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). Recent developments have focused on X-ray spectroscopy measurements under multiple extreme and complex conditions and scanning XAS/XMCD imaging using a nanofocused X-ray beam. These techniques are available for user experiments, and further developments are ongoing.

In FY2021, the following developments were mainly undertaken: (1) the extension of the X-ray emission spectrometer to the high-energy region beyond 16 keV was studied at experimental hutch 1, and (2) a visible light monitoring system was installed to determine the target position of the sample at experimental hutch 2.

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

Experimental hutch 1, located about 48 m away from the undulator source, is mainly used in the XMCD measurements under extreme conditions and in the XES measurements. In FY2021, the X-ray emission spectrometer was studied in order to extend available emission energy to the high-energy region beyond 16 keV.

The XES spectrometer installed at BL39XU is a so-called Rowland-type spectrometer in which the sample, spherically bent analyzer crystals, and X-ray detector are mounted on a Rowland circle. Therefore, the emitted X-rays from the sample are monochromatized and focused in the Bragg reflection geometry. For high-energy X-rays, the efficiency of the X-ray reflection is generally higher in the Laue geometry than in the Bragg geometry. However, it is difficult to efficiently collect (focus) X-rays emitted radially from the sample in the Laue geometry. To check for the possibility of XES measurements in the conventional Rowland-type geometry, the XES spectrum of Rh $K\alpha_1$ emission was observed by using a combination of five Si 12 12 0 analyzer crystals with a PILATUS 100K (Si elements of 320 μm thickness) detector. Figure 1 shows the XES spectrum of Rh $K\alpha_1$ in a $CeRh_3$ polycrystalline sample. The accumulation time is 10 s per energy point. The energy resolution is about 2.5 eV at 20.23 keV estimated using the elastic scattering X-rays. The XES intensity is relatively lower than the results for the $K\alpha_1$ emission of 3*d* transition metals (emission energy is located at 5–

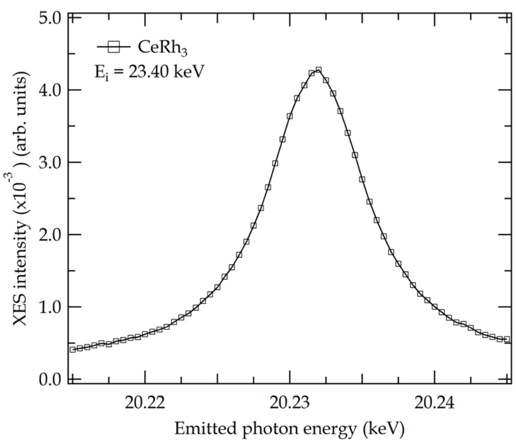

Fig. 1. XES spectrum of Rh $K\alpha_1$ emission in a $CeRh_3$ polycrystalline sample. The incident photon energy is 23.40 keV.

9 keV). The reasons are clearly the low X-ray reflectivity from the analyzer crystals and the low sensitivity of the Si detector elements. However, it is confirmed that the XES spectra can be measured, although the measurement efficiency is low.

High-energy resolution fluorescence detected (HERFD) X-ray absorption spectrum (XAS) can also be measured using the conventional XES system. Figure 2 shows the HERFD-XAS spectrum at the Rh *K*-edge in $CeRh_3$. The fluorescence XAS spectrum measured simultaneously is also shown in the figure for comparison. The structures in the HERFD-XAS spectrum are clearly observed owing to the lifetime-broadening-suppressed effect [1]. Therefore, it was confirmed that the conventional HERFD-XAS method can be used in the high-energy region above 20 keV, except that the efficiency of the emission intensity is low.

The efficiency can be improved by replacing the detector. The result of replacing the PILATUS detector with the PiXirad-2 detector (CdTe elements

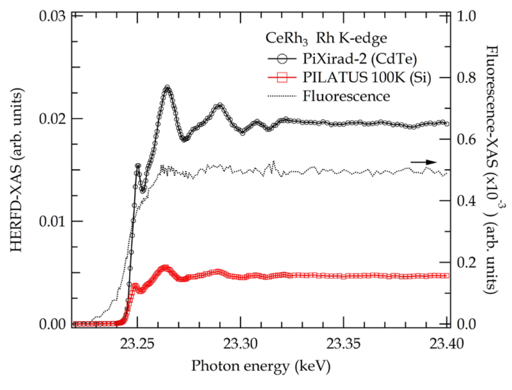

Fig. 2. HERFD-XAS spectra at Rh *K*-edge in $CeRh_3$. Red and black marks represent the data obtained by the PILATUS 100K and PiXirad-2 detectors, respectively. The black dotted line denotes the XAS spectrum in the conventional fluorescence method obtained by the silicon drift detector.

of 650 μm thickness) is also shown in Fig. 2. In general, the efficiency of the CdTe element at 20 keV is 100%, whereas that of the Si element of the PILATUS detector at 20 keV is only 28%. Therefore, it is expected that the efficiency of the PiXirad-2 detector will be by 3.6 times higher than that of the PILATUS detector. In fact, the efficiency of the PiXirad-2 detector is experimentally estimated to be 4.2 times higher than that of the PILATUS detector from the results shown in Fig. 2. Consequently, the XES measurements above 20 keV are possible on a practical level using the PiXirad-2 detector with CdTe elements.

3. Experimental station for X-ray nanospectroscopy

Experimental hutch 2, 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 hutch 2 [2]. Kirkpatrick and 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.

Recently, an X-ray spectroscopic imaging method [3] has been actively used in addition to the X-ray nanoprobe XMCD. In particular, a highly efficient imaging method using X-ray fluorescence (XRF) and XAS is also conceivable with the effective use of a nanofocusing X-ray beam. However, the positioning of a small sample or target, such as a device element, the microstructure of materials, and the active point of a catalyst, is desired. To more easily locate the target position for X-ray irradiation, a visible light monitoring system (microscope) was installed at experimental hutch 2.

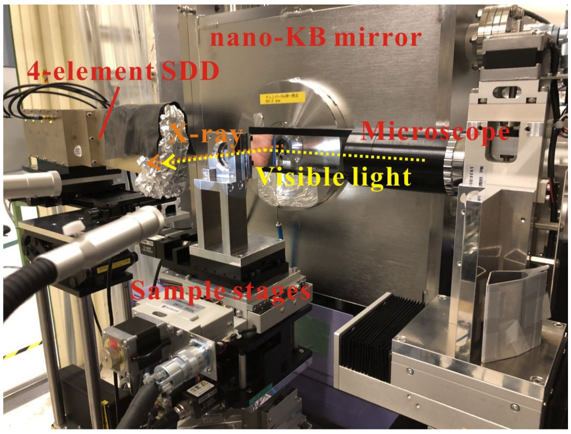

Fig. 3. Visible light monitoring system installed at experimental hutch 2.

Figure 3 shows a photograph of the system. A visible mirror tilted at an angle of 45 degrees with a hole that allows focused X-rays to pass through facilitates X-ray irradiation to the target position and also allows the sample to be constantly monitored. The monitoring system provides a minimum spatial resolution of ~4 μm, facilitating the search for the tiniest sample. The system can be used for the quick alignment of target position in nanoprobe XAFS measurements. In the near future, a highly efficient imaging system will be completed by installing a multi-element silicon drift detector (SDD).

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