BL10XU
High Pressure Research

1. Introduction

The high-pressure beamline BL10XU is dedicated to monochromatic X-ray diffraction experiments of materials under extreme conditions. This beamline has two specialized experimental hutches: a laser-heated diamond anvil cell (DAC) hutch and a cryogenic-temperature DAC hutch. Hydrides have become one of the important materials in many technological fields. The number of high-pressure research studies on hydrides, such as metal–hydrogen systems, has rapidly increased over the past five years, and the fundamental properties of pressurized hydride compounds have been studied for application to novel materials development and Earth and planetary sciences. Recently, high-temperature superconductivity has been demonstrated in hydride materials in high-pressure experiments, such as sulfur hydride, lanthanum hydride, and carbonaceous sulfur hydride with their critical temperatures above 200 K. Now, there is an urgent need for understanding the mechanism of high-temperature superconductors. Therefore, to advance the in situ X-ray observation of hydride materials under extreme conditions, the installation of a hydrogen gas-loading system for the DAC had been strongly required. In addition, a new multichannel collimator system has been designed and installed to collect X-ray diffraction data with low background from, among others, low-Z materials, liquid/amorphous materials, and tiny samples under multiple megabar to terapascal pressures. Details of these developments are discussed in this report.

2. High-pressure hydrogen gas-loading system for DACs

The new gas-loading system that can operate up to a maximum pressure of 200 MPa at room temperature was developed in FY2020, which can handle hydrogen, helium, neon, argon, nitrogen, and their gas mixtures. Figure 1 shows a schematic diagram of the high-pressure gas-loading system. Gas is supplied from gas cylinders and pressurized by an air-driven, oil-free, two-stage compressor. It usually takes about 5–10 min to compress hydrogen up to 200 MPa while maintaining the inlet pressure of the compressor at 5–6 MPa. The DAC is placed in a high-pressure vessel.

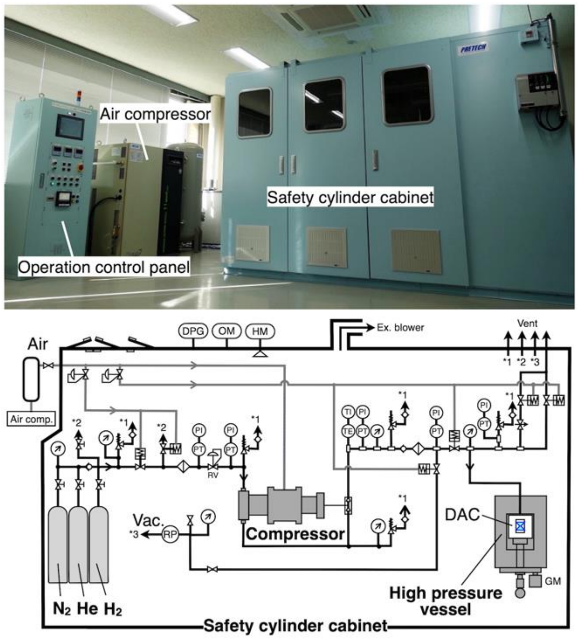

Fig. 1. Photograph and schematic diagram of the hydrogen gas-loading system.

The high-pressure vessel is made of tough-hardening steel, which has both high hardness and high tensile strength, for resisting hydrogen. The inner dimensions of the vessel are 80 mm in diameter and 75 mm in height, allowing to accommodate various types of DAC. The high-pressure vessel has a built-in drive shaft connected to a motor, and it allows the DAC to clamp directly from outside the vessel. Except for an operating control panel, the gas cylinder, compressor, and high-pressure vessel are placed in a safety cabinet to prevent an accidental release of hydrogen and a lack of oxygen. The system has been used successfully to load 200 MPa hydrogen, helium, or nitrogen into DACs as a pressure-transmitting medium for synchrotron radiation high-pressure experiments.

3. Multichannel collimator for structural studies of liquids and low-Z materials at high pressures

High-pressure X-ray diffraction data obtained using DACs include high-intensity Bragg spots single-crystal diffraction and Compton scattering from diamonds in addition to the diffraction from the sample. The mirrors for laser heating and windows of the cryostat also increase the background signals, and this is a serious problem for users. Moreover, the diffraction and scattering excluded from the sample yield a poor signal-to-background ratio.

To reduce the background signal intensity, we developed a multichannel collimator (MCC) for high-pressure X-ray diffraction measurement using DACs. We used tantalum blades for multislits, as shown in Fig. 2, because they must be sufficiently shielded from high-energy X-rays such as 62 keV scatterings around the sample environment. The inner slits of 1 mm thickness are located at 90 mm from the sample position, and the slit width is 0.071 mm. The outer slits of 3 mm thickness are located at 190 mm from the sample position, and the slit width is 0.316 mm. These slits can cover a diffraction angle of $\pm 30^\circ$ in 2 theta. We performed the X-ray diffraction measurement with the MCC and FPD detector. We collected 50–150 2D images with an acquisition time of less than 1 s per image. During the measurement, the MCC was oscillated to scan the entire diffraction angle, and finally, recorded data were merged into one diffraction data. With the developed MCC, the signals from mirrors for laser heating and windows of the cryostat can be completely removed from the X-ray diffraction data. In addition, the scattering intensities derived from diamond anvils are reduced by half above 10° in 2 theta. These studies focused on liquid/amorphous and low-Z materials in DACs have experimental difficulties. In the near future, using the developed MCC system, we believe that studies under extreme conditions can be accelerated.

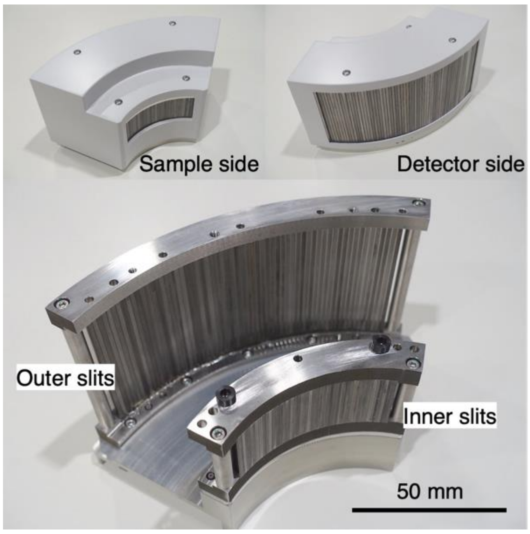

Fig. 2. Photograph of the developed MCC.

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