

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 microspectroscopy, full-field X-ray microspectroscopy, and ultra-trace-element analysis [1]. 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. In FY2024, BL37XU operated smoothly and almost all users completed their user time as scheduled. In addition, the following project was undertaken: the installation of an automatic switching system for full-field X-ray microspectroscopy.

2. Upgrade of full-field X-ray microspectroscopy

The optics used in full-field imaging-type measurements have chromatic aberration, and the focal length of the condenser zone plate (CZP) varies from 403 mm (5 keV) to 1,209 mm (15 keV),

and the focal length of the Fresnel zone plate (FZP) varies from 62 mm (5 keV) to 185 mm (15 keV). Therefore, it was necessary to reposition and readjust the configuration whenever the absorption edge to be measured was changed. A new chamber for illumination optics, a stone surface plate, and an adjustment mechanism for imaging optics were installed, which enabled multiscale measurement by switching between projection type and imaging type, along with an automatic switching system for absorption edges. Figure 1 shows the layout of the full-field multiscale X-ray microspectroscopy measurement system.

The new chamber for illumination optics has two X-ray windows: one serves as a helium path for the projection type and the other accommodates the center beam stop, CZP, and order-sorting aperture for the imaging type. These can be switched to align with the X-ray optical axis. The CZP can be translated up to 1 m along the optical axis, enabling switching between the Ti K-edge (4.9 keV, $f = 395$ mm) and Y K-edge (17 keV, $f = 1,370$ mm),

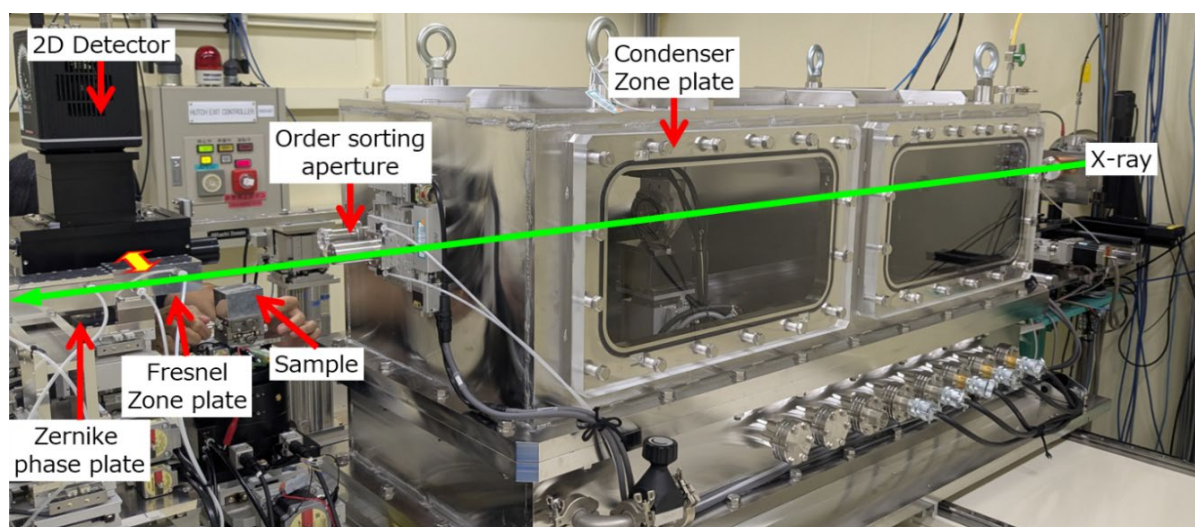


Fig. 1. Layout of full-field multiscale X-ray microspectroscopy measurement system.

depending on the absorption edge.

To suppress vibration during measurement, a stone surface plate was installed. Because the CZP continuously rotates and generates vibrations, the illumination chamber was isolated from the plate. A sample stage and an adjustment mechanism for imaging optics were mounted on the plate, which also incorporates a long-stroke X-stage to switch between a 2D detector for the projection type and an FZP for the imaging type. This configuration, combined with the illumination chamber, enables multiscale measurements in both projection and imaging modes.

The adjustment mechanism for imaging optics allows the FZP to move up to 80 mm along the optical axis. This travel range covers several practical configurations, for example, from the V K-edge (5.5 keV, $f = 68$ mm) to the Pt L_3 -edge (11.5 keV, $f = 142$ mm), and from the Pt L_3 -edge (11.5 keV, $f = 142$ mm) to the Y K-edge (17 keV, $f = 210$ mm).

Although the FZP does not provide as wide an energy-switching capability as the CZP, this range is considered sufficient. In practice, switching across a wider energy span would also require the adjustment of the higher-harmonics rejection mirror and changes to the imaging optics.

For precise alignment, a θ_x - θ_z stage is installed beneath the Y stage to adjust the FZP position. This ensures that the focal point remains fixed even when the Y stage moves to accommodate changes in focal length (Fig. 2). With this mechanism, imaging-type QXAFS measurements can be performed simply by linking the Y stage to the quick-scan function of the double-crystal monochromator.

Previously, the practical energy range was limited to 6.5–15 keV owing to atmospheric

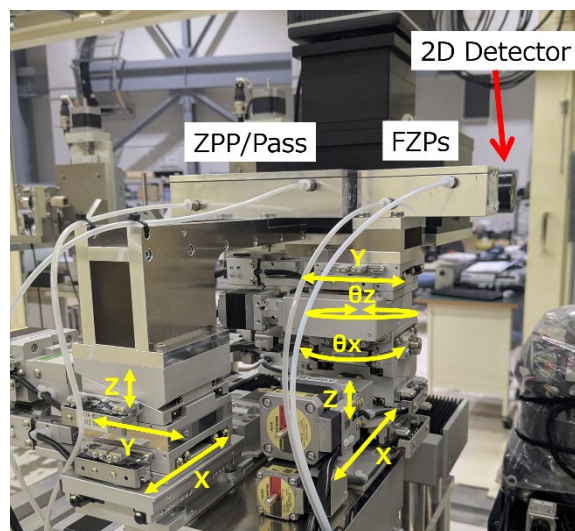


Fig. 2. Adjustment mechanism for imaging optics.

The arrows show the travel direction of each stage.

absorption and the diffraction efficiency of the optics. With this update, most of the X-ray path has been converted to helium, making it possible to access energies below 6.5 keV that were previously unmeasurable. As a result, imaging-type microspectroscopy at the V K-edge has been successfully achieved.

Multiscale full-field microspectroscopy is now possible with the projection type offering a field of view of 1 mm and a resolution of 1 μm , and the imaging type providing a field of view of 50 μm and a resolution of 80 nm. The absorption edges can be switched automatically via the control software. This new system is expected to significantly broaden the applicability of imaging spectroscopic measurements.

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References:

- [1] Sekizawa, O. & Nitta, K. (2024). *SPring-8/SACLA Annual Report FY2023*, 68–70.