BL46XU
Engineering Science Research III

1. Introduction

BL46XU is a beamline with an undulator light source dedicated to promoting the utilization of synchrotron radiation by the industry. A multi-axis X-ray diffractometer is installed in the first experimental hutch (EH1). In addition, the open space in EH1 can be utilized for various experiments of X-ray imaging, microfocus X-ray diffraction, and so on. The second experimental hutch (EH2) has a hard X-ray photoelectron spectroscopy (HAXPES) system [1, 2]. The following instrumental improvements were carried out in FY2021: 1) a measurement technique of X-ray diffraction and scattering was developed utilizing a hexapod and 2) an upper load-lock system was installed in the HAXPES system.

2. Optics and performance

The light source is a standard in-vacuum undulator at SPring-8, and the optics adopt a liquid-nitrogen-cooled Si (111) double-crystal monochromator. 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 monochromator and the mirrors to achieve incident X-rays with fine energy resolution. Figure 1 shows the beamline layout of BL46XU.

3. New equipment and developments

3.1 Sample stage using a hexapod in X-ray diffraction and scattering measurements

BL46XU has a HUBER multi-axis X-ray diffractometer installed in EH1. This apparatus has been used by researchers from the industry and industry–academia collaboration sectors in various scientific fields. Because the purposes of their experiments were varied, the sample stage of this multi-axis X-ray diffractometer should be relatively large (approximately 20 cm × 20 cm × 20 cm) and have a high load capacity (approximately 10 kg). In recent years, the needs of users have diversified, and there is an increasing demand for specialty-stage-dedicated experiments using large and heavy instruments for the control of sample environments.

To fulfill this requirement, a large hexapod was introduced as a sample stage for X-ray diffraction and scattering measurements that require

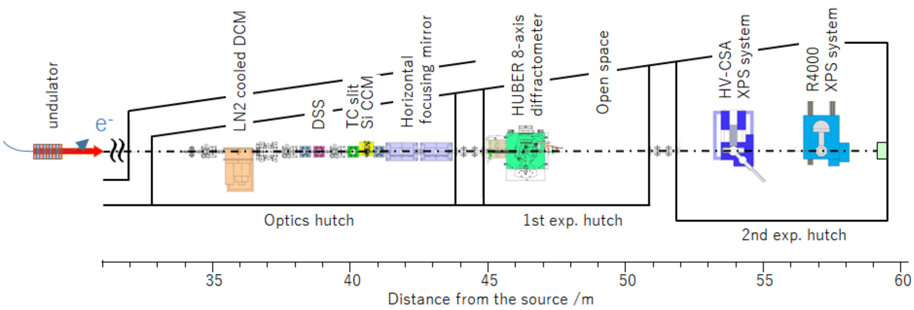

Fig. 1. Beamline layout of BL46XU.

a wide space and a high load capacity. The hexapod consists of a six-axis actuator system of X, Y, Z, RX, RY, and RZ, providing flexible sample position control with high precision and a high load capacity of 250 kg. It also has the advantage that users can select the rotation center of the stage where the X-ray beam passes through. The hexapod can be utilized for X-ray diffraction and scattering measurements in combination with a two-dimensional detector instead of multi-axis X-ray diffractometers, although the hexapod has limited motion and angle ranges. After the hexapod was delivered at the end of 2020, we made many offline adjustments, and X-ray diffraction measurements were started in 2021. Here, we showed the use of the hexapod in X-ray diffraction measurements as follows: (1) experimental setup used for high-throughput 2D-GIXD measurements (Fig. 2(a)), (2) *in situ* XRD measurement setup equipped with a compact vacuum deposition chamber for organic thin films (Fig. 2(b)), and (3) *in situ* XRD measurement setup with a high-vacuum X-ray oven for sample heating (Fig. 2(c)). In 2022, this hexapod was relocated to BL13XU EH2 and operated in combination with a two-dimensional detector handled by a robot arm.

3.2 Upper load-lock system for high-throughput HAXPES equipment

Because of the plan of the automated sample exchange system of HAXPES at BL46XU in FY2023, it is necessary to improve the mechanical controllability, accuracy, and reliability of the sample loading system. Because it was difficult for the conventional horizontal load-lock system currently used to meet the requirement mentioned

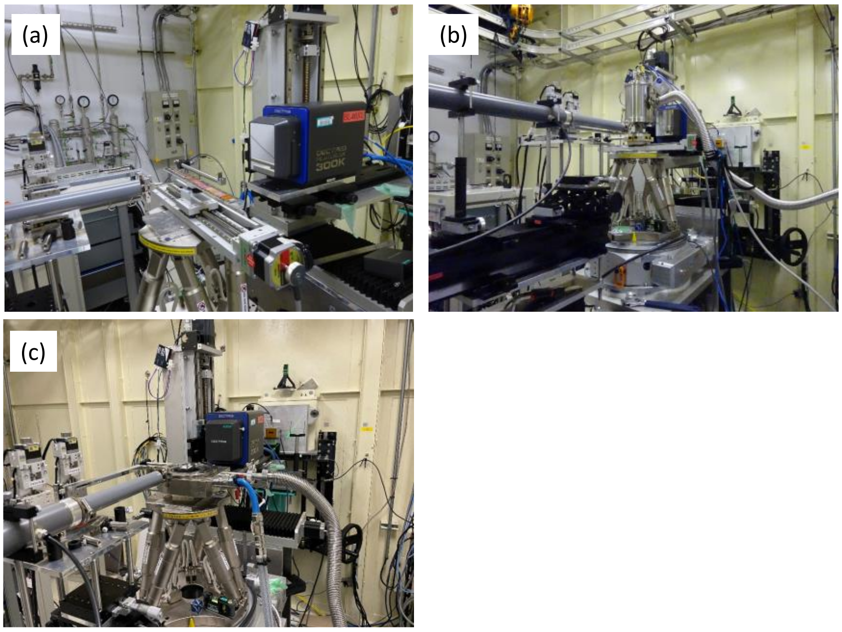

Fig. 2. (a) Experimental setup used for the high-throughput 2D-GIXD measurements, (b) *in situ* XRD measurement setup equipped with a compact vacuum deposition chamber and (c) *in situ* XRD measurement setup with a high-vacuum X-ray oven for sample heating.

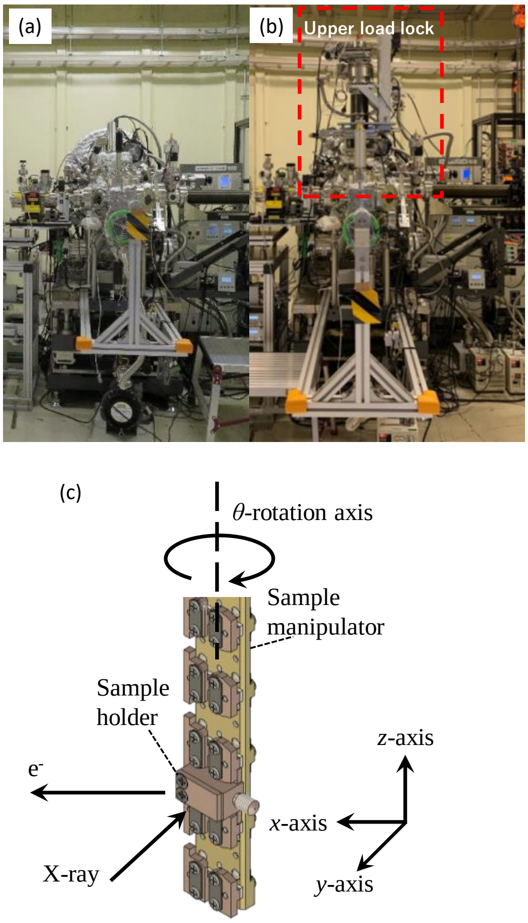

Fig. 3. (a) Conventional chamber system, (b) new HAXPES chamber system equipped with the upper load-lock system, and (c) measurement geometry.

above, we introduced an upper load-lock system into the HAXPES chamber at the end of FY2021. Figure 3(b) shows an overview of the HAXPES chamber system equipped with the upper load-lock system in comparison with that without the load-lock system (Fig. 3(a)). We have successfully installed the upper load-lock system, as shown by the red dashed square in Fig. 3(b). This upper load-lock system provided good mechanical controllability for sample manipulation by its mechanism shown in Fig. 3(c). The sample positions are controlled by motorized stages of the *x*-axis, *y*-axis, *z*-axis, and *θ*-rotation axis. These motorized stages are controlled by the SES program, which is the HAXPES operating program made by Scienta Omicron. Automatic measurement can be performed by registering multiple measurement positions on samples by the SES program. In the conventional horizontal load-lock system, incidence angles of X-rays to sample surfaces were fixed by the tapered shape of a conventional hexagonal sample holder. This new system enabled the fine tuning of the incidence angle of X-rays to the sample surface by the *θ* -rotation axis. Moreover, in the conventional horizontal load-lock system, the sample manipulation rod was equipped horizontally, so that the repeatability for the control of sample positions was affected by the deflection of the rod, which originated from gravity. In this new system, because its sample manipulation rod equipped vertically is not affected by gravity, the repeatability for the control of sample positions can be improved.

Figure 4(a) shows the sample mount system on the manipulator of the upper load-lock system. Sample holders #1–#4 are for normal HAXPES measurements. Sample holder #5 made of Mo materials is for the *in situ* measurement under heating. This sample holder is equipped with a heater and can heat samples up to 600 °C. The sample holder shown in Fig. 4(b) is for a wide sample. The size of the sample holder is 15 mm × 20 mm. This wide sample holder enables us to apply the upper load-lock system to various sample configurations for HAXPES measurement more widely than the conventional horizontal load-lock system. A newly equipped upper load-lock system is opened to beamline users from the 2022A1 period.

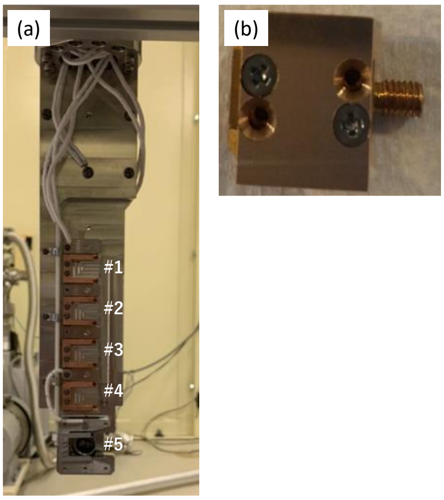

Fig. 4. (a) Sample mount system on the manipulator and (b) sample holder for a wide sample.

Yasuno Satoshi, Koganezawa Tomoyuki, Kajiwara Kentaro, Seo Okkyun and Kumara Loku Singgappulige Rosantha

Industrial Application and Partnership Division, JASRI

References:

[1] Oji, H. Cui, Y.-T. Son, J.-Y. Matsumoto, T. Koganezawa, T. & Yasuno, S. (2015). *J. Surf. Anal*. **21**, 121–129.

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