XAFS at the Canadian Light Source

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Abstract. Canadian Light Source Hard X-ray Micro-Analysis Beamline (HXMA, 06ID-1) is a hard X-ray spectroscopy beamline currently under commissioning. The source of the beamline is a superconducting wiggler covering 5 to 40 keV. The primary optics include a cryogenically cooled double crystal monochromator (Si 111 and 220), white beam vertical collimating and toroidal focusing mirrors. End station experimental capabilities include XAFS (Ge solid state detectors), microprobe (Kirkpatrick-Baez mirrors, Ge solid state detector and image plate area detector), and diffraction (Huber psi-8 and powder diffraction setups, with diamond anvil cell high pressure sample environment). Commissioning status for the XAFS capabilities is described.

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INTRODUCTION

The Canadian Light Source (CLS) 06ID-1 Hard X-ray Micro-Analysis Beamline (HXMA), or originally called CLS General Purpose XAFS Beamline, was proposed as a facility beamline [1] to the phase I CLS construction plan at the initial suggestion from the CLS Facility Advisory Committee (FAC) on which the late Prof. D. E. Sayers served as a member. The initial design concept is to deliver a stable high throughput XAFS beamline to the spectroscopy user community, particularly those in the geochemistry, materials sciences and the mining sector of the industry. With the increasing demands from the user communities the scope of the project was later expanded twice to include the microprobe and diffraction capabilities. These later additions of the capabilities led to a higher demand to the brilliance performance of the beamline.

CLS is a mediate energy 3rd generation synchrotron radiation (SR) facility (2.9 GeV, 500 mA, 170.88 m circumference, horizontal emittance 18.1 nm-rad) [2]. To serve the broad users' requirement for the SR spectral range and with consideration of the spectroscopic nature of the applications, we opted to use a short-period wiggler insertion device to source this beamline (63 pole, 33 mm period, 2 T) [3], which has a critical energy of 10.7 keV and a relatively narrow horizontal radiation fan of about 2 mrad. The designed HXMA BL spectral range is 5 to 40 keV which covers most of hard X-ray XAFS required, the beam collimation and focus by the optics permitting synchrotron-radiation grade diffraction and the capability of generating micron size of beam for microspectroscopy. In this paper, the beamline optics design is briefly summarized and the current XAFS commissioning activity is described with some preliminary testing results.

DESIGN AND COMMISSIONING

The optical layout of the HXMA beamline is schematically shown in Fig. 1. The primary optics include water-cooled vertical silicon collimating mirror: 1.2 m optical length, exchangeable between Rh and Pt coating stripes by mirror tank transverse motion, maximum absorbing thermal power of 500 W; Kohzu CMJ-1 DCM: selectable Si 111 or Si 220 crystal pairs exchanged by transverse motion of the vacuum housing, indirect liquid nitrogen cooling for the monochromator 1st crystal (max 500 W absorbing thermal power) [4] and the 2nd crystal [5]; water-cooled silicon toroidal focusing mirror: 1.15 m optical length, exchangeable Rh and Pt coating stripes, maximum absorbing thermal power 200 W. This layout permits three SR beam operation modes: (1). the mirror-mono-mirror mode (5 -30 keV); (2). mono-only mode (5-40 keV); and (3). mirror-mirror focused pink beam mode

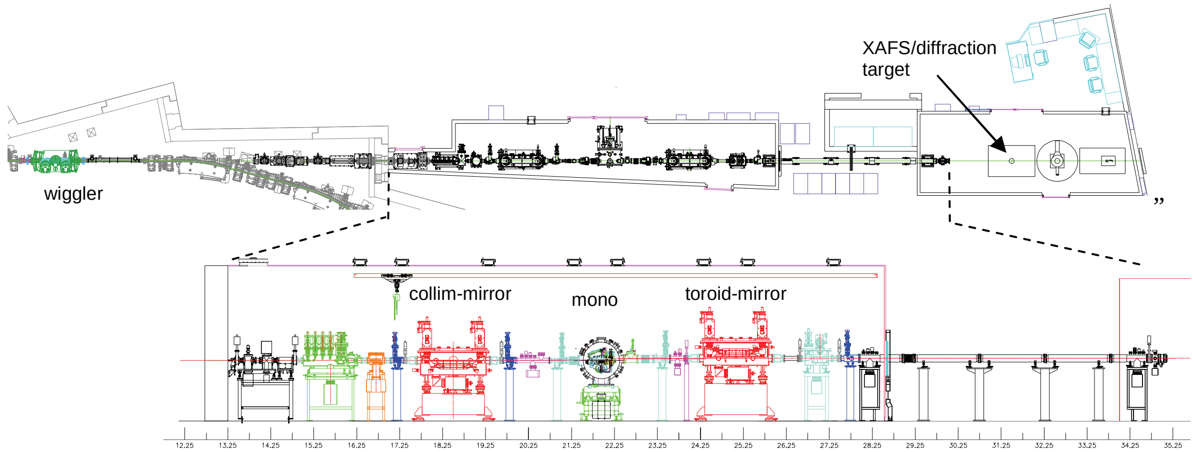

FIGURE 1. Schematic layout of HXMA beamline. Top: plan view including a portion of the CLS storage ring components, the ratchet wall, foot-print of the two hutches, a sample user preparation area, and the user experimental control area. Bottom: elevation view of the beamline optical components, with collimating-mirror, monocharomator and toroidal focusing mirror identified.

(1600 W broad band pink SR output at the beamline exit window). Any one of these three operational modes can be applied to XAFS or diffraction experiments. For microprobe application we adopt the Kirkpatrick-Baez (KB) mirror bender design of Eng *et al.* [6]. The focus point of toroidal mirror (XAFS/diffraction target point in Fig. 1) is taken as the source for the KB optics. The beamline monochromator harmonics rejection relies on using a metal coated float glass mirror capable of tilting and vertical translation (not shown). In the commissioning, monochromator 2nd crystal detuning was used to reduce the beam harmonic content. In the experimental hutch there are three permanent experimental setups (upstream XAFS table, diffraction table, and downstream microprobe table, see Fig. 2) which are all mounted on a longitudinal translation rail. The diffraction station includes a Huber psi-8 diffractometer.

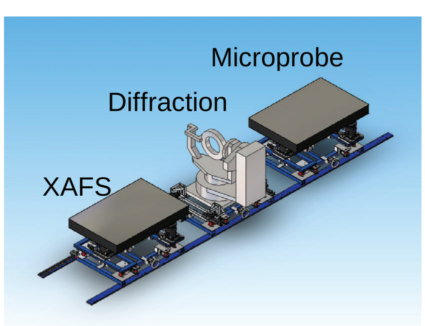

FIGURE 2. Experimental hutch layout. The toroidal mirror focusing point is shared by the XAFS and diffraction stations by longitudinally translating the stations.

More detail of the beamline optics design and specifications is described elsewhere [7]. Briefly, the typical XAFS running mode of the beamline is to use the combination of collimating mirror, monochromator, and toroidal focusing mirror, where the fixed-exit double crystal monochromator is in a quasi channel cut mode during a single XAFS scan. Figure 3 shows some preliminary testing results of this operation mode,

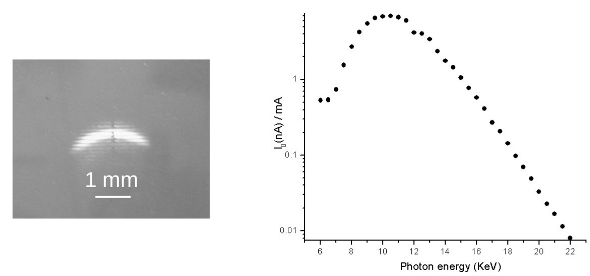

FIGURE 3. Preliminary results on the mirror focused monochromatic beam properties. Left panel: a photograph image of a focused beam near Cu K-edge. The pre-mono white beam entrance slits opening was at 2.5 mm (vertical) x 4.0 mm (horizontal), which represents ~1/4 of the 1.6 mrad horizontal wiggler radiation fan. Right panel: spectral coverage of the mirror-mono-mirror mode of HXMA beamline, with Si 220 mono-crystal, Rh mirror coatings, and N_2-filled 6” ion chamber (normalized to per mA of storage ring current).

The design of the CLS HXMA wiggler source, with its emphasis on the spectral brilliance by reducing the magnetic periodicity, leads to a rather limited deflection coefficient (K=5.91) of the device, i.e. the undulator harmonic features in the spectrum become a concern for spectroscopic applications such as XAFS.

A de-phasing measure of adding random thickness shims (up to 1 mm thickness) between the superconducting coils was applied to generate a RMS phase angle of 36.4 ° [3], but this still leaves clearly detectable undulator oscillations for photon energies below 10 keV (Fig. 4a). However, when a wider vertical acceptance of the wiggler radiation cone is taken and the beam is conditioned by the mirror-mono-mirror combination at these low photon energies, the effect is greatly reduced as shown in Fig. 4b. So far no distortion of the XAFS data have been noticed due to these residual undulator features.

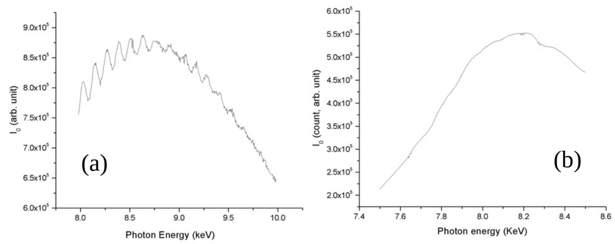

FIGURE 4. I_0 spectra below 10 keV. (a), no-focusing case, with a white beam slits of 1 mm vertical opening; (b) mirror focused case with a white beam slits of 2.5 mm vertical opening.

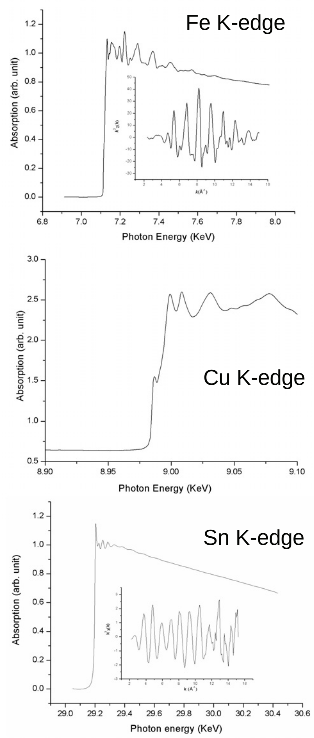

FIGURE 5. Several exemplary spectra of standard foil samples acquired in transmission mode. The inserts in the upper and lower panel show the background removed XAFS data in k-space. The measurements were taken using Si (220) mono crystals and Rh coating stripes on the collimating and toroidal focusing mirror.

The superconducting wiggler of the HXMA line was commissioned in Feb., 2005 and the first XAFS spectrum from HXMA beamline was recorded in July 2005. There have been limited user accesses while commissioning on the other aspects of the beamline being carried out. In the meanwhile an EPICS-based data acquisition system has been developed at CLS for the beamline. Figure 5 shows a few standard spectra spanning over most of the spectral range covered by the beamline. Initial results indicate that the crystal mounting and cooling mechanism [4,5] performs as expected, i.e. no crystal-strain induced resolution loss could be detected. Initial optical system stability appears to be satisfactory and this will be further improved by using dynamic beam position monitoring in the experimental hutch. The mirror collimating and focusing optimization is ongoing, which will provide the source point for the microprobe setup of the beamline.

SUMMARY

The construction and commissioning of a superconducting wiggler-based hard X-ray (5 -40 keV) spectroscopy beamline at the Canadian Light Source is described. The primary function of the beamline is to perform high quality XAFS measurements, while the expanded functions include KB-mirror based microprobe and various diffraction setups.

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