Sources, Beamlines and Optics

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The XAFS beamline BL01B1 at SPring-8

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An x-ray absorption fine-structure (XAFS) spectroscopy beamline, BL01B1, was installed at a bending magnet source at SPring-8 and has been open to users since October 1997. It was designed for XAFS experiments covering a wide energy range. Position tables and automatical control programs were established to adjust the x-ray optics and achieve the designed performance of the beamline under each experimental condition. This has enabled conventional XAFS measurements to be made with a good data quality from 4.5 to 110 keV.

Keywords: XAFS; high-energy; beamlines.

1. Introduction

The bending magnet source at SPring-8 has a high brilliance, low emittance, and a high photon flux up to 100 keV. The beamline BL01B1 is dedicated to XAFS measurements in a wide energy range from 4.5 to 110 keV for a variety of research topics (Emura *et al.*, 1995), and has been open to users since October 1997 with a storage ring current of 20 mA, i.e., 1/5 the target current. The performance of the x-ray optics characterized using synchrotron radiation light will be reported elsewhere. The results show that the target specifications for the measured beam have almost been completely achieved except for sagittal focusing: a photon flux of 10^{9}-10^{11} phs/s with $\Delta E/E$ of $<2\times10^{-4}$, a vertical beam size focused by a mirror of < 0.2 mm, and a ratio of the higher harmonics contaminant of $< 10^{-5}$ with mirrors.

To achieve the designed performance of the beamline in a wide energy range, the beamline optics should be adjusted to the optimal position for each experiment. Because rearranging the monochromator and/or mirrors involves the realignment of many components, such rearranging can be done a few times per day. To achieve quick and easy adjustment, we prepared tables at the positions of the optical components and developed automatic control programs. This report gives an overview of the beamline status and some representative results highlighting the performance of BL01B1.

2. Beamline

The source is the SPring-8 bending magnet of type B1: σ_x=0.182 mm, σ_y=0.058 mm, and σ_y'=0.065 mrad (@ 10 keV). The critical energy is 28.9 keV. The photon flux of the source calculated using code SPECTRA (Tanaka & Kitamura, 1998) is shown in Fig. 1. The front end section is installed in the shielding wall, and is separated from the transport channel section by two Be windows 0.25 mm thick.

The x-ray optics and their arrangement for BL01B1 are standard for SPring-8 bending magnet beamlines (Fig. 2) (Goto *et al.*, 1998), and are well suited for XAFS experiments.

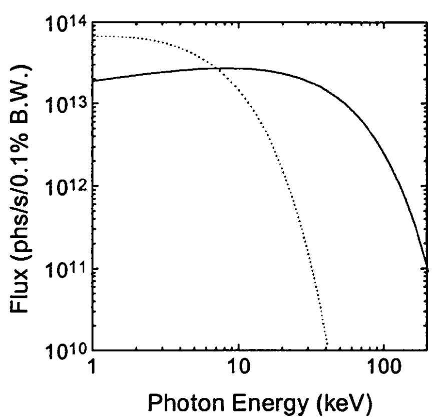

Figure 1
Calculated photon flux of bending magnet source at SPring-8 (solid line) and KEK-PF (broken line).

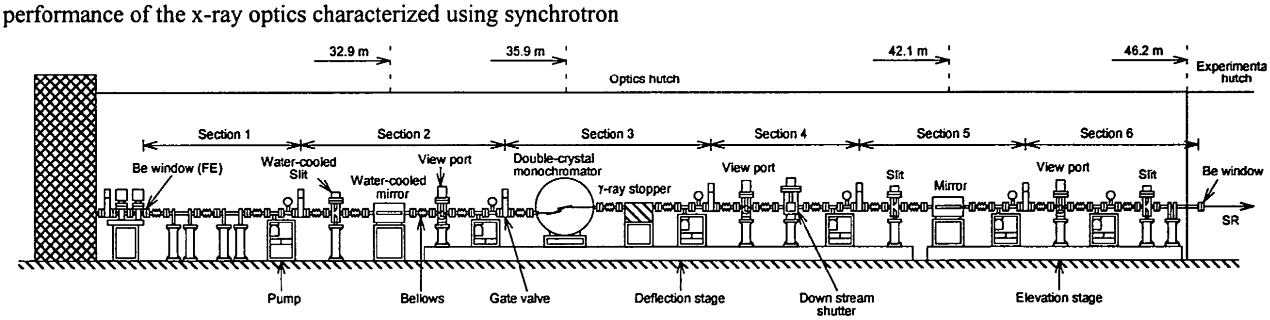

Figure 2
Schematic layout of transport channel of BL01B1.

Briefly, the optics consist of a first slit, first vertical collimation mirror, fixed-exit double-crystal monochromator, γ-ray stopper, second slit, second vertical refocusing mirror, and third slit.

To cover a wide energy range without breaking the vacuum chamber, the monochromator is of the adjustable inclined double-crystal type (Kohzu Co.) with a single pair of Si(311) crystals (Uruga *et al.*, 1995). The [011] axis is in the scattering plane. Both of the crystals rotate around the [011] axis to get a net plane (hkk). The Bragg angle ranges from 3 to 26 degrees, enabling photon energies of 4.5-37 keV with Si(111), 8.6-70 keV with Si(311), and 13.5-110 keV with Si(511). The first crystal is of the water-cooled fin type, and the second crystal is of the indirect water-cooled flat type. A bent second crystal to focus the beam in the sagittal direction is under development.

The mirrors are also used for higher harmonics rejection. The glancing angle of the mirrors is adjusted from 1.5 to 10 mrad to determine the cut-off energy. The mirrors are removed from the beam axis over 40 keV. Both of them are bent by cramp rotation type bending mechanics (Tanase & Endo, 1997). The bending radius is from 6 to 45 km for the first mirror and from 1 to 7 km for the second mirror. The substrates are side-water cooled Si for the first mirror and SiO_2 for the second mirror. The coating material is rhodium and the length is 1000 mm.

To follow the reflected beam, the components downstream of the first mirror to the second mirror were installed on a deflection stage. The components downstream of the second mirror to the Be window were installed on an elevation stage. Both stage positions are controlled by a motor-driven axis, with the measured height of the deflection stage at the down end having a deflection angle accuracy of about 10 microradians.

In the experimental hutch, measurement equipment have been prepared for standard XAFS measurements, including ionization chambers, a Lytle detector, a cryostat, electric furnaces, and a slit. The slit is located upstream of the first ionization chamber to restrict the measurement beam. The beam height changes from 0 to 250 mm together with the glancing angle of the mirror. To follow the beam, the measurement equipment are mounted on the Al honeycomb base, which can be adjusted by a vertical translation stage.

3. Control of XAFS measurements in a wide energy range

The main optics rearrangements include 1) the change of the net planes of the monochromator crystals and 2) the change of the glancing angle of the mirrors, which is followed by more than ten alignment steps. To complete the process of alignment, we have developed an automatic adjustment program for the optical elements using commercial LabVIEW code. For those optical elements whose positions are reproduced with the desired accuracies, their positions have been tabulated for use when the elements are adjusted. For those optical elements requiring fine adjustment using a synchrotron beam, we have developed an automatic scanning routine, i.e., peak search for rocking curves.

Briefly, the change of the net plane of the monochromator crystals involves the following operation: the rotation of the crystals around the [011] axis to get the new net plane, parallelism adjustment for the crystals, and the establishment of a fixed exit beam condition. The parallelism of the crystals can be achieved by the peak search for rocking curves routine. The fixed exit beam is obtained by correcting the deviation of the inclination angle of the crystals around the [011] axis. The magnitude of the deviation is calculated by the beam geometry using the horizontal beam position measured by slit scanning in the experimental hutch.

The change of the glancing angle of the mirrors is followed by the adjustment of the curvature of the mirrors and by the correction of the positions of the deflection and elevation stages to the tabulated positions. The second mirror, second and third slits, and vertical translation stage in the experimental hutch are adjusted by vertical scanning.

It takes about 30 minutes to complete the alignment, either the change of the net plane of the monochromator crystals or the change of the glancing angle of the mirrors, about the same amount of time to obtain typical XAFS data of 500 points.

4. Experimental results

Some XAFS experimental data at BL01B1 are shown bellow. All of the data were obtained in the transmission mode at room temperature, with an ring current of from 15 to 20 mA.

In the energy region lower than several keV, the photon flux of the source was low even compared with the second generation source as shown in Fig. 1. The source flux at the energy corresponding to the third harmonics was about one order of magnitude larger than the first harmonics. Accordingly, it is inherent that the mirrors are used both as a collimation device and as a higher-harmonics rejecter.

Figure 3 shows Ti K-edge spectra for TiO_2 anatase. The mirrors were inserted with a glancing angle of 6 mrad. A sufficient photon flux beam with a low higher-harmonics contaminant ($<10^{-5}$) enabled us to get good S/N ratio data up to $k=16$ A^{-1}. In the figure, the pre-edge profile indicates that the collimation device functioned well to provide a good energy resolution estimated to be < 1.4 eV. To obtain the same energy resolution and higher-harmonics contaminant without the mirrors, it would be necessary to use a narrow slit and detuned

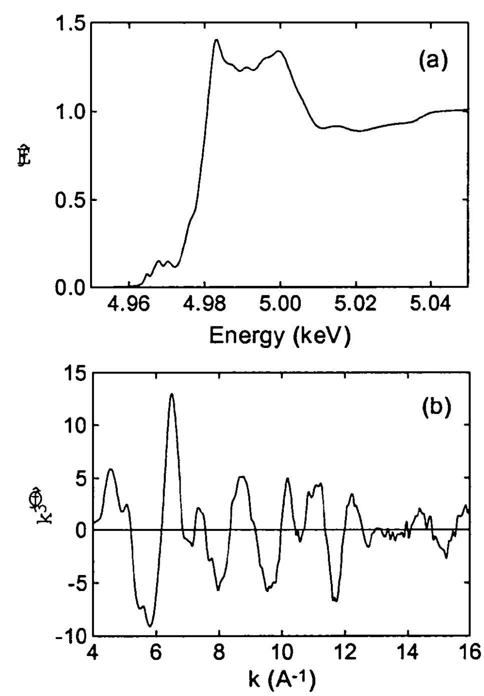

Figure 3
(a) XAFS spectrum near the Ti K edge of TiO_2 anatase at room temperature. (b) EXAFS function above the Ti K edge.

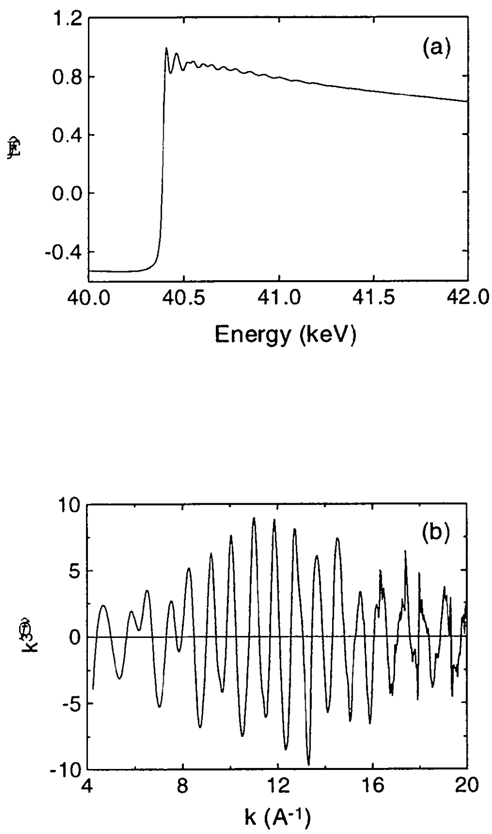

Figure 4
(a) XAFS spectrum near the Ce K edge of CeO_2 at room temperature. (b) EXAFS function above the Ce K edge.

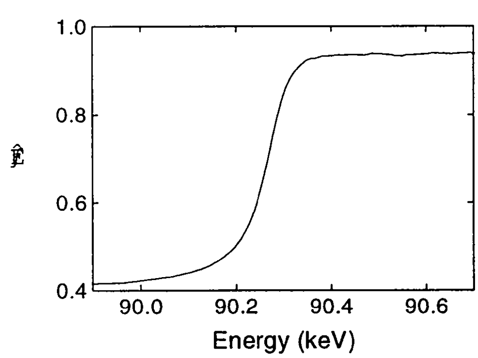

Figure 5
XAFS spectrum near the Bi K edge of Bi2212 at room temperature.

monochromator, which would reduce the photon flux about one order of magnitude.

The moderate energy region (ca. 20 to 50 keV) may be the most effective in BL01B1 because of the high photon flux. Figure 4 shows XAFS spectra for CeO_2. The energy resolution was estimated to be about 4 eV from the geometry of the source and slit. The low noise EXAFS function was obtained up to k=20 A^{-1}.

In the high energy region, the Darwin width is narrow (< 1 arcsec above 30 keV) for Si(311) and Si(511) diffraction planes in comparison with the rotation error of the first crystal during the energy scan. The XAFS spectra were measured while the crystals were kept in parallel by using a digital piezo actuator. Typically, several more minutes are necessary for each XAFS scanning.

Figure 5 shows XAFS spectra for a super conductor Bi2212, which is heaviest element to have been measured at BL01B1. The energy resolution was estimated to be about 15 eV from the geometry of the source and slit. An edge jump was clearly seen but the edge was blunt and the EXAFS signal was small; this is consistent with the theory on the short life time of a K hole. The XAFS of heavy atoms has being studied systematically at BL01B1 (Nishihata et al., 1998).

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