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].

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References

[1] C. Gourier, J. Daillant, A. Braslau, D. Luzet, C. Blot, G. Grübel, J.F. Legrand, G. Vignaud, M. Alba, F. Rieutord and A. Gabriel, ESRF Newsletter 25, 34-37 (1996).

[2] W.H. de Jeu, E.A.L. Mol, G.C.L. Wong, J.M. Petit and F. Rieutord, ESRF Newsletter 29, 10-12 (1997).

[3] F. Rieutord, R. Simon, R. Conradt and P. Müller-Buschbaum, Europhys. Lett. 37, 565 (1997).

[4] see ESRF highlights 1995/1996, p33 and 35.

[5] An article on this subject will be found in the forthcoming publication of ESRF highlights.

[6] H. Bulou, A. Barbier, G. Renaud, B. Carrière, R. Baudoing-Savois and J.P. Deville, Surf. Sci. 377, 90 (1997).

[7] O. Robach, G. Renaud, A. Barbier and P. Guénard, to appear in Surf. Rev. Lett. (1997) and O. Robach, Phd thesis, University of Grenoble, 1997.

[8] P. Guénard, G. Renaud and B. Villette, Physica B 221, 205 (1996) and P. Guénard, PhD thesis, University of Grenoble, 1996.

[9] A. Barbier and G. Renaud, to appear in Surf. Sci., 392 L15-L20 (1997).

[10] Y. Soldo, J.L. Hazemann, D. Aberdam, E. Pernot, M. Inui, J.F. Jal, K. Tamura, J. Dupuy-Philon and D. Raoux, J Phys. IV, C2-983 (1997), and accepted in Phys. Rev. B.

[11] C. Revenant Brizard, J.R. Regnard, J. Mimault, D. Duclos and J.J. Faix, J. Phys. IV, C2-325 (1997).

[12] C. Revenant Brizard, J.R. Regnard, J. Mimault, O. Proux, B. Dieny and B. Mevel, J. Phys. IV, C2-1111, 1997.

[13] C. Meneghini, M. Maret, M.C. Cadeville and J.L. Hazemann, J. Phys. IV, C2 1115-1117.

[14] A. Manceau, M. C. Boisset, G. Sarret, J.L. Hazemann, M. Mench, Ph. Cambier, R. Prost, Env. Sci. & Technol., 30, 1540 (1996) .