

## BL37XU (Trace Element Analysis)

### 1. Introduction

BL37XU is a hard X-ray undulator beamline for trace element analysis. It is used for various X-ray spectroscopy methods like scanning X-ray microspectroscopy, full-field spectroscopic imaging, depth-resolved XAFS, and ultra-trace element analysis. Using 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. There were no serious troubles in BL37XU in 2018, and almost all users completed their user time as scheduled.

In FY2018, the following features and pieces of equipment were installed to improve high-speed scanning micro XRF/XAFS measurements: (1) X-ray coaxial observation optical microscopy to simply adjust samples and simultaneous observations during X-ray measurements, and (2) a new Kirkpatrick-Baez (KB) mirror system to expand user activities in the higher-energy region.

### 2. X-ray coaxial observation optical microscopy

Previously, the scanning X-ray microspectroscopy was performed using sample position coordinates determined with an optical microscope located outside the experimental hutch, but this had problems with sample position repeatability. Therefore, an X-ray coaxial observation optical microscope (ULWZ-200M; Sigma Koki) was installed to adjust the sample position smoothly and accurately.

Figure 1 shows the layout of the scanning X-ray microspectroscopy measurement system. The

optical microscope has an aluminum flat mirror with 2-mm pinhole, which is inserted between the KB mirrors and the sample holder. The optical magnification can be changed from  $\times 0.58$  (FOV: 13.8 mm) to  $\times 7$  (FOV: 1.1 mm) by a remote control for easy sample adjustments. The X-ray passes through the pinhole and the optical microscope can be used during scanning X-ray microspectroscopy measurements. Consequently, the sample adjustment is simplified and simultaneous observations during X-ray measurement are available.

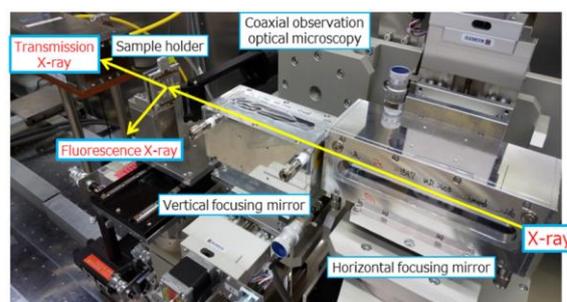


Fig. 1. Layout of the scanning microscopic XRF/XAFS measurement system.

### 3. High-energy 100-nm focusing KB mirrors

The previously available focused beam size of the scanning X-ray microspectroscopy measurement at BL37XU was 100 nm at 4.5–15 keV <sup>[1]</sup> and 300–500 nm at 15–37.5 keV <sup>[2]</sup>. In these conditions, it is impossible to separate the rare earth elements *L*-line XRF and 100-nm imaging of a fifth periodic element, which is important in materials research and catalytic reactions. To enable a 100-nm microspectroscopy imaging of these elements, we developed the KB mirrors (JTEC) for high-energy regions.

Table 1 shows the design parameters of high-energy 100-nm KB mirrors. The vertical source point is an insertion device (source size: 14.9  $\mu\text{m}$ ), while the horizontal source point is a secondary source slit (source size: 9.4  $\mu\text{m}$ ) located at the directly attached flange downstream of double-crystal monochromator (DCM). A surface of the mirror is striped with Rh and Pt coatings, and the coating can

be selected according to the measurement target. The X-ray reflectivity at 50 keV is about 40% using the Pt coating surface, and a 100-nm focused beam is available up to an energy of about 55 keV. Figure 2 shows the focused beam profiles at 50 keV by a knife-edge test with a Ta blade. Table 2 shows examples of a 100-nm-focused beam using surface of Pt coating. The photon flux of 100-nm-focused

Table 1. Design parameters of the focusing mirrors

|                              | Horizontal focusing mirror             | Vertical focusing mirror               |
|------------------------------|--|--|
| Surface shape                | elliptic                               |  |
| Substrate material           | silicon                                |  |
| Surface coating (thickness)  | stripes of Rh and Pt (100 nm)          |  |
| Mirror size                  | $200 \times 50 \times 50 \text{ mm}^3$ | $150 \times 50 \times 50 \text{ mm}^3$ |
| Glancing angle at center     | 1.6 mrad                               | 1.5 mrad                               |
| Distance from undulator      | 76,815 mm                              | 77,000 mm                              |
| Distance from secondary slit | 33,900 mm                              | 34,085 mm                              |
| Focal length                 | 360 mm                                 | 175 mm                                 |
| Acceptance size              | 320 $\mu\text{m}$                      | 225 $\mu\text{m}$                      |
| Height error                 | < 0.8 nm RMS                           |  |
| Surface roughness            | < 0.35 nm RMS                          | < 0.5 nm RMS                           |

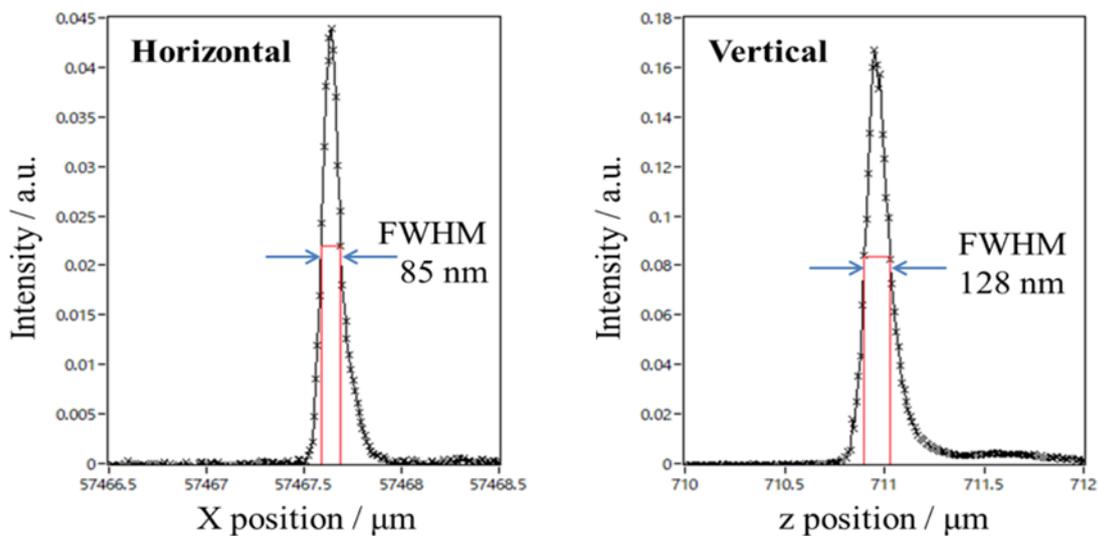


Fig. 2. Focusing beam profiles at 50 keV.

Table 2. Typical beam size and photon flux

| Energy / keV | Monochromator   | Beam size / nm |          | Photon flux / photons s <sup>-1</sup> |
|--------------|-----------------|----------------|----------|---------------------------------------|
|              |                 | Horizontal     | Vertical |                                       |
| 7            | Si(111)         | 128            | 110      | 9.3×10 <sup>9</sup>                   |
| 15           |                 | 99             | 103      | 2.6×10 <sup>10</sup>                  |
| 20           |                 | 169            | 114      | 3.1×10 <sup>10</sup>                  |
| 30           |                 | 91             | 93       | 6.9×10 <sup>9</sup>                   |
| 37.5         |                 | 93             | 112      | 5.1×10 <sup>9</sup>                   |
| 45           | Si(511)-Si(333) | 152            | 80       | 1.2×10 <sup>8</sup>                   |
| 50           |                 | 85             | 128      | 4.5×10 <sup>7</sup>                   |

beam is 10<sup>9</sup>–10<sup>10</sup> photons/s at 15–37.5 keV, which use Si(111) DCM, and 10<sup>7</sup>–10<sup>8</sup> photons/s at 37.5–50 keV, which use Si(511)-(333) DCM. Consequently, a 100-nm spatial resolution two-dimensional scanning microspectroscopy measurement is available over a wide energy range of 4.5–55 keV, and BL37XU will be used for various (trace) element measurements.

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#### References:

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 [2] K. Nitta and O. Sekizawa, *SPring-8/SACLA Annual Report 2017*, 68 (2018) [in Japanese].