

## 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 <sup>[1]</sup>. The synchrotron light source and X-ray optics design 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  $\mu$ m), XES [high-energy-resolution fluorescence detected XANES (HERFD-XANES) and resonant inelastic X-ray scattering (RIXS)], simultaneous time-resolved QXAFS/XRD, 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* nano QXAFS-CT imaging system using advanced KB mirror

A nano CT-XAFS imaging system using a Fresnel zone plate (FZP) in BL36XU was constructed in 2014 <sup>[1]</sup>. We newly constructed a nano XAFS-CT measurement system using an advanced KB (AKB) mirror developed by RIKEN/Osaka Univ. (Prof. Yamauchi Lab.) <sup>[2]</sup> in collaboration with Osaka Univ. AKB mirrors have no chromatic aberration, namely, the focal length is constant with no dependence on the X-ray energy and the reflection efficiency is more than 4 times that using FZP. The performance of the AKB mirror enables rapid QXAFS-CT measurements by measuring two-dimensional transmission QXAFS images at each projection angle of the sample, which gives higher quality XAFS spectra of each three-dimensional pixel than that by CT-XAFS measurement (CT measurement at each X-ray energy). To further improve the measurement efficiency, we introduced a fiber optic plate (FOP) coupled with an X-ray sCMOS camera (Hamamatsu Photonics, pixel resolution: 6.5  $\mu$ m) as a two-dimensional X-ray image detector. We relocated the modified detector booth to 9.8 m downstream of the AKB mirror (Fig. 1) and placed

the FOP-coupled sCMOS camera in the detector booth; this realized an optical magnification ratio of 230 by the AKB mirror and an X-ray image pixel size of 30 nm. *In situ/operando* nano QXAFS-CT measurements of material samples have been successfully conducted using this system.

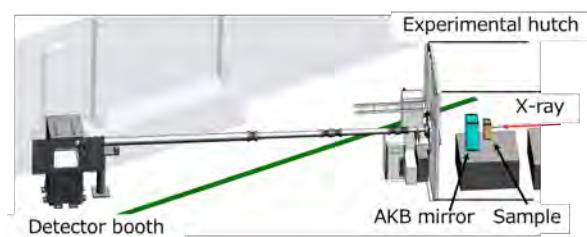


Fig. 1. Layout of detector booth and experimental hut for nano QXAFS-CT imaging.

## 2-2. High-quality fluorescence mode QXAFS measurement system using CITIUS detector

We constructed a high-efficiency fluorescence QXAFS measurement system using a two-dimensional X-ray pixel detector (DECTRIS, Eiger 1M) and applied it to *in situ* time-resolved QXANES measurement with a time resolution of 20 ms in BL36XU [3]. With the cooperation of the RIKEN Detector Team, we have been constructing a new fluorescence-mode QXAFS measurement system using the CIRIUS detector [4] to realize a faster (higher time resolution) and higher quality fluorescence QXAFS measurement. The CITIUS has an energy resolution (ca. 600 eV by droplet analysis, ca. 350 eV by single photon event analysis) that realizes XAFS spectra with high signal-to-background ratios by selectively measuring the X-ray fluorescence of the target element separated from background X-rays. The CITIUS works with a high frame rate (17.4 kHz),

which enables time-resolved QXAFS measurement with a time resolution more than five times that measured with Eiger.

Feasibility studies of CITIUS 280K (Fig. 2) showed that the QXAFS measurement of dilute samples can be conducted 10–100 times faster than with multi-element semiconductor detectors such as SDDs and Ge detectors. We are constructing a user-friendly and easy-to-use measurement system using CITIUS.

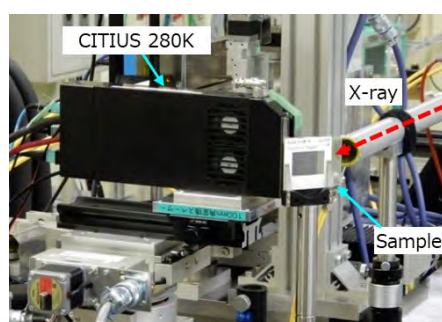


Fig. 2. Experimental setup of fluorescence-mode QXAFS measurement system using CITIUS 280 K detector.

URUGA Tomoya<sup>\*1,\*2</sup>

<sup>\*1</sup>Physical and Chemical Research Infrastructure Group, RIKEN SPring-8 Center

<sup>\*2</sup>Research Project Division, JASRI

## References:

- [1] Uruga, T. et al. (2019). *Chem. Rec.* **19**, 1444.
- [2] Matsuyama, S. et al. (2019). *Opt. Express* **27**, 18318.
- [3] Yamamoto, T. et al. (2023). *Adv. Sci.* **10**, 2301876.
- [4] Hatsui, T. et al. (2014). *SPring-8-II Conceptual Design Report*.