# BL46XU
# HAXPES II

## 1. Introduction

BL46XU is a beamline with an undulator light source. Until FY2021, two experimental apparatus, which were a multi-axis X-ray diffractometer in the first experimental hutch (EH1) and a hard X-ray photoelectron spectroscopy (HAXPES) [1,2] apparatus in the second experimental hutch (EH2), had been installed in this beamline. The multi-axis X-ray diffractometer was relocated to BL13XU at the end of FY2021, and BL46XU was reorganized as a beamline dedicated to HAXPES measurement techniques from FY2022. Therefore, the name of the beamline was changed to "HAXPES II". The layout of BL46XU in FY2022 is shown in Fig. 1. The main upgrading of the beamline, for example, improving the optical components, installing new apparatus (an ambient-pressure HAXPES apparatus), and relocating the apparatuses, will be carried out in the next FY.

The following instrumental improvements were carried out in FY2022: 1) an auto–sample exchange system was installed in the HAXPES apparatus and 2) the efficiency of improving the intensity of detected photoelectron signals by focusing the X-ray beam was studied using a bimorph mirror system.

## 2. Optics and performance

The light source is a standard in-vacuum undulator at SPring-8, and a liquid-nitrogen-cooled Si (111) DCM is adopted in the optics. The tunable energy range is 5.5–37.5 keV. To eliminate harmonics, two Rh-coated mirrors (70 cm in length, horizontal reflection direction) are placed in the most downstream part of the optics hutch. The mirrors can be bent for horizontal light focus. A Si (111) channel-cut monochromator is placed between the DCM and the mirrors to achieve incident X-rays with fine energy resolution.

## 3. Instrumental improvements

### 3.1 Auto–sample transfer system for high-throughput HAXPES

For the purpose of developing an automated measurement system of HAXPES at BL46XU, an auto–sample exchange system was introduced to the auto–sample transfer system of the HAXPES apparatus at the end of FY2022.

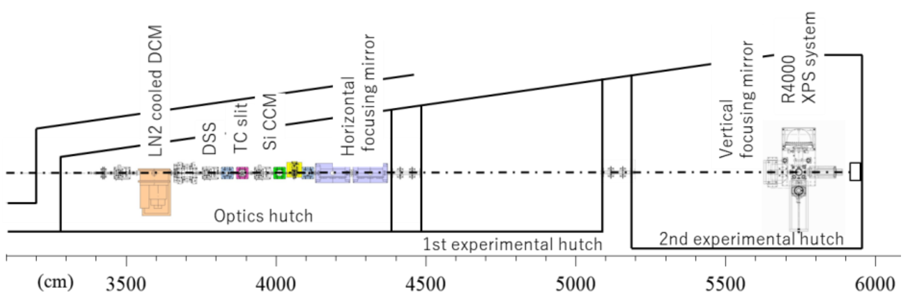


Fig. 1. Beamline layout of BL46XU.

Figure 2 shows a comprehensive overview of the auto–sample transfer system. The auto–sample exchange system consists of (1) an analysis chamber and (2) a lower load-lock chamber, which is shown in Fig. 2(c), equipped at the bottom of (1). The samples are transferred from (2) the lower load-lock chamber to (1) the analysis chamber using (3) the six-axis manipulator.

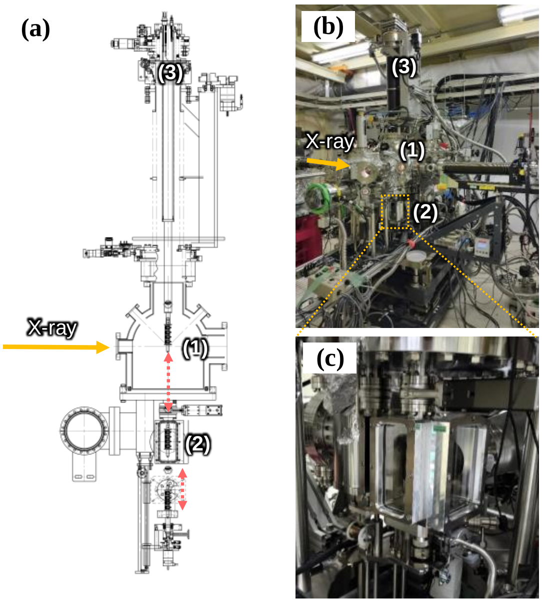


Fig. 2. (a) Schematic illustration and (b) photograph of auto–sample transfer system for HAXPES, including (1) analysis chamber, (2) lower load-lock chamber, and (3) six-axis manipulator. (c) Enlarged photograph of load-lock chamber.

The photographs in Fig. 3 show the details of this system. The sample holders with samples are set on the sample holder stocker shown in Fig. 3(b). As shown in the figure, four sample holders can be set on the sample holder stocker. The sample holders are common with those for BL09XU to optimize usability for users of both HAXPES beamlines. The sample holder stocker is set in the lower load-lock chamber. The lower load-lock chamber can accommodate four sample holder stockers. The six-axis manipulator approaches the sample holder stockers from above the lower load-lock chamber and transfers them into the analysis chamber without disrupting the vacuum, as shown in Fig. 3(a).

This system allows for the simultaneous introduction of a substantial number of samples into the vacuum, thereby enabling efficient sample transport and measurement. In FY2023, we are in the process of commissioning the automated sample transfer and measurement system. We aim to have the automated HAXPES measurement system fully operational by FY2024.

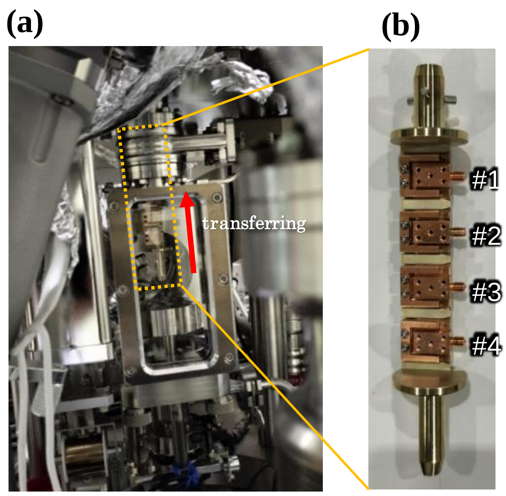


Fig. 3. (a) View of the six-axis manipulator transferring a sample holder stocker from the lower load-lock chamber to the analysis chamber. (b) A sample holder stoker.

### 3.2 Feasibility study on the efficiency of improving photoelectron signals by modifying

**the optics for focusing the X-ray**

In the upgrade of BL46XU scheduled for the next FY, the optical component for focusing the incident X-ray beam will be modified to improve the efficiency of detecting photoelectron signals. In this FY, we carried out a feasibility study on the efficiency of the modification mentioned above by using a bimorph mirror to focus the X-ray.

Under the standard optical condition of BL46XU of FY2022, the incident X-ray beam size at the sample position of the HAXPES apparatus is 0.03 mm in the vertical direction and 0.2 mm in the horizontal direction, which is wide in the horizontal direction. In the standard conditions for measurements in the HAXPES apparatus, the samples with plate shapes are set so that their sample surfaces are parallel to the vertical plane containing the direction of the incident X-ray beam. To irradiate sample surfaces with the X-ray, it is required to tilt the sample surfaces in the horizontal direction. That is, the X-ray-irradiated areas on the sample surfaces should be elongated horizontally. In the case that the beam size of the incident X-ray is wide in the horizontal direction, the efficiency of detecting photoelectrons should deteriorate because the elongated X-ray-irradiated areas on sample surfaces extend beyond the detection area of the objective lens of the HAXPES apparatus. Therefore, reducing the X-ray beam size in the horizontal direction is essential to improve the efficiency of measurement in the HAXPES apparatus.

For this feasibility study, we utilized a

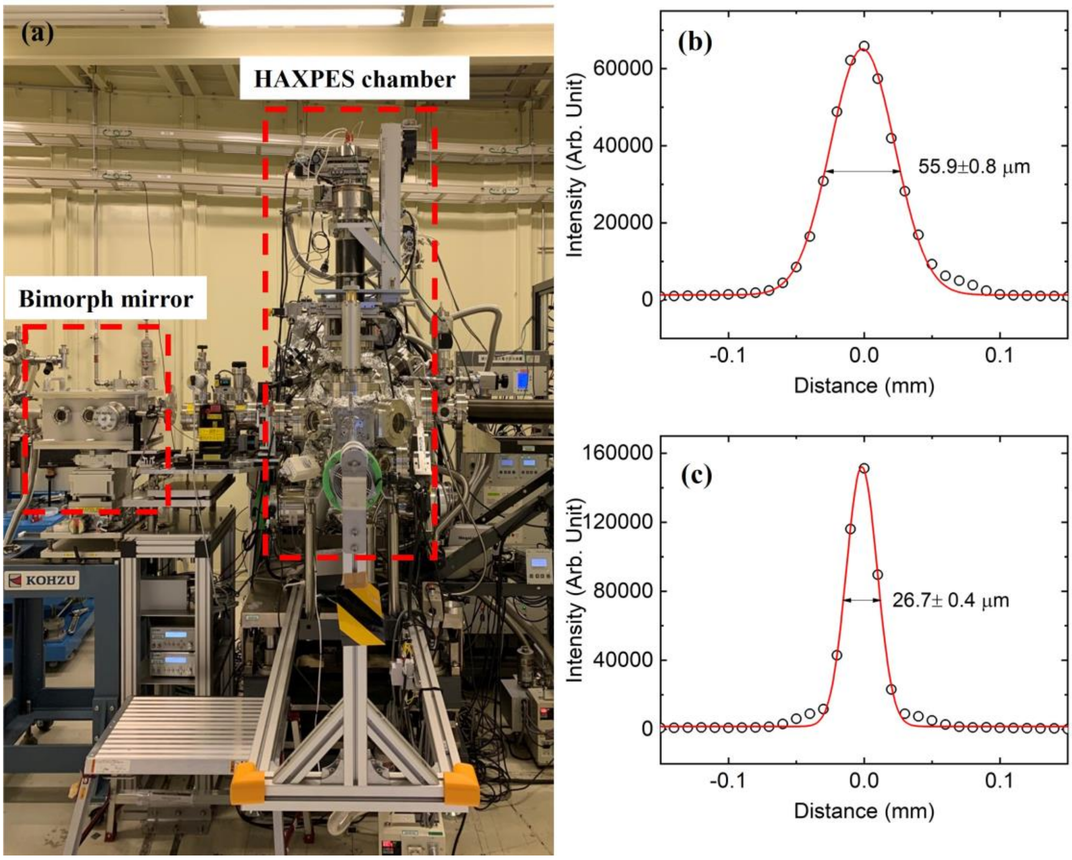


Fig. 4. (a) HAXPES system with the bimorph mirror in BL46XU beamline, and focused X-ray beam sizes at the sample position along the (b) horizontal and (c) vertical directions.

bimorph mirror system that had been used in the BL47XU beamline until FY2021. The bimorph mirror system was installed ahead of the analysis chamber of the HAXPES apparatus, as shown in Fig. 4(a). Figures 4(b) and 4(c) show the focused X-ray beam sizes at the sample position, along the horizontal and vertical directions, as observed by wire scan. The determined focused X-ray beam size, evaluated by fitting the profiles to the Gaussian equation, is 0.027 mm (vertical) × 0.056 mm (horizontal). These results are close to those obtained from the BL47XU beamline. The X-ray beam flux measured by the ion chamber just above the upstream side of the sample position before the installation of the bimorph mirror was 43,000 counts/s, while that measured after installing the bimorph mirror was reduced to 24,000 counts/s. Moreover, the flux density of the beam increased as a result of focusing the X-ray beam by means of the bimorph mirror system and was estimated to be 1.3 $\times 10^{14}$ photons/s/mm$^2$. Figure 5 shows the HAXPES spectra of Au films before and after the installation of the bimorph mirror, measured with a 2° incident angle. The integrated intensity of the Au 4f spectra ($4f_{7/2}$ and $4f_{5/2}$ peaks) increased significantly from 500,000 to 2,400,000 counts. This means a photoelectron signal intensity increase of over fourfold compared with that obtained using the previous optics system.

These results indicated that the throughput of HAXPES measurements in BL46XU can be improved by upgrading the optics for horizontally focusing the X-ray beam.

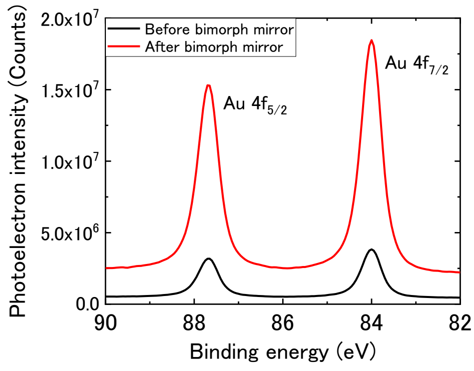


Fig. 5. HAXPES spectra of Au films before and after installation of bimorph mirror measured at Au 4f.

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**References:**

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[2] Yasuno, S. Oji, H. Koganezawa, T. & Watanabe, T. (2016). *AIP Conf. Proc*. **1741**, 030020.