



Materials science	
Current designated sector:	Facility goes to:
ID11	ID11

1.1 ID CARD

ID11 performs experiments across the range of materials chemistry and physics, using primarily diffraction methods to investigate the structure of materials over many length scales, from charge densities to the microstructure of bulk materials. The high X-Ray energy and high flux of ID11 permit experiments requiring high time and/or spatial resolution, via the use of fast detectors and micro- or nano-focusing optics. Typical issues addressed include fundamental crystallographic problems, properties of mechanical materials, and materials of interest to the energy economy, such as batteries, fuel cells and gas-storage compounds.

1.2 SCIENTIFIC CASE

The scientific programme at ID11 covers a large range of research fields and several different experimental techniques, although diffraction experiments are predominant. Most experiments probe materials' responses to outside conditions and are thus carried out in situ, with time resolution ranging from milliseconds to hours. Our current aim is to carry out the various classes of experiments *simultaneously* on a single sample, thus obtaining a complete picture of the sample over several length scales in terms of diverse characteristics, such as orientation, strain state, stoichiometry, etc. This we refer to as **simultaneous hierarchical characterisation**.

The high flux, high energy and focusing capabilities at ID11 allow several different classes of experiments to be performed. **Single crystal studies** fall broadly into two areas: structure solution and/or refinement of complex microcrystals, with up to 5000 refinable parameters, e.g. (Barboiu et al, 2005), and studies of fine structural details such as spin or oxidation state ordering, e.g. (Blake et al, 2001), or charge density studies. Our current aim is to be able to perform such studies on polycrystalline samples via methods we are developing to extract single crystal quality data from polycrystalline samples (Vaughan et al, 2005).

Whereas fast reversible reactions may be studied by stroboscopic techniques, most phenomena of interest to materials science are irreversible, and require one shot techniques to be studied. **High time resolution studies** of samples under the

influence of heat, pressure, chemical potential, etc. For the highest time resolution (currently ~1 ms) or for poorly crystalline samples, e.g. (Yavrai et al, 2005), powder diffraction methods must be used. Higher precision single grain isolation methods may be applied when slower (i.e. minutes) time resolution is satisfactory (see, (Juul Jensen, et al 2006), for a recent review of the methods we have developed).

We therefore propose to continue the evolution of the beamline to address several scientific themes. Development of **simultaneous hierarchical characterisation** has been considered to be the top priority since the beamline review of 2003; this axis was further strengthened by the recommendations of the latest review panel. We now carry out experiments which simultaneously probe length scales from Ångstroms (crystallography) to mm (sample size) by using multiple detectors. Crystal structure, microstructure, orientation, position and shape can be simultaneously characterized for polycrystalline samples, giving the actual distribution (rather than just averages) of these properties in the sample, as well as the relationships *between* grains, and their time evolution.

In situ chemistry/materials science experiments are carried out using powder diffraction when very high time resolution (down to ms) is required, or if the samples are amorphous or nanocrystalline. Many of our research programmes, for example on hydrogen storage materials, batteries and fuel cells, are linked to the high priority evolving energy economy. Heightened performance of such systems often relies on subtle structural features and as such is ideal for study at the ESRF.

Demand for **PDF** experiments is growing rapidly on ID11. Complimentary to the high resolution PDF experiments performed on ID31 (and similar to the case on ID15), we carry out mainly time-resolved local structure studies using two-dimensional detectors, including experiments in extreme conditions with levitated samples with very high time resolution.

Other spatially resolving quantitative diffraction imaging methods such as Topo-tomography and extinction contrast tomography (ECT) (King et al, 2008) are means of extracting high quality grain maps from well-crystallised materials, adding another tool to the box of available techniques. The ongoing diffractometer upgrade will allow both of these techniques to be practiced in a standard way.

There is a growing demand for **ultra-high precision crystallography measurements**, such as electron density studies, perturbation crystallography, very accurate measurement of displacement factors or diffuse scattering analyses. Ongoing upgrades on the beamline will make it fully adapted to performing these studies. The particular conditions for ultra-high resolution crystallography are stable and homogeneous flux at high energies, without (extreme) focussing. These are the same needs as the PDF and ECT techniques described above, as well as of other methods practiced on ID11 such as X-Ray holography (Kopecký et al, 2008).

1.3 PROJECT HISTORY

The current CDR is essentially an extension of the MATSCI CDR in the Purple Book, extended to include recent developments in ultra-high resolution crystallography and related fields stemming in large part from the last beamline review in October 2008. ID11 was the first beamline proposed to be extended, and

has been a pilot project for the upgrade in general. The timeline for the upgrade is given in §1.6.

1.4 BASIC TECHNICAL CONSIDERATIONS

For several years we have been in the process of a major upgrade which has made ID11 the first of the new set of long beamlines; as such there are many technical issues to resolve in order to fully exploit the possibilities made available by this evolution. In particular careful consideration was taken in order to minimise the effects of vibrations, temperature gradients and other potential instabilities. The expertise obtained during the ID11 upgrade will be invaluable for subsequent beamline upgrades.

The criterion for many of our techniques to be applied is that a sufficiently small quantity of sample be present in the beam for diffraction spots to be differentiable. As we wish to work with arbitrary samples, we need to be capable of producing beams of the smallest possible sizes. Optical developments, many of which have come from the ESRF, have produced sub-100 nm beams; the challenge now is to be able to exploit such beams, which requires beam monitoring and sample metrology to the same accuracy. We have thus developed our new experimental station with these technical criteria in mind. It is our aim to both extend our current methods toward nanoscale resolution and integrate their application, such that we can simultaneously characterise the crystal, micro- and meso-structural characteristics – that is, their structure from the Ångström to the millimetre scale.

In the initial phase, “moderate” (i.e. 0.5 – 2 μm) focusing optics consisting of bent Laue crystals, a KB multilayer mirror system, and a two compound refractive lens “transfocators”, which allow a tunable focus between about 0.5 μm and 1 mm by insertion of lenses in the beam, were implemented. Complete redesign and reconstruction of the primary optics, in particular with the addition of an ultra stable, horizontal double bent Laue monochromator, along with accompanying vibration suppression and temperature control now ensures that we are able to work with an unperturbed vertical source, a unique situation. The smallest focus has been achieved by nano-lenses made with silicon wafer technology. During our initial tests we achieved 140 nm focus at 45 keV with this device.

With the implementation of a new cryogenic undulator in January 2010, we expect to gain a factor up to eight times more flux at high energy with respect to the current source, ?? (*not clear*) which should be added to the increase in gain (up to a factor of 100) due to the implementation of the in-vacuum transfocator earlier this year. For detectors, current detector technology does not offer a clear improvement over our fibre-optic coupled CCD camera (Frelon). However, the ongoing development of high energy pixel detectors, hopefully within the next five years, will present a clear improvement in the quality of data which could be collected.

1.5 REFERENCES

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