BL39XU
Magnetic Materials

1. Introduction

BL39XU is a hard X-ray beamline mainly dedicated to the study of magnetic materials and strongly correlated electron systems. Techniques include X-ray absorption spectroscopy (XAS), X-ray magnetic circular dichroism (XMCD), and X-ray emission spectroscopy (XES). Recently, developments in these methods have been gradually progressing toward upgrading the beamlines in anticipation of SPring-8-II. The upgrade of BL39XU will start in July 2023.

In FY2022, the following developments were mainly undertaken: (1) the extension of the X-ray emission spectrometer to the high-energy region beyond 16 keV, and (2) the development of the 7-elements Si drift detector (SDD) for high-efficiency X-ray fluorescence detection.

2. Experimental station for X-ray spectroscopy under multiple extreme conditions

Experimental hutch 1 (EH1), located about 48 m away from the undulator source, is mainly used for the XMCD measurements under extreme conditions and for the XES measurements. In FY2022, the X-ray emission spectrometer was studied in order to extend available emission energy to the high-energy region beyond 16 keV, which continued from the last fiscal year.

In FY2021, the extension to the emission X-ray energy of 20.24 keV (around rhodium (Rh)-$K\alpha_1$ emission) was successfully accomplished using a combination of Si 12 12 0 reflections with a CdTe-element detector [1]. This year, XES and high-energy-resolution-fluorescence-detected XAS (HERFD-XAS) measurements for indium (In)-$K\alpha_1$ emission (~24.21 keV) were performed in order to extend the measurement to higher energy regions beyond the Rh-$K\alpha_1$ emission energy.

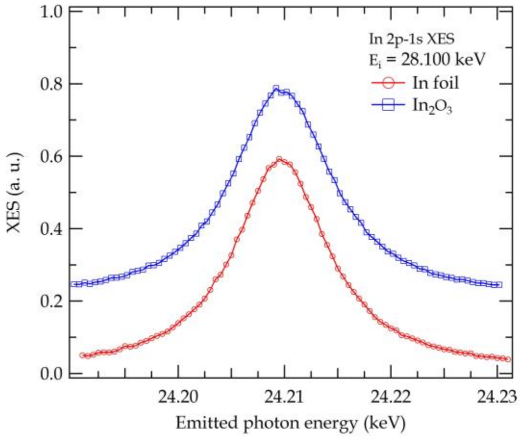

Fig. 1. XES spectra of In-$K\alpha_1$ emission in In foil and In_2O_3 powder. The incident photon energy was 28.10 keV.

In-$K\alpha_1$ XES spectra were obtained by a combination of five InSb 15 15 5 analyzer crystals with a PiXirad-2 detector with a CdTe element of 650 μm thickness. Figure 1 shows the XES spectra of In-$K\alpha_1$ in In foil and In_2O_3 powder. The spectra shown in Fig. 1 are obtained with an accumulation time of 8–10 s per energy point; therefore, it is estimated that a similar accumulation time is required to obtain a high signal-to-noise ratio for the data quality. The energy resolution is about 1.9 eV at 24.2 keV, estimated using the elastic scattering X-rays.

HERFD-XAS can also be measured using the XES system. Figure 2 shows the HERFD-XAS spectra at the In *K*-edge in In foil and In_2O_3 powder.

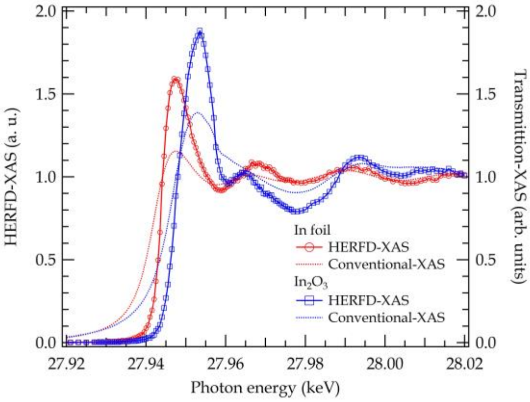

Fig. 2. HERFD-XAS spectra at In *K*-edge in In foil and In$_2$O$_3$ powder. The dotted lines denote the XAS spectrum in the conventional transmission method.

For comparison, conventional-XAS spectra are shown in the figure. The spectra are obtained with an accumulation time of 30–40 s per energy point. The structures of the HERFD-XAS spectra are clearly observed because of the lifetime-broadening-suppressed effect as well as the result at the Rh *K*-edge [1].

A PILAUS detector with a Si element was used and its sensitivity and efficiency were compared with those of the PiXirad-2 detector. However, emission X-rays were not detected by the PILATUS detector because of the extremely low sensitivity. In conclusion, XES/HERFD-XAS measurements above 20 keV require a detector with CdTe elements in terms of sensitivity.

3. Experimental station for X-ray nanospectroscopy

Experimental hutch 2 (EH2), located about 76 m away from the undulator source, is mainly used in XAS/XMCD nanospectroscopy. Since FY2011, a scanning hard X-ray nanoprobe has been developed for XAS/XMCD microscopy in EH2 [2]. Kirkpatrick–Baez (KB) mirror optics can be used to generate a focused X-ray beam with a typical spot size of 100 nm × 100 nm in an energy range of 5–16 keV.

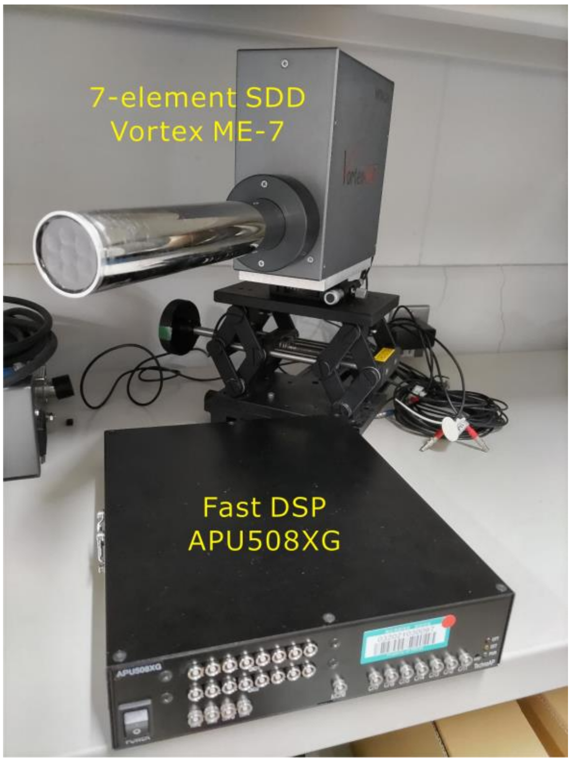

Fig. 3. Photograph of high-efficiency X-ray fluorescence system, 7-element SDD, and fast DSP installed at BL39XU.

Recently, X-ray spectroscopic imaging methods have been widely used, and a high-efficiency imaging method using X-ray fluorescence (XRF) and XAS are also important for the effective use of nanofocused X-ray beams.

To improve the efficiency of X-ray fluorescence, a new 7-element SDD (Voltex ME-7; Hitachi High-Tech Science America, Inc.) was installed as shown in Fig. 3. In order to suppress count loss, a fast digital signal processor (DSP, APU508XG; TechnoAP Co., Ltd.) was also installed. The total active area for fluorescent X-rays is increased from 260 to 350 mm^2 (×1.35), compared with a previous 4-element SDD (SiriusSD 4CH-SDD; RaySpec Ltd.). The maximum practical count rate achieves 1 Mcps/element or higher. The efficiency is equal to or much better than those of previous systems [4-element SDD and DSP (FalconX4; XIA LLC) combination]. Furthermore, the CUBE preamplifier maintains a high energy resolution at a high counting rate of 1 Mcps/element or higher, thus improving the energy resolution from 240 eV of the 4-element SDD to 190 eV. Therefore, the fluorescence peaks are well resolved, allowing for highly sensitive XRF analysis. In addition, the new DSP is equipped with a high-speed communication protocol of 10 Gbit/s, which enables batch acquisition of fluorescence spectra with more than 800 frames/s/CH.

The commonality of systems is important from the perspectives of risk management and efficient use of beamline resources. The SDD system is fully compatible with the system at BL37XU installed in FY2021 [2]. The objectives of standardization are (1) to allow users to use beamlines without the need to be aware of their differences, and (2) to allow immediate alternative use in the event of equipment failure. In the future, more convenient systems will be built to standardize control software.

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References:

[1] Kawamura, N. Higashi, K. & Osawa, H. (2022). *SPring-8/SACLA Annual Report FY2021*, 68–70.
[2] Nitta, K. Sekizawa, O. & Suga, H. (2022). *SPring-8/SACLA Annual Report FY2021*, 65–67.