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HIGH-TEMPERATURE AND HIGH-PRESSURE EXPERIMENTAL STATION BL04B1 AT SPRING-8

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Most of the Earth's interior is a high-pressure, high-temperature environment, which makes high-pressure experimental techniques essential for studying Earth and planetary science. High-flux synchrotron radiation X-rays are important for in situ observations because the samples used in high-pressure experiments are generally very small (a few millimeters to a few microns), and X-ray absorption by the pressure medium and anvil is high.

The SPring-8/BL04B1 beamline uses white X-rays emitted from bending magnets as a light source, enabling energy-dispersive X-ray diffraction measurements and X-ray radiography observations. The beamline also features a compact Si(111) double-crystal monochromator, which enables angle-dispersive X-ray diffraction measurements using monochromatic X-rays of 30–60 keV and monochromatic X-ray radiographic observations. The SPEED-1500 Kawai-type high-pressure apparatus (DIA-type block, optical hatch 2) and SPEED-Mk.II Kawai-type high-pressure apparatus (D-DIA-type mold, optical hatch 3) are installed, both with a maximum load of 1500 tons (Fig. 1). The SPEED-Mk.II can also be used for high-pressure and high-temperature experiments above 30 GPa using sintered diamond anvils and high-pressure deformation experiments using the D-RAM (differential ram that can move independently of the main ram). For deformation experiments, it is necessary to obtain Debye rings all along the azimuth angle to measure deviatoric stresses. For this purpose, BL04B1 is normally equipped with a large-area CCD detector (ϕ200 mm, SX200, Rayonix), and a high-resolution X-ray beam monitor enables direct observation of minute sample deformations through X-ray absorption images.

An example of a high-pressure experiment conducted at BL04B1 is the development of an elastic wave velocity measurement technique under very high pressure and temperature conditions. The lower mantle occupies about 60% of the Earth's total volume and is the primary arena for mantle dynamics, such as mantle convection and mantle plumes. However, the chemical composition and thermoelastic properties of lower-mantle minerals are not fully understood, and the driving forces of mantle convection and plumes remain unknown. Therefore, ultrasonic techniques have been improved to measure elastic wave velocities of lower-mantle minerals under high pressure and temperature conditions up to about 30 GPa and 1600 °C, which correspond to the upper part of the lower mantle.

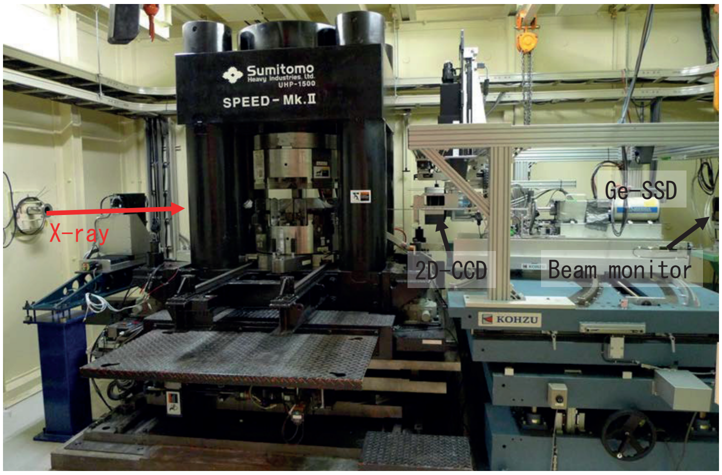

Figure 1 The D-DIA type multi-anvil high pressure apparatus SPEED-Mk.II installed at BL04B1 and its detectors. Synchrotron radiation X-rays enter from the left side, pass through the sample in the high-pressure cell, and X-ray diffraction and X-ray transmission images are acquired by the detector on the right side.

High-pressure and high-temperature experiments were performed using a SPEED-1500 Kawai-type multi-anvil apparatus. A set of eight TF-05 carbide anvils (Fuji Dies Co., Ltd.) with a 3-mm truncated length was used as the second stage anvil. TiB_2+BN, which has good X-ray transparency, was used as the heater material. The pulse echo overlap method was used for elastic wave velocity measurement. A $LiNbO_3$ ultrasonic resonator attached to the second-stage anvil was driven by a 40–90 MHz high-frequency signal to generate ultrasonic waves, and the reflected echoes from the sample were observed using a digital oscilloscope. To obtain ultrasonic echoes from samples smaller than 1 mm, an input signal amplifier and an input–output signal isolator were newly developed and installed. The sample length was directly determined under high-temperature and high-pressure conditions using X-ray radiography images acquired by a high-resolution CCD camera. The generated pressure was determined by lattice constant determination from the X-ray diffraction pattern of the pressure reference material (MgO) by the energy dispersion method, and the pressure was calculated using the equation of state.

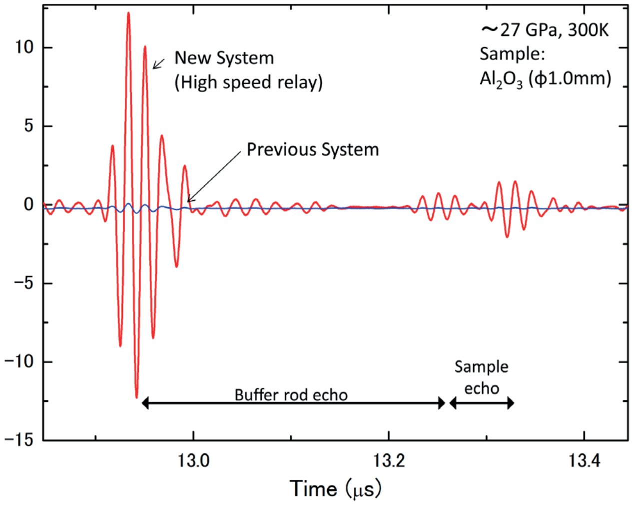

Figure 2 Waveform of ultrasonic echoes of alumina samples under high pressure.

The experimental results demonstrated that the ultrasonic echoes obtained from the new system were considerably stronger than those from the previous system and that they were sufficiently strong for analysis of even small samples (Fig. 2). Using this system, elastic wave velocities of Al_2O_3 were successfully measured over a wide range of temperatures and pressures, and thermoelastic parameters were accurately determined (Fig. 3). The bulk modulus (K_{0S}) and shear modulus (G) along with their dependence on pressure (P) and temperature (T) were estimated as K_{0S} = 251.2 (18) GPa, $\partial K_S/\partial P$ = 4.21 (10), $\partial K_S/\partial T$ = −0.025 (1), G = 164.1 (7), $\partial G/\partial P$ = 1.59 (3), $\partial G/\partial T$ = −0.021 (1). These values were found to be in good agreement with the previous experimental results obtained at low pressure (Higo et al. 2018).

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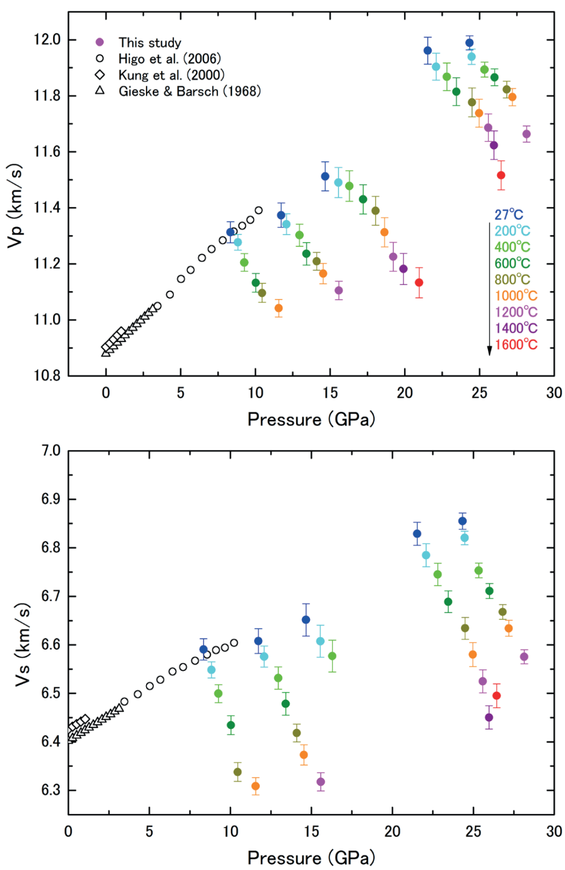

FIGURE 3 Elastic wave velocities of alumina samples under very high pressure and temperature. **TOP**: longitudinal wave, **BOTTOM**: transverse wave.

REFERENCES

Higo Y, Irifune T, Funakoshi K (2018) Simultaneous high-pressure high-temperature elastic velocity measurement system up to 27 GPa and 1873 K using ultrasonic and synchrotron X-ray techniques. Review of Scientific Instruments 89: 014501, doi: 10.1063/1.4993121

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