ESRF	**UPGRADE PROGRAMME – PHASE I** **CONCEPTUAL DESIGN REPORT**

Beamline for surface and interface science	
Current designated sector:	Facility goes to:
ID03	ID10B

1.1 ID CARD

ID03 is dedicated to the characterisation of the structure and the morphology of surfaces. It gives the possibility of performing surface crystallography, grazing incidence small angle scattering, coherent diffraction imaging and photocorrelation measurements. Studies can be performed in situ and in real time. Typical scientific fields concern the growth of advanced materials and the relation between their properties and their structure and/or morphology. One important activity deals with the study of catalytic properties in real pressure/material conditions. The submitted proposals are mainly from the surface and interface community, with an increasing number covering chemistry and soft matter.

1.2 SCIENTIFIC CASE

The first surface X-ray diffraction (SXRD) experiments were carried out in the 1980s with the aim of solving the three-dimensional terminating structure of solids or determining the adsorption geometry of atoms deposited under UHV conditions.

This technique made an enormous progress with the development of dedicated X-ray synchrotron sources, because of the increase in the available flux and collimation which allows shorter acquisition times and larger signal to noise ratios. The development of the instrumentation came together with advances of the theory resulting in the possibility of solving complex systems with a large number of atoms in each unit cell. In the meantime the concept of “surfaces” evolved from the classical solid/UHV interface to more complicated systems such as liquid-solid and solid-solid interfaces.

The present surface diffraction beamline optics has been updated during 2006 in all its components gaining in stability, energy tuning, focalisation and flux. The scientific fields, covered by the beamline, have grown in time and for the middle and long term plans we intend to further develop the existing instrumentation in order to cover the following scientific topics:

Surface crystallography under UHV, and not UHV conditions. The characterisation of a crystallographic structure termination or the adsorption geometry of atoms and molecules is fundamental for the comprehension of the system properties. Surface X-ray crystallography is a unique tool giving the possibility to determine the detailed structure of a buried interface, which is not achievable with other techniques. The complexity of the studied system is growing with time and it is now possible to solve ordered surface structures involving a large number of atoms, as in the case of fullerene molecules on noble metal surfaces where the results gave unique information on the adsorbate-surface bonding and on the deformation induced on the substrate (Felici, 2005). In the future this technique will be mainly applied to solve the structure of complex molecules on surfaces and to highlight the role of the interaction between the substrate and the adsorbate in the formation of the new system. For this purpose we need a specialised chamber where it is possible to handle organic compounds.

Moreover, in order to be able to respond to the demand of large communities such as those dealing with oxides and semiconductors, we need specialised chambers where they can characterise their systems and reproduce the growth conditions they have in their own laboratories.

Together with the development of specialised investigation chambers we shall extend the use of 2D pixel detectors which enormously reduce the acquisition time giving the possibility of measuring large data sets in a short time.

Time dependent phenomena at surfaces are a class of experiment where it is crucial to measure the evolution of the surface structure together with its morphology during a chemical or physical process (e.g. growth or corrosion or tribology). For this purpose we will develop new data acquisition methodologies based on the simultaneous use of several area detectors, which is an effective way to acquire more useful information in a limited time. The experiments will require a simultaneous recording of diffraction intensities, providing the structure of the surface, together with grazing incidence small angle scattering giving information on the morphology of the surface. New classes of experiments will then be possible, such as the study of phase transition between two surface phases, characterisation of the dynamic friction between two solids (Rubinstein, 2004), initial stages of corrosion and the evolution of surface nanostructures. The time resolution we plan to achieve with the new beamline should extend down to 0.1 s or less.

Amongst the processes, which require a real time and in situ analysis, we shall continue providing instrumentation and support for the characterisation of **reactions at surfaces**, which occur only at well defined thermodynamic conditions. Recent technical developments have made it possible to overcome the so-called pressure gap allowing surface X-ray diffraction (SXRD) to be used to understand the role of the active catalysts. Time dependent SXRD experiments showed the crucial active role played by the metal oxide during the real working conditions of the catalytic reactions, whilst the oxide was previously believed to be a pollutant and inhibitor of the reaction (Ferrer et al, 2007). With the present upgrade we shall install a new vertical diffractometer where it will be possible to precisely manipulate large and heavy specific equipment. This will give us the opportunity of enlarging the range of reactions which can be studied and of reaching reaction conditions more similar to those in industry in order to understand the reaction at an atomic level and then to optimise the catalyst performance and life time. For this same reason we shall optimise the instrumentation in order to be able to study catalysts based on nanomaterials in porous oxides.

Reduced dimensionality objects have structural, electric and optical properties different from the homologous bulk objects. For instance nanodots of transition metals composed of a few tens of atoms generally have a first neighbour distance which is 10 to 20% smaller that the corresponding infinite bulk structure (Mironets et al, 2008). For a better control of their properties it is necessary to correlate their structural properties with their morphology. Moreover, the growth of these systems is still carried out in a quasi-empirical fashion because of the limitations encountered by using standard laboratory techniques in following the morphology or the structure in real time. Morphology is normally determined by scanning probe microscopy which cannot be generally defined a real-time technique, whilst the structure of the deposited material is followed by RHEED analysis, which only works for perfectly crystalline materials. The new surface and interface beamline must be able to provide the user community with instrumentation where it is possible to follow the growth of low dimensionality structure in situ in order to correlate the structure at an atomic level with the morphology and, possibly, with the peculiar characteristics of the system (luminescence, magnetic properties, etc.). For this purpose a new experimental chamber directly coupled with the beamline vacuum will be realised. This will minimise the scattering from any window crossed by the X-ray beam, which is in turn the limiting factor to study overlayers with a nominal thickness of few hundredths of a monolayer.

1.3 PROJECT HISTORY

The ID03 surface diffraction experiment was one of the first ESRF beamlines open to the user community and started its activity at the end of 1993.

The future of a dedicated beamline to the surface structure characterisation was deeply discussed during early 2004 and led to the organisation of a specific workshop held at the ESRF during September 2004. The European scientific community was very clear in demanding the refurbishment of the beamline and the SAC in November 2004 endorsed this request. A full refurbishment of the beamline was carried during 2005 and 2006.

The main needs of the users have been summarised in the SURF CDR of the Purple Book and the present document follows the main guidelines already presented and discussed by the user community.

At the moment the beamline is a reference in the surface science X-ray community. Its upgrade described below takes into account the users' needs which can be foreseen for the years to come.

1.4 BASIC TECHNICAL CONSIDERATIONS

The basic structure of the beamline will not differ substantially from the newly refurbished ID03. It will be equipped with two hutches in a row. The incident beam energy will be between 5 and 35 keV. The beamline must be windowless to limit the background and to preserve the coherence of the source. This implies the front end must be UHV.

The ID10 sector must be a 7 m long canted undulators section allowing the use of two standard length undulators. The choice of the period and technology (i.e. standard, revolver or in vacuum) will be made with the Insertion Device Group. At present two U22 or U20 undulators seem to be the most appropriate choices.
The typical focus size will be tuneable depending upon the experiment and ranging from a focal spot of about 50 microns with a flux exceeding 10^{14} ph/s in the case of diffraction experiment, when the sphere of confusion of diffractometer is going to be the limiting factor, to 1 micron or less in the case of nano-imaging at surfaces. In order to achieve these numbers we need to slightly modify the present ID03 monochromator in order to install a multilayer, and to substitute the ID03 main mirrors because of the different focal length of the new beamline.

Both hutches will be equipped with high resolution diffractometers. In the first one we plan a new vertical geometry station with decoupled sample and detector movements. In the second hutch we shall install the current diffractometer coupled with new UHV systems. Both hutches will also be equipped with 2D pixel detectors for diffraction and GISAXS experiments.

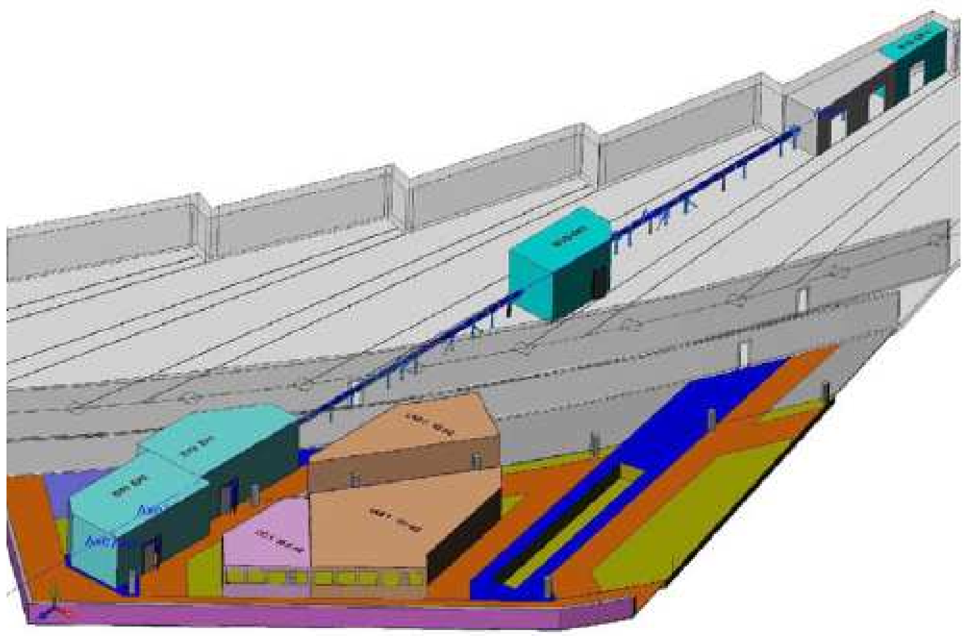
The available techniques at the beamline will be: surface diffraction, grazing incidence small angle scattering, anomalous diffraction, coherent diffraction imaging and photocorrelation spectroscopy.

The uniqueness and speciality of the beamline is the in situ preparation and characterisation of the systems under study. For this reason, both experimental apparatus will be able to host user chambers. The diffractometers must have enough flexibility in order to be able to align samples with a sub-micrometric precision.

Modern surface science needs the correlation of surface characteristics such as magnetic properties, optical activity, friction, reactivity, etc, with its structural and morphological properties. With this aim in mind we need to foresee useful complementary techniques in the experimental chambers and a side laboratory where the experiments can be carefully prepared and samples can be characterised in detail before and after the experiments.

In particular for the flow reactor in EH1, we plan to have an in situ AFM and an in situ IR spectrometer for the characterisation of the surface morphology and of the molecules bonded to the surface whilst we do the X-ray diffraction. In the UHV chamber in EH2 we would like to have a small SEM (complete SEMs, which can be mounted on a CF63 flange, are nowadays available) and a new electron analyser. In the side laboratory we need UHV chambers where samples can be prepared before the experiments and characterised after the measurements. For most of the needs we will use the existing chambers but we strongly need a dedicated UHV STM interfaced with our sample holder standard where the sample surface structure can be easily imaged. The specifications of the new experimental chambers and beamline side facilities will be decided jointly with the user community.

A layout of the beamline is shown in the following figure. We have tried to minimise the cost in reusing the existing hutches of the Troika C beamline.



1.5 REFERENCES

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