BL04B1 High Pressure and High Temperature

The BL04B1, “High Pressure and High Temperature” is a white X-ray beamline from a bending magnet. The use of high energy (20~150 keV) is suitable for energy-dispersive diffraction experiment using a solid state detector (SSD) and imaging experiment using a CCD camera. This beamline is designed to conduct research on the structure and properties of materials in the Earth's interior under high-pressure and high-temperature conditions. High-pressure and high-temperature experiments are performed using two large-volume presses: SPEED-1500 and SPEED-Mk.II, both of which are installed in the tandem experimental stations (Experimental hutch 1, hutch 2). These large-volume presses are constructed by double-stage (Kawai-type) high-pressure vessels with two kinds of the anvils; the tungsten carbide (WC) and the sintered diamond (SD). In the present system, 30 GPa or 50 GPa has been reached with the WC (26 × 26 × 26 mm^3) or the SD (14 × 14 × 14 mm^3) over 2500 K, and each high-pressure and high-temperature condition is corresponding to the upper or the lower mantle region in the Earth.

Area of research

Determination of phase relation, Equation of state of mantle, Viscosity of melts, Kinetics of mineral transformation, Rheology of mantle minerals, Structure of melts and glasses at high pressures

Keywords

Scientific field

High-pressure, Geophysics

Equipment

Large volume press, Energy-dispersive X-ray diffractometer, High-speed CCD camera imaging system

Source and optics

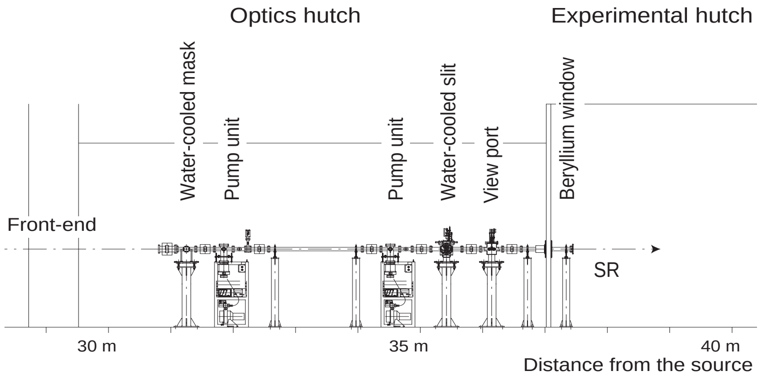
X-rays at sample

Energy range	20 ~ 150 keV (white)
Beam size	0.05 × 0.05 ~ 10 × 10 mm^2
Beam divergence (V)	< 0.05 mrad
Beam divergence (H)	< 0.5 mrad

Experimental stations

High pressure mineral physics (Experimental hutch 1, hutch 2)

The BL04B1 has one optics hutch, and two experimental hutches. Two experimental hutches have been built in tandem configuration, both of which are dedicated to the large presses: Experimental hutch 1; the SPEED-1500, and Experimental hutch 2; SPEED-Mk.II. The layout of the BL04B1 is shown in Fig. 1. The BL04B1 has no monochromators or focusing mirrors, and thus only white X-rays are available over the energy range of 20-150 keV. Each experimental hutch has a horizontal goniometer for the energy-dispersive X-ray diffraction using white X-rays. These goniometers are attached to the large volume high-pressure apparatuses, the SPEED-1500 and SPEED-Mk.II, and thus *in situ* X-ray diffraction experiments can be carried out at high pressure and high temperature conditions.



Schematic view of beamline

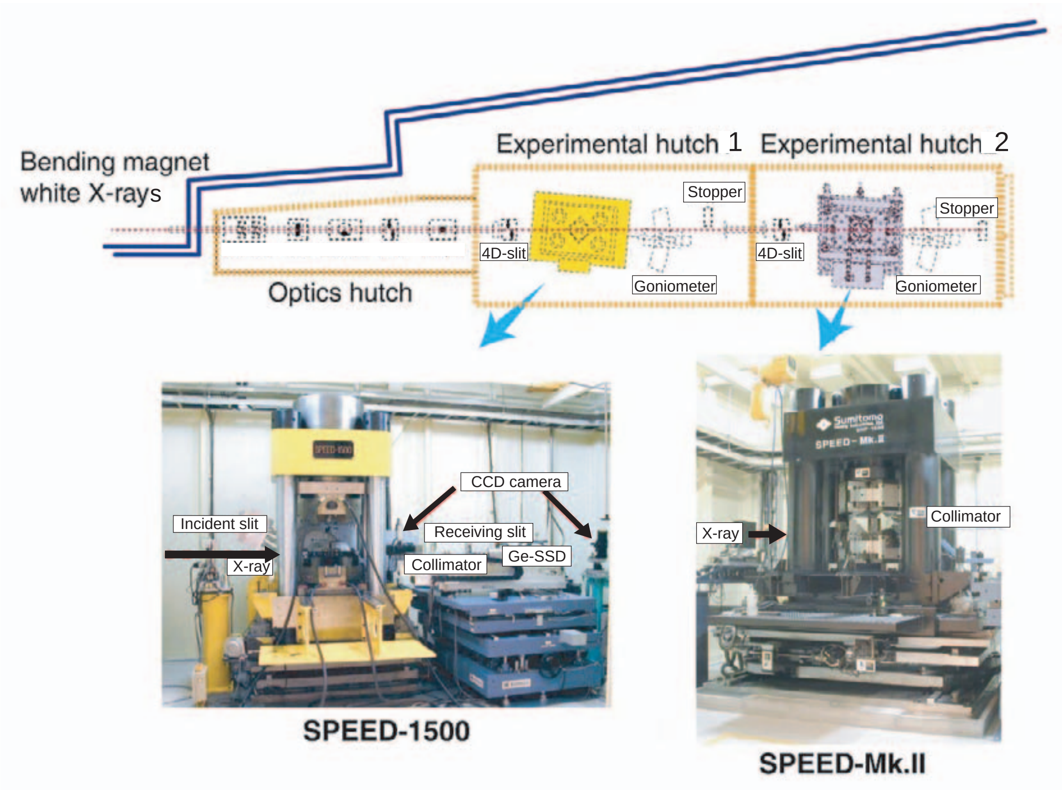


Fig. 1. Layout of BL04B1 (SPEED-1500 and SPEED-Mk.II)

The two large-volume multi-anvil presses, SPEED-1500 and SPEED-Mk.II, are double-stage type (Kawai-type) compression with 1500-ton ram force. The outside first-stage consists of six cubic type anvils in the [100] axis direction. The inside second-stage consists of eight cubic anvils, each of which has one corner truncated into a triangular face. The eight faces form an octahedral cavity. The eight second-stage anvils that are compressed by the six first-stage anvils compress an octahedral shaped pressure medium (Kawai-type high pressure vessel: Fig. 2). In the present system, 26 × 26 × 26 mm^3 tungsten carbide (WC) or 14 × 14 × 14 mm^3 sintered diamond (SD) anvils can be used as the second anvils (Fig. 3). Each generated maximum pressure is about 30 GPa with the WC anvils and 50 GPa with the SD anvils (Fig. 4).

In order to carry out the energy-dispersive X-ray diffraction on the Kawai-type system, holes are cut in the first-stage anvils to allow the X-ray beam to pass through. The incident white X-ray beam from the first-stage passes through the gaps between the second-stage anvils in a horizontal plane (Fig. 5). The horizontal diffractometer covers a range of 2θ angles between -7 and 23°. The incident beam can be as low as 50 × 50 μm^2 and the diffracted X-rays are detected by Ge-SSD.

On the other hand, CCD camera has been installed on the both of large-volume presses for X-ray radiography measurements. The incident white beam irradiates the sample cell through the anvil gap, and an image is projected on the fluorescence screen (YAG crystal). This image is then magnified and detected by a high-speed CCD camera (Fig. 6). Real-time images are captured and recorded at very short time intervals.

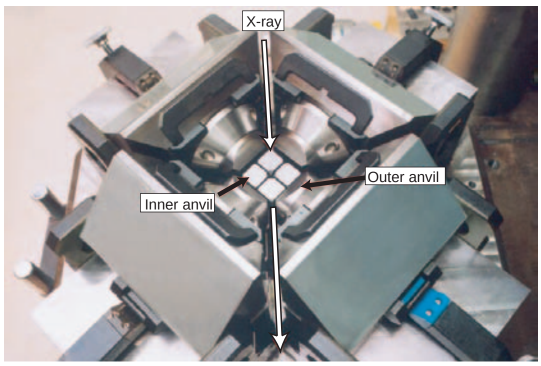


Fig. 2. Kawai-type high-pressure vessel ([100] direction)

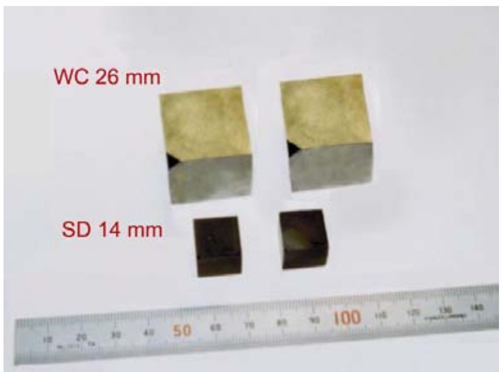


Fig. 3. Inner anvils of WC and SD cubes

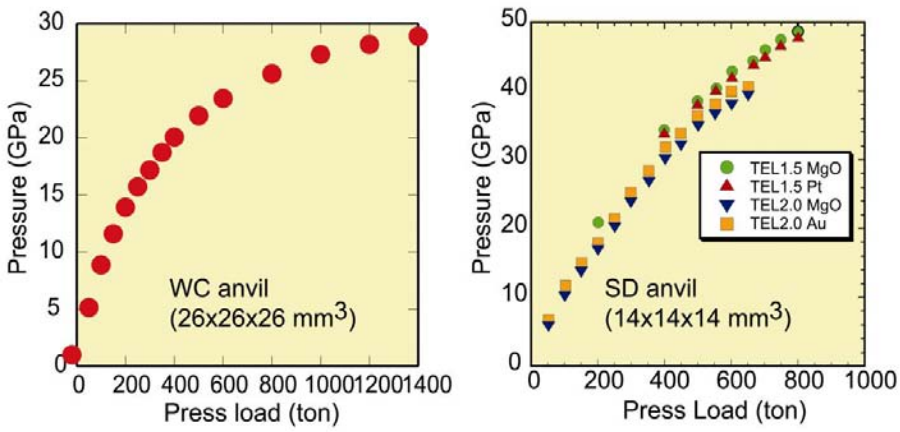


Fig. 4. Comparisons of the pressure generations using WC and SD anvils

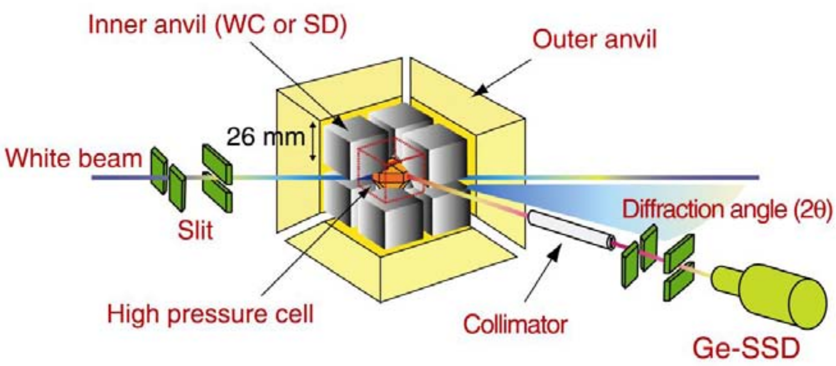


Fig. 5. Energy-dispersive X-ray diffraction measurement

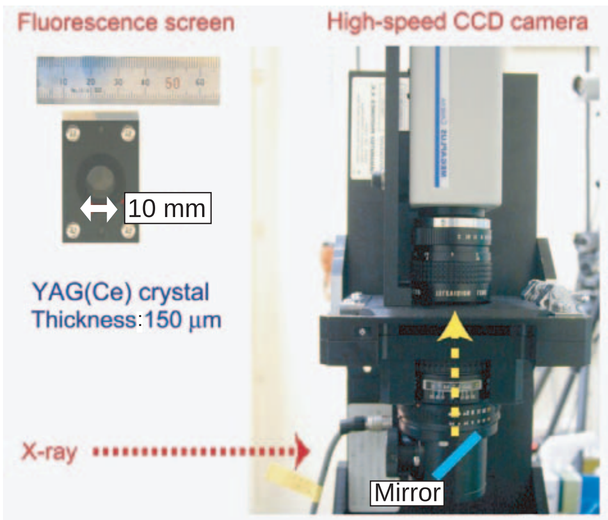


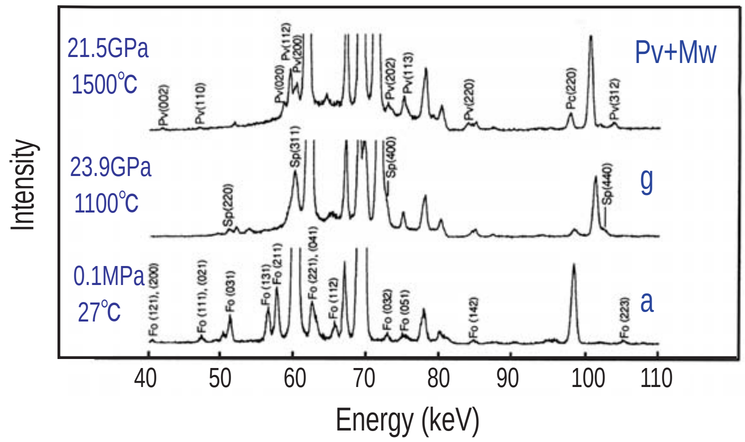
Fig. 6. High-speed CCD camera system for X-ray radiography

Examples of results

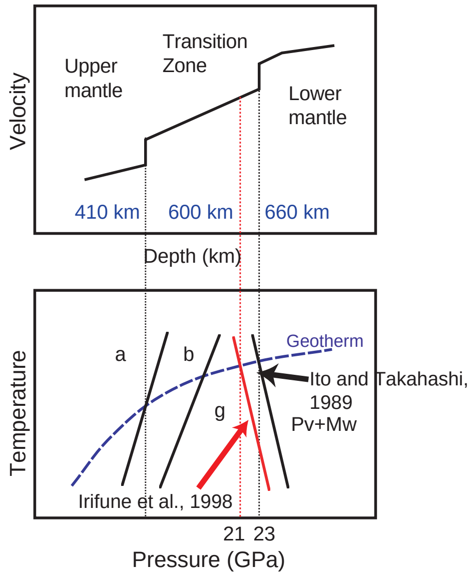
The BL04B1 has been widely used in studying the structures and properties of minerals corresponding to the mantle region in the Earth's interior. *In situ* X-ray diffraction experiments have been made for precise determination of the phase boundary or the equation of state of the important minerals in the mantle. As the first results of in situ X-ray diffraction measurements, the postspinel transition in Mg_2SiO_4 olivine was clearly observed and its phase boundary was determined at a near depth of 660 km discontinuity in the Earth's mantle (Fig. 7). In addition to the diffraction study, the X-ray radiography technique has been applied to the measurements of the viscosity of melts. The viscosity experiments using the "falling sphere method" have been successfully made and the precise viscosities of magmas, outer core, or some important melts have been determined as a function of pressure (Fig. 8). Utilizing the diffraction and radiography techniques, following studies have been conducted with mantle minerals and melts;

- Determination of phase relation, equation of state (EOS) and crystal structure,
- Structure of melts and glasses,
- Kinetics of the phase transition,
- Viscosity
- Rheology

These studies will provide new significant information into the current status, origin, and evolution of the Earth.

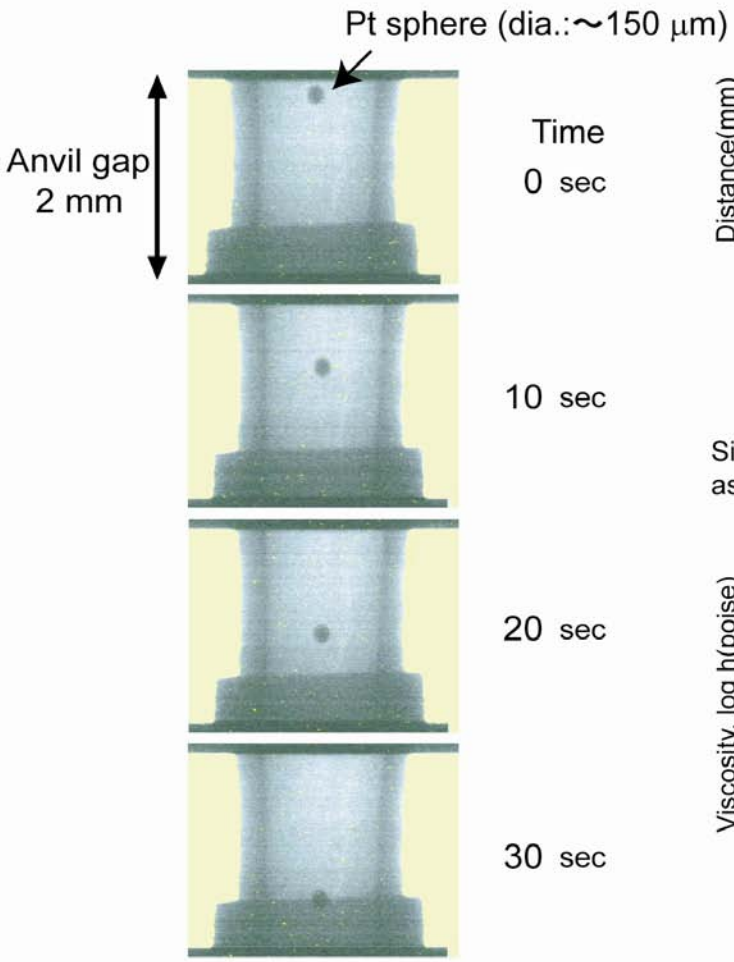


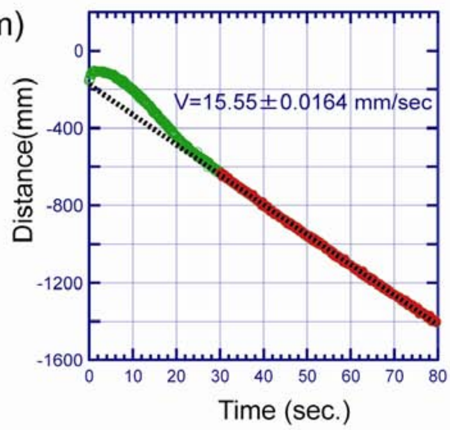
X-ray diffraction patterns of Mg_2SiO_4 olivine at various P/T conditions.



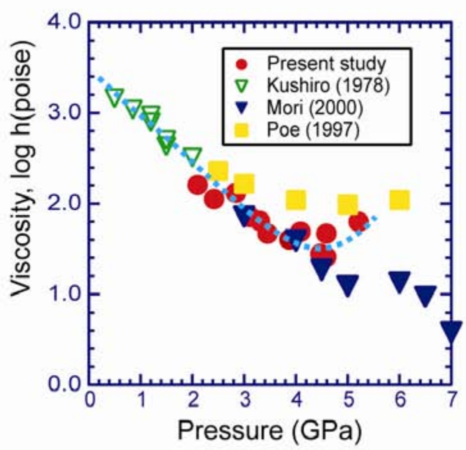
Phase boundary between the spinel and the post spinel phases.

Fig. 7. The phase boundary of Mg_2SiO_4 olivine determined by *in situ* X-ray diffraction measurements. (*T. Irifune et al., Science, 279, 1698 (1998)*.)





Sinking distance of a Pt sphere in albite melt as a function of time (2.8 GPa, 1700℃).



Real-time images of Pt sphere sinking in albite ($NaAlSi_3O_8$) melt detected by a CCD camera.

A comparison of the pressure dependence of the viscosity of albite melt.

Fig. 8. Viscosity measurements of albite ($NaAlSi_3O_8$) melt using an X-ray radiography falling sphere method. (*K. Funakoshi et al., J. Phys. Condens. Matter., 14, 11343 (2002)*.)

Facilities

Large-volume press (LVP)

SPEED-1500

- Maximum load: 1500 ton ram force
- High-pressure vessel: Kawai-type ([100] direction)
- Inner anvil material: Tungsten carbide (WC), Cube size: $26 \times 26 \times 26$ mm^3
- Pressure & Temperature: 30 GPa, 2500 K
- Measurements: Energy-dispersive XRD (horizontal goniometer 2θ: -7 ~ 23 degree), High-speed CCD imaging (Maximum image size: 4×4 mm^2), Ultra high-speed CCD camera (Maximum image size: 2×2 mm^2)
- Sample: Powder

SPEED-Mk.II

- Maximum load: 1500 ton ram force
- High-pressure vessel: Kawai-type ([100] direction)
- Inner anvil material: Sintered diamond (SD), Cube size: $14 \times 14 \times 14$ mm^3
- Pressure & Temperature: 50 GPa, 2500 K
- Measurements: Energy-dispersive XRD (horizontal goniometer 2θ: -7 ~ 23 degree), High-speed CCD imaging (Maximum image size: 4×4 mm^2)
- Sample: Powder

Detector

- Pure Ge solid state detector (active area of 1 cm^2 and thickness 10 mm)
- Ion chamber
- High-speed CCD camera (Size: 640 × 480 pixels, Resolution: 7 μm/pixels, Recording time: 30 frames/sec)
- Ultra high-speed CCD camera (Size: 640 × 480 pixels, Resolution: 4 μm/pixels, Recording time: 125 frames/sec)

Applications

- Determination of phase relation
- Equation of state of mantle
- Viscosity of melts
- Kinetics of mineral transformation
- Rheology of mantle minerals
- Structure of melts and glasses

Contact information

Kenichi FUNAKOSHI
SPring-8 / JASRI
1-1-1 Kouto, Mikazuki-cho, Sayo-gun, Hyogo 679-5198
Phone : +81-(0)791-58-0832
Fax : +81-(0)791-58-0830
e-mail : funakosi@spring8.or.jp