BL10XU
High Pressure Research

1. Introduction

BL10XU is a beamline specialized for high-pressure X-ray diffraction (XRD) measurements using diamond-anvil cells (DAC) for structural analysis and the investigation of physical properties by multimodal measurements under extreme conditions. BL10XU is equipped with an in-vacuum undulator and a liquid-nitrogen-cooled double-crystal monochromator. The monochromator mounts two switchable Si (111) and (220) crystals and can deliver a 10^{-4}-bandwidth X-ray beam ranging from 6 to 62 keV. BL10XU has employed a combination of several X-ray compound refractive lenses made of various materials, and they can achieve 200 μm to 800 nm X-ray beam spot sizes. BL10XU has two experimental hutches. In experimental hutch 1 (EH1), single crystal and powder XRD measurements can be carried out at cryogenic temperatures (> 7 K) using a 4 K GM cryostat. Raman spectroscopy spectra can also be collected from the upstream side of the sample simultaneously with XRD measurements. In experimental hutch 2 (EH2), the primary methods are XRD measurement at exceedingly high temperatures (<6000 K) through a double-side laser heating system, and X-ray diffraction measurement performed under ultra-high pressure (over 300 GPa) utilizing a submicron focused X-ray.

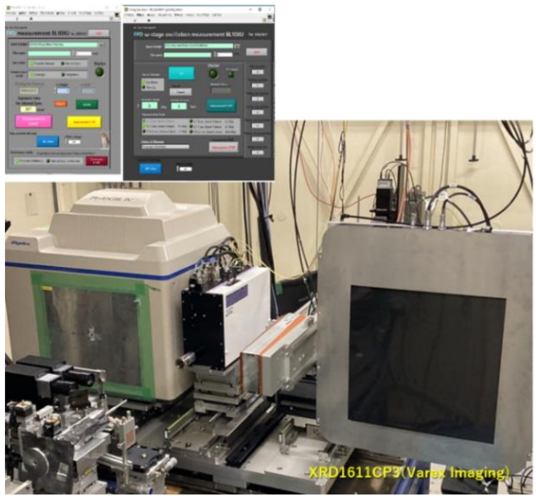
In recent years, we have focused on development and improvement to make the XRD measurement platform of BL10XU more efficient and precise. Although each scientific field needs its own unique setup, it is essential for all users in any scientific field to obtain high-quality data easily and quickly. Therefore, in FY2020, the software that serves as the basis for the unified control of all instruments has been updated. In FY2021, a dual gas-pressure control system was developed to remotely control the sample pressure with precision. A large-area flat-panel detector was installed this year to achieve higher angular resolution and more efficient measurements. In addition, flat-top laser optics has been installed for homogenized laser heating. Here, the details of these developments and upgrading are introduced in the following sections.

2. New large-area flat-panel detector and development of XRD measurement systems

Obviously, the X-ray detector is one of the most crucial components of the X-ray diffraction platform. Both experimental hutches of BL10XU have been equipped with large-area imaging plate detectors (Rigaku R-AXIS IV++, reading resolution 0.1 mm, area size 300 × 300 mm^2) and an X-ray flat-panel detector (Perkin Elmer, XRD0822 CP23, pixel size 0.2 × 0.2 mm^2, area size 204.8 × 204.8 mm^2). The imaging plate has a large area, high reading resolution, and a wide dynamic range. Conversely, flat-panel detectors are characterized by their high sensitivity and a rapid imaging speed of 25 Hz. For instance, the imaging plate has been employed for precise structural analyses of both single-crystal and powder samples. On the other hand, flat-panel detectors have been the choice for observations of crystal structural changes under varying external conditions. Furthermore, starting from FY2020, a CdTe hybrid pixel array X-ray detector (X-Spectrum, LAMBDA CdTe 750k, with a pixel size of 55 μm × 55 μm and an area size of 512 × 1528 pixels) was introduced. This allows for high-speed measurements up to a maximum of 2000 Hz. On the other hand, while the imaging plate continues to be one of the superior detectors, when used at BL10XU, taking measurements while oscillating the sample often required more than 10 minutes for a single data set, making efficient measurements challenging. During the 10-minute measurement time, temperature and pressure often varied. Moreover, the imaging plate in EH2, in particular, is a detector introduced in 2006, and issues associated with its aging have increasingly arisen.

Consequently, in FY2022, we introduced a large-area flat-panel detector, Varex XRD1611CP [Fig. 1(a)]. This detector possesses a pixel size of 0.1×0.1 mm^2 and an area size of 41 cm × 41 cm, offering a resolution surpassing that of the existing imaging plate detectors. The angular resolution is equivalent to 0.01°. The distance between the sample and the detector can be adjusted from 300 to 470 mm. One-shot measurements are possible with a minimum exposure time of 267 ms. Fig. 1. (b) shows the results of the precise structure analysis of CeO_2 at 30 keV (±5° oscillation × 2 rounds and measurement time of about 60 seconds). By achieving synchronization with the oscillation stage, XRD measurements intended for structural analysis, which previously took over 10 minutes using the imaging plate, can now be completed in about one minute. As shown in Fig. 1(a), as a result of the software development and establishment of an automatic detector switching system by the end of FY2022, this detector and its associated measurement system have been available to users since the start of the 2023A period. Our objective for the next fiscal year is to implement automatic sequence measurements using this detector while changing both temperature and pressure.

(a)



(b)

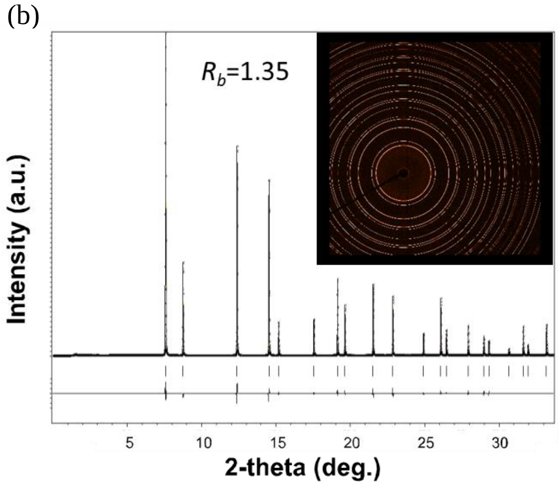


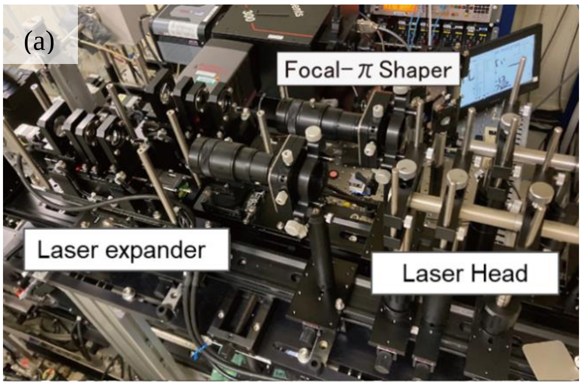
Fig. 1 (a) Results of software development and construction of an automatic detector switching system by the end of FY2022. (b) Results of precise structure analysis of CeO_2 at 30 keV (±5° oscillation × 2 rounds and measurement time about 60 seconds).

3. Upgrade of optics for heating area uniformity in laser heating systems

As mentioned above, at BL10XU, a double-side laser heating system incorporating two lasers is available for high-temperature experiments. For laser heating experiments and to enhance the reliability of its temperature measurements, it is crucial to maintain a uniform heating region and to minimize aberrations in the temperature measurement/monitoring optics.

At BL10XU, custom-designed focusing and imaging lenses have been introduced to reduce chromatic, distortion, and wavefront aberration in the central wavelength range of 600–800 nm. Efforts have been devoted to enhancing the precision of the temperature measurements. However, regarding uniform heating, because of the aging of the laser homogenizer we have been using, achieving uniform heating has become difficult. Depending on the experimental conditions, a temperature nonuniformity of up to 20% relative to the standard X-ray spot size of 10 μm at BL10XU had been observed. A temperature gradient in samples during heating not only degrades the accuracy of XRD measurements and physical property measurement data but can also potentially lead to compositional heterogeneity within the sample. Moreover, in recent years, the number of challenges related to the field of material synthesis has been increasing, and there is a widespread demand to heat samples uniformly over as broad a laser heating area as possible. However, it was inherently impossible to shape a laser diameter of more than φ40 μm using the existing homogenizers.

To achieve flat-top laser heating, at the end of FY2021, we designed and introduced a new optical system comprising a combination of a focal π-shaper (Focal-πShaper 9_1964, AdlOptica) and a laser expander [Fig. 2. (a)]. The focal π-shaper is an optical system that achieves flat-top heating at the focal position using a focusing lens. The motorized laser expander, custom-designed for BL10XU by OptoSigma, is installed just after the π-shaper. By employing the motorized laser expander, users can change the heating spot size without entering the hutch. Fig.2 (b) displays temperature measurement profiles of a platinum foil that was test-heated. Combining these components has enabled flat-top heating of samples ranging from φ15 to 100 μm at the sample position. This system has been available to users since the 2022B term. The enhanced



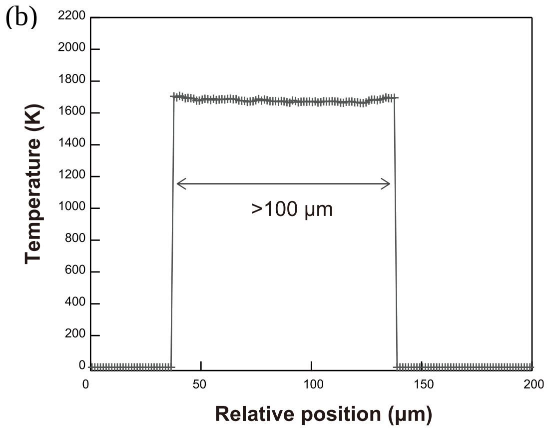


Fig. 2 (a) New optical system comprising a focal π-shaper (Focal-πShaper 9_1964, AdlOptica) and a laser expander. (b) Temperature measurement profile of platinum foil during test heating.

uniformity of heating and user-friendliness have been very well-received.

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