# Newsletter



Nanoparticles were made by Renaissance artisans





### **Editorial**

3 EIROforum, ESRF and the European Research Area

### Feature news

- 4-5 Renaissance artists decorated pottery with nanoparticles
  - 6 Secrets of fish orientation discovered at the microfocus beamline
  - 7 FAME38 makes the first steps to facilitate sample alignment The ESRF collaborates with DESY
- 8-9 Night-time at the ESRF: a world of its own
- 10 New Head of the French delegation in the Council The Users' Meeting 2004: brace up for a leap forward
- 11 Nobel Prize winner in chemistry used ESRF biocrystallography facilities High demand for beam time in 2003

## **Interview**

12-13 Åke Kvick - Flying free in a world of science

## Selected scientific highlights

- 14-17 Latest research results at the ESRF
  - 18 News from the CRGs

### Scientific article

19-20 3D Structure and (Dis-)order in Photonic Crystals by Microradian Synchrotron X-ray Diffraction

## Visiting a beamline

21 ID08: Multitechniques in a multinational beamline

## **Gallery of events**

- 22 Students day: PhD students show their work to the other ESRF scientists
- 23 Experts on hard X-ray photoelectron spectroscopy share their expertise

## **Science & Society**

24 Discovering science (Fête de la Science)



## EIROforum, ESRF AND THE EUROPEAN RESEARCH AREA

wice a year the Directors General of seven major European intergovernmental research organisations - the EIROs meet as a collaboration coordination council - the EIROforum assembly - to discuss topics of mutual interest. The primary aim of this collaboration is to work together to promote the quality and impact of European research. The member organisations of the EIROforum - CERN, EFDA-JET, EMBL, ESRF, ESA, ESO and ILL - cover a very diverse range of scientific activities, but share substantial experience and expertise fundamental and applied research and in the operation of large international research infrastructures.

In its charter, signed in 2002, EIROforum stressed the importance of joint discussion, the optimisation of resources and facilities, and the coordination of outreach activities. To 'put flesh on the bones' of these aims, a number of thematic working groups were set up. Of particular importance to the ESRF are the *Instrumentation* and the *Outreach and Education* working groups.

The Instrumentation group has focussed on three areas: detectors, data acquisition and visualisation, and neutron diagnostics. A concrete realisation of this collaboration is the IDEPHIX FP6 proposal, submitted to the European Commission (EC) with the ESRF as coordinating organisation. This

ambitious proposal aims to develop pixel detectors for synchrotron radiation use, as well as for applications in medical and security imaging. The EIROforum dimension arises from the close collaboration on this proposal, uniting the detector competences of ESRF and CERN, with a large number of academic and industrial partners.

EIROforum has been very active where Outreach issues are concerned. As well as information events describing the research of the EIROs at the European Parliament and the FP6 launch event, the Outreach group has organised important communication events such as Physics on Stage; the next of the series, Science on Stage, will take place in Grenoble in October 2004. All of the EIROforum members have joined together to produce the European Science Teachers Initiative (ESTI) proposal to the EC, aimed at employing the know-how and facilities of our large research infrastructures to give support and encouragement to the teachers of science throughout Europe. So the ESRF is an active and fully engaged member of the EIROforum partnership. Where does the European Research Area (ERA) come into this? The ERA was launched in March 2000 at the European Council meeting in Lisbon. Two years later, this time in Barcelona, the European Council set Europe the goal of becoming the 'leading knowledge-based economy' by the year 2010. This is a very important



The European Research Commissioner, P. Busquin (4th from left), signs the EC-EIROforum Statement of Intent in Brussels on 27 October 2003. From left to right are C. Carlile (ILL), L. Maiani (CERN), C. Cesarsky (ESO), P. Busquin (EC), J. Pamela (EFDA-JET), W.G. Stirling (ESRF), J.-J. Dordain (ESA), and F. Kafatos (EMBL).

commitment to research - and to research funding - by the European nations. To achieve the Barcelona goals it is estimated that a level of spending on R&D of 3 % will be required. Most nations are currently far from this.

The EIROs - including the ESRF - have a key role to play in the future development of the European Research Area. They represent some of the best of European science and are all world leaders in their particular fields. A few weeks ago in Brussels, the European Commissioner for Research, Philippe Busquin, and the Directors General of the seven EIROforum members signed an important statement of intent to work together toward the creation ERA (see photo above). The EIROforum members and the EC have agreed on a number of joint initiatives, judged by Commissioner Busquin to be "a new step towards the creation of the European Research Area" which "will play a decisive role in promoting the quality and consistency of European Research".

The EIROforum which started out three years ago as an informal discussion forum between the managements of several research institutes is now becoming a major player in crucial developments that will affect all of us in research in Europe.

Watch this space!



A XVI century dish from Deruta, with gold lustre decorations.

## Feature news

# RENAISSANCE ARTISTS DECORATED POTTERY WITH NANOPARTICLES

he nanoworld is not just a modern high-tech trend. Centuries ago, our ancestors were already using nanoparticles of silver and copper to decorate pottery. The first examples dated from the IX century AD in Mesopotamia. Now the ESRF has helped in the elucidation of the amazing techniques used in Renaissance pottery to make it glitter.

Pottery from the Middle Ages and Renaissance Era often has gold and copper-coloured metallic reflections and iridescence. These effects are called lustre and originate from a metallic film that was applied to the transparent surface of medieval glazed pottery. Centuries later, the lustre is still visible thanks to the high quality of the film and its resistance to atmospheric oxidation and burial weathering.

Samples of XVI century pottery from Deruta. EXAFS measurements were carried out on the red and gold luster indicated by arrows.



But how could this film create these peculiar optical effects? The answer is in the composition of the film itself, with silver and copper nanoparticles, dispersed homogeneously in the glassy matrix of the ceramic glaze. To create these nanoparticles, artisans put a mixture of copper and silver salts and oxides, together with vinegar, ochre, and clay, on the surface of previously-glazed pottery. The object was then placed in a kiln where it would have been heated to about 600°C in a reducing atmosphere. The high temperature caused the glaze to soften, and then the copper and silver ions would have migrated into the outer layers of the glaze. The reducing atmosphere reduced ions to metals, which then came together forming the nanoparticles that give the colour and optical effects.



Positioning of the ceramic samples in the vacuum chamber.

Several techniques have been used to characterise the chemical and physical properties of these films. Compositional detailed information was obtained by Rutherford Backscattering Spectrometry (RBS), whereas the optical properties were investigated by optical absorption in the visible-ultraviolet region. By electron microscopy (TEM and SEM) it was possible to image the metallic particles present in the glaze and X-ray diffraction patterns were collected to determine the crystalline phase of the particles. In order to determine the valence state and to describe the local atomic environment around the metallic species, researchers came to the ESRF to carry out experiments of X-ray absorption spectroscopy on BM08 (GILDA CRG beamline). This in particular permitted the amorphous oxide phase for the metals to be evidenced, which had not been revealed by the previous experimental investigations.

## About the colours

Different recipes were used to obtain different colours, ranging from goldlike to copperlike. Red comes as a result of the copper ions' migration and reduction. In the case of gold shades, in principle only silver is needed, but craftsmen used both copper and silver. Researchers are still trying to find out why they used both materials and how they did this, since copper needs higher

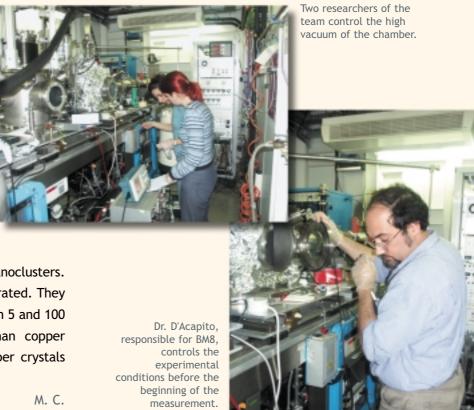
temperatures than silver in order to get nanoclusters. Silver and copper nanocrystals are well separated. They are quasi-spherical, with a diameter between 5 and 100 nanometres. Silver nanocrystals, larger than copper ones, appear grouped together among copper crystals and close to the glaze surface.

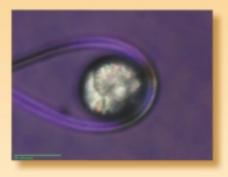
## Looking back...

The technique of lustre was developed by the Islamic culture in Mesopotamia, during the IX century. Muslims were not allowed to use gold in artistic representations, therefore they had to find a way to create the same effect without using real gold. So they found a solution by using the lustre technique. It arrived in Spain during medieval times, following the expansion of Arabian culture. From there it was introduced into the centre of Italy, where it was exploited to produce polychrome lustre Renaissance pottery.

### And forward...

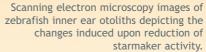
The lustre technique shows that craftsmen had a technological and empirical knowledge of materials science and were ahead of their times. The information obtained from this technique can help to better understand the process that led to uniform reproducible and long-lasting metalglass nanocomposites. Nanocomposites consist of a mixture of two or more components, where a polymeric matrix is reinforced by addition of other components such as metals or fibres of nanometric dimensions.







Otoliths are normally round stone-like structures. After the reduction of activity of the gene, they become star-shaped crystals. The pictures were taken with the microscope of the ID13 microdiffractometer.





# SECRETS OF FISH ORIENTATION DISCOVERED AT THE MICROFOCUS BEAMLINE

Institute recently teamed up to study how a single gene in fish influences the biomineralisation of otoliths. The stone-like otoliths are located in the fish's inner ear and are involved in the balance and hearing processes. The previously unknown gene has been given the name starmaker as the reduction of its activity results in star-like otoliths. Humans have a similar gene, which is also implicated in hearing, as well as teeth formation. Synchrotron light was used to uncover changes occurring in the structure of otoliths following gene alteration. An article on this research was recently featured in Science magazine.

The experiment consisted of reducing the activity of the *starmaker* gene and hence its encoded protein in order to see how it influenced the otolith formation in zebrafish. As a result of this modification, their otoliths, which are normally smooth, round, stone-like structures became elaborate star-shaped crystals. That is the reason why the gene has been named *starmaker*. Synchrotron light experiments showed that this change is also associated with a change in calcium carbonate crystal structure. After the reduction of *starmaker* activity, most of the fish had difficulty in orienting themselves quickly in fast-moving water. This is the first time a change in the otolith has been induced and tested for behavioural defects in live animals.

The team of researchers used X-ray microdiffraction techniques developed for protein crystallography at the microfocus beamline, ID13, at the ESRF. The experiment was performed on different types of otoliths of less than 30 µm in diameter. The goal was to determine which crystal polymorph was present in each type of otolith. The impressive issue is that just by modifying one single gene, there was a change of the calcium carbonate polymorphs at room temperature. "This is an important breakthrough and there are currently many teams from different scientific fields working on this kind of biomineralisation experiments. The final aim is to develop artificial materials based on information from biological systems, that is, to mimic nature", explains Christian Riekel, the scientist responsible for ID13 and one of the authors of the article.

This gene is being studied because it is a counterpart of the human gene responsible for hearing loss and teeth formation (DSPP or *dentin sialophosphoprotein*). Both of them encode many negatively-charged amino acids, although they have a different structure. In humans, "the mutation of the DSPP protein is probably involved in biomineralisation of teeth in a similar way that *starmaker* mediates crystal formation in otoliths", explains Teresa Nicolson, the principal investigator.

M. C.

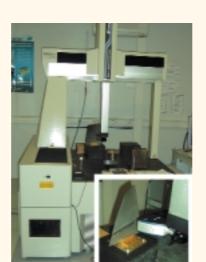
## FAME38 MAKES THE FIRST STEPS TO FACILITATE SAMPLE ALIGNMENT

Materials Engineering) laboratory in November 2002, work has been progressing to increase the support available to engineering users. Although there is an emphasis to users in the strain/stress field, support is also available for materials science experiments generally. Complementing the on-line support that is being provided, an off-line laboratory is

available, which provides advanced metrology tools, data analysis facilities and support from the FaME38 team.

A particular objective of FaME38 is to improve the efficiency of engineering experiments that are performed at the ESRF. Often a significant amount of beamtime is spent fixturing and aligning a sample on the beamline and generating the appropriate SPEC measurement 'macros'. The FaME38 philosophy is that this type of work should be performed off-line, and if possible, prior to an experiment. The FaME38 metrology laboratory is

equipped with a high resolution Coordinate Measurement Machine (CMM) that allows sample shapes to be determined to an accuracy of 2  $\mu$ m. In order to achieve



The FaME38 Coordinate Measuring Machine (CMM), which is equipped with an advanced non-contact laser metrology head (inset).

correlation between the sample coordinates on the CMM and the beamline, a standard flexible fixturing system has been developed. The system is based on a beamline-mounted baseplate with a half threaded, half dowelled hole matrix, and a range of sample mounting tools that have a matching hole pattern. The methodology that is being applied means that a sample is measured on the FaME38 CMM and, when it is transferred to the beamline, the relative position of the sample with respect to the measuring or 'gauge' volume is known. The reduction in sample alignment time is significant and the need to use X-ray or optical sample alignment is minimised. In fact, once the 'gauge' volume has been determined at the beginning of an experiment, all further alignment,

mounting and planning should be performed off-line.

Furthermore, work is currently underway to create tools that will enable the experimental simulation to be performed away from the beamline. It is intended that this will be available via the Internet to users, prior to an experiment. By combining a Computer Aided Design (CAD) knowledge of the beamline environment and accurate sample models available from the CMM or CAD it will be possible to perform an experiment 'virtually' without actually having access to a beamline. In this manner it will be possible to resolve conflicts in

positioning and generate appropriate measurement 'macros'. For further information contact the FaME38 team (FaME38@esrf.fr).

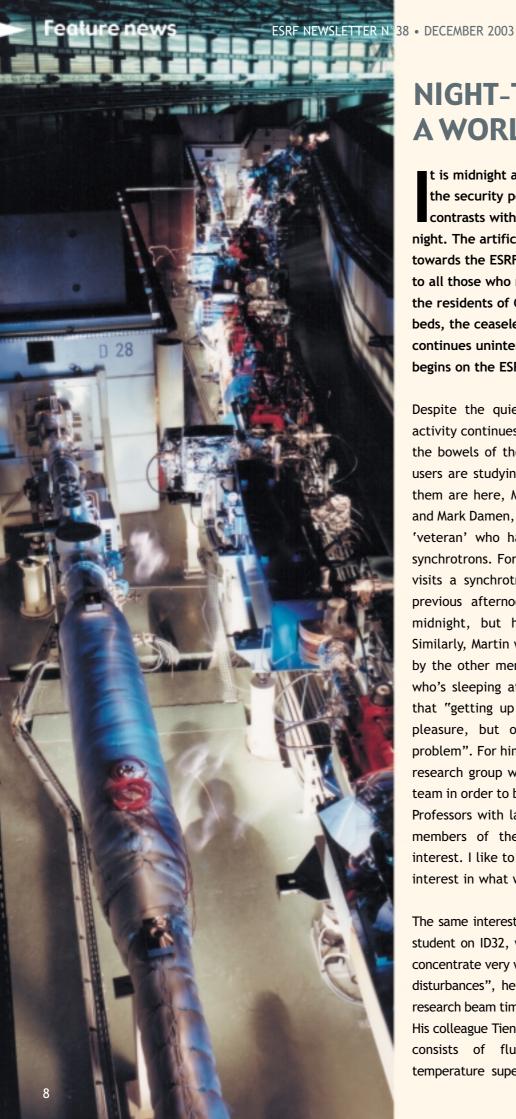
**DARREN HUGHES** 

## THE ESRF COLLABORATES WITH DESY

The directorates of DESY and ESRF assisted by group leaders met in Grenoble earlier this year to discuss details of a collaboration between the two institutions. DESY has currently two projects underway: the X-ray Free Electron Laser (XFEL) and the upgrade of PETRA to a synchrotron light source (6-7 GeV, 100-200 mA, 30 x 3  $\mu$ m² electron beam size and 13 undulator stations). The German Federal Ministry of Education and Research will fund half of the investment of both projects. The remaining funds must come from other countries so that

the projects will have a truly international character.

The discussions included technical and scientific subjects such as machine and beamline control systems, insertion devices, beamline automation and instrumentation, as well as matters regarding staff exchange, fellowships, administration, outreach and education. Whereas in general the mutual benefits of collaborative activities are obvious, much effort is needed to formulate the details of a collaboration that satisfies both parties.



## **NIGHT-TIME AT THE ESRF:** A WORLD OF ITS OWN

t is midnight at the ESRF and the presence of the security personnel at the site entrance contrasts with the surrounding dark and silent night. The artificially lit corridor that stretches towards the ESRF serves as an impassive witness to all those who make their way to work. Whilst the residents of Grenoble lie tucked up in their beds, the ceaseless pursuit of scientific discovery continues uninterrupted. And so another night begins on the ESRF night shift...

Despite the quietness that reigns outside the ESRF, activity continues - and sometimes quite frantically - in the bowels of the facility. On BM26, a team of Dutch users are studying some protein samples. Only two of them are here, Martin Feiters, the head of the team, and Mark Damen, a PhD Student. Martin is a synchrotron 'veteran' who has done experiments at many other synchrotrons. For Mark, however, it is the first time he visits a synchrotron. Having been to bed during the previous afternoon, he has only just woken up at midnight, but he reckons he hasn't slept a lot. Similarly, Martin will go to bed at 3am and be replaced by the other member of the team, Marjolijn Roeters, who's sleeping at the moment. Martin Feiters admits that "getting up in the middle of the night is not a pleasure, but once you are working there is no problem". For him, it is all a question of structuring the research group well: "You need to come with a larger team in order to be able to man the station at all times. Professors with large groups just assign these tasks to members of their team irrespective of their own interest. I like to come with people that really have an interest in what we are doing".

The same interest is what keeps Sebastian Thiess, a phD student on ID32, wide awake at an ungodly hour. "I can concentrate very well in these conditions since I have less disturbances", he explains. Tonight he is using in-house research beam time for some measurements for his thesis. His colleague Tien Lin Lee is helping him. The experiment consists of fluorescence measurements of high temperature superconductors using the ID32 beamline.

ESRF NEWSLETTER N°38 • DECEMBER 2003

Sebastian has an average of six night shifts every two months. He is so devoted to science that he doesn't really mind working at night. He adds that "the beamline does not have any daylight, so when I am here I don't even know whether it is day or night". Mark Damen agrees and compares the synchrotron to a submarine.

For Steve Quarless, nights are much more obvious. "I don't like nights, it is too hard on the body", he says. Today he is the Experimental Hall Operator throughout the night. He has this timetable for two nights in every ten days. The Experimental Hall Operator's job is to ensure that everything works well and safely on the beamlines. During the night, he does a tour of the Hall to check that everything is going smoothly. He also attends to calls from scientists who are experiencing problems with beamline equipment. Apart from having a good general technical knowledge, the Experimental Hall Operator "must know how to deal with people", says Steve. Users come for a short time and their maximum concern is that their experiment runs well. At night, there are much fewer technicians and scientists than during daytime. Therefore, the Hall Operator is a big help whenever a problem appears. Steve Quarless says that "if you cannot solve it, you have to be very diplomatic with them". Sebastian Thiess explains: "Working at nights can be very stressful because there are no technicians or staff to help you whenever you have a problem". On the other hand, for Mark Damen, "it is nicer to find a solution yourself instead of just asking straight away".

This lack of people can make one feel the solitude even more. The Storage Ring status screen is a good reflection of how much life there is at the ESRF. As the night goes by, it shows more beamlines on automatic mode. The automatic delivery mode is a means of making the beamlines work with minimal human intervention. In this case an experiment has been pre-programmed by the scientists.

The coffee machines spread around in the ring are on service all night long. In fact, they constitute a very important means to staying awake. For the team of Dutch users, it's not only coffee but also food that is important: "It is important to have good provisions of food and drink so that you do not get problems with the irregular



Sebastian Thiess working in his experiment at night on ID32.

working and eating/drinking times", explains Martin Feiters.

In this special atmosphere of night shifts, the ESRF has some 'guardian angels' who oversee that everything runs correctly: the technicians in the Control Room. They control the correct functioning of the machine and the onsite security. Their role at night is of critical importance to the extent that the authority commanded by the Control Room Operatives replaces that of the Director General. Tonight Benoit Joly is 'taking care' of the ESRF. He monitors the numerous alarms that may be set off (often by mistake), as well as the electricity infrastructure, the water system and the general security of the site. In the unlikely event of problems that even he cannot deal with, he has a list of experts who are on permanent 'stand by' and can be contacted at anytime in case of emergency. But luckily, emergencies are very rare.

It's almost morning at the ESRF and there are a few scientists still working. Sebastian Thiess looks more awake than ever, his tiredness 'kidnapped' by his desire for new and interesting data. He is still busy; only a few hours of beam are left. He, as the other remaining scientists, is determined to use his beam time as best as he can and measure until the last photon of the morning. After that, the night workers of the ESRF climb into their beds. Although tired, they may be satisfied by another successful experiment or disappointed by the complications they might have encountered, but always dreaming of the discovery that their new data could potentially bring... •

## NEW HEAD OF THE FRENCH DELEGATION IN THE COUNCIL

he Council meeting due to take place in December will see a new Head of the French Delegation, Michel Lannoo. He has been appointed by the two French member organisations of the ESRF, i.e. the CNRS and the CEA, and will replace Jean-Paul Pouget. To obtain a better knowledge of the ESRF, he visited the facility in September, discussed with Management and had an extended guided tour of the beamlines.

Michel Lannoo is the Scientific Director of the Department of Physical Sciences and Mathematics of the CNRS. He is a physicist and has considerable experience in the field of nanoscience: in 1999 he was appointed Director of the 'nanostructures' incentive action



Dr Lannoo listens to Odile Robach's explanation on ID03 during his visit to the ESRF.

launched by the French Ministry of Research, and a year ago he became the Director of the 'nanosciences-nanotechnologies' research programme.

Besides the Director General, the ESRF Council is the essential decision-taking body of the ESRF. Each of the eight contracting parties (France is one of them) appoints a delegation composed of up to three delegates to the ESRF Council. The Council meets at least twice a year. It decides important issues of company policy and may issue instructions to the Director General.

M. C.

## THE USERS' MEETING 2004: BRACE UP FOR A LEAP FORWARD

An invitation from the Head of the Users' Organisation

n February 2004 Grenoble will be more than ever the place to be for all European scientists interested in synchrotron radiation and X-rays. From Monday 9 to Friday 13, the annual ESRF Users' Meeting and three topical workshops will offer everyone a great opportunity to enjoy exciting new science and to debate on future directions. For its 14th edition, the format of the Meeting will be revised with the aim of promoting exchanges of points of view among scientists.

The plenary meeting will move to an entirely new format, from Tuesday afternoon to Wednesday noon, with a programme especially devised to promote the interaction between the participants and with the ESRF staff. The users community will be asked to identify its long-term scientific needs, its opinion on a strategic document on the future of the facility, prepared by the ESRF Management. The 2004 Meeting will be the ideal forum to exchange new ideas, and to ignite a broad debate on crucial decisions that will influence our research, and that of our younger colleagues, for a long time to come.

There will be a large emphasis on parallel sessions, organised according to the scientific interest. Guided

Marco Grioni

discussion will start there on specific and common problems, and their conclusions will contribute to set up the agenda of a panel discussion that will close the meeting on Wednesday. But before then there will be time for inspiring talks, a poster session where all posters will be available on-line ahead of time, the much awaited Young Scientist Award ceremony, and the latest scientific and technical news from the beamlines. Last but not least, this time we will play at home. The ESRF site will in fact host all scientific sessions and, needless to say, a memorable banquet.

I cordially invite all users to attend the 2004 meeting. Update your scientific wish list, imagine your research in 10 years from now - dreams are allowed from time to time - and come to Grenoble to share the excitement.

Head of the Users' Organisation

## NOBEL PRIZE WINNER IN CHEMISTRY USED ESRF BIOCRYSTALLOGRAPHY FACILITIES

he 2003 Nobel Prize in Chemistry was recently awarded to Roderick MacKinnon, who conducted some of his research on ID13, the microfocus beamline at the ESRF. He earned the accolade for his studies on the structure and

mechanics of ion channels. The prize was shared with Peter Agre, who was recognised for the discovery of water channels.

Ion channels are tiny pores that stud the surface of all of our cells. These channels are made of proteins that let ions such as potassium, calcium, sodium and chloride molecules pass by. The opening and

closing of these channels release ions, moving electrical impulses from the brain in a wave to their destination in the body. Roderick MacKinnon was a visiting scientist on ID13 several times in the year 2000 and worked on potassium and chloride channels.

The scientist worked mainly with the microgoniometer in the beamline, developed by the ID13 team in collaboration with EMBL scientists.



Roderick MacKinnon

Professor Roderick MacKinnon, Head of the Laboratory of Molecular Neurobiology and Biophysics at the Rockefeller University in New York, has also conducted his research at other synchrotrons, all in the United States. The fact that part of his research took place at the ESRF, in Europe, is a sign of the thriving situation of this European facility. It also shows its spirit of

collaboration and openness towards American scientists.

M. C.

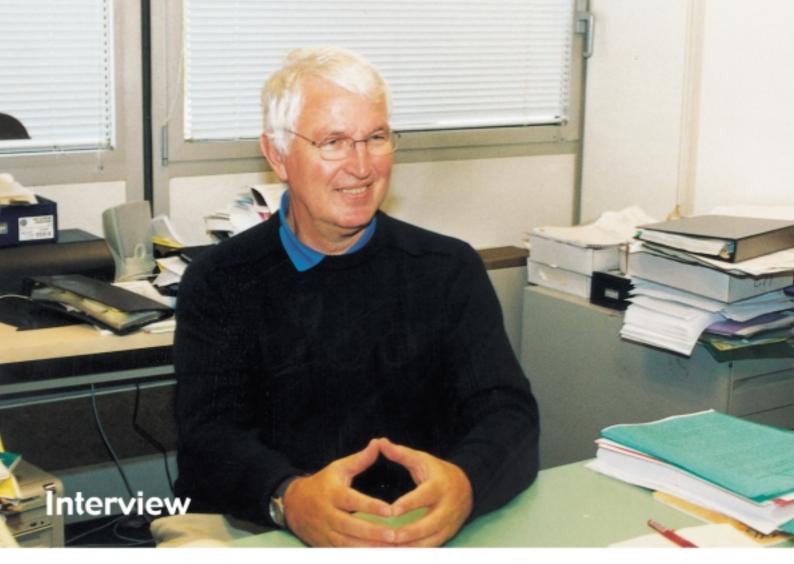
## HIGH DEMAND FOR BEAM TIME IN 2003

total of 752 new applications for beam time arrived at the Users' Office for the last deadline, in September, all of them submitted electronically. These applications include a request for 11993 shifts of beam time for the next scheduling period for user experiments, from March to August 2004. All in all, in the two rounds of 2003, a total of 1622 proposals were submitted, 8% more than the previous year. Unfortunately, only 769 will actually be allocated beam time.

The largest number of new proposals (135) were related to studies of structures in the Hard Condensed Matter area, as has been the case in past years. The least demanded area was medicine, with 21 proposals. Nevertheless, this discipline is beginning to be more and more attractive for experiments, so the

demand increases. Therefore, Management decided to have a Medical Committee for the first time in the last September round. A team formed by four experts, including medical doctors who work with synchrotron light, decided which proposals would be accepted. "This new committee worked extremely well", explains Roselyn Mason, Head of the Users' Office, "and it is foreseen to have two more experts in the team for the forthcoming March round". The other eight Review Committees that assess the applications on the basis of scientific merit also met recently at the ESRF. They recommended projects for beam time on the 30 ESRF and 10 CRG beamlines open to users during the first half of 2004. However, the results were not yet made public by the closing of this Newsletter.

M. C.



## **ÅKE KVICK**

## Flying free in a world of science

His very first publication was an article in a Swedish journal about a new bird species that had just appeared in his country. He was 14 years old and loved watching the birds fly free, with energy, discovering the world and with the will of living life at its maximum. Just like a bird, Åke Kvick has always been a free spirit as a result of an insatiable curiosity for the world. Indeed, curiosity is the main driving force for his career in science: "I like to work on something I don't know where it exactly will lead and that's what science is, finding out the unknown", he acknowledges.

e carries more than three decades of science on his back, but science with very different aspects: research, teaching, and lately, managing and being editor of a scientific journal. He defines his career path as 'atypical' for a scientist: "most researchers stay in one University for ever and, with time, become an authority in one field, whereas I've changed my speciality several times". Even if he

admits that this has hurt his career, he doesn't regret it. In fact, this is linked to his personality, eager for the discovery of new things and ready to take calculated risks. Every time he has had an attractive possibility of a change of direction in his career, he would think: "This is a challenge, let's see if I can succeed". Thanks to this philosophy, he started with biochemistry at the university, "I wanted to understand how

molecular reactions work", and after the first year of chemistry at Uppsala University, he became a teaching assistant in inorganic chemistry. Soon afterwards, he spent a decade in New York, where he constructed the diffraction beamline at the National Synchrotron Light Source. Then he moved to the alpine city of Grenoble where he participated in the birth and growth of the ESRF. His incentive to go to the facility was his desire to

learn more about chemical reactions. He became more and more involved in the synchrotron world. He kept a strong link to his home country, Sweden, by belonging to the board of Maxlab, the synchrotron facility in the town of Lund, where he is still involved.

## **Keeping the roots**

Despite his links with his country, where does he feel at home after all the moving from one country to another? He admits his home is Sweden, where he goes often, mainly for work. After getting used to milder temperatures, he doubts he could live there all year long, but he sees himself retired and living on the farm he has in the countryside in Sweden with his wife. He speaks Swedish to his wife and two children, aged 22 and 32, French in town and English at work. Speaking Swedish is a way of feeling at home, although he assures that it is much faster for him to read (usually currently the Head of the Materials Science group at the ESRF, as well as the Head of the Experiments Division Secretariat. He is also a the member οf Experimental Division Management Board and co-ordinator for the ESRF users' Scientific programme and the Management Information System. "Isn't management considered by scientists as a degradation one's career somehow?", asks the interviewer. "It is a very important aspect for an institution like this one", he assures. And adds: "It won't give me any credits like a publication would do, but I get personal satisfaction". It is the kind of job that suits him at his age (61): "when you are 30, a researcher has to be selfish and competitive to get success in science, but at my age I can do this kind of job because I am more mature and established, and I can act convinced of what I do thanks to my wide experience in broad scientific areas".

### A BORN TRAVELLER

There are two agendas on his office desk. A big one for the office and a small one for travelling. Where to? His latest trip was to Brazil, for work, and then he went to Canada. Thanks to his former position as Responsible for **Swedish** Research assistance to developing countries, he travelled to most of Latin America some years ago. However. he also eniovs travelling closer to Grenoble: whenever he can, he goes bird watching in the Camargue or takes the car, the Michelin Guide and improvises a getaway weekend with his wife somewhere.

## "Management is a very important aspect for an institution like the ESRF"

non-scientific books) and write in English rather than in Swedish. His ability to adapt to new cultures has been the key to the success in his life: "Life has always been very good to me; the only negative aspect is that it is not easy for the family, they've suffered from my career".

## Managing responsabilities

A career that has led this senior scientist to take management responsibilities at the ESRF. He is His gift for listening to people and dealing with them is an important asset in his position. Being able to take decisions is an outstanding quality he has proven to have. "Decisions are tough, but the worst thing you can do is not to choose", he acknowledges. His whole life has been a result of taking risks by deciding things that maybe weren't the most obvious, and all in all, he doesn't regret it: "Whenever I have to take a decision, I don't look back".

M. C.

Fig. 1: 3D rendering of a diatom cell:
Top: external surface of the diatom
(yellow) and of the glue (white)
reconstructed by conventional
transmission tomography.
Bottom: 3D rendering of the
distribution of Bromine (red)
revealed by making transparent
the external surface.

## Selected scientific highlights

## X-RAY IMAGING GROUP

# ID22 - Non-destructive Quantitative 3D Elemental Microanalysis combining X-ray Transmission, Fluorescence and Compton Helical Microtomography

B. Golosio, A. Somogyi, A. Simionovici, P. Bleuet, J. Susini ESRF - Laboratoire des Sciences de la Terre de l'ENS Lyon, France

The understanding, control and modification of environmental, chemical and biological processes necessitate their thorough investigation and knowledge. Certain trace elements (e.g. Cr. Se, Fe, Zn, Mn) may play a very important - but usually not fully understood - role in these processes. Therefore, nondestructive, quantitative, 3D, in situ investigation of the trace element distribution at (sub)micrometre resolution and the study of the relationship of the elemental composition with morphological features would be of interest for several research fields.

There are several methods (e.g. absorption-, phase contrast-, Compton-tomography) that provide information about the internal structure of a sample at the (sub)micrometre scale. However, only limited number of techniques (e.g. X-ray fluorescence tomography, energy filtered transmission electron

tomography, PIXE tomography) are capable of revealing information about the internal elemental composition. Among these methods only X-ray fluorescence tomography can be considered as non-destructive.

In order to fulfill the above described requirements, a new non-destructive X-ray technique has been developed at the ID22 beamline by combining simultaneous transmission, fluorescence and Compton microtomography [1]. The combination of the information of the three tomographic techniques provides a precise quantitative reconstruction of the elemental composition and internal structure of the sample. To date X-ray fluorescence, Compton and the recently developed combined tomography methods have been applied to the reconstruction of one or only a few separate slices of a given sample, mainly due to time constraints. This approach does not provide the required truly volumetric

reconstruction since the structural details of the intermediate space between the slices are lost. In order to overcome these constraints a truly 3D combined XRF/Compton/transmission tomography was performed by a helical scan of the sample through the microbeam. It has been proved that for a given measurement time, helical CT provides better longitudinal resolution than the conventional CT. A set of planar projections can be obtained from the raw helical scan data via various interpolation techniques, *e.g.* by the fullscan interpolation method, which was used in this study. The quantitative reconstruction of each planar slice has been obtained by the Integrated Tomographic Technique (ITT) [1].

Figure 1 shows an application of this method in the field of biology. The reconstructed structure and elemental composition of a single diatom cell is shown in this figure. The total longitudinal translation during the spiral scan was 72  $\mu$ m. By the fullscan interpolation method 2  $\mu$ m slice-to-slice separation has been obtained.

The authors are grateful to A. Homs, R. Tucoulou and S. Labouré for help with the acquisition software and experimental setup and to L. Lemelle for providing the diatom sample and for her help during the sample preparation.

[1] B. Golosio, A. Simionovici, A. Somogyi, L. Lemelle, M. Chukalina, A. Brunetti, J. Appl. Phys. **94**(1), 145-156 (2003).

## HIGH RESOLUTION AND RESONANCE SCATTERING GROUP

## ID16 – Iron Partitioning in Earth's Mantle: Toward a Deep Lower Mantle Discontinuity

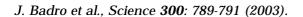
J. Badro, G. Figuet, F. Guyot Université Paris VI, Institut de Physique du Globe, France

J.-P. Rueff Université Paris VI, France

V.V. Struzhkin Carnegie Institution of Washington, USA

G. Vankó, G. Monaco ESRF

We measured the spin state of iron in ferropericlase  $(Mg_{0.83}\ Fe_{0.17})O$  at high pressure and found a high-spin to low-spin transition occurring in the 60- to 70-GPa pressure range, corresponding to depths of 2000 kilometres in Earth's lower mantle. This transition implies that the partition coefficient of iron between ferropericlase and magnesium silicate perovskite, the two main constituents of the lower mantle, may increase by several orders of magnitude, depleting the perovskite phase of its iron. The lower mantle may then be composed of two different layers. The upper layer would consist of a phase mixture with about equal partitioning of iron between magnesium silicate perovskite and ferropericlase, whereas the lower layer would consist of almost iron-free perovskite and ironrich ferropericlase. This stratification is likely to have profound implication for the transport properties of Earth's lowermost mantle and hence to strongly affect whole-Earth dynamics.



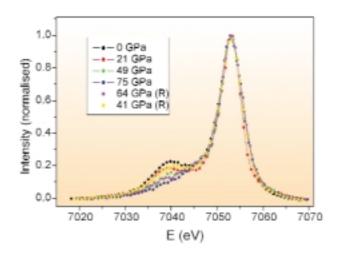


Fig. 2: X-ray emission spectra collected on ferropericlase ( $Mg_{0.83}$  Fe<sub>0.17</sub>)O at different pressures. The presence of a satellite structure ( $K\beta$ ' line) on the low-energy side of the iron main emission line ( $K\beta_{1,3}$  line) is characteristic of a high-spin 3d magnetic moment.

This structure collapses at high pressure upon compression and then reforms upon pressure decrease [spectra with (r)]. The system is in the high-spin state at 36 GPa, in a high-spin low-spin mixture at 49 and 58 GPa, and in the low-spin state at 75 GPa. The pure low-spin component appears between 58 and 75 GPa.



## **MATERIALS SCIENCE GROUP**

## ID15 - Depth-Resolved Investigation of Friction Stir Welds using a Novel Strain and Phase-Mapping Technique

R.V. Martins Risø National Laboratory, Denmark V. Honkimäki ESRF

Friction stir welding (FSW) is a solid state welding process in which a spinning tool is forced along the joint line, heating the abutting components by friction, and producing a weld joint by large plastic mixing (stirring) of material from the two components. In the framework of the EU funded JOIN-DMC project (Joining Dissimilar Materials and Composites by Friction Stir Welding) the residual strains in FSW of dissimilar materials are investigated, applying for the first time a novel strain-mapping technique recently developed at ID15. The novel technique provides depth-resolved information on intensity and position of all the Bragg reflections emanating from the sample. The depth resolution is achieved by the use of a spiral slit, which consists of twelve equidistant concentric spiral apertures cut into a W plate. A sketch of the experimental setup is shown in Figure 3.

This technique allowed us to map the weld zone for the first time, in a non-destructively manner with depth-

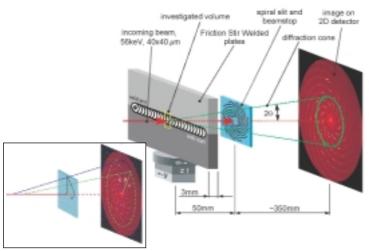


Fig. 3: Sketch of the experimental setup with spiral slit and online image plate scanner on ID15B. In the lower part of the figure the principle of the spiral system is shown with one of the twelve spiral cuts: The radii of the diffracted rays are measured as a function of the angle  $\varphi$ , which gives the radius of the ray at the position of the spiral slit. By knowing these two radii at the constant distance it is straightforward to get the origin of the ray and the diffraction angle  $2\theta$ , which gives the strain.

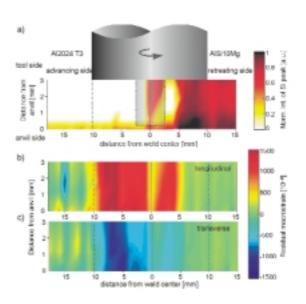


Fig. 4: a) Distribution of Si phase in the weld zone. For a better comprehension the welding tool is shown schematically, where the grey shaded area corresponds to the position of the tool pin that was plunged into the material during the weld process. b) Depth resolved residual macrostrains in parallel to weld in Al phase. c) Depth resolved residual macrostrains in across weld in Al phase.

resolution, material composition and strain distribution. The results presented here relate to a FSW between AA2024 (single phase Al alloy) and AlSi10Mg (an alloy containing 10 wt.-% Si particles). The signal from the Si phase can be used as an indicator for the mixing of the two welding partners as shown in Figure 4a. It can be observed that a significant amount of AA2024 is protruding in the AlSi10Mg side, while the AlSi10Mg material is dominant close to the weld centre. Large tensile strains in Al phase are observed in parallel to the weld line (Figure 4b) and across the weld line (Figure 4c) the residual strains are largely compressive. When comparing Figure 4a with 4b and 4c it can be seen that a close correlation exists between the strain distribution, the material distribution, and the tool position.

[1] The Welding Institute (TWI); W.M. Thomas, E.D. Nicholas, J.C. Needham, M.G. Murch, P. Temple-Smith, and C.J. Dawes: PCT World Patent Application WO 93/10935. Filed: Nov. 27, 1992 (U.K. 9125978.8, Dec. 6, 1991). Publ.: June 19, 1993. [2] D.P. Field, T.W Nelson, Y. Hovanski, K.V. Jata, Metallurgical and Materials Transactions A, 32A, 2001.

### MACROMOLECULAR CRYSTALLOGRAPHY GROUP

## ID14-2 - Crystal Structure of Human Cytochrome P450 2C9 with Bound Warfarin

P.A. Williams, J. Cosme, A. Ward, H.C. Angove, D. Matak Vinkovic, H. Jhoti Astex Technology, Cambridge, UK

Cytochrome P450 proteins (CYP450s) are membrane-associated haem proteins that metabolize physiologically important compounds in many species of microorganisms, plants and animals. Mammalian CYP450s recognize and metabolize diverse xenobiotics such as drug molecules, environmental compounds and pollutants. Human CYP450 proteins CYP1A2, CYP2C9, CYP2C19, CYP2D6 and CYP3A4 are the major drug-metabolizing isoforms, and contribute to the oxidative metabolism of more than 90% of the drugs in current clinical use. Polymorphic variants have also been reported for some CYP450 isoforms, which has implications for the efficacy of drugs in individuals, and for the co-administration of drugs. The molecular basis of drug recognition by human CYP450s, however, has

remained elusive. Here we describe the crystal structure of a human CYP450, CYP2C9, both unliganded and in complex with the anti-coagulant drug warfarin. The structure defines unanticipated interactions between CYP2C9 and warfarin, and reveals a new binding pocket. The binding mode of warfarin suggests that CYP2C9 may undergo an allosteric mechanism during its function. The newly discovered binding pocket also suggests that CYP2C9 may simultaneously accommodate multiple ligands during its biological function, and provides a possible molecular basis for understanding complex drugdrug interactions.

Nature 2003 Jul 24;**424**(6947):464-8. Epub 2003 Jul 13.

## SOFT-CONDENSED MATTER GROUP

## ID10B - Confinement of Biomaterials in Microfluidic Devices

T. Pfohl, A. Otten *University of Ulm, Germany* B. Struth, A. Snigirev, O.Konovalov *ESRF* 

The interactions of macro-ions with oppositely charged multivalent ions in aqueous solutions draw wide interest from biologists, chemists, and physicists. The investigations are mainly inspired by the fascinating macromolecule DNA: centimetre-long DNA-molecules are condensed into micron-size nuclei of eukaryotic cells with controlled localised DNA decondensation for an effective expression of genes. In first experiments at ID10B the time evolution of condensation reactions of biological macro-ions induced by multivalent ions could be investigated by X-ray small angle scattering. Using a micro-fluidic focusing device (Figure 5) the macro-ions could be oriented in the micro-focused liquid jet. The alignment of the material improved the characterization of these liquid crystalline-like materials (obtaining the lattice symmetry) with an excellent time resolution and the use of only minimal sample volumes.

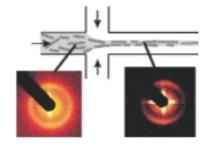


Fig. 5: Sketch of the alignment of an aqueous DNA-solution in a micro-fluidic focusing device. Real 2D-scattering patterns of the DNA-solution in the inlet channel and in the micro-focused liquid jet are also shown. Such first successful X-ray small angle experiments were carried out with a focused X-ray beam (in a first time use of Fresnel zone plate at the ID10B beamline [1], beam diameter at focus ~ 6 µm).

[1] B.Struth, A.Snigirev, O.Konovalov, A.Otten, R.Gauggel, T.Pfohl, Application of microfocussing at a none specific beamline, SRI 2003 proceedings, accepted, 2003.

## **News from the CRGs**

The British beamline for macromolecular biology, **BM14**, reports on the successful installation of their new 3x3 mosaic CCD detector delivered recently by MAR-USA. The detector has an active area of 225 mm by 225 mm making it one of the largest CCD X-ray detectors available for use by the protein crystallographers in Europe.

The installation and commissioning of the Spanish CRG's (BM16) experimental station for macromolecular crystallography has been completed within schedule. The result is a well performing and user-friendly beamline providing easier access to Spanish users since September 17. During the first run of operation, 8 proposals were allocated over 36 shifts. An increasing demand for beam time for research in the field of structural biology and genomics on this station is expected from the Spanish community based on the experience at BM14 during the past years. During the next run the end station for small angle scattering will be commissioned. First ESRF users on BM16 are expected in March 2004.



Fig.1: The XMaS in-vacuum slit system and how it can be 'vacuum integrated' with the 'tube slits' to form a beam collimator.

The British CRG for magnetic X-ray scattering (XMaS), BM28 further improved their experimental equipment. Efforts continue to reduce attenuation losses caused by air paths and vacuum closing screens. To this end a number of developments have been completed including a new in-vacuum slit assembly for use on the diffractometer  $2\theta$  arm, shown in Figure 1. The maximum opening aperture of this screen, formed from four tungsten blades, is 12 mm x 12 mm. Each jaw can be independently positioned to within 2 µm and all axes have limit switches to prevent mechanical damage. A cyberstar detector has been acquired, also for use on the  $2\theta$  arm, with a vacuum flange adaptation that allows integration of the detector into the same vacuum path—saving 2 kapton windows and an air gap.

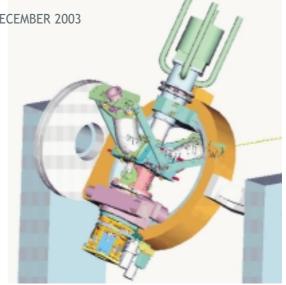
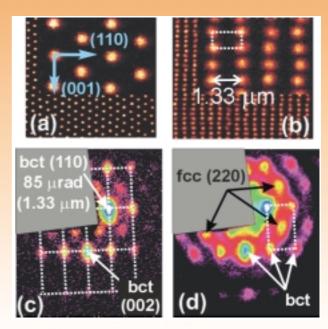


Fig. 2: An evacuated sample environment that incorporates a magnet and sample cooling from a displex that is mechanically isolated from the sample.

Progress has been made upstream of and, in some instances, around the sample for certain experimental setups in order to allow the application of a magnetic field at low temperatures and remove entry and exit window absorption from the beam path. An in-vacuum magnet-cryostat has been designed and made. The main body of the vacuum vessel is supported on the XMaS XYZ mount on the  $\chi$  circle to allow for precise sample alignment. To ensure efficient cooling, the coil of the electro-magnet is mounted ex-vacuum, below the vacuum vessel. The iron magnet yoke passes through seals in the base of the vacuum vessel. The tips of the adjustable magnet pole pieces, inside the vacuum vessel, support a copper sample stub via three ceramic balls to minimize thermal losses. An ARS DE202 displex is mounted on the Huber  $\chi$  cradle on rubber anti-vibration dampers and intrudes into the vacuum vessel through a bellows. A short copper braid connects the sample stub to the tip of the second stage of the cryostat. The maximum scattering angle allowed within the constraints of the bellows is 30 degrees in  $\theta$  and 60 degrees in  $2\theta$ . Figure 2 shows a schematic of this experimental setup.

A 100  $\mu$ m thick diamond has been purchased to produce circularly-polarised X-rays for low-energy experiments. It has been commissioned in an experiment with the energy set at the uranium M4 edge, 3728 eV. At this energy, it transmits 20% of the photons in contrast to only 1% for the 300  $\mu$ m thick diamond in BM28's possession. Integration of the phase plate into the vacuum leads to a further gain of two orders of magnitude in flux at these energies, *i.e.* more than 10³ flux gain overall.

Fig. 1: Body-centred tetragonal colloidal crystal: (a) A confocal laser microscope xy-image of a single hexagonal plane. (b) A z-average of lateral particle positions in four crystal planes. 11 keV µrad-XRD patterns of a crystal before (c) and after (d) drying and silicon infiltration. (a)-(c) are measured in the same crystal while for the sample used in (d) optical techniques are not applicable.



## Scientific article

# 3D STRUCTURE AND (DIS-)ORDER IN PHOTONIC CRYSTALS BY MICRORADIAN SYNCHROTRON X-RAY DIFFRACTION

A.V. Petukhov Van't Hoff laboratory, Debye Institute, Utrecht University, The Netherlands J.H.J. Thijssen, A. Imhof, A. van Blaaderen Soft Condensed Matter, Debye Institute, Utrecht University, The Netherlands

I.P. Dolbnya BM26 DUBBLE CRG, ESRF
A. Snigirev, I. Snigireva, M. Drakopoulos ESRF

-ray diffraction at microradian angles (μrad-XRD) is readily achievable at the ESRF and is able to characterise 3D structure and order of photonic crystals with a lattice spacing above one micrometre.

## Photonics: large-scale crystals

In photonic crystals the refractive index varies periodically on length scales comparable to optical wavelengths [1]. With a sufficient contrast and a proper 3D lattice a photonic band gap can be opened, which will allow one to manipulate light similar to semiconductors manipulating electrons. An important area of application is infrared telecommunications, operating at wavelengths of 1.3 and 1.5 µm, where silica fibres display high transport performance. Fabrication of such materials is challenging and one possible route is through self-organisation of colloidal particles. By tuning the interparticle interaction

potential, various lattices can be achieved [2]. By filling dried colloidal crystals with silicon and etching out the silica, 'inverted crystals' of silicon can be made, which have the desired band gap [3].

To check whether the photonic crystal still possesses the intended structure and a high degree of order, one can no longer rely on optical tools since the light/lattice coupling is too strong. X-rays are therefore one of the few tools, if not the only one, available to elucidate the internal 3D structure and order [4,5]. The issue addressed here is: can one further extend XRD to include crystals with spacing above 1  $\mu$ m? Moreover, can one exploit diffraction to probe order on distances of tens to hundreds of periods?

## Microradian 3D Crystallography

At **BM26B** (DUBBLE) X-ray crystallography methods were successfully applied to photonic super-micrometre

structures. An example is given in Figure 1c, which presents a diffraction pattern of a wet body-centred tetragonal (bct) crystal, which is self-assembled in an external electric field [2]. The rectangular arrangement of bright reflections (highlighted by white dotted lines) reflects the bct-stacking of close-packed layers (cf. panel 1b). By measuring patterns at different orientations, the full 3D structure was accessed. Panel 1d presents a diffraction pattern obtained from a similar bct crystal, which was dried and then filled with silicon. The rectangular arrangement of the strongest reflections is still recognisable, but reflections typical for close-packed (fcc, rhcp) structures [4,5] are also apparent. Furthermore, one can see about 6% reduction of the average period and a strong increase of the scattering background, which indicates creation of defects in the crystal. Moreover, one can detect broadening of the intrinsic  $2\sigma$  width of the crystal reflection to about  $0.6~\mu m^{-1}$  (or,  $11~\mu rad$  in angular terms). The latter, however, is at the edge of the resolution power of BM26B.

## Pushing the limit further

The middle sketch in 'Diffraction and Coherence' points out the bottleneck of the setup at BM26B. Due to beam focusing, the coherence properties of the X-ray beam degrade towards the sample position and  $l_{tr}$  shrinks to about 10 µm. A simple solution is to let X-rays freely propagate to the sample. With typical ESRF parameters  $(d/L \sim 10^{-6})$ , a transverse coherence length  $l_{tr}$  up to 50 - 100 µm can be reached, which is sufficient to determine the order parameters of photonic crystals in great detail. Figure 2 illustrates that a few microradians resolution can indeed be achieved [6]. The experiment was performed at BM05 using a test 2D Ni grid with a period as large as 12.5 µm.

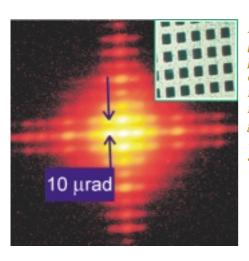
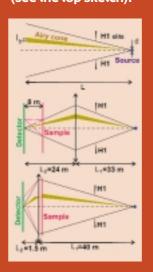


Fig. 2: 10 keV µrad-XRD pattern and optical microscope image of a Ni grid with 12.5 µm pitch and 5 µm bar.

### Diffraction and Coherence

Diffraction appears as a result of interference of scattered X-ray waves, which must be coherent with each other. This can be fulfilled easily in the longitudinal direction (i.e., along the beam) for a small diffraction angle [5]. Troublesome are the random fluctuations of the phase front in the transverse direction, which must be much smaller than the tiny X-ray wavelength  $(\lambda \sim 1 \text{ Å})$  in order to probe positional correlation of scattering objects. A freely-propagating X-ray wave is coherent within the so-called Airy cone of size  $l_{tr} = L\lambda/d$ (see the top sketch).



To resolve diffraction at µrad angles, one has to focus the beams at the detector. As illustrated in the middle panel, at BM26B the focusing element is far upstream from the sample. Another scheme utilising a compound refractive lens [7] with a much shorter focal length was used in the experiment at BM5 as sketched at the bottom.

To summarise, our results demonstrate that µrad-XRD is readily achievable at the ESRF even without a Bonse-Hart camera, which is typically used in an ultra-small-angle X-ray scattering (USAXS) setup. µrad-XRD was shown to fully cover the needs of photonics and to yield a wealth of information on the 3D structure and (dis)order.

The authors would like to thank D. 't Hart, K. van der Werf, R. Schropp (UU), Y. Jun, and D. Norris (Univ. of Minnesota, USA) for their contributions to sample preparation, H.N.W. Lekkerkerker, G.J. Vroege (UU) and W. Bras (ESRF) for inspiring discussions, J.P. Hoogenboom, D. Detollenaere, J. Jacobs, J. Hoszowska and J.-Y. Massonnat for technical support during the experiments at BM26B and BM5.

[1] J. D. Joannopoulos et al., Photonic Crystals: Molding the Flow of Light (Princeton University Press, Princeton, 1995).

[2] A. Yethiraj and A. van Blaaderen, Nature, 421, 513 (2003).

[3] Y. A. Vlasov et al., Nature 414, 289 (2001).

[4] M. Megens et al., ESRF Newsletter 29, 8 (1997); W. Vos et al., Langmuir 13, 6004 (1997).

[5] A.V. Petukhov et al., PRL, 88, 208301 (2002); PRL, 90, 028304 (2003); ESRF Highlights 2002, p. 19.

[6] M. Drakopoulos et al., submitted to PRL (2003).

[7] A. Snigirev et al., Nature, 384, 49 (1996); B. Lengeler et al., APL, 74, 3924 (1999).



## Visiting a beamline

# ID08: MULTITECHNIQUES IN A MULTINATIONAL BEAMLINE

he Dragon Beamline or ID08 is probably one of the richest in techniques at the ESRF. With five endstations for different soft X-rays techniques, ID08 investigates the magnetic and electronic properties of nanoclusters, ultrathin films, superconductors and semimetals. And it has a very intense scientific activity.

They come from France, Italy, Romania, Slovakia, Sweden, United Kingdom and soon Brazil. This is not a new committee of United Nations, but the international team of ID08, a two years old soft X-rays beamline. One of the main attractions of ID08 is that it features almost as many techniques as nationalities, and this seems to work fine: "ID08 is reasonably productive in the number and quality of papers published", says the scientist Nick Brookes.

ID08 has an intense source of polarised soft X-rays used to probe magnetism in a diverse range of systems with X-rays magneto-optical techniques and to study the electronic structure of materials using X-rays and photoelectron emission techniques. Indeed, users have a very diverse and specific choice of techniques to study their samples: X-rays magnetic circular dichroism, spin-polarised X-rays photoelectron spectroscopy, soft X-rays resonant magnetic scattering, soft X-rays angle resolved and high energy resolution photoelectron spectroscopy and X-rays emission spectroscopy. "We work in four different areas", explains Nick Brookes, "all of them are quite different, but at the same time, they are complementary". Each of

The ID08 team: (from left to right) Gilles Retout (technician), Federica Venturini (PhD), Stefan Stanescu (postdoc), Celine Denadai (postdoc), Kenneth Larsson (engineer), Peter Bencok (scientist) and Nick Brookes (scientist).

the scientists on the beamline is specialised mainly in one of the techniques, so everyone is quite essential. This is maybe one of the reasons why this beamline receives many more proposals than it can handle. This makes the Dragon Beamline very busy. Busy not only running experiments, which normally last six days, but also doing the setting up in the endstations. In fact, for some experiments, such as the ones on surfaces, one needs to start the preparation of samples a week before the experiment begins. So it often happens that a group prepares the setting up while another is working. Some users bring their own instruments, others use those of the beamline. A group from the Dipartimento di Fisica del Politecnico di Milano (Italy) has its own instrument (AXES) in the beamline. It is also open for general users.

This frantic activity leaves the team little time to publish their own scientific papers. "If we were a bigger team, we would find more time to sit in our offices and write our own papers", explains Nick Brookes. Even though a small team, the seven people on ID08 get on very well. They have a beamline meeting every fortnight, but they see each other every day for lunch. It is the perfect occasion to discuss any issue about work, or any other subject, such as the latest results in soccer or international news.

M. C.

## The reason for Dragon

ID08 is one of many Dragon soft X-ray beamlines in the world. Its name is inspired by the Dragon synchrotron beamline on the VUV ring at the National Synchrotron Light Source (NSLS) in Brookhaven National Lab, USA. The Dragon was the first soft X-ray monochromator delivering resolving power above 10,000 - it opened the very productive field of high energy resolution soft X-ray spectroscopy, and its flexible and modular design allowed the first soft X-ray magnetic circular dichroism measurements. The choice of a Dragon design for ID08 was influenced by energy resolution issues and reliability of operation with variable polarisation soft X-rays radiation. The NSLS Dragon was constructed in 1987 by the present Director of the National Synchrotron Radiation Research Centre, in Taiwan, Chien Te Chen, and by Francesco Sette, today at the ESRF. In those days, Nick Brookes was working on the neighbouring beamline.



Luciana Cappello, PhD student on ID01, shows her poster to Jürgen Härtwig.

## STUDENTS DAY: PHD STUDENTS SHOW THEIR WORK TO THE OTHER ESRF SCIENTISTS

hD students had the opportunity of getting together with the rest of the scientists at the ESRF and learning from each other in the first edition of the Students Day at the ESRF. More than 100 participants found out about the science done by those at the first stages of their scientific career thanks to talks and posters.

There are 55 PhD students at the ESRF, ten of them from the Collaborating Research Groups (CRG). You can spot them easily, they are the often stressed young people that look haggard, not because of an excess of partying, but because of long periods of time dedicated to the development of their theses. This autumn, for the first time, a day was dedicated exclusively to them at the ESRF.

The Students Day offered the best scenario for the PhD students to present their work and provided a platform for a lively scientific exchange amongst colleagues. The main features of the day were 14 talks from students. The Heads of each scientific group gave a short

presentation of the group and some highlights of the talks. As the ESRF covers so many fields, the talks went from the studies of eye lenses, to the deformation mechanism of polymers, the reason for the beginning of corrosion or how to fight brain tumours with Synchrotron Radiation. All these exciting subjects led to a great interaction between the audience and the speakers.

Apart from the talks, over thirty posters filled the entrance hall of the Central Building with research results and questions whose answers will soon fill the pages of much longed-for theses. The PhD student Daniel Sapede, who divides his working time between the ILL (Institut Laue Langevin) and ID13, was awarded

for his poster entitled 'Structure and Dynamics of Spider Silk: A neutron and X-ray scattering study'. He received the book 'Elements of Modern X-ray Physics', by Jens Als-Nielsen and Des McMorrow, and a bottle of champagne. Michael Krisch, organiser of the event, was very satisfied with the participation of the students: "All the talks and posters were extremely carefully prepared and showed high quality".

M. C.

## EXPERTS ON HARD X-RAY PHOTOELECTRON SPECTROSCOPY SHARE THEIR EXPERTISE

ard X-ray PhotoElectron Spectroscopy (HAXPES) is a new technique that is only just beginning to be exploited. In mid September, 62 experts from eleven different countries met for the first time and shared their knowledge and experience in this field at the HAXPES-2003 workshop, hosted by the ESRF.



The 'family' picture.

At the ESRF Experiments Division Science Days in Annecy in the year 2000, Hard X-ray PhotoElectron Spectroscopy (HAXPES) had been adopted as one of the issues for the ESRF Medium Term Scientific Programme. Currently, there is one beamline at the ESRF, which uses this technique: ID32. ID16 is planning experiments in this field starting February 2004. Worldwide activities in this area are still incipient and there are only a few papers published. The workshop at the ESRF showed that the field is nevertheless in a state of a dynamic development aiming at bulk sensitive XPS studies with electron energies beyond 10 keV and meV resolution. Thus, the vast majority of the presented work was brand-new and unpublished.

The different contributions illustrated well the major advantages for material science and basic physics research as well as the challenges of the HAXPES technique. At present, the probing depth of about 10 nm and a spectra resolving power of 30000 are limited only by the available spectrometers.

The talks were followed by lively and fruitful discussions, which continued during the two poster sessions. Issues such as cross-sections, angular resolution, multipole contributions, as well as possibilities and difficulties in the spectrometer design were intensely debated. The award for the best poster was given to Sebastian Thiess, PhD student on ID32, for his poster entitled 'Hard X-ray Photoelectron Spectroscopy up to 14.5 keV on Au and C and YBa $_2$ Cu $_3$ O $_{(7-\delta)}$  single crystals'. The jury consisted of Charles Fadley, University of California Davis (USA), Stefan Hüfner, Universität Saarbrücken (Germany), and D. Phil Woodruff, University of Warwick (United Kingdom).

The workshop was very enriching and stimulating for the HAXPES community. This positive feeling led some colleagues from Japan, where significant activities in this field are evident, to volunteer to organise the second HAXPES workshop in Japan, probably in two years time. •

JÖRG ZEGENHAGEN



The poster session.

