An XAFS Beamline at the SAGA Light Source

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Abstract. A new hard X-ray beamline, BL15, has been designed and constructed at the SAGA-Light Source. The beamline is optimized for industrial applications of the synchrotron light. X-rays with photon energies from 2.1 keV to 14.2 keV are delivered to the experimental station passing a fixed-exit double-crystal Si(111) monochromator and a bent cylindrical mirror. Basic experimental equipments for XAFS measurement, high resolution diffractometry, various kinds of X-ray imaging and energy-dispersive diffractometry have been prepared for the station. From our initial commissioning and performance testing of the beamline, we show that BL15 can perform XAFS measurements.

Keywords: Synchrotron radiation, SAGA Light Source, Beamline, Double Crystal Monochromator, Bent Cylindrical Mirror, Experimental Station, XAFS.
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INTRODUCTION

Saga prefecture, a local government in the Kyushu district of Japan, operates a synchrotron light facility, named Kyushu Synchrotron Light Research Center, for regional industries to develop advanced industrial technologies as well as to promote fundamental material sciences [1]. It is expected that the facility will play the role of an Asia-wide technology interchange center for light-related industries.

The light source, named SAGA-LS, is a 1.4 GeV storage ring with a circumference of 75.6 m [2]. The critical energy of the photons from the bending magnets is 1.9 keV. Twenty beamlines at maximum can be set up around the ring. The Saga prefectural government has prepared three beamlines; a white-light / EUV beamline, a soft X-ray beamline and a hard X-ray beamline. Another VUV / soft X-ray beamline connecting to an undulator has been installed by Saga University. These beamlines are under commissioning and partly opened for trial use of the synchrotron light.

BL15 is the hard X-ray beamline. The beamline is designed to facilitate industrial applications as well as academic research based on the characterization of materials.

In the following, we describe the outline of the beamline optics and the experimental station, including some results of performance testing.

BEAMLINE

The BL15 consists of a front end in the ring tunnel, a transport channel and an experimental station in the experimental hall. The experimental equipment is set up in an experimental hutch with dimensions of 6 m (length) × 3 m (width) × 3 m (height).

The front end is evacuated by ion pumps and titanium sublimation pumps to achieve an ultra-high vacuum (10^{-8} – 10^{-9} Pa), since it is directly connected to the storage ring of 10^{-9} Pa. The front end also shields the experimental stations from the *Bremsstrahlung* during electron injection into the storage ring. A Be window with the thickness of 0.125 mm is mounted at the exit port of the front end, separating the vacuum in the storage ring from the 10^{-6} Pa vacuum in the transport channel yet allowing X-ray transmission. The transport channel is equipped with a monochromator to provide monochromatized X-rays for the experimental station. The schematic view of the beamline is given in Fig. 1. It consists of vacuum and optical components. Turbo-molecular pumps evacuate all transport channel components to 10^{-5}–10^{-6} Pa, preventing contamination of the optical

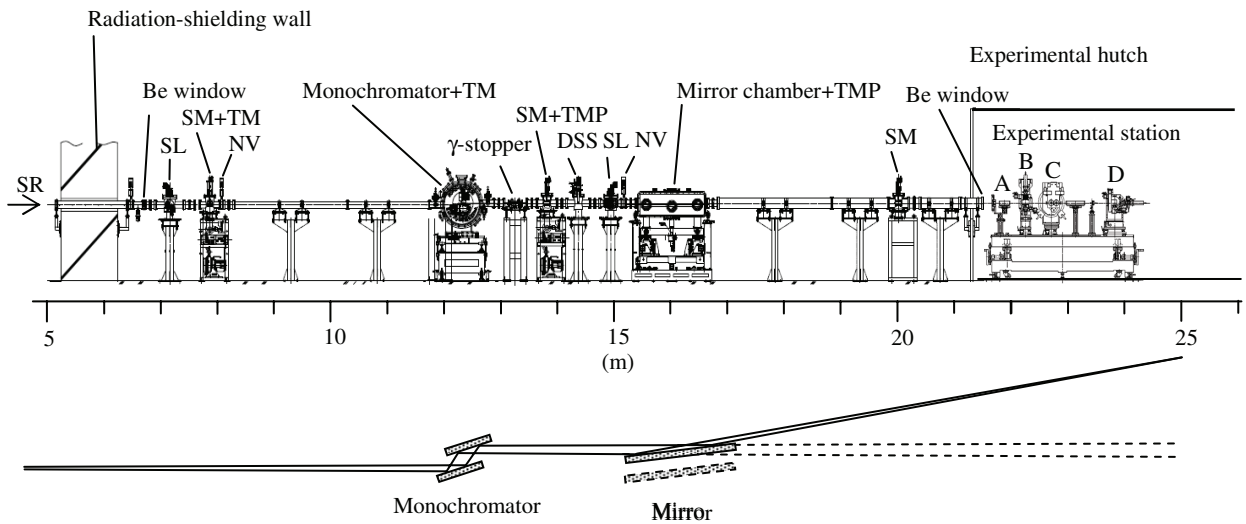

FIGURE 1. Side view of the beamline BL15. TMP: turbo-molecular pump; SL: 4-jawed slit; DSS: downstream shutter; SM; screen monitor; NV: pneumatic gate valve. The Arabic numerals under the figure show the distance from the source point.

components and X-ray absorption by ambient gas. Be windows, 0.25 mm and 0.125 mm thick, at the end of the transport channel transmit X-ray to the experimental hutch, and separate the transport channel vacuum from the low vacuum (about 1 Pa).

The X-rays form the front end hit the first Si(111) crystal of the fixed-exit double-crystal monochromator. The main rotational axis coincides with the surface plane of the second Si(111) crystal. A water-cooled holder cools the first crystal. The monochromatized X-rays are focused using a bent cylindrical mirror located downstream from the monochromator. X-rays with photon energies from 2.1 to 14.2 keV are available at the experimental station. The mirror is coated with rhodium and the angle of incidence is 4 mrad, which corresponds to a cutoff energy of 16 keV. The values for the meridian radius of curvature R_m and sagital radius of curvature R_s are 2880 m and 46.0 mm, respectively. The mirror focuses the beam into a sub-millimeter spot at the focal point 25 m from the light source with the photon flux of more than 1×10^{11} photons/sec at 8 keV [3,4].

EXPERIMENTAL STATION

The experimental station is composed of four units A, B, C, and D as seen in Fig. 1. Unit A consists of a receiving slit and an ionization chamber. The incident beam from the transport channel is apertured by the receiving slit and the photon flux is monitored by the ionization chamber. The main components of the Unit B are a pulse-motor driven X - Z stage and a θ - 2θ stage mounted on the X - Z stage. Using these stages, we can perform θ - 2θ type powder diffraction measurement and X-ray reflectivity measurement. Unit C is an ultra high precision goniometer, which has two parallel ultra high resolution rotation axes driven independently by two tangent bar mechanisms. The resolution is 0.01"/step. This unit is used for the high precision X-ray imaging using X-ray imaging interferometry. Unit D is an off-centered 4-circle diffractometer. Various kinds of detectors such as ionization chambers, germanium single-element solid state detector, NaI (Tl) type scintillation counters, imaging plates, Lytle type detector and conversion electron yield detector are also available in the experimental station. By combining these units and detectors, various experiments as described above can be performed. In particular, XAFS spectra are measured in transmission mode, X-ray fluorescence mode and electron yield mode using these detectors.

PERFORMANCE TEST

We have measured some reference samples at BL15 in order to evaluate the initial performance of the beamline. The obtained Cu K-edge EXAFS spectrum of a Cu foil at BL15 of SAGA-LS is shown in Fig. 2 (a). For comparison, the spectrum measured at BL7C of KEK-PF is shown in Fig. 2 (b). The inserts show the X-ray absorption near edge structure around the K absorption edge. Both spectra were measured in transmission mode. From the magnitude and frequency of the oscillations, it can be seen that the EXAFS spectrum of Cu foil obtained at SAGA-LS is in good agreement with that measured at KEK-PF. The pre-edge peak at 8979 eV was observed in both spectra with similar resolution. These results indicate

that the SAGA beam line is functional at this point and for the initial samples.

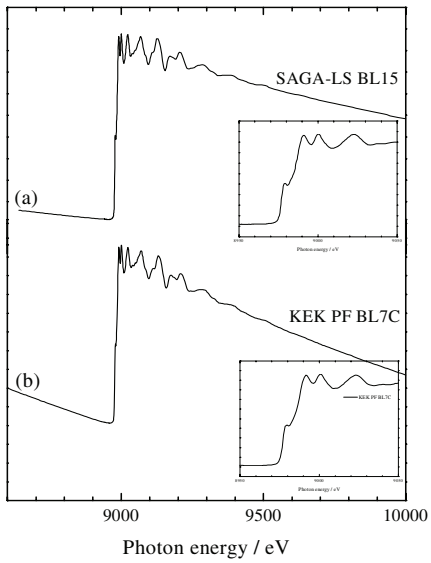

FIGURE 2. Comparison of Cu K-edge XAFS spectra measured at (a) SAGA-LS BL15 and KEK-PF BL7C.

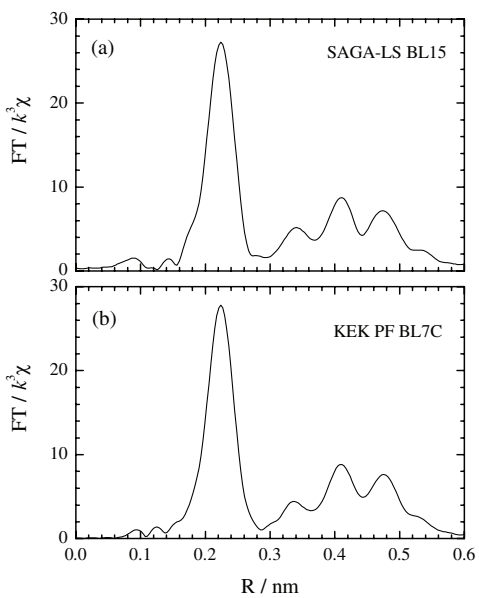

FIGURE 3. Fourier transforms of the EXAFS spectra shown in Fig. 2.

Figure 3 shows the radial distribution functions obtained from Fourier transform of $k^3\chi(k)$ in the EXAFS spectra shown in Fig. 2. This result displays clearly that the first neighbor coordination is about 0.22 nm far from the central Cu atom and two characteristic features of face-centered-cubic Cu structure appear at about 0.41 nm and 0.48 nm which correspond to the third and fourth coordination, respectively. Quantitative analysis for the data shown in Fig. 3 (a) was performed for the first to the third coordination shells. The resultant R-factor was about 3%. The bond length and σ^2 of the first shell were 0.2556(5) nm and less than 0.0001 nm^2, respectively. These results indicate that the XAFS station at BL15 of SAGA-LS is in working status.

SUMMARY

We have designed and constructed an X-ray beamline named BL15 for use in the characterization of industrial materials at SAGA-Light Source. The beamline covers the photon energy range from 2.1 to 14.2 keV. Beamline optics consists of a fixed-exit Si(111) double-crystal monochromator and a mechanically bent cylindrical mirror for vertical and horizontal focusing. In the experimental hutch, the experimental station can be set up for X-ray absorption fine structure (XAFS) measurement, high resolution diffractometry, topographic imaging and energy-dispersive four-circle diffractometry. XAFS spectra measured at BL15 has the initial performance for industrial applications. The use of the XAFS measurement system of the beamline began at the beginning of March, 2006.

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