BL36XU
RIKEN Materials Science II

1. Introduction

BL36XU is the RIKEN Materials Science II beamline constructed by the University of Electro-Communications, Institute for Molecular Science, and Nagoya University with the support of RIKEN and JASRI under a New Energy and Industrial Technology Development Organization (NEDO) program for the development of polymer electrolyte fuel cells (PEFCs). Construction was completed in November 2012, and user operations began in January 2013. BL36XU became a RIKEN beamline in March 2020 and serves users of RIKEN proposals (40–50% of beamtime), project proposals (NEDO PEFC project and CREST Innovative Measurement and Analysis project) (40–50%), and general proposals (10%).

BL36XU consists of an in-vacuum-type tapered undulator and two channel-cut monochromators having a channel-cut Si (111) crystal and a Si (220) crystal, which are tandemly arranged to cover an energy range from 4.5 to 35 keV [1]. The design of the synchrotron light source and X-ray optics is a SPring-8 standard. BL36XU provides time-resolved quick scan XAFS (QXAFS, time resolution of 10 ms), full-field/scanning XAFS imaging (spatial resolution of 100 nm–1 μm), XES [high-energy-resolution fluorescence detected XANES (HERFD-XANES) and resonant inelastic X-ray scattering (RIXS)], simultaneous time-resolved QXAFS/XRD, ambient-pressure HAXPES, and pink beam experiments under *in situ* experimental conditions.

Available X-ray detectors are fast ionization chambers, a four-element silicon drift detector, a two-dimensional pixel array detector, and indirect X-ray imaging detectors. Equipment for controlling the sample environment includes a cryostat (4 K-RT), reaction gas supply and removal equipment, a high-temperature gas cell (RT-1000 K), a fuel cell, and power generation equipment.

2. Recent activities

2-1. *In situ/operando* spatially resolved pump-probed XAFS measurement system

A Rh-coated horizontal focusing elliptical mirror, which so far had been used for AP-HAXPES experiments, was relocated to a position upstream of the experimental base plate. The combined use with vertical focusing mirrors (M3 and M4) enables the use of a high-flux two-dimensional focused X-ray beam of 35 (h) × 20 (v) μm for experiments on various samples. The glancing angle and focal distance of the elliptical mirror respectively are 4 mrad and 1 m in the energy range from 4.5 to 15 keV and 2 mrad and 0.5 m in the energy range from 15 to 30 keV.

Using a high-flux 20–40 μm beam, we developed a spatially resolved pump-probe XAFS measurement system in the fluorescence mode for *in situ*/*operando* analysis of chemical and electrical reactions of dilute samples. Figure 1 shows the experimental setup of the measurement system. Fluorescence X-rays from the sample were detected using a two-dimensional pixel detector (Eiger 1M, Dectris). The minimum time resolution is 330 μs, limited by the maximum frame rate of the two-

dimensional pixel detector (3 kHz). A Soller slit and a filter were placed between the sample and a two-dimensional pixel detector to improve the signal-to-noise ratio of the data by removing background X-ray scattering from the sample.

The *operando* simultaneous UV-VIS and spatially resolved pump-probe XAFS measurement system successfully expands the research target for mapping spatially inhomogeneous electrochemical reactions in electrochromic devices during a stepwise external voltage change with time and spatial resolutions of 1 ms and 35 μm, respectively.

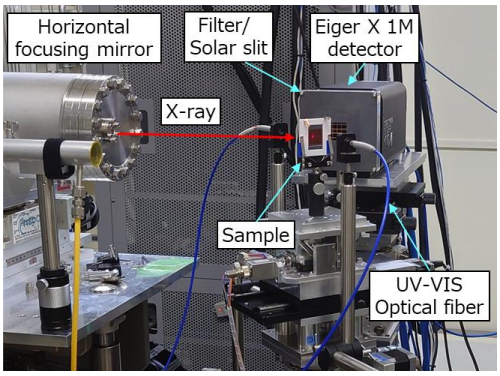

Fig. 1. Experimental setup of *operando* simultaneous UV-VIS and spatially resolved pump-probed XAFS measurement system.

2-2. *In situ/operando* time-resolved nano QXAFS measurement system

We developed a compact alignment system for two sets of Kirkpatrick–Baez (KB) mirrors for low-energy (4.5–15 keV) and high-energy (15-30 keV) regions. Figure 2 shows a schematic layout of the measurement system. The low- and high-energy mirrors are switched using translation stages and their glancing angles are precisely aligned using tangential bar rotary stages, which enables continuous measurement of nano-XAFS in low- and high-energy regions without the need to attach and detach mirrors.

Using a nanobeam, we conducted *in situ/operando* time-resolved nano QXAFS measurements for mapping spatially inhomogeneous dynamical structural changes in a single crystal under high temperature. The single-crystal samples were stably mounted on a SiN membrane held in a temperature-controlled sample cell. The measurements were successfully conducted with time and spatial resolutions of 100 ms and 200 nm, respectively, at 300–450 K.

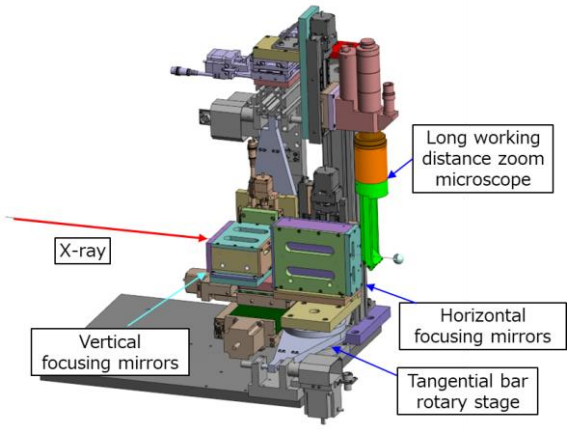

Fig. 2. Schematic of layout of alignment system for two sets of KB mirrors for low- and high-energy regions.

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Reference:

[1] Uruga, T. et al. (2019). *Chem. Rec*. **19,** 1444–1456.