Generation of pressure over 1 Mbar in the Kawai-type multianvil apparatus

High-pressure experiments are one of the most useful methods for studying the earth's interior, meaning that the generation of pressure is of essential importance. In earth science, the Kawai-type multianvil apparatus has been widely employed in the clarification of the structure and state of the deep Earth (cf. [1]). The most marked advantage of the Kawai-type multianvil apparatus is the capability to compress a large volume sample (>1 mm^3) in an octahedral pressure medium under a quasi-hydrostatic environment through compression with eight cubic anvils. Moreover, by adopting an internal heating system, the sample can be heated homogeneously and stably, which make it possible to obtain qualitative physical and chemical properties of minerals under high-pressure and -temperature conditions. In the conventional Kawai-type multianvil apparatus, in which tungsten carbide (WC) is used as second-stage anvils, however, the attainable pressure has been limited to ~28 GPa. In the late 1980s, sintered diamond, which is two times harder than WC, became available for the second-stage anvils of the Kawai-type multianvil apparatus instead of WC [2]. In the early 1990s, therefore, the experimental pressure was raised to slightly higher than 30 GPa by using cubic sintered diamond anvils with an edge length of ~10 mm. In 1998, sintered diamond cubes with an edge length of 14 mm were introduced, which made it possible to squeeze the Kawai-cell of eight cubic anvil assembly at a load two times higher than when using 10 mm edge cubes. Through the use of 14-mm-edge-length sintered diamond anvils and the optimization of the pressure medium and the gasket, the attainable pressure has been expanded to higher than 40 GPa (e.g., [3]). As shown in Fig. 1, the pressure range has been significantly increased since 2004. The discovery of post-perovskite phase strongly motivates our technique of pressure generation using the Kawai-type multianvil apparatus.

The high-pressure and high-temperature *in situ* X-ray diffraction experiment was conducted at the white X-ray beamline **BL04B1** to test the pressure generation. The Kawai-type high-pressure cell composed of eight sintered diamond cubic anvils with an edge length of 14 mm and truncated edge length (TEL) of 1.0 mm was set in the DIA-type press (SPEED-Mk.II/Madonna). Two types of sintered diamond anvils were tested: one is the "C-grade" sintered diamond anvil with 8% Co as a binder and the other is the "C2-grade" sintered diamond anvil with 7.5% Co as a binder (both sintered diamond anvils are supplied by Sumitomo Electric Industries, Ltd.). The strength of the C2-grade anvil is slightly higher than that of the C-grade. The fracture strength (TRS) and Young's modulus of C2-grade anvil are reported to be 1.25 GPa and 941 GPa, respectively, whereas those of the C-grade anvil are 1.15 GPa and 893 GPa, respectively (informed from Sumitomo Electric Industries, Ltd.). As illustrated in Fig. 2, a cylindrical TiB_2+BN+AlN heater was placed in the octahedral pressure medium of MgO+5% Cr_2O_3 with an edge length of 4.1 mm. Pressure was determined from the volume of Au mixed into the sample, using the equation of state for Au [3].

Pressures up to ~110 GPa were successfully generated using the cell assembly shown in Fig. 2 with the C2-grade anvils. The attainable *P-T* range of the Kawai-type apparatus has been extended in the present study, which is ~15 GPa higher than

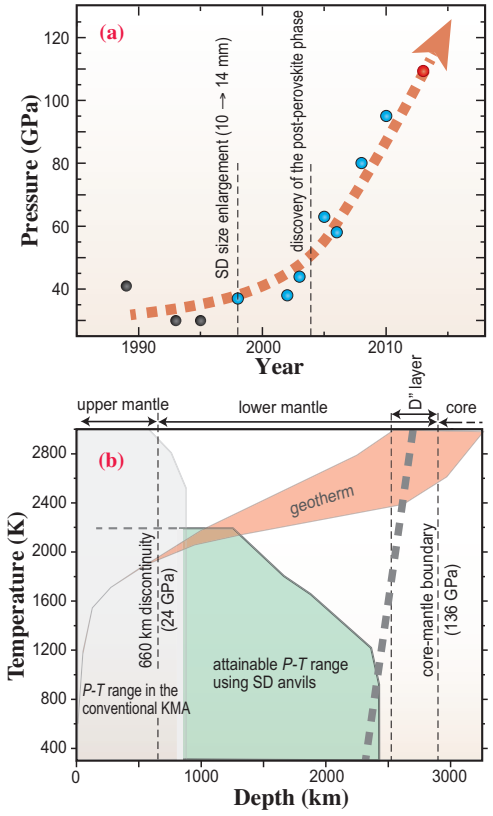

Fig. 1. **(a)** Evolution of the attainable pressure in the Kawai-type multianvil apparatus using sintered diamond for second-stage anvil (thick dotted line). **(b)** Depth-temperature diagram showing the attainable pressure and temperature range of the Kawai-type multianvil apparatus, together with the phase boundary between perovskite and post-perovskite in $MgSiO_3$ (thick solid line).

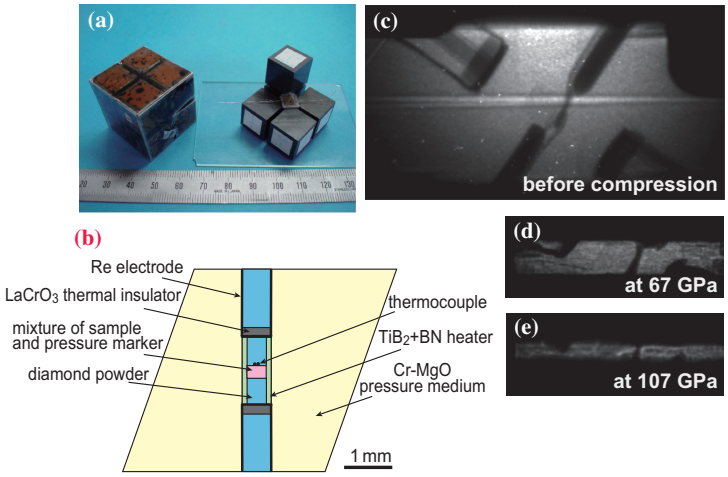

Fig. 2. **(a)** Kawai-type high-pressure cell comprising of eight sintered diamond cubic anvils. **(b)** Schematic illustration of furnace assemblies used in the present study. **(c)-(e)** X-ray radiographs showing the inside of cell assembly through anvil gap before compression, at 67 GPa, and at 107 GPa, respectively.

in the previous study, as shown in Fig. 1. This pressure range is now close to those of the D" layer (Fig. 1). In order to generate higher pressure, high-quality sintered diamond is of essential importance. In addition, enlargement of the sintered diamond anvil size is demanded because a load more higher press than 10 MN should be applied in the present cell assembly. At present, the maximum press load for a 14 mm cube is limited to ~10 MN owing to the limitation of the strength of WC of the first-stage anvils. Another key point may be further optimization of the cell assembly. For example, Tange *et al.* [4] showed that the use of Al_2O_3 as a pressure medium generates pressure more effectively than when using the Cr-doped MgO adopted in the present study.

Stishovite was compressed in the pressure generation test. During compression, the second-order structural transition from stishovite ($P4_2/mmm$) to the $CaCl_2$-type ($Pnnm$) phase was observed. The transition is shown in the diffraction patterns in Fig. 3. A peak assigned to be (211) line of stishovite split into a doublet with peaks assigned to the (121) and (211) lines of the $CaCl_2$-type phase. The splitting, however, was not clear at pressures around 55 GPa. Assuming the ambiguity is due to the coexistence of both phases, the transition pressure is estimated to be ~55 GPa. From the change in the axial ratios of stishovite to the $CaCl_2$-type phase, the transition pressure is also estimated to be ~55 GPa. The transition boundary in this study is consistent with the results previous studies (e.g., [5]).

In SiO_2, the α-PbO_2 structure (*Pbcn or Pnc2*) was reported as the next high-pressure polymorph of the $CaCl_2$-type. Murakami *et al.* [6] showed the stability of the α-PbO_2-type phase to be higher than ~100 GPa, and determined the phase boundary to be P (GPa) = 98 + (0.0095 ± 0.0016) × T (K). In the compression experiment on stishovite, the sample was annealed at 109.0 GP and 900 K for 15 min. Although the annealing pressure was 2.5 GPa higher than that at the phase boundary proposed in the previous study, no sign of transition to the α-PbO_2 type was observed. This indicates that the annealing temperature and duration are not sufficient to promote the phase transition because of slow transition kinetics, or the real phase boundary is higher than that reported in the previous study.

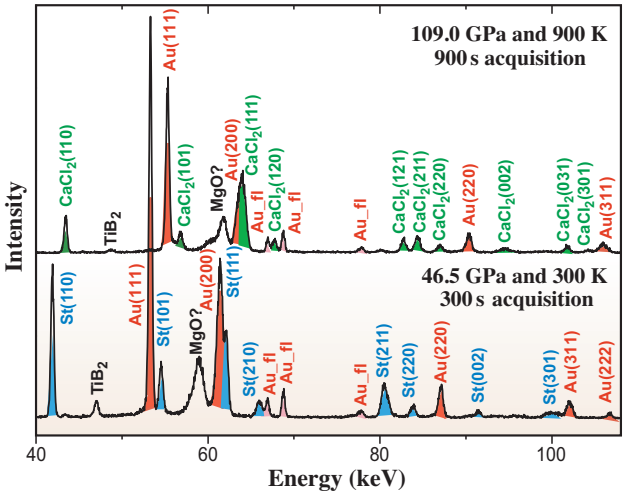

Fig. 3. Representative diffraction profiles for stishovite (st) at 46.5 GPa and 300 K (bottom profile) and the $CaCl_2$-type phase ($CaCl_2$) at 109 GPa and 900 K (upper profile).

Daisuke Yamazaki

Institute for Study of the Earth's Interior,
Okayama University

E-mail: dy@misasa.okayama-u.ac.jp

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