BL09XU
HAXPES I

1. Introduction

BL09XU is an X-ray beamline with a 32-mm-period standard linear undulator. In FY2021, BL09XU took on activities of hard X-ray photoelectron spectroscopy (HAXPES) previously carried out at BL47XU and was reorganized as a beamline dedicated to HAXPES [1]. In this upgrade, all optics except for a liquid-nitrogen-cooled double-crystal monochromator (DCM) with Si 111 reflection were upgraded to state-of-the-art ones specialized for HAXPES experiments for conducting more advanced HAXPES applications, such as resonant HAXPES [2] and three-dimensional spatial-resolved chemical bonding analysis [3]. For example, two pairs of double channel-cut crystal monochromators (DCCMs) with Si 220 and Si 311 reflections were introduced to realize HAXPES analyses with a total energy resolution below 300 meV at any incident photon energy of 4.9–12 keV while satisfying a fixed-exit condition. X-ray phase retarders with two diamond crystals enable us to control the polarization state with a high polarization degree above 90% in a wide energy range of 5.9–9.5 keV. In FY2023, (1) a non-atmospheric-exposure sample conditioning and transport system and (2) web applications for HAXPES and optical system control were introduced.

2. Non–atmospheric-exposure sample conditioning/transfer system

In studies on the development of battery materials such as a fuel cell and a lithium-ion battery, it is necessary to prepare, transport, and measure samples without atmospheric exposure to prevent sample degradation. Since the HAXPES system is under the vacuum condition, sample preparation and transportation of the prepared samples to the HAXPES system must be carried out in a non–air-exposed manner. Previously, sample preparation, such as mounting the sample on a sample holder for HAXPES measurements, was carried out using a glovebox at another beamline and the samples were introduced into the HAXPES instrument using a transfer vessel, as reported in Annual Report FY2016 [4]. On the other hand, the number of measurements for samples without air exposure is increasing year by year, partly due to the demands for the more efficient use of energy for the achievement of a carbon neutral society. Furthermore, these experiments often involve the measurement of a large number of samples with different preparation conditions. The special sample holders used in previous transfer vessels had a small mounting area and only two samples could be mounted and up to four sample holders could be stored in the vessel, so only eight samples could be introduced into the HAXPES instrument at once. To meet the growing demand for the measurement of nonexposed samples, [i] regular operation of the glovebox was started and [ii] a compact transfer vessel capable of storing a large number of samples was developed.

A gas-flow glovebox, GBJF080, manufactured by Glovebox Japan Inc. was installed in FY2021, and regular operation was newly started with the introduction of an inert gas recycling purification system, GBJPWS3, manufactured by the same company. This instrument is connected to the glove box and removes oxygen and moisture generated inside the glove box using a catalyst and adsorbent and performs recycling and purification operations. It is capable of removing both oxygen and moisture of less than 1 ppm, making it suitable for many battery materials.

Furthermore, a compact transfer vessel was developed, which can be equipped with five sample holders that can accommodate the large number of samples used in normal HAXPES measurements. For samples of the usual size of 3 mm × 5 mm, about 15 samples can be mounted in each sample holder, making it possible to introduce a total of 75 samples. The use of this vessel enables efficient measurements, because the sample transfer to the measuring chamber can be completed just once. In addition, the vessel was made compact for introduction into a typical glove box with a pass box of about 250 mm on each side. The linear motion of the previous vessel was removed, and the removable sample bank stored in the vessel is gripped by a separately prepared transfer rod when transferring samples. This makes the new vessel simple, merely a long vacuum tube with a gate valve and flanges, and it is compact in size with a footprint of 205 mm square.

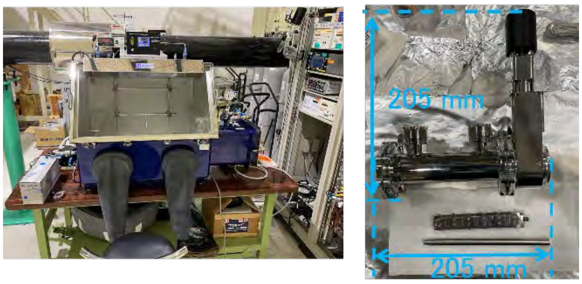

Fig. 1. Glove box system installed in BL09XU and the developed compact transfer vessel.

3. Web applications for HAXPES and optics control

For the beamline upgrade in FY2021, most of the optics instruments and the positioning mechanism of the HAXPES instrument were switched to be controlled by BL-774 [5], which was built on a Python-based platform. On the other hand, user interface programs to control these instruments were created in LabVIEW. However, such software could not fully utilize BL-774's function of coordination between devices. Therefore, new equipment control software that can make full use of the BL-774's functions was developed.

The developed software is entirely based on Python. The main functions, such as coordinated operation between multiple devices, are built within the framework of BL-774, and the excellent task management by BL-774 can be utilized. The functions are called from a web application based on the Python web framework FastAPI. Web applications can distribute resources such as memory in the computer used for displaying the programs, greatly reducing the load on the server that controls the instrument. Furthermore, by using the browser's tab function, multiple web applications can be switched in a single browser. This has the advantage that, in the future, the display area will be compact even if many software applications are used when conducting remote experiments from outside of SPring-8. Examples of developed programs are as follows.

Stage Scan: Single-axis scanning of any drive axis and monitoring X-ray or photoelectron intensity.

Set Sample Position: Registering and moving the sample position.

Control Optics: Adjusting the undulator gap and the rotation angles of DCM and DCCMs corresponding to the incident X-ray energy.

Attenuator Control: Controlling the thickness of the Al and Si attenuators, which attenuate the X-ray intensity.

2D Mapping: Two-dimensional scanning of the sample position within the sample surface or in a plane perpendicular to the X-ray axis and monitoring X-ray or photoelectron intensity.

Sequence Measurement: Sequential measurements in combination with HAXPES measurement and optical instruments.

By utilizing the above programs from the "Sequence Measurement" program, it is possible to perform resonant HAXPES measurements and automatic measurements that couple control of the sample position and attenuator conditions with HAXPES measurements. These programs are applied to various experiments at SPring-8 to improve usability for users.

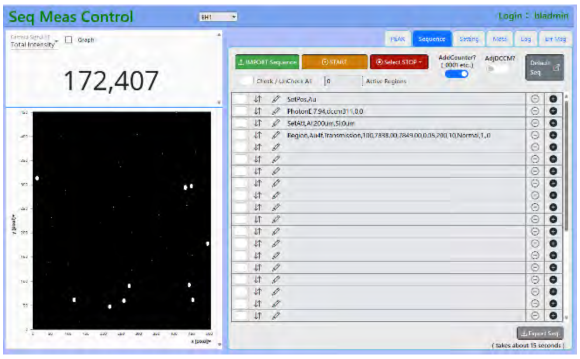

Fig. 2. Captured image of the web application for HAXPES and optics control.

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References:

[1] Yasui, A. Takagi, Y. Osaka, T. Senba, Y. Yamazaki, H. Koyama, T. Yumoto, H. Ohashi, H. Motomura, K. Nakajima, K. Sugahara, M. Kawamura, N. Tamasaku, K. Tamenori, Y. & Yabashi, M. (2023). *J. Synchrotron Rad.* **30**, 1013.

[2] For example, Maeda, K. Sato, H. Akedo, Y. Kawabata, T. Abe, K. Shimokasa, R. Yasui, A. Mizumaki, M. Kawamura, N. Ikenaga, E. Tsutsui, S. Matsumoto, K. Hiraoka, K. & Mimura, K. (2020). *JPS Conf. Proc.* **30**, 011137.

[3] For example, Oshime, N. Kano, J. Ikenaga, E. Yasui, S. Hamasaki, Y. Yasuhara, S. Hinokuma, S. Ikeda, N. Janolin, P. E. Kiat, J. M. Itoh, M. Yokoya, T. Fujii, T. Yasui, A. & Osawa, H. (2020). *Sci. Rep.* **10,** 10702.

[4] Yasui, A. & Ikenaga, E. (2017). *SPring-8/ SACLA Annual Report FY2016*, 88.

[5] Nakajima, K. Motomura, K. Hiraki, T. N. Nakada, K. Sugimoto, T. Watanabe, K. Osaka, T. Yamazaki, H. Ohashi, H. Joti, Y. Hatsui, T. & Yabashi, M. (2022). *J. Phys.: Conf. Ser.* **2380,** 012101.