ESRF NEWSLETTER

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SYNCHROTRON RADIATION 50 years

INTERNATIONAL CONFERENCE

Highlights in X-Ray Synchrotron Radiation Research

17-21 November 1997
Grenoble, France (see page 3)

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Photography by:
C. Argoud, S. Bertini, S. Berard and T. Le Bihan.

The Conference on «Highlights in X-Ray Synchrotron Radiation Research»

In 1947, the first report of direct visual observation of synchrotron radiation from a 70 MeV machine in the General Electric Research Laboratories appeared in the Physical Review. To celebrate the fiftieth anniversary of this momentuous event, and to provide a timely review of the status of the field after the start of operation of third generation sources around the world, ESRF organized an international conference on «Highlights in X-Ray Synchrotron Radiation Research». The conference was held in Grenoble from 17 to 20 November 1997, and was accompanied by a symposium on Structural Biology and followed by the ESRF Users' Meeting and by the general meeting of the European Synchrotron Radiation Society. Specialized workshops were also organized immediately before and after the conference. More than 500 attendees convened and provided a responsive audience who animated discussion sessions throughout the meeting.

The format chosen for the conference was to have a series of plenary invited talks, at least 35 minutes long, most of them grouped so as to provide a broad overview of a highlighted field of science. Lively poster sessions, complementing the plenary talks, concluded three of the four conference days.

On the first day, after a short welcome from Y. Petroff, Chairman of the Organizing Committee, B. Batterman of Cornell University took up the challenge of taking the audience on a trip through fifty years of synchrotron radiation in just about 40 minutes. This was no easy task, and the lively discussion at the end of his presentation included questions challenging his omission of one or another contribution to the history of the field. In the following presentation J. M. Filhol, ESRF Machine Director, reviewed the performance of 3rd generation sources and gave an outlook on what could be expected from the 4th generation sources, which are at present on the drawing board.

The x-ray investigation of magnetic systems has been undergoing a qualitative and quantitative explosion in recent years, thanks to the wavelength selection and polarization properties allowed by synchrotron radiation. The early part of the conference featured plenary talks devoted to an in-depth discussion of the status and the perspective of magnetic studies with synchrotron radiation.

After a review of the theoretical foundations of the different techniques by M. Blume of Brookhaven, talks by C.T. Chen (SRRC, Taiwan), P. Carra and J. Goedkoop (ESRF) discussed magnetic circular dichroism, and the experimental and theoretical breakthroughs which made this technique so popular and useful for the study of ferromagnetic systems. E. Kisker (Düsseldorf University) discussed methods based on photoelectron detection, in particular the imaging and element-sensitive capabilities. G. Sawatzky (Groningen University) discussed an area in which synchrotron radiation shows great promise of becoming a very important technique, but has not yet experimentally proven it: orbital order in transition metal oxides, where the orbital degeneracy of d-electrons can be removed by formation of a superlattice of orbital orientations, with dramatic effects on magnetic structure and excitations.

On Tuesday morning, three more talks devoted to magnetic studies, specifically by magnetic scattering, were presented. D. Gibbs (Brookhaven) reviewed work on many different systems, and also emphasized the emerging field of surface magnetic scattering. This technique was discussed in detail by S. Ferrer of ESRF who showed the recent results on the Co/Pt system obtained at ID3. M.J. Cooper (Warwick University) described recent Compton scattering studies of the spin density.

In the next group of talks, S.K. Sinha (APS, Argonne) and G. Gruebel (ESRF) discussed and exemplified the use of the partially coherent character of undulator x-ray radiation in the scattering studies of dynamics and fluctuations in condensed matter. This is certainly one of the most novel aspects of third generation sources, and, in spite of the many beautiful results presented, one which has yet to deliver its full promise of exciting new knowledge. In another field which has known a fast development of synchrotron radiation studies, soft-condensed matter, J. Daillant (Saclay) discussed grazing incidence studies of liquid surfaces and interfaces, and A.J. Ryan (University of Sheffield) and M. Stamm (MPI Mainz) reviewed recent applications to polymer science. The last two plenary talks of the day were devoted to inelastic scattering with ultra-high energy resolution. F. Sette (ESRF) described the spectacular results of ID16 on the dispersion of collective excitations of liquids and amorphous systems, measured with meV resolution in previously inaccessible regions of the energy-momentum plane. E. Burkel (University of Rostock) presented results on crystalline solids, including high-pressure investigations of phonons in He.

On Wednesday, W. Hendrickson of Columbia University described progress in the investigation of biomolecular structures using the MAD method, which is taking place thanks to dedicated beamlines and to the quality of undulator radiation. J. Trümper (MPI, Garching) delivered a fascinating presentation of x-ray astrophysics, with splendid pictures of celestial bodies from our small moon to immense galactic clusters, and showed how progress in x-ray instrumentation is widening and deepening our view of the Universe.

Coming down to earthly applications of synchrotron radiation, J. Schneider (Hasylab) described the use of very high-energy x-rays from Petra undulators to perform beautiful experiments on the critical region surrounding the structural transition of $SrTiO_3$, and to elucidate the «two length-scales» behavior induced by structural defects in the near surface regions of the sample. E. Gerdau (University of Hamburg) reviewed nuclear resonant scattering of synchrotron radiation, which is still delivering new and sometimes surprising applications of the extremely monochromatic beams it produces, which have therefore longitudinal coherence lengths of many meters.

The remaining talks of the day explored theoretical and experimental advances in high-pressure physics. On the theoretical front R. Martin (University of Illinois) described the present knowledge of the phase diagram of hydrogen, and the long-standing effort to pinpoint and, hopefully, observe its insulator-to-metal

USERS' MEETING

The annual Users' Meeting took place on 21 November 1997, following the four-day SR50 Conference. It was organized around one plenary session and three mini-workshops on X-Ray Imaging, Time-Resolved Scattering and Absorption, and also Industrial Applications of Synchrotron Radiation.

The «Young Scientist of the Year» award, which is given every year by the Users' Organization for outstanding work done at the ESRF by a scientist 35 years of age or younger, was offered this time to M. Thoms (from Erlangen University) for the development of a new image plate detector, an order of magnitude faster than other detectors of this kind.

The new Council of the Users' Organization has been elected for the next two years: P. Armand, F. Boscherini, J. Cockroft, M. Frey, A. Kaprolat, D. Nicholson, with M. Cooper as chairman.

M. Thoms receives his award from the hands of S. Pascarelli, Chairwoman of the Users' Organization.

The new Council of the Users' Organization.

transition, with its many implications for fundamental solid state physics as well as planet physics; and M. Parrinello (MPI Stuttgart) reported *ab initio* molecular dynamics simulations of water and ice, where the hydrogen bonds play a fundamental role. In particular, he discussed the phase transformations of ice at high pressure. On the experimental side, P. Loubeyre (University of Paris VI) described the large impact of 3rd generation synchrotron radiation sources on single crystal structural studies of light elements like hydrogen and helium at Mbar pressures; H.K. Mao (Carnegie Institute, Washington) reported how the simultaneous development of synchrotron sources and of high pressure instrumentation such as diamond anvil cells equipped with Be windows and improved control of pressure and temperature are allowing the investigation of materials under conditions approaching those in the deep core of the earth. The same message came through the talk of D. Andrault (Institut de Physique du Globe, Paris), describing experiments on Fe at 2500 K temperatures and Mbar pressures and their implications for geophysics.

The final day of the conference comprised a presentation by S. Doniach (Stanford) on time-resolved SAXS studies of protein folding and then a series of talks devoted to x-ray imaging. Doniach reported how proteins, after an initial rapid collapse to a metastable state, approach the final configuration with a single rate constant of order 10-100 ms, a time scale accessible to time-resolved SAXS investigations. The technique allows for example to follow the evolution of the radius of gyration along the folding pathway.

Imaging methods are very important because, among the many applications of synchrotron radiation, they can be particularly important for industrial problems and for problems of great public interest such as medical imaging. The progress in imaging based on phase contrast was discussed in the presentations by U. Bonse (University of Dortmund), S. Wilkins (CSIRO, Australia), A. Momose (Hitachi, Japan) and A. Snigirev (ESRF). Bonse reported on three-dimensional tomographic images reconstructed from the phase shifts suffered by a beam going through the sample in different directions, as measured with an interferometer, and showed the advantages of phase contrast over absorption for biological specimens composed of low-Z elements. Wilkins discussed the different experimental configurations and the theoretical foundations of phase contrast image reconstruction, with the goal of formulating the optimum conditions under which a particular feature of the sample can be imaged. Momose described in particular some steps towards the application of phase contrast methods in vivo, for which minimization of the dose is of course a crucial condition, to be obtained by optimizing the equipment and the selection of the x-ray wavelength. Snigirev presented a broad overview of different imaging and focusing techniques exploiting the partial coherence and the high collimation of undulator beams, and their application to the characterization of samples, of optical elements and of the coherence properties of the beam itself.

P. Spanne (ESRF) discussed medical imaging with hard-x-ray synchrotron radiation and the efforts underway to exploit coherence and diffraction properties for radiography. Finally, J. Gronkowsky (University of Warsaw) and J. Baruchel (ESRF) discussed the status and the challenges of topography. Gronkowsky reported on the simulations of topographic images, where new algorithms have led to substantial progress in recent years. Baruchel reported on the new scope of topographic investigation with third generation sources. In particular he reported results clarifying the growth mechanisms and the role of defects in quasi-crystals.

In summary, the Conference provided the participants with an overview not only of the recent results obtained by synchrotron radiation sources but also of the scientific fields in which context they are taking place. The talks by theoreticians in particular allowed an appreciation of the meaning and relevance of synchrotron radiation experiments in relation to the open questions at the frontiers of research.

M. Altarelli

ESRS PRIZE GIVING

On the occasion of fifty years of synchrotron radiation, the European Synchrotron Radiation Society (ESRS) awarded a prize of 1500 ecus for an outstanding contribution to synchrotron radiation science. The winner was M.I. McMahon from Daresbury for his work in the high pressure field.

M.I. Mc Mahon, winner of the ESRS prize.

Biology Symposium

The Biology Symposium was held on 19 and 20 November and ran in parallel to the main SR50 Conference. However two plenary lectures were held in common. Firstly, W. Hendrickson (New York) outlined how the Multiple Wavelength Anomalous Dispersion technique had provided a new approach to the «phase problem» in macromolecular crystallography, leading to the elucidation of structures which had not been possible with conventional methods. In this technique the ready tunability of synchrotron radiation was a key feature. Secondly, S. Doniach (Stanford) explained how small-angle x-ray scattering could be used to study the biologically important problem of how polypeptide chains fold to yield functional, three-dimensional macromolecules. The symposium, itself, concentrated mainly on biological macromolecules whose structures had been determined by protein crystallographic methods and comprised four themes; virus structures, RNA/DNA complexes, membrane proteins and time-resolved studies of reaction mechanisms. A highlight in the virus section involved a description of the blue tongue virus by D. Stuart (Oxford). This double stranded RNA virus, which is one of the most virulent among ruminants, has a core particle with a diameter of about 800 Å and an overall molecular weight of 100 MDa, thus providing a severe technical challenge with regard to diffraction data collection using synchrotron radiation and structure determination. Structural interme-diates in the life cycle of icosahedral viruses were described by M. Rossmann (West Lafayette) and L. Liljas (Uppsala) explained structural studies on the mechanism of assembly of small RNA phages using coat protein mutants and RNA complexes. In the second section an undoubted highlight was the structure of the nucleosome core particle of chromatin undertaken by T. Richmond (Zürich) and colleagues. The structure shows how four pairs of histone fold domains are responsible for organising 121 base pairs of DNA whilst extensions of the histone fold bind the termini of the DNA superhelix and help to stabilise the overall structure. Other speakers described principles of RNA-protein interaction (T. Steitz, Yale), how class II aminoacyl-tRNA synthetases work (S. Cusack, EMBL, Grenoble), recognition and repair of mismatched DNA base pairs (L. Pearl, London) and crystallographic studies of eukaryotic gene expression (S. Burley, New York). The elucidation of the structures of membrane proteins provides further stern challenges for synchrotron x-ray crystallographic techniques. J. Rosenbusch (Basel) explained how the key step of obtaining suitable crystals, albeit of small dimensions, can be achieved with reference to the growth of bacteriorhodopsin in a cubic lipid gel phase. N. Isaacs (Glasgow) described the elegantly symmetric ring structure of the light harvesting complex from photosynthetic bacteria which has enabled an understanding of the process of photosynthesis and T. Schirmer (Basel) described the structures of transmembrane proteins, general porins to maltoporin, which allow solutes and sugars (specifically malto-oligosaccharides) respectively, to pass through outer cell membranes. The final session was devoted to studies of reaction mechanisms using very fast time-resolved measurements.

I. Schlichting (Heidelberg) described the characterisation of intermediates in the reaction cycle of cytochrome P450(cam), M. Wulff (Grenoble) explained the development of the technology required to undertake such studies and K. Moffat (Chicago) highlighted studies on the millisecond to nanosecond time scale on heme proteins and the photoreactive yellow protein.

The symposium provided a first class example of how x-ray structural analysis of macromolecules using synchrotron radiation is making a vital contribution to our understanding of life processes; all the speakers are to be congratulated on the excellence of their presentations.

P. Lindley

K. Moffat (Chicago) in the final session.

Applications for Beam Time Arrive from Labs World-Wide

Scientists from laboratories in Europe, the US and Japan requested a total of 8469 shifts on beamlines at ESRF for the first seven months of 1998. In total, 581 applications arrived for the September 1997 deadline for beam time on the 28 ESRF and 4 CRG beamlines which will be scheduling user experiments during this period.

The Review Committees, whose members are external scientific experts in a range of fields, met at the ESRF on 23 and 24 October 1997 to assess the applications. Following discussions lasting one and a half days, 387 proposals were finally selected and allocated a total of 4914 shifts or 1638 days of beam time. Of these, four new long-term projects were accepted and allocated beam time for a two-year period. **Figure 1** shows the results of these allocations per Review Committee. Slightly more than 66% of proposals were successful this review round, compared with an average of 45% over previous rounds. The higher success rate this time reflects the increasing number of beamlines available, and a slightly longer scheduling period of 7 months.

The Committees commended users on the higher standard of proposals. This largely reflects the experience which experimental teams are acquiring on the beamlines, and an increasing interest in the kinds of challenging projects which can be undertaken given the instrumentation available and the specific qualities of the beam at the ESRF.

The beamlines most frequently requested this round were ID16, for inelastic scattering; ID12B, the Dragon spectrometer with polarization selectivity; ID2, the high-brilliance beamline, which combines applications in small-angle x-ray scattering and macromolecular crystallography; ID32, for SEXAFS and standing waves, and ID18, the nuclear resonance beamline.

Figure 2 shows the number of shifts allocated compared with shifts requested, per scheduling period, since the beginning of user operation in September 1994. It should be noted that the second scheduling period each year to date has been slightly shorter than the first, so that fewer shifts have been allocated and scheduled during the second half of each year.

Potential users are reminded that the next deadline in 1998 for applications for beam time is 1 September. During the corresponding scheduling period the available beam time for macromolecular crystallography will increase significantly, since two additional experimental stations on ID14 will be operational. Further, a second beamline dedicated to elastic scattering for electronic excitations, ID28, will also begin commissioning, so that by the end of the period the full complement of 30 publicly financed beamlines will be operating with users. The British CRG beamline XMaS, for magnetic scattering, will accept first users from April 1998. Details of the beamlines and instrumentation available, proposal forms and experimental report forms are available at the ESRF Web site: http://www.esrf.fr, or from the User Office.

R. Mason

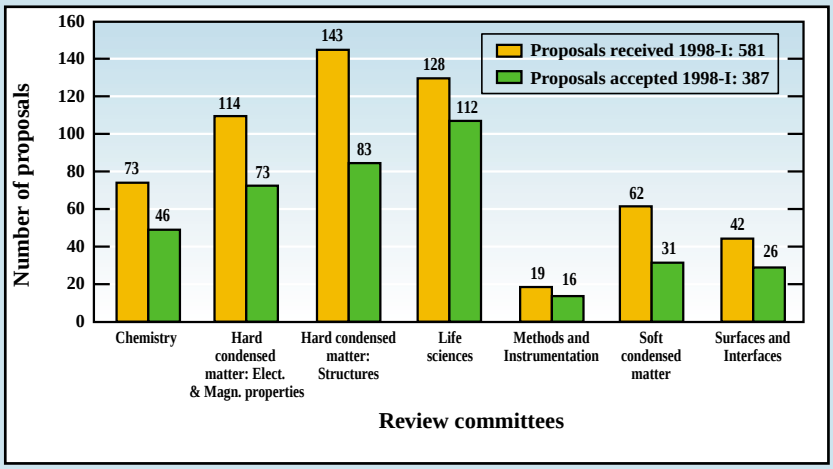
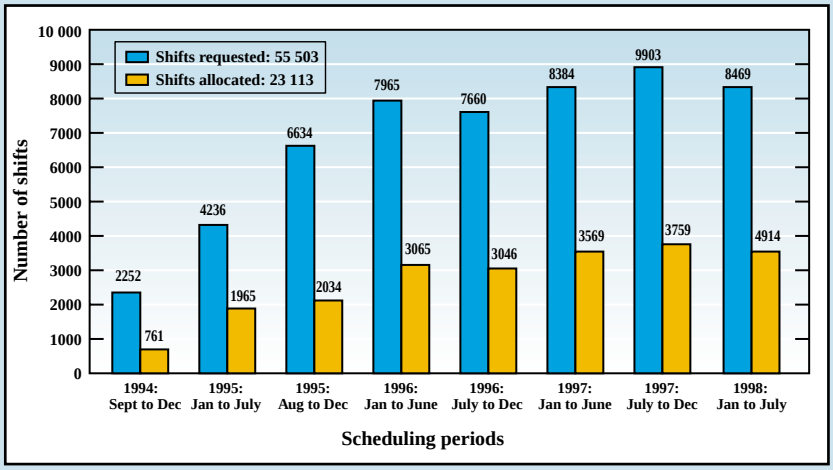


Fig. 1: Proposals submitted and allocated beam time, per Review Committee.

Fig. 2: Total number of shifts of beam time requested and allocated, per scheduling period: September 1994 to July 1998.



«Powder Diffraction at Synchrotrons»

This workshop was held at the ESRF on 11 and 12 December 1997. It was organized by A. Fitch and Å. Kvick and featured 12 invited speakers from Europe and the US (H. Fuess, TU Darmstadt - A. Fitch, ESRF - D.E. Cox, BNL - D. Louër, Univ. Rennes - R.J. Cernik, Daresbury - G. Artioli, Univ. Milano - P. Norby, Stony Brook - T. Wroblewski, Hasylab - P.J. Webster, Salford - L.B. McCusker, ETH Zürich - W.I.F. David, Rutherford - A.K. Cheetham, Univ of California). The latest developments in structure determinations from high-resolution powder data, materials characterization of industrial importance and *in situ* studies of reactions and processes using powder techniques were discussed. A concluding discussion session outlined the hardware and analysis development needs for the future. The workshop was attended by about 70 people of which more than 50 scientists came from outside the ESRF.

A. Fitch/Å. Kvick

«Ultra-Fast Kinetics of Molecular Assemblies»

On 15-16 January 1998 the ESRF hosted a workshop titled «Ultra-fast kinetics of molecular assemblies» with the aim of reviewing progress on time-resolved diffraction experiments on photosensitive proteins and to examine whether this technique is applicable to smaller scale chemical systems. 15 invited speakers covered fields ranging from protein crystallography, optical spectroscopy to future ligth and electron sources.

K. Moffat, who pioneered ultra-fast time-resolved diffraction on proteins with the scientists on ID9, presented new data of the first intermediate in the photocycle of the yellow protein PYP. Electron density maps of the first nanosecond intermediate show that the shaft of the light sensing chromophore is compressed and that the stored energy is released in a large rotation of the chromophore. These real-time data were contrasted by new static measurements by L. Getzoff, who used the «cool then activate» approach. In this way one can study intermediates preceding an activation barrier which becomes insurmountable at low temperatures. She showed beautiful monochromatic electron density maps at true atomic resolution. R. Hochstrasser gave a overview of ultra fast laser spectroscopy showing the strength and weakness of optical techniques. He showed that optical spectroscopy probes extended electronic levels which are often poor indicators of atomic structure. By contrast x-rays interact with core levels and are therefore effective structural probes. C. Shank from Lawrence Berkeley Laboratory showed how they generate 300 fs x-ray pulses at 0.4 Å by 90 degree Thompson scattering between infrared terawatt pulses and electrons from a linac. With a brilliance of $2x10^5$ photons/mm^2/mrad^2/s in a 15% bandwidth, it has been possible to study the onset of disorder in a ISn crystal from a 100 fs temperature jump. In the final session about future x-ray ligth sources, B. Wiik from Desy described the TESLA project, a proposal to construct two 16 km linacs running at 250 GeV, one for electrons and one for positrons. In the 1 x 1 nm^2 collision point, the high energy physics hope to produce the energy density a picosecond after the big bang! The instrument will allow high-energy physicists to study the top quark and to search for the still theoretical Higgs Boson, the carrier of mass. The interest to the x-ray community is the free electron side station. B. Wiik showed that a free electron laser will produce 100 fs coherent x-rays with 10^{12} photons per bunch, 10^4-10^5 times the present level at the ESRF.

M. Wulff

ESRF at «Physique 97» in Paris

The ESRF is well-known as a public research institute but probably less known as a facility open to industrial research. To accompany the development of the ESRF industrial policy, it is necessary to give the industrialists more information about the scientific potential of synchrotron radiation for industrial applications.

A good opportunity to accomplish this was given by the exhibition «Physique 97» which was held in Paris from 7 to 9 October 1997. The ESRF decided to participate in this exhibition and the operation was managed by the Industrial Relations Office, with support from the Information Office.

An exhibition such as «Physique 97» attracts a large number of scientists as well as visitors from industry (the ESRF stand received more than 1000 visitors over the three days, 50% from industry, 50% from research institutes).

In addition, this exhibition being principally commercial (most of the exhibitors are companies proposing technological and scientific equipment), it is a perfect place to find partners for the marketing of instruments developed at the ESRF.

The ESRF stand consisted of:

- scale models to show and explain the functioning of the machine and of beamlines;
- posters (4 general posters about the ESRF, 3 posters about technical developments and 5 posters about recent scientific results);
- some instruments developed at the ESRF (the FRELON camera, monochromator crystals and the cryogenic cooling system for monochromators).

The Delegate of the French Minister of Public Education, Research and Technology, M. Descusse (right) shows a strong interest in the remarkable scientific results and developments explained by J. Doucet (second from right).

The stand thus combined information provided by the posters with more animated areas: the interactive scale models, the demonstration of the FRELON camera in real operation and of course lively discussions with the ESRF staff present on the stand. General and technical documents were also distributed in large quantities during the three days.

The ESRF stand was particularly appreciated by the visitors who could find all kinds of information. It was also strongly congratulated by the organizers of the exhibition (Société Française de Physique).

J. Doucet

Workshop «Chemistry and Surfaces» for Industrialists

4 June 1998, ESRF, Grenoble

In the series of workshops «ESRF for Industry», which are organized for industrialists from European companies, a workshop dedicated to «Chemistry and Surfaces» will take place at the ESRF on 4 June 1998. For the first time, it will be held together with a similar and complementary workshop «ILL for Industry», on 5 June 1998.

The morning program will comprise a general presentation of the ESRF, a description of the various modes of access to the facilities and presentations of the possibilities of characterization of materials and control. The topics are: diffraction analysis of powdered materials; chemical reactions studied by absorption techniques; surface, interface and thin-layer analysis using synchrotron radiation; as well as new opportunities in micro-imaging techniques (tomography, x-ray microscopy, phase-contrast technique). In the afternoon, a visit of the beamlines will be organized during which the participants will benefit from informal discussions with scientists from the ESRF.

27TH AND 28TH COUNCIL MEETINGS

The ESRF Council met on 17 October 1997 in Paris in order to finalize the wording of the Arrangement between the ESRF and the Government of Portugal on the long-term scientific use of synchrotron radiation (see also ESRF Newsletter N° 29, p. 3). The approved Arrangement was signed on 12 November 1997 in Lisbon and became effective on 1 January 1998.

At its meeting on 4 and 5 December 1997 in Grenoble, the Council approved the Medium-Term Scientific Program submitted by Management which, apart from the continuous efforts to improve the radiation source and the various beamline components (e.g. optics, sample environment, data acquisition), comprised some specific projects aiming at an extension of experimental options at the ESRF, such as a TRXF station for silicon wafer mapping, the rebuilding of the MAD beamline on an insertion device location and the establishment of a second nuclear resonance scattering station.

The Council discussed its position vis-à-vis further Collaborating Research Group beamlines (regarding their scientific objectives and the burden on the ESRF general infrastructure). It decided that for any further CRG beamline, the CRG in charge will have to bear the full investment cost of the corresponding beamline front-end, and set 16 CRG beamlines branches as the threshold for the next review of the situation. (Currently 13 branches are in operation or under construction as compared with 34 branches of ESRF public beamlines.)

The Council noted some planned building construction measures, the most important being the extension of the Guest House by a third wing, possibly with the participation of the Institute Laue Langevin.

The budget for 1998, unanimously approved by the Council, provides for an expenditure level of 417 420 kFF, covered by Members' contributions of 396 000 kFF and 21 420 kFF of other income.

The Council took note of the Medium-Term Financial Estimates presented by Management and considered the expenditure and income profiles suggested for the period up to 2002 as a reasonable guideline for further developing these estimates and the associated scientific program.

With a view to the end of the ESRF construction period, the Council approved a revised version of the Financial Rules. It also adopted a first part of «Guidelines for a Readjustment of Contribution Rates», essentially dealing with the assessment of the scientific use which is made of the ESRF by the scientific communities of the various Contracting Parties. (In the case of a lasting and significant imbalance between use and contributions, the ESRF Convention provides for the option of a readjustment of the contribution rates).

Finally, the Council slightly amended the Procedures for beam time allocation in order to enable Management to better accommodate projects involving industry-based proposers and providing potential for industrial applications.

K. Witte

GEORG VON KLITZING
NEW CHAIRMAN OF THE ESRF COUNCIL

At its 26th meeting on 10 and 11 June 1997, the Council elected G. von Klitzing as its Chairman for the period from 1 January 1998 to 31 December 1999. He succeeds Prof. F. Menzinger who has chaired the ESRF Council since January 1996.

G. von Klitzing, 64, has studied law and economics. His first contact with research and science administration was in 1962 when he entered the former German Federal Ministry of Nuclear Energy (from which arose, after several intermediate steps, the BMBF of today) where he was involved in European Space Research.

G. von Klitzing's career after this was marked by a succession of periods at European organizations such as CERN, ESRO and EURATOM, and tasks within the Federal Ministry of Research and Technology. During these latter periods he was a member of various committees at European level (European Communities, JET Council, Working Group «International Fuel Cycle Evaluation», ...).

From the end of 1991 to mid-1997, G. von Klitzing was Deputy Chairman of the Board of Directors of the Forschungszentrum Jülich, i.e. the German Member of the ESRF.

During this period he became involved in ESRF matters, first as a member of the German delegation to the Council, from mid-1993 to mid-1995 as Chairman of the Administrative and Finance Committee and, for the last two years, as Vice-Chairman of the ESRF Council.

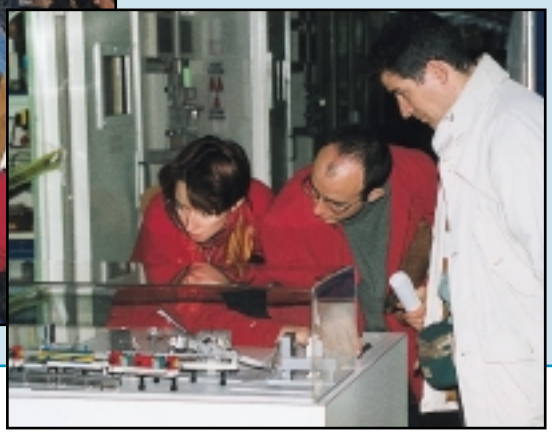
The new Vice-Chairman of the Council is Dr Paul E. Zinsli from the Swiss Federal Office of Education and Science, who - with more than ten years of service - has been the senior Head of Delegation at the ESRF.

Open Days

After nearly four years without an Open Day, the ESRF opened its doors again and attracted about 2000 people on 14 and 15 March 1998.

On their tour through the ESRF, the visitors had the opportunity to discover 12 «stations», where ESRF staff were posted and gave explanations: There was a general presentation, and they could visit the machine (the roof of the storage ring had been partly removed) and several beamlines (surfaces, materials, biology and medicine). The visitors could also attend a demonstration, on a scale model, of a new experimental station for industrial use in micro-electronics. The visitors stayed 3 hours on average and seemed delighted with their visit.

Looking into the storage ring.



Following explainations on a scale model.

Theatrical Itinerary «Lumières»

On the initiative of the director of a theatrical company in Grenoble, six actors and one musician were involved in a «theatrical itinerary» through the ESRF, mixing art and science. From 20 to 31 March 1998, twice a day, this theatrical itinerary was proposed to a group of about 35 people, who could hear about science and synchrotron light, as well as poetic, philosophical and literary texts.

This project was strongly supported by institutions such as the Rhône-Alpes region, the Grenoble town council and the Isère department council.

It had an excellent impact in the media, both in the press (30 announcements and articles) and on radio/TV (about 25 broadcasts). Foreign newspapers, radio and TV also showed a great interest in this event. On Thursday 19 March, for the last rehearsal, we welcomed correspondants from Ny Teknik (Sweden), La Tribune de Genève (Switzerland), TV3 Barcelona and El Correo (Spain), Giornale di Brescia (Italy), NRC Handelblad (The Netherlands) and RTBF (Belgium).

VACANCIES AT THE ESRF ON 20 MARCH 1998

	Ref	Subject
SCIENTIST	2170	High brilliance
POST-DOC	PDID15B M5	
	PDID9	Diffraction
	PDID21	X-ray microscopy
	PDID10A	Troika group
ENGINEERS	6106	Buildings/infrastructure
	2206	Software support
	CDD/CH	Digital electronics
TECHNICIAN	2521	Nuclear resonance
OTHER	6103	Survey and alignment

Previous postdoctoral experience with synchrotron radiation is essential.

If you are interested, please send us a fax (+33 (0) 4 76 88 24 60) or an e-mail (brink@esrf.fr) with your address, and we will provide you with an application form. You can also print out an application form on the World Wide Web http://www.esrf.fr

X-Ray Scattering and Magnetism

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Magnetism has become a major field of investigation with synchrotron radiation. In particular, the highlights of the ESRF for 1996/1997 state that «The use of x-rays for the investigation of magnetic properties of matter is a relatively recent field», and then continues with 11 pages of various examples of new effects measured at the ESRF in this field. A large fraction of the allocated beam time is spent on studying various aspects of magnetism. To focus on this activity a workshop was organized at the ESRF and held over the weekend of 15/16 November 1997 before the large SR50 conference. Some 90 attendees came to hear speakers over the day and a half of meeting.

M. Blume (BNL, USA) opened the conference recalling the fundamental cross-sections and showing how the general non-resonance cross-section was modified by the introduction of resonant terms. B. Gyorffy (Bristol, UK) and M. Brooks (Karlsruhe, Germany) discussed new directions in theory stimulated by synchrotron experiments. We heard reports of experiments using both non-resonant (Th. Brueckel, Jülich, Germany; J. McCarthy, ESRF) and resonant techniques (C. Detlefs, Ames, USA; S. Langridge, Rutherford Lab, UK) that gave representative examples in the field of scattering from synchrotrons in Hamburg, Brookhaven and Grenoble, and included new experiments to look for «orbital ordering» in perovskite materials (S. Ishirara, Sendai, Japan). Later the first day we turned to dichroism with a theory talk by P. Carra (ESRF) on the sum rules, and contributions by G. van der Laan (Daresbury Lab, UK), A. Rogalev (ESRF), and the extension of dichroism to photoemission (G. Kaindl, Berlin) and magnetic EXAFS (G. Schütz, Wurzburg, Germany). These talks showed that techniques using the dichroic signal are rapidly expanding and that more advances can be anticipated.

After a long first day, the participants were rewarded with a fine dinner in town, and the second day started in earnest at 9am (on Sunday) with contributions on surfaces and films. N. Bernhoeft (ILL), D. Gibbs (BNL, USA), G. Helgessen (Kjeller, Norway) and C. Sutter (ESRF) showed the variety of that are now being examined with scattering, almost all using resonant techniques and involving rare-earths and actinides. The workshop ended with reviews of inelastic scattering (F. Sette, ESRF) and perspectives for the future (M. Altarelli, ESRF). These last talks introduced new areas and showed that, despite the considerable impact of synchrotron studies on magnetism, the field is rapidly expanding and the future looks bright and challenging.

All participants could enjoy a fruitful exchange of ideas and experiences. The interest and involvement of the audience was reflected in lively discussions. Although there were no neutron talks per se in the workshop, all attendees were aware of the significant impact neutrons have had, and continue to have, on magnetism. It was appropriate that much of the complementary nature of the photon probe should be emphasized in the Chadwick Amphitheatre of the Institut Laue Langevin – under the stern gaze of the discoverer of the neutron. It is planned to organize a common workshop in the near future together with the neutron community on the complementary use of neutrons and x-rays in the study of magnetism.

We illustrate the broad scientific range of the workshop with the following two articles that expose recent advances in the field, although they are not directly related to magnetism.

In a first paper M. Altarelli describes the physics of the ordering of electronic orbital occupancy, which shows some similarities with magnetic order, and N. Bernhoeft et al. present the competition between the coherence length of the probe and correlation length scales in the sample that lead to new effects in x-ray and neutron scattering experiments.

DETECTION OF ORBITAL ORDER IN TRANSITION METAL OXIDES BY RESONANT X-RAY SCATTERING

M. ALTARELLI
ESRF, EXPERIMENTS DIVISION

The transition metal oxides have fascinated chemists and physicists for many decades with their intriguing structural, magnetic and electronic properties. One of the remarkable phenomena displayed by a number of these compounds is the Jahn-Teller effect, in which an orbital degeneracy of the transition metal ion is lifted by a low-symmetry lattice distortion, which «chooses» one of the degenerate states. In a lattice of such ions one can encounter a cooperative

Jahn-Teller effect, in which the occupation of the degenerate orbitals displays long-range order, characterized by a well-defined wavevector. For example in $LaMnO_3$, the Mn ions are partitioned in two sublattices, in which the $3d_{(3x^2-r^2)}$ or, respectively, the $3d_{(3y^2-r^2)}$ orbitals are occupied. It turns out that orbital order can be favoured not only by the consideration of lattice distortions, but also by other mechanisms, notably by magnetic interactions. This is because the relative orientation of occupied d orbitals in neighbouring ions determines the strength and sign of the indirect exchange (superexchange) interaction between their spins.

Recently, in view of the high current interest on transition metal oxides stimulated by the discovery of cuprate high-temperature superconductors and of colossal magneto-resistance in the manganates, orbital order phenomena have been paid much attention. One aspect which was discussed during the ESRF workshop on Magnetic Scattering is the possibility of a direct observation of orbital order by resonant x-ray scattering at the K edge of the transition metal. This could give a direct handle to the order parameter of this peculiar kind of ordered state, and a measure of its temperature dependence, and the relationship of its onset to structural or magnetic phase transitions, thus contributing to an understanding of its origin. In fact there is up to date no satisfactory way to observe and measure the orbital order parameter, although in principle non-resonant diffraction should detect the corresponding change in the shape of the electron density. The corresponding signal is however expected to be weak (less then 0.1 electrons per unit cell).

In some recent theoretical work done at the ESRF by M. Fabrizio, M. Altarelli and M. Benfatto, the idea is to exploit the sensitivity of resonant scattering of polarized x-rays to the availability of empty intermediate states of the appropriate symmetry. In an orbitally-ordered system, they are available on one sublattice but not on the other. In this work the particularly intriguing case of V_2O_3 was considered; in this compound, the wavevector of the orbital order has never been experimentally measured and is not unambiguously determined by theory; recent indirect evidence, is in some cases, interpreted as strongly supporting the existence of orbital order, and in some others as putting the very existence of orbital order in question. The conclusion of the Authors, which has not yet been verified, is that orbital order should be directly observable by resonant scattering and therefore all controversy could be settled by experiments.

In experiments performed by Y. Murakami et al. on $La_{0.5}Sr_{1.5}MnO_4$ as well as on $LaMnO_3$, a signal at the wavevector of the expected orbital order was detected, which was strongly resonant at the dipole edge corresponding to the Mn 1s to 4p transitions. In theoretical work done in Sendai by S. Ishihara and S. Maekawa, the sensitivity to orbital order was ascribed to a modulation of the energy of the empty 4p levels depending on the selective occupation of the 3d orbitals. These results of the Japanese groups were reproduced in Brookhaven by D. Gibbs and M. Blume. In our opinion, more work is called for, in order to verify whether the 4p level energy modulation is controlled by other factors or not, such as the displacement of the ligand oxygens.

In summary, the workshop provided a very timely opportunity to discuss the contribution of x-ray scattering to the investigation of orbital order, bringing together the groups involved in this problem at the ESRF, at NSLS and in Japan. ■

PROBE COHERENCE VOLUMES AND THE INTERPRETATION OF SCATTERING EXPERIMENTS

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One of the significant features of synchrotron radiation, especially that from 3rd generation insertion devices, is the small-energy bandpass of the emitted radiation. In the case of undulator beams monochromatized by Si crystals at the ESRF, the wavelength bandpass ($\Delta\lambda/\lambda$) attains $\sim 10^{-4}$. As a result the photon beam exhibits partial coherence with a longitudinal coherence length $\xi_L = \lambda/(\Delta\lambda/\lambda)$ of the order of 1 μm for 1 Å x-rays. The possibility of coherence over such dimension is fundamental to the experiments involving «speckle» patterns at the ESRF, but it is also an important consideration when conventional scattering experiments are performed, especially when the quantity ξ_L becomes comparable to the dimensions of the material being studied, or with the extinction length; the latter being written as $1/\mu$ and corresponding to the length over which absorption reduces the intensity of the radiation by a factor 1/e. Within a coherent volume the *amplitudes* rather than the intensities of the scattering process must be added. We emphasize here that these ideas are independent of the perfection of the crystal scattering the photon beam, i.e. they apply equally to both the kinematic and dynamic theories of diffraction, although the treatment in the two cases is, of course, different.

The standard procedure for correcting for absorption involves correcting the intensities diffracted from a volume of scattering, i.e. the interference between the amplitudes is first calculated to give the beam amplitude for a given reflection, and then the absorption correction is made on the final intensities. This is appropriate when the extinction length

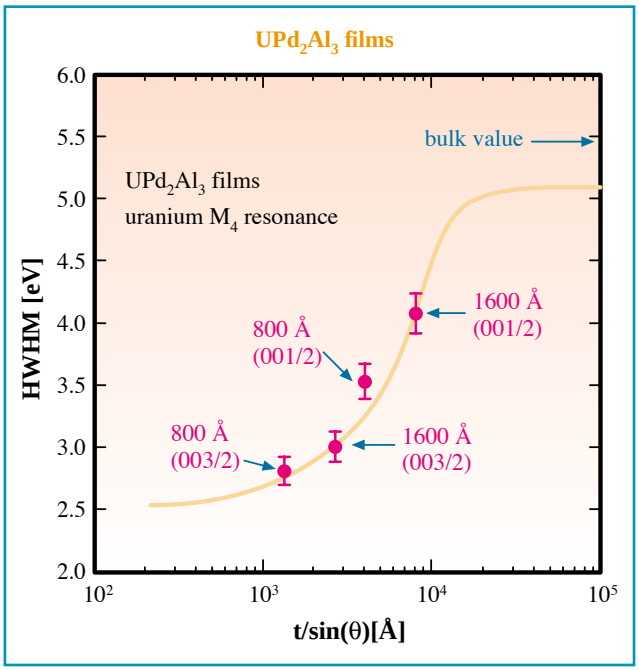


Fig.1: Energy half-width of the antiferromagnetic peaks (all at T = 4 K) at the M_4 absorption edge as a function of optical path length (t = film thickness, and θ is the Bragg angle for a reflection) of the photon beam in the thin films of UPd_2Al_3. The dashed curve is the numerical simulation assuming that the ordering is established throughout the film thickness.

is large and the coherence volume is small, $1/\mu > \xi_L$, such as when a laboratory source is used and the wavelength is far from an absorption edge. However, it is inappropriate when resonant scattering experiments (i.e. the photon energy is tuned to an absorption edge) are performed at a synchrotron source. For example, at the actinide M edges $\mu \sim 20{,}000$ cm^{-1} so that $1/\mu \sim 0.5$ μm and clearly $1/\mu < \xi_L$ in this case.

These ideas have been pursued and numerical routines developed to simulate possible situations. Space does not permit a complete description of the theory, but we will illustrate how these ideas influence scattering patterns actually observed on the magnetic diffraction beamline, ID20. Magnetic resonant scattering occurs when the photon beam is tuned to a particular resonance and a large enhancement of the (usually weak) magnetic cross-section occurs. The largest such effects have been found to be associated with actinide M edges, when the enhancement of the magnetic cross-section can reach a factor of $\sim 10^6$. At the same time the absorption is such that the probe becomes essentially one of the near surface region. In this extreme situation, numerical simulations show that the exact form of the scattering, especially its energy dependence, which changes the extinction length, will depend on the *spatial localisation of the scattering centres*. This is a new concept, coupling the spatial aspect within the sample being examined to the energy dependence of the scattering intensity.

The first example is taken from experiments on thin films of the antiferromagnet UPd_2Al_3. The films have thicknesses (t) 800 and 1600 Å and were grown epitaxially on the (111) surface of $LaAlO_3$ by co-evaporation of the elements. The antiferromagnetic (AF) order of the compound is alternating (001) layers of ferromagnetic spins so that the new Bragg peaks produced by the AF repeat distance are $(0, 0, {}^{n}/_{2})$where n = odd integer. Figure 1 shows the results of measuring the energy-dependence of the scattered intensities for two different values of n, and for the two films. There is a clear tendency for the narrowest energy widths to be achieved when the photon beam has the shortest optical path in the material. The dashed curve is the result of numerical simulations considering that the probe and sample transverse coherence may be neglected; as shown, the simulations capture the essential behaviour. At T = 4 K these films are ordered throughout their thickness, and provide a good test of the theory because of their limited thickness. Just below the ordering temperature T_N only the top part of the films orders magnetically and this may be seen by a 20% *narrowing* of the energy width of the $(0, 0, {}^{1}/_{2})$ reflection for the 1600 Å film. The best fit to this energy profile suggests that only the top 600 (± 50) Å of the film is magnetically ordered at this temperature. The lack of homogeneity is assumed to arise from the 0.15% interfacial strain between the film and the substrate.

The second example is taken from experiments on another uranium antiferromagnet UAs. The experiments are performed on an infinitely (several mm) thick crystal. In Figure 2, we show the energy profile as a function of photon energy when the crystal is near T_N. The profile clearly shows split peaks. This cannot arise from ordering just at the surface, as was seen above in the films, and which always gives rise to a narrowing of the energy width. Instead in this case there is a non-diffracting layer of some 400 Å at the

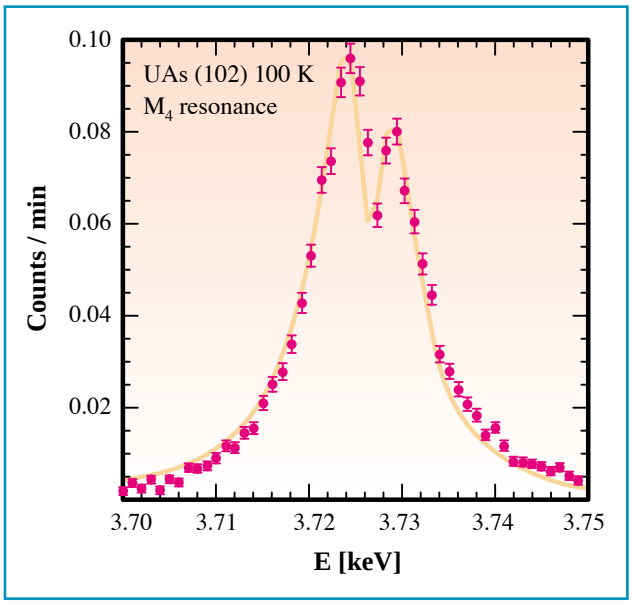


Fig. 2: Energy profile of an off-specular antiferromagnetic reflection from UAs taken near T_N on ID20. The solid line is a fit to the data employing the model described in the text and assuming that there exists a non-diffracting layer of 400 Å of the same material on top of the crystal. This belongs to another domain and so diffracts into a different direction.

surface of the UAs crystal that actually corresponds to another magnetically-ordered domain not probed by the off-specular (102) reflection. The solid line is a fit, and it reproduces the data very well.

In conclusion, we have introduced the idea that the «probe coherence volume» plays an important part in the interpretation of diffraction data taken at the ESRF. This represents a re-examination of assumptions conventionally used to interpret diffraction data. In most cases the probe coherence volumes are small – special efforts have to be made to increase the monochromaticity of the beam, which usually result in an unacceptable loss of intensity. However, these new conditions are encountered with synchrotron sources, especially with the new undulator beams, and care must be taken to understand the consequences. In the presence of strong absorption, new effects arise and we have shown that in certain cases the *energy* dependence of the intensity may be used to identify the *spatial* localization of the scattering centres. This opens the way for further experiments exploiting the partial coherence of the incident x-ray beam. The coherence of x-ray sources and the narrow bandpass beams that are available from undulators at 3rd generation sources make these effects more qualitatively important than previously thought. Discussions during the workshop made this point clear and further emphasised the new information that can be obtained from experimental results.

MEETINGS ON X-RAY IMAGING

X-ray imaging was one of the main topics of the series of meetings held in November 97 in Grenoble.
They started with the SR-50 celebration of the 50th anniversary of synchrotron radiation, where the last day was nearly completely devoted to imaging.
The User's Meeting followed, one day after; it included a plenary talk on the in-situ phase tomographic investigation of damage in a composite alloy, (J.Y. Buffiere), and a «mini-workshop» on imaging. The satellite meeting on x-ray imaging held the next day at the ESRF concluded these scientific events.
The 'imaging' mini-workshop, organized by J. Baruchel within the frame of the User's Meeting, gave a general view on what has been achieved at the ESRF (M. Schlenker's review paper), and on the big variety of techniques which have been developed at the various beamlines partly or totally devoted to imaging (BM5, A. Souvorov; ID17, P. Spanne; ID19, J. Härtwig; ID21, J. Susini; ID22, C. Raven). The use of phase contrast was highlighted by several speakers, and the specificities of the beamlines emphasized. A presentation of the available detectors (J.P. Moy) and two short scientific highlights, on the joint use of phase radiography and diffraction imaging to investigate defects in quasicrystals (L. Mancini), and on the peculiar deformations of the ice of the Antartica (P. Duval) concluded this mini-workshop.
The satellite meeting held at the ESRF was originally intended to bring together the various users of the 'long' ID19 beamline. ID19 was the first operational beamline completely devoted to x-ray imaging. The idea was, 16 months after the official aperture to users, to show what was possible, which topics were already investigated and what are the main results. The success of the meeting went beyond what was originally expected: more than 80 scientists attended the sessions and more than 50 posters were displayed. These posters presented an important fraction of the work performed on ID19, but also imaging work performed on other ESRF beamlines (BM5, ID22) and even other synchrotron radiation facilities (Daresbury, Hasylab). This one-day meeting started with a presentation on the new possibilities already implemented. It was followed by a series of short oral presentations of the posters. The main of the afternoon was devoted to an animated poster session, and a visit, for the interested people, of the ID19 and ID22 beamlines.
The highlights and trends of this meeting were extracted by B. Tanner. His, of course subjective, but quite largely shared, highlights list included the first microfluorescence mapping, the microtomographic work (bones in absorption, Al-SiC composites and insects using phase radiography), the combination of phase and diffraction imaging (defects in quasicrystals, domains in lithium niobate), the applications of diffraction imaging to magnetic materials and, through Moire imaging, to semiconductors.
The three short papers which follow partly illustrate these highlights.

J. Baruchel

COMBINING DIFFRACTION TOPOGRAPHY AND PHASE IMAGING

J. Baruchel, P. Cloetens*, M. Drakopoulos, L. Mancini, P. Rejmánková-Pernot, A. Snigirev, I. Snigireva, A. Souvorov
ESRF, Experiments Division
* also at EMAT, RUCA, Antwerp, Belgium

This work results from collaboration between ESRF staff members and scientists from other institutes: J. Gastaldi and E. Reinier (CRMC2, Marseille), J.P. Guigay and M. Schlenker (Lab. L. Néel, CNRS, Grenoble), Z. Hu and P. Thomas (Univ. of Warwick), P. Moretti (Univ. C. Bernard, Lyon).

The deliberate combination of phase and Bragg-diffraction imaging has recently led to important results, giving a new impulse to the use of these techniques. The experiments are being carried out on several beamlines: BM5, ID11, ID19 and ID22.

In the classical version of x-ray diffraction imaging ('topography') the single-crystal specimen is set to give Bragg reflection(s), and a detector placed as near as possible records the diffracted beam(s). Contrast shows up whenever the crystal includes features that locally affect the Bragg reflectivity. Thus crystal defects (dislocations, inclusions, ferroelastic domains,...) are made visible and can be characterized. The investigation in transmission of thick crystals often requires restricting one dimension of the beam («section» topography): the image then originates, in a first approximation, from a virtual slice of the crystal. When working at the ESRF an additional parameter can be varied to obtain further information: the 'propagation' distance between sample and detector.

The 'propagation' parameter also allows phase radiography and tomography to be performed in a very simple way. The 'propagation' technique takes advantage of the small size of the x-ray beam sources at ESRF, yielding a very small divergence α of the beam as seen from any point in the specimen. This results in a high degree of spatial coherence, characterised by the transverse coherence length $l_c = \lambda/2\alpha$, where λ is the x-ray wavelength. Local variations in the x-ray optical path-length in the sample, i.e. phase variations across the beam, then produce contrast through Fresnel diffraction. In the case of negligible absorption the image is uniform when the detector (x-ray film in our case) is close to the sample. As the detector is moved further away, contrast builds up, and features on different length scales are brought out more conspicuously depending on the specimen-distance D. The edges of an inhomogeneity of size s are outlined when $D < s^2/\lambda$, whereas interference fringes occur, the image being a hologram, for further increase of D.

The investigation of defects in Al-Pd-Mn single quasi-crystals is a first case where the combination of topography and phase radiography allowed a breakthrough. It is now familiar that quasicrystals, non-periodic solids with long-range order, exist, and produce fine diffraction peaks for x-rays just as do crystals. Therefore Bragg-diffraction imaging is applicable to single quasicrystals too, and it has already produced some information about the structural defects in these materials, with topology quite different from that of crystals. Figures 1a and b show a region of a single quasicrystal slab, imaged in simple transmission, with no strong Bragg reflection excited. In the enlargement (Figure 1a) a clear delineation of internal holes with a shape featuring, just as etch pits in crystals, the icosahedral point symmetry of the material, and elongated precipitates, are observed. Figure 1c shows a topograph of the same region as in Figure 1b. Loop shaped defect images, corresponding to distorted areas, are visible. The geometrical relations between the two, apparently quite different, images of the same object can be determined by recording a phase radiograph with the sample set for a strong Bragg reflection (Figure 1d). Joint use of these images allows to determine the relationships between the holes or precipitates and the complicated defects around them [1].

In conventional Bragg-diffraction imaging, the surfaces of the samples (and/or monochromator) should be strain free, but no specific demand is made on

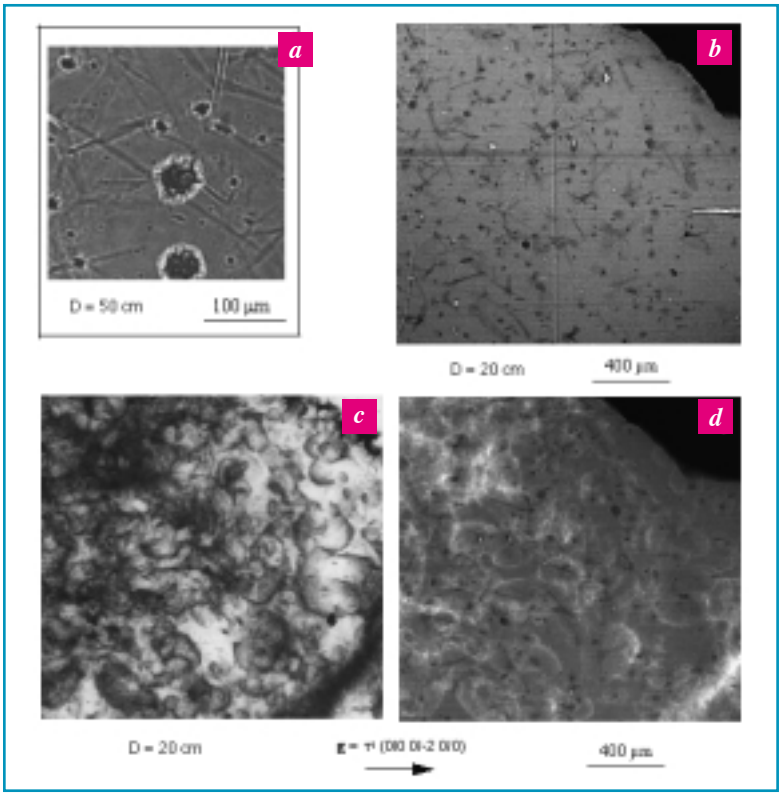


Fig. 1: Images of an Al-Pd-Mn quasicrystal recorded at $\lambda = 0.35$ Å a) phase radiograph (D = 0.5 m). In b), c) an d) the same region of the sample is presented (D = 0.2 m) b) phase radiograph with no strong Bragg diffraction excite; c) topograph (diffracted beam) and d) combined phase radiograph and topograph (transmitted beam); sample set for the τ^3 (0/0 0/-2 0/0) reflection.

their flatness. This is no more true when dealing with high quality crystals and using a coherent x-ray beam. The diffraction image then carries phase information, yielding extra contrast when the optical path length is irregular. Faint scratches on the surface of silicon crystals used as monochromators were shown to produce strong phase images which are not visible in 'classical' topography. The same approach was used to reveal thin oxide strips on reflections where the associated distortion does not produce contrast [2].

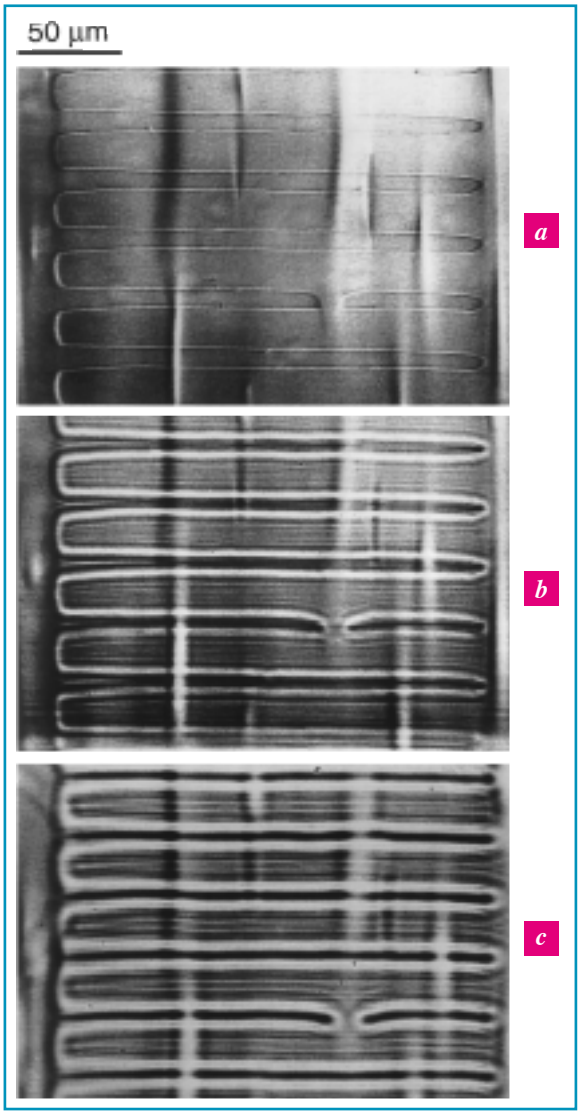
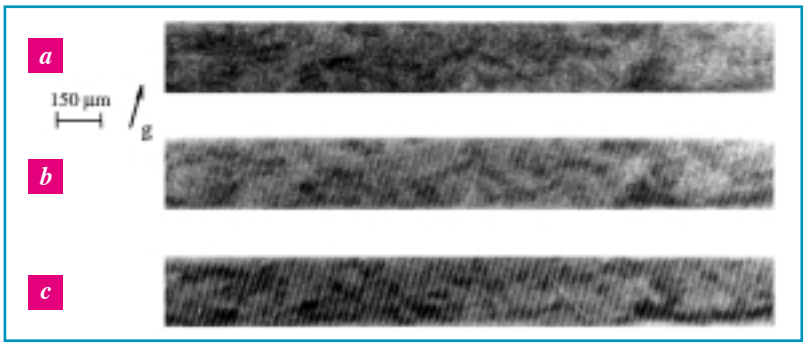


Fig. 2: Images of inversion domain walls in a $LiNbO_3$ crystal recorded using the 006 reflection (reflection geometry), λ = 1 Å, at different sample-to-film distances D: a) 0.5 cm b) 20 cm c) 50 cm. Note the interference fringes which build up as the distance is increased.

Fig. 3: Section topographs (transmission geometry) of a periodically poled $LiNbO_3$ crystal using the 274 reflection, λ = 0.24 Å, recorded at different sample-to-film distances D: a) 0.11 m, b) 0.51 m, c) 1.67 m, g is the projection of the diffraction vector on the detector.



The combination of phase and diffraction imaging led to spectacular results with beams Bragg-diffracted by periodically poled lithium niobate crystals. These crystals exhibit large optical nonlinearities and are widely used for electro-optic and acousto-optic applications as well as for optical frequency conversion. Poling produces a periodic spatial distribution of the sign of the nonlinear optical coefficient, crucial for the quasi-phase matching required for optical harmonic generation. When parts of the coherent x-ray wave front diffracted from domain inverted regions are split and subsequently overlap, they interfere [3]. Figure 2 shows a series of images at different sample-to-film distances, in Bragg (reflection) geometry. The fringes show the phase origin of the phenomenon. The inverted ferroelectric domains exhibit structure factors with very similar modules, but different phases. This gives rise to contrast mechanism, used to image the domain distribution in the bulk in Laue (transmission) geometry as shown on Figure 3. Many reflections involve nearly no effect of domain-related lattice distortion. Contrast then mostly originates from the phase shift between the structure factors in the domains. It is thus possible to extract, from images recorded at different distances, a direct measurement of this phase difference [4]. It was on the other hand shown that the contrast of the domain images can be enhanced by the application of an electric field [5].

The above examples show some of the unique possibilities resulting from the combined use of Bragg and Fresnel diffraction imaging techniques. This is clearly an emerging new field.

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IN SITU DAMAGE ASSESSMENT IN MICRO-HETEROGENEOUS MATERIALS USING HIGH RESOLUTION X-RAY TOMOGRAPHY

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Metal Matrix Composites (a metal reinforced by ceramic particles or fibers) combine good mechanical properties and a low density. Hence, those materials are very attractive for automotive applications where a gain in weight is highly desirable. However, the poor fracture properties of those materials have greatly restricted, so far, their use in industrial applications. Therefore, in the last years, much attention has been paid to the study of fracture processes of Metal Matrix Composites (MMC). For instance, in situ mechanical tests have been used by several authors and provide very interesting information on the loss of ductility of MMC. However, the relevance of surface examination has to be checked by destructive observations within the material through time consuming techniques like serial sectioning which are not free of artefacts. To overcome this problem, we have used phase contrast tomography, to obtain three dimensional images of damage within materials under stress.

The damage mechanisms of an Aluminium/Silicon Carbide (Al/SiC) composite strained in tension have been studied *in situ* by phase contrast tomography using a specially designed tensile testing device. The phase contrast technique enhances the contrast between the aluminium matrix and the SiC particles with respect to classical attenuation-based tomography. The steady development of cracks in the SiC particles has been monitored on the same sample for increasing values of plastic deformation. The use of phase contrast enables to visualize cracks with an opening lower than 0.5 microns in the SiC reinforcements. For the first time, a quantitative comparison of the damage mechanisms observed at the surface and those observed in the bulk, by a non destructive method, is presented.

EXPERIMENTS

High-resolution x-ray tomography experiments were carried out at the ESRF in Grenoble on the «topography» beamline ID19. Images of damage induced by a tensile test within a MMC (aluminium matrix reinforced by silicon carbide (SiC) particles) have been obtained.

Because of the low difference in the x-ray attenuation of aluminium and SiC, classical transmission tomography, based on attenuation laws, was hardly able to discriminate between matrix and reinforcement. To improve this contrast, the very high lateral coherence of the beamline ID19 was used to produce **phase contrast images** through a very simple experimental set-up [1]. A Fast Read-Out Low Noise CCD detector, developed at the ESRF, was placed at about 1 metre behind the sample to record edge diffraction patterns resulting from discontinuities in the material such as interfaces between reinforcement and matrix or strain-induced cracks. Figure 1 shows a three-dimensional reconstruction obtained from the recorded phase images, where the reinforcing particles are clearly visible.

The studied material was a 6061 aluminium alloy reinforced with 10 % in volume of silicon carbide (SiC) particles with an average size of 120 μm. The material was produced through a rheocasting route and subsequently extruded at high temperature. A special tensile testing device was built in order to record tomographic scans of the sample under load. The frame of the tensile testing device, made out of a PMMA tube, gave negligible absorption on the 2D images along the 180° rotation of the sample during the scan. Small double-shouldered tensile specimens with a cross-section of 1.5 * 1.5 mm^2 and a gage length of 5 mm were used. The sample surfaces were mechanically polished using SiC paper and diamond paste down to 1 μm. The tensile tests were carried out at room temperature using a constant crosshead displacement rate of 150 μm.min^{-1}. Several scans were performed on the same sample at the initial state and after several increments of plastic deformation. During the scan, the position of the crosshead was maintained constant. Before and after the mechanical tests, the sample was observed in a Scanning Electron Microscope (SEM) operated at 20 keV.

RESULTS

Reconstructed 3D volumes show clearly the initiation of damage and its evolution within the material. Internal pores, resulting from the manufacturing process, were observed in the material at the initial state in the vicinity of the SiC particles. At the surface, those pores had been filled by the polishing process and, therefore, only a few of them were detected in the SEM. Thanks to the phase contrast technique, cracks with an opening down to 0.5 μm - i.e. well below the voxel size of 6.5 μm^3 - could be detected in the reinforcing particles.

From a qualitative point of view, the damage mechanisms observed in the bulk did not differ from those observed

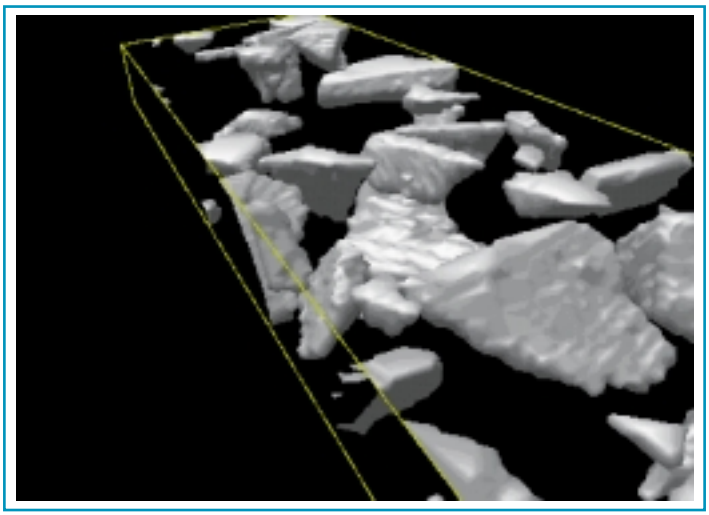

Fig. 1: Three dimensional reconstruction of some reinforcing particles in the interior of an Al/SiC composite. The average size of the particles is 120 µm.

at the surface. Schematically, the evolution of damage as a function of plastic strain can be described as follows:
1 cracking of SiC particles,
2 reinforcement/matrix decohesions,
3 propagation of processing-induced pores.

Representative images of the evolution of damage in the sample during the tensile test are presented in Figure 2.

From a quantitative point of view, however, the present experiment shows that the number of cracked SiC particles as a function of the plastic strain is substantially larger in the bulk than at the surface. Finite element modelling of the deformation process of a two-phase material is now being carried out to try to account for the observed differences between the bulk and the surface. ■

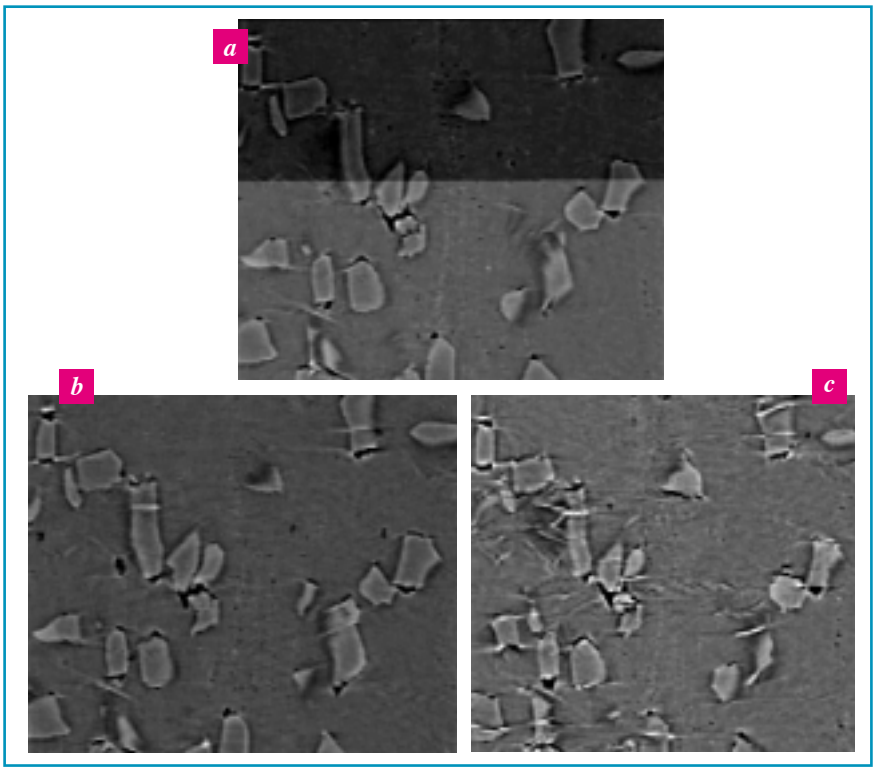

Fig. 2: Reconstructed images of the interior of the sample at the initial state (a) and after two steps of plastic deformation (b and c). A tensile stress was applied along the vertical direction. Some cracks in the particles are indicated by white arrows.

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BRAGG DIFFRACTION IMAGING OF MAGNETIC CRYSTALS: NEW RESULTS FROM NOVEL BEAMS

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INTRODUCTION

Bragg diffraction x-ray imaging (x-ray topography) produces direct-space images of crystal defects, domains, or just thickness variations in single crystals, via the inhomogeneous intensity of the Bragg-diffracted beams.

The advent of synchrotron radiation brought high intensity making real-time imaging possible and at high energy machines a large flux of high energy photons, allowing the observation of thick samples in transmission geometry. On third-generation machines, the small emittance of the source again provides new possibilities. Because image blurring remains small even when the detector is placed far from the sample, particularly at the long (145 m) beamline of ID19, the specimen-detector distance becomes a new adjustable parameter. It extends the range of options traditionally available: white beam vs monochromatic beam imaging, projection vs section topography, selection of wavelength and choice of the Bragg reflection.

We briefly present results obtained at the ESRF, using beamline ID19, on magnetic materials: the oldest magnetic material known, magnetite Fe_3O_4; hæmatite α-Fe_2O_3, manganese phosphide MnP, with a wealth of magnetic phases; and iron borate $FeBO_3$. Although the scattering process used is Thomson (charge) scattering and provides information on the magnetic moments only via the lattice distortion, quite spectacular results were obtained.

PROBING SYMMETRY: THE LOW-TEMPERATURE PHASE OF MAGNETITE

Magnetite, cubic above its Curie point and a ferrimagnet at room temperature, exhibits at 120 K a transition affecting its electric and magnetic properties. Below this Verwey transition, the crystal symmetry is either monoclinic or even triclinic, implying that a single crystal splits into many domains or twins. Magnetite is also magnetoelectric at low temperature, with ferroelectricity coupled with ferrimagnetism.

The domain structure was followed across the transition, using white-beam topography. The least complicated situation prevailed, as expected, when the transition was passed in a magnetic field favouring one of the easy <001> magnetization directions, or the nearest easy directions [001]. Even then, however, so many domains were superimposed on any projection topograph that, contrary to standard usage, this provided no overall view. White beam section topographs, in which the effectively investigated region was a ribbon, some 20 μm thick across the 0.8 mm thick sample, provided clues to the coexisting domains, their shape and arrangement.

Figure 1a shows a typical result, corresponding to one Laue spot, to be compared with the single straight line a single domain would yield. Unraveling this data involved first the determination of matching lines, associated with adjacent domains in the specimen; then the measurement of the geometric splitting of the corresponding lines associated with the relative rotation of the lattice planes for a number of Laue spots; and comparison with calculation. The outcome is a plausible model, shown in Figure 1b, of monoclinic domains, with all walls satisfying the conditions of neither magnetic nor electric charge distributions, and no long-range elastic stress. Supplementary contrast is observed, and is the first unambiguous evidence of extra distortions associated with triclinic symmetry.

The ability to vary the sample-to-film distance, to obtain high sensitivity to lattice plane rotation at large (40 cm) distances, and to confirm the assignment of matching elements at smaller distances, proved essential.

Fig. 1: a) Section topograph in white beam. Low temperature phase of magnetite. T = 110 K, B = 300 mT; 593 reflections, λ = 0.3 Å, $\mu t \approx 1$. b) Model for the monoclinic twins in the sample.

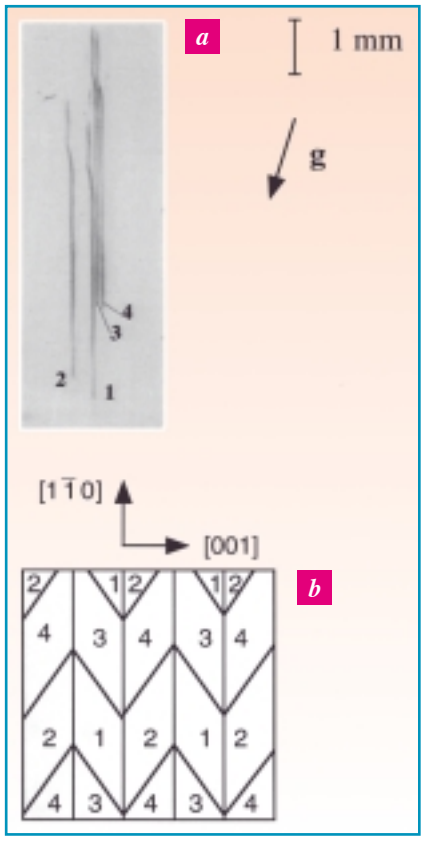
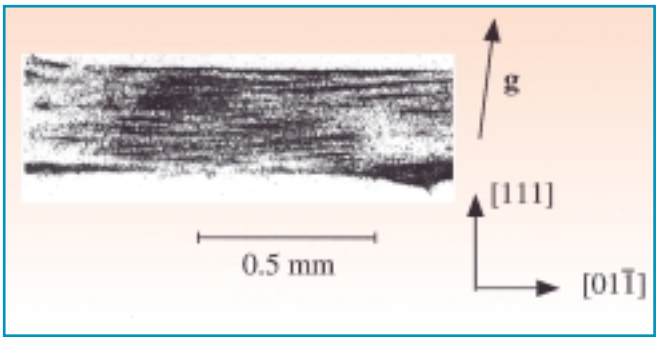


Fig. 2: White beam section topograph on a 1.2 mm thick hematite sample. 21 reflection, λ = 0.22 Å, $\mu t \approx 0.5$. Demagnetized state.



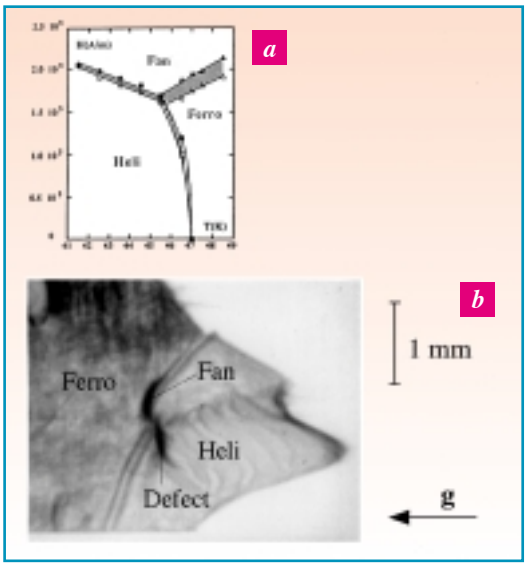


Fig. 3: a) Magnetic phase diagram, indicating the range in applied field of the coexistence on the video monitor. b) White beam topograph at the triple point in MnP. 020 reflection.

AN UNUSUAL MAGNETIZATION PROCESS: HÆMATITE

Hæmatite at room temperature is a weak ferromagnet, in which the magnetic moments are almost antiparallel and lie in the low anisotropy basal plane of the trigonal structure. Under an in-plane magnetic field, samples parallel to this (111) plane fail to show domain walls. The white beam section topography investigation of a very good crystal provides the clue to this riddle (Figure 2): the walls are pinned along (111), and magnetization occurs through rotation of the magnetic moments. This is quite different from the standard process prevailing in materials with more sizeable anisotropy, where magnetization would proceed through the displacement of walls, which would furthermore be highly unlikely to take this maximum area orientation.

MAGNETIC PHASE COEXISTENCE: MANGANESE PHOSPHIDE MNP

Orthorhombic MnP features a magnetic phase diagram with ferromagnetic, helimagnetic, and fan structures all within reach with moderate temperatures and fields. Synchrotron radiation topography can reveal the coexistence of two or three of the magnetic phases because each involves slightly different lattice distortions. In the standard representation, the magnetic phase diagram as an H,T plot shows coexistence lines. The experimental plot in the H_o,T plane, where H_o is the applied field, shows bands instead of lines (Figure 3a). This is related to the different spontaneous magnetization in each phase. A range of values of H_o corresponds to the same value of $\mathbf{H} = \mathbf{H}_o + \mathbf{H}_{dem}$ because the demagnetizing field $\mathbf{H}_{dem}$ changes continuously as one of the coexisting phases grows. In the same way, the triple point is spread into a (small) area in the H_o,T plane. Figure 3b shows a projection topograph obtained at this triple «point» and a schematic assignment of the various parts of the image to the different phases. One of the noteworthy features is that the fan phase nucleates within the heli-ferromagnetic interface.

VISUALIZING MAGNETOELASTIC RESONANCES IN THE MHZ RANGE: IRON BORATE

Iron borate is trigonal, isomorphous with haematite and features strong magnetoelastic coupling. White beam projection topographs were recorded while a very good quality (111) single crystal plate, 50 μm thick, was excited into an elastic resonance by a 1.3 MHz a.c. magnetic field superimposed on a small perpendicular d.c. field, both in-plane. The vibration of the magnetic moments induces standing acoustic waves. In this imaging mode, the predominant effect is that of lattice plane rotation of maximum amplitude ϕ_o. A membrane type vibration leads to focusing of different components of the beam. Figure 4 shows some of the results obtained from one Laue spot as the excitation amplitude, hence the rotation amplitude ϕ_o, was varied. Very similar pictures (ESRF Highlights 1996-97) were obtained as a function of the sample-film distance L. This is in accordance with the formal theoretical description where the relevant parameter is the product $L\phi_o$, and again points to the importance of the sample-to-detector distance as a new free parameter provided by the small emittance at the ESRF. Such an experiment gives access to the main characteristics of the vibration: amplitude, shape, polarization, wavelength and sound velocity. In addition this effect could be used to obtain a high frequency pulsed, «monochromatic», and focused x-ray beam. ■

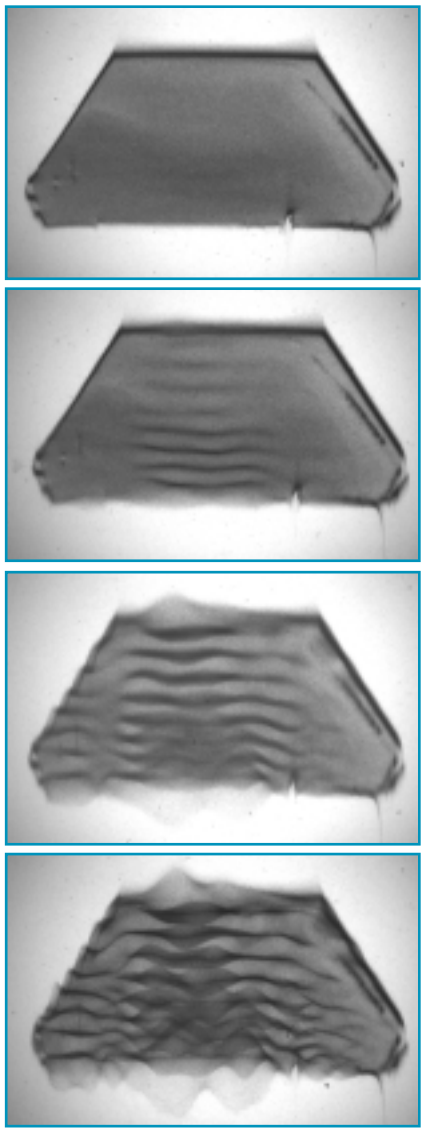


Fig. 4: White beam topographs of the reflection in $FeBO_3$ 119 cm after the sample as a function of the excitation amplitude; the resonance pattern reveals the time integrated images of the pulsed, focused x-ray beam.

High Pressure Workshop

High-pressure studies in science, more particularly in the physical and geophysical sciences, have greatly benefited from the development of new techniques at synchrotron radiation sources in the last 15 years. The availability of very high-brilliance radiation at the ESRF since 1993 has largely contributed to this development, and to the popularization of the use of the pressure parameter in many fields, especially in those of structural studies and crystallography in extreme conditions of pressure and temperature. With the setting up of the High-Pressure Commission of the International Union of Crystallography (IUCr) at the Seattle congress in 1996, the success of the ESRF high-pressure program, and the first high-pressure experiments at Spring-8 and APS in 1997, the time was right to organize the first workshop of the new commission. It was held at the ESRF from 21 to 23 November 1997. 27 scientists representing the main research groups active in these fields across the globe accepted to give very interesting presentations on the latest results and developments at synchrotron radiation facilities and in associated fields. The program was divided into ten sessions. The first two were concerned with non-structural techniques. They provided an overview of the latest developments and results in magnetic dichroism studies, Mössbauer spectroscopy, inelastic scattering, x-ray absorption, EXAFS of single crystals, and diffraction combined with spectroscopy. This was followed by two sessions on extreme conditions covering laser and resistive heating in angle- (ADX) and energy-dispersive (EDX) diffraction, extreme pressures, and structural studies of low-Z materials in these conditions. After talks on liquids and large-volume presses, attention was given to solving and refining structures with a comparison between neutron and x-ray techniques, a report of the work done recently at the ESRF on nitrogen, and a provocative comparison of the respective powers of ADX and EDX. After an exciting session on strength, elasticity and kinetics, all hot topics at the moment as the animated accompanying discussions showed, the last two sessions concentrated on 2-D data and detectors, and 3rd generation sources. The former covered 2-D data analysis, the «Fastscan» detector (see articles on pages 27 and 30) and pixel detectors. The latter included a review of the ESRF highlights, a presentation of the Spring-8 high pressure facilities, and an overview of the main technical and scientific projects in high pressure at APS, this including a proposal for a new 'multi-techniques' sector dedicated to high pressure research. To round things off, an exciting forward looking view of the new science and techniques pointing over the horizon left much of the audience with even bolder expectations. The program also contained several very interesting discussion sessions which contributed greatly to the success of the workshop. Over 90 participants attended the meeting, including eleven young scientists who benefited from travel awards provided by the IUCr. The organizers and HP Commission members thank the IUCr for this financial support. Finally, I thank all those who contributed to making this a great event, and an important step in the development and success of the field of high pressure research. I cannot name them all here, but special thanks are due to the HP Commission, and more particularly to its chairman, Prof. R. Nelmes, for his invaluable help with the program.

D. Häusermann

LASER HEATING UNDER PRESSURE: A BRILLIANT JOURNEY TOWARDS THE CENTRE OF THE EARTH

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INTRODUCTION

The study of the deep Earth has been motivating generations of scientists who have to take up the challenge given by the extreme conditions existing at the centre of the Earth: over 300 GPa (3 million bar) and 5000 Kelvin. As the study of the propagation of elastic waves created by earthquakes only allow the determination of the density profile of our planet, there remains the all important problem of the determination of the chemical composition and crystalline structures existing in the deep Earth, as these govern the Earth global exchanges (thermal regimes, convection drifts, plate tectonics...). Thus relationships have to be established between chemical composition, crystalline structure and specific volume over the whole range of pressures and temperatures existing within our planet, and x-ray diffraction is by far the best technique to obtain reliable structural and molar volume data on the compounds and materials of interest. Consequently, as very high pressures can only be generated in extremely small samples, the high brilliance of the ESRF, coupled with advances in detector, optics and high pressure technologies, has stimulated the rapid development of techniques for collecting structural data on geophysical samples in extreme conditions of P and T. We report here some recent results from a project concerned with in-situ x-ray diffraction studies of laser-heated samples under pressure [1], and the examples selected are studies devoted to the two major constituents of the deep Earth: iron and $MgSiO_3$ perovskite.

EXPERIMENTAL DEVELOPMENTS

Before this project, structural data on materials in laser-heated diamond-anvil cells (DACs) were obtained using white-beam energy-dispersive diffraction, a technique which suffers from an intrinsic low resolution and poor crystallite statistics (hence unreliable intensity data) due to the small window of diffraction space sampled, but thanks to the high brilliance of the ESRF, it is now possible to combine monochromatic angle-dispersive diffraction and image-plate detectors to collect quality data up to pressures and temperatures in excess of 90 GPa and 3000 K. This is achieved by focusing the high brilliance beam produced by two phased 40 mm period undulators [2] using single-electrode bimorph mirrors [3] on the High Pressure beamline (ID30). The resulting focal spot of about 8 μm x 15 μm (FWHM) is compatible with the size of the laser-heated hot spot, and the wavelength of the monochromatic beam, selected by a water-cooled channel-cut Si(111) monochromator in the 0.4 to 0.5 Å range, is well matched to the aperture of custom-built DACs. Combining these beam characteristics with an experimental set-up especially designed for the project, and consisting of TEM00 CO_2 and multimode YAG lasers, optical set-ups for on-line P,T measurements and alignment of the sample and beam, large aperture DACs allowing in-situ P,T measurements and full 4θ data collection, for the first time it has been possible to collect angle-dispersive diffraction data on image-plates during the laser-heating of samples only a few micron thick.

A NEW-PHASE OF IRON

Iron being the dominant constituent of the Earth's core, information on its behavior at high P and T is fundamental in Earth sciences, but despite numerous studies on this subject [4] there is still much uncertainty about its structure in the P,T conditions relevant to the core. The accurate determination of the phase diagram of this element is indeed an experimental challenge because of the extreme conditions involved, and even below 100 GPa, recent x-ray diffraction experiments have led to conflicting results on the structure of its β-phase. Thus that region of the phase diagram, which was previously regarded as simple, is in fact complicated and clearly in need of new experimental data. Indeed, as mentioned by Anderson [4], the final choice between the ε and γ phases for the core depends on the outcome of future studies aiming at proving the existence of the β-phase and identifying its crystallographic structure.

The iron phase diagram up to 100 GPa and 2700 K has thus been studied using the best diffraction technique

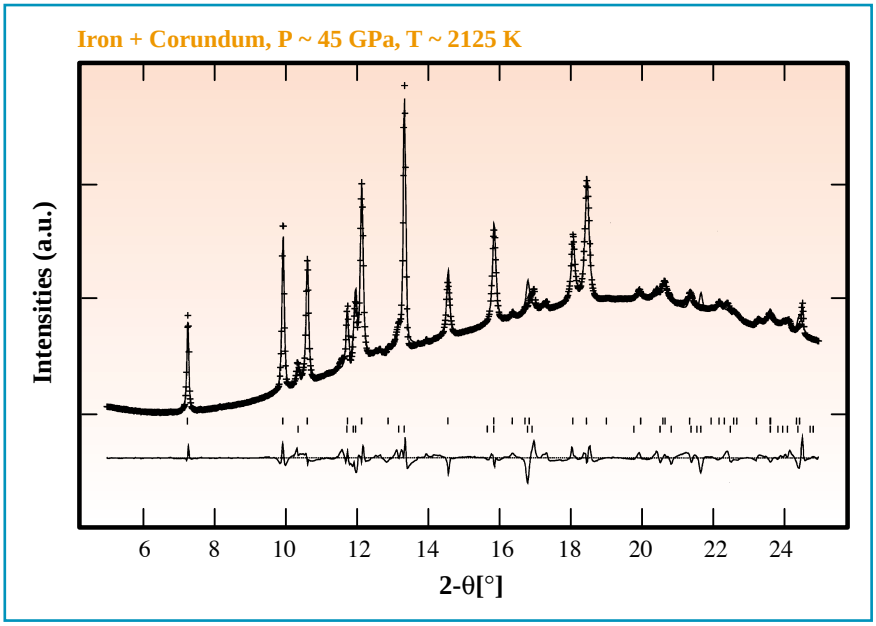

Fig. 1: Full structure refinement of a spectrum recorded at 2125 K, 44.6 GPa. The cell is orthorhombic with space group Pbcm and iron location {0.239, 0.472, 0.25}. Observed, calculated and difference spectra are shown, with lower ticks for iron and upper ticks for Al_2O_3.

currently available, and these results introduce strong constraints on its structures at high P and T. Having significantly improved both the resolution and the reliability of the data, it has been possible for the first time to perform full structural Rietveld-refinement in these extreme conditions of P and T (Figure 1). The space group determined is Pbcm, and the atomic topology is close to that of the ε-hcp phase. The structure is also closely related to the lower pressure, high-T polymorph, γ-fcc. The high-T polymorph appears unquenchable at moderate pressures, but the spectra of the back-transformed γ-phase show some anomalies which can explain ambiguities reported in previous structure determinations [5].

To eliminate possible artifacts introduced by the pressure transmitting medium (corundum, which is also used as thermal insulator), the occurrence of the orthorhombic lattice in a SiO_2 medium was also checked. This is illustrated in Figure 2 which shows results obtained up to about 100 GPa. Unfortunalely at this pressure the sample was not sufficiently insulated from the diamonds, and it was too difficult to obtain *in-situ* spectra during stable laser heating. However, spectra quenched from about 2500 K clearly show the doubling of the 100 and 101 lines of the hcp-lattice, here again an evidence of a transformation at high P and T. All the experimental lines are explained by an orthorhombic lattice similar to that previously observed, and in contrast with results obtained at moderate pressures, the structure of the high-T polymorph is now preserved after the quench. At this pressure, the orthorhombic lattice is found to be about 1% denser than ε-iron.

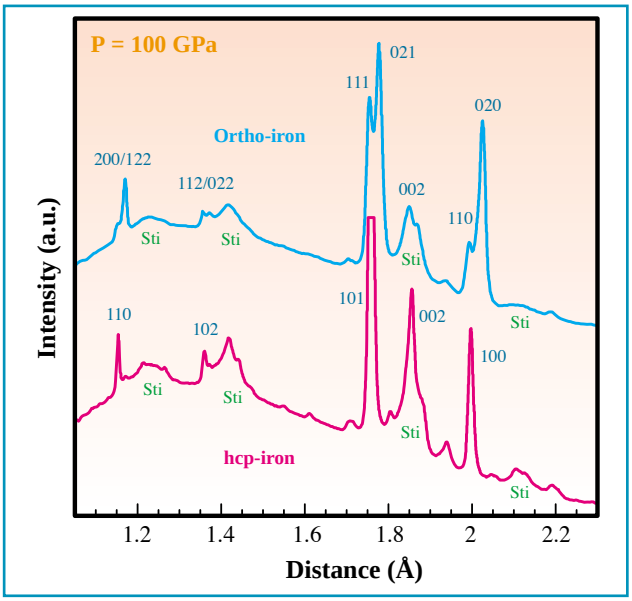

Fig. 2: Diffraction spectra of hcp and orthorhombic iron recorded at 100 GPa in a SiO_2 medium (labeled Sti). The 101 reflection of ε-iron is truncated for better clarity. The top spectrum, quenched from about 2500 K, clearly shows the doubling of the 100 and 101 ε-iron lines, evidence of the phase transition toward the orthorhombic phase.

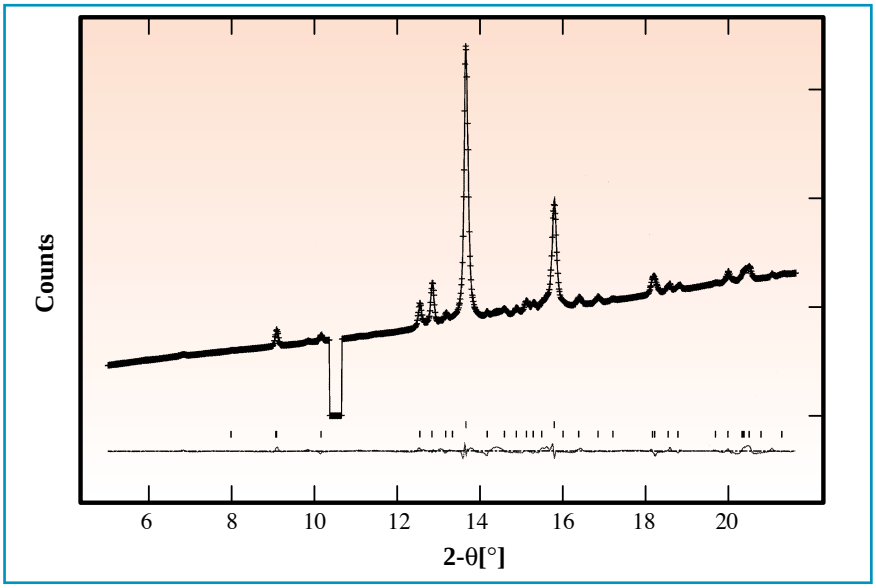

Fig. 3: Full Rietveld structure refinement of a diffraction spectrum of $MgSiO_3$ at 86 GPa and 2310 K integrated from an image plate exposed for 10 minutes using a monochromatic beam focused to 10 µm x 20 µm. Sample and platimum (the pressure calibrant) reflections are shown by lower and upper ticks respectively.

THE STRUCTURE OF MGSIO$_3$ PEROVSKITE

The perovskite form of (Mg,Fe)SiO_3 being currently accepted as the dominant phase of the Earth's lower mantle (700 to 2900 km deep), its equation of state (EOS) plays an important role in many fields of geophysics. It is however presently impossible to choose between the perovskite-pure and perovskite-magnesiowüstite (Mg, Fe)O models for the Earth's lower mantle on the basis of the existing data, and in-situ high P and T diffraction is certainly the only method available to measure correctly its EOS and solve the stuctural problem. Previous studies were conducted in the stability field of the perovskite, but energy-dispersive diffraction and large-volume presses limited the performance and P,T ranges to 30 GPa and 2000 K respectively. Using the technique described earlier, our measurements on $MgSiO_3$ perovskite were extended to 86 GPa and 2700 K. Here however the new on-line image-plate detector (the «Fastscan» [6]) now available on the ID30 beamline was used for the data collection.

Silicate perovskite $MgSiO_3$ samples were synthesized from synthetic $MgSiO_3$ enstatite crystals or synthetic $MgSiO_3$ glass mixed with platinum powder, and once loaded in a large aperture DAC, the starting materials were transformed at high P using either the CO_2 or the YAG infrared lasers, depending of the pressure transmitting medium. The temperature was determined by analyzing the thermal emission spectra recorded during the diffraction measurements and the pressure conditions were calculated from the EOS of platinum, used here as internal pressure calibrant [7].

Le Bail profile refinements were performed on the diffraction patterns to obtain reliable high P, high T cell parameters for the sample and the pressure calibrant up to 86 GPa and 2700 K, and the most remarkable result was that Rietveld structural refinements were successfully carried out on selected patterns in these extreme conditions [8] (Figure 3). This gave for the first time

precious structural information on these compounds, as for instance the first observation of the increase of the internal distortion of the SiO_6 octahedra with increasing pressure in a powder sample. Furthermore, the data analysis allowed us to identify a set of thermoelastic parameters to constrain the compositional model of the Earth's lower mantle. Assuming that the thermoelastic parameters obtained from this study are applicable to perovskites with moderate iron content, then the comparison of the density and K_T profiles calculated for a mixture of perovskite and magnesiowüstite with those obtained from the PREM [9] model indicates that a pure perovskite lower mantle is very unlikely. On the other hand, a very good match between the PREM density and K_T profiles is obtained for a mixture of 83 vol% $(Mg_{0.93}, Fe_{0.07})SiO_3$ perovskite and 17 vol% $(Mg_{0.79}Fe_{0.21})O$ magnesiowüstite [8].

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DENSITY MEASUREMENTS OF LIQUID IRON ALLOYS AT HIGH PRESSURES: TOWARDS A BETTER UNDERSTANDING OF THE PLANETS

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Physical properties of iron-based liquids are of much interest to better understand both the current state of planetary cores and their formation during the differentiation of planets. Here we present the first experiments performed on metallic liquids in the Fe-Ni-S system, which might be relevant at least to the terrestrial outer-core and the martian core. Using a large-volume press apparatus (a Paris-Edinburgh press), the P-T range of 0-4 GPa and 20-1250 °C was explored by measuring the absorption profiles, hence density, of samples using high-energy x-rays. Equations of state of liquid iron alloys are therefore on the way to be determined, along with accurate melting-phase diagrams as a function of pressure and temperature relevant to geophysical conditions.

GEOPHYSICAL INTERESTS

Density measurements of Fe-based liquids at pressure and temperature relevant to planetary cores are essential to model accurately the core composition and convection. This should help resolve two important geophysical issues: the generation of the Earth's magnetic field and the thermal history of the planet.

Also relevant to these measurements is the differentiation of planets, i.e. at first order, the individualization of a metallic core towards the center of the planet. All these phenomena refer to the liquid state of core materials, which concerns at least the outer terrestrial core, but also probably Mars, Venus and some Galilean satellites such as Ganymede for example, as a substantial magnetic field (roughly a

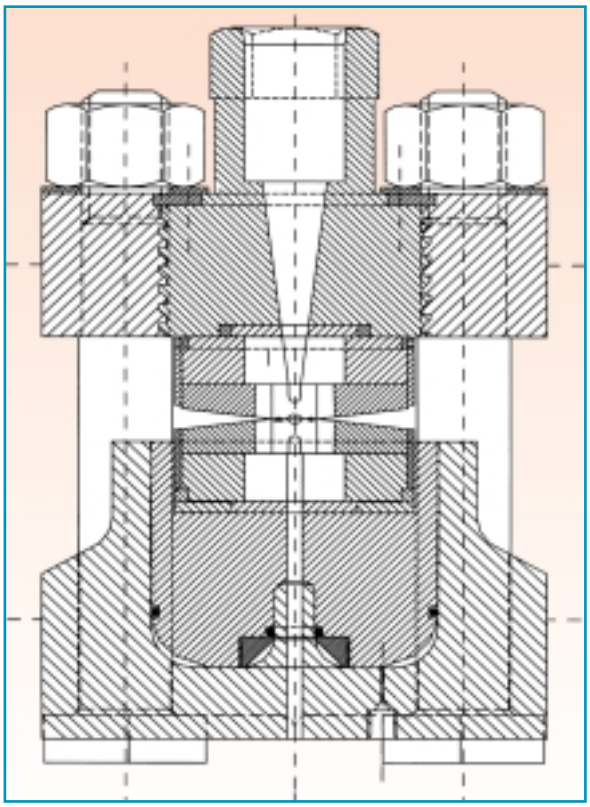

Fig. 1: The Paris-Edinburgh press (model V4). Using a force of 300 tonnes, 300 mm³ of material (gasket, heater, calibrant, sample...) are compressed so that the maximum pressure of the central 100 mm³ of sample is 12 GPa. The device weighs approximately 50 kg and measures 20 cm by 20 cm by 25 cm. With high-resistivity graphite heaters, the sample temperature is raised to 1600 °C by passing a current of 100 A through the anvils, giving a maximum power of 250 W.

tenth of the Earth's field in intensity) was recently detected by the NASA's Galileo spacecraft.

Seismic waves, generated by earthquakes, have revealed to be powerful probes of density and elastic properties of the interiors of the Earth, and soon of the Moon and Mars. Indeed, seismic data predict that the terrestrial core is composed of an iron-nickel alloy up to 90 %, plus 10 % of a light element, which is mainly supposed to be either oxygen, silicon, sulfur, or carbon, or a mixture of these elements. The composition of the core can also be assessed using geochemical models of planetary formation, and in fact the consensus which emerges now is that sulfur seems to be quite an ubiquitous component of metallic cores, but in varying proportions (up to 25 wt % for the Martian core for example, and less than 4 wt % in the Earth's core).

The P-T domain of interest differs from one planet to another, ranging from as high as 130-330 GPa and 4500-5500 K for the Earth, to 8-12 GPa and 1500-2000 K for Ganymede. In the experiments discussed below, we have reached a P-T range of 5 GPa and 1200 °C, which is already of great interest for such geophysical and astrophysical problems, even though it needs to be extended to more realistic conditions.

PRELIMINARY RESULTS

Using a large-volume press apparatus (known as the Paris-Edinburgh press - see Figure 1), the P-T range of 0-4 GPa and 20-1250 °C was explored, and high-energy x-ray absorption data (λ = 0.21 Å) were collected *in-situ* using the method developed by Katayama et al. [1] for the study of liquid tellurium. These measurements were carried out on the ESRF High Pressure beamline (ID30) using radiation from the 70 mm period wiggler. The pressure and temperature conditions were determined by computing the intersections of the isochoric lines for hexagonal BN (the sample is contained in a hBN cylinder) and γFe, just before melting. The temperature was also calibrated against the power delivered by the system using these isochoric lines, which allowed us to determine an empirical law for T as a function of the input power, and then to extrapolate it to higher temperatures when the Fe-S alloy was molten.

Disappearance of crystalline diffraction peaks allowed a precise determination of the melting point of samples, while their density was obtained from the x-ray absorption curve (Figure 2) as x-ray absorption obeys the Beer-Lambert law:

$$I/Io = \int_{beam\ size} exp\ (-\mu_{liq}\rho_{liq}(X) - \mu_{env}\rho_{env})\ dx$$

where I is the intensity of the x-ray beam, μ the mass-absorption coefficient, ρ the density, and 'env' stands for the sample environment.

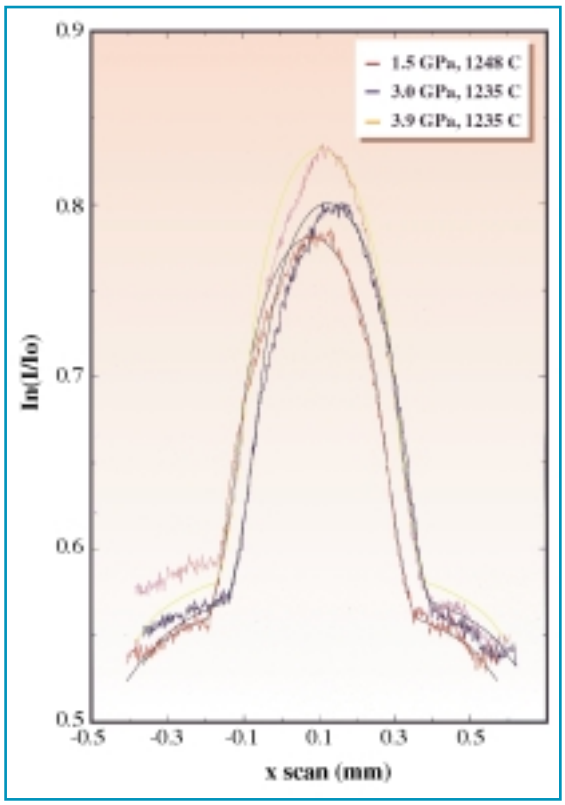

Fig. 2: X-ray absorption scans of 73 % Fe-27 % S liquids. The logarithm of I/Io is fitted according to the Beer-Lambert law (continuous lines, see text) at different pressures (1.5, 3.0 and 3.9 GPa), and around 1250 °C; the background signal corresponds to the sample environment.

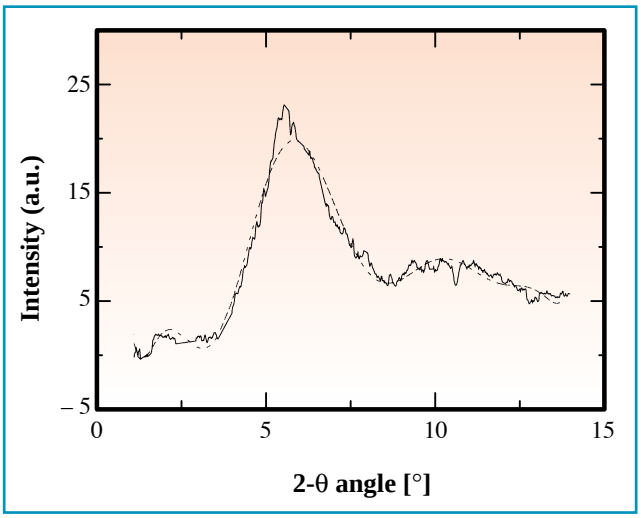

Fig. 3: 1-D integrated diffraction spectra of 73 % Fe-27 % S liquid at 3.9 GPa and 1235 °C. They are fitted with a polynomial function which shows the first and second rings of the radial distribution function.

During our first set of experiments, liquids were obtained in the Fe-S system by mixing Fe and FeS powders to have 25wt % of sulphur. It appears that the presence of sulfur dramatically reduces the bulk modulus, Ko, as Nasch et al. [2] already noticed when carrying out an ultrasonic interferometry investigation of molten Fe-5 %Ni-10 %S at ambient pressure. Indeed, they obtained a value of 63 GPa for Ko, while it is 110 GPa for pure liquid iron, and the value we obtained in these experiments is as low as 27 GPa with 2.5 times their quantity of sulfur. This result could be of considerable geophysical and astrophysical importance, if confirmed in our future studies. In particular, it could be used to determine the maximum sulfur content in the Earth's core, and soon in the Martian core, by direct comparison with core bulk moduli derived from seismological data.

As mentioned earlier, absorption and diffraction data were collected simultaneously, so that in addition to the observation of the disappearance of the crystalline state, the diffraction data gave a first order approximation of the radial distribution function. The integrated diffraction data (the «Fastscan» detector was also used here to collect 2-D images - see article by Fiquet et al. on page 26) of liquid 73%Fe-27%S at 3.9 GPa and 1235 °C (Figure 3) show a broad peak at around 5.5° in 2θ, which corresponds to an interatomic distance of 2.1 Å for the position of the first neighbours, and we can also distinguish a second weaker peak at about 10.5° in 2θ.

Lastly, and of great importance, is the high-precision measurement of the equations of states of the products of metal oxidization. This is carried out using the same high-pressure device, but in a conventional diffraction mode, and also with image plates. Here again the «Fastscan» detector is used, and an example of integrated spectra is shown in Figure 4. Images are collected by first illuminating the sample and its surroundings, then only its surroundings by translation of the whole press, and the difference spectra obtained are of high enough quality to allow analysis by Rietveld refinement. This opens up the possibility of studying the equilibria between liquid metals and solid oxides along planetary P-T profiles. These equilibria are the first order parameters of the establishment and evolution of redox states in the planetary interiors, and these eventually determine the nature of the fluids at the surfaces. Considering the promise of these preliminary results, we are now planning to explore the Fe-Ni-S-O system at higher pressures (up to 8-10GPa) and for various other compositions. ■

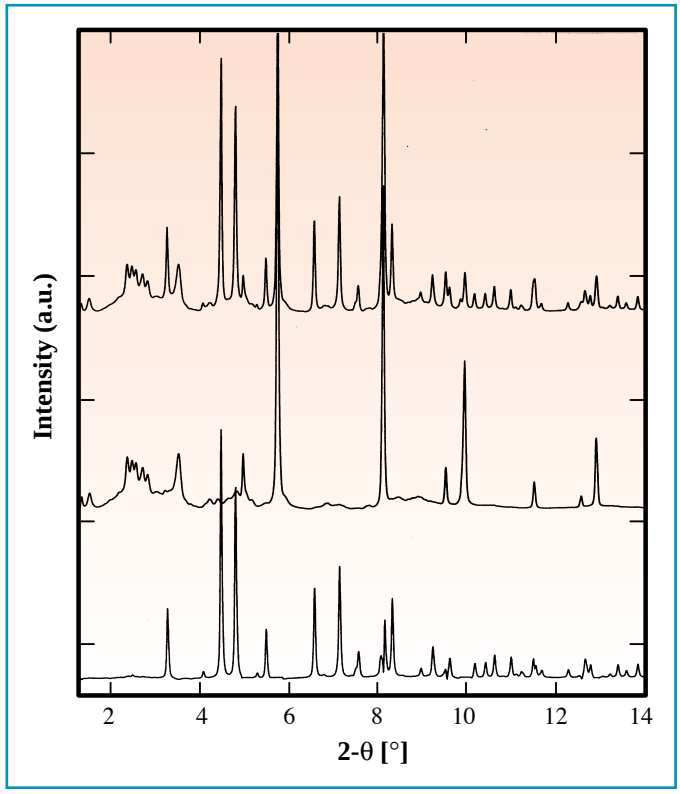

Fig. 4: Diffraction spectra of Fe_2O_3 integrated from 2-D «Fastscan» images obtained in 20 seconds using unfocussed high-energy radiation. Top: sample, calibrant and gasket, middle: calibrant and gasket, bottom: recovered Fe_2O_3 spectrum suitable for Rietveld analysis.

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A Euroconference and NEA Workshop

on Speciation, Techniques, and Facilities for Radioactive Materials at Synchrotron Light Sources

will be held at the ESRF, Grenoble,
4-6 October 1998

This meeting will introduce the applications of synchrotron-based analytical techniques to environmental and radionuclide scientists, provide a forum for teaching and scientific discussion, and establish a possible cooperative scientific network. Invited speakers will give introductory lectures on the types of information that can be obtained from synchrotron-based techniques and report the latest results on radionuclide work.

Persons who are interested in applying synchrotron radiation can obtain necessary information from lectures on protocols which are in place for radionuclide research at several synchrotron storage rings throughout the world and during a tour of the ESRF.

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COLLABORATING RESEARCH GROUPS: A REVIEW

In 1988 the ESRF had its first discussions with external users to consider how an increase in the number of beamlines at the facility might be achieved. The ESRF had been designed and funded as a facility which would have essentially insertion device (ID) x-ray sources, but it was clear that scientists from the member countries who had no access or insufficient access to beamlines at National Synchrotron Radiation Facilities could benefit from the use of the radiation generated by the bending magnets (BM) in the storage ring. These sources, although less intense than the ID sources can, with appropriate focusing optics, produce x-ray intensities at the sample position which exceed that found in many national facilities. The Council agreed at its June 1990 meeting that scientists from the member countries could form groups to exploit these BM sources provided that this did not divert either manpower or funds from the main ESRF program. These groups are called Collaborating Research Groups (CRG). Several groups were formed in a very short period of time, and their scientific programs together with beamline designs were submitted to the Science Advisory Committee (SAC) for formal approval in 1991 (see Newsletter Nr 9). Formal contracts with the ESRF were signed soon afterwards and construction of the first CRG beamlines in the experimental hall started in 1992. The ESRF supports the CRG program, and provides x-rays free of charge in return for use of the instrumentation on the CRG beamlines for 1/3 of the scheduled beam time. The ESRF also provides the front-ends which transport the x-rays from the source to the beamlines. In the future the cost of the ESRF front-ends will be refunded by the CRG.

The CRG program is now well established, with 4 CRGs having operated a user program since the end of 1994, and with 4 more groups having beamlines in an advanced stage of commissioning. A 9th CRG was approved by the Council at its June 1997 meeting, and the ESRF is aware that three possible further projects are under discussion. In view of the burden on the general ESRF infrastructure by further beamlines, the ESRF Council decided at its December 1997 meeting to set 16 independent CRG beamline branches as the threshold for the next review of the situation by the Council. The scientific aims of each of the first 9 CRGs are given in Table 1.

The GRAAL experiment at the ESRF also operates as a CRG although it is dedicated to nuclear physics measurements, and does not use synchrotron radiation (see more details on page 41).

The user operation of the ESRF started in September 1994, and the shifts (8-hour running periods) allocated for user experiments on the operational CRG beamlines, up to the end of 1997, are shown in Figure 1. In total, 1723 shifts have been carried out for public users and 4014 shifts have been allocated to the CRGs «private» users. The total of 5737 compares with 18029 shifts being available for public users on the ESRF beamlines during the same period. Using these scheduling figures as a measure, we see that the public user program has been increased by nearly 10% due to the presence of the CRGs, and that the total scientific activity at the ESRF was increased by over 30% during the four-year

Table 1

CRG	NATIONALITY	STUDIES	OPERATION DATES SINCE	FORESEEN
BM1 (SNBL)	Swiss/Norwegian	Multipurpose	Jan. 95	
BM2 (D2AM)	French	Materials / Biology	Sept. 94	
BM7 (GRAAL)	Italian	Photoproduction of particles	Mid. 95	
BM8 (GILDA)	Italian	Multipurpose	Sept. 94	
BM20 (ROBL)	German	Materials / Radiochemistry		Sept. 98
BM25 (SPLINE)	Spanish	Multipurpose		2001
BM26 (DUBBLE)	Dutch / Belgian	Multipurpose		Sept. 98
BM28 (XMAS)	British	Magnetic scattering		April 98
BM30 (FIP)	French	Proteins structure		Sept. 98
BM32 (IF)	French	Interface studies (multipurpose)	Sept. 94	

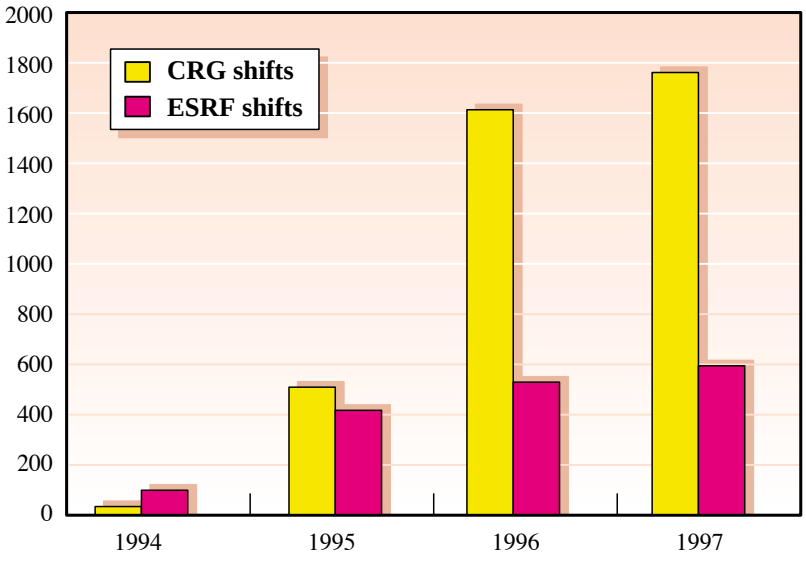

Fig. 1: shifts on CRG beamlines.

start-up period. It is expected that the CRGs will be able to maintain a significant contribution to the scientific life of the ESRF as more beamlines become operational during 1998.
This extra activity is of course not obtained «free of charge», the total extra-capital investment by the countries having one or more CRG beamlines will be about 200 MFF by the time the ten groups all have operational beamlines. In addition, the salaries of beamline staff and the travel costs for about 1350 CRG users who have come to the ESRF so far, to carry out 450 CRG proposals, are funded from the CRGs' own budgets.

CRG infrastructure - ESRF/CRG collaborations

At the inception of the CRG program, the CRGs formed a «club» as a forum for sharing new ideas, for discussing the difficulties involved in working in a «foreign» laboratory, and as a means of communicating with the ESRF. This quickly led to the ESRF Council's agreement to the appointment of a CRG liaison engineer to be responsible for advising the CRGs on all technical matters relating to the installation and operation of their beamlines. When the first beamlines became operational, the CRGs started to need help with the internal ESRF administration of their own user programs and collectively provided funding for employment of secretarial assistance. This user oriented work has now increased and currently two part-time secretaries are employed. During the last year, CRGs have also been collectively funding a technician post to assist with various construction and user-oriented tasks on their beamlines. These staff are employed by the ESRF on behalf of the CRGs.

The ESRF and the CRGs have co-operated in two more significant areas. The first follows an initiative by the Swiss/Norwegian CRG to split the 6 mrad bending magnet radiation fan into two to enable two beamlines to operate independently. Other groups asked for a 9 mrad wide radiation fan to be delivered to the experimental hall. The ESRF subsequently redesigned part of the front-end so that this could be achieved, and this development has greatly eased the space problems associated with the installation of two sets of optical components in the confined space available in a single optics hutch. Three CRGs have now chosen this 9 mrad option, despite having to pay for the additional front-end costs involved.
The second area of co-operation has been the provision of office and laboratory accommodation for the CRG staff and users. As previously stated, the original ESRF plans did not include any provisions for the CRGs. Through the CRG Club, the CRGs jointly expressed a need for accommodation «as close as possible to the beamlines». This is why there are now some buildings projecting outside the experimental hall. The ESRF Technical Services Division made the planning and construction of these buildings possible, while the ESRF Administration together with the CRGs made long-term financial agreements ensuring there would be no cost to the ESRF.
With the move from the construction phase of the ESRF towards routine operation, the CRGs are, through the CRG Club, extending the co-operation between themselves, and where possible, with the ESRF. Since the start of the CRG program, the ESRF has recommended that the CRGs adopt ESRF technical solutions where possible as such a policy has many advantages. The CRGs are now building up a stock of spare parts which are commonly available, and it is hoped that in the near future this concept will be extended to other areas, e.g. participation in a detector pool at the ESRF.

I. Kilvington

SNBL (Swiss-Norwegian Beamline) on BM1

From left to right: P. Pattison, K. Knudsen, H. Emerich, W. Van Beek; in the car: H.P. Weber.

The Swiss-Norwegian facility is split into two branch lines: one dedicated to single-crystal diffraction (2.5 mrad fan of radiation, focused beam, high-resolution monochromator) and the other to powder diffraction, EXAFS and topography (1 mrad fan of radiation, unfocused, channel-cut monochromator). To illustrate the kind of research carried out on the two beamlines, we present and discuss one recent investigation from each branch line.

A NEW TECHNIQUE: PHYSICALLY ESTIMATED TRIPLET PHASES

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Information on phase is lost in the general diffraction experiment with hard x-rays, a fact which gives rise to the so-called phase problem in crystallography. This is in contrast to diffraction of visible light or electrons, where the scattered beams can be collected by suitable lenses, to provide directly a reconstruction of the scattering object. However, even with hard x-rays, phase information can be retrieved from specially designed experiments.

Electron diffractionists have known for about 50 years that phase information is available as interference effects between several beams diffracted simultaneously in *perfect crystals* (n-beam diffraction) [1, 2]. Experimental evidence was obtained later with x-rays [3]. This direct, physical approach to structure-factor phase acquisition became of much greater practical importance when several groups [4, 5, 6] in the early 1980's found that phase effects can be significant also with truly *mosaic crystals*. Mosaic, or non-perfect crystals are vastly more abundant than the perfect specimens.

STATUS

Theoretical descriptions of n-beam x-ray diffraction have been developed by several authors on different levels of approximation; for a review see Weckert and Hümmer [7]. In a recent contribution Thorkildsen and Larsen have considered the influence of various parameters describing the radiation and its interaction with a finite, non-perfect diffracting crystal, on the solution of the Takagi equations for three-beam diffraction [8]. The experimental side has grown very strongly over the past decade, first and foremost through the diligent work of Weckert, Hümmer and collaborators in developing highly sophisticated instrumentation and measurement techniques. Triplet phases can now be estimated physically even for crystals of macromolecules. At present, phases have been acquired successfully for at least 5-6 different proteins.

EXPERIMENT

The n-beam experiment in its simplest form comprises three beams (n = 3), one incoming and two diffracted beams. A very simple representation is shown in Figure 1. In the crystal the incident beam $\mathbf{K_0}$ can be scattered into the $\mathbf{K_H}$ direction by two different pathways, either by the direct wave diffracted at the lattice planes H, or by the *Umweg* wave diffracted first at lattice planes L, and then at the H-L planes. In analogy with holography experiments the total wavefield in direction $\mathbf{K_H}$ will depend both on the amplitudes of the component waves - F_H, F_L and F_{H-L} - and the phase difference $D = (f_L + f_{H-L}) - f_H$. The latter quantity can be rewritten as a phase sum $F_3 = f_{-H} + f_L + f_{H-L}$, a so-called three-

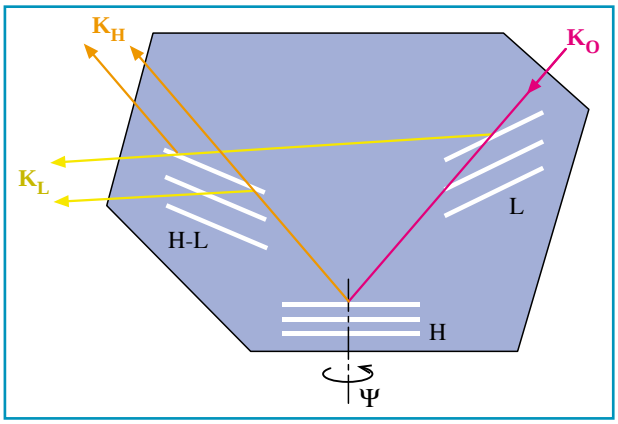

Fig. 1: A three-beam diffraction case illustrated in real space. K_0 is the incoming beam, K_H and K_L are the primary and the secondary diffracted beams, respectively.

phase structure invariant, which is a physically unique quantity of the crystal. In the experiment the crystal is rotated about the normal to lattice planes H, such that the planes L are brought successively into and out of the diffraction position, while reflection H remains fully excited. During this rotation, called a Y-scan, the primary diffracted intensity I_H is recorded. The interference between the diffracted beams which contains information about the triplet phase F_3, is projected out as a characteristic modulation of the intensity profile. In addition to this phase dependent «signature», the primary diffracted intensity may be enhanced (*Umweganregung*) or depleted (*Aufhellung*) by phase independent contributions from the other beams.

APPLICATIONS

Physically estimated triplet phases (PETP) have several interesting applications, of which only a few have been realized so far. An obvious application is to initiate the determination of molecular structures. Apparently, only one unknown small structure has been solved from PETP [9]. Feasibility studies have shown that solution of macromolecular structures is within reach. Other applications include determination of absolute structure and studies of quasicrystals [7].

A NEW APPLICATION

Phases are much more sensitive to small changes in structure than are the structure-factor amplitudes that can be derived from the intensities measured in the standard diffraction experiment. Therefore, PETP could presumably be used as well to discriminate between several possible and closely-related models of a structure. This novel application was explored in a study of the complex a-D-glucose ∑ NaCl ∑ H_2O (6:3:3) in space group $P3_1$, with unit-cell volume V = 4180 Å^3. A structure of the complex has been published (Model CA) [10]. However, there exists one alternative solution (Model GE) [11]. We found from refinements that the primary difference in structure is an interchange of the Cl and water O positions, which is accompanied by smaller, mainly translatory displacements of glucose rings, and a reorientation of water molecules and one of the glucose OH groups. This leads to a reversal of the polarity in chains of H-bonds along the polar **c** axis. The changes in structure imply significant changes in phase for about 50% of the structure factors. In contrast, triplet phases being sensitive to the model are extremely scarce. Thus, only about 0.05% of the calculated three-phase invariants with amplitudes suitable for phase measurement had a triplet-phase difference $|DF_3| > 30^o$! By measuring several *model-sensitive* three-phase invariants and comparing with those calculated for the refined models CA and GE we expected initially to be able to identify one correct model of the pair.

EXPERIMENTAL

Thirteen crystals, all cut from one large single crystal of the complex were subjected to physical phase estimation which involved studies of model-sensitive triplet phases. Each phase assignment was based on the intensity profiles mapped out in Y-scans of the pair of triplets -H/L/H-L and H/-L/-H+L, corresponding to phases $+F_3$ and $-F_3$, respectively. Two distinct right-handed cell matrices are possible in this space group. It was ascertained that the choice of unit cell of the crystals was internally consistent and in agreement with the indexing of the native data sets CA and GE. In total 309 pairs of three-beam interference profiles were collected for the thirteen crystals. The measurements included 89 different triplets.

RESULTS

The estimated triplet phases have been compared with the parent values calculated for the refined structure models CA and GE. A statistical analysis provides highly significant evidence that two structures are present, but do not coexist in the same crystal. Six crystals are in agreement with model CA, seven crystals fit the GE model. The estimated random error in the phase assignment is 19.7o; for all crystals the model with the largest mean variance can be rejected at a significance level $p << 0.001$. The results are presented in Figure 2. The cell parameters of the two structures are equal within 1 esd., as determined with synchrotron radiation, λ = 1.0000 Å.

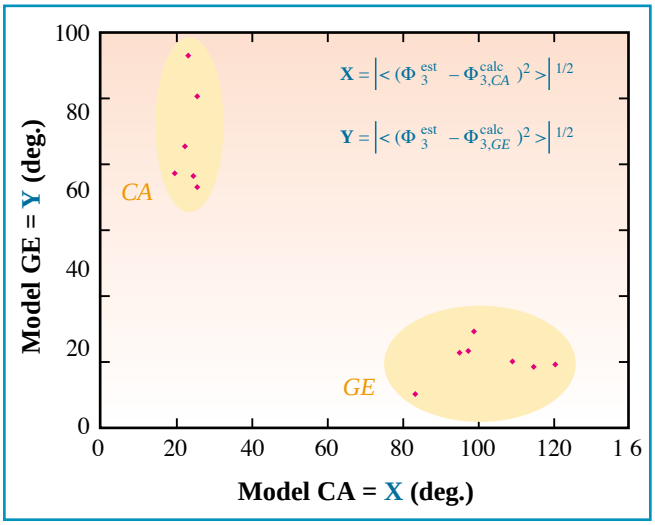

Fig. 2: Classification regions given for the mean errors calculated relative to the two models.

As an example, the intensity profiles for two triplets, one model insensitive with $|DF_3| \sim 20^o$ (a), and one model sensitive with $|DF_3| \sim 170^o$ (b) , are shown in Figure 3 for both structure models. In each pair, the upper profile corresponds to the triplet -H/L/H-L. The modified intensity is plotted relative to the intensity at the two-beam level, I_H = 1.0, as a function of Y. Note the inverted asymmetry and the reversal of high/low amplitude from top to bottom profile between the two triplet pairs of Figure 3(b), in accord with phase assignments differing by about 160°. A full account of this work will be published [12].

The Y-scans were carried out on a 6-circle diffractometer (Prof. Hümmer, Univ. of Karlsruhe) located on the Swiss-Norwegian Beamline.

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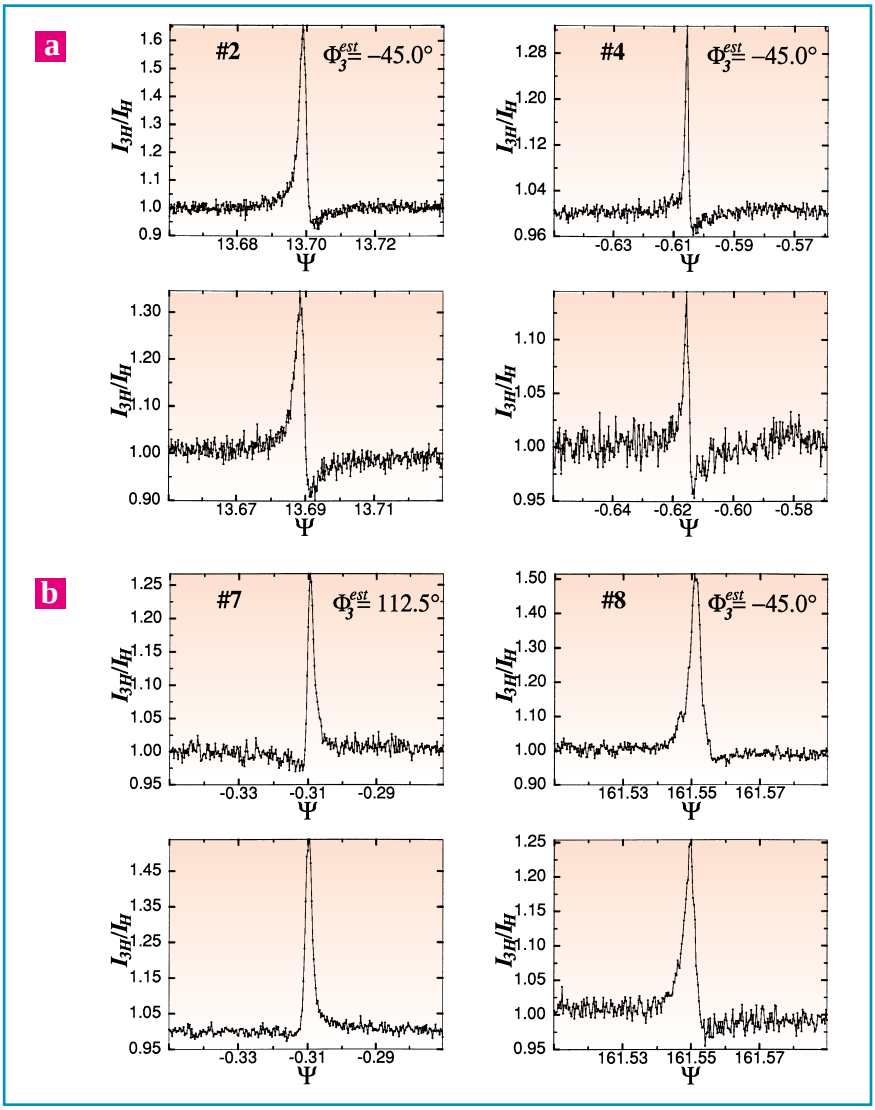
Fig. 3: Y-scan profiles of four triplet pairs. Estimated triplet phase, F3est, and crystal serial no. are given in the upper profile of each pair. (a) Triplet 3 0 -4/ -2 4 -1/ -1 -4 5, F3CA = -48o (Cryst. #2), F3GE = -65o (Cryst. #4). (b) Triplet -2 -2 -3/ 1 4 -7/ 1 -2 10, F3CA = 105o (Cryst. #7), F3GE = -66o (Cryst. #8).



EXAFS: HYDRIDE TREATMENT CATALYSTS

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EXAFS is the only method suitable to determine the local structures of bulk, non-crystalline materials under arbitrary atmosphere temperature and pressure conditions. From an experimental point of view, EXAFS is a particularly simple technique; essentially, one measures x-ray absorption spectra of the samples of interest as a function of energy; the skill is mostly in the analysis of the spectra, and progress has come mostly from computer-intensive modelling. The technique has been widely used to investigate the structure of supported catalysts. Up to recently, only the contribution from the first shell of atoms (mostly M-O) to the EXAFS spectra was analyzed; this yielded the coordination number of the absorber atom. However, with the recent development of multiple scattering theory, the analysis of the contribution from the higher shells (mostly M-M interaction) has become possible; it

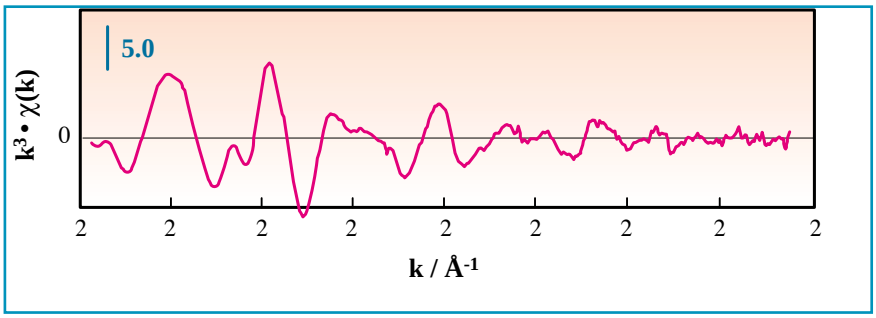

Fig. 1: k^3-weighed EXAFS function of HTC-1.9 catalyst in the oxidic state. Obtained from the raw absorption data by subtracting background and normalizing to one absorber atom.

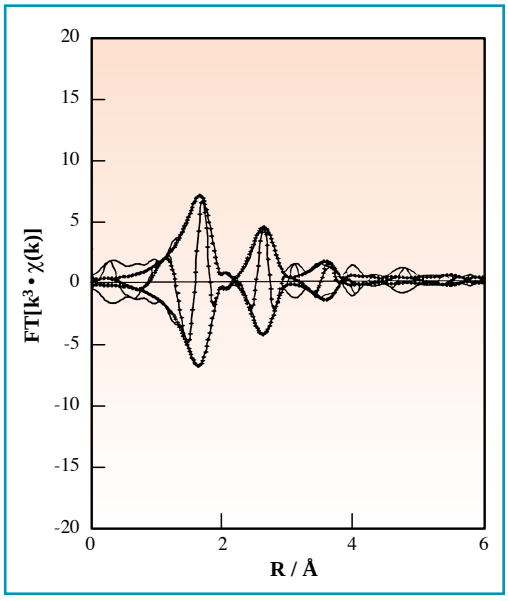

Fig. 2: Fourier-transformed k^3-weighed EXAFS function. This radial distribution function reveals the number of atoms located at distance R from absorber atom (coordination number); the distance displayed is about 0.2 - 0.5 Å shorter than the actual distances due to a phase factor.

yields information about the shape and sizes of catalytic particles adsorbed on surfaces as well as on the interaction between particle and support surfaces. Prins and his group (ETH-Z) have recently characterized Hydride Treatment Catalysts (HTC), using EXAFS amongst other methods. The salient features of these catalysts are their high specific nickel surface area, together with their high reducibility. This uncommon combination of high nickel dispersion with low (but effective) interaction of metal with support raised questions as to how the Al_2O_3 support stabilizes the metal particles. Absorption spectra were collected in the oxidic, reduced and passivated states. Figure 1 and Figure 2 show the results obtained for the oxidic state. The Ni cations in the NiO particles are surrounded by 6 oxygen anions as in bulk NiO; the Ni-O distance (2.04 Å) inferred is close to the distance found in bulk NiO (2.09 Å). As Ni loading of HTC was reduced, the coordination number due to the Ni-Ni contribution decreased dramatically. This small coordination number for Ni-Ni suggests that the NiO particles on the Al_2O_3 support are small. From a comparison with various NiO particle models of varying size and configuration (ranging from Ni (111) layer to cubic NiO (100) particles) it was concluded that the NiO particles in the oxidic catalysts were built up of successive (111) layers. This shape of the NiO particles implies that they interact strongly with their Al_2O_3 support. This was confirmed by the observation of a Ni-Al contribution at low Ni loading. A possible model for such a particle is shown in Figure 3. ■

SOME HISTORY

The Swiss-Norwegian collaboration was born at a workshop hosted by the ESRF in Frankfurt, Germany, in February 1989. On this occasion, in a hallside encounter, C. Riekel (ESRF) suggested to H.-P. Weber, University of Lausanne (UNIL), and F. Mo, University of Trondheim (UNIT), that they pool their resources and construct a bi-national beamline at the ESRF.
On the Norwegian side, this idea was quickly given form by F. Mo and D. Nicholson in a proposal that involved additional commitment from UNIT to basic and applied research in physics, chemistry and materials science. The then Rector, Prof. R. Lenschow, and Director H. Skaar enthusiastically supported the proposal and submitted it for approval to the Collegium of the University. On 5 February 1990, UNIT, as the first institution, allocated 2 mill. NOK towards the construction of the Swiss-Norwegian beamline.
The Swiss side (next to H.-P. Weber, G. Chapuis and M. Fehlmann) was at first surprised by the alacrity with which their partners-to-be had converted a good yet vague idea into a fully-fledged project. However, when the Swiss counterparts (at first UNIL and the Swiss Federal Institute of Technology in Zurich) realized how modest the beam time fraction, to which Switzerland, as a full member of the ESRF, was entitled, they quickly moved into action and started raising funds. The ensuing Swiss-Norwegian Beam Lines Consortium was approved and fully funded by the respective national research councils and universities by the autumn of 1990. The universities of Oslo, Berne, Geneva, Stavanger College and the Swiss pharmaceutical industry (Hoffmann-LaRoche, Novartis) joined as full consortial members at a later date.

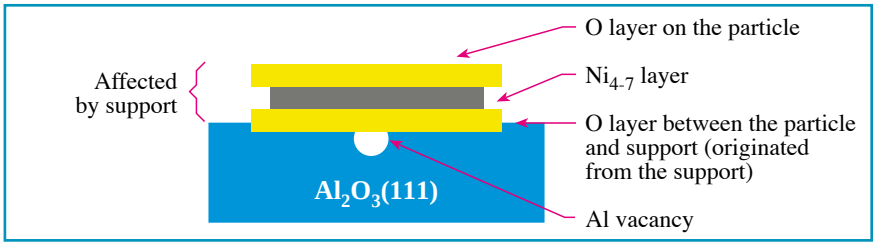

Fig. 3: Possible structure of the NiO particles.

D2AM (French Beamline) on BM2: Protein Crystallography and Materials Science

From left to right: P. Carpentier, M. Pirrocchi, S. Arnaud, J.F. Berar, B. Caillot, J.P. Simon and M. Roth.

The D2AM beamline has been operated up to now as a «double» beamline, dedicated to two different communities, who exchanged instruments around every 6 weeks: on the one hand protein crystallography using the multiwavelength anomalous method and, on the other hand, materials science using diffuse anomalous scattering at small and wide angles.

PROTEIN CRYSTALLOGRAPHY

Numerous structures have been quickly solved using data collected with the protein crystallography instrument or with the help of these data. The beam intensity is very high, D2AM being one of the most intense European beamlines for this application apart from the ESRF undulators, ID2, ID9 or ID14. But since the beam comes from a bending magnet, high intensity is obtained from beam focusing. Therefore the beamline is not very well suited for experiments with crystals having unit cell parameters above 300 Å, as they require very low divergence. A MSC™ cryogenic system is attached to the goniometer; it allows one to work at temperatures of – 170 °C on crystals obtained by flash *in situ* cooling or on pre-cooled crystals: very few protein crystals can be irradiated at room temperature without damage. The x-ray image intensifier with CCD detector gives high quality data but the exposure time cannot be increased over 100 s; typical exposure times are between 5 and 60 s according to crystal size and quality with a dead time of 15 to 20 s between images.

New and original research carried out on the beamline is concerned with the study of the structure of the anomalous diffraction near an absorption edge with protein to use numerous data taken at wavelengths near an absorption edge. This implies direct relative scaling of all the data sets, which gives as a secondary result a measurement of the crystal fluorescence. This should be a very powerful phasing method, but it requires a very large amount of data, as at least 12 sets at different wavelengths have to be collected [1, 2]. The beamline has also been used for mosaicity studies; a specific software has been developed; these measurements have been used to analyse the advantage of growing crystals in microgravity [3].

MATERIALS SCIENCE

The materials science group extends its activity over a wider domain. One part is concerned by small-angle scattering, the other with crystallographic activities. The instruments were built not only for anomalous scattering but also to improve the resolution and the dynamic range available on laboratory instruments. This goal has been reached. The following report does not represent the whole activity but gives some light on specific themes. D2AM initiated DAFS spectroscopy at the ESRF and has transformed it into a technique accessible to outside users: applied to mutilayers, this site-resolved spectroscopy allows the measure of the inter-diffusion between layers. Phason disorder has been studied in AlPdMn quasicrystals, diffuse scattering was recorded over more than 8 orders of magnitude; the results can be well reproduced by the elasticity theory of quasicrystal, considering only long wavelength phason fluctuation [4-6]. The mechanical properties of superalloys are related to a large volume fraction of an ordered phase that is coherent with the disordered fcc matrix. Their processes are linked to the kinetics of systems far from equilibrium. The NiCrAl system appears as a model alloy: the first states after quench from a homogeneous alloy have been studied both by small-angle and wide-angle anomalous scattering. In this ternary alloy, the first overall ordering is a high Cr content located on Al sites; during growth of precipitates, Cr is replaced by Al. Meanwhile, the composition of the matrix is fairly

constant. This behavior should be interpreted as a nucleation of the disordered phase after preliminary ordering of the whole, and gives an experimental answer to the question: «what occurs first: ordering or phase separation?». The relation to models is, however, less straightforward: theoretical models have been confined to binary systems.

Studies of dynamics around a phase transition have also been performed, showing how alloys come back to their thermodynamic equilibrium; this kind of experiment requires both a very high resolution and a high intensity. An example is given hereafter.

M. Roth, J.F. Berar and J.P. Simon

SOME HISTORY

The building of the French CRG beamlines, D2AM and IF, started simultaneously in 1990 with the first ESRF beamlines. They were established with the participation of the Rhône-Alpes region, the Isère Department and the «Ministry of National Education and Scientifc Research». Both the CEA and the CNRS are in charge of these beamlines, providing their financial support and manpower for construction and exploitation.

The D2AM beamline is the result of two independent initiatives that converged in their technical requirements. M. Roth (LCCP/IBS) required an instrument dedicated to the collection of three-dimensional atomic structure data of protein crystals using the multiwavelength anomalous diffraction method: this implies frequent wavelength changes as well as high intensity on the sample. J.P. Simon (LTPCM/INPG-CNRS) and a group of materials science laboratories from Grenoble and Orsay were interested in investigating the diffuse anomalous scattering at small and wide angles. They needed a more versatile instrument but with demanding optics requirements: both the harmonic purity and the signal-to-noise ratio had to be improved. This explains why two communities have shared D2AM. However, the situation will be improved in 1998 when the new French FIP-CRG beamline will be completed.

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[6] *ESRF-Highlights 96/97, p. 38-39.*

THE HIGH TEMPERATURE PROJECT

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2 LURE, ORSAY (FRANCE)

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4 LABO. DE CRISTALLOGRAPHIE, GRENOBLE (FRANCE)

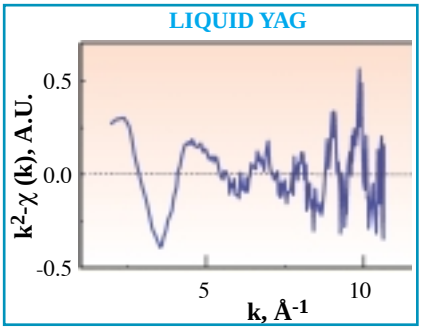
First containerless characterization using combined XAFS and XRD on refractory oxides at very high temperature are reported. We have performed *in situ* experiments at the D2AM beamline on solid and liquid refractory oxides by using a laser heating system and aerodynamic levitation. This paper describes the experimental device and presents some of the first results recorded during dedicated shifts.

A two crystal monochromator – Si(111) in our experiments – provides a monochromatic x-ray beam in the 5-25 keV energy range. The beam is focused to a size of 0.5 x 0.5 mm^2 by bent mirrors and by the second crystal of the monochromator. The wavelength can be continuously adjusted as needed in DAFS experiments, which makes it possible to record absorption and diffraction data in exactly the same sample conditions. We have developed a set-up based on laser heating and aerodynamic levitation in order to gain an insight of materials at very high temperature. The aim of the experiment is to investigate the structural properties, especially short and long-range order, in partially disordered specimens. The analysis chamber, which has been designed for working on the 7-cradle goniometer of the D2AM station, consists in an isolated cell performing *in situ* experiments combining x-ray absorption and diffraction. Up to now, three teams had used levitation (electromagnetic, aerodynamic or acoustic) associated with suitable heating (electromagnetic, laser ir-radiation) [1-3], but no one has combined two or more among these x-ray spectroscopies. Our first measurements at the ESRF have been carried out in November 1997. We studied the structure of some solid and liquid refractory oxides at temperatures up to 2750 °C: ZrO_2, melting temperature T_m = 2715 °C; Y_2O_3, T_m = 2440 °C; YAG, yttrium aluminum garnet, T_m = 1945 °C; Gd_2O_3,, T_m = 2440 °C; Ho_2O_3, T_m= 2420 °C and Er_2O_3, T_m = 2420 °C.

The levitation of the spherical sample is performed with a gas jet (argon or helium) to steadily bear a spherical sample in an energy well. A convergent-divergent diffuser creates a stable vortex ring that traps the sample. This levitator allows the sample to float in a central position independently of any contact. A similar system has been used for shaping the spherical sample from compact powders that have been used in x-ray characterization experiments. The heating is obtained by irradiation of the sample by a continuous wave (cw) 100 W CO_2 laser (SYNRAD). For security reasons, the invisible CO_2 laser beam was displayed by a red He-Ne laser beam. Our device has been configured in order to provide a large open solid angle for the photodiodes used as detectors for XAFS experiments in fluorescence mode.

X-ray absorption spectra have been recorded in the fluorescence mode with large area silicon photodiodes (Hamamatsu). The position of the detectors has been adjusted close to the sample in order to achieve an optimum fluorescence signal emitted by the sample. The photodiode supports were water-cooled. Beryllium windows have been used to transmit x-rays and to absorb visible light. Figure 1 illustrates

Fig. 1: $k^2\chi(k)$ EXAFS oscillations at the yttrium K-edge for a liquid drop of YAG (yttrium aluminum garnet). Oscillations above k = 8 $Å^{-1}$ are the signature of the Y-Y bonds.



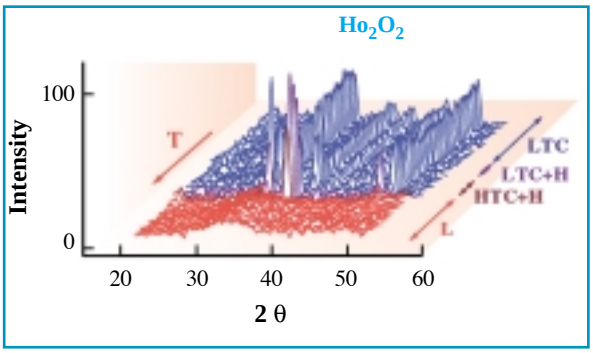


Fig. 2: 50 successive real-time diffraction patterns recorded during the cooling of a drop of liquid Ho_2O_3. The record time is 50 ms per diagram. L = liquid, HTC = high-temperature cubic phase, LTC = low-temperature cubic phase, H = hexagonal phase.

the $k^2\chi(\kappa)$ function of the yttrium K-edge recorded at 2200 °C above melting of an yttrium aluminum garnet (YAG) sample. The signal-to-noise ratio seems to be sufficiently high to exhibit the EXAFS oscillations above k = 8 $Å^{-1}$ characteristic of the yttrium-yttrium bond.

In order to perform time-resolved diffraction measurements during heating and cooling, we used a Position Sensitive Detector (PSD) developed by J.-F. Berar [4]. A gas (argon) detector is used with an angular aperture of 30° (angular resolution in 2θ of 0.1°) located at a distance of 130 mm of the spherical sample. The measurement of the position of a photon results from the determination of the delay time between two signals emitted by the incoming photon. The 50 diffraction patterns presented in Figure 2 have been recorded during the cooling time of 2.5 s (50 ms per diagram) of a liquid drop of Ho_2O_3, showing the solidification of the drop. From the diagram, we suggest the following phase transitions: the first, towards a mixing of high-temperature cubic and hexagonal phases, the second towards a mixing of low-temperature cubic and hexagonal phases and the last, towards a low-temperature cubic phase.

We have also recorded diffusion spectra on various liquid oxides with a scintillator coupled to a photomultiplier. The spectra were recorded on an angular range of 120° giving complementary information to the pair function data calculated by EXAFS analysis. The interest of measurement of x-ray diffusion oscillations is due to the reliability of the information obtained at low k values.

We can safely conclude through these first encouraging results that gas levitation associated to laser heating is a potential tool for contactless processing of refractory materials with particular properties controlled by *in situ* synchrotron analysis at high temperature. The real-time characterization by diffraction allows the follow-up of phase transition phenomena as rapid as 50 ms.

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ACKNOWLEDGEMENTS

The contribution of D. Ruffier and Y. Auger in the construction of the analysis cell is highly acknowledged. We would like to gratefully thank the ESRF staff. This work was financially supported by a contract «XI ème plan Etat / Région-Centre».

GRAAL on BM7: Gamma-Ray Beam

GRAAL does not exploit synchrotron radiation but utilizes gamma-rays produced by Compton backscattering of photons from the circulating electrons, for studies in nuclear and particle physics. As such, it is not a typical CRG beamline, for example no beam time is made available to general ESRF users.

Atoms, molecules and more complex structures are studied almost uniquely by the long-range electromagnetic interactions, while the much smaller nuclei and elementary particles have been investigated mostly by the short-range strong and weak interactions. In fact, if the strong, hadronic interaction yields the largest cross-sections and therefore the highest event rate, photon-induced reactions have several advantages, and high-energy gamma beams can be good probes of nuclei.

The first polarised and tagged gamma-ray beam was obtained by the backward Compton scattering of laser light against high-energy electrons circulating in a storage ring (Adone, in Frascati). After this successful start, other similar beams entered into operation at Novosibirsk and Brookhaven. When the ESRF was originally proposed, it was evident that its high energy and low emittance would make it the best machine to produce such a beam and therefore a proposal was immediately submitted and included in all ESRF presentations. It was named Graal.

The Graal experiment is now in full operation. It produces a gamma-ray beam with a maximum energy of 1470 MeV and an intensity of about 10^6 photons per second. It has a 98% linear polarisation at the maximum energy and a resolution of 16 MeV fwhm.
The main parts devoted to the production and monitoring of the beam are:

- a laser electron interaction region situated in one of the short straight sections of the storage ring;
- a tagging system, to measure the position and therefore the energy of the electrons which have interacted with the laser photons. It is located at the exit of the storage ring dipole which follows the interaction region;
- a vacuum system to connect the machine vacuum pipe to the cabin where the laser is located;
- a laser cabin with the optical bench which supports the laser and its optics;
- an adjustable lead collimator, a clearing magnet and a second, fixed, lead collimator to collimate the gamma-ray beam and clear it of the background of undesired photons and electrons;
- a liquid hydrogen (or deuterium) target;
- three intensity, energy and position monitors for the gamma-ray beam.

The detector consists of:

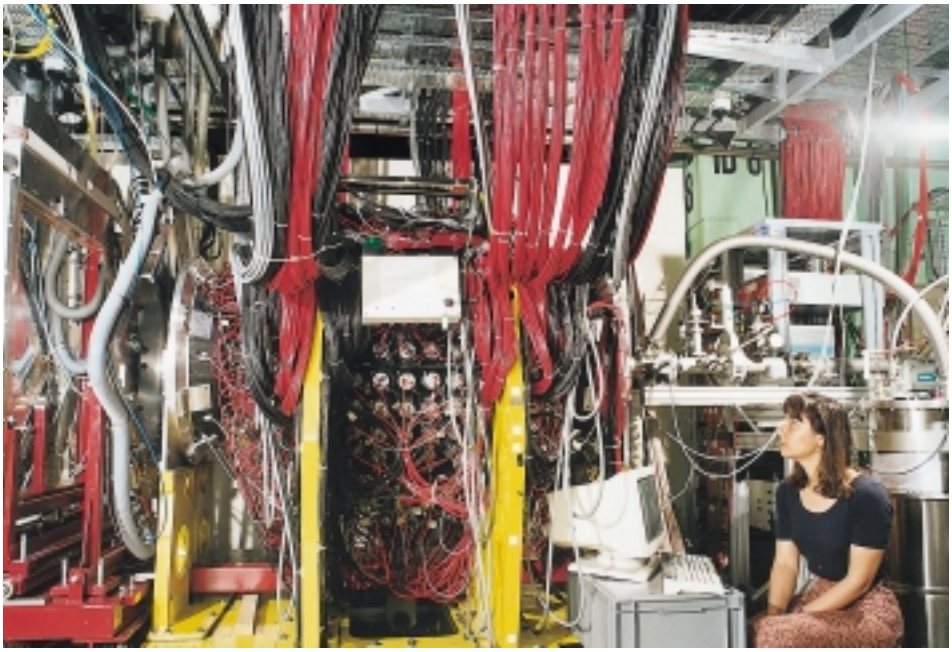
- a BGO crystal ball made of 480 BGO crystals for the detection of high-energy gamma-rays and medium-energy protons; it covers a polar angle between 25° and 155°;
- two cylindrical wire chambers located around the target, inside the crystal ball, followed by a barrel made of 32 plastic scintillators. The wire chambers and the barrel are used to measure the angles and the ionization of the charged particles entering the BGO;
- two plane wire chambers and three walls of plastic scintillators to detect particles emitted in the forward direction, at $\theta < 25°$.

This very complicated apparatus requires various expertises: storage ring physics, laser optics, gamma-ray beam handling, cryogenic targets, organic and inorganic scintillator detectors and wire chambers, plus fast electronic, data acquisition electronics, data handling, etc. This expertise is provided by a large international collaboration which includes about 50 scientists from Italy, France, Russia and the USA.

The beam polarization asymmetries have been measured for the photo-production of π°, π^+, 2 π° and η. A more detailed report on these measurements will be given in a future issue of the ESRF Newsletter.

C. Schaerf
and D. Rebreyend

The BGO crystal ball surrounding the cryogenic target on Graal.



GILDA (Italian Beamline) on BM8

1 CNR Roma
2 INFM-Genova
3 University of Parma
4 INFN Frascati
5 University of Trento
6 University of Roma «Tor Vergata»
7 University of Cagliari
8 CNR Trento
9 University of Roma «Roma Tre»

F. d'Acapito[1], S. Colonna[2], S. Pascarelli[2], G. Antonioli[3], A. Balerna[4], A. Bazzini[3], F. Boscherini[4], F. Campolungo[4], G. Chini[5], G. Dalba[5], G. I. Davoli[6], P. Fornasini[5], R. Graziola[4], G. Licheri[7], C. Meneghini[2], F. Rocca[8], L. Sangiorgio[4], V. Sciarra[4], V. Tullio[4] and S. Mobilio[9]

The Italian CRG beamline GILDA is financed by three major Italian public research institutes (Consiglio Nazionale delle Ricerche CNR, Istituto Nazionale per la Fisica della Materia INFM and Istituto Nazionale di Fisica Nucleare INFN). It has been operational since September 1994 and is mainly dedicated to the investigation of local structure. To this purpose, x-ray absorption spectroscopy as well as x-ray diffraction are used on the beamline in the energy range 5-50 keV.

The beamline GILDA consists of four experimental hutches. The first experimental hutch, in the 1:3 focal configuration which ensures the maximum flux, is dedicated to X-ray Absorption Spectroscopy (XAS). The wide energy range of the beamline makes GILDA well-suited for XAS investigations on heavy elements with the possibility to access the K absorption edges from Ti (Z = 22) to Eu (Z = 63). Experiments are carried out in transmission, fluorescence and total electron yield modes. For the study of highly diluted samples a 7-element high-purity Ge detector is currently used (current limit sensitivity: 10^{14} at/cm^2). Surface experiments in total reflection mode (ReflEXAFS) are performed in a dedicated experimental chamber.

The second hutch, in 1:1 focal geometry, is dedicated to x-ray scattering and diffraction. It is equipped with a two-circle diffractometer with an angular step of 0.28 arcsec and a reproducibility of 2 arcsec. Crystal analyzers, solid-state detectors and scintillators are used to perform anomalous scattering on amorphous materials, and powder diffraction with an instrumental angular resolution lower than 0.01 degree. The installation of an Image Plate detector is foreseen. Anomalous x-ray reflectivity spectra have also been collected. Finally, Diffraction Anomalous Fine Structure (DAFS) spectra have been successfully recorded: soon also this technique will become available for users.

A third experimental hutch at the end of the beamline is available for users who wish to install their own apparatus. An ultra-high vacuum chamber fully equipped for surface preparation and characterization is available for EXAFS investigations in total and partial electron yield mode.

SCIENTIFIC ACTIVITY

The scientific activity of GILDA mainly deals with the study of local structure around atoms present in trace amounts. XAS is a well-suited technique for this: among the experiments performed we recall here the investigations on InAsP/InP and InGaAs/InP superlattices, impurities in semiconductors (Er:Si, Ga:a-Ge, As:Si, Co:Si), impurities (Ag, Cu, Er, Eu, Pr) in various kinds of glasses, metallic active sites in proteins. Catalytic systems have been investigated in cells for *in situ* treatments. The 1997 ESRF-Highlights booklet reported the GILDA studies performed on the metal insulator phase transition in colossal magnetoresistance manganites as well as epitaxial overlayer of InGaAs/InP.

X-ray scattering experiments on amorphous and polycrystalline samples have been performed in the conventional and anomalous modes to obtain a description of the local and medium range order of the whole material and around a particular component. Experiments carried out in this field provided the local order around Eu and Sr in Eu-doped Sr-metaphosphate glasses, around the metallic components of FeNiB amorphous alloys and around Zr in Zr-based ceramics. Other kind of experiments performed were anomalous powder diffraction on nanocrystalline metallic clusters dispersed on a pumice substrate, anomalous x-ray reflectivity in InAsP/InP superlattices and DAFS on $ZnAl_2O_4$ and $ZnFe_2O_4$ spinels.

LOCAL ATOMIC ORDER AROUND ER IN ER-BASED LUMINESCENT MATERIALS

Er doped crystalline Si, exhibiting optical luminescence at 1.54 μm that corresponds to the absorption minimum of commercial optical fibers, represent a promising material for optoelectronical applications. The luminescence arises only from Er^{+3} ions bound to O atoms (usually present in Si as impurities) and the effect is absent in Er-doped high-purity Si. A new way to obtain a high concentration of Er^{+3} active centers consists in performing a double implantation of Er and O ions on a crystalline Si substrate. The as-implanted material undergoes then several annealing treatments to recrystallize the implanted region. Unexpectedly, the as-implanted material exhibited a barely detectable luminescence, strongly increased by the annealing process. To understand this phenomenon, an investigation of the local atomic order around the Er^{+3} ions

was performed using the Extended X-ray Absorption Fine Structure (EXAFS) technique at the Er L_{III} edge. The typical amount of Er ions in these samples was around $2*10^{15}$ Er/cm^2 (roughly 2 ML). In the as-implanted material (process A - Figure 1a) the position of the first FT peak corresponds to an Er-Si coordination with no evidence of Er-O bonds. Er and O do not react, probably due to the extremely reduced diffusion constant of Er in the Si network, and the correspondent luminescence is very low (Figure 1b). The spectra of the annealed samples (B and C) on the other hand show a peak corresponding to an Er-O bond and the luminescent yields are much stronger. Upon annealing, the crystal-amorphous boundary moves towards the surface, creating a region of high atomic mobility where the implanted species can react together and give rise to the active centers.

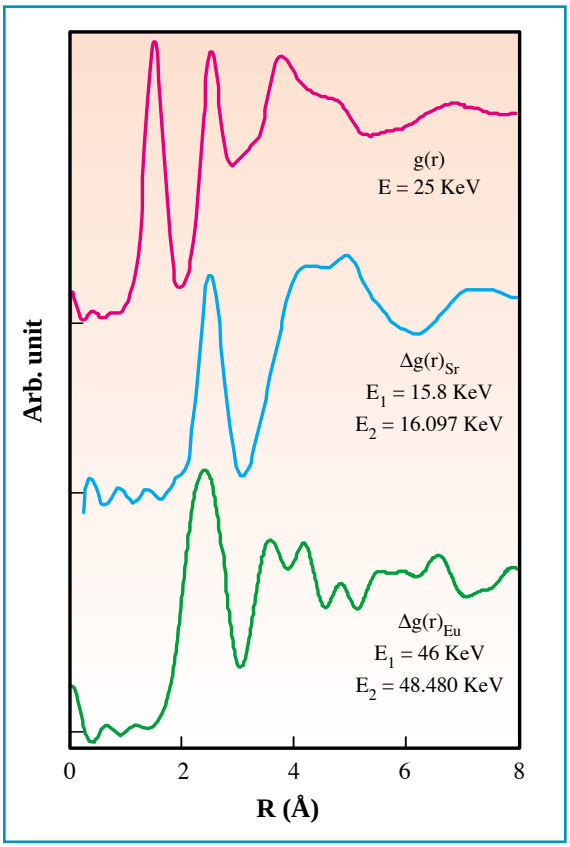


Fig 2: The upper panel reports the Total Correlation Function g(r) for $Eu_{0.1}Sr_{0.9}(PO_3)_{2.1}$ obtained at 25 keV. The middle panel shows the Differential Correlation Function Δg(r) obtained at the Sr K edge and in the lower pannel is reported the Differential Correlation Function Δg(r) obtained at the Eu K edge.

LOCAL STRUCTURE OF GA-DOPED A-GE:H THIN FILMS

One of the major issues in the physics of amorphous semiconductors is the understanding of microscopic mechanism of substitutional doping in hydrogenated amorphous Si and Ge thin films doped with group III and V elements. In this work hydrogenated amorphous Ge thin films doped with different Ga concentrations have been investigated by EXAFS spectroscopy. The films were prepared by rf sputtering from a c-Ge target, dopants were added using a co-sputtering technique. The typical thickness of the films was around 3 μm, with a Ga amount ranging between 5 x 10^{14} and 1.3 x 10^{17} at/cm^2. Data were collected in fluorescence mode. From the quantitative analysis, the coordination number of Ga at the lower concentration was found to be 4, in

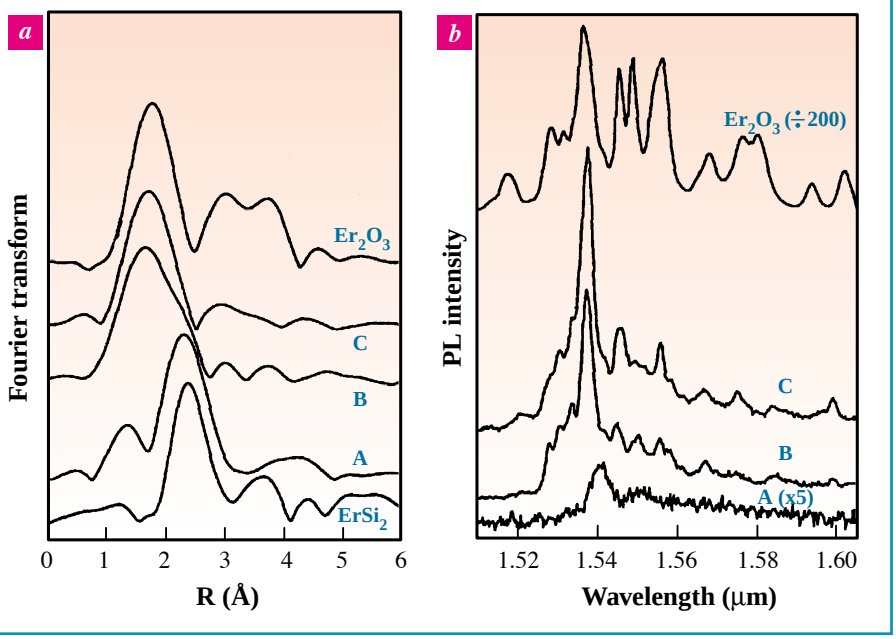


Fig 1: a) Fourier transforms of the EXAFS spectra compared with model compounds for the Er-Si bond ($ErSi_2$ crystal, peak at ~2.5 Å) and the Er-O bond (Er_2O_3 crystal peak at ~2 Å). b) High resolution photoluminescence spectra at 15 K with a 200 mW laser pump. Capital letters indicate the thermal processes: A = 450°C-30min, B = A+620 °C-180min, C = B+900 °C-30s.

agreement with the hypothesis of substitutional site of the dopant in the Ge tetrahedral network. A sharp decrease in the mean coordination number from 4 to 2.8 occurs when the dopant concentration rises from 4.5 x 10^{15} to 4.5 x 10^{16} at/cm^2 suggesting a Ga site modification at high concentration.

SR AND EU DOPED METAPHOSOPHATE GLASSES

Anomalous Wide Angle X-ray Scattering (AWAXS) and extended x-ray absorption fine structure (EXAFS) have been used to investigate the structure of Eu doped Sr metaphosphate glass (Figure 2). In order to investigate both the local structure around Eu and the modifications of metaphosphate network induced by doping two glass samples, a-$Sr(PO_3)_2$ and a-$Eu_{0.1}Sr_{0.9}(PO_3)_{2.1}$, were studied at the Sr (16.107 keV) and Eu (48.517 keV) K edges. The resulting model of the host Sr-metaphosphate matrix is consistent with a description of the structure in terms of Sr^{2+} ions interposed between PO_4 chains. All distances and coordination numbers related to a distribution of PO_4 tetraedra, interconnected via two bridging oxygens, fall within the expected results. However, the location of strontium is more problematic, and the coordination number $N_{Sr-O} \sim 5$ suggests an irregular distribution of its first coordination shell or an equal number of four-fold and six-fold coordinated Sr atoms. The data obtained on both samples demonstrated that the host metaphosphate matrix is unchanged by doping. The determination of the local structure around Eu by AWAXS at the Eu K edge has been a special issue of this experiment as it is the first attempt to perform an AWAXS experiment at such energies. The choice of one of the three Eu-L edges is also possible to detect the Eu environment, but several problems arise in this case: for the low energy, the reciprocal-space extension of the data is not enough to allow a good resolution of the Eu coordination shells. Moreover, the three L absorption edges are very close each other, making the measurements more difficult for the lower contrast between the scattering factors of Eu and high fluorescences emission. AWAXS results demonstrated an asymmetric environment around Eu with 7.5 O at 2.32Å and 1.5 at 2.62 Å.

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ACKNOWLEDGEMENTS

We are grateful to F. Berti, A. Gasparoli, D. Gasperetti, P. Gennara, P. Recchia, A. Rossi and G. Silvestrin for technical support in the construction and commissioning of the x-ray absorption set-up.

ROBL (German Beamline) on BM20: Structural and Radiochemical Investigations

W. Matz, N. Schell, H. Funke and G. Bernhard

The beamline is located at the bending magnet BM20.
The two experimental stations, operating alternatively, are designed for radiochemical investigations with x-ray absorption spectroscopy on the one hand and structural investigations of materials by x-ray diffraction and reflectivity on the other hand.

To fulfil its purpose, besides the optics hutch and a small beamline workshop, ROBL has two experimental stations: the radiochemistry hutch (RCH) and the materials research hutch (MRH). The RCH is built as a radiochemical laboratory of type B to perform experiments with radioactive samples having an activity up to 185 MBq (5 mCi). Such a dedicated installation is unique in the world at a synchrotron radiation source.

THE RADIOCHEMISTRY HUTCH (RCH)

The Institute of Radiochemistry is interested in research for risk assessment and remediation of radionuclide contaminations related to uranium ore mining and milling, of environmental contamination resulting from world-wide nuclear weapon production activities and nuclear accidents. Furthermore, it is interested in research for risk assessment of potential nuclear waste repositories. A basic molecular-level understanding is required to quantitatively describe the mechanisms of radionuclide transport in the environment. XAS is a powerful technique to study speciation and complexation of radionuclides in solutions, adsorption processes at the solid-water interface of radionuclide solutions with geological and biological materials.

The radiochemistry hutch (RCH) is designed to handle radioactive samples (solid or liquid) with an activity of up to 185 MBq (5 mCi). The samples contain the actinides from Th to Am as well as Ra, Po and Tc.

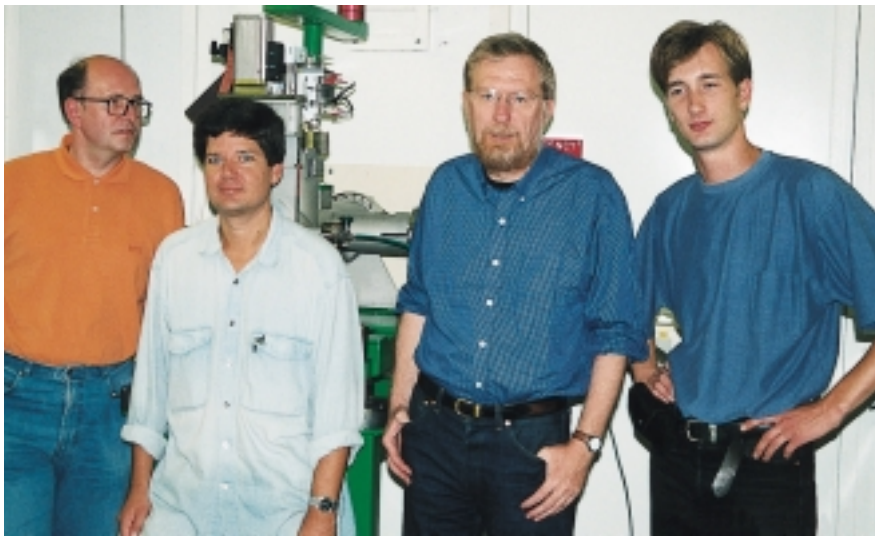
The main experimental method will be x-ray absorption spectroscopy (XANES and EXAFS) in both the transmission- and the fluorescence mode (for diluted samples). A closed cycle He cryostat allows for studies in the temperature range of 4-300 K. Quick EXAFS for time-dependent studies and microfocus x-ray absorption spectroscopy for spatially-resolved studies are planned. Different detection systems (ion chambers of different lengths, Ge solid state fluorescence detectors, fluorescent x-ray ion chamber detectors) will be available.

All radioactive samples will be delivered, stored and handled according to the safety regulations agreed upon with the ESRF. Only specially trained staff will be allowed to operate this experimental station. However, our expertise and help can be made available to collaborators.

Staff situated in Rossendorf from left to right: S. Dienel, D. Pröhl, W. Oehme, R. Schlenk, W. Matz, P. Reichel, M. Betzl, M.A. Denecke, V. Brendler, G. Hüttig, F. Eichhorn, G. Bernhard, T. Reich, J. Claußner.

SOME HISTORY

In the beginning of the year 1995 the Forschungszentrum Rossendorf (FZR) near Dresden, Saxony, signed a contract with the ESRF to establish the German CRG ROBL in order to enhance the research capabilities of the biggest research institution of the new Länder in reunified Germany. Building and running of ROBL will be carried out by close co-operation of the Institute of Radiochemistry and the Institute of Ion Beam Physics and Materials Research as well as the Central Department of Research and Information Technology of the FZR. Since May 1996 a small permanent staff is residing at the ESRF to supervise and contribute to the building progress. The construction phase in the ESRF experimental hall started in May 1996.
Tests and preliminary experiments by FZR groups started early in 1998. The start of scheduled user operation of ROBL is planned for September 1998.



Local staff of ROBL at the ESRF with project leader W. Matz (left): N. Schell (Materials Research), H. Funke (Radiochemistry) and U. Strauch (technical service).

THE MATERIALS RESEARCH HUTCH (MRH)

The Institute of Ion Beam Physics and Materials Research will use ROBL within its scientific program for the identification and characterization of the modifications of surfaces, interfaces and near surface layers including phase formation produced by ion beam techniques. Research topics of the institute are hard coatings, nanoclusters, biocompatible materials and advanced semiconductors.

In collaboration with universities and other research institutes from the region structural investigations of melts, amorphous solids and phases in metal and semiconductor nanometer-multilayers as well as the analysis of the real structure of single crystallites or texture are planned. But also other users are invited to perform experiments in MRH in collaboration with the FZR.
The basic equipment in MRH will consist of a high-precision 6-circle Huber goniometer, several detectors (scintillator, energy sensitive photodiode, two-dimensional PSD of CCD-type) and a high-temperature chamber up to 2000 °C. ■

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[1] More information is available under http://www.fz-rossendorf.de. Postal address: Forschungszentrum Rossendorf, P.O. Box 510119, D – 01314 Dresden.
[2] ESRF Beamline Handbook; also at http://esrf.fr/exp_facilities/BLHB.htm.
[3] Contact ESRF residents of ROBL at robl@esrf.fr.

ACKNOWLEDGEMENTS

We would like to thank the ESRF for their support in beamline planning and installation matters, and the ILL for agreeing to the temporary storage of future radioactive samples.

SPLINE (SPANISH BEAMLINE) ON BM25

At its July 1997 meeting, the ESRF Council has approved the construction of the Spanish CRG beamline (Spline) financed by the CICYT (Comisión Interministerial de Ciencias y Tecnología). The main goal of the Spanish CRG x-ray beamline is to satisfy the needs of the Spanish scientific community and give it access to a third generation synchrotron radiation facility to perform x-ray absorption and diffraction experiments in a broad energy range.

The Spanish beamline will be split into two lines: one branch will be allocated on the soft edge (A) and the other on the hard edge (B) of the BM25 bending magnet.

BRANCH A:

- High-resolution powder diffraction (HRPD) including anomalous dispersion.
- X-ray absorption spectroscopy (XAS) and x-ray standing waves (XSW).

BRANCH B:

- Macromolecular crystallography including MAD
- Single crystal diffraction and diffraction from interfaces
- X-ray diffraction/scattering camera for non-crystalline specimens. ■

The hutches are due to be constructed in January 1999. It is intended that the first station will be ready for user operation in January 2001. Full operation will start in the second half of 2002.

More in ESRF Beamline Handbook; also at http://esrf.fr/exp_facilities/BLHB.htm

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DUBBLE (Dutch-Belgian Beamline) on BM26

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With support from
I. Cerjak, D. Glastra van Loon, P. Lassing, B. Munneke, J. Derks, M. Konijnenburg,
M. Borsboom, H. Alberda, R. Bakker, F. Udo
and several other technicians, engineers and scientists

Although neither Belgium nor the Netherlands have a national facility for the use of synchrotron radiation, researchers from these countries have been using synchrotron radiation as a tool for a long time. Lure, Desy and Daresbury in particular have regularly been hosts for scientists from these countries. The Netherlands Research Council (NWO) has been an active promoter of the use of synchrotron radiation notably through its formal involvement in Daresbury where two beamlines were constructed and several other Dutch/English projects received financial support. For over ten years NWO-funded scientists were part of the staff in Daresbury. When the CRG program at the ESRF was approved attention shifted towards Grenoble. After extensive consultations with possible users a consortium was formed consisting of NWO, the universities of Utrecht and Amsterdam and AMOLF from the Netherlands, and NFWO and the universities of Leuven, Antwerp, Gent and Brussels in Belgium. The decision was made to build two beamlines, which after the decision from the ESRF to support the development of the wide 9 mrad front-ends, will be equipped with full focusing optics on both beamlines. The experimental techniques that will be implemented are interface diffraction, powder diffraction EXAFS and time-resolved simultaneous SAXS/WAXS. This reflects the need of a large part of the Dutch and Belgian user community. The DUBBLE project will considerably enhance the number of shifts available to Dutch and Belgian users at the ESRF.

The design and construction is done mainly at the Amsterdam-based institutes AMOLF and NIKHEF. NIKHEF is the Dutch high-energy physics institute which had already been involved in the construction of synchrotron radiation beamlines. A team of four scientists and two technicians is responsible for the building and commissioning of the beamlines but there is generous support from AMOLF and NIKHEF so that the expertise and effort from several engineering disciplines is available. The collaboration between the high-energy physics and synchrotron radiation experts is quite unique, especially in the collaboration on position-sensitive detectors. Much is expected of this.

Besides the optics hutch there are two experimental hutches. The first will house the EXAFS and powder diffraction equipment, and the second the interface diffraction and SAXS/WAXS. The latter hutch is rather large in order to house the long optical bench for SAXS and to be able to mount large sample environments used for instance in polymer processing but also to house the large six-circle interface diffractometer.

The project is, after some slight delay, in an advanced state and it is expected that the first technique, time-resolved SAXS/WAXS, will become operational in the first half of 1998, followed by interface diffraction shortly afterwards. Later in 1998 the construction of the EXAFS and powder diffraction stations will start. This will be completely finished in 1999. At the moment two staff members are permanently based in Grenoble. This number will increase to five in 1998 when more equipment, assembled in Amsterdam, is ready to be mounted on the beamlines. It is also foreseen that more universities will join the consortium in the coming years.

XMAS (UK Beamline) on BM28: X-ray Magnetic and High-Resolution Scattering

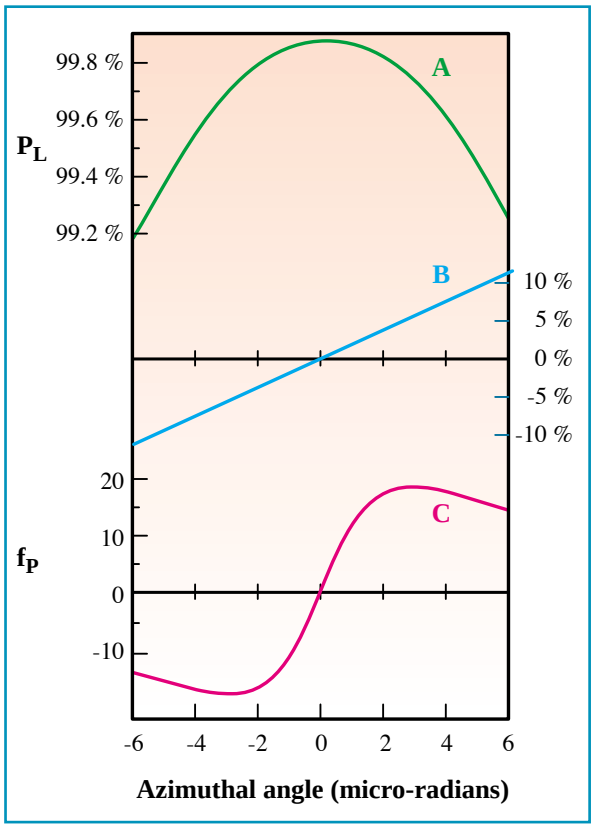
This project was conceived back in 1990 when M. Cooper, B. Stirling and G. Stirling (no relation) were emptying a bottle of wine at a Daresbury Users' Meeting. Three years were spent in refining and revising an application to the UK Science Research Council (EPSRC); the budget grew by a factor of five from our first naive estimate. The proposal was eventually accepted by EPSRC and funds allocated for construction of the beamline in 1994. It was not until January 1996 that the first hutch was erected on the floor in front of dipole D28 and the construction began in earnest. A grant to run it was thankfully forthcoming from the EPSRC earlier this year and on 19 September the transition to the new funding was marked by further emptying of wine bottles. As is usual in these circumstances, the «opened» beamline is still being commissioned and we expect to take our first really independent users in Spring 1998.

The acronym XMaS (**X**-ray **Ma**gnetic **S**cattering) was invented by B. Forsyth, but if you think it is corny take a moment to savour those of some of our continental European colleagues (have Dubble never heard of *double Dutch?*). Our objective is to focus a few areas of physics that can peacefully co-exist on a single beamline. It is designed primarily for single-crystal diffraction studies, especially magnetic diffraction studies, but also other forms of high-resolution single crystal diffraction, white beam studies and powder diffraction will be possible. The UK scientific community waiting to use it is big enough to justify the expense but small enough to ensure that users can expect to win sufficient

time to sustain their research programs. The beamline has some features that are different from other dipole lines at the ESRF. First, it is aligned on the soft end of the dipole in order to maximize the flux that we get at 3-4 keV. This is specifically in response to our interest in the magnetic structure of the actinides and transuranic materials whose compounds have fascinating magnetic properties: their M-edges are at these low energies.

The beamline is also a white beam station because the only viable method developed to date for measuring the magnetic form factors of ferromagnets is based on the use of white beam. In fact as part of the commissioning exercise we have characterized the polarization of the source in a white beam diffraction study of the magnetic form factor of nickel. It is the angular distribution of the electron trajectories at the source point that governs the beam polarization. Our study showed that the linear polarization, P_L, of the beam from the dipole source D28 is 99.85 ± 0.01% at 10 keV; the corresponding figures at 5 and 15 keV are 99.88 and 99.83, respectively. The 10 keV result, as a function of the angle out of the orbital plane at which the source is viewed, is shown in Figure 1 together with the circular polarization, P_C, and the factor $f_P = P_C/(1-P_L)$, which «magnifies» the ratio of magnetic to charge scattering in the white beam method (see for example, Collins *et al*, Phil. Mag. **B65**, 37 1992). The high degree of polarization helped us to measure magnetic form factors out to the (18,0,0) reflection (Laundy et al: in print Journal of Synchrotron Radiation).

Fig. 1: Curve A is the linear polarisation P_L calculated from measurements of Bragg peaks observed at a scattering angle of 90° in the horizontal plane at the XMaS beamline as a function of the azimuthal angle. The degree of circular polarisation, P_C, is shown in curve B and f_P (= $P_C/(1-P_L)$) is the factor that enhances the ratio of magnetic to charge scattering. The benefit of a highly polarised source is self evident and the curve shows that an angle of 2-3 microradians is optimum for the white beam experiment.



The subjects identified for study by the CRG's user group (there are over a dozen groups in the UK who plan to use the XMaS facility) fall into two broad categories. First, magnetic materials: the magnetic structure of lanthanide and actinide metals, compounds and multi-layers using resonant exchange scattering, and their magnetic and structural phase transitions will be investigated. Second, non-magnetic materials: lattice distortions in bulk material and real (i.e. non-UHV) surfaces and interfaces.

Examples from commissioning experiments are shown below. Figure 2 presents the intensity of the (0,0,2+q) magnetic satellite of holmium as a function of energy at the L_{III} resonance. The resonant enhancement at the M_{IV} edge of uranium is exemplified by Figure 3 for a mixed UAs/USe single crystal, just below its Neel temperature. The beamline appears to be working according to specification.

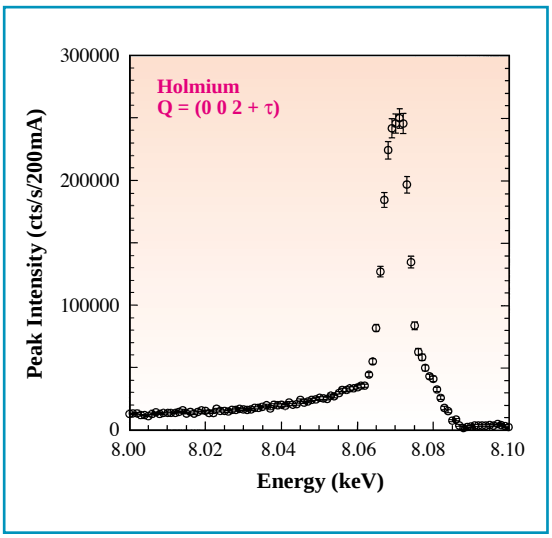
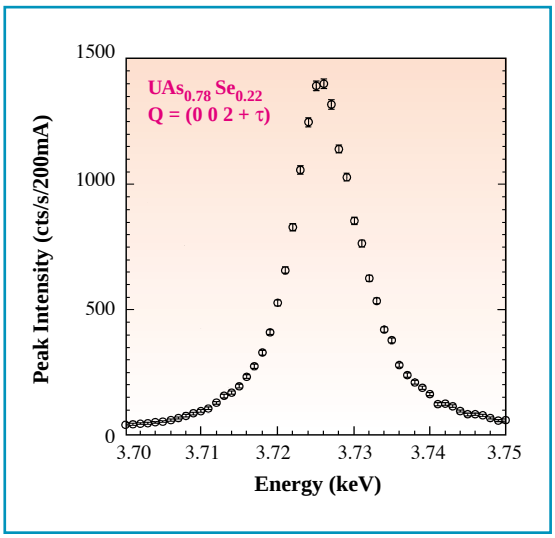


Fig. 2: Resonant enhancement at the L_{III} absorption edge of holmium.

Fig. 3: The uranium M_{IV} resonance for a UAs/USe sample, just below T_N.



FIP (French Beamline)on BM30: Investigation of Protein Structures

FIP is a French CRG beamline under construction at the BM30 front-end. It will be dedicated exclusively to biological macromolecular crystallography. FIP will replace the biocrystallography station at the CRG beamline D2AM, where it used only half of the available beam time, sharing the space with materials science experiments.

The move of biocrystallography from D2AM to FIP has started in March 1998. However FIP will come into full operation only by the end of 1998. What can be foreseen at present is the following: in September 1998, the beamline will be ready with the same equipment and similar beam characteristics as it was on D2AM, and we will start to invite selected external users for data collection. But with respect to D2AM, two new developments are prepared: 1) using cryo-cooling on the first monochromator crystal and 2) using piezoelectric actuators for bending a long U-shaped crystal. The first improvement will allow the extension of the range of accessible x-ray photon energy up to 25 keV, and the second one should make the focusing of the beam faster and more efficient by sagittal bending. New detectors will also be installed on the beamline. Next September we will start installing the cryo-cooling of crystal 1 of the monochromator and probably an image plate detection system. Later in the year, a new CCD detector will be installed replacing the XRII-CCD used on the D2AM station since 1995. As another improvement with respect to D2AM, the beamline will have its own cold room as part of the small crystal preparation laboratory located on the beamline itself.

The gain in beam time for data collection will contribute to improve the quality of the collected data in two aspects, first more time for data collection, of course, i.e. better statistics, but secondly more time for careful data collection preparation. It will also allow, hopefully, the development of two time-consuming new applications, namely 1) kinetic studies of biological reactions produced in the crystals, by collecting monochromatic data after flash-cooling of the crystals in intermediate states of reaction, and 2) the fine study of the x-ray diffraction in proteins with heavy atoms near the absorption edge, which requires collecting data at a high number of wavelengths on the absorption edge.

M. Roth

IF (French Beamline) on BM32: Interfaces

The word «interface» has a very wide meaning, and interfaces may be found in nearly everything, from materials to living beings. Hence a large number of researchers from very different scientific fields have been interested in the project from the beginning, which is illustrated by the large diversity of experiments that have been performed concerning new materials, soft condensed matter, environmental science or biology.

The beamline has been focused on two complementary techniques: Glancing x-ray diffraction, which probes the long range order, and x-ray absorption spectroscopy (XAS) which probes the short-range order. This resulted in three experimental stations, one equipped with a multi-purpose goniometer (GM), one dedicated to x-ray absorption spectroscopy (XAS) experiments, and one devoted to surface x-ray diffraction in ultra-high vacuum called SUV. The two first instruments were commissioned in 1994, and the last one in 1996. The details of the beamline and experimental stations can be found in the ESRF Beamline Handbook.

Because of its very versatile design, the GM instrument is a user-oriented facility that fits the needs of a wide community. The research on the «GM» diffractometer has been balanced between studies of « soft » and « hard » condensed matter.

In the field of soft-condensed matter, many experiments have been devoted to the study of monolayers such as amphiphilic films on liquids [1] and of free-standing liquid crystal films [2], intended to test theories on the elastic properties of membranes with full calculation of the fluctuation spectrum, from macroscopic to molecular sizes, including various kinds of interactions. Liquid crystal films supported by solid substrates like MoS_2 have also been investigated to determine the structural evolution with temperature and correlate it to the observations of atomic force microscopy, thus providing insight on the mechanisms of inter-molecular and substrate/molecule interactions. These studies open up large perspectives on the influence of dimensionality and substrate adhesion on order phase transitions in liquid-crystals. Other experiments have been devoted to the 2D transitions of alcohol layers adsorbed at the air/water interface, with a particular attention to the influence of chirality, opening the

SOME HISTORY

The project of a French CRG beamline dedicated to the study of interfaces was initiated in the framework of the French «*Programme d'accompagnement de l'ESRF*». It was supported by several laboratories from the National Center For Scientific Research (CNRS, Laboratoire de Cristallographie, Grenoble), the Atomic Energy Commission (CEA, DRFMC, Grenoble and DRECAM, Saclay) and the University of Grenoble (Spectrométrie Physique). Part of the financial support also came from the Isère department and the Rhône-Alpes region.

It was the very first CRG project to be presented - and accepted - by the ESRF, in early 1990. The IF beamline, one of the first available to users, came into operation in 1994. Experiments have been performed since, with 1/3 of the available beam time being allocated by European committees through the usual application for beam time at the ESRF, and 2/3 being allocated by a French committee through a similar procedure working at the French level.

way for studies on cholesterol. Another important subject has been the study of the growth and wetting properties of adsorbed films of rare gas, with the first direct observation of a pre-roughening transition at the origin of the surface melting, which is in turn at the origin of bulk melting [3, 4].

In the field of hard-condensed matter, numerous and diverse experiments have been performed, concerning the structure of thin layers such as semiconductors (ZnTe, CdTe, GaN) or magnetic alloys (FePd), oxide layers (ZrO_2) or metal/oxide interfaces (Ag/MgO). Surface x-ray diffraction experiments were also performed when UHV is not necessary, like on the oxidised surface of gold, thus bringing new insight to the physics of metallic oxidation. Less classical experiments such as Refl-EXAFS on $NbSe_2$ layers or resonant x-ray magnetic diffraction on ErFeB magnets were also performed.

The SUV station allows reflectivity, surface x-ray diffraction and absorption to be performed in ultra-high vacuum (UHV), either on clean surfaces or on thin films. Most experiments have consisted in studying the evolution during their growth or during anneals of the structure and morphology of thin metallic films elaborated in situ on different kinds of substrates, metallic or refractory, with a thickness varying from a fraction of a monolayer to 100 Å or more. An example is the combination of the three techniques to characterise ultra-thin metastable Pt-Co alloys formed on the Pt(111) surface by different routes, either by co-deposition of Co and Pt or by annealing of a thin Co deposit, in relation to their properties of perpendicular magnetic anisotropy which make them promising candidates for high density magneto-optic recording [5]. The growth of the analogous system, Pt on Co(0001), was also investigated at different temperatures [6], with the same objective of linking the structure and morphology to magnetic properties. Another research program is devoted to oxide surfaces and metal/oxide interfaces, which are of interest because they are involved in many applications, and because the theories to describe them are still under development. Model systems such as the surface of alumina [4] or of magnesium oxide and its interface with different metals like Ag, Pd or Ni [7, 8] have been investigated, with emphasis on the epitaxial relationships, the growth mode and the lattice parameter relaxation during the very early stages of deposition. The introduction of coherency defects such as stacking faults or interfacial misfit dislocations and their reordering for large deposits and upon annealing were characterised in detail. The ultimate goal is to better understand, and improve, the bonding between such dissimilar materials. Other oxide surfaces such as NiO(111) [5, 9], are also studied because of their interesting magnetic properties.

The XAS station is now used by a very wide community, in biology, geochemistry, environment, electrochemistry, catalysis, magnetism and material sciences. All the groups to which beam time has been attributed have been able to realise their experiments (more than 25 publication with reviewers). Among them, let us cite three examples that are connected to instrumental developments. The dynamic focusing, allowing to keep a 300 x 300 mm^2 spot size on the sample all over the EXAFS spectrum, has been successfully used in many experiments such as in the high-pressure and high-temperature investigation on liquid selenium near the critical point (390 bars, 1620 °C), where a semiconductor to metal transition occurs [10]. Recently, a total electron yield detector with He gas flow at atmospheric pressure has been developed and successfully tested [11]. It was successfully used to analyse the evolution during annealing of the nanostructure of CoxAg1-x, NixAg1-x heterogeneous alloys exhibiting giant magnetoresistance [12], as well as in an investigation of epitaxial (111) CoPt3 films aimed at understanding the microscopic origin of their perpendicular anisotropy [13].

A new germanium solid state multi-detector has recently been commissioned, which opens the way for studies of highly diluted samples down to about 100 ppm. The next paper gives an example of its use in environmental science [14].

G. Renaud

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ENVIRONMENTAL SCIENCE

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In recent years, environmental problems such as pollution of air and soil have become increasingly important, even for everyday life. For these reasons, the ESRF has been involved in studies concerning environmental science. In particular, absorption experiments are an indispensable complement to laboratory techniques and, sometimes, the only method able to bring unambiguous answers to environmental problems.

Recently, new possibilities to carry out absorption experiments on highly diluted samples have been available on beamline BM32. It is possible to study a diluted sample down to about 100 ppm, which is a very important target, allowing the study of samples which were, to date, inaccessible. The detector is quite user-friendly: it is possible in an easy way and quite rapidly to change the energy selected intervals of detection of the fluorescent photons.

Furthermore, the dynamic focusing of the x-ray beam allows a 300-300 μm^2 spot size to be kept on the sample all over the EXAFS spectrum. This permits us, firstly, to considerably enhance the intensity of incident photons on the sample, which is essential in the case of highly diluted systems, and, secondly, to study small samples even in grazing incidence.

DIRECT DETERMINATION OF THE SPECIATION OF HEAVY METALS IN SOILS BY EXAFS

The input of heavy metals in the environment, and specifically in soils and industrial or domestic urban wastes, endangers living organisms. Assessing the risk associated with their presence is a prerequisite for designing recovery techniques of contaminated sites and preventing future contamination in the case of landfills. In either case, the risk associated with the presence of heavy metals depends primarily on their *speciation*. The notion of speciation is taken in its broadest sense and includes metal characteristics, such as electronic structure (oxidation state, nature of chemical bonds) and chemical state: its association with the organic or inorganic fraction, the nature of the functional groups to which the metal is bonded, fixation by minerals (clays, oxides...) and the crystal chemical mechanism of this fixation (adsorption vs. lattice substitution), or, more simply, precipitation of the pollutant as a salt (sulfate, carbonate, phosphate...), an oxide or a silicate. This chemical state determines the intrinsic toxicity of the host matrix (soil or waste), as well as the mobility of metals in the environment to either surface and ground waters, living organisms (plants, micro-organisms, meso-fauna) or the atmosphere.

Determining the speciation of heavy metals is a difficult task because of their relatively high dilution and the structural and chemical complexity of host materials. Classical approaches are based on the «selective» or the «sequential» dissolution of soils components and can be classified as possibilities offered by EXAFS spectroscopy for determining the speciation of metals. In contrast to previous methods, EXAFS is a «direct method» since it can be applied to non-disturbed, polymetallic and polyphasic materials. The potential of the EXAFS spectroscopy method will be illustrated with the determination of the speciation of heavy metals in lichens contaminated by different sources of pollution of lead and zinc.

UPTAKE MECHANISMS FOR THE FIXATION OF HEAVY ELEMENTS BY LICHENS

Lichens are currently used as biomonitors of atmospheric and soil pollution. The absorbing capacity of lichens for different heavy metals has been widely studied, but little is known about the localisation and retention mechanisms of metals in the thallus. Three different uptake mechanisms are supposed to exist: (1) entrapment of metal-rich mineral particulates originating from atmospheric aerosols (mineral phase) (2) extracellular complexation with polysaccharides of the cell walls, oxalic acid or lichenic acids (3) intracellular uptake by complexation with cysteine-containing proteins (metallothioneins) (Figure 1).

EXAFS measurements on contaminated lichens are a difficult task because (i) metals are generally diluted (500-1000 mg/kg), and (ii) lichens may absorb a variety of metals, thus creating a multi-element system. This study has been possible thanks to the Canberra multidetector and the dynamic focusing allowing a detection limit for trace elements presently

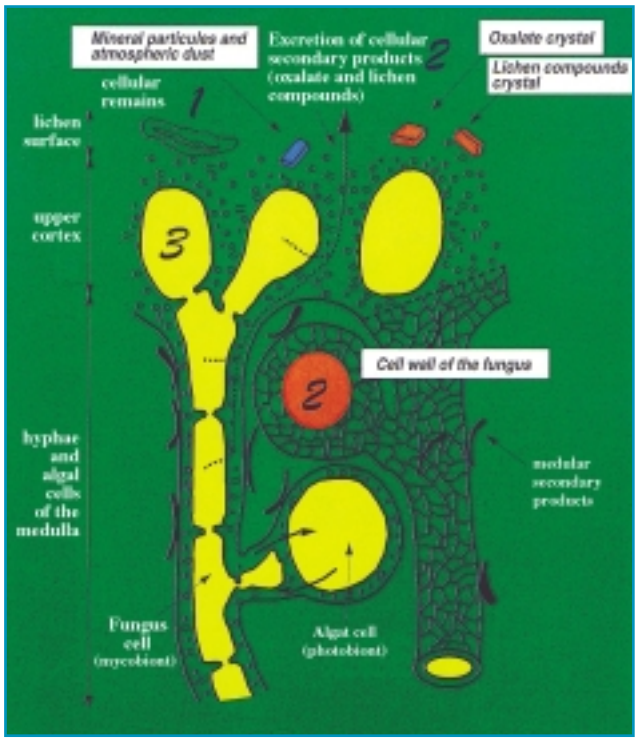

Fig. 1: Three different uptake mechanisms: (1) entrapment of metal-rich mineral particulates (2) extracellular complexation, (3) intracellular.

comprised between 100 and 500 mg/kg. Two lichens from two French sites were exposed to different pollutions: Xanthoria parietina, a saxicolous lichen growing on rocks near a tetraethyl and tetramethyl lead factory, and Diploschistes muscorum, a terricolous lichen collected on Pb-Zn tailings near a Zn extraction plant.

Comparisons of EXAFS spectra for lichens and for reference compounds (Figure 2) show that in the two lichens studied, metals are not included in mineral phases, but are complexed by organic ligands. The comparison with Pb or Zn-cysteine reference compounds enable us to reject the intracellular trapping mechanism for the two lichens. Therefore, three possible complexing agents could be involved in metal accumulation within the lichens: lichenic acids, macromolecules of the cell wall, or oxalic acid exudates.

• Diploschistes: in this lichen, Pb and Zn (Figure 3) were found to be bioaccumulated by the same mechanism, i. e. by precipitation of Pb, Zn oxalate. The production of oxalic acid is probably enhanced by the pollution stress.

• Cladonia: in this lichen, Zn is complexed by lichenic acids but not within the cell wall.

This study reveals that in both lichens, cells are protected from toxicity by extracellular complexation of heavy metals, but the strategies differ: in Diploschistes, Pb and Zn are accumulated through an enhanced excretion of oxalate, which precipitates toxic elements such as insoluble salts, whereas in Xanthoria, Pb is complexed to carboxylic groups of the fungal cell walls. We conclude that hyper-accumulation of metals results from a reactive mechanism of organic acid production, whereas metallo-tolerance is achieved by a passive complexation to existing functional groups.

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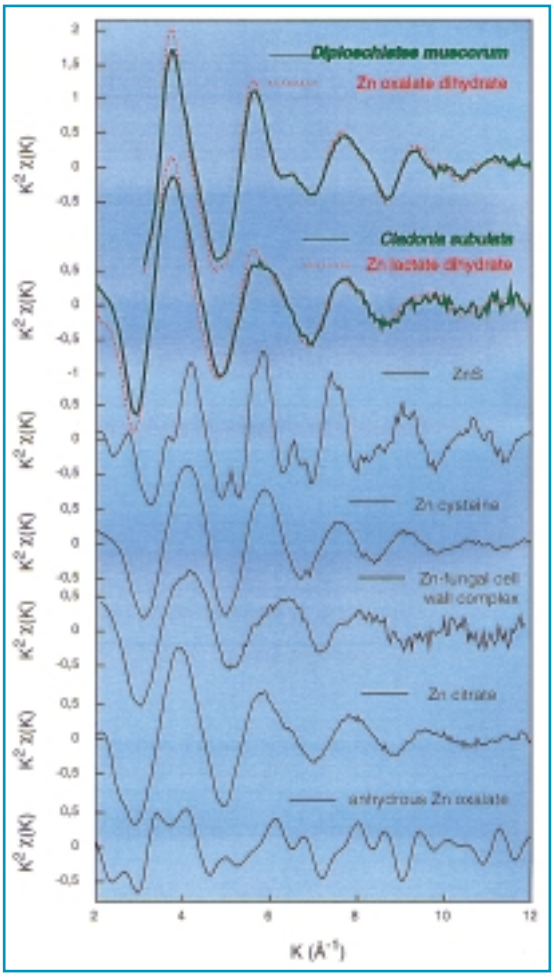

Fig. 2: Comparison of Zn K edge EXAFS spectra for the two lichens and some reference compounds.

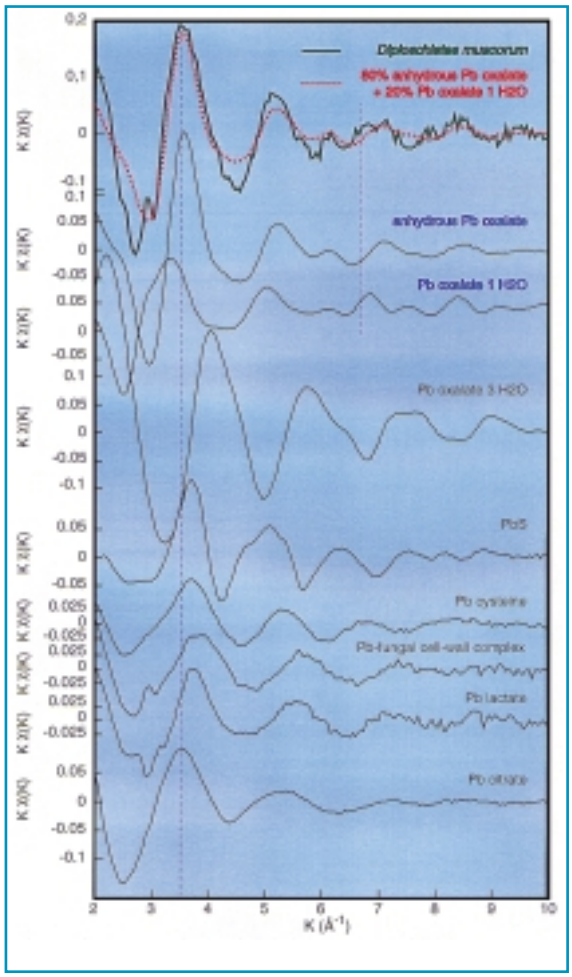

Fig. 3: Comparison of Pb L_{III} edge EXAFS spectra for a lichen and some reference compounds.

REAL-TIME PHASE-CONTRAST IMAGING

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2 ESRF, COMPUTING SERVICES DIVISION

Real-time x-ray imaging experiments, with typical evolution times in the 0.1 second range, are now possible at several ESRF beamlines. The FRELON (Fast REad-Out LOw-Noise) CCD camera, with 14-bit dynamic range and 1024 x 1024 pixel area, developed at the ESRF [1] is used on many of these beamlines. The standard solution to read this type of CCD camera data is using a SDV card directly plugged in the Sun workstation, and unified SPEC software. But this solution has not been able until now to take advantage of the full read-out speed of the FRELON camera (20 MPixels/s).

We present, in this short report, real-time phase images recorded on the ID19 ('Topography') beamline, using a VME system based on the VCCD3 card which allows one to record about 20 images / second on the complete area and with the full dynamic range. This system was developed in the Computing Services [2].

THE VCCD3 CARD

The VCCD3 VME card has on-board memory for image rebuilding, organized in two banks. While one bank is written/filled with the incoming pixels from the FRELON camera (new image), the other bank (previous image) is read/flushed into the VME memory card. The VCCD3 card allows:

- reading out the FRELON camera data over all 4 optical fibers to achieve the shortest possible read-out time (20 images/seconde).
- displaying a live image on a video display for monitoring purposes.
- summing up the data from the consecutive images of shorter duration when required.
- acquiring data in a continuous mode using the VME memory as a ring buffer; a pre-defined number of images can be stored after an interesting event (trigger).

The VCCD3 card stores the acquired images in a memory card, while BIT3 adaptor cards and fast link (25 MBytes/s) allow the online display program to be run on the Sun workstation and the data to be transferred in an efficient way. On the software side, the dedicated OS-9 device driver and server, as well as a special SPEC application, were used to both control the acquisition and to transfer the data.

FIRST RESULTS

The FRELON camera was equipped with an optics leading to a pixel size of 10 μm, a compromise allowing us to have reasonably short exposure times while retaining a fair spatial resolution. An energy of 20 keV was selected by the double-crystal (Si 111 in Bragg symmetrical geometry) vertical monochromator. For the first tests with a pure absorption object (stainless steel tip), the sample was located just before the x-ray converter (a Gd_2O_2S:Tb scintillator screen). For the final series of images of phase objects (gas bubbles in beer), they were placed at around 2.5 m from the camera in order to obtain the phase image through propagation (see papers by Buffiere *et al.*, and Baruchel *et al.*, in this Newsletter, pages 18 and 20). At this distance, we were in an edge detection regime, where black/white contrast appears at the border of the gas bubbles.

The images were corrected by subtracting the 'dark current' (without beam), and the 'flat field' images (with beam but without sample). A «fast» shutter (7 mm x 7 mm) was used to cut off the x-ray beam during the read-out time.

We tried to recreate the conditions of a real experiment in which we would have phase objects (thus, invisible in absorption) which move in some unpredictable way and whose behavior we wanted to track at a given moment. We found all these characteristics in the gas bubbles which appear inside a glass of beer (of course Champagne would also have been great, but it is more expensive). We took a series of data on several samples, using an exposure time of 50 ms which, together with the read-out time, yields a lapse of time of 100 ms between two consecutive images.

Figure 1 shows, as an example, a series of images recorded over a bubble source inside the beer. The emerging bubble is recognized when it has a diameter of ~ 30 μm. We can perfectly track its movement as it grows up to ø ~ 150 μm and its speed increases.

CONCLUSIONS

By using the FRELON camera at its full read-out speed, as carried out with the VCCD3 card, we could for the first time perform real-time phase-contrast imaging. The real-time display proved to be crucial for the success of the experiment.

The implementation of a fast read-out system well integrated within the standard ESRF hardware and software appears to be essential for the real-time image acquisition, which is of increasing interest for many users.

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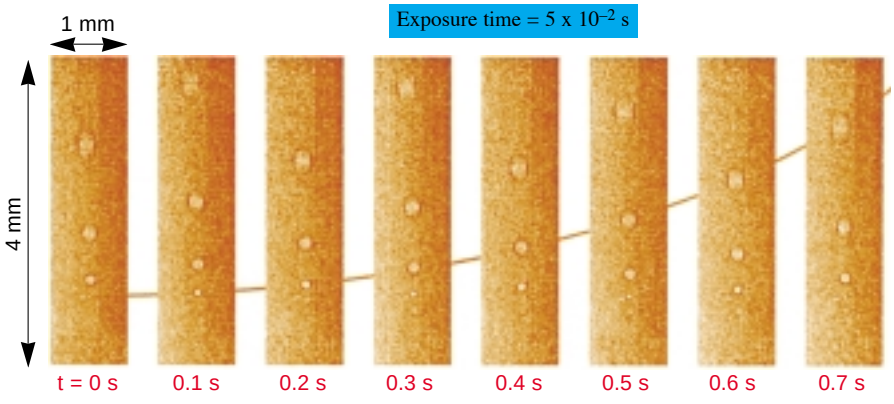
Fig. 1: Phase-contrast images showing the formation and evolution of gas bubbles inside a glass of «white» beer (a Belgian beer which is opaque).



MECHANICALLY INDUCED INFLUENCES ON THE STORAGE RING BEAM

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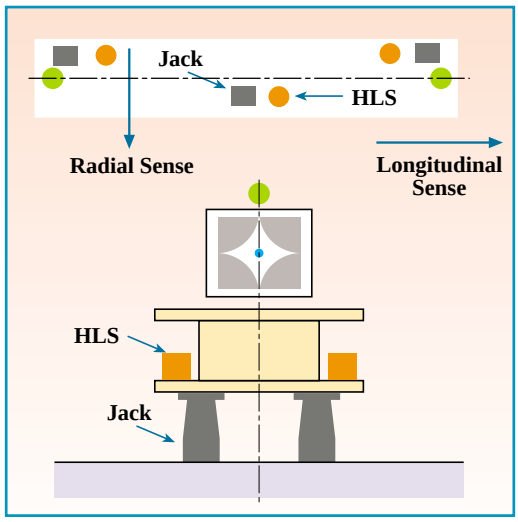
Magnet alignment errors are responsible for different types of perturbations in the ESRF storage ring.
The main effects are:
• Quadrupole magnet positioning errors inducing closed orbit distortions,
• Quadrupole tilt error inducing emittance coupling into the vertical plane.
These perturbations are normally compensated by magnetic correctors (horizontal or vertical steerers, skew quadrupole corrector magnets). Thanks to the good initial alignment of the machine, these correctors are used at a small fraction of their nominal strength. Alternatively the remote controlled motorised jacks supporting the magnet girders can also be used to minimise some of the misalignment errors:
• Vertical quadrupole positioning
• Quadrupole tilt (though this motion is correlated with an undesired horizontal displacement of the magnets).
The interest of correcting mechanically these errors may be:
• The analysis of the correction gives indications on the residual errors (amplitude, distribution) after the alignment of the machine,
• Ensuring a certain level of correction could permit the reduction of the strength of the magnetic correctors and at the same time improve their resolution. This would be beneficial for the fine adjustment of the beam position.
Several experiments have been performed recently to check these two correction procedures.

The remote control of the jacks, together with the Hydrostatic Levelling System (HLS), which cross-checks the effective girder displacement, enable these tests to be performed with the tunnel closed and with 5 mA of stored beam. The net advantage is that the effect (beneficial or not) of the girder displacements on the electron beam is assessed on-line. This beam-based alignment technique is a long awaited dream of accelerator physicists around the world. The different experiments were performed within a few hours and the girder's position and beam trajectory were put back at the end of the tests.

Fig. 1: position of the HLS and jacks on a typical storage ring girder.



CONTROL OF GIRDER MOVEMENTS

Figure 1 shows the position of the HLS and jacks on the G10, G20 and G30 girders. Longitudinal tilt motion is a rotation about the middle jack on a girder in the sense of the travel of the beam. Radial tilt motion is about the center of the girder in the sense perpendicular to the beam travel. Vertical movements are made on the G10, G20 and G30 girders.
Jack movements are calculated from longitudinal and radial tilt values issued from calculations of beam parameters. These tilt values are translated into movements for the three jacks under each girder. Corresponding expected HLS readings at the jack positions are also calculated and used as a control for these movements. The difference between the jack movement and the expected HLS reading is the precision of the movement. In practice, the HLS readings must be processed to eliminate the wave effect due to the motion of water in the pipes, and the possible discontinuities in the readings created by blockages in the water system.
For a 40 μm peak movement of the jacks, the residual standard deviation is 1.3 μm which represents both the precision of the 288 jack movements and the natural evolution in time of the storage ring girders.

VERTICAL BEAM CLOSED ORBIT DISTORTION

Calibration of girder displacements

Eighteen jacks were moved independently by 10 μm. The motion was checked by deducing the displacement from the beam position readings on the electron Beam Position Monitors (BPM) all around the storage ring. There is an agreement of better than 1 μm between the requested jack movement, the HLS readings and the beam response.

Closed orbit correction using girder displacements

The response matrix of the vertical closed orbit to girder motion was computed for the theoretical machine. In simulation analysis, it was confirmed that pure longitudinal rotation of girders was more efficient than pure translation. It was therefore decided to correct the machine using only girder rotations. The correction method was exactly the same as the one used for magnetic steerers (SVD method).
The procedure used in the test was:
• Start from a perfectly corrected machine,
• Reduce the number of steerer correction vectors so that the vertical orbit blows up significantly ($z_{rms} < 300$ μm),
• Measure the vertical closed orbit and compute the mechanical correction,
• Apply the mechanical correction,
• iterate.
The results were obtained after four

Table 1

	Nb. vectors left on steerers	rms. orbit before movements (μm)	rms. orbit after movements (μm)	rms. steerer strength (mA)
1st iteration	32	269	191	76
last iteration	3	493	215	21

iterations (see Table 1).
Figure 2 shows the displacements corresponding to this partial alignment:
The full cancellation of the steerer currents could not be tested because of lack of time, but should be straightforward. The rather large residual orbit is due to the choice of a limited number of correction vectors to avoid too large movements of girders.

HORIZONTAL/VERTICAL EMITTANCE COUPLING

The horizontal/vertical betatron coupling is responsible for the major part of the vertical emittance of the storage ring. It is defined as the ratio *k* of the vertical to the horizontal beam emittances:

$$\varepsilon_z = k\,\varepsilon_x$$

In a perfect machine coupling does not exist. It is a result of magnet imperfections and alignment tilt errors. Since the brilliance is inversely proportional to the coupling, its reduction is a way of optimising the performance. This is usually done by powering skew quadrupole correctors. Alternatively, coupling can be varied by tilting girders in the radial sense. The horizontal displacement linked to the tilts also induced undesired horizontal closed orbit distortions which were corrected by the standard orbit correction system.

Calibration of a harmonic tilt

The coupling is mainly sensitive to the excitation of the resonance close to the betatron tune difference ($\nu_x - \nu_z = 25$ in the case of the 4 nm lattice). So a systematic transverse tilt was applied to the girders on a corrected machine, according to this 25th harmonic:
The value of the peak angle was varied between 0 and 1 mrad and the coupling was measured using the pinhole camera. The results show a large deviation (factor 2 to 3) from the prediction of the theoretical model. This discrepancy is not understood at this point.

Correction of the main harmonic with the storage ring girders

This was applied on the low βz optics for which the tune difference is ν_x-ν_z = 22. A harmonic 22 tilt was applied to a machine without corrections and was varied experimentally to cancel the excitation of the coupling resonance:

rms tilt angle (mrad rms)	Phase (°)	Coupling
0	0	34 %
0.21	19	4.5 %

The residual coupling value is similar to what can be obtained with magnetic correction of a single resonance. However, the rms tilt value of 0.21 mrad (see Figure 3) introduced in the machine is very large. It cannot represent the compensation of a residual harmonic component with such an amplitude in the girder alignment. It probably compensates another coupling source (individual positioning of magnets on girders, magnetic tilt angle,…).

CONCLUSION

Several experiments have been carried out using high precision jacks installed under the SR girders relating mechanical motion to vertical closed orbit and coupling:
• the calibration of the translation of one girder,
• the complete correction of the machine by movements made to imitate the action of the steerers,
• the calibration of a harmonic excitation of a coupling resonance as a function of girder tilts,
• the compensation of the main coupling resonance.

The correction of coupling shows a sensitivity of the machine much higher than predicted and movements larger that what could be derived from alignment tolerances.

The correction applied for the vertical closed orbit agrees perfectly with the modelling and results in motions compatible with the residual alignment errors. This may be applied for the next machine realignment (performed with beam after the next winter shutdown) and could allow a further reduction of the rms vertical closed orbit errors. It could also allow the achievement of a low rms orbit (~ 200 μm) without any magnetic correction which would be a record for such a high focusing storage ring. ■

Fig. 2: Girder displacement after correction.

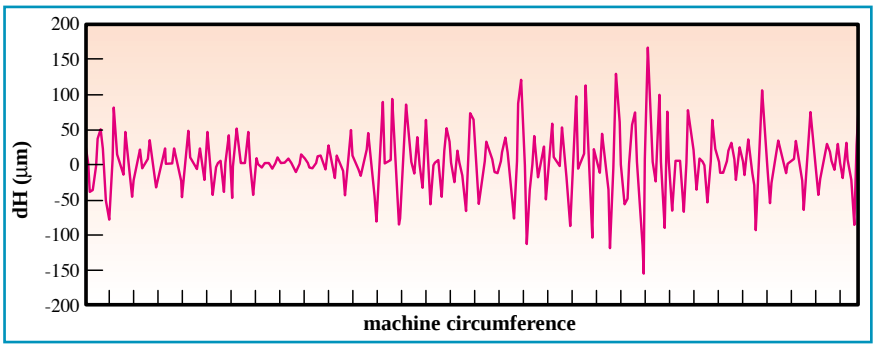
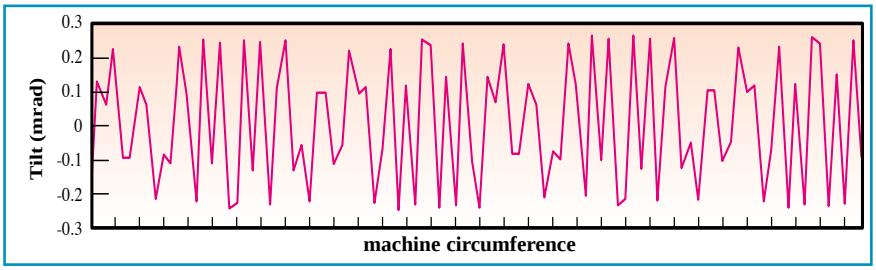


Fig. 3: Tilt movements used for coupling correction.



Events

Open Days
14-15 March 1998

In the optics lab.

About 2000 people visited the ESRF on this occasion and had the opportunity to meet with the ESRF staff.

(See page 11)

On ID14, a beamline for protein crystallography.

Theatrical itinerary «Lumières»
from 20 to 31 March 1998

6 actors and 1 musician created a new kind of visit at the ESRF, mixing Science and Art.

(See page 11)

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