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

Powder diffraction	
Current designated sector:	Facility goes to:
ID31	ID31

1.1 ID CARD

ID31 investigates the crystal and micro-structures of materials prepared in either powder or polycrystalline form. The submitted proposals cover mainly chemistry, hard condensed matter (crystals and ordered systems) and applied materials and engineering. Typical scientific questions concern the characterisation of advanced materials with magnetic, superconducting, catalytic or hydrogen-storage properties. Systems in the realms of solid state chemistry, advanced aerospace alloys or even structural biology are also studied.

1.2 SCIENTIFIC CASE

ID31 delivers very accurate high resolution powder diffraction data for the study of powders, polycrystalline and amorphous materials, thin layers and coatings, etc. over the energy range of 6–61 keV with a wide range of sample conditions, e.g. temperatures from 3K to 1600ºC. There is also a programme of residual strain mapping in engineering components, particularly made from Ti and Al alloys used in aerospace applications, and from thin steel. The diffraction patterns from ID31 have high angular accuracy, owing to the diffracted-beam analyser crystals which eliminate aberrations due to sample positioning and beam penetration that affect conventional diffraction geometries with a scanning slit or position-sensitive detector. In the latter, the 2θ angle is inferred from the position in space of the slit or pixel, whereas an analyser crystal defines a true angle as the X-rays transmitted must satisfy the Bragg condition at the crystal's surface. The analyser crystals, coupled with the highly collimated incident beam and the mechanical integrity of the diffractometer, also define the high angular resolution of the beamline, with an instrumental contribution to the peak widths of $\approx 2 - 3\times10^{-3}$ ° 2θ. The resolution ($\Delta d/d$) is almost independent of the X-ray energy, so that high-resolution studies can be carried out at hard energies above 30 keV on a wide range of absorbing materials in transmission mode, especially metallic and inorganic systems. The use of a spinning capillary sample in transmission for powder diffraction studies greatly reduces problems of texture and preferred orientation thus yielding accurate diffraction intensities. Working at harder energies also allows the acquisition of data to high values of *Q*. This is advantageous for practically all crystalline substances, and also for the analysis of poorly crystalline materials via the atomic pair distribution function (PDF), a technique of growing importance. PDF analysis can be used to characterise the structures of materials that are far from ideal with regard to their crystallinity, e.g. amorphous, highly-defective and nano materials, many of which are of practical as well as scientific relevance.

Thus ID31 is a versatile instrument whose scientific programme can be classified into:

Structural studies: the solving and refining of crystal structures, exploration of the structure of glasses, and atomic pair distribution function (PDF) analysis;

In situ studies: materials evolving with temperature, time, voltage, etc. during phase changes, solid-state chemistry, electrochemistry, etc.;

Anomalous scattering: distinguishing between neighbouring elements in a material, via the K or L edge (sometimes both) of all elements above Ti;

High throughput studies: involving many samples, synthesised with different compositions, or under varied preparation conditions, etc. A sample-changing robot allows automated screening of up to 50 capillaries in succession, and is compatible with studies in the temperature range 80K–1250K;

Quantitative analysis: owing to the high angular resolution, diffraction patterns from complex mixtures with many contributing phases can be analysed; moreover, with the excellent statistical quality and low background, the detection of phases present in very low proportions is possible;

Microstructure: detailed analysis of peak shapes yields information about the microstructure or chemical homogeneity (e.g. David et al, 2007) of a material. Since the instrumental contribution is so small, the observed peak shapes are dominated by the sample;

Residual strain: measurements of residual strain, either by the $sin^2\psi$ technique, or by mapping peak positions from within the bulk and surface of a sample, via a gauge volume defined with slits and the angular acceptance of the analyser crystal;

Grazing incidence and reflectivity: measurements from thin films and surfaces.

The evolution of the beamline entails:

1) Reinforcement of its high-energy capabilities, providing significantly more flux above 30 keV, with the option to push the upper energy range further, towards 90 keV. This will improve the instrument's performance and time resolution in the energy range where the majority of experiments are already carried out, enhance the acquisition of data for high quality PDF analysis, and extend the range and thickness of materials that can be analysed for strain mapping. With 90 keV, access to the K edges of elements in the third transition-metal series (e.g. W, Au and Pt with important technological properties) will then be accessible for anomalous diffraction and PDF studies; currently we are limited to the low-energy L edges with the associated problems of absorption. More flux above 40 keV range would also greatly improve measurements at the lanthanide K edges, where we have needed very long scan times when carrying out anomalous scattering studies on lanthanide-containing glasses which have important applications.

2) A second theme involves improvements to the high-resolution detectors to improve peak shapes, angular and time resolution, thus allowing faster processes to be investigated, and higher throughput, at high angular resolution. An upgrade of the diffractometer is also foreseen to enable larger samples, faster scanning, and more complex sample environments to be mounted, as well as the heavier detector systems.

ID31, operating at hard energies and with its very high angular resolution, is complementary to the capabilities of ID11, ID15 (current and future high-energy, white beam instrument), and to the future UPBL2.

1.3 PROJECT HISTORY

The original high resolution powder diffraction beamline at ESRF (Fitch, 2004?? 1994?) was built on a bending magnet, BM16, and had an upper energy of 40 keV. The beamline review of May 2000 recommended the transfer of the beamline to an undulator to exploit the one to two orders of magnitude (depending upon energy) improvement in flux that would result from a horizontally-collimated beam, and an increase in the operational energy range to 60 keV. ID31 welcomed its first users in June 2002, using the original diffractometer from BM16. The beamline optics were redesigned (and simplified) for the undulator source, originally planned as two in-vacuum undulators, but finally settling on three 11-mm-gap in-air undulators owing to constraints at the time on the installation of in-vacuum devices around the storage ring. In the Purple Book the CDR POW outlined the development of ID31 over the medium term. The ideas presented there were endorsed by the beamline review of November 2007 and are closely followed in the current document.

1.4 BASIC TECHNICAL CONSIDERATIONS

The evolution is foreseen as follows:

1) Provision of at least one in-vacuum undulator. With a 6 m straight section one of the U32 in-air undulators is replaced with an in-vacuum U22. Thus the source will be one in-air U35 + one in-air U32 + one in-vacuum U22. This will increase the hard-energy performance significantly whilst maintaining tunability throughout the current energy range. A further evolution could involve replacing the two remaining in-air undulators with a second in-vacuum device, depending on how user demand for different energy ranges evolves. The Pb hutch is already thick enough to allow this.
2) Axially-resolving detectors. Detection efficiency could be increased by an order of magnitude by widening the axial (horizontal) acceptance of the detectors behind the analyser crystals. Normally the peaks would become broadened and asymmetric on the low-angle side of the peaks at low 2θ as a result of the curvature of the Debye-Scherrer cones. However, by using a horizontally-mounted one-dimensional PSD, the axial position of each arriving photon is measured, (so its position around the cone is known), allowing its true 2θ position to be determined. In this way, as well as increasing counting efficiency, resolution and peak shapes are improved as compared to the 4-mm-wide receiving aperture used today. The challenge here is to have linear photon-counting PSDs, with a spatial resolution of around 500 – 1000 µm, ≈50 mm wide, sensitive to the hard energies used on ID31, thus made in GaAs or zinc-cadmium telluride, etc. (Medipix 3?). The solution already exists for operation up to 20 keV with the Si-based Mythen detector from Dectris. This low-energy option would be very useful for studying radiation-sensitive samples such as organometallics, pharmaceutical molecules and proteins, which are also studied at present on ID31.

3) To take the extra weight of the new detectors a replacement, stronger, faster, though equally accurate, powder diffractometer is required. The current

diffractometer has operated reliably since 1996, but is limited in its load-carrying capacity and scanning speed, which will be insufficient for the increase in throughput and efficiency provided by the new detectors.

4) White-beam refractive-lens transfocator. In the Purple Book, when the location of beamlines was not yet fixed, it was noted that high resolution powder diffraction might be better on a high-beta sector of the ring. Given that it will remain at ID31, which is a low-beta sector, improved horizontal collimation could be obtained by refractive lenses before the monochromator via a transfocator, as on ID11. Moreover, the possibility then of focusing at hard energies would improve the spatial resolution in strain-mapping experiments, currently limited to around 200 μm horizontal by slitting down the beam. Focusing at hard energies was envisaged in the original ID31 design and the space needed is reserved in the optics hutch.

5) ID31 has no dedicated lab space, apart from one of the control cabins set aside for capillary manipulation and mounting. With users currently running tens, sometimes hundreds, of specimens for an experiment, conditions are far from optimal. ID31 shares the materials science group lab, on the opposite side of the ring near ID15, but this is too distant and unsuitable for intensive work. Thus the provision of a 15 – 20 m^2 sample preparation and manipulation laboratory near the beamline is essential.

6) The beamline review of 2007 recommended the acquisition of a two-dimensional detector which, coupled with improved hard-energy performance, would be used for fast PDF measurements, where the whole dataset is measured in a single shot, rather than by scanning the detector bank (as at APS). In this respect the beamline would then offer capabilities for both "quality PDF" and "fast PDF" experiments. Thus an area detector such as the Pixium or similar is a further option in the evolution of the beamline. A curved, one-dimensional PSD over 90° 2θ, efficient for hard radiation, might be another option.

7) With the improved high-energy capability, allowing penetration through greater thicknesses of metals, it will be possible to study larger components for residual strain mapping. Thus is could be advantageous to construct a single-detector vertical-axis diffractometer dedicated to these studies, able to accommodate larger and heavier samples.

8) Partnership for materials science, Fame38. A partnership for materials science with ILL, perhaps also incorporating the Fame38 facility in support of engineering strain scanning, etc., would enhance the complementarity between hard X-rays at the ESRF (ID31, ID11, ID15 white beam and UPBL2) with neutrons, for a wide range of studies (engineering, strain scanning, low temperature magnetic ordering, complex sample environments and in situ studies, to mention but a few). [see the CDR for UPBL2]

1.5 REFERENCES

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