

BL04B1

High-Temperature and High-Pressure Research

1. Introduction

BL04B1 is designed for high-temperature and high-pressure experiments using a large-volume press and is mainly used in earth and planetary science and the synthesis of new materials. BL04B1 operates as a bending magnet beamline and offers the capability to conduct energy-dispersive X-ray diffraction measurements and X-ray radiography observations using white X-rays. The X-rays emitted from the bending magnet are directly introduced into the experimental hutch. White X-rays with a wide energy range up to 145 keV are utilized in measurements. This beamline is also equipped with a compact Si(111) double-crystal monochromator, which makes it possible to perform angle-dispersive X-ray diffraction measurements and X-ray radiographic observations using monochromatic X-rays with the photon energy between 30 and 60 keV. These high-energy X-rays allow us to conduct X-ray observations for samples surrounded totally by materials such as in high-pressure vessels.

The beamline has two experimental hutches (classified as optical hutches) operating in tandem. A large-volume press with a maximum load of 1500 tons is installed in each hutch. These large-volume presses make it possible to carry out high-pressure and high-temperature experiments. The SPEED-1500 Kawai-type high-pressure press with DIA-type guide blocks is installed in the upstream hutch, while the SPEED-Mk.II Kawai-type high-pressure press with D-DIA-type and D-111-type guide blocks is installed in the downstream hutch. SPEED-Mk.II has differential rams (D-RAM) inside, which move independently of the main ram, and we can conduct deformation experiments under high-pressure and high-temperature conditions. By utilizing the large-volume presses with high-energy X-rays, we can routinely carry out the *in situ* observations of materials under high-pressure and high-temperature conditions up to 100 GPa and 2500 K in the beamline.

In FY2023, we focused on improving the user interface, as well as addressing the aging of the measurement and heating systems.

2. Optical Hutch 2 (SPEED-1500): Improvement of control software

The incident slit with the He chamber was installed in FY2022 (see the FY2022 annual report for details). At that time, the stepping motor controller

was changed to PM16C-16, produced by TSUJI ELECTRONICS, which is widely used at SPring-8, so that it can be controlled by a general procedure. A four-quadrant slit with low risk of collision was used, and position and limit sensors were installed outside the He chamber to considerably reduce the effects of scattered X-ray damage. In postinstallation operation, the failure rate of position sensors and limit sensors was actually significantly reduced. Since stable operation was achieved, the control program for the high-pressure press stage, including this incident slit, was automated and sequential control was put into practical use.

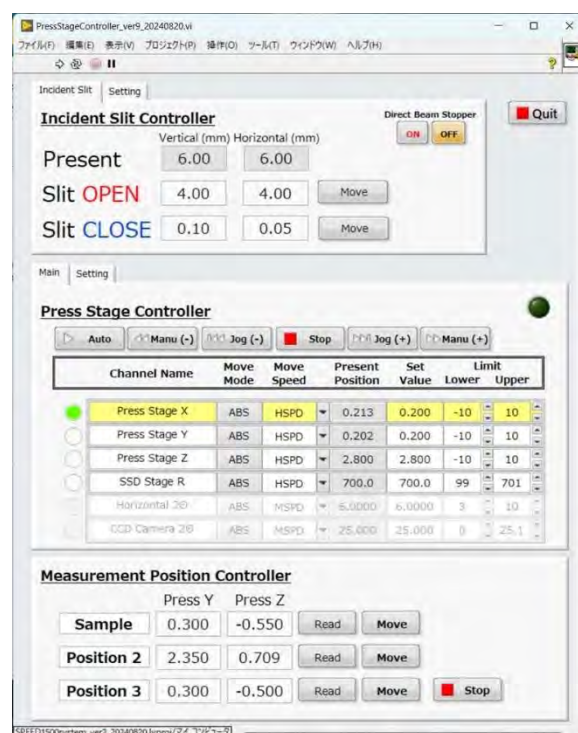


Fig. 1. Newly developed control software for incident slit and press stage at optical hutch 2, BL04B1.

Figure 1 shows the newly developed control software for the incidence slit and high-pressure press stage. This software allows the user to highlight only the axes needed during the experiment and to save up to three sample positions, preventing user error and improving the visibility of each axis. In addition, values of frequently used settings such as slit size for radiography imaging

(Wide) or XRD measurement (Small) can be saved and moved to the setting position with a single click.

Figure 2 shows the sequential control software that can automatically control the incident slit, high-pressure press stage, Ge-SSD, X-ray camera, and ultrasonic measurement system. This software allows the users to set the order of control and control values as desired. This software is particularly useful in ultrasonic velocity measurement experiments, where it is necessary to acquire XRD, image, and ultrasonic data quickly and without error under the same temperature and pressure conditions.



Fig. 2. Newly developed control software for incidence slit and press stage at optical hutch 2, BL04B1.

2. Optical Hutch 3 (SPEED-Mk.II): Improvement of heating control system

In recent years, large specimens for industrial applications and heating with large heaters for the precise determination of phase equilibrium boundaries have been frequently utilized. Experiments using such large heaters generally require poor thermal insulation and need a large power source for heating. In this upgrade, we installed an AC regulated power supply with a maximum power of 3 kW, which is 1.5 times the existing power supply (2 kW) (Figure 3).

On the other hand, heating systems using an AC power supply use a transformer to step down the voltage. However, because of aging, the transformer's leakage current had increased, resulting in a power loss of approximately 500 W even under no-load conditions. Figure 4 shows the relationship between the primary voltage applied without load and leakage current. It can be seen that the newly introduced transformer, shown in Fig. 3, significantly improves the leakage current. A switcher with the existing power supply was also installed and the software was updated. This

upgrade is expected to further stabilize the power control of high-resistance heaters.



Fig. 3. Newly installed transformer at optical hutch 3, BL04B1.

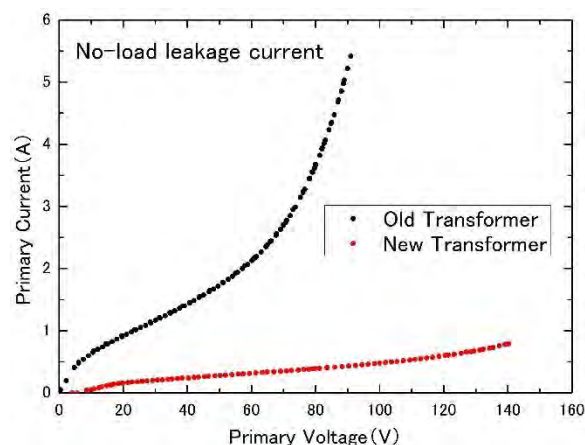


Fig. 4. Leakage currents of old and new transformers.

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