BL37XU
Trace Element Analysis

1. Introduction

BL37XU is a hard X-ray undulator beamline for trace element analysis and chemical/elemental imaging dedicated to various X-ray spectroscopy methods such as scanning X-ray micro-spectroscopy, full-field X-ray micro-spectroscopy, and ultratrace element analysis [1,2]. By these methods, research is actively conducted to elucidate the properties and functions of materials through analyses of the morphology, element distribution, chemical state, and local structure. In FY2021, BL37XU operated smoothly and almost all users completed their user time as scheduled. In addition, the following two components were upgraded: (1) harmonic rejection mirrors and (2) silicon drift detector (SDD).

2. Upgrade of harmonic rejection mirrors

At BL37XU, high-quality spectroscopic measurements have been performed by using harmonic rejection mirrors to increase monochromatic purity. In recent years, the deterioration of the harmonic rejection mirrors has resulted in striped beam profiles, which causes difficulty in nanofocusing with Kirkpatrick–Baez (KB) mirrors and non-uniform illumination in full-field X-ray micro-spectroscopy measurements. Therefore, the harmonic rejection mirrors were updated at the end of FY2020. The mirror coating was changed from conventional Pt/Rh stripes to Pt/Ru stripes, but as before, the angle of incidence and coating surface can be selected according to the cutoff energy and absorption edge used. Figure 1 shows the beam profiles of the direct beam and with the harmonic rejection mirrors observed before (top) and after (bottom) the mirror upgrade. When using the upgraded harmonic rejection mirrors, the beam profile is almost the same as the direct beam profile, which makes it possible to use the monochromatic beam having a uniform distribution.

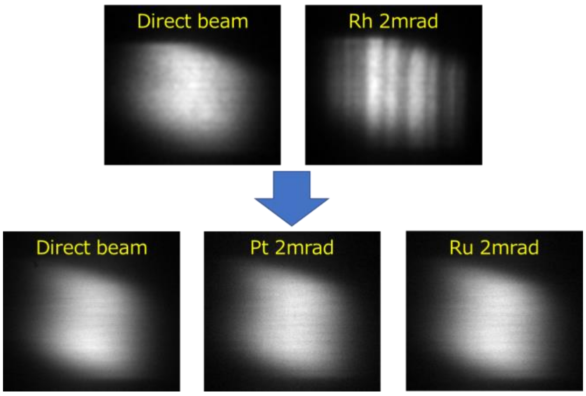

Fig. 1. Beam profiles of direct beam and with harmonic rejection mirrors. The top image is from the old mirrors (Term: 2019B, ID gap: 12.246 mm, FE slit: 0.3×0.3 mm, energy: 22.68 keV) and the bottom images are from the new mirrors (Term: 2021A, ID gap: 13.958 mm, FE slit: 0.5×0.5 mm, energy: 9.11 keV).

3. Upgrade of silicon drift detector

In the scanning X-ray micro-spectroscopy using a 100 nm X-ray beam focused by KB mirrors, a Ge solid-state detector (Ge-SSD) and a four-element SDD are used to detect fluorescence X-rays. Each existing detector had advantages and disadvantages. The Ge-SSD (EGX10-06-CP5-PLUS-WC; Mirion Technologies, Inc.), which was installed in FY 2019 [2], has an almost 100% detection sensitivity up to nearly 100 keV, but owing to the single element, its use is limited to measurements up to a maximum counting rate of about 1 Mcps. In contrast, the four-element SDD (Vortex-ME4; Hitachi High-Tech Science America, Inc.) decreased the detection sensitivity at energies above 10 keV, but a maximum counting rate of about 3 Mcps was available.

In recent years, the energy resolution of the SDD has deteriorated because of aging, and it has become difficult to use the SDD under the high counting rate condition, so we have upgraded it to a new seven-element SDD (Vortex-ME7; Hitachi High-Tech Science America, Inc.). The CUBE preamplifier maintains a high energy resolution at a high counting rate of 1 Mcps/element or higher, and the detector head size is the same as that of the conventional SDD, but the number of elements has been increased to seven, which enables its use at counting rates up to about 20 Mcps. Figure 2 shows the X-ray fluorescence (XRF) spectrum of Fe foil. The seven-element SDD has an energy resolution of 140 eV, which is higher than that of the conventional four-element SDD (275 eV). Also, under the high counting rate condition (peaking time: 0.2 us, input count rate: 1 Mcps), the energy resolution was 189 eV.

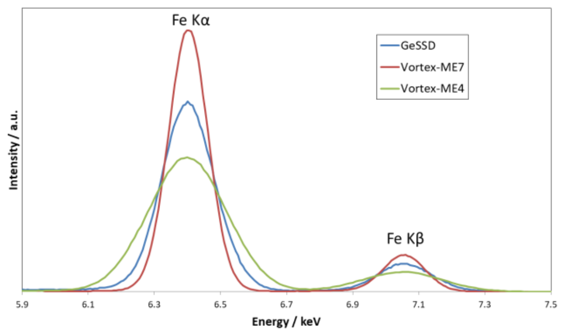

Fig. 2. XRF spectrum of Fe foil (peaking time: 1 µs, input count rate: 10 kcps).

Figure 3 shows the XRF spectrum of the standard reference material (SRM610, NIST). Each detector is located at the same position. Although the detection efficiency of the SDD decreases above 10 keV, the detection capability of the seven-element SDD is comparable to that of the Ge-SSD up to about 30 keV owing to the multiple elements. In addition, some peaks that could not be separated by the conventional four-element SDD owing to the low energy resolution can be clearly separated by the seven-element SDD, enabling a wide range of applications from low to high energies. The digital signal processor (DSP) was also updated along with the SDD. Previously, the maximum number of regions of interest (ROIs) was only 16 for on-the-fly scanning, but now, the full X-ray

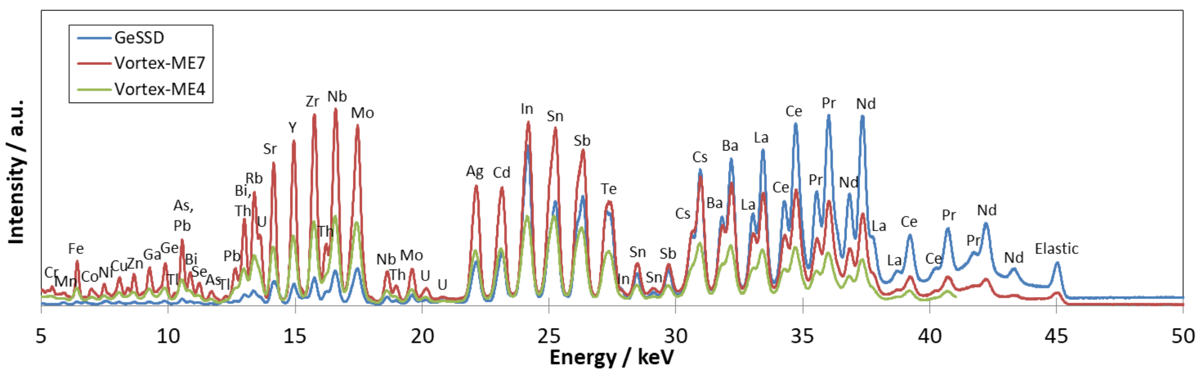

Fig. 3. XRF spectrum of SRM610 (incident X-ray energy: 45 keV).

fluorescence spectrum can be obtained at all points, enabling high-precision 2D XRF/XAFS measurements. The DSP also has a high-speed 10G-Ethernet communication protocol, which enables high-speed measurements with more than 800 frames/s/ch.

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References:

[1] Nitta, K. Sekizawa, O. & Suga, H. (2021). *SPring-8/SACLA Annual Report FY2020*, 67–68.
[2] Nitta, K. & Sekizawa, O. (2020). *SPring-8/SACLA Annual Report FY2019*, 62–63.