BL10XU
High Pressure Research

1. Introduction

BL10XU/SPring-8 is dedicated to the visualization of crystal structure via *in situ* X-ray diffraction (XRD) measurements targeting high-pressure and microscopic samples using diamond anvil cells (DACs). It utilizes a vacuum-sealed undulator as the light source and switches the crystal planes of the monochromator between (111) and (220) to monochromatize the light, allowing the use of extremely high-brightness and high-energy X-rays (6–62 keV). The samples in the DAC are microscopic, ranging in size from hundreds of microns to several microns. At BL10XU, multiple sets of X-ray compound refractive lenses are used to provide focused X-ray beams ranging from millimeter to submicron sizes [minimum 0.8 mm (H) × 0.8 mm (V), FWHM]. BL10XU has two experimental hutches: in experimental hutch 1 (EH1), single-crystal and powder X-ray diffraction measurements are conducted at ultralow temperatures (>7 K) using a 4 K GM cryocooler; in experimental hutch 2 (EH2), XRD measurements are performed at room temperature and high temperatures using a resistance heating system (<1500 K) or a laser heating and radiation temperature measurement system (<6000 K), as well as under ultrahigh pressures utilizing submicron-sized X-ray beams.

In recent years, BL10XU has focused on developing and improving the XRD measurement system utilizing focused X-rays to make it more efficient and precise. In FY2020, the software forming the basis for the unified control of all devices was updated. In FY2021, a dual gas pressure control system for the precise remote control of sample pressure and a new optical system for uniform laser heating were developed. In FY2022, a large-area flat-panel detector was introduced to achieve higher angular resolution and efficient measurements. This fiscal year, to further enhance the precision of XRD measurements using the developed systems, a harmonic rejection mirror was introduced, floor stabilization work was carried out, and motor drivers were relocated outside the hutch to ensure uniform temperature within the hutch. The details of these developments and improvements are introduced below.

2. X-ray optical mirrors for high harmonic rejection

At BL10XU, numerous users have been employing 30 keV X-rays from the Si 111 double-crystal monochromator to obtain X-ray diffraction (XRD) data. However, the beamline has not yet been equipped with an X-ray optical mirror, resulting in data contamination from intense single-crystal reflections of the diamond anvil (single-crystal diamond) of the DAC, as well as reflections from the sample's harmonics. This contamination complicates peak searches for polycrystalline and single-crystal samples, frequently impeding experiments. To mitigate harmonic contamination, the double-crystal monochromator was detuned for structural analysis purposes. Nevertheless, this detuning significantly reduced the fundamental wave intensity to less than one-half to one-tenth of the optimal level, necessitating prolonged exposure times and leading to an inefficient measurement environment. To advance future ultrahigh-pressure experiments and structural analysis research, an X-ray optical mirror was installed to eliminate harmonics. Concurrently, the TC slits in the optical hutch were replaced with high-precision slits, consistent with recent SPring-8 standards. In addition, the beamline network was transferred to a new instrument control platform, BL-774, with the installation of this mirror.

Two X-ray optical mirrors were installed to achieve parallel X-ray emission, with mirror chambers positioned approximately 45 m from the light source at the downstream end of the optical hutches, each housing one mirror. The specifications of the mirrors are as follows: the Si mirror is 700 mm in length, 70 mm in width, and 50 mm in thickness, with a 24-mm-wide Pt-coated area. The reflection angle is approximately 2 mrad. The Si-only area is employed for harmonic rejection when utilizing fundamental X-rays of 10–15 keV (e.g., for imaging), whereas the Pt-coated surface is used for XRD measurements with fundamental X-rays of around 30 keV. The mechanical components, including the chamber, are of standard specifications equivalent to those used in BL13XU and other upgraded beamlines. The upstream mirror (M1) reflects X-rays towards the ring side, and the downstream mirror (M2) reflects them towards the hole side, ensuring the X-ray optical axis is ultimately aligned parallel to the ring side.

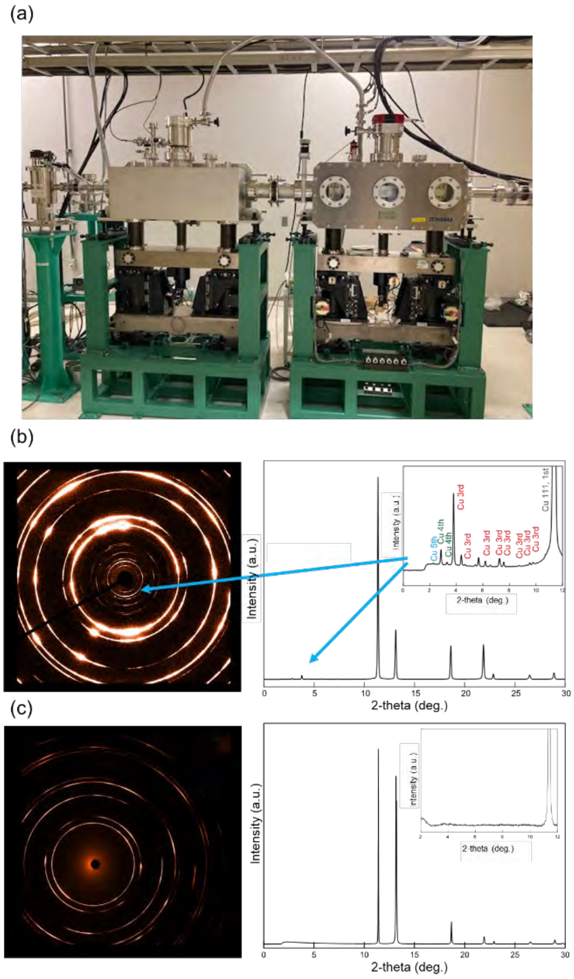

Fig. 1. (a) Mirror chamber located in the optical hutch. (b), (c) 2D and 1D XRD patterns of copper foil acquired without and with harmonic rejection mirror, respectively.

Figure 1 shows a photograph of the higher harmonic rejection mirror chamber units installed in the optics hutch, along with the 2D and 1D XRD results of Cu foil as a sample, where the data before and after the mirror installation were compared. In both cases, X-rays with an energy of 30 keV, focused down to approximately 10 μm, were used to obtain XRD data of the Cu foil. The mirror angle was adjusted to 2 mrad and used. The introduction of the mirror effectively eliminates the harmonic-derived XRD peaks observed at low scattering angles prior to its installation. Software for adjusting the mirror and a vacuum path for shifting the optical axis of the X-rays have been installed, and the mirror is now in regular use. In the future, we plan to utilize harmonic-free X-rays using mirrors and combine them with large-area flat-panel detectors and high-speed photon counting detectors to further improve the efficiency of high-pressure XRD measurement and to realize applied measurements such as multigrain XRD measurement.

3. Stabilization of focused X-rays: Reduction of floor vibration and temperature uniformity in the experimental hutch

As explained at the outset, BL10XU provides users with micro- to submicron focused X-rays using X-ray refractive lenses for the XRD measurement of small samples in the DAC. To optimize the use of micro–X-rays, it is essential to ensure the high stability of the X-ray optical elements and sample position. Therefore, at BL10XU, the floor, which had become unstable due to aging, has been repaired. Figure 2 shows images of a 60-μm-diameter pinhole mounted on the sample stage before and after the floor was repaired, illustrating the displacement of the pinhole when a person approaches the sample stage and then moves to a location 50 cm away from it. Before the floor repair, there was a displacement of more than 6 μm, whereas after the repair, there was almost no displacement.

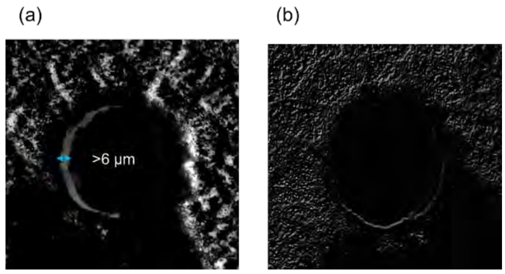

Fig. 2. Images of a 60-μm-diameter pinhole mounted on the sample stage (a) before and (b) after the floor was repaired.

Additionally, the SPring-8 standard motor driver and controller, which had been the primary heat sources, were relocated outside the experimental hutch to achieve a more uniform and lower temperature within the hutch. Previously, the temperature in the hutch had reached nearly 30 °C, exceeding the optimal operating temperature range of the detectors. This temperature discrepancy between the inside of the hutch and the outside air caused significant inhomogeneity. In EH2, motor drivers and controllers (for more than 80 axes), constituting the majority of the heat sources, were installed at the back of the hutch. To accommodate this, a deck was installed on the ceiling to secure the 19" racks, and the motor driver cables were extended by 20 m each.

Figure 3 shows the room temperature at the back of the experimental hutch before and after the relocation of the motor driver. It can be seen that after the motor driver was relocated, the room temperature decreased by about 3 °C, and the stability of the room improved. Thus, efforts to improve the stability of X-rays, laser mirror optics, and stages will lead to the stabilization of low Z elements, e.g., hydrogen, and ultrahigh-pressure experiments. It can expect to generate more data in the future.

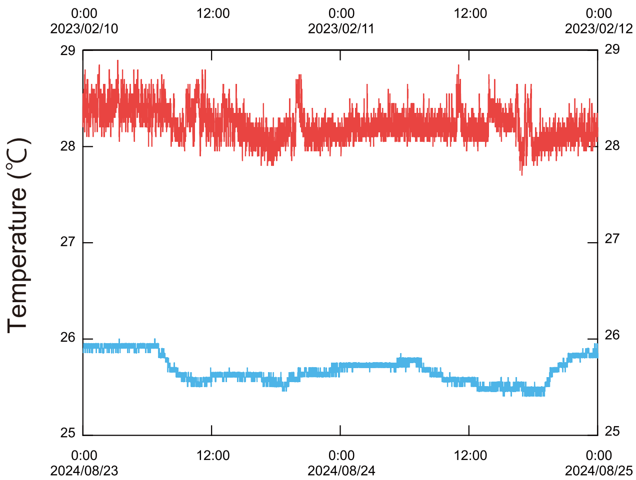

Fig. 3. Room temperature in EH2 before (red) and after (blue) removal of motor drivers.

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