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 ultra-trace-element analysis [1]. 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 FY2023, BL37XU operated smoothly and almost all users completed their user time as scheduled. In addition, the following project was undertaken. A new illumination and imaging optics was installed for full-field transmission micro-spectroscopy.

2. Upgraded Optics of Full-field Imaging-type X-ray Micro-spectroscopy

The imaging-type CT-XAFS required a very long measurement time of more than 10 hours in the past, but with the installation of the fiber optic plate (FOP)-type detector in FY2022, the exposure time was reduced to 150 ms, which is 1/6 of the conventional exposure time, and the measurement time was reduced to about 2 h [1]. Although the measurement time has reached a practical level, it is still too long for analyzing, for example, the reaction process of a lithium-ion battery [2], and further reduction of the measurement time is desired. Therefore, we optimized the optical elements to improve the throughput.

A new 6-layer beam-shaping condenser zone plate (BS-CZP) was introduced as an illumination optical element. Table 1 and Fig. 1 show the parameters and diffraction efficiency of BS-CZP, respectively. Compared with the conventional 3-layer BS-CZP [3], the 6-layer BS-CZP reduces the focusing area by a factor of 4, from about 100 μm × 100 μm to about 50 μm × 50 μm, but the number of segments is about four times larger, and the intensity is expected to increase. Since the real field of view that can be measured is about 50 μm in the optical configuration of BL37XU, even if the focusing diameter of the BS-CZP is reduced, the effect on the measurement area is considered to be negligible. The Fresnel zone plate (FZP) described below was also designed to match this thickness, because the use of 5 keV is being considered for the future, and the parameters were chosen to have diffraction efficiency peaks at lower energies.

Table 1. Parameters of BS-CZPs.

BS-CZP	Zone material	Zone thickness	Number of segments	Focusing size
3-layer	Ta	2000 nm	60	<100 μm
6-layer	Ta	1500 nm	258	<50 μm

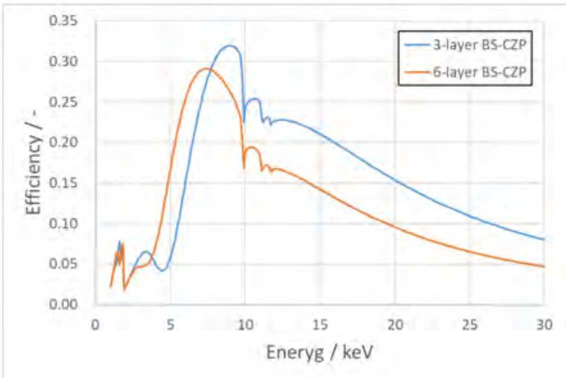

Fig. 1. Diffraction efficiency of BS-CZPs.

An apodization-type FZP [4] was installed as the imaging optics. Table 2 and Fig. 2 show the parameters and diffraction efficiency of FZPs, respectively. Conventional binary-type FZPs have a flat pattern thickness, whereas apodization-type FZPs have a Gaussian shape, which allows for a thicker pattern near the center of the element with wide pattern spacing. This will make it easier to increase the diffraction efficiency of the FZP, and image contrast is also expected to be improved by making it a Gaussian beam optical system. By installing a high-aspect-ratio FZP, the width of the outermost zone increased slightly from 50 nm to 80 nm. However, the change in the outermost bandwidth is not expected to have much effect on practical measurement, since a resolution of about 100 nm is sufficient for the current CT-XAFS measurement. The diffraction efficiency was improved by about 10% at 6.5–15 keV because the zone was thicker; thus the throughput was increased by a factor of 1.5 times.

Table 2. Parameters of FZPs.

FZP	Zone material	Zone tickness	Outermost zone width	Diameter
binary-type	Au	850 nm	50 nm	300 μm
apodization-type	Ta	1500 nm	80 nm	190 μm

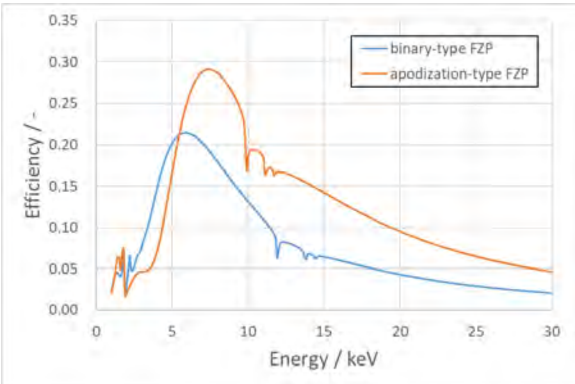

Fig. 2. Diffraction efficiency of FZPs.

The exposure time of 20 ms provides the same level of incident intensity as a conventional 150 ms exposure, and the throughput has been improved by a factor of about 7. Figure 3 shows μt images of X-ray chart patterns and line profiles of 100 nm L&S. Good image quality is obtained with an exposure time of 20 ms. It was also confirmed that the apodization-type Gaussian beam optics significantly improved the contrast of the μt image from 0.15 to 0.24. The chart pattern material is Au with a thickness of 550 nm, and the theoretical value of μt is 0.244 at 7.7 keV. The new optics shows that the correct absorption coefficient is obtained at the line part, although the 100 nm L&S is not completely isolated. Even with an exposure time of 20 ms, a clear image that was superior to that of conventional 150 ms exposure was obtained.

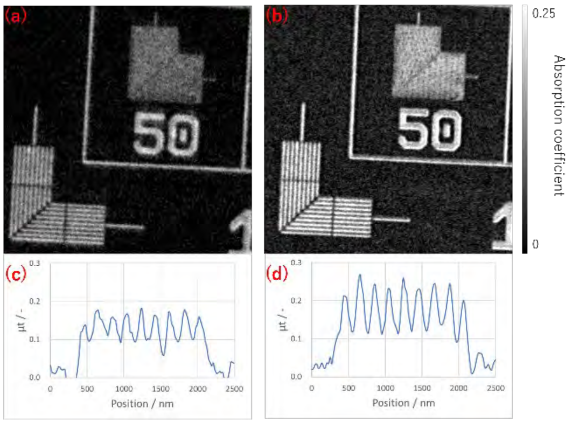

Fig. 3. X-ray chart patterns: (a), (c) conventional optics (exposure time: 150 ms), (b), (d) new optics (exposure time: 20 ms). (a) and (b) are μt images, (c) and (d) are line profiles of 100 nm L&S.

The throughput was improved by updating the optical elements, and the exposure time was successfully reduced to 1/7 of the conventional exposure time. Combined with the FOP-type detector installed in FY2022, the exposure time was reduced to 1/40, enabling imaging-type CT-XAFS measurements in less than one hour.

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