Physicochemical Analysis (BL39XU)

1. Introduction

The Physicochemical analysis beamline BL39XU is a hard X-ray undulator beamline that is mainly used for X-ray magnetic absorption/scattering (XMAS), X-ray micro-spectrochemical analysis in materials and biology, and ultra trace element analysis.

In 1999, the following features and pieces of equipment were added to BL39XU: (1) the photon flux has been increased because the storage ring current is now up to 100 mA, (2) the peak energy of the undulator spectrum can be tuned within ±10 eV to the X-ray energy selected by the Si 111 monochromator, (3) a piezo actuator was installed in the monochromator to quickly tune the first crystal parallel to the second one, (4) for polarization-dependent EXAFS spectroscopy, the angle of an X-ray phase retarder (XPR) crystal can be tuned using a fit function which correlates the Bragg condition of the XPR with the X-ray energy, (5) three stages of a diffractometer for XMAS measurements were improved by mounting stepping motors, and (6) a pair of elliptical mirrors was designed and fabricated for X-ray fluorescence spectromicroscopy. In this report, the current status of BL39XU is described as it stands at the beginning of 2000.

2. X-ray Source and Optics

The storage ring has been operated at a ring current of 100 mA since June 1999, so that the photon flux has increased to 1×10^{13} ph/s after the monochromator. To make an investigation into the heat load of the first crystal of the monochromator, some characteristics of the monochromatized X-ray beam, such as the rocking curve width, the beam profile and the photon energy, were measured as a function of the FE slit's aperture and the operating current of the storage ring. These characteristics were mostly conserved at a ring current between 10 and 100 mA as far as the aperture of the FE slit was up to 0.75×0.75 mm^2. However, when the aperture of the FE slit was set at 1×1 mm^2 at 100 mA, the incident photon flux was less and the width of the rocking curve was broader than at 70 mA. The reason for this is considered to be that the thermal degradation of the first crystal was imperfect. The first crystal of the monochromator currently used at BL39XU is a first model of the pin-post crystal [1]. A new model of the pin-post crystal with an improved watercourse [2] could efficiently reduce the heat load at the 100 mA storage ring current, and the photon flux would increase as high as allowed by the current increment. A new-type pin-post first crystal will be installed in September 2000.

For EXAFS spectroscopy using an undulator source, the undulator gap must be controlled by synchronization with the monochromator to maintain the incoming X-rays at maximum intensity. To control the gap correlated with the X-ray energy, the following function has been used,

$$G = \alpha \ell \mathrm{n}\left(\frac{\beta}{E} - 1\right) + \gamma, \qquad (1)$$

where, G (mm) represents the undulator gap, E (keV) is the X-ray energy, α, β, and γ are fitting parameters. A fit of the function to the measured result of the X-ray energy of fundamental harmonics with the gap value yields $\alpha = -4.988$, $\beta = 18.65$ and $\gamma = 13.72$. The peak energy of the undulator spectrum could be tuned to the optimum energy of X-rays using the Si 111 reflection within ±10 eV between 6 and 16 keV by using this function with these parameters.

A piezo actuator has been installed in the monochromator to quickly tune the first crystal parallel to the second one. The tunable range is 30 arcsec. This piezo-tuning system is similar to that which has been adopted at BL01B1 [3]. The intensity of the monochromatized X-ray beam is monitored using an ionization chamber placed at the most upstream position in the experimental hutch, and the parallel condition of the double-crystal arrangement is decided by a fast scan using the piezo actuator within 1 s. By adopting this piezo-tuning system together with the control of the undulator gap, the intensity of incident X-rays is maximized and almost constant over several keV. It takes 1.5 s to adjust the undulator gap and the parallelism of the monochromator.

An XPR [4,5] of diamond (111) crystal has been used to generate circular or vertically linear polarization. Crystals of different thickness (0.34, 0.45, 0.73, 2.7 and 4 mm) used in the 220 Laue or 111 Bragg transmission geometry cover a wide energy range of 5.7–16 keV. To perform polarization dependent EXAFS experiments, the angle of an XPR is controlled according to a polynomial fit function which correlates the measured Bragg condition of the XPR crystal with an X-ray energy between 6 and 16 keV. A circular (vertical linear) polarization rate higher than 90% (70%) could be obtained using an XPR crystal of appropriate thickness with an appropriate offset angle.

3. Experimental Station

3.1 X-ray Magnetic Absorption / Scattering

Three stages of a diffractometer [6] used for XMAS measurements have been improved by equipping stepping motors. The improved stages were a horizontal translation stage perpendicular to the beam axis (X), a

horizontal (X_{4c}) and a vertical (Z_{4c}) translation stage for the 4-circle goniometer. The resolutions of X, X_{4c} and Z_{4c} stages were 3, 1 and 3 μm/pulse, respectively. This improvement enabled us to align the sample or the polarization analyzer crystal with the incident X-ray beam more precisely in a shorter time. This was useful for experiments using the diffractometer, in particular, measurements of resonant magnetic scattering including polarization analysis.

The influence of a stray field of a superconducting magnet (SCM) of 7 T on an electron orbit in the storage ring was investigated. A significant drift of the electron orbit was observed as the magnetic field of the SCM increased. The deflection of the orbit can be corrected by using the steering magnets on the storage ring if the variation of the magnetic field is slow enough. In a case where the direction of the magnetic field is parallel to that of the incident X-rays, i.e. the configuration of XMCD, the allowable sweep rate is less than 0.7 T/min. The rotation rate at 7 T is allowed to be slower than 9 degrees/min. An SCM of 10 T with a liquid He recondensing system will be installed at the beginning of 2001.

3.2 X-ray Fluorescence Micro-Spectrometer

To realize an energy-tunable X-ray microbeam for spectromicroscopy in the hard X-ray region, a pair of elliptical mirrors (Kirkpatrick and Baez (KB) mirror) was designed and fabricated [7,8]. Preliminary evaluation of the KB mirror was carried out using 10 keV monochromatized undulator radiation. Alignment of the mirrors was assisted by a beam monitor system composed of a scintillator and a CCD, and a beam size of 2×4 μm^2 was obtained with the photon flux of 1×10^{10} ph/s. A smaller beam size may be expected with optimum alignment, and the characterization of trace elements with the spatial resolution will be carried out using X-ray fluorescence (XRF) analysis and X-ray absorption fine structure measurements with an XRF yield method.

References

[1] M. Kuroda *et al.*, J. Synchrotron Rad. **5** (1998) 1211.

[2] H. Yamazaki *et al.*, submitted to SRI2000 Conf. Proc.

[3] SPring-8 Annual Report 1998 (1998) 42.

[4] K. Hirano *et al.*, Jpn. J. Appl. Phys. **30** (1991) L407.

[5] M. Suzuki *et al.*, Jpn. J. Appl. Phys. **37** (1998) L1488.

[6] H. Maruyama *et al.*, J. Synchrotron Rad. **6** (1999) 1133.

[7] S. Hayakawa *et al.*, X-ray Microscopy, edited by W. Meyer-Ilse, T.Warwick and D. Attwood, p.92, American Institute of Physics 2000.

[8] S. Hayakawa *et al.*, submitted to XAFS-XI Conf. Proc.

Light Source	
Type	In-vacuum undulator
Undulator period	λ_μ = 32 mm
Number of periods	N_{period} = 140
Tunable range	5 – 70 keV (fundamental to 5th)
Peak brilliance	2×10^{19} ph/s/mrad^2/mm^2/0.1%b.w. (100 mA)
Total power	11 kW (at 5 keV, *K*=2.3)
Power density	470 kW/mrad^2

X-rays at Sample	
Energy range	5 – 37 keV
Energy resolution	~ 2×10^{-4} *
Photon flux	1×10^{13} ph/s *
Beam divergence	< 0.1 mrad
Beam size	0.5×1.3 mm^2 (V×H)
Linearly polarization	99.9%
Circularly polarization	> 90% **

* XY slit at front-end 0.5×0.5 mm^2, X-ray energy 7.74 keV.

** with using a diamond X-ray phase retarder

Facilities in Experimental Station

(1) For magnetic scattering / absorption

- Diffractometer (3-axes diffractometer and 4-axes goniometer)
- Electromagnet and power supply (H_{max} = 0.6, 1.1, 2.0 T with 45, 20, 10 mm gap, respectively)
- Cryostat and vacuum pump
  - Cryostat A (T = 15–300 K, fitted with the electromagnet)
  - Cryostat B (T = 15–300 K, fitted with the 4-axes goniometer)
- Channel-cut Si 333 and 331, and LiF 220 analyzer crystals
- Ionization chambers and their electronics
- NaI scintillation counter and its electronics
- Si (Li) detector and its electronics
- Si photodiode

(2) For microscopic analysis

- X-ray fluorescence micro-spectrometer
  Vacuum chamber, Pin hole device, Precision sample stage, PSPC with wavelength-dispersive crystal analyzer, Si (Li) detector, and its electronics
- CCD camera monitor system

(3) For ultra-trace element analysis

- Grazing-incidence reflectometer
  Vacuum chamber, Goniometer stage of 0.005 arcsec/step, Glove box, Ionization chamber, Si (Li) detector, and its electronics

(4) Others

- Diamond X-ray phase retarder (0.34, 0.45, 0.73, 2.7, and 4 mm in thickness)
- Lock-in amplifier, Function generator, Current amplifier, Digital multimeter
- Pen recorder, Oscilloscope
- Four jaws slit, Replaceable attenuator, Light chopper (chopping frequency : 5–20,000 Hz)
- Vacuum and He pipes for X-ray path
- Oil-free scroll pump
- Monitor camera system
- Clean bench
- Optical microscope
- Dewar vessel for LN_2
- Magnetometer
- Desiccator and vacuum pump
- Ultrasonic cleaner