B18: A core XAS spectroscopy beamline for Diamond

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Abstract. During the past twenty years, XAS has progressed from being a technique only suitable for specialists to become a widely applicable tool. This situation has resulted from the steady development of reliable spectrometers and new generations of software for data analysis. B18 will be a general purpose XAS beamline on Diamond. It will cover a wide energy range (2 to 35 keV), with a monochromator designed to carry out both conventional and QEXAFS measurements. The main design novelty is that the low and high energy optic branches will run in parallel and the appropriate branch for a given experiment will be selected by changing the position of the slits, instead of by moving the optical elements. This will allow us to develop a very high level of automation in the operation of the instrument. Detection systems will include transmission, fluorescence and electron yield. Experience shows that considerable value is added by combining techniques. Therefore provision has been made for wide angle X-ray diffraction studies to be incorporated into the beamline architecture. The instrument will offer a variety of sample environments and the flexibility to integrate set-ups designed by the users. Hence, B18 will be able to contribute to research programs across a wide range of scientific disciplines, e.g. solid state physics and materials, catalysis, chemistry, soft matter, surfaces and biomaterials. The instrument will open to first users in April 2010.

1. Introduction

XAS is nowadays widely recognized as a mature X-ray technique, with a wide range of applications across scientific disciplines. As a result, there is a need at modern synchrotrons to provide robust and highly automated XAS beamlines, designed to be used by both experienced and occasional users. The design of B18 at the Diamond Light Source has been done with this goal in mind. By April 2010, there will be three spectroscopy beamlines operational at Diamond: a micro-focus instrument (I18 [1]), a high intensity XAS beamline (I20 [2]) and B18, a general purpose instrument that will complement the more specialized I18 and I20. To that end, B18 will be built on a bending magnet and have a wide energy range (2 to 35 keV covering K-edges from P to I and LIII-edges from Y to U) that will overlap with the ranges of the other two spectroscopy beamlines.

2. Optics design

The optical configuration has been chosen to optimize the efficiency and reliability of the beamline without compromising the flux. The beam focusing will facilitate sample mapping or high throughput
experiments where the sample size is relatively small. The design is based on three main optical elements: a collimating mirror, a fixed-exit double crystal monochromator and a double toroidal focusing mirror. In addition, a pair of harmonic rejection mirrors will be used for low energy operations. To cover the large energy range required (from 2.05 to 35 keV), two crystal sets and two different mirror coatings are necessary.

The bending magnet source provides a wide (3 mrad) beam fan. This allows us to collect two separate beam fans in the horizontal plane, each 0.9 mrad wide and separated by 1.56 mrad. The two beams will follow different optical paths and will be independently focused at 37.5 m from the source. This simplification will improve stability and reduce the number of alignment operations. Indeed, to select the correct branch path it will be necessary to operate only the horizontal slits and exchange the monochromator crystal positions. There will be no need to translate the mirrors but the design requires two different positions for the sample.

After the front-end, the white beam will be collimated by a flat, cylindrically bent mirror at a fixed incidence angle (2.3 mrad to ensure operation up to 35 keV). The mirror will be coated with two stripes (Cr and Pt), one for each beamline branch. This mirror will always collect the full 3 mrad beam power, as delimited horizontally and vertically by the primary slits, to reduce deformations of the surface due to asymmetrical power loads. In addition, with Diamond running in top-up mode, the thermal load on the optical elements will be constant with time and will improve the overall stability. The beam branch selection will occur at the secondary slits, placed just before the monochromator.

The monochromator is a bounce-down, double crystal and fixed exit unit. It will host two directly water-cooled flat crystal sets, Si(111) and Si(311) cut, interchangeable in-vacuum. The monochromator Bragg axis will be driven over large angular range (6° to 70°) by an in-vacuum, water-cooled brushless DC motor, to allow for fast and repeated continuous operation, with a maximum speed of about 1 deg/s. The monochromator will operate both in step-scan and in continuous (QEXAFS) scan modes. In the latter case in particular, in the high energy (> 7 keV) range, the monochromator will be preferentially used as a pseudo-channel cut, the beam height change being compensated by the following focusing mirror.

Figure 1. Schematic representation of the layout for B18. The drawing presents the main optical components (collimating mirror, monochromator, focusing mirror, harmonic rejection mirrors) and the main diagnostics elements (slits, attenuator filters, diagnostics units and removable fluorescence screen and cameras). Distances from the bending magnet source to the optical elements are also shown. The experimental table is divided in a high vacuum section for low energy experiments, followed by a hard X-ray section, where experiments can be performed in air.

The monochromator is followed by a double bent cylinder focusing mirror. The cylinders are coated again with Cr and Pt. The cylinder axes are slightly divergent (1.56 mrad), to reduce the need for alignment of the mirror in the yaw direction when changing the working beamline branch. The focusing geometry is in the 2:1 configuration [3] that allows compensating in a first approximation the optical aberrations, giving a stable and symmetrical spot size. The focus (100 x 250 μm FWHM approximately, including mirror slope errors) is nominally placed at 37.5 m from the source, but the
beamline will be able to move the focus position in a limited range (+/- 1 m) without affecting substantially the focus quality.

A pair of harmonic rejection (HR) mirrors with two coatings (Ni and Pt) is located in the experimental hutch, and will be inserted in the optical path when operating below 15 keV. This unit, designed in-house, includes the final monochromatic beam slits and the diagnostics unit similar to the one placed downstream of the monochromator, based on a metal foil array and photodiodes to measure the X-ray fluorescence from the foils in the high energy regime, and total electron yield for low energy operations.

The expected flux at the sample positions is of the order of $5 \times 10^{11} \text{ph/s/(Si111 BW)}$, comparable with similar bending magnet beamlines at third generation sources (BM29 at the ESRF, SuperXAS at SLS). B18 will have a gain of a factor of approximately 2 with respect to beamline 9.3 at Daresbury and a significantly improved flux density thanks to the focused beam configuration.

3. Experimental station
Experiments will take place on a large experimental table (Figure 3), providing 5 degrees of freedom, that will need to be aligned horizontally to the selected beam branch.

The experimental space can be divided into two areas. The upstream UHV section will be separated from the beamline HV environment by a thin polymer window (the beamline is windowless up to that point) to allow experiments down to the soft X-ray regime (2.05 keV) while preserving the beamline vacuum from contaminations. The vacuum vessel is fitted with a sample manipulator with motorized vertical and rotation degrees of freedom. Detection modes will include total electron yield, a gas microstrip fluorescence detector for high counting rates [4] and a 4 element silicon drift detector (SDD). Measurements down to liquid nitrogen temperatures will be possible as a standard. Downstream, a beryllium window (100 µm thick) separates the vacuum chamber from the following section to allow higher energy experiments to be conducted in-air. An X-ray position monitor placed after the Be window will be used to monitor the beam stability and provide a feedback position signal to the monochromator for the hard X-ray range, while drain current measurement from the final monochromatic beam slits will be used in the soft X-ray range.

**Figure 2.** Simulation of the beam focus (100 x 250 µm FWHM – H x V). The major plot ticks are placed at 200µm intervals.

**Figure 3.** Experimental table model. On the right-hand side is the vacuum chamber for experiments in the soft X-ray region. The default experimental breadboard and detectors (ionization chambers and monolithic Ge detector) are represented in the hard X-ray section on the left.
The hard X-ray section will be mounted on a large (2.4 m x 1.2 m) optical table. Three ionization chambers will be installed on standard rails. A 9-element monolithic germanium detector [5] with XSPRESS-II [6] readout and a 4 element silicon drift detector will be available for fluorescence detection. B18 will also give the possibility to undertake combined XAS-XRD experiments, using a compact microstrip detector system for X-ray diffraction measurements that will cover a 60° angle. The angular resolution (assuming no contribution due to the size of the sample) will be limited by the beamline vertical divergence, and is expected to be 0.03°. We have designed a large and flexible sample space in order to facilitate the installation of large sample environments. Experimental equipment will be mounted by default on the experimental table using standard 600 x 600 mm breadboards, with 480 mm distance between the table surface and the beam position. B18 will provide as standard a high throughput, top loading, liquid nitrogen cryostat (expected to be able to load about 50 samples simultaneously) and furnaces. In addition, we expect our users to bring specialized equipment that will combine XAS measurements with other techniques. To that end, the services available in the experimental hutch beyond the commonly available water cooling, compressed air, power and spare connections to the data acquisition systems, will include liquid nitrogen, gas lines and fume extraction systems.

4. Conclusion
B18 will be the new general purpose XAS beamline at the Diamond Light Source. First users are expected on the beamline on April 2010. It will be competitive in terms of flux and flux density with respect to equivalent beamlines at other sources. The target of the instrument is to provide efficient operation over the whole energy range. This will be achieved thanks to the simplification of the alignment operations made possible by the adoption of a configuration with two beam branches, which will enable a high degree of automatic optics optimisation. Quick-EXAFS will be available as a default. Detection techniques available will be fluorescence for dilute samples (9 elements Ge detector and 4 elements SDD, gas microstrip for low energies), ionisation chambers for transmission measurements and Si microstrip for wide angle X-ray scattering experiments.

The sample environment will be flexible enough to allow for user designed set-ups and there will be provision for high throughput sample environments and combined detection modes to make the most efficient use of the allocated beamtime.

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