Construction of a Wide-range High-resolution Beamline BL05 in NewSUBARU for Soft X-ray Spectroscopic Analysis on Industrial Materials

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Abstract. A material analysis beamline which is designed to useable in the wide energy range, 50–4000 eV for the various industrial demands, was constructed at BL05 in NewSUBARU for industrial companies’ use in cooperation with the Laboratory of Advanced Science and Technology for Industry (LASTI), University of Hyogo and well-informed researchers in the industrial world. BL05 consists of two branch lines, which is mounted with a double crystal monochromator at BL05A and grating monochromator at BL05B. XAFS measurements in the total electron yield mode and partial fluorescence yield mode using a silicon drift detector can be performed at the both branch lines. In addition, the photoelectron spectra can be measured at BL05B. The maintenance and management of BL05 are performed by Synchrotron Analysis LLC (SALLC), which is a consortium of user companies of BL05, under the supervision of the LASTI staff. If industrial users want help with measurements at BL05, operators of SALLC can assist them.

1. Introduction
The X-ray absorption fine structure (XAFS) technique in the soft X-ray region has attracted much attention from the industrial world as a powerful tool for investigating the electronic and geometric structures of industrial materials. In addition, photoemission spectroscopy (PES) technique is also an effective means for researching the electronic structure of material surfaces in the soft X-ray region. Thus, there is a need for the analysis ability in the soft X-ray region to increase with the development of nanotechnology in the industrial fields. A new beamline was constructed in response to such a demand in the industrial world. The decision on the optical design and specifications of end stations was made in cooperation with the University of Hyogo and industrial companies joining Synchrotron Analysis Limited Liability Company (SALLC). SALLC was established for the development and innovation of advanced industrial science and technology using BL05. This beamline is anticipated to
prosper the co-research with University of Hyogo and industrial companies, and expected to advance the analysis abilities of industrial companies.

2. Beamline description

NewSUBARU was constructed for the light source in the vacuum UV and soft X-ray region on the SPring-8 site. The circumference of the storage ring is 119 m and its electron energy is 1.0-1.5 GeV.[1] A wide-range high-resolution beamline was constructed at BL05 of NewSUBARU. BL05 consists of two branch lines for use in the wide range from 50 eV to 4000 eV. BL05A, which is mounted with a double-crystal monochromator, can be used in the energy range of 1300-4000 eV. On the other hand, BL05B is mounted with a variable-line-spacing grating monochromator, which is designed to cover the energy range of 50-1300 eV. The incident beam from the bending magnet as a light source is provided for two branch lines through different windows of a mask, which is mounted at the just downstream of storage ring. Therefore, these two branch lines can be employed simultaneously. At BL05A, the XAFS spectra in the total electron yield (TEY) mode and fluorescence XAFS measurement using SDD (SII Vortex) can be performed. The fluorescence XAFS spectra of wet or liquid samples can be measured at the end station filled with He gas. At BL05B, the XAFS spectra in the TEY mode and partial fluorescence XAFS spectra using SDD (Ourstex) can be measured in a high vacuum chamber. In addition, the photoelectron spectrum can be measured using spherical electron analyzer (VG Sienta, R3000) in an ultra high-vacuum chamber. The chambers can be replaced by each other within 1 hour. The intensity of incident X-rays was detected using the photocurrent coming from a gold or a nickel mesh, which was mounted at the front of measurement chambers.

2.1. BL05A

A layout of the BL05A is shown in Fig. 1. Acceptance angle is 7 mrad. The optical system consists of a first mirror (M0), a double crystal monochromator (DXM) and a focusing mirror (M1). InSb crystals and Si crystals are prepared for a double crystal monochromator. Toroidal mirrors (R = 229.2 m, r = 69.8 cm, Ni coating) are used as a pre-mirror and a focusing mirror. Incident angles into mirror are 89°.

![Fig. 1 Optical layout of BL05A](image)

2.2. BL05B

A layout of BL05B is shown in Fig. 2. Acceptance angle is 3 mrad. The constant-deviation monochromator consisting of a demagnifying spherical mirror and a variable-line spacing plane grating (VLSPG), which can provide high resolution, simple wavelength scanning with fixed slits, was mounted on BL05B. The optical system consists of a first mirror (M0), a second mirror (M1), an entrance slit (S1), a pre-mirror (M2), and three kinds of plane grating (G), an exit slit (S2) and a
focusing mirror (Mf). M0 and M1 are cylindrical mirrors (R = 171.9 m). M2 is the spherical mirror (R = 229.2 m) Mf is the toroidal mirror (R = 114.6 m, r = 61.1 cm). Each mirror is coated by Au. Incident angles in to M0, M1, M2, and Mf are 88°, 88.25°, 88.25°, and 89°, respectively. The deviation angle of the monochromator is 175°. The VLSPG achieves high resolution in the extreme ultraviolet region by diminishing various kinds of aberration. In the case of the VLSPG, the groove parameters can be expressed in the following form by expanding the groove density, N, with coefficients a_i [2]:

\[ N(w) = N_0 (1+a_1w+a_2w^2+a_3w^3+...) \]  

where \( N_0 \) is the groove density at the center of the grating and \( w \) is the distance from the center of the grating along the direction of light traveling. Aberrations, such as defocus, coma, and spherical aberration, were computed with the present geometrical and ruling parameters. For BL05B, we use three kinds of grating with groove densities of 100, 300 and 800 grooves/mm at the center of the grating, \( N_0 \) and the space variation parameters \( a_1, a_2 \) and \( a_3 \) are determined to \(-3.0727 \times 10^{-4}, 7.1 \times 10^{-8} \) and \(-1.7 \times 10^{-11} \), respectively, to minimize the aberration.

\[ \text{Fig. 2 Optical layout of BL05B} \]

2.3 Sample stage and sample holder
For the industrial demands, large-area sample holders and large-capacity holder stockers were prepared. Samples holder is a disk of the 49 mm diameter and sample can be mounted in the 25 mm \( \times \) 25 mm square in area on the sample holder. Identical sample holder can be used at BL05A and BL05B. Sample stage can be controlled x-, y-, and z-axis and incident angle \( \theta \) using a stepper motor. Sample holder can be stocked in the vacuum load lock chamber and can be transferred to sample stage using a transfer rod. The 8 pieces and 16 pieces sample holders can be stocked in the load lock chamber at BL05A and BL05B, respectively.

3. Expected performance
Figure 3(a) and 3(b) show the expected diffraction efficiency and the calculated resolving powers of BL05B. Fig. 3(a) depicts that BL05B covers the energy range of 50-1300 eV using three kinds of grating. Resolving powers were estimated at entrance and exit slit widths of 50 and 100 \( \mu \)m with the slope error limit. A total resolving power of about 3000 can be realized in the entire energy region. On the other hand, a total resolving power is expected about 5000 in the entire energy region in BL05A.

Two sheets of observed spectra are shown in Fig. 4(a) and (b) for an example. The S K-edge NEXAFS spectra of several sulfur containing materials measured at BL05A with TEY mode are shown in Fig. 4(a).[3] The changes of energy position and spectral shape correspond to the environments of sulfur atom can be seen. The Ti L-edge NEXAFS spectra of TiO2 measured at BL05B with TEY mode are shown in Fig. 4(b). The difference in peak intensity of L3 between anatase and rutile was obviously distinguished as reported in a reference. [4]
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