BL10XU
High Pressure Research

1. Introduction

BL10XU is an undulator beamline at SPring-8 dedicated to angular-dispersive X-ray diffraction (XRD) measurements using monochromatic X-rays and area detectors at high pressure in a diamond anvil cell (DAC). In recent years, the number of high-pressure experiments in the multi-megabar pressure region (200 GPa or higher) has increased not only in deep Earth interior studies but also in high-pressure physics and chemistry. Even under such extreme conditions, the pressure must be precisely determined for high-pressure XRD. Furthermore, high-pressure research using high-energy X-rays is expected to evolve to realize time-resolved high-pressure XRD experiments under a fast compression or high-pressure and -temperature conditions, as well as high-pressure XRD measurements for liquids with a low scattering power (e.g., oxide and silicates). To meet these requirements, in FY2019 an optical and confocal Raman spectroscopy probe system was developed for precise pressure determination, and a photon-counting hybrid pixel array detector was installed for high-energy X-rays. This report details these advances.

2. Developed online Raman spectroscopy system to combine with X-ray diffraction

In high-pressure experiments, pressure determination is a fundamental issue. The ruby luminescence method and the diamond anvil Raman gauge are widely used for pressure measurements in high-pressure experiments with DACs. The latter is mainly utilized when high-pressure regions above 100 GPa or other pressure markers are unavailable. However, the non-confocal Raman probe optics, which was initially installed, has made collecting high-quality Raman spectra from samples smaller than 50 μm in the multi-megabar pressure region challenging due to the strong stress-induced fluorescence increase from diamond anvils of the DAC and the large Raman probe spot. In FY2019, a micro-Raman probe unit with confocal optics was developed to solve these challenges and to acquire Raman spectra with a high signal-to-noise ratio at a high pressure over 200 GPa (Fig. 1).

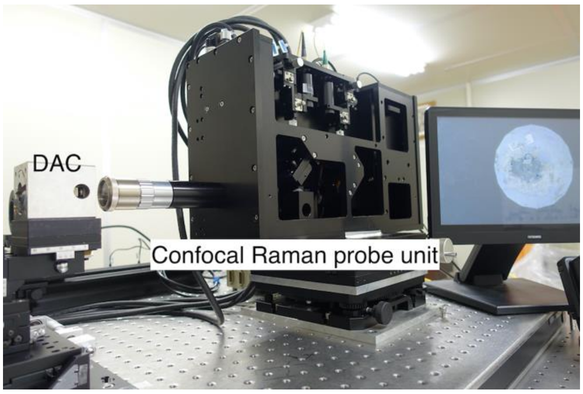

Fig. 1. Photograph of the fiber-based Raman probe unit with a confocal optical system.

The newly developed Raman probe unit has the following features: (1) confocal optics, (2) spherical aberration–corrected objective lens for the diamond anvil, and (3) dual excitation lasers. These features allow a micro-focused laser probe with a spot size of 2–3 μm and a depth resolution of less than 35 μm. Depending on the sample and experimental conditions, two different excitation lasers with wavelengths of 532 nm and 633 nm are available. In the confocal optics of this Raman probe unit, a motorized aperture is adopted at the conjugate focus.

Thus, the aperture size can be continuously controlled. Preliminary results indicated that the signal-to-ratio of the Raman spectrum was improved several times compared to that using the previous non-confocal Raman spectroscopy system. The optimization of the optical system of the Raman probe unit will be continued for online utilization.

3. Photon-counting hybrid pixel detector for high-energy XRD experiments

Two types of area detectors, an image plate (IP) detector and a flat panel detector (FPD), have been used for high-pressure XRD with high-energy X-rays (20–62 keV). The IP detector is utilized mainly for precise crystal structure analysis requiring accurate determination of the XRD peak intensities with a high angular resolution over a wide angular region. The FPD is a suitable device for conducting moderately fast XRD experiments and real-time monitoring under extreme conditions. In addition, the photon-counting hybrid pixel detector LAMBDA system with a high-Z pixel sensor of Cadmium Telluride (CdTe) from X-Spectrum GmbH was newly installed. The LAMBDA CdTe 750k is a Medipix3-based pixel array with a small pixel size (55 μm × 55 μm) and a format of 1528 × 512 pixels. The detector can acquire images up to 2000 frames per second with a 12-bit counter depth, which is 100 times faster than the FPD. This allows time-resolved high-energy XRD experiments on the millisecond timescale such as the accurate determination of *P*–*V*–*T* structure relations at a fast compression and during a chemical reaction under high pressure and temperature. In another mode of the detector, noise-free images can be collected with 24-bit depth for a signal range of 0–16 million photon hits per pixel. This mode provides an excellent signal-to-noise ratio and should realize crystallography of low-Z materials at high pressure, non-crystalline XRD studies for high-pressure small samples, and measurements of melting curves at high pressure and temperature.

In FY2019, the construction of measurable environments, including software development, performance tests, and trial measurements, were performed (Fig. 2).

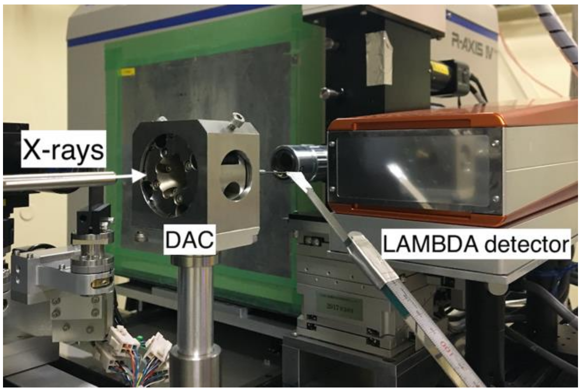

Fig. 2. Experimental setup of the detector during a trial measurement for high-pressure XRD.

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