

## BL10XU (High Pressure Research)

### 1. Overview

BL10XU is a public beamline dedicated to high-pressure X-ray diffraction measurements using monochromatic X-rays. The scientific fields cover high-pressure physics and chemistry, materials sciences, and Earth and planetary sciences. Integration of a high-pressure apparatus diamond anvil cell (DAC) and cryostat/laser heating allows X-ray diffraction experiments ranging from cryogenic temperature to over 3,000 K to be performed under high pressure conditions. Over the last two decades, we have been developing X-ray focusing optics, diffractometers, and detectors suite for high-pressure X-ray diffraction. High-pressure and high-temperature X-ray diffraction using the laser-heated DAC technique is well established for studying crystallography, phase relation, and  $P$ - $V$ - $T$  equation of state of deep Earth materials. Recently, the number of high-pressure and high-temperature synthetic studies has increased rapidly using this technique. Studies include hydrogen-rich materials called hydrides. Precise laser-heating-controlling techniques and accurate temperature measurements are required to enhance X-ray diffraction under extreme conditions over 5,000 K and to synthesize new materials. Additionally, there is a growing need for observations of pressure-induced phenomena of liquid/amorphous materials, including changes in the coordination number and polyamorphism. To meet these requirements, we developed a laser heating system and a high-energy X-ray focusing refractive optics in FY2018. Here, the details are described.

### 2. New developments in the laser-heating system with *in situ* synchrotron high-pressure X-ray diffraction

The stability of the laser spot and fast temperature measurements are key components to *in situ* high-pressure and high-temperature X-ray diffraction measurements. The conventional online laser heating system installed at BL10XU made it very difficult to keep the laser spot in one position for a long time. However, a long exposure time is necessary to determine the temperature.

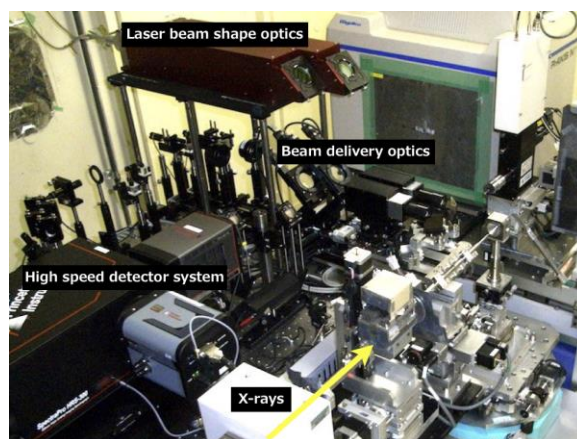


Fig. 1. Photograph of the *in situ* laser-heating system for high-pressure X-ray diffraction with the DAC.

In FY2018, we introduced a few modifications to improve the laser pointing stability and temperature measurements for laser heating. Improvements included installing high-power laser optics capable of accurate beam shape control, laser beam delivery optics to a sample in the DAC, and high-speed CCD detectors (Fig. 1). In the new beam delivery optics, we changed the mounting method for X-ray

transparent silver-coated glassy carbon mirrors, adopted the gimbal mechanism in the mirror mounts, and placed a 100-mm-thick honeycomb optical table to meet the requirements for rigidity, vibration isolation, system stability, and user-friendliness for laser heating. This setup also controls the laser beam spot with a precision of a few  $\mu\text{m}$ . Currently, we are optimizing the flat-top optics for a high-power laser beam and developing an interface program that combines temperature measurements and diffraction pattern collection.

### **3. X-ray-focusing refractive optics at a high energy of 62 keV**

High-energy focused X-ray probes are essential for high-pressure X-ray diffraction with a DAC involving minute samples. An X-ray focusing technique using a compound refractive lens (CRL) was developed over the last two decades at the SPring-8 BL10XU beamline. To realize high-pressure X-ray diffraction using high-energy monochromatic micro X-ray beam, X-ray-focusing SU8-CRL manufactured by synchrotron deep X-ray lithography was installed in experimental hutch 2. SU8-CRL, which has a focal length of 500 mm and a physical aperture size of 80  $\mu\text{m}$ , is used to focus the 62-keV incident X-ray. In combination with a pre-focusing aluminum CRL installed in optics hutch, the beam size, which is defined as the full width at half maximum (FWHM) of the intensity profile, is approximately 10  $\mu\text{m}$  both in the horizontal and vertical directions at the sample position in the experimental hutch. The flux is  $3 \times 10^{10}$  photons/s at a storage ring current of 100 mA when using an incident slit size of 80  $\mu\text{m} \times 80 \mu\text{m}$  and a 30- $\mu\text{m}$  cleanup pinhole. This new X-ray probe will enhance amorphous/liquid X-ray diffraction

measurements, and should allow for a deeper understanding of the Earth's liquid outer core and non-crystalline structures.

### **4. Next-generation hard X-ray undulator**

A new in-vacuum undulator, which consists of a magnetic attractive force cancellation system, was built as an insertion device of the X-ray source at BL10XU. Performance tests were launched in 2018. This undulator realizes synchrotron-based multi-probe measurements using X-ray energy ranges that could not be achieved using the previous hybrid-type undulator with a shorter period suitable for high-energy X-ray generation. The new device will be installed at BL10XU in FY2019.

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