XAFS (BL01B1)

1. Introduction

The beamline BL01B1 was constructed to study XAFS (X-ray absorption fine structure) spectra in a wide energy range (3.8-113 keV) by various techniques [1]. The beamline facilities have been stably opened to users under accumulation ring operation of 100 mA mode. XAFS measurement has been taken for the elements from Ca K (4.0 keV) to Bi K (92 keV). This report describes the current status of BL01B1 including newly installed facilities. For an outline of the construction plan of BL01B1, see Ref. [2].

2. X-ray Optics

In the optical hutch of BL01B1, the main X-ray optics are a first collimating mirror, double-crystal monochromator and second focusing mirror in the vertical direction at the sample position (Fig. 1). The configuration and components are standard for the SPring-8 bending magnet beamline of the first construction phase. The beamline design is well suited to the XAFS experiment, *i.e.*, a fixed exit beam with high energy resolution, very low level of higher harmonics contaminants, and vertical focusing in a wide energy range.

The monochromator is an adjustable inclined double-crystal type. The net plane of crystals can be switched between Si(111), Si(311) and Si(511) without breaking the vacuum chamber. The operation is completed within 30 min by semi-automatic computer control. The drive range in the Bragg angle of crystals was extended from 3-26 to 3-32 deg by remodeling the water plumbing in the vacuum chamber. This extended the energy range to the lower side covered by each net plane, *i.e.*, Si(111): 3.8-37 keV, Si(311): 7.1-72 keV, Si(511): 11.2-113 keV. In the overlapping energy range, an adequate net plane can be selected by estimating the photon flux and energy resolution.

The heat load on the first crystal was estimated to be lower than 10 W at 100 mA of ring current for the typical beam cross section, *i.e.* 10 (H) $\times$ 6 (V) mm^2 with the first mirror and 10 (H) $\times$ 1 (V) mm^2 without the first mirror. This heat load is not severe, thus allowing an indirect water cooled flat Si(311) crystal to be used for the first crystal.

The mirrors are 1 m long and coated with rhodium. The substrates are Si and SiO_2 for the first and second mirror, respectively. The figure of the mirrors surface is a meridional bent cylinder, which is a good approximation of the ideal parabola figure in the case of a very small curvature. The mirrors were inserted in the transport channel in an energy range under 40 keV. The glancing angle of mirrors was adjusted to achieve the higher harmonics contaminants of less than 10^{-5} with a first harmonic reflectivity of more than 70 %. The realignment of mirrors and related facilities were completed within 30 min by semi-automatic computer control. The mirror systems performed well without any significant drift by heat load under 100 mA of ring current. The focused beam size in the vertical direction was 0.06-0.2 mm (FWHM). The measured energy resolution, $\Delta E/E$, was about 2×10^{-4} with Si(111), 6×10^{-5} with Si(311), and 3×10^{-5} with Si(511).

The position sensitive ionization chamber [3] made alignment of the monochromator easy in order to quickly achieve the fixed exit beam condition. The beam position fluctuation at the sample position was controlled within $\pm$ 0.01 mm during XAFS measurement.

The photon flux at the sample position was 10^9-10^{11} phs/sec at 3.8-113 keV.

3. Experimental Hutch

3.1 Measurement Equipment

The standard XAFS measurement is the transmission mode using ionization chambers. Three lengths of the ionization chambers, *i.e.*, 65, 170 and 310 mm, were prepared. The electric current of the ionization chamber was amplified to voltage and then transformed to frequency and counted. For the gas-flow type ionization chambers, He, N_2, Ar, Kr and their mixture were prepared. The Xe-gas-closed types were also prepared for high energy measurement.

For the fluorescence mode's XAFS, a Lytle detector and a Ge single-element solid state detector (Ge SSD) were prepared. The Lytle detector has a solar slit of Al and an ionization chamber of 50 mm long. The element of the Ge SSD has an effective area of 200 mm^2 and a thickness of 10 mm. It is possible to absorb more than 95 % of incident photons in the energy range from 3.8 to 113 keV.

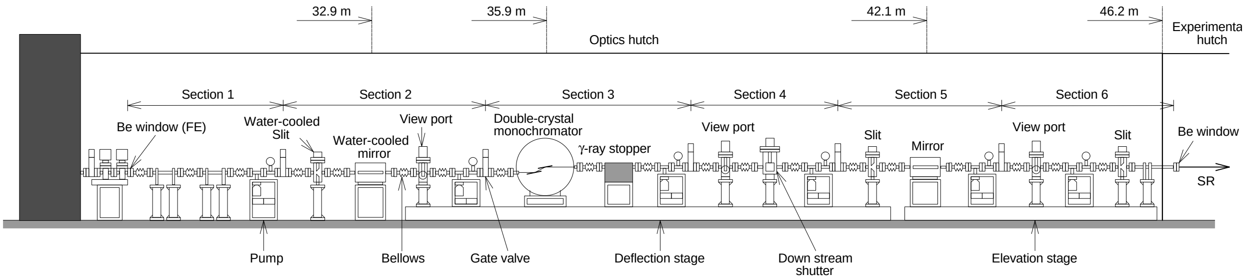

Fig. 1. Schematic view of BL01B1 transport channel.

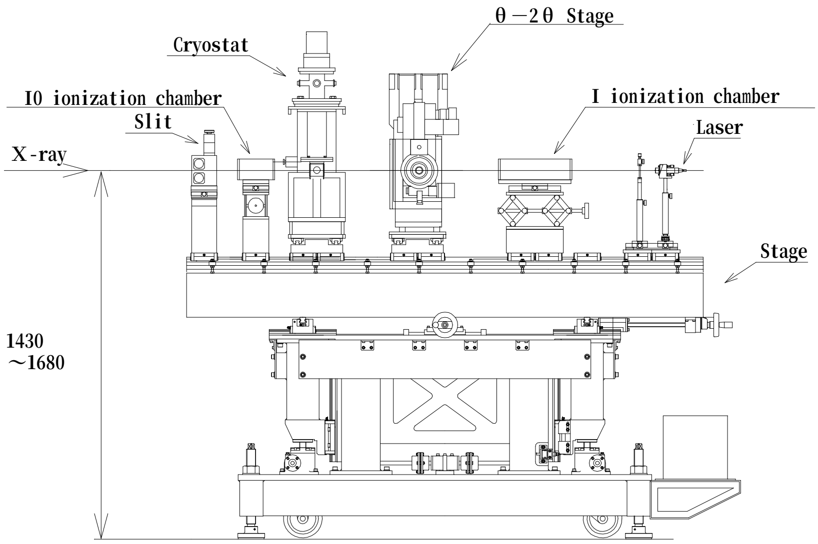

Fig. 2. Facilities in experimental hutch.

3.2 Sample Position Adjustment Stages

A pulse motor driven xz stage was prepared for the sample position adjustment. The step is 0.001 mm/pulse for both axes. The maximum load is 15 kg. A θ-2θ stage was prepared for XAFS measurement under a low glancing-angle arrangement and for the crystalline sample. It includes a pulse motor driven z stage. The steps of these stages are 0.005 arcsec/pulse for the θ axis, 0.001 deg/pulse for the 2θ axis, and 0.001 mm/pulse for the z axis. The maximum load is 20 kg.

3.3 Sample Temperature Controller

A cryostat was prepared for sample temperature control between 6.5 and 300 K. The cooling power is 8 W at the sample stage of 20 K. The cooling rate is about 50 min from room temperature to 6.5 K. During the cooling operation, the cryostat vibrates at about ± 0.01 mm. To prevent the vibration from propagating to the measurement equipment, antivibration rubber was inserted under the cryostat.

Two furnaces were prepared for the control of high temperature. A water cooled electric furnace controls the sample temperature up to 1,070 K with a stability of ± 1 K. An electric muffle furnace controls the temperature up to 1,870 K (in preparation). The cryostat and furnaces have capton windows for the transmission and fluorescence modes.

3.4 Measurement Facilities Arrangement

The measurement facilities were arranged on a vertical translation stage (Fig. 2). The upper base of the stage is an Al honeycomb (1.2×2 m^2). The height of the stage is controlled by the pulse motor drive so that it follows the incident beam. The stroke is 250 mm and the step is 0.0005 mm/pulse.

4. Control

The X-ray optics are controlled from a personal computer through the beamline workstation. Also, the measurement facilities in the experimental hutch were controlled from this computer linked with X-ray optics. The control programs are written in a commercially available application software program (LabVIEW, National Instruments Co.)

In XAFS measurement, error occurs on the Bragg angle of the first crystal of the monochromator during the scanning motion, which reduces the beam intensity, especially in the high energy region. To overcome this problem, the Bragg angle of the first crystal is corrected by a piezo actuator, which takes about 1 sec for adjustment. Also, after the XAFS scanning motion of the monochromator, a waiting time for the start of data accumulation is necessary to stop the vibration of the monochromator. Another 2-3 sec/data point is required to measure the XAFS data.

References

[1] T. Uruga *et al.*, J. Synchrotron Rad., in press.
[2] S. Emura *et al.*, SPring-8 Annual Report 1996, 82 (1996).
[3] K. Sato *et al.*, SPring-8 Annual Report 1998, (1998).