BL39XU
X-ray Absorption and Emission Spectroscopy

1. Introduction

BL39XU is a hard X-ray beamline primarily dedicated to investigating the electronic states of a wide variety of materials, including those relevant to magnetism, strongly correlated electron systems, catalysts, and environmental samples. The principal techniques employed are X-ray absorption spectroscopy (XAS), X-ray magnetic circular dichroism (XMCD), and X-ray emission spectroscopy (XES). The upgrade of the BL39XU beamline toward SPring-8-II was initiated in July 2023 [1], and in parallel with the renewal of the optics, these methods have been making gradual progress.

The commissioning following the upgrade was conducted up to July 2024, primarily involving the performance evaluation of the optics and assessments at the experimental stations. In this report, the recent upgrade and commissioning results of BL39XU are presented, focusing on the following: (1) beamline optics, featuring a fixed-exit optical system with advanced polarization control, and (2) experimental hutches (EHs), including enhanced X-ray spectroscopy under multiple extreme conditions at EH1, a newly constructed station for X-ray emission spectroscopy with improved resolution at EH2, and advanced nanospectroscopy with reduced higher harmonics and upgraded detectors at EH3.

2. Beamline optics in optics hutch

The higher-harmonics cut mirror (HCM), located downstream of the monochromator, serves not only to remove high-order reflections from the monochromator but also to maintain the optical axis by employing triple reflection. As a result of its adjustment, the vertical beam fluctuation has been suppressed to within 55 μm for changes in glancing angle ranging from 2 to 8 mrad. Consequently, the adjustment of the focusing optics installed at the experimental station is facilitated, since the X-ray axis remains unchanged when the glancing angle of the HCM is varied.

A double X-ray phase retarder (DXPR) system is installed downstream of the HCM to control X-ray polarization. Before the upgrade, polarization control relied on a single-crystal diamond and was limited to circular, horizontal, and vertical linear polarizations. The upgrade introduced a two-stage phase retarder with a rotation axis centered on the X-ray axis in the second stage, enabling improved vertical linear polarization and the rotation of the linear polarization plane. In addition, as shown in Fig. 1, each stage can accommodate three diamond

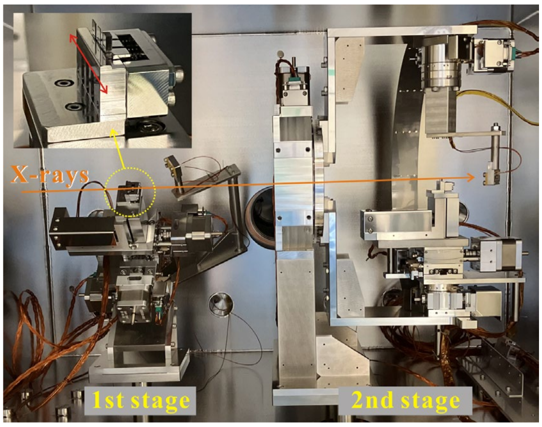

Fig. 1. Photograph of the DXPR system. The 1st stage is fixed at a 45° tilt relative to the beam axis, while the 2nd stage can rotate around the beam axis within a range from −10 to 100°.

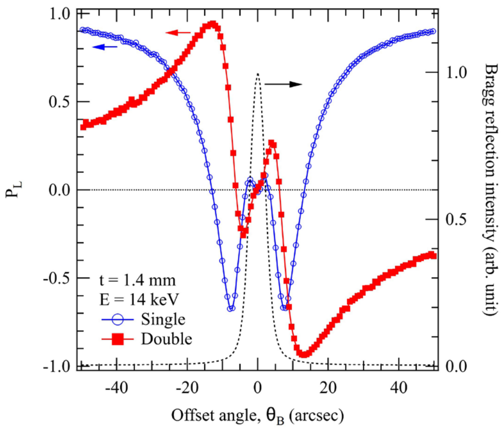

Fig. 2. Degree of linear polarization at 14 keV produced by a single and a double XPR system using diamond crystals with a thickness of 1.4 mm.

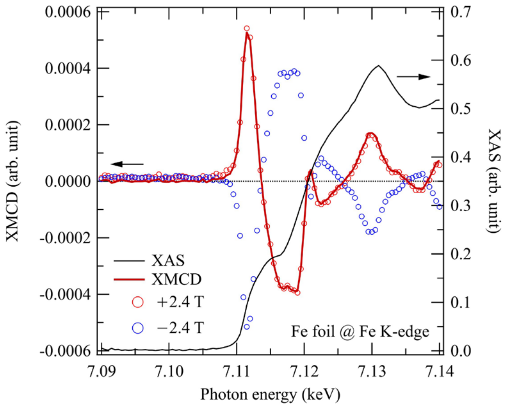

Fig. 3. XMCD spectra at the Fe *K*-edge in a 4-μm-thick Fe foil. The spectra were obtained under a magnetic field of 2.4 T at room temperature.

crystals that can be switched as needed. With up to six crystals of different thicknesses installed, appropriate crystals can be selected over a wide energy range without breaking the vacuum, thereby providing highly polarized circular and vertical polarizations.

Figure 2 shows the degree of vertical polarization at 14 keV obtained using two phase retarders with a thickness of 1.4 mm, compared with that obtained using a single retarder. The degree of linear polarization was estimated using a simple polarization monitor with Kapton scattering [2]. With two phase retarders, the degree of vertical polarization is observed to exceed 0.9, whereas with a single retarder it is around 0.7. At present, the crystals available for use as double phase retarders are limited to those with thicknesses of 0.31 and 1.4 mm, but we plan to introduce additional crystals of different thicknesses in the future.

3. Experimental station for X-ray spectroscopy under multiple extreme conditions (EH1)

EH1, located approximately 48 m from the undulator source, is primarily used for X-ray absorption spectroscopy under various extreme conditions. At BL39XU, variable X-ray polarization can be achieved using the DXPR system. After the upgrade, new Kirkpatrick–Baez (KB) mirrors were installed for high-pressure experiments, allowing the beam to be focused to 10 μm horizontally and 1 μm vertically.

During the commissioning following the upgrade, the performance of XMCD measurements was verified. Figure 3 shows the XMCD spectrum at the Fe *K*-edge in Fe foil, obtained using the circular polarization modulation method [3]. The results indicate that the data quality after the upgrade is comparable to that obtained before the upgrade. Furthermore, while the maximum applied magnetic field at room temperature was previously limited to 2.0 T, the upgrade has extended this to 3.5 T (1.5 T at low temperatures). The superconducting magnet, providing fields up to 7 T at temperatures as low as 2 K, also remains available. This enhancement is expected to further advance X-ray spectroscopy under multiple extreme conditions.

4. Experimental station for X-ray emission spectroscopy (EH2)

EH2, located approximately 66 m from the undulator source, was newly constructed as part of the upgrade for X-ray emission spectroscopy (XES) and high-energy-resolution fluorescence-detected (HERFD) XAS measurements. The XES spectrometer [4], previously installed in EH1 before the upgrade, was relocated to this new hutch. In addition, a Wolter mirror [5] with a focusing size of 15 μm horizontally and 1 μm vertically was installed, enabling micro-area XES/HERFD-XAS measurements and two-dimensional (2D) imaging.

The performance of the XES spectrometer was evaluated before and after the upgrade. Figure 4 shows an example of the HERFD-XAS spectrum at the Ir *L_3*-edge of IrO_2. Overall, the performance has been maintained; however, the beam reduction at the emission point achieved by the dedicated focusing optics has improved the overall energy resolution of the system by approximately 10%. At present, this system is equipped only for temperature-dependent measurements from room temperature down to low temperatures (~3 K), or for experiments at high pressures. In the future, operando/*in situ* measurement and high-temperature environments will be developed to extend its applications and promote wider use.

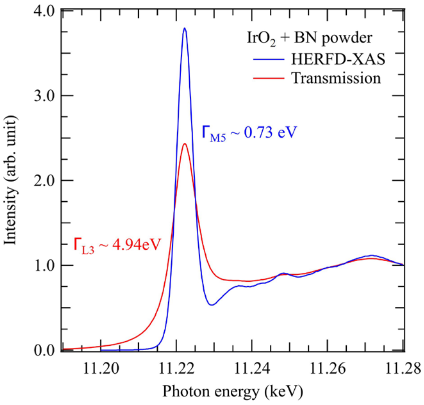

Fig. 4. HERFD-XAS spectrum at the Ir L_3-edge of IrO_2 diluted with BN powder, obtained using five Ge (800) analyzer crystals. For comparison, a conventional XAS spectrum obtained in transmission mode is also shown.

5. Experimental station for X-ray nanospectroscopy (EH3)

EH3, located approximately 76 m from the undulator source, has been used for XAS/XMCD nanospectroscopy since FY2011 [6]. No changes were made to the focusing optics or XAS/XMCD measurement instruments after the upgrade. However, the installation of the HCM has reduced higher-harmonic components in the low-energy region below 6 keV. As a result, high-precision nano-XAS/XMCD spectroscopy and imaging for elements such as Ti, Ce, and Nd, which are used in practical devices and catalysts, are expected to be realized.

Enhancements in the sensitivity of nano-XAS/XMCD spectroscopy and their imaging are also in progress. A seven-element SDD and a DSP have been newly introduced, and the system is being reconstructed to incorporate these devices. In addition, an expansion of the sample environments is under consideration. In the future, temperature-dependent measurements are expected to be realized.

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