# Physicochemical Analysis

Shunji GOTO
Naomi KAWAMURA
Motohiro SUZUKI

## 1. Introduction

Physicochemical analysis beamline BL39XU is a public beamline for groups involved with X-ray magnetic absorption and scattering, spectrochemical analysis, and medical applications.

BL39XU is one of the standard hard X-ray undulator beamlines. The beamline optics and the experimental station of BL39XU are shown in Fig. 1. The major part of the beamline was constructed until September 1997 and test operations including adjustment of the beamline optics and radiation surveys around the radiation shielding hutches were started on September 28. We were also able to carry out similar construction and commissioning processes to the preceding beamlines.

## 2. Beamline

### *Undulator*

An in-vacuum undulator with a period length of 32 mm and a period number of 140 has been installed and generates linearly polarized X-rays ranging from 5 to 70 keV (fundamental to fifth peak).

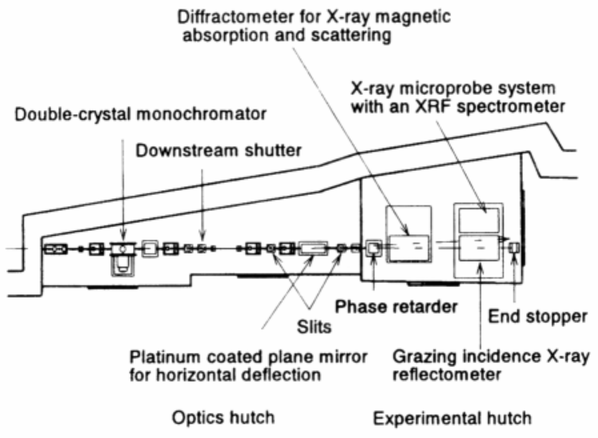

Figure 1. Beamline optics and experimental station of BL39XU.

### *Monochromator*

A rotated inclined double-crystal monochromator is placed at 36 m from the source to eliminate high heat loads. Photon energies from 5 to 17 keV can be obtained by combining the fundamental peak of the undulator and Si 111 reflection, and higher energies up to 37 keV can be obtained by using the third peak and Si 111 reflection. The beamline provides us with X-rays with an energy resolution DE/E of 10-4, and photon flux of 1012 photons/s/mA according to the calculation for perfect crystals.

The monochromator stages, and crystals were aligned based on the same procedure with which the optics group had been established. The pin-post directly water-cooled crystal was mounted as the first crystal.

After the alignment of the stages and energy calibration, we checked the beam shift perpendicular to the beam direction at the sample position. We found the movement of the beam to be 1 mm in the horizontal direction with 7 to 37 keV scanning. One difficulty for the fixed-exit was due to an inclined geometry of 80 degrees, and also due to imperfection of the pin-post crystal. It was difficult to find the exact Bragg peak due to broadening of the rocking curve and spotted beam. Moreover, a small deviation from the Bragg angle enlarged the change of the reflected beam direction mainly in the horizontal direction about ten times larger; this is inherent for dynamic diffraction at an inclined geometry [1]. Further study is necessary for the fixed-exit, including the establishment of alignment process and a beam position measurement method.

In addition, we found extra horizontal beam divergence due to imperfection of the pin-post crystal at an inclined geometry found in other undulator beamlines [2].

### *SR spectra*

Preliminary measurement of undulator spectra was done during the beamline commissioning period. We used an NaI scintillation counter for the spectrum measurement. We measured scattered photons in the air downstream from the beryllium window. From this, we obtained the absolute intensity passing through the monochromator and beryllium window, by correction of the absorption by the window and air, the scattering amplitude of air, and the scattering volume by the slit system.

Figure 2 shows measured and calculated spectra of an undulator gap of 12 mm; the front-end XY-slit size is 0.5 × 0.5 mm$^2$. An extreme discrepancy between them can be observed in the peak intensity (about one-fifth the calculated intensity) and line-shape of the spectra. Spectrum improvement, however, was found after tuning of the storage ring operation and insertion device.

Detailed results will be shown elsewhere. Furthermore, we can expect twice the intensity by improving the pin-post crystal fabrication process in the future.

*Horizontal deflection mirror*

A platinum-coated fused quartz plane mirror for harmonics rejection is located at 44 m away from the source. The SR beam is deflected to the horizontal direction to keep the beam height constant. The glancing angle can be changed from 0 to 10 mrad with a cutoff energy down to 8 keV. The mirror reflects 7- to 30-keV photons with a reflectivity of 70% and a third harmonics-to-fundamental reflectivity ratio of 10-3.

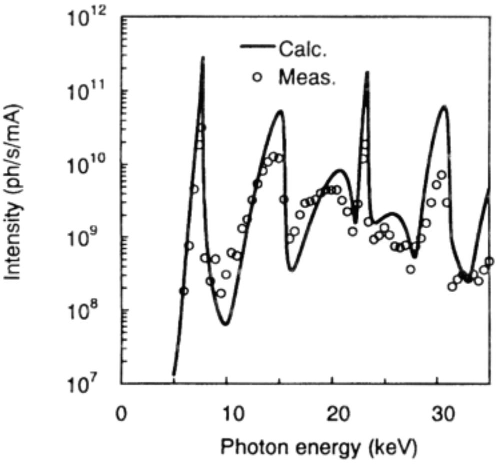

Figure 2. Photon flux spectrum at BL39XU observed in a preliminary measurement for an undulator gap of 12 mm and a front-end XY-slit of 0.5 × 0.5 mm$^2$

The moving mechanism of the downstream vacuum components from the mirror was designed to follow the reflected beam. The slit 1.1 m apart from the mirror and the beryllium window 2.2 m apart from the mirror move perpendicular to the direct beam with a moving ratio of 1:2, and they pass the reflected beam at their centers. We plan to install a mechanism, align it, and evaluate the reflectivity and third harmonics-to-fundamental reflectivity ratio from the beginning of 1998.

Until the end of 1997, beamline tests and experiments were carried out without a mirror.

*Phase retarder*

In addition to the standard optics, BL39XU has an X-ray phase retarder at the end of the beamline (inside an experimental hutch) to produce either circular polarization or vertical polarization.

The goniometer and stages for the X-ray phase retarder were designed and installed downstream from the beryllium window at the experimental hutch in November. After alignment of the system and evaluation of the polarization, the retarder was used for experiments on X-ray magnetic scattering and absorption.

A 0.7-mm-thick diamond crystal of a (111) plane is used. The transmission of the 111 Bragg case reflection and 220 Laue case reflection was evaluated. We checked the alternation of helicity of the circular polarization, and the production of the vertical polarization, by changing the offset angle around the Bragg angle. For example, we obtained values for the degree of circular polarization up to 99.5% for the 220 Laue case at a photon energy of 7.12 keV with 15% transmittance.

When the phase retarder is used in the region of a few keV to 30 keV, we must optimize the crystal, reflection plane, and thickness, from the viewpoints of transmittance and controllability of polarization (controllability of offset angle). The alternation of the helicity using a piezo-actuator is being planned by Suzuki. Additionally, stages are being prepared to follow the reflected beam from the mirror.

Experimental results of the phase retarder are reported elsewhere [3].

## 3. Experimental station

Apparatuses are located in the experimental hutch as shown in Fig. 1. The phase retarder is located just downstream from the beryllium window. The diffractometer for X-ray magnetic absorption and scattering is located next to the phase retarder on a sliding table. At the end of the hutch, an X-ray microprobe system with an XRF spectrometer [4] is placed on a bench with a sliding table. A grazing incidence X-ray reflectometer will be placed on the same bench. These three apparatuses can be set on the beam and the beam time can be shared. Detailed are shown in the following group reports.

We prepared vacuum pipes and helium pipes between the beryllium window and each of the apparatuses to prevent air scattering and absorption.

### *Apparatus to be prepared*

### *(a) Electromagnet and cryostat for X-ray magnetic absorption and scattering*

The basic specifications of an electromagnet and a cryostat for X-ray magnetic absorption and scattering have been fixed and these items are being manufactured. They will be mounted on the w-axis of the diffractometer. The electromagnet is of the normal conducting type and it will supply 0.6 T at a 45-mm gap, 1.3 T at a 20-mm gap, and 1.9 T at a 10-mm gap.

The cryostat will refrigerate a sample down to 20 K within 80 minutes. Three sample holders will be prepared for the electromagnet gaps. These will be installed in the experimental hutch and will be used from April 1998.

### *(b) Grazing incidence X-ray reflectometer*

A grazing incidence X-ray reflectometer was designed by Sakurai [5]. This will be mounted on the same bench as the X-ray microprobe system with an XRF spectro-meter. Installation will be in February 1998.

## References

[1] R. C. Blasdell and A. T. Macrander, Nucl. Instr. Meth. A347, 320 (1994).

[2] M. Yabashi, in this report.

[3] M. Suzuki et al., in this report.

[4] S. Hayakawa et al., to be published in J. Synchrotron Rad. 5 (1998).

[5] K. Sakurai et al., to be published in J. Synchrotron Rad. 5 (1998). Rad (1998).