BL14B2
XAFS II

1. Introduction

BL14B2 is a bending magnet beamline at SPring-8 dedicated to research by industrial and academia users conducting X-ray absorption spectroscopy (XAS) and X-ray imaging measurements. Various measurement systems have been developed to realize easy and high-throughput operations of XAS measurements. In the last two years, a V–F converter system with high output frequency and a fully automatic pellet sample preparation system has been developed in BL14B2 [1,2]. In FY2023, we developed a new X-ray diffraction (XRD) measurement system using a two-dimensional detector for *in situ* XRD-XAS measurements.

2. Development of XRD measurement system for *in situ* XRD-XAS measurements

Recently, in the field of electrochemistry, the demand arose to investigate changes in the chemical state, local structure, and crystal structure under various electrochemical reactions. XAS measurements provide information on chemical states and local structures, and XRD measurements reveal crystallographic information such as lattice parameters, crystalline size, and crystal orientation. In FY2023, we developed a new XRD measurement system utilizing a two-dimensional detector, and we combined this system with the XAS measurement system to conduct an *in situ* XRD-XAS measurement.

Figure 1 shows the (a) schematic illustrations and (b) photograph of the developed XRD-XAS measurement system. A two-dimensional detector (PILATUS 300K) is used to collect the XRD pattern.

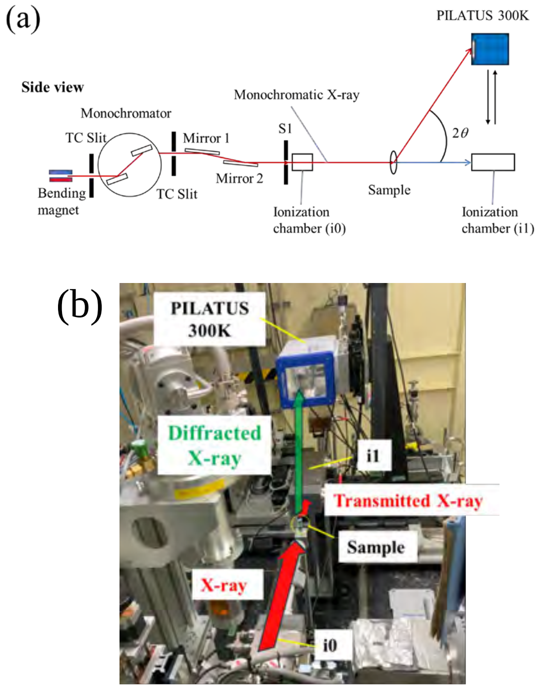

Fig. 1. (a) Schematic illustration and (b) photograph of the XRD-XAS.

The detector is fixed on an auto–linear Z-stage located downstream of the sample. The linear Z-stage can move to any desired height, enabling the acquisition of the required diffraction profile. The distance between the sample and the detector (camera length) can be adjusted manually in the system.

Figure 2 shows the (a) XRD and (b) XAS patterns of CuO powder. CuO was chosen as a test sample for XRD-XAS measurement. During XAS measurement, the XAS spectra were collected at the Cu-*K* absorption edge in the transmission mode. The X-ray energy and camera length during XRD measurement were set to 8.9 keV and 466.3 mm, respectively. The camera length was calibrated using CeO_2 powder diffraction. As a result, we could obtain XAS and XRD profiles in a measurement time of about 10 min. This XRD measurement system can be combined with transmission and fluorescence XAS measurement mode, making it adaptable not only for bulk samples but also for flat samples such as thin films. In FY2024, we plan to open for public users and to conduct *in situ* XRD-XAS measurements during electroreduction reactions.

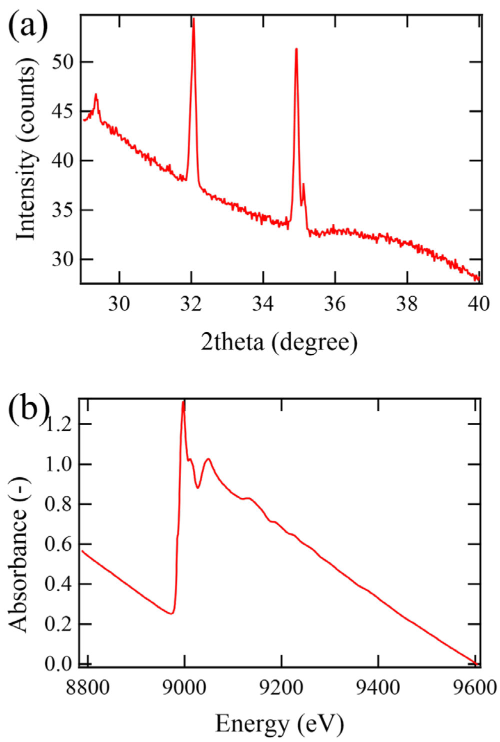

Fig. 2. (a) XRD pattern and (b) Cu-K edge XAS spectrum of CuO test sample.

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References:

[1] Ofuchi, H. & Honma, T. (2021). *SPring-8/SACLA Annual Report FY2021*, 45–47.

[2] Ofuchi, H. & Honma, T. (2022). *SPring-8/SACLA Annual Report FY2022*, 41–43.