

High Temperature and High Pressure Research Beamline, BL04B1

The BL04B1, “High Temperature and High Pressure Research Beamline,” is a beamline for Earth and materials sciences equipped with large multi-anvil devices. The white X-rays in a high-energy range (~20 - 150 keV) are available for energy-dispersive X-ray diffraction experiments using a solid-state detector (SSD) and for X-ray radiography experiments using a CCD camera. This beamline is designed particularly for conducting researches on the precise in situ X-ray measurements of the crystal structures and physical properties of minerals under high pressure and high temperature conditions. High-pressure and high-temperature experiments are performed using two large multi-anvil devices: SPEED-1500 and SPEED-Mk.II (Fig. 1), both of which are installed in tandem at the experimental stations [1]. These devices operate double-stage (Kawai-type) high-pressure vessels with two types of the second-stage anvils: cubic tungsten carbide (WC) and sintered diamond (SD) anvils (Fig. 2). Pressures up to 30 GPa or 60 GPa have been achieved with the WC (26 x 26 x 26 mm³) or SD (14 x 14 x 14 mm³) anvils above 2500 K, respectively, which cover the depths of more than 1500 km in the Earth’s interior.

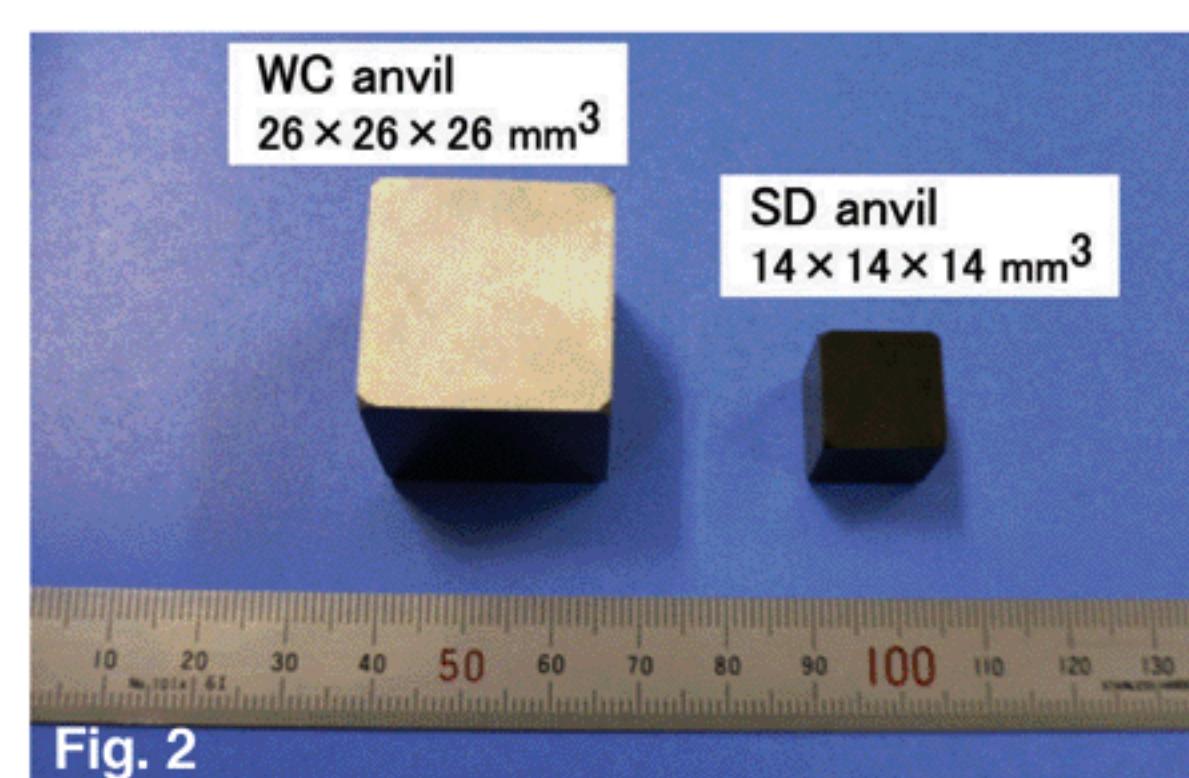
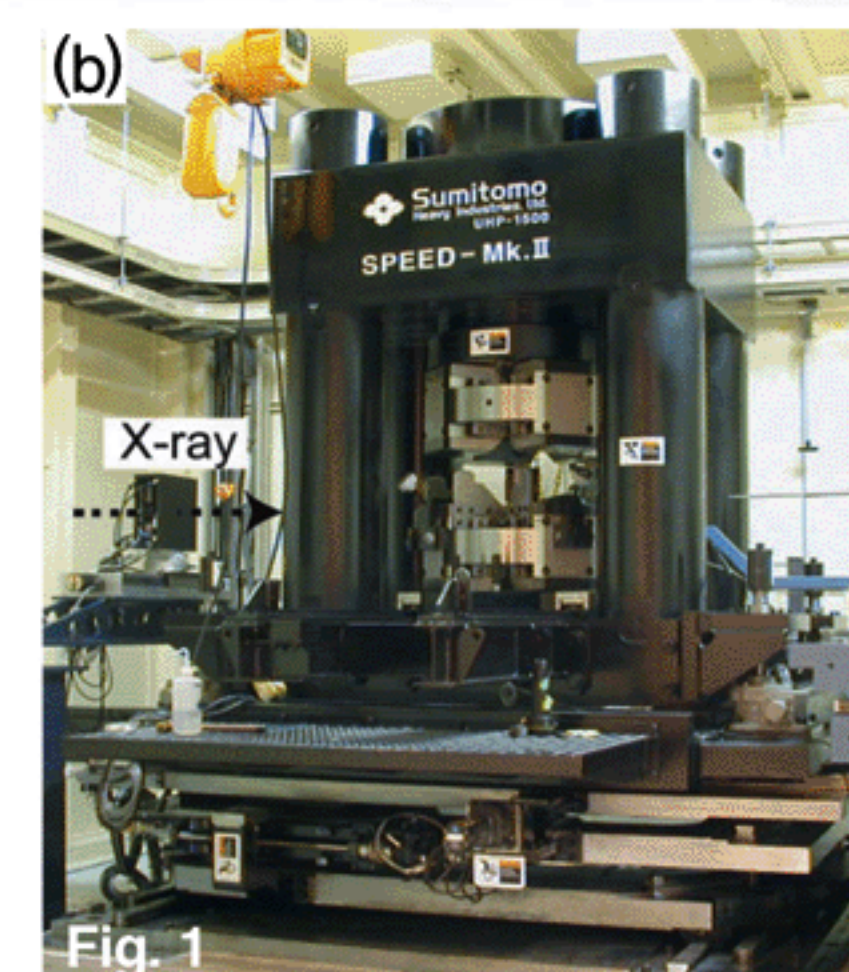
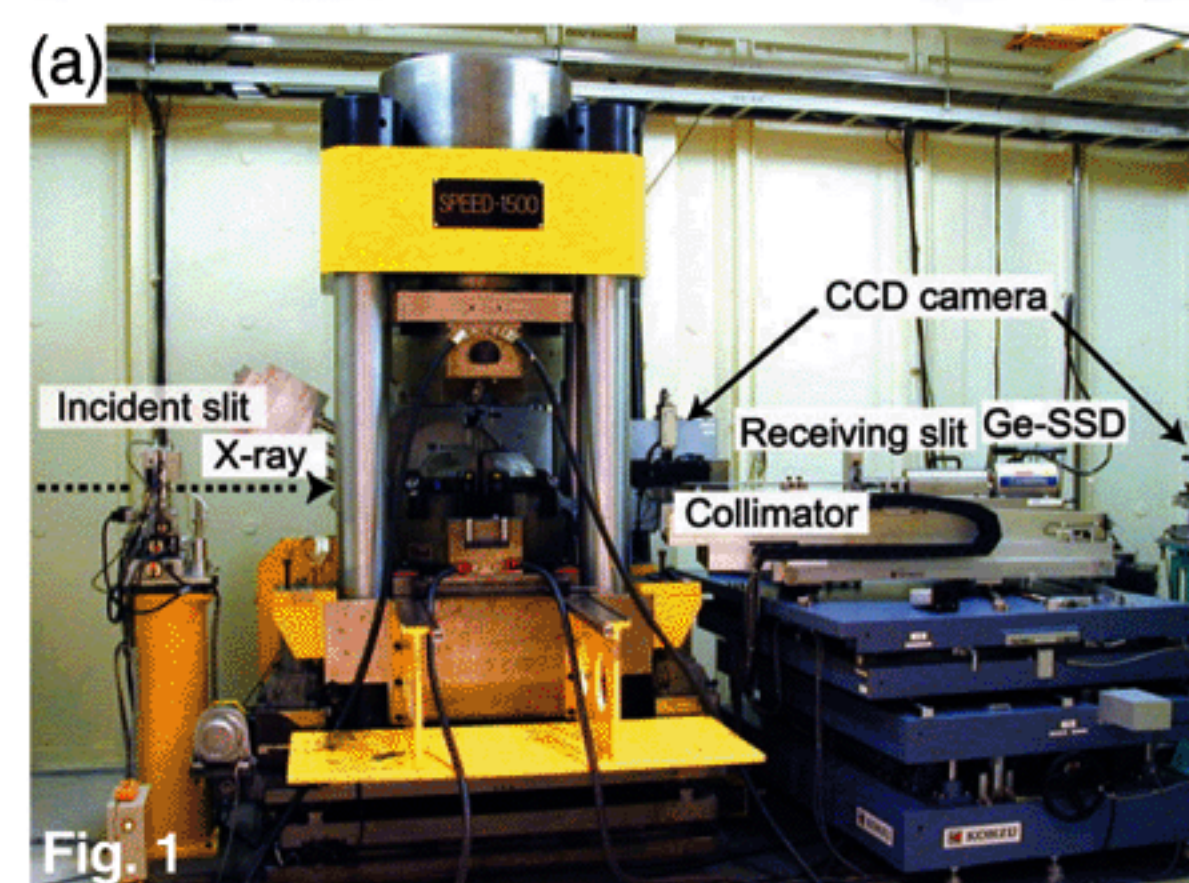


Fig. 1 Photographs of large multi-anvil devices installed at BL04B1: (a) SPEED-1500, (b) SPEED-Mk.II.

Fig. 2 Second stage anvils: tungsten carbide (WC: 26 x 26 x 26 mm³) and sintered diamond (SD: 14 x 14 x 14 mm³) cubes.

An example of high pressure studies with sintered diamond anvils: wurtzite-rocksalt transformation in GaN

The pressures produced in Kawai-type multi-anvil devices have been generally limited to ~30 GPa because of the plastic deformation of tungsten carbide (WC) anvils used for second-stage anvils. Thus, sintered diamond (SD) anvils, which are much harder than these conventional anvils, were introduced to overcome this limit. A Kawai-type multi-anvil device at Spring-8, SPEED-Mk.II, was custom-designed for this purpose [2]. By developing the techniques using a combination of synchrotron radiation and the SD anvils (14 x 14 x 14 mm³ cube), pressures higher than 60 GPa have been successfully generated at room temperature (Fig. 3). An in situ X-ray diffraction study on the phase transitions in GaN was performed using this technique [3], and the onset of wurtzite-rocksalt transformation in this material was observed at 54 GPa and 300 K and at 51.4 GPa and 750 K, suggesting a negative Clapeyron slope of -170 K/GPa (Fig. 4). A rocksalt-type GaN was found to be quenchable to the ambient conditions. It is suggested that a noticeable change in electric resistance may not be accompanied by the wurtzite-rocksalt transformation. Moreover, the sluggishness of the reaction observed in the present study also suggests that this phase transformation in GaN is unsuitable for being used as a pressure reference point.

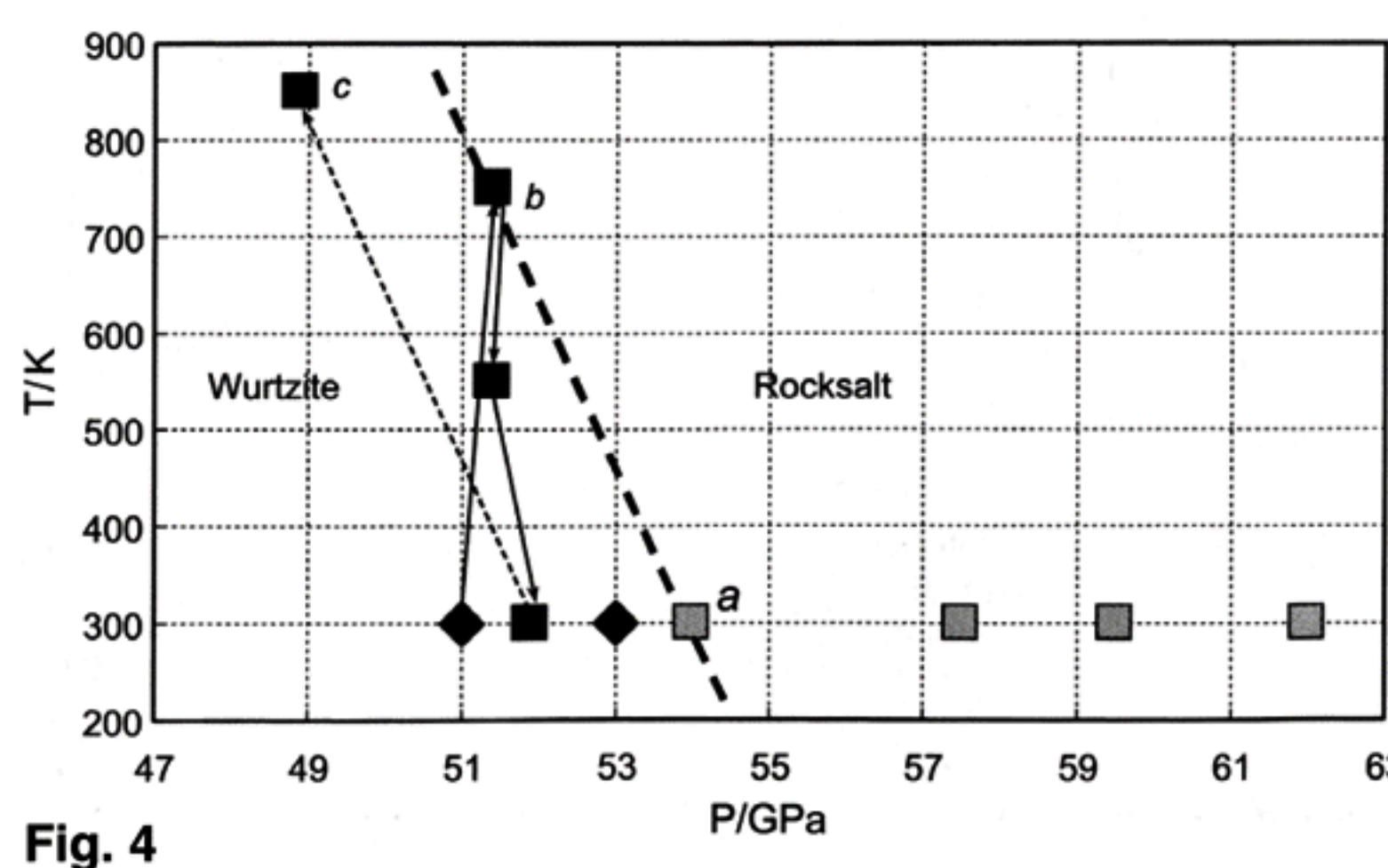
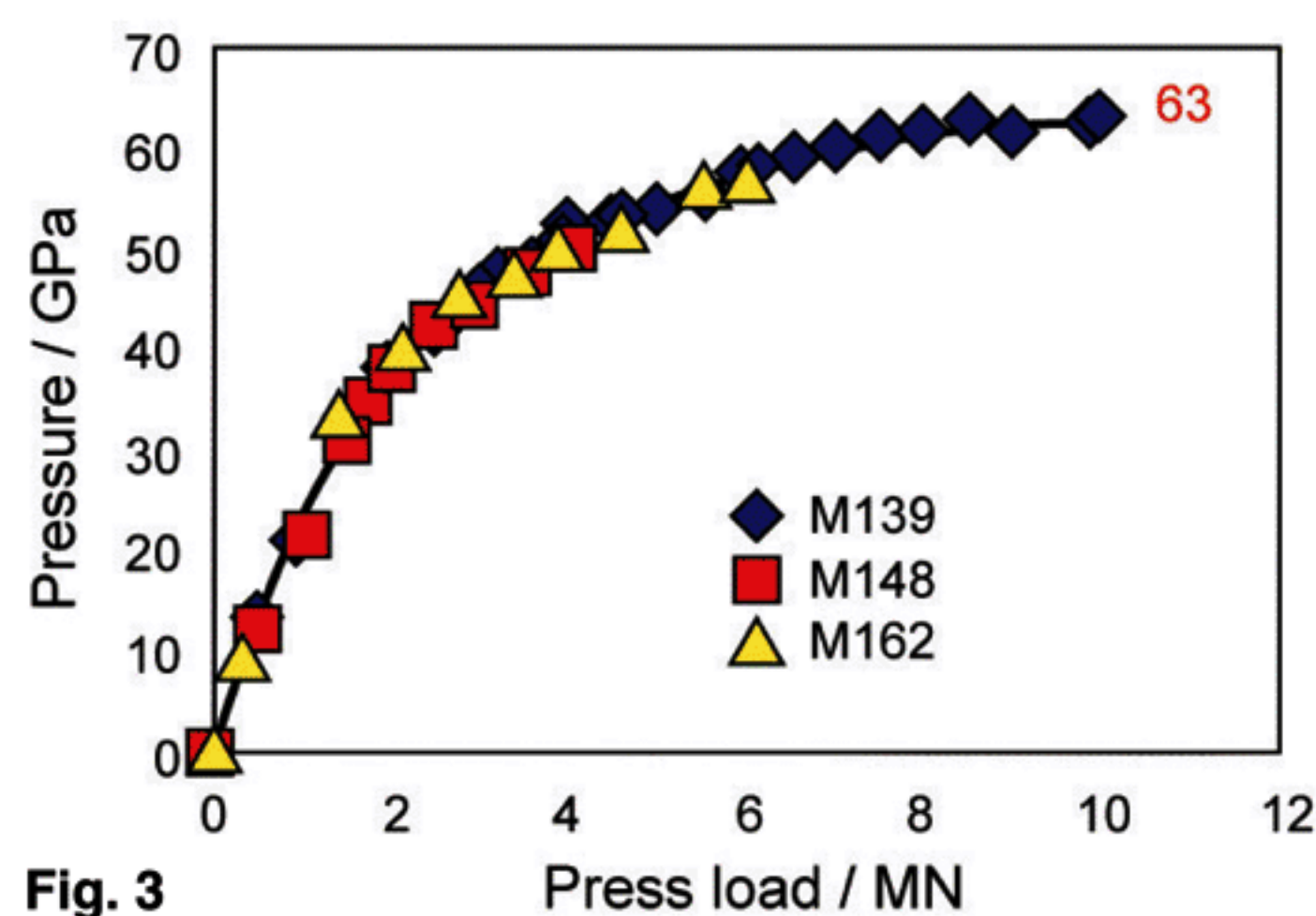


Fig. 3 Examples of pressure generation using SD anvils (14 x 14 x 14 mm³ cube) with truncated edge length of 1.5 mm at room temperature. The generated pressures are determined from the MgO scale (Jamieson et al., 1982).

Fig. 4 Phase transitions in GaN determined using in situ X-ray observations with SD anvils [3]. a and b denote the conditions of the onsets of wurtzite-rocksalt transformation at 300 and 750 K, respectively. The solid diamond, solid square, and gray square show the P, T conditions where wurtzite type, rocksalt type, and composite of wurtzite and rocksalt type are formed, respectively. The dashed line is a tentative phase boundary.

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