BL10XU
High Pressure Research

1. Introduction

BL10XU is a beamline designed for high-pressure experiments using a diamond anvil cell (DAC) and X-ray diffraction (XRD) studies of crystal structures and physical properties under high pressure. The beamline is equipped with an in-vacuum undulator as a light source, and monochromatic X-rays in the energy range from 6 to 62 keV with $\Delta E/E \sim 10^{-4}$ or less are available by switching between Si 111 and 220 reflections with a liquid-nitrogen-cooled double-crystal monochromator. The X-ray compound refractive lens system consisting of a combination of lenses made of glassy carbon, aluminum, and SU8 is employed to focus the X-rays. BL10XU has two experimental hutches: hutch 1 is for single-crystal and powder XRD measurements at cryogenic temperatures (down to 7 K) using a 4K-GM refrigerator, and hutch 2 is for very high temperatures (up to 6000 K) using a laser heating and radiation temperature measurement system and under very high pressure over the entire temperature range with sub-micro-focused X-rays.

Recently, the need for high-pressure experiments using DAC has expanded in various scientific fields. However, until now, DAC experiments have required users to have advanced skills. In FY2021, we focused on pressure control and started to develop a device that enables both precise compression and decompression experiments with simple user operation. In addition, as an advanced characteristic, a function for rapid compression and decompression experiments has been added.

2. Two-line gas pressure control system for remote compression-decompression experiments

Compression and decompression are unavoidable procedures in conducting DAC experiments. In the past, many users have increased and decreased pressure manually using screws and a gearbox outside of the experimental hutches, which required entering and exiting the hutches every time they needed to pressurize or depressurize the DAC. One option is to use a membrane-driven DAC to avoid frequent entry and exit into the hutches. The membrane-driven DAC is a type of DAC in which a membrane (like a metal balloon) is attached to the piston side of the DAC, gas pressure is applied to inflate the membrane, and the piston is driven to pressurize the sample. In BL10XU, a single gas line pressure control system for increasing pressure has been installed; however, the following problems exist: (1) Conventional gas pressure control systems have not had a remote-control function and required the direct operation of a device installed near the hatch entrance when boosting pressure. (2) In addition, owing to the resistance between the DAC piston and cylinder, it was difficult to control pressure in decompression processes by only gas pressure leakage from a membrane. Therefore, it was difficult to study the phase equilibrium precisely in decompression. Also, in low-temperature experiments, the pressure is often observed to increase owing to the thermal contraction of the DAC. As mentioned above, it is almost impossible to decrease pressure precisely; therefore, users had to constantly monitor the sample pressure.

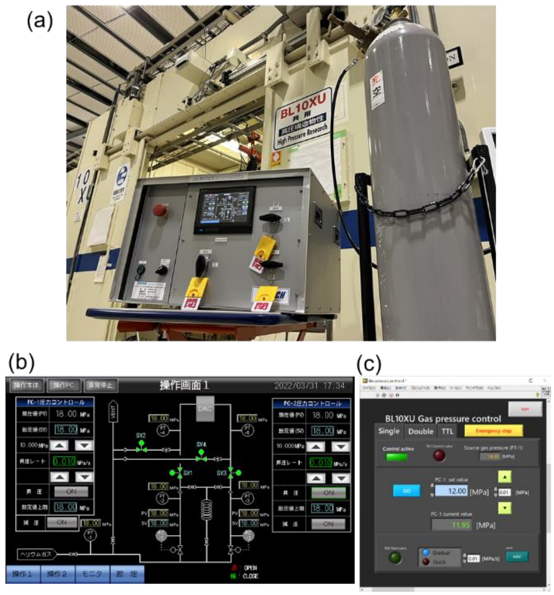

Fig. 1. (a) Developed two-line gas pressure control system, (b) touch panel on the main unit, and (c) control software based on LabVIEW.

To resolve these experimental difficulties, the two-line gas pressure control system has been developed in FY2021. Figure 1 shows a photograph of the exterior, the touch panel of the main unit, and the control software created based on LabVIEW. The gas pressure from the gas cylinder is reduced to the target pressure through the gas pressure regulator and the mass flow controller in the gas pressure controller, and it is supplied to the DAC at the specified flow rate value.

With this system, a maximum gas pressure of 18 MPa can be safely supplied. Both the pressure regulator and the exhaust section are electrically operated open/close valves, which can be remotely controlled by the control panel on the main unit and the user GUI from control PCs. The device is equipped with two control systems for applying gas pressure. With the combined new gas control system and a developed double-membrane-driven DAC, a DAC that enables depressurization control by pushing the cylinder side with a membrane can achieve not only conventional compression experiments but also precise decompression-control experiments. This new gas pressure controller can be easily synchronized with XRD measurement equipment via the main control system of BL10XU. In the future, we will establish a pressure feedback control feature using this device and prepare for the automation of compression and decompression experiments.

Furthermore, the new gas pressure controller has an open/close mechanism for each solenoid valve using TTL signals, enabling rapid pressurization and depressurization experiments. Recently, there has been a need for experiments under dynamic and extreme conditions, such as meteorite impacts, to explore the Earth itself and the origin of water and amino acids. Such dynamic conditions are also important to synthesize new novel materials. In FY 2020, we installed a sub-millisecond XRD measurement system with LAMBDA 750k (X-spectrum, DESY). We incorporated the new gas controller in the sub-millisecond XRD measurement system with LAMBDA 750k. Figure 2 shows the results of a rapid compression experiment with gold (Au) and sodium chloride (NaCl), which are widely used as pressure markers in high-pressure experiments using DACs. XRD 2D images were collected with focused X-rays at 30 keV of ~8 μm (H) × 8 μm (V) using an X-ray CRL lens, and the exposure time is 1 millisecond. A B1 to B2 phase transition of NaCl was observed about 1 s after the TTL signal. The analysis results show that rapid compression experiments can be performed at speeds of 100 GPa/s or higher. After further upgrading the control software, the system is scheduled to be used starting from 2022B period.

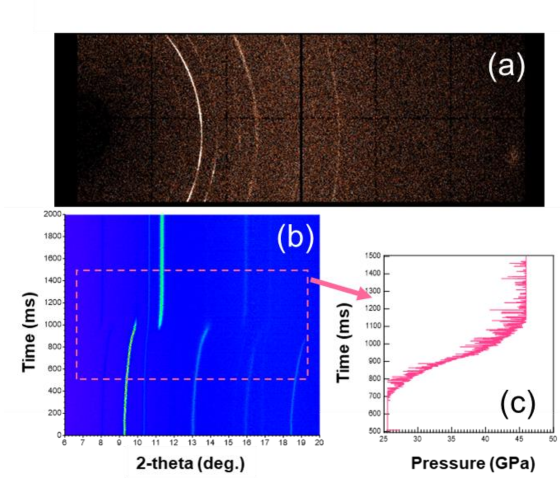

Fig. 2. Test results of a rapid compression experiment with gold and salt using the new gas pressure controller. (a) In situ XRD 2D image collected continuously on LAMBDA 750k at 1 kHz. (b) Time-resolved data of Au and NaCl under rapid compression. (c) Time dependence of sample pressures determined using *P*-*V* equation of state of Au.

Kawaguchi Saori and Kadobayashi Hirokazu
Powder Diffraction & Total Scattering Team, Diffraction and Scattering Division, Center for Synchrotron Radiation Research, JASRI