Physicochemical Analysis (BL39XU)

1. Introduction

The physicochemical analysis beamline BL39XU is a hard X-ray undulator beamline that is mainly used for studying X-ray magnetic absorption/scattering (XMAS), X-ray micro-spectrochemical analysis in materials and biology, and ultra trace element analysis. This beamline has been open to public use since October 1997.

In 1998, several features and pieces of equipment have been added to BL39XU: (1) photon flux has been increased by starting storage ring operations at 70 mA, and (2) a plane mirror for higher harmonics rejection, (3) an electromagnet and a cryostat for XMAS experiments, (4) an X-ray microspectrometer for X-ray fluorescence (XRF) analysis and (5) a grazing-incidence reflectometer for ultra trace element analysis have been installed. This article describes these new features and the current status of BL39XU at the beginning of 1999.

2. Insertion Device and Optics

BL39XU is equipped with an in-vacuum type linear undulator [1] (λ_u= 32 mm, N = 140) and a rotated-inclined double crystal monochromator [2] . The features of the X-ray beam, located in the experimental hutch, are summarized in Table 1. The tunable range of the undulator gap is from 9.6 to 50 mm. A combination of the fundamental and third harmonics with the Si(111) reflection of the monochromator covers the energy range from 5.7 to 37 keV. It was confirmed that the monochromator with a pin-post water-cooled first crystal can efficiently reduce high heat load at the 70 mA storage ring current; this also maximizes photon flux as high as allowed by the current increment. Tuning of the undulator gap to the monochromator is also possible. One can set both the undulator gap and the crystal angle of the monochromator to an appropriate value, giving just the X-ray energy needed for use to a beamline PC.

A plane mirror placed after the monochromator has been installed to reduce the amount of higher harmonics. This mirror is made of 0.7 m-long fused quartz coated with 200 Å chromium as a binder and 500 Å platinum at the top. An X-ray beam is deflected in the horizontal plane while maintaining the beam height. The glancing angle can be changed from 2 to 9 mrad with a cutoff energy as low as 8 keV. For using high-energy X-rays, the mirror can be removed from the beam axis. Measured reflectivity curves at various energies indicate a 40 Å ,'short-wavelength' platinum roughness. Nevertheless, the mirror has a high low-angle reflectivity. From 6 to 20 keV, the third-harmonics-to-fundamental reflectivity ratio can be reduced to be less than 10^{-3} by adopting an appropriate glancing angle.

In addition to the standard optics, BL39XU has an X-ray phase retarder (XPR) [3, 4] at the end of a beam transport channel inside the experimental hutch. Diamond(111) crystals of various thickness (0.45, 0.73 and 4 mm) can be mounted as XPR and tuned to provide circular polarization over a wide energy range (6-15 keV). The circular polarization rate is more than 90% using a 0.73 mm-thick crystal at 7.12 keV [4] . Since the maximum vertical polarization rate is 80% at the same energy, further studies are necessary to improve this value.

3. Experimental Station

3-1 Apparatus for X-ray Magnetic Absorption / Scattering

A diffractometer with an electromagnet and a cryostat has been developed for experiments on magnetic scattering in the horizontal plane as well as on X-ray magnetic circular dichroism (XMCD). The diffractometer consists of a triaxial goniometer and a 4-circle goniometer. The triaxial goniometer carries a sample with the electromagnet on its ω-circle, a scintillation counter or a solid-state detector on its ω-arm. To make a full linear polarization analysis, either Si(333) or Si(331) channel-cut analyzer crystals can be mounted on the 4-circle goniometer on a robust 2θ-arm of the triaxial goniometer; these crystals can rotate about the X-ray beam scattered by the sample.

Table. I X-rays at BL39XU

energy range	5.7 - 37 keV
resolution in $\Delta E/E$	~ 1×10^{-4}*
flux at sample	7×10^{12} ph/s (70 mA)*
beam size at sample	0.5×1.3 mm^2 (VxH)*
higher harmonics content	< 1×10^{-4}
linear polarization	99.9%
circular polarization	> 90%**

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

**with a diamond phase retarder, X-ray energy 7.12 keV.

The electromagnet is of the normal conducting type and generates a magnetic field of 0.6 T at a pole piece gap of 45 mm, 1.1 T at a 20 mm gap, and 2.0 T at a 10 mm gap. The field polarity is reversed in 1.7 s by changing the polarity of the feeding current. The field direction can be changed to parallel, perpendicular, or tilted by 45° with respect to the X-ray beam. The cryostat can cool a sample down to 20 K within 90 min, and the controllable temperature range is from 20 to 300 K.

In addition, an accurate data acquisition system for XMCD experiments has been developed by means of a helicity-modulation technique [5] . This system makes it possible to measure XMCD signals as small as 10^{-5} within 20 s for one data point.

A superconducting magnet (SCM) of 10 T has been designed for XMCD and non-resonant magnetic scattering experiments. The SCM will be mounted on the (ω-circle of the diffractometer. A liquid helium recondensing cooler can operate the SCM for 14 days without supplying additional coolant. The sample temperature is controllable between 1.5 and 300 K. A motor-driven two-axis manipulator can change the sample orientation. The SCM will be installed in the experimental hutch and used from October 1999.

3-2 X-ray Fluorescence Micro-Spectrometer

An X-ray fluorescence micro-spectrometer [6] has been developed for XRF analysis and spectroscopy with 100 to sub-μm spatial resolution. This apparatus comprises a pinhole device for collimating X-rays and precision stages for scanning a sample. To collect XRF signals, either an energy-dispersive spectrometer (EDS) using an Si(Li) detector or a wavelength-dispersive spectrometer (WDS) using a fiat analyzer crystal with a position-sensitive proportional counter are attached.

Ultra trace element analysis and micro-XANES studies using the EDS at BL39XU has the advantage of good counting statistics over those with second generation synchrotron radiation sources. Several studies have been carried out using the EDS, such as analysis of impurities contained in a synthetic diamond and imaging of the distribution of trace transition metals in a biological specimen. Preliminary experiments with the WDS, which were applied to standard metal foils, indicate that XRF spectroscopy with 2-60 eV energy resolution is feasible.

X-ray focusing mirrors in a Kirkpatric-Baez configuration will be added in the vacuum chamber of the micro-spectrometer in September 1999. The focused beam size on a sample will be less than ϕl μm while the photon flux is expected to be ten times as high as that without the mirrors. By using the focusing mirrors, an XRF imaging of 100 ppb diluted elements with sub-μm spatial resolution will be possible. Also, an X-ray polarization microscope is a feasible application by combining the X-ray micro-spectrometer and an XPR.

3-3 Grazing Incidence Spectro-Reflectometer

A grazing incidence spectro-reflectometer [7] has been equipped for surface/interface studies by means of total reflection X-ray fluorescence (TXRF) as well as specular/non-specular X-ray reflection. A single-axis goniometer is used to rotate a sample. The angular resolution is 0.005 arcsec and the reproducibility is better than 0.04 arcsec. This reflectometer is also intended for use in ultra trace element analysis by TXRF. To improve the signal-to-background ratio as well as to reduce stray fluorescent X-rays, a low pressure chamber and an oil-free pump are adopted, and a teflon surface finish is added to each part around the sample and the XRF detector. By using the reflectometer, contamination of transition metals can be detected on the order of 10^9 atom/cm^2 on the surface of a silicon wafer .

The commissioning of the reflectometer was completed in May 1998. The following experiments are currently planned: (1) ultra trace chemical analysis of metals in a water drop, (2) nm-scale surface topography by means of X-ray surface scattering, and (3) interface evaluation in thin films by combining TXRF and diffuse scattering.

References

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