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Design of powder diffraction beamline (BL-I11) at Diamond

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Abstract. We present the design of a synchrotron x-ray beamline (BL-I11) dedicated to powder diffraction experiments, currently under construction at Diamond Light Source (DLS). An in-vacuum undulator will provide an intense and highly collimated x-ray source and with the necessary optics, a high purity beam of low energy-bandpass x-rays in the range 5-30 keV will be delivered at the sample. The diffraction instrument is designed to have the flexibility to house a variety of sample environments and to have two detection systems to collect high quality diffraction data, i.e. multi-analysing crystals for high angular resolution experiments and a fast position sensitive detector for time-resolved studies. BL-I11 will be a powerful state-of-the-art dedicated user facility for high resolution (*d*-space and time) powder diffraction studies of polycrystalline materials under ambient and non-ambient conditions.

Introduction

Now in its final construction stage, Diamond is the UK's new 3rd generation synchrotron radiation (SR) facility. The SR machine will comprise a 3 GeV electron storage ring, injected from a 100 MeV linac through a full energy booster synchrotron. Initially, seven phase I beamlines will be open to users in January 2007, followed by a further suite of 15 similarly high performance state-of-the-art phase II beamlines, the first of which will be the powder diffraction beamline BL-I11. The scientific aims of this beamline are very broad and cover a wide range of topics in materials science, physics, chemistry, earth sciences, engineering, pharmaceuticals, archaeology, industrial processing and others. Researchers who use synchrotron x-ray powder diffraction thus form a large and very diverse user community. However, their common requirements, as identified by our User Working Group (UWG), are for a beamline that combines (a) high flux over a wide energy range, (b) high resolution (*d*-space and time) and (c) experimental flexibility, e.g. interchangeable diffraction geometries and versatile sample environments. The beamline must also provide the resolving power to "probe" deep into sample structures ($\Delta d/d \sim 10^{-3}$-10^{-4}), to be able to "clock" rapid changes ($\Delta t \sim$ ms-s) as they occur and to perform resonant diffraction in order to solve complex structures with low "normal" electron contrasts. The key parameters and specified performance deliverables required from the beamline are summarised in table 1. This paper describes the beamline design concepts needed to meet these requirements. The design specifications are aimed at not only satisfying existing scientific demands, but to provide the capability of facing new challenges and to open up new areas of research.

Table 1. Expected Deliverables from Beamline BL-I11

Energy range (wavelength)	5-30 keV (0.4-2.5 Å), continuously tuneable
Energy resolution (ΔE/E)	1.5×10^{-4} at 10 keV (Si111 monochromator)
Energy stability	0.5 eV at 10 keV or less
Beam size at sample (FWHM)	~0.8 mm (vertical) x 5.0 mm (horizontal)
Beam divergence @ 10 keV (FWHM)	30 µrad (vertical) x 100 µrad (horizontal)
Photon flux at sample at 300 mA	10^{13} photons/s @ 10 keV
Resolution, Δ2θ (analysing crystal)	0.005° at 10 keV
Time resolution with PSD	ms-s per powder pattern

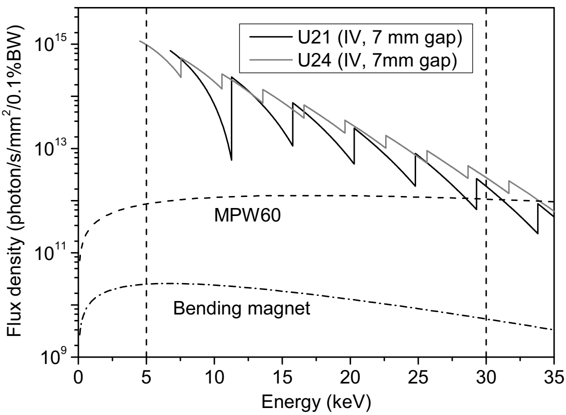

Figure 1. Flux density output of bending magnet, multi-pole wiggler (MPW) with 60 superconducting poles and 2 m long IV undulators. All calculations were done with 300 mA ring current.

X-ray source

Recently constructed powder beamlines are sourced by insertion devices rather than bending magnets in order to deliver the highest possible flux at the sample. For example, X04SA (Swiss Light Source) [1], MPW6.2 (Daresbury SRS) [2] and ID31 (ESRF) are either multi-pole wiggler or undulator beamlines. Figure 1 shows the predicted flux per unit area (mm^2) at the sample (45 m from source) produced by typical insertion devices in the Diamond synchrotron. The vertical dashed lines indicate the minimum (5 keV) and maximum (30 keV) energy requirement for BL-I11. Within this range, the low divergent x-ray beam produced by an in-vacuum undulator (IVU) clearly meets the requirements of Table 1. The matching of the beam cross-section (~1 mm vertical and 5 mm horizontal) to the sample size (at ~45 m from source) also means that the beamline does not require complicated focusing optics and will make operation much easier, particularly when frequent wavelength changes are required. With the appropriate choice of IVU (e.g. U21: device length=2 m, period length= 21 mm or U24: device length=2 m, period length=24 mm) with 7 mm magnet gap to cover the energy range, the beamline will deliver high flux beam at the sample (~10^{14} photon/s/0.1%bw at 10 keV). The beam from the IVU will be of low divergence, 30 (vertical) by 100 µrad (horizontal), and thus provides the basis for delivering the features of fast and high resolution data collection.

Beamline layout

The beamline has two hutches: (i) optics - to house the necessary components to "condition" the beam straight from the source, (ii) experimental (end station) - to house the diffraction instrument, sample tables/environmental cells and detector systems. A schematic of these is shown in figure 2 indicating the main components and their approximate distances from the source. The optics hutch will be approximately 4 m high, 10 m long and 2.5 m wide. The machine ratchet wall will form the inner hutch walls, with remaining walls constructed from steel and lead panels. The floor area will be ~30 m^2. The large experimental hutch will be approximately 9.7 m long, 5.0 m wide, narrowing to 3.9 m, and 4.0 m in height, located downstream of the optics hutch and constructed entirely from steel and lead panels.

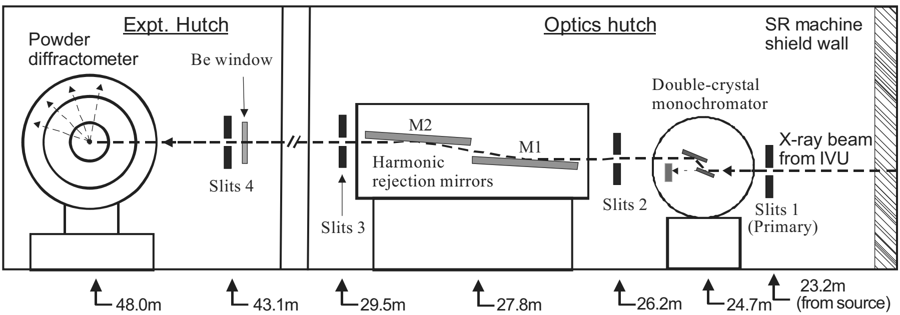

Figure 2. Schematic representation of Beamline I11 showing the two hutch arrangement.

Design optics

The optics hutch will house slits systems, monochromator and harmonic rejection mirrors. The primary slits will "trim" and define the white beam produced by the IVU. The most important issue for the optical design is the monochromator position, which could be either before, or after, the mirror system, if the latter is used as a collimating device. There are advantages and disadvantages associated with either option. However, both were recently tested on ID09 at the ESRF [3], leading to the conclusion that the "mono-first" geometry gives better performance and stability. There is much evidence from SR sources worldwide that greater stability is achieved by placing a cryogenic monochromator before the mirror, provided vertical divergence is not a major problem - i.e. beams from undulator sources. Based on this, the monochromator forms the first BL-I11 optical element, located as close as possible to the source. Silicon crystals of (111) cut are capable of meeting the beamline's energy range and low bandpass requirements and a double-crystal monochromator (DCM) has been specified to allow for a fixed exit-height beam geometry and requiring no movement of the mirrors and other downstream components each time the energy is changed. Due to the high heat load of the IVU beam, liquid N_2 cryogenic cooling will be employed. Depending on the final outcome of finite element analysis, an appropriate crystal cooling scheme will be adopted, e.g. direct cooling or indirect cooling through a Cu block in contact with the bottom surface or side surfaces of the 1st crystal.

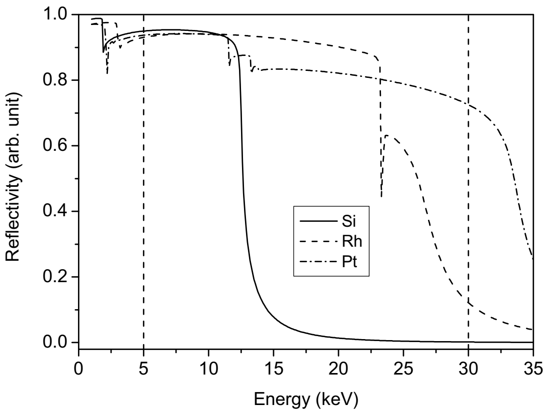

Figure 3. Reflectance versus energy of Si, Rh and Pt surfaces.

Powder diffraction relies on vast numbers of crystallites being distributed at random orientations in the diffracting lozenge. The Scherrer effect of particle size broadening of diffraction peaks means that powder specimens should be packed with grains typically sub-micron to a few microns in size. Thus to ensure good sampling statistics, the irradiated specimen area is typically a few mm^2. Focusing of the undulator beam is therefore unnecessary for this beamline. However, inclusion of a mirror assembly after the DCM will make a significant contribution to harmonic rejection by reducing the problematic high energy photons that can contaminate low energy experiments. Although it is possible to reduce harmonic radiation by misaligning the second monochromator crystal, to fine-tune the monochromator in this way ran contrary to the requirement for a simple user-friendly beamline. To adequately cover the required energy range, a double bounce mirror (DBM) assembly with three stripe surfaces of Si, Rh and Pt element has been devised. If the operational angle is sensibly fixed at ~0.15°

(~2-3 mrad), then, the three coatings can adequately cover the large energy range (figure 3): (i) Si coating for energies below 10 keV, (ii) Rh coating for energies between 10 and 22 keV and (iii) Pt coating for energies between 22 and 30 keV. In addition to reducing further the harmonic contamination, the DBM geometry has the added advantage that the beam will be offset vertically by the small mirror gap instead of being deflected at the angle of reflection if a single mirror was to be used. The original beam will be slightly “perturbed” by the DBM, but a decent final beam is predicted from ray-tracing simulation, e.g. the beam profiles shown in figure 4. The beam profile (right) was simulated with the mirror surface at a fixed incident angle of 2.5 mrad using a 500 Å Rh coating of 5 Å roughness (rms), 2 μrad sagittal and 4 μrad tangential sloping error (rms).

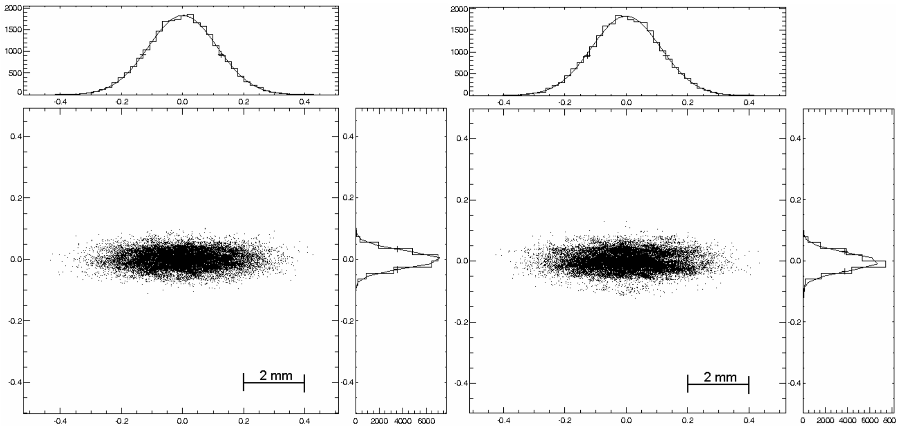

Figure 4. The 20 keV beam at 45 m from the DCM (left) and with the double-bounce mirror (right).

End station

In the experimental hutch, the centre-piece will be a large heavy duty diffractometer with three co-axial, high precision rotary stages (θ-, 2θ- and δ-circle). Powder specimen in capillary, flat-plate holder or small sample cell can be attached onto a small xyz-table which is mounted on the inner θ-circle. Low-high temperature apparatus (cryogenic devices and furnaces) and variety of sample stages including a large table to support bulky environmental chambers and high throughput equipment (robotic arm) will also be available. Specimen will be aligned at the centre of rotation of the diffractometer which will itself be aligned with the position of the x-ray beam. For high resolution operations, the large 2θ-circle will provide the scanning of positive angles (above the beam) via the use of multi-analysing crystals (MAC) which will “filter” the x-rays diffracted by the sample. The MAC system is based on the 9-crystal design of Hodeau et al [4] for ID31 at the ESRF. For BL-I11 however, 36-45 crystals and detectors will be deployed to speed up data collection. These will be grouped into 4-5 MAC stages. The detailed design and functionality of these arms are described elsewhere [5]. For time-resolved work a wide-angle position sensitive detector (PSD) mounted on the δ-circle will be used. The advantages of mounting the PSD on a separate circle are to eliminate changeover time between experiments and to be able to operate the two systems simultaneously or independently. The PSD has been specified to perform fast data collection (e.g. 1 powder pattern/ms) with a 70º aperture and an intrinsic angular resolution of 0.01º. The end station and the detector systems will be described in future publications.

Concluding remarks

We have designed an x-ray powder diffraction beamline sourced by an in-vacuum undulator at Diamond Light Source which, when complete, will be a powerful facility with which to study the structural properties of materials. The combination of in-vacuum undulator with beamline optics will deliver a high quality beam in terms of both flux and purity to the sample. The large experimental hutch, heavy duty diffractometer, and modular sample components will make the changeover of equipment and the configuration of experiments quick and flexible. This versatile beamline will have the capacity to accommodate small, medium and large environmental cells allowing a wide range of experiments to be carried out under ambient and non-ambient conditions. High angular- and time-resolution data will be collected through the use of the two independent detection systems. The beamline is currently at the beginning of its installation stage and is due to be completed by the end of 2007. Commissioning of the optical and end station components will start in early 2008, with user access scheduled for Jul-Aug 2008.

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