields of research, complementing the well-established inelastic neutron and light scattering techniques. An important application concerns the high-frequency dynamics of disordered systems in a previously unexploited energy-momentum region. As the most prominent example we cite the still ongoing inelastic



Scheme of the experimental techniques covered by the beamlines of the High Resolution and Resonance Scattering group. ID18 and ID22N are dedicated to nuclear resonance applications with nuclear inelastic scattering (NIS), nuclear forward scattering (NFS), nuclear small angle scattering (NSAS), nuclear quasi-elastic scattering (NQES), and synchrotron radiation based perturbed angular correlation (SRPAC). ID26 is dedicated to absorption spectroscopies such as EXAFS and XANES and together with ID16 (partly) to electronic excitations applying resonant and non-resonant X-ray emission spectroscopy (RXES, XES). Finally, ID28 and ID16 (partly) are dedicated to high-resolution non-resonant inelastic X-ray scattering (NR-IXS).

X-ray scattering studies on the water-ice system, which provided invaluable insights concerning sound propagation and attenuation, relaxational phenomena and similarities between its amorphous, crystalline and liquid phases.

The recently developed method of nuclear inelastic scattering for measurements of the density of vibrational states allowed for isotope-specific and, therefore, site-specific investigations of vibrational properties of solids, glasses, and proteins. This provided decisive results for the understanding of the structure and dynamics of macromolecules and functional properties of proteins. Extending the time scale of accessible processes, the time-resolved experiments on nuclear resonant scattering with nanoseconds time resolution gave access to slow dynamics in the pico- to msec range. The combined application of coherent and incoherent nuclear scattering allowed the separation of various dynamical processes such as translational and rotational diffusion.

Complementing the conventional absorption spectroscopies, resonant and non-resonant inelastic X-ray scattering techniques allowed the study of electronic excitations with an increasing selectivity, thus unravelling weak excitation channels, electron correlation and subtle valence changes. The ability to study the electronic structure of highly-diluted systems greatly enhanced the investigation of catalytic reactions in industry and nature. Fundamental processes such as dioxygen production in photosynthesis can now be studied with unprecedented selectivity. With advancing instrumental developments it will become possible to study electrons in chemical reactions on a pico-second time-scale. The combination of high-energy resolution with a microfocused beam will allow one to map the chemical state of an element across a sample under *in situ* conditions.

Moreover, these advanced studies can be performed on very small samples (down to 10^{-5} mm^3) allowing investigations under extreme conditions such as high-pressure and of nano-structured materials. Studies under extreme conditions of temperature and pressure give access to thermodynamic, elastic, electronic, and magnetic properties of materials of interest in fundamental and applied research. In particular, the study of iron and iron bearing compounds had a major impact in Earth and Planetary science. Other investigated systems range from elemental solids such as graphite, samarium, and plutonium to advanced materials like 'unusual' and high-temperature superconductors, nano-particles, and giant-magneto-resistance films.

Microscopic studies of magnetism with time-resolved nuclear resonant scattering shed new light on magnetic properties of solids. Measurements have made it possible to establish magnetic phase diagrams in extended pressure and temperature ranges for strongly correlated electron systems, for instance. Applying the probe-layer technique provides sub-monolayer sensitivity in magnetic and dynamic investigations.

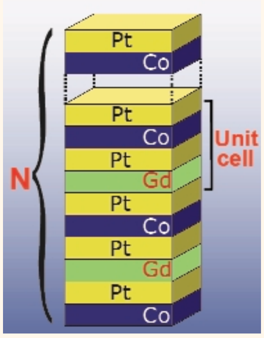
The continuous improvements of the X-ray source, X-ray optics and the spectrometers will advance the present limits in terms of energy and time resolution, sample size and dilution, and pave new avenues towards the study of dynamic, electronic, and magnetic properties of surfaces and in confined systems, low-energy electronic excitations and long-time atomic dynamics. In particular, exploiting the excellent time structure of the synchrotron light in combination with a challenging fast detector development programme will access new time windows in the world of slow and fast dynamics of electronic, magnetic, and structural properties. ●

RUDOLF RÜFFER & HRRS GROUP

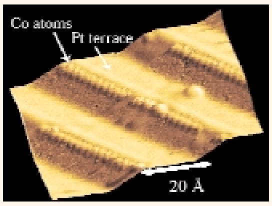
X-RAY ABSORPTION AND MAGNETIC SCATTERING GROUP

The present group has evolved over the past ten years from the original X-ray Absorption group to the present five beamlines, following the inclusion of the Magnetic Scattering beamline in 2002. This evolution will surely continue in the coming years as we strive to improve further.

Ten years ago the 'beamline 6' project, led by José Goulon, was comprised of two branches covering the X-ray ranges 2-20 keV (ID12A) and 0.5-1.5 keV (ID12B). The idea was to exploit X-ray polarisation (both circular and linear) dependent spectroscopy. This was highly successful and led to separating the soft X-ray branch line to ID08 and the full utilisation of the ID12 straight section for hard X-rays in January 2001.

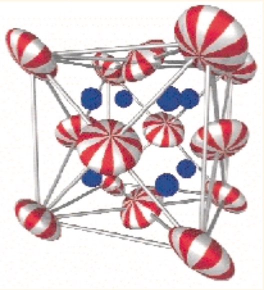


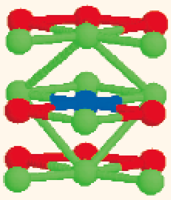
On ID12 there have been many significant contributions in X-ray magneto-optics *e.g.* X-ray magnetic circular (linear) dichroism and X-ray resonant magnetic scattering. X-ray optical activity has also been explored revealing new phenomena such as X-ray natural circular dichroism, X-ray magnetochiral dichroism and non-reciprocal X-ray magnetic linear dichroism. For the future we can expect many new developments particularly in 'pump-probe' type measurements.



In the soft X-ray range, dichroic effects have been exploited to study magnetic thin films. Today magnetic impurities and dimensionality effects can be studied with thousandths of a monolayer sensitivity. Large strides have been made in understanding transition metal oxides utilising X-ray emission, X-ray photoemission spectroscopies and new tools such as soft X-ray magnetic scattering. In the future the time domain and the coherence of the beam wait to be fully exploited.

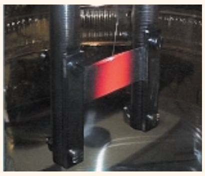
The ID20 beamline was specifically designed for X-ray magnetic scattering. In the past years many ground-breaking experiments have been carried out using resonant and non-resonant X-ray scattering from single crystals and thin films. More recently, the use of X-ray polarisation analysis (both circular and linear) and angular dependence measurements (azimuthal scans) have yielded a rich variety of new results. They have in turn widened the research field and stimulated theoretical activity. This is particularly true for understanding multipole transitions and the magnetic ground state properties of strongly correlated electron systems. New sample environments, high-pressure, low temperature and the recent custom 10 Tesla magnet expand further the horizon for the future years.





Turning to the Extended X-ray Absorption Fine Structure (EXAFS) beamlines, ID24 was the first undulator based dispersive EXAFS beamline and its fast time resolution and small spots have allowed XAS studies of dynamical processes and high-pressure work.

In addition, the inherent stability has opened up areas such as X-ray magnetic circular dichroism at high pressure. Today the time resolution is 10-100 times better than originally envisaged, with studies of non-repetitive kinetic processes in the msec range and much faster for repetitive processes. Future trends include combining other in situ time-resolved spectroscopies such as IR, UV/VIS. Also micro-XAS, even under high pressure (5 x 5 microns^2 spot) and differential XAS methods including temperature, pressure and magnetic field 'jump' methods could be exploited.



Working closely with ID24 is the general purpose of EXAFS beamline BM29, which has been at the centre of many of our activities. The beamline utilises its exceptional luminosity and stability for state-of-the art conventional experiments and to extend the technique into a wider range of sample environments. Today BM29 is a highly automated beamline able to perform experiments over a wide energy range (4-76 keV) and providing a variety of possibilities including high-pressure, high and low temperatures, liquid samples, refleXAFS, temperature scans.

The future will bring the integration of more complementary techniques and this will be helped by the closer synergy of activities between ID24 and BM29, thereby providing possibilities not available separately. ●

NICK BROOKES

HISTORY OF THE CRGs

In 1990, the ESRF Council agreed that the Collaborating Research Groups (CRG) could be formed. They would be able to construct beamlines at ESRF bending magnet sources to improve access to synchrotron radiation for scientists from countries lacking, at that time, national sources, providing that this did not divert either manpower or funds from the ESRF's own programme.

In return for the X-ray photons being delivered to the CRGs free of charge, they have to make 1/3 of their beam time available for ESRF users.

CRG	BL	NATIONALITY	TECHNIQUES, SCIENTIFIC AREAS	OPERATION SINCE
SNBL	BM01A	Swiss-Norwegian	Single Crystal Diffraction	1995
	BM01b	Swiss-Norwegian	Powder Diffraction + EXAFS	1995
D2AM	BM02	French	Hard Cond. Matter, Structure + Anomalous Scattering	1994
GILDA	BM08	Italian	EXAFS + Diffraction	1994
	BM14	British	Diffraction (Macromolecular Crystallography)	2001
	BM16	Spanish	Diffraction + Scattering (Macromolecular Crystallography + SAXS)	2003
ROBL	BM20	German	EXAFS (Radiochemistry) + Diffraction/Reflectometry	1998
SPLINE	BM25A	Spanish	EXAFS + Powder Diffraction	2005
	BM25B	Spanish	Surface/Interface + Single Crystal Diffraction	2005
DUBBLE	BM26A	Dutch-Belgia	Protein Crystallography + EXAFS	2003
	BM26B	Dutch-Belgia	SAXS and WAXS + Interface Diffraction	1998
XMAS	BM28	British	Magnetic Scattering (Materials Science)	1998
FIP	BM30A	French (1)	Diffraction (Protein Structures)	1999
FAME	BM30B	French	EXAFS (Materials/Environment)	2002
IF	BM32	French	Surface and Interface Diffraction (Interface Studies/ Soft Matter Materials)	1994
GRAAL	BM07	Italian	Photoproduction of particles, using Gamma-rays produced by Compton back-scattering	1995

(1) With a 10% involvement of the Belgium FNRS

One of the first CRGs to become operational was BM32. It is devoted to surface and interface studies and addresses, together with researchers from nearby local research laboratories and industry, the characterisation of interfaces within microelectronic devices, tackling fundamental problems like molecular bonding between silicon wafers. Grazing incidence X-ray diffraction complemented with small angle scattering yields information about the morphology or topography on nanometre scale. The beamline will be refurbished to aim at sub-micrometre beams and microdiffraction next year.

The Swiss-Norwegian CRG beamlines became operational in 1995, after a decision to split the beam from bending magnet BM01 into two independent lines. Over the years, the data collection rate improved by an order of magnitude and the experiments have become more complex. After having made substantial investments in improved detector technology, this beamline will now look at new X-ray optical configurations to open up more scientific opportunities for their community.

The British CRG for magnetic scattering (XMaS) had its first users in 1998. Its experimental equipment has been updated over the past few years and now comprises sample environments covering a temperature range from 1 to 800 K, a wide-access magnet of 1 Tesla and a MAR CCD camera. The beamline will soon be equipped with a 4 Tesla warm-bore magnet together with a narrow-bore cryostat covering 1-300 K. The science at XMaS is dominated by magnetism studies (mostly resonant), followed by surface studies and correlated systems.

A rather particular CRG, due to the necessary safety precautions, is the German Rossendorf beamline, which is equipped to perform X-ray absorption spectroscopy on alpha-decay radionuclides. The principal task is the chemical speciation of actinides and other radionuclides, stimulated by interest in the fields of basic aqueous chemistry, migration and biogeochemistry of radionuclides. Scientific highlights related to these recent developments are the identification and characterisation of unstable U(IV) species in aqueous and chloride solutions and of U(IV) and U(VI) species in soils contaminated by U mining and DU penetrator ammunition.

The Dutch-Belgian beamline has been operational since the end of 1998 with its SAXS/WAXS station and since 2003 with a second branch offering (time-resolved) EXAFS techniques. It produces a steady stream of publications making use of experimental equipment ranging from a 10 Tesla superconducting magnet to a 30 Tesla pulsed magnet, from flame boxes for the study of soot formation in live flames to more mundane equipment like cone and plate rheometers and tensile testers. Light scattering, differential scanning calorimetry, di-electric spectroscopy etc. are some of the tools that can now be used simultaneously with X-ray experiments.

The most recent beamline to join the CRGs is the former ESRF beamline BM16, which was purchased by the Spanish Ministry of Science and Technology. Since 2003, after a major refurbishment, it is dedicated to macromolecular crystallography and small angle scattering. One of the earliest experiments carried out on BM16 led to the *ab initio* structure determination of a DNA-drug complex. A further Spanish multi-purpose beamline will become operational at the beginning of 2005. ●

EDITED BY AXEL KAPROLAT

TEN YEARS OF USERS' ORGANISATION HISTORY, TOO

The historical records on the beginning of associative life of ESRF users are scarce. In the early days of User Operation the urgency and excitement for the brand new science clearly eclipsed concerns about future synchrotron radiation historians!

The original charter (1997) is the first official document to mention an 'Association des Utilisateurs' which was registered according to French law. Remarkably, one of the paragraphs of this document is entitled 'radiations' - but only to enumerate reasons for 'exclusions' from the organisation! Less officially, but with great enthusiasm, an organisation of ESRF users was already active from 1994. H.-P. Weber (Lausanne and Swiss-Norwegian CRG beamline) played then the role of main catalyst. His 'CRG club' rapidly evolved into a full-scale Users' Organisation (UO), which was immediately supported by the ESRF management.

The newly-born UO certainly did not waste time. It was already involved in the organisation of the 1995 ESRF Users' Meeting. On that occasion, displaying some flair, the UO presented the first Young Scientist Award to Francesco Sette, who was later to take up the position of Director of Research. One year later a very successful 'Science at the ESRF' conference made history by inaugurating the present format of the Users' Meeting, with associated satellite workshops, but also for a memorable exhibition of Polynesian dancers. The UO is still closely associated with the Users' Meeting, which it organises with the User Office. Each annual meeting has been a unique scientific and social experience, and the UO has strived to follow the evolution in the scientific interests of the users' community. Recent editions, in particular, have put an increasing emphasis on scientific and technical discussion workgroups. Over the years, the international reputation of the Young Scientist Award has grown, and its winners already make up a remarkable list of successful scientists.

Today, the ESRF Users' Organisation is an independent body representing more than 5000 registered ESRF users, from all of Europe and the rest of the world, and acts as a direct link to the ESRF's directors. Its mission is to identify and bring to the attention of the ESRF Management issues of interest and concern for the users. The UO has also a direct link to the Science Advisory Committee (SAC), and an UO representative is traditionally invited to attend SAC meetings. Ordinary business is conducted by a Users' Organisation Committee, in charge for 2 years, which reflects as much as possible the broad spectrum of research done at the ESRF and the geographical composition of the user community.

The Users' Organisation has been a partner of the ESRF management in discussions on science and policy issues, and is currently involved in talks over the long-term scientific perspectives of the facility. It is also very much concerned with more mundane problems, which nonetheless touch the everyday life of users. Keeping up the standards of the local canteen has been a permanent battle, with occasional successes like the 'V (for vegetarian) – day', still celebrated by many users. The recent network installation for Internet access of the guest house was also a highly-appreciated improvement of the 'user environment'.

The fruitful collaboration between the ESRF and its users is the result of the genuine and continuing interest of the ESRF management for users issues and opinions, and of the personal commitment of all the individuals who served in the Users' Organisation committees. It is up to old and new users to continue this tradition, and to use their Organisation as an effective tool for the next crucial ten years, and beyond. ●

M.GRIONI WITH H.-P. WEBER, S. PASCARELLI,
M. COOPER, K. HÄMÄLÄINEN

15th ESRF Users Meeting
8–9 February 2005

The annual ESRF Users Meeting will be held on 8 and 9 February and will feature

- a presentation and discussion of technical and strategic long-term options for the facility

- the Young Scientist Award and talk a special celebration of ten years of User operation.

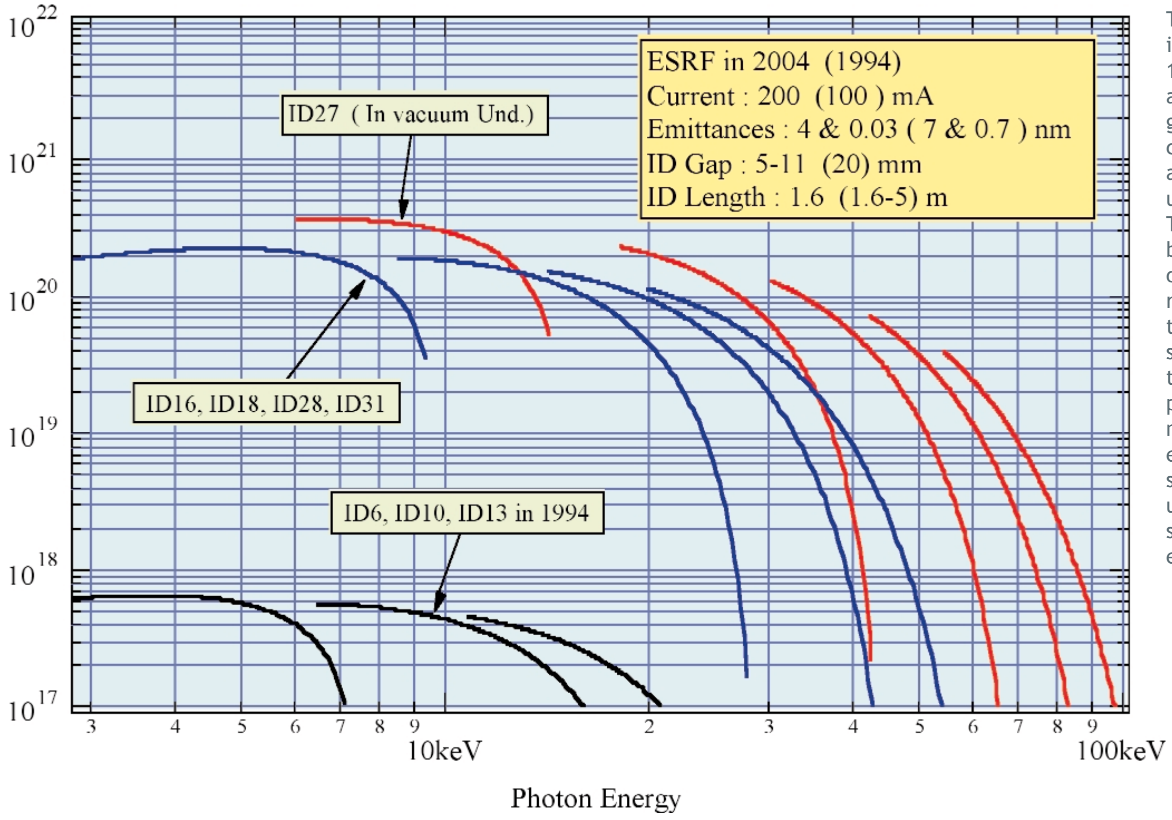
Satellite workshops:

Synchrotron Radiation in Art and Archaeology

Synchrotron radiation techniques provide powerful new ways to investigate records of our physical and cultural past. The purpose of this workshop is to discuss and explore the current and potential applications of synchrotron science to problems in archaeology and art conservation. Bringing together key members of the synchrotron community and experts in the disciplines of Archaeology, Archaeological Science, Art Conservation and Materials Science. This interdisciplinary workshop aims to report the latest research accomplishments, highlight ongoing projects, and catalyse new interactions between fields.

New Science with New Detectors

It is becoming increasingly clear that the next major advance in synchrotron science will come via drastically improved or revolutionary detector concepts. This workshop will look at the future science at synchrotron radiation facilities, and discuss the requirements for the detection systems needed.



The brilliance generated in 2004 by a 5 m long, 11 mm gap undulator and a 4 m long, 6 mm gap in-vacuum undulator compared to that available from the early undulators in 1994. The increase in brilliance is about 2.5 orders of magnitude and results from upgrades to the lattice, the RF system and ID technology, as well as progress in beamline mechanical and thermal engineering. Thanks to small-gap operation the undulator spectra have shifted to higher energies.

The Machine

THE MACHINE FROM 1994 TO 2004: FROM INFANCY TO MATURITY

The ESRF was the first third generation light source in the world. The first beam was stored in the ring in early March 1992 and the facility was opened to European scientists in 1994. Since that time, the characteristics of the beam delivered to the beamlines have evolved dramatically.

The most classically used figure of merit for third generation synchrotron sources is the brilliance (also called brightness, or spectral brightness) which defines the number of photons produced per second per unit surface of the source, per unit solid angle and per unit energy bandwidth. Figure 1 presents the brilliance reached in 2004 as compared to that achieved in 1994. The 2.5 order of magnitude difference stems from upgrades carried out to the lattice, the Radio Frequency (RF) system and the Insertion Devices (ID), as well as from other engineering developments.

In 1994, the original zero dispersion, double bend achromat lattice delivered a horizontal emittance close to 7 nm with less than 10 % coupling to the vertical plane. Using different quadrupole and sextupole settings, a modified lattice with a 4 nm emittance was implemented and operated in User Service Mode (USM) in 1995. The shrinking of the emittance was obtained by introducing small dispersion in the straight sections. In 1996, the lattice was further modified to reduce the vertical beta function to about 2.5 m in all ID straight sections. This move did not immediately increase the brightness but had far ranging consequences. Besides increasing the effective vertical aperture of the lattice and therefore increasing the lifetime and making the injection somewhat easier, most importantly it reduced the activation of the chamber and the generation of Bremsstrahlung radiation in the beamlines, opening the way up to very low gap insertion devices. Further work was undertaken to understand and minimise the vertical emittance. As a result a value of 20-30 pm has been routinely achieved in USM over the past few years whereas a value of less than 10 pm was achieved for a few days in a row using a feedback technique.

Between 1992 and November 1995, the beam current in USM was raised progressively from 100 to 200 mA. In August 1997, a third pair of RF cavities provided the margin and redundancy required for the long term operation at 200 mA . In those days, for reasons of beam stability, the 200 mA was only delivered in 1/3 filling mode with a lifetime of around 10-20 hours. It took some time, by means of achieving a precise temperature control of the RF cavities, and after hours of Machine Dedicated Time (MDT), to finalize a stable 2/3 filling mode, and later on a uniform filling mode. In 2004, these uniform and 2/3 filling modes correspond to 66% of USM with a lifetime of 80 hours being delivered in uniform. Raising the current to 300 mA is an active subject of development for the years to come. In addition, many other filling modes have been developed and implemented in USM. While most synchrotron light sources operate in user mode in a single or a few bunch filling mode pattern, it is a special feature of the ESRF to deliver many different kinds of filling modes in USM, optimised for various types of experiments. These include the single bunch mode (now replaced by a 4 bunch mode), the 16 bunch mode and the hybrid mode (with 24 trains of 8 bunches occupying 2/3 of the circumference with 1 mA per bunch and a single 5-6 mA current bunch in the middle of the remaining 1/3). In response to a request from the nuclear scattering beamlines in particular, a high contrast between the filled and unfilled buckets is served with routine values always in excess of 10^6 and the ability to reach a few 10^{10} (not a single electron in the other buckets).

At the same time a vigorous and on-going effort was set up to develop and upgrade the Insertion Devices (ID). In 1994, the minimum gap of an undulator was 20 mm. Although this figure seems very conservative these days, one must remember that there existed then little expertise world wide, with storage rings operated with a large number of small gap undulators. Thus a number of accelerator experts were concerned about the position and phase space stability of the electron beam under the combined effect of complicated undulator magnetic fields, their unpredictable errors and the expected large wake field generated by the small vertical apertures. These problems were addressed one by one and as soon as 1994 the first ID segment with a 16 mm gap was installed, followed by many others. Later 11 mm gap undulators were built followed in 1999 by the first in-vacuum undulator operating at a minimum gap of 5 mm. The magnetic gap reduction implied a shrinking of the undulator period and a shift of the undulator spectrum to a higher photon energy. At this point it is worth mentioning a debate that took place in the early 1990s on the optimum energy required to cover the 2-20 keV photon energy range with undulator radiation. At that time it was thought that 6 GeV was a rather low energy, so APS and Spring8 were consequently built with a 7 and 8 GeV energy respectively. The improvement in ID technology, in which the ESRF has been a major actor, has greatly reversed this way of thinking and one now sees the new national 3 GeV light sources aggressively designed to compete with the ESRF in this photon range. However, the effective jump in performance from longer and smaller gap IDs took more time than expected for two main reasons. Firstly, higher brilliance at higher photon energies implies a higher white beam heatload. The front-end group had been subject to a heavy workload of implementing and commissioning all thirty ESRF public beamlines with a design that was validated but unable to withstand the power delivered by a 5 m long, 11 mm gap undulator operated with a ring current of 200 mA. In 2000, a new front end was tested that could withstand ID power in excess of 15 kW and a power density higher than 400 kW/mr2 which corresponds to a 2 kW/mm2 of power at normal incidence on the first absorber of a beamline. Simultaneously the beamline monochromators were upgraded using diamond crystals and/or cryogenic cooling. These upgrades are presently being generalised to all beamlines (ESRF Newsletter nr.39). Of particular importance is the fact that the new beamline front-end design is equipped with an 0.3 mm thick, CVD diamond window replacing the original beryllium/graphite assembly. The diamond can take a higher heatload and stay vacuum tight. In addition, contrary to the graphite or the unpolished beryllium, it does not generate small angle scattering and preserves the ultra small emittance of the photon beam. The second reason was that, as the ID vacuum chambers being developed needed to be compatible with a large quantity of existing undulator segments, the ID chamber could not be equipped with an antechamber, and the pumping performed from the extremity induced a large pressure bump in the middle of the ID vessel, resulting in excessive Bremsstrahlung downstream in the beamline. Two directions were explored to remedy this effect. First, the in-vacuum undulator technology pioneered and developed on a large scale at Spring8 was also developed at ESRF. The second direction consisted in collaborating with CERN to qualify, and later obtain the licence for, the process of Non Evaporable Getter (NEG) coating of the ID vacuum chambers. The NEG film of a fraction of a micron is deposited on the wall of the chamber by magnetron sputtering. This film made of Titanium, Zirconium and Vanadium pumps the residual gas and reduces the secondary emission. It results in a much shorter conditioning time of the chambers and a lower ultimate pressure irrespective of the material constituting the chamber (aluminium, stainless steel…). In 2004, 16 of the 28 straight sections equipped with IDs have received a NEG coated vessel and a NEG coating facility is operating in house.

In 2004, there are 68 ID segments installed in the ring, but as the result of the upgrade sequence around a 100 magnetic assemblies have been designed, built and commissioned by the ID group. Originally the undulators had phase errors around or larger than 5 degrees resulting in a reduced brilliance on the high harmonics. In 1994, the ID group invented the phase shimming process which since then has been implemented on all undulators at the ESRF and in many places around the world with a typical maximum phase error of 1-2 degrees rms resulting in nearly ideal performances on all harmonics lower than 21. Many different types of exotic IDs have been produced such as several Helios type and Apple II type variable polarization undulators as well as a fast switching helical undulator, several high field asymmetric wigglers and quasi-periodic undulators. The latest development has been the commissioning of the 8th in-vacuum undulator segment and the operation of a revolver type undulator prototype.

Thanks to the long lifetime (~80 hours in uniform filling mode), injection only needs to be made twice a day and has been carried out since 1997 in topping up mode. Nevertheless until 2003, for safety reasons, all beamline shutters needed to be closed during injection. This resulted in a large variation of heatload in the beamline imposing delays which, depending on the beamline, could be as long as one hour before data acquisition could resume following a re-injection. Subsequent to a detailed study by the radio protection group, this limitation has been overcome and the refilling of the ring is now made without closing the front-end, with most beamlines continuing data acquisition during injection.

From the very beginning of User Service Mode (USM) operation in September 1992, the beam delivered to the beamlines has been quite stable as defined by the existing standards of second-generation light sources. Nevertheless, as more advanced experiments were performed on the beamlines, some beam motion smaller than the original target of 10 % (compared to the beam size) was reported to be harmful. A detailed investigation showed that most of the motion originated from the mechanical Eigen mode of the girders supporting the quadrupole magnets. Such resonating modes are excited by ground motion. To eliminate the induced beam motion, special damping links made from a visco-elastic material were developed, tested and fixed on all 96 girders. The beam motion has since then been reduced by more than 100 at the 7 Hz frequency, where the motion is at its maximum. Further stabilisation has been achieved by implementing a global vertical position feedback, as well as several local horizontal feedbacks. In particular, the ID24 beam (the most demanding beamline in terms of stability) is routinely stabilized to 0.3% of the size in the horizontal plane (within a 1 sec integration time). Further improvements of the global feedback are under way.

Simultaneous to this continuous upgrade and improvement in performance, a considerable effort has been put into increasing the reliability of the facility. A systematic analysis of all failures has been carried out to set up the necessary preventive maintenance. In 1994 and 1995, a large afternoon storm could leave the machine without beam for a duration that sometimes exceeded 12 hours. In 1996, the High Quality Power Supply (HQPS) was commissioned. It consists of 10 accumulator rotating wheels coupled to a generator and a diesel engine, each generating 0.8 MW of electrical power. The HQPS is capable of smoothing the voltage and phase drops of the main during storms, thereby keeping the beam stable in the ring. As a result of the extensive failure analysis and preventive maintenance, the beam availability has been kept at a level higher than 96 % in the last four years.

I have only briefly covered the visible part of the iceberg. Looking in more detail, one could equally see a dramatic progress in many other areas such as beam diagnostics, control, alignment, vacuum, operation … This major upgrade in performance since 1994 has been accomplished thanks to the hard work and dedication of many engineers and technicians from the Machine, Technical Services and Computing divisions.

PASCAL ELLEAUME

From left to right, up: Gavin Vaughan, Jonathan Wright, Åke Kvick, Magnus Bostrom, Larry Margulies, Becky Plappert, Jean-Michel Reynal. From left to right, down: Silvia Capelli, Mona Moret and Andy Götz. Missing in the picture: Nicoleta Lupu, Michel Rossat, Thomas Buslaps, Roland Taffut.

Visiting a beamline

ID11 FACES MATURITY MOVING TO NANOSCIENCE

ID11 is one of the oldest beamlines at the ESRF. Throughout a decade of existence it has constantly been updated in order to best fulfil its users' expectations. Today, the ID11 team looks to the future with an important extension of the beamline: moving towards nanoscience.

The beamline is mainly dedicated to diffraction experiments at higher energies in materials science. Gavin Vaughan, the scientist in charge, defines it as a 'flexible beamline', where they do research for metallurgy, polymers, chemical engineering, chemistry and fundamental research.

Despite being one of the oldest beamlines at the ESRF, it now faces a challenging project for the future. Starting next year, the beamline is going to be extended by 50 metres. The aim is to decrease the focal size, that is, to move to the nanoscience world. The optics for this extended beamline are already being developed. During the two years of construction, experiments will still be carried out on ID11.

These major changes are only possible because there is a great team behind them, not only on the beamline, but also in the support groups. They have lunch together every day and once a year at least they go skiing. They also have a tradition of bringing back the worst alcohol every time they go on a mission.

Fruit of the good atmosphere on ID11 is the book that has just seen the light. It is about the techniques developed by the ESRF and the Risø National Laboratory (Denmark), which has a scientist at the ESRF 75% of its time. The book is called 'Three dimensional X-ray diffraction microscopy', and has been written by Henning F. Poulsen. This collaboration is "very successful", according to Åke Kvick, head of the Materials Science group.

ID11 is a big team of 14 people. Three new scientists have joined the team sometime in the last month: two post-docs working for different European Projects and a trainee.

There is an English expression that links happiness with pigs. According to the team, users are happy with them. That is the reason why the mascot of ID11 is a pig (see the picture).

The pig of ID11.

The energy of the team will surely help them to deal with all the work foreseen for the next three years. After that time, ID11 will become an even better beamline for, at least, another ten years.

M. C.

Key moments in the history of the ESRF

30 YEARS OF ESRF OR HOW A DREAM TURNED INTO REALITY

The inauguration of the ESRF on 30 September 1994 marked the end of the ESRF's construction period, which itself had started on 1 January 1988. However, the conception of the ESRF began much earlier, almost twenty years before the inauguration.

Conception and construction

First discussions on a European synchrotron radiation facility started in the 70's. A series of studies, more and more detailed and assembled in books of different colours, marked the various stages of what would become the ESRF. The first of them (black, blue, yellow) were compiled by working groups under the auspices of the European Science Foundation, followed by the green European Synchrotron Radiation Project Report (1984). The Foundation Phase team in Grenoble finally produced the Red Book (1987), which was adopted as the reference document for construction. Pending an agreement on the legal texts and on contribution rates, construction was launched on the basis of a three-page protocol, signed on 22 December 1987. Only one year later, on 16 December 1988, final versions of the intergovernmental Convention and the Statutes were signed in Paris by the research ministers of the eleven countries that constituted the ESRF so far. The ESRF company was established as a French société civile on 12 January 1989.

A year later civil engineering work started on the site whilst 60% of the equipment for the accelerator had already been ordered. The installation of the booster synchrotron began in November 1990. The pre-injector (linear accelerator) was delivered at the beginning of 1991; it accelerated the first electrons in May. In August/September the commissioning of the booster synchrotron started with a first beam around the machine on the first day and acceleration to 3 GeV a couple of days later. On 12 November 1991 the ESRF injector reached its operational energy of 6 GeV for the first time.

At the end of 1991 all the technical buildings were completed. The initial construction programme referred to a facility with 30 beamlines, essentially on insertion devices as radiation sources. Given that the storage ring provided the potential for further beamlines to be built on bending magnets, the ESRF Council agreed that groups from research institutes based in the Contracting Party countries (Collaborating Research Groups) could install their own beamlines and use this bending magnet radiation.

Electrons were injected into the storage ring for the first time on 17 February 1992. Eleven days later the machine team succeeded in getting electrons to circulate. In June the target value for the current of the stored beam (100 mA) was achieved. The first undulator trials in July confirmed the high beam stability and record brightness. By November 1992 all the target values for the storage ring (*e.g.* lifetime, brilliance, stability) were achieved or exceeded. At the first three user beamlines, installed during the autumn of 1992, components were tested with X-ray beam.

Improving the performance

The storage ring was complemented by further insertion devices and front-ends; and the quality of operation of the machine and its performance continuously increased over the year. A High Quality Power Supply system was installed in order to prevent beam losses due to voltage drops on the mains (frequent during thunderstorms). By the end of 1993 six ESRF beamlines (and four CRG beamlines) were commissioned with beam, whilst a further five had already been assembled. Innovative devices for X-ray optics were successfully tested. Six review committees were established in accordance with the beam time allocation policy adopted by the Council. The Director General and the Unions signed the Collective Agreement and regulations for shift work.

While in 1994 the machine team was pushing the performances of the source beyond the initial target specifications and ensuring a high standard of reliability during user service operation, commissioning of the first set of beamlines entered its final phase. A satellite building for the first long beamline (Topography) was completed and construction work for a guest house started.

Test experiments confirmed the extraordinary progress made possible by the ESRF in a large number of research areas, even in fields that had not been put forward when the scientific case for the ESRF was set out (for instance, coherent radiation with sufficient flux, very high pressure on light materials, or inelastic scattering with very high resolution). Regular allocation of beam time took place for the first time for the period September-December 1994. By June 1994 the scientific programme for the first 26 (of 30) ESRF beamlines had been determined, seven CRG beamlines (plus a special high-energy physics experiment) had been approved and applications for another two were under investigation. In September 1994, the ESRF inauguration ceremony took place in the presence of research ministers and representatives from the twelve countries of the contracting parties.

Further development during ten years of User Operation

Parallel to the construction of further beamlines, regular user operation took place on those beamlines already completed. In 1995, there were a total of 1149 user visits for 339 experimental sessions with altogether 3703 shifts. The corresponding numbers for 2003 are 5140 user visits for 1282 experimental sessions with altogether 14273 shifts. In the meantime the number of operating beamlines has increased to 30 (ESRF) + 10 (CRG) and the quality of the beam has exceeded initial specifications by several orders of magnitude. ●

KARL WITTE

Getting the countries together

In October 1984 France and Germany proposed the implementation of the European synchrotron in France and agreed to assume a major share of the financing. They invited other countries to join. Within France, the decision fell in favour of Grenoble, to the disappointment of those rallying for Strasbourg, which for a long period had been the only French site proposal. On 10 December 1985 the signing of a Memorandum of Understanding (MoU) brought five countries (France, Germany, Italy, the United Kingdom and Spain) together in the synchrotron adventure. For the start of construction Switzerland and the four Nordic countries joined the group. The legal texts were also signed by Belgium (December 1988), joined in 1991 by The Netherlands as partner of the BENESYNC consortium. Presently the ESRF has still got the same twelve Contracting Party countries, while (so far) six further countries (Portugal, Israel, Austria, Czech Republic, Hungary and Poland) have been associated through bilateral agreements.

Interview

CHRISTIAN RIEKEL

Creating science

Christian Riekel holds the honour of being the first scientist recruited at the ESRF. When he arrived here from Germany the synchrotron facility was still just a project on paper. Today, eighteen years later, he admits to be rooted to the ESRF, but prefers to look ahead rather than evoking the past: "If you don't keep yourself up to date, you'd better stop being a scientist", he says. At present, he is the Leader of the Soft Condensed Matter group, as well as the head and 'father-figure' of the Microfocus Beamline. During a distinguished career spanning the last three decades, he has published more than 180 scientific articles, covering a range of subject areas.

You've lived in Grenoble for almost two decades. How much do you enjoy life here?

I have adapted well to the French way of life (my wife is French) and Grenoble is the perfect town to practise my favourite sports such as cycling and skiing. Indeed, after all these years I could open a museum of skis with all the skis I have at home.

The reason you came to Grenoble was science. What does science mean to you?

Science is certainly more than just a job. A scientist doesn't mind working long hours, because he or she gets compensated by gaining a better understanding of things. Scientists have to explore new areas. Some research will work, some won't, nevertheless the importance is to maintain scientific curiosity. Fortunately the ESRF is an ideal environment to 'wake up' one's scientific curiosity.

How do you keep that scientific curiosity alive?

It is important to always further one's chosen field. For this you have to combine the best science - one of the biggest assets of the ESRF is the in-house research programme - with the best instrumentation, which is constantly being developed. You can only get the best result if you work with a great team, like in my case.

How do you like your new task as Group Leader?

It gives me better opportunity to shape the future of the Soft Condensed Matter arena and I also receive new information before other people do, which is a bonus. I try to favour information exchange among the scientists themselves.

The Microfocus beamline is one of the first of the ESRF. How has it changed since the early days?

The Red Book (*i.e.* the Foundation Phase Report) from the ESRF didn't predict that the Microfocus beamline could reach beam sizes that are now routinely employed. At the present time the beamline works with routine techniques but it still remains very specialised. The improvements in the beamline have always been favoured by the fact that the Directors of the ESRF have given me freedom to develop new approaches. At the end of the day, scientists must to be creative and have new ideas to further their field.

How do you see the future of science?

It is very important to explore new areas and work with researchers coming from different disciplines. Flexibility is the key word and scientists have to learn to work with each other. That is why we have to seize opportunities for collaboration. Long-term projects and partnerships will become more and more important. ●

M. C.

Spider silk being studied on ID13 as the spider produces it.

SPIDERWORLD

He is the spiderman of the ESRF. Spider silk has been one of his research subjects in the last few years. The aim of this study is to model this material, which combines the properties of high flexibility with strength. "It is the old quest of understanding nature", explains Christian Riekel. His experiments on spider silk are famous at the synchrotron, especially because of what could be called 'the set-up', which consisted of a living spider producing silk *in situ*. The beamline has become well-known thanks to the results of these experiments. On ID13 there is a plastic spider on the door of the control room which welcomes the users. Another spider, this made in Africa with tin and wires, keeps a pile of business cards safe on his desk. Spiders are definitely welcomed on ID13.

The spider on ID13, the Microfocus beamline.

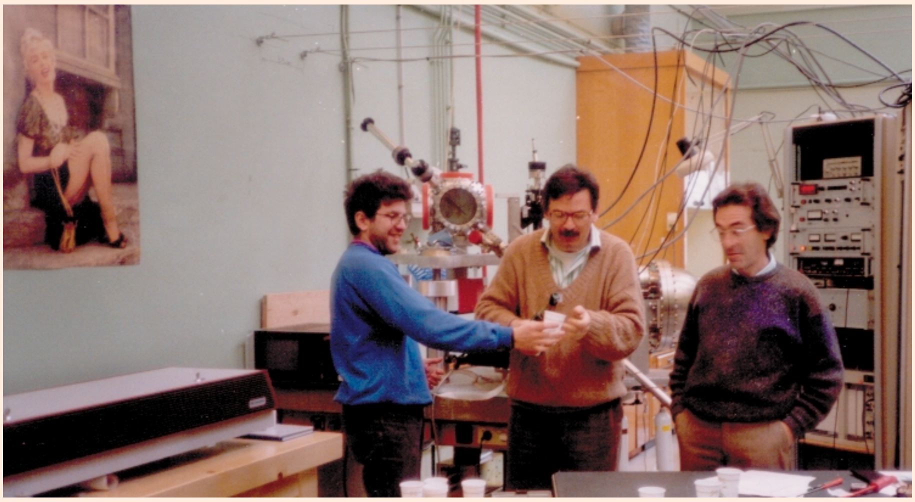
The way we were

The vibrating floor of the Experimental Hall

In 1992, when the ESRF was just starting its runs, we discovered that the floor in the Experimental Hall was unstable. To correct the problem, holes were drilled one metre apart and concrete was injected into the sub-soil under the slabs.

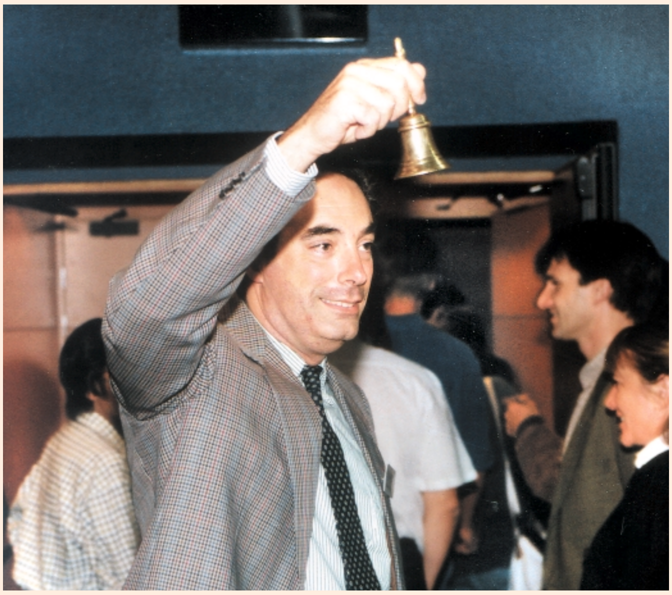
Jean-Louis Laclare,

Project Leader then Machine Director from 1986 to 1996 (left), in discussion during the 1993 Users' Meeting.



The first ESRF lab,

with the first UHV system, the first laser and the first ion pump. These weren't the first drinks, though!!! From left to right: Marylin Monroe over a YAG laser, a young Spanish student, now a regular user, the Laser Deposition system for diamond films, Fabio Comin and Salvador Ferrer.



The workshop bell

The bell is a very convenient instrument to announce the end of a coffee break in a conference or workshops. This is the former Director of Research, Massimo Altarelli, trying to get people back to work.

From left to right: Ian Kilvington, Federico Boscherini and Settimio Mobilio talking at the 1993 **Users' Meeting**.

On **the Inauguration day**, 30 September 1994, the Experimental Hall was turned into a huge auditorium. This event marked the completion of the construction of the radiation source and the first set of beamlines and also the opening of the ESRF's doors to scientific users.

A bunch of young scientists on one of the first beamlines, with a background full of cables.

Louis Néel, founder of the Polygone Scientifique, where the ESRF is located, visiting the installations in June 1990. In the picture he is holding a walking stick and is surrounded by the former directors of the ESRF, representatives of the CNRS and of the city of Grenoble.

Empty Experimental Hall In the early days, the Experimental Hall seemed like an enormous cycle track.

Carl-Ivar Brändén (left), Director of Research from 1992 to 1997, in discussion with **Yves Petroff** (right), Director of the ESRF from 1993 until 2000.

The Users' Organisation awarded Francesco Sette, now Director of Research, with the Young Scientist Award in 1995. In the picture you can see Francesco Sette (right) and Hans Peter Weber (left), from the Users' Organisation and now scientist in charge of the Swiss-Norwegian beamline.

The ESRF has organised five **Open days** throughout its history. This photo was taken in the last Open Day, in 2003.

José Baruchel and coworkers were given, very soon after the first available beam, a night shift on ID11 to carry out a 'white beam topography' experiment. This team proceeded the same way they used when working in other facilities: first to place a green paper in front of the sample and then, after letting the beam in, know where the beam was. They forgot to set the filters and clearly underestimated the ESRF beam power: not only the paper was completely burnt, but a hole produced in the studied crystal (melting point 120°C) along the beam path. Luckily, it wasn't a unique crystal! In the photo you see José holding **the perforated crystal**, and in the second you can see the team. From left to right: Petra Pernot, Jürgen Härtwig, José Baruchel and Federico Zontone.

ULRICH BONSE

« The ESRF matched our expectations and even exceeded them »

Professor Ulrich Bonse has witnessed the first crucial moments of the life and gestation of the ESRF. He participated in the conception of the facility and was a member of the Council for five years. He is also the first user who carried out experiments at the ESRF.

When you participated in the European Science Foundation (ESF) *ad hoc* committee during the seventies, what were your expectations of the future European Light Source and how were they matched?

We expected to design and finally lead to completion the best dedicated synchrotron radiation (SR) source we could imagine. The key properties of a 'best' source were: wide energy range, precise and easy energy selection, high flux respective brightness, stability, convenient accessibility, all combined with awareness to scientific and technological innovation of any kind one could think of. The initiative was with the ESF sub-committee on synchrotron radiation. It had to be European because it was clear from the beginning that sufficient resources (financial, personal) could not be raised by a single country.

I guess at the beginning most of us were realistic enough in judging the latter requirement a chance combined with an impediment. Consequently, the choice of the building site was carefully circumvented in the scientific and technical studies that followed (and left to the 'politicians' to be decided later!) This procedure helped to perform very thorough studies, which confirmed that such a source could become a really exceptional tool for science, technical development, and innovation in general. Our expectations were mostly matched and in some respects definitely exceeded by the facility that emerged in the early nineties.

What was your position concerning the choice of the site for the ESRF?

My position was that the site should be excellent from all points of view, *i.e.* technical requirements like tectonic properties, power requirements, and from other aspects like human resources, scientific / technical environment, accessibility for serving all Europeans as evenly as possible etc. Obviously no site could score best in all categories. The idea therefore was to seriously compare upcoming site applications (*e.g.*, Risø , Dortmund, Triest, Strasbourg, Grenoble....) and select the best site on the grounds of objective criteria. The Dortmund site proposal (1982) followed a suggestion of the German synchrotron radiation user community at a meeting in Hamburg.

However, at this point big politics entered the scene. Initially the German (federal) government - relying on existing reviews lacking timely update - considered large scale synchrotron radiation not even worthwhile running for. Other and for scientists unforeseeable and partly amazing factors (like political elections in France) suddenly played the key role. The outcome is well known.

How did you succeed in becoming the first user at the ESRF in 1994? What was your experiment about and how was the experience?

Actually it was already in December 1992. The machine group had set up a diagnostic beamline at ID06 for orbit and insertion device studies. They reported to the Council about it adding that beam time (however very limited) could already be offered to users capable and willing to work at such a rudimentary line. This was no problem for me and my group working for years at DESY and later at DORIS with mostly our own equipment developed 'at home'. F. Busch and F. Beckmann packed our apparatus, and instead of going up to Hamburg drove down to Grenoble. Supported by M. Krisch, we had our machine (consisting of a double crystal monochromator with cooled first crystal in front of a tomographic scanner) installed at the diagnostic line after about five days. Within another week or so we were able to make some of the first micro-tomographic studies on microcallus in human bone biopsies obtained from Dr. G. Delling of the University Hospital Eppendorf in Hamburg.

All this was possible only with the kind understanding of the acting director general Ruprecht Haensel and the excellent help and enthusiasm from Pascal Elleaume and his colleagues. Worth noting is that while the commissioning of the ESRF storage ring was several months if not a year ahead in schedule, the opposite had to be said of the beamlines and experimental stations. Therefore the Council was extremely keen to see 'synchrotron radiation experiments' begin.

What memories do you keep of the first years of the ESRF (when you were member of the ESRF Council and a user)?

For me the work on the Council was hard and frequently frustrating. This had to do with the fact that the Council – at least in its early years – was a highly political body. I was usually one of at most two or three scientific members on the Council. As such I was at the beginning surprised of how many words could be made on – seemingly - 'nothing'. The discussions dealing with nothing were, as I learnt by and by, a misunderstanding of my own. To my surprise, hard disagreeing discussions dissolved frequently into brief and total agreement the next day, *i.e.* when in the evening the bargains between the main actors had been arranged behind the scene, thus 'saving' the public further trouble (and decisions!).

An important matter was the 'international' school for the children of employees at the ESRF stemming from other member countries. Compared to CERN, where such a school was completely normal, the French authorities saw always insurmountable formal difficulties. Whenever the subject was raised again it proved impossible to find out exactly whether at the very beginning the French side had promised to establish such an institution or not. (Unfortunately, 'nothing-discussions' on this controversy never mutated to total agreement the next day). I do hope this is no longer an issue.

As a remarkable success, I recall when - with the help of two colleagues - I could persuade the Council to establish the rule that scientists from abroad with approved experiments receive financial support from the ESRF for travel and accommodation if they need it. Today this seems to be ordinary matter but it was not so at the beginning.

As a user from Germany I had the opportunity to perform a number of quite successful experiments with my group. The synchrotron radiation source was excellent and kept us usually very busy when we were there. Likewise we enjoyed on the whole competent, efficient and agreeable support by the respective staff members. Though it is worth mentioning that we never were the 'standard user': we always had to build or provide essential experimental equipment of our own and carry it with us (usually for micro-tomography and interferometry).

How much do you think the ESRF has changed over its ten years of regular operation?

I followed the ESRF performance in recent years, although not very thoroughly. I had contact to colleagues at the ESRF when I worked at the ILL in 2003-2004. The synchrotron radiation source performance is really outstanding and excellent. My impression is that the general operation of the ESRF has become routine by now, of course.

How do you see the future of the ESRF at the present time, while several national synchrotron facilities of the third generation have been or are being constructed?

There has always been a competitive relation between the ESRF and national synchrotron radiation sources. I believe that competition will definitely increase with more 3rd generation national sources in operation. This is likely to result in less users coming to Grenoble unless some additional and well-grounded motivation(s) to use the ESRF comes (come) up.

In your career, you've combined the possibilities of synchrotron radiation and neutrons facilities. How complementary do you think these tools are?

X-rays and neutrons are complementary in a number of ways. However, the importance of this is frequently overestimated. An example: It does not follow that synchrotron radiation and neutron facilities automatically gain very much by sharing the same site. My personal experience: Having worked in both fields, experiments rarely would have profited by the fact, that – in principle – the other sort of work could have been done at the same location.

You have a broad scientific career behind you. Even at the present time, being retired, you still produce publications. What does carrying out research work mean for you?

For me carrying out research work has two major stimulants: one could be described as curiosity to find out the structure of things combined with the urge to improve them or to improve the use of them. The other stimulant is to show and tell what I find, or, with other words, to share this with students / collaborators, other humans in general.

What, in your view, should be done to fight against the disaffection of the young for science and scientific careers?

All I can say briefly is to encourage to continue with patience and intelligence to explain and illustrate the benefits and necessities of science and its careers to the young and society in general in as many respects as possible. However, be always competent and honest and keep strictly to the truth while doing so. – Do not give up!

M. C.

Fig.1: SEM image of Al-Cu-Fe icosahedral quasicrystalline grains.

Scientific article

USING SYNCHROTRON RADIATION X-RAY IMAGING TO INVESTIGATE POROSITY IN QUASICRYSTALS

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Introduction

Quasicrystals (QCs) were discovered in 1984 by Dany Shechtman and his collaborators Ilan Blech, John Cahn and Denis Gratias *[1]*. They exhibit orientational symmetries forbidden for crystals (fivefold for instance, as indicated by the pentagonal facets of the quasicrystalline grains shown on figure 1) and a long range quasiperiodic translational order. Contrary to what was thought at the very beginning, the quasicrystalline arrangement is a stable one.

The icosahedral QCs were shown to have properties that could appear surprising when taking into account their metallic components (Al-Pd-Mn, for instance). These unusual properties include weak electric and thermal conductivity, bad wetting and low friction coefficient, and high resistance to corrosion and oxidation. In addition, experience has proved that QCs grains can produce sharp diffraction patterns.

Porosity is one of the most frequently encountered defects in QCs. The sizes of the pores are very big when comparing to those observed in metals like Al, and 'locally' discrete sizes were reported. This raises several questions, which include the influence of these pores on the unusual QCs properties, and more fundamentally, their origin and relationship with the structure and growth process.

Characterisation of the porosity

In the last ten years a number of X-ray imaging studies have been performed at the ESRF to characterise the porosity in Al-Pd-Mn QCs. The origin of this porosity was investigated by observing its behaviour under thermal treatment and its formation in the course of a solidification process. This was possible because of the high degree of coherence of the ESRF beams that allowed phase images to be easily recorded.

Figure 2 shows radiographs of a Al-Pd-Mn QC grain, performed at three different sample-to-detector distances. The smaller distance (figure 2a, 13 mm) corresponds to the 'absorption' image, where large pores are faintly visible, the intermediate distance (figure 2b, 100 mm) to the 'edge enhancement' regime that allows in addition the observation of the small pores, and the largest distance (figure 2c, 500 mm), where the interference fringes are clearly visible, substantially increase the contrast. The schematic drawing shown on figure 2d indicates that what is observed is an icosahedral

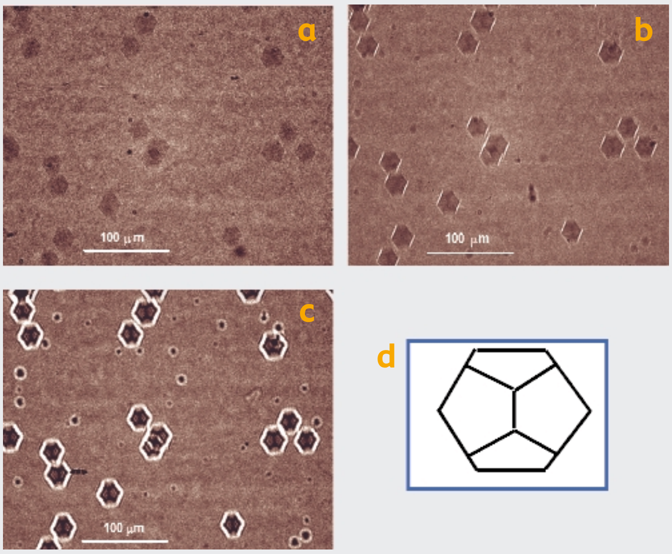


Fig.2: Phase contrast images of pores in a quasicristalline grain of Al-Pd-Mn , as a function of the distance a) 13 mm b) 100 mm c) 500 mm. The X-ray beam is parallel to a twofold axis of the QC, and the icosahedral pores are therefore seen as sketched in d).

pore projected along the twofold axis. Figure 3 shows the same type of radiograph with the incoming beam along the fivefold axis: it shows an apparent tenfold symmetry of the pores, except for those lying on the surface, where only the fivefold symmetry is observed, and sizes of the pores that can be as big as 50 µm. From these images, and microtomographic ones, it can be deduced that the total porosity is in the 0.2% range.

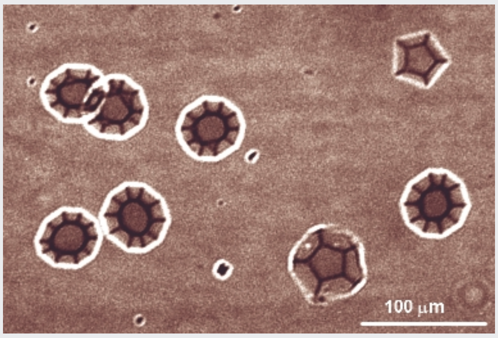


Fig.3: Phase contrast radiograph showing the pores projected along the fivefold axis.

Phase radiographs images (figure 4a), and monochromatic diffraction topographic images (figure 4b) of the same region of an as-grown $Al_{68.6}Pd_{24.7}Mn_{6.7}$ QC, were compared. This comparison indicates that the pores are associated with 'inclusions-like' strains, which show conspicuously on the topograph as black-white contrast (fig. 4b insert) *[2]*.

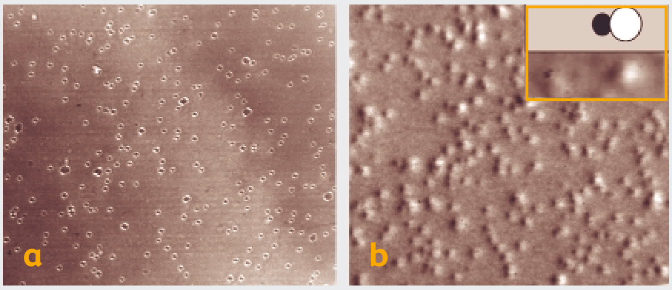


Fig.4: Comparison of a phase radiograph and a diffraction topograph showing that the pores are associated with an 'inclusion-like' strain field.

Origin of the porosity

The origin of such big pores remains an open question. Two main ideas were evoked to try to explain their occurrence: a growth tiling frustration *[3]*, or a condensation of vacancies *[4]*.

The images of the same region of an $Al_{68.6}Pd_{24.7}Mn_{6.7}$ grain before and after an in-vacuum annealing process show that the pore sizes and the strain field around the pores diminish. This evolution was shown to be compatible with a vacancy diffusion model *[5]*. On the other hand the real-time X-ray imaging observation of the Al-Pd-Mn quasicrystalline growth from the melt shows that the big (> 1 µm) pores do not form immediately but in the course of a further heating (figure 5). This fact is under further investigation, and is expected to give clues to explain the origin of the porosity.

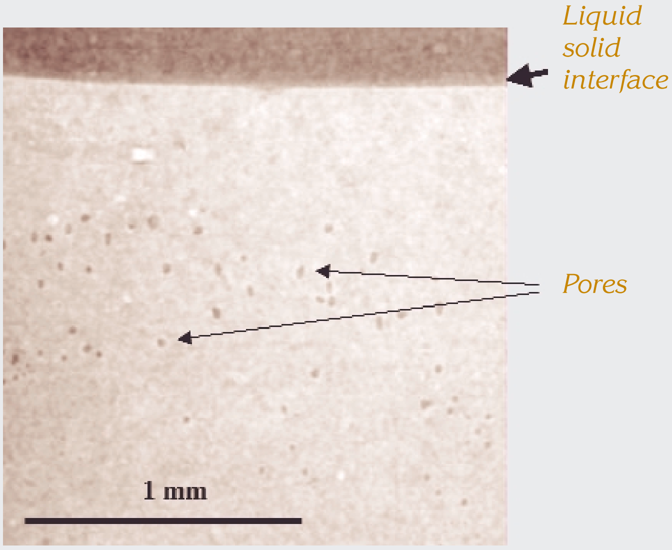


Fig.5: In situ observation of Al-Pd-Mn QC, which shows that pores grow during a temporary remelting in the course of a solidification experiment

*[1] D. Shechtman, I. Blech, D. Gratias and J.W. Cahn, Phys. Rev. Lett., **53**, 1951 (1984)*
*[2] L. Mancini, E. Reinier, P. Cloetens, J. Gastaldi, J. Härtwig, M. Schlenker, J. Baruchel, Phil. Mag. A. **78**, 1175 (1998)*
*[3] L. Mancini, C. Janot, L. Loreto, R. Farinato, J. Gastaldi And J. Baruchel Phil. Mag. Letters **78**, 159 (1998)*
*[4] C. Beeli, T Gödecke and R. Lück, Phil. Mag. Let. **78**, 339 (1998)*
*[5] S. Agliozzo, J. Gastaldi, H. Klein, J. Härtwig, J. Baruchel, E. Brunello, Phys. Rev. B **69**, 144204-1-9 (2004)*

Latest News

Structure of a component of chocolate unravelled

Think about a piece of chocolate. Imagine it melting in your mouth. The sensation is delicious. Now think of the same image, but this time the chocolate is covered by a white film on its surface. This white film is produced when chocolate is poorly crystallised or when it is stored under the wrong conditions. Here is where scientists come into the picture. Researchers from The Netherlands working at the ESRF try to avoid this white layer, called fat bloom, by studying the structure of chocolate. Their aim is to optimise the pleasure of eating it. They sound out the structure of a component of cocoa butter and also the crystal structure of the most common form of cocoa butter in chocolate, a result of great importance for chocolate production.

There is a lot of science in the process of making chocolate. Dark and bitter sweet chocolate contain from 31 to 38% of cocoa-butter, 16 to 32% of cocoa powder and 30 to 50% of sugar. Cocoa butter determines the physical properties of the chocolate. It has a high degree of crystallinity and may crystallise in six different crystalline forms in the course of the production process. This process includes tempering, which consists of repeatedly heating the chocolate to a specific temperature and then cooling it down. It aims to bring the cocoa butter in one of the most stable crystal forms. The different crystalline phases are numbered from phase I to the most stable phase VI. The lower-numbered phases are unstable and do not give a good product, but manufacturers nowadays manage to set the chocolate in phase V. Nevertheless, even this chocolate phase can suffer from phase transition during storage, resulting in fat bloom. This explains the importance of crystallising the chocolate properly.

A team of scientists from the University of Amsterdam, with help of the ESRF, has made a major step forward by identifying for the first time the crystal structure of one of the three main triglycerides that make up chocolate butter. The triglyceride, called SOS, is a cis-mono-unsaturated type and represents one quarter of the chocolate butter. This breakthrough helps in better understanding the melting behaviour of cocoa butter and better controlling the production process. According to Dr. René Peschar, first author of the paper, "This work is expected to be highly relevant to confectionery research and industry and the first step to a better understanding of the mechanism of the fat bloom phenomenon at the molecular level."

The researchers used the synchrotron light to collect data from which they determined this structure using the X-ray powder diffraction technique. They also stored completely molten cocoa butter at room temperature (around 22°C) for several weeks to get the phase V. Then they studied it at the ESRF with the same technique and managed to construct a crystal structure model of this cocoa butter phase V. "It is impossible to get these results with laboratory data; you really need a synchrotron facility because of its superior data quality", explains Dr. Peschar, from the University of Amsterdam.

The chocolate research based on data measured at the ESRF has also had impact on industry. The Dutch machine manufacturing company 'Machinefabriek P.M. Duyvis' acquired a patent concerning an improved method of making chocolate that is based on the results of experiments carried out by the Dutch researchers at the ESRF over the last few years. The company built a prototype, tested and fine-tuned it together with the University of Amsterdam and a major European chocolate producer. The company is situated in the middle of the Zaanstreek, a region hallmarked by a huge diversity of foodstuff manufacturers and processing more than 20% of the world's cocoa bean crop. ●

M. C.

Peschar et al. Crystal Structures of 1,3-Distearoyl-2-oleoylglycerol and Cocoa Butter in the ß(V) Phase Reveal the Driving Force Behind the Occurrence of Fat Bloom on Chocolate, J. Phys. Chem. B, Web Release Date : September 14, 2004.

Schenk and Peschar. Understanding the structure of chocolate, Rad. Phys. and Chem. 71 (2004) 829-835.

New insight into aluminium

Aluminium is a metal widely used in industry; therefore the more that is known about it, the more effectively it can be used. Researchers at Risø National Laboratory in Denmark and the ESRF have filmed in 3D the changes in the bulk of deformed aluminium after annealing for the first time. The results give a new insight into this metal and contradict classical assumptions.

Take a can. The aluminium that you see has been processed before having the shape of a cylinder. In a first stage, the aluminium is deformed. The energy is concentrated in its bulk. Then it goes through a process of annealing to get the shape of the can. In the annealing process, grains grow in the bulk. Up to now, there was a general assumption that the grains grow smoothly and in a regular shape. With the power of the X-rays at the ESRF, researchers have proved that the grains grow very irregularly. These changes of aluminium are of great importance for manufacturers in order to know how to process it to get certain properties, such as more strength. The experiment is a real breakthrough in the field, since the previous studies on metals were in 2D and focused on the surface. The team has achieved measurements that go into the bulk, which has a very different structure than the surface. They followed the grain as it grew after annealing the metal. "Individual grains don't behave like average and having a look at the local scale will help to create a better model", explains Lawrence Margulies, one of the authors of the paper.

The in situ measurements were done using the 3D X-ray diffraction microscope at the ESRF. The sample had a pre-annealing period of one hour at 260°C. Afterwards, it was put in a furnace that raised the temperature from 270° C to 290°C. Researchers took 73 snapshots of the grain during almost 30 hours and made a movie where one can clearly see the irregular growth of the grain in a micrometre spatial resolution. ●

M. C.

Schmidt et al., Watching the Growth of Bulk Grains During Recrystallization of Deformed Metals, Science 2004, 305: 229 – 232.

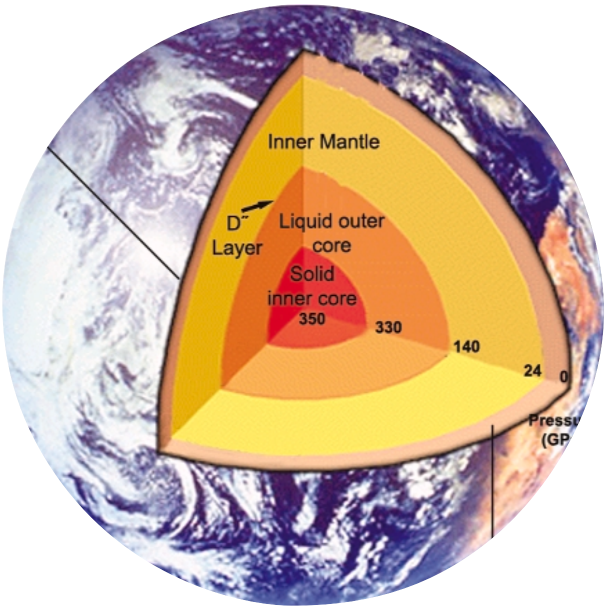
What goes on underneath your feet?

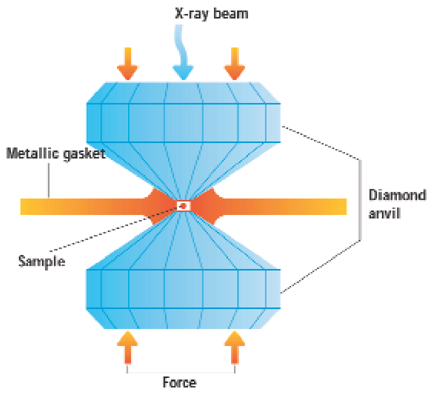
It is generally assumed that heat from Earth's core and mantle, due to the low thermal conductivity of the latter, is transferred to the outer part mainly by convection. This implies swirling movement of an immense amount of hot material, which is behind the dynamics of Earth's interior. Understanding the details of this is of great interest since it can explain natural phenomena such as earthquakes, volcanoes, movements of tectonic plates and formation of mountains. A team from the University of Paris and the ESRF have found out that iron-bearing magnesium silicate perovskite, the Earth's most abundant mineral, transforms, when pressure is applied, to a state where radiation could play a far more important role in heat transfer in the lowermost part of the mantle. This would change our vision of the dynamics of the deep Earth and would suggest that the material at these depths is more static than currently thought.

Earth's lower mantle is formed mainly by two components: magnesium silicate perovskite and magnesiowüstite. The first one, the subject material of this research, occupies 80% of the mantle. Therefore, it is indispensable to explore how this behaves at high pressure. Iron in perovskite is in a magnetic (high-spin) state at atmospheric pressure, the electronic properties of which are mainly responsible for this mineral being opaque to infrared radiation (heat). The team performed the experiment at various pressures and found electronic transitions which show that iron becomes non-magnetic (low-spin) at significantly lower pressure than previously thought. This pressure (or depth) is consistent with that of the D˝ layer, the deepest part of the lower mantle which is also the most mysterious and uncharacterised layer in the Earth, which separates Earth's liquid metallic core below from the solid silicate mantle above.

The most striking consequence of the revealed electronic transition is an increased transparency of the material to the near-infrared radiation (where the core and mantle radiate most of their thermal energy). The sample became more transparent to heat above 70 GPa (bottom third of the mantle) and almost completely transparent above 120 GPa (D˝ layer above the core-mantle boundary); this is more than one million times greater than atmospheric pressure. Increased transparency is the reason why these researchers suggest that in the deep Earth, radiation plays a larger role with respect to convection in transferring heat.

During the experiment, the researchers took a virtual trip inside the Earth by reproducing the conditions of Earth's mantle at the ESRF. They placed the sample of iron-bearing perovskite between the two diamond tips of a diamond-anvil cell and subjected them to pressures from 20 to 145 GPa (*see image above*). By using x-rays, they could extract information from the sample and its behaviour under those conditions. ●





M. C.

Badro et al. Electronic Transitions in Perovskite: Possible Nonconvecting Layers in the Lower Mantle, Science, 2004; 305: 383-386.

Gallery of events

Exchanging views with scientists from outside...

One tends to think that in the summertime there is not much activity. This is not strictly true for scientists, since many workshops and conferences take place in this season. At the ESRF, in June, a workshop on Ultrafast Structural Dynamics with pulsed X-rays reviewed progress done during the last five years in picosecond pump and probe experiments in physical, chemical and biological systems made at the ESRF and at other X-ray sources.

In July, the Polymorphism in Liquid and Amorphous Matter (POLIMAT) joint ESRF-CECAM international workshop focussed on new trends in the simulation and experimental studies of liquid and amorphous matter.

When the summer was reaching its last days, research on surfaces and interfaces, which is now carried out on ID03 and ID32, was discussed, with the aim of providing unique, world-class facilities for the years to come. This event coincided with the tenth anniversary of the very first users of the ESRF, who took data at the surface diffraction beamline ID03.

...and from inside

The internal life of he ESRF has also been very active these last months. The beauty of ESRF science shone during the Science Days, which took place in Aussois, an alpine spot near Italy. According to Sine Larsen, Director of Research, "this was an occasion to interact socially about science". Indeed, during the three days of the event, there was time for scientific presentations, and also for informal discussions during the coffee breaks, meals or with a beer in the bar.

In September, the Experiments Division Students day took place following the success of the first edition last year. This day was an opportunity for PhD students, primarily in their 2nd or 3rd years, from CRG and ESRF beamlines, to present their work to their peers and to other members of staff. The highlight of the workshop was the overall quality and maturity of the presentations. Following the presentations, a 'poster clips' session gave all of the 28 poster-makers an opportunity to show their work.

M. C.

CREATING SPECTROMETERS

A simple experiment was used to teach children the meaning of the unfamiliar word ‘spectrometer’. Matchboxes containing slits and some grating paper were used to demonstrate that light is made of many colours. The location of this experiment was this year’s Fête de la Science in Grenoble. This event is an annual French science festival, which is held in October and is designed to bring together scientists and the general public.

In addition to the posters and scale model of the ESRF, visitors could discover the experiments carried out on beamline BM30B by a team from the Laboratoire de Géochimie de l’Environnement of the University Joseph Fourier who work with plants that accumulate heavy metals.

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