

## Mineralogy of the Lower Mantle Investigated Using Sintered Diamond Multianvil Apparatus

The Kawai-type, in other words, 6-8 double-staged, multianvil apparatus (KMA) is one of the most popular experimental devices, which can reproduce highpressure and high-temperature conditions relevant to the Earth's deep interior. KMA has the advantage of generating stable high P-T conditions with high reproducibility and large sample volume. Because of these characteristics, KMA has played an important role in the high-pressure Earth sciences, particularly in understanding the mineralogy of the upper mantle, mantle transition zone, and uppermost lower mantle (e.g., Ref. [1]). However, generally, pressures available in KMA are limited to ~30 GPa owing to the limitation in the hardness of the tungsten carbide (WC) used as second-stage anvils. The second-stage anvils are one of the most important parts in KMA, which concentrate the large press load (up to ~15 MN in the case of KMAs installed at BL04B1) generated by a hydraulic power unit into a sample put into the center of the apparatus.

In order to overcome this limitation in the pressure generation range of KMA, experimental techniques using sintered diamond (SD) as the material of secondstage anvils have been developed in Japan. The experimental techniques using SD anvils in KMA allowed studies to be carried out beyond the limit of ~30 GPa, but pressures have been limited to 40 GPa using conventional high-pressure cell assemblies optimized for experiments with WC anvils. This limitation in pressure generation using SD anvils has been overcome by changing the materials used for the gaskets and pressure medium, and the pressure range has since been expanded to 60 GPa. Recently, after further testing the materials for the pressure medium and anvils while optimizing the high-pressure cell assembly, the pressure range has been expanded to 80 GPa (Fig. 1) [2]. This pressure value corresponds to ~1900 km depth in the Earth's interior, and now, we can investigate the mineralogy of the lower mantle, which is found from 660 to 2900 km depth in the Earth, with the broad pressure range using KMA. In the next section, we introduce recent results obtained using the SDMA techniques.

As mentioned above, current high-pressure techniques using SDMA allow us to achieve pressures found in the middle part of the Earth's lower mantle. The Earth's lower mantle contains more than half the mass of the planet, and the system MgO-FeO-SiO<sub>2</sub> is fundamental to our understanding of its constituents and dynamics because the total amounts of MgO, FeO, and SiO<sub>2</sub> make up more than 90 wt% of the bulk silicate Earth. As a result, the phase relations in the system

MgO-FeO-SiO<sub>2</sub> have been extensively investigated using high-pressure and high-temperature techniques (e.g., Ref. [3]). We performed a series of high P-T experiments in the system MgO-FeO-SiO<sub>2</sub> under deep lower mantle conditions for the first time using KMA [4]. Phase relations in the system MgO-FeO-SiO<sub>2</sub> were investigated between 22 and 47 GPa at 1500 and 2000°C using SDMA to clarify P-T effects on the phase relations. A (Mg<sub>0.5</sub>,Fe<sub>0.5</sub>)SiO<sub>3</sub> clinopyroxene synthesized at 1 atm and 1250°C under a controlled oxygen fugacity was used as the starting material. After the high P-T experiments, the recovered samples were analyzed with an electron microprobe and by analytic transmission electron microscopy and X-ray diffraction measurements using synchrotron radiation at BL10XU to determine the phases present in the sample and its chemical composition.

After the analyses of the recovered samples, it was confirmed that the three phases of  $(Mg,Fe)SiO_3$ perovskite (Pv), (Mg,Fe)O magnesiowüstite (Mw), and stishovite (St) coexisted in all the run products. Since each sample is in a three-component system, the coexistence of three phases (Pv + Mw + St) at a given pressure and temperature indicates that Pv and Mw have univariant compositions assuming chemical equilibrium. The univariant compositions of Pv and Mw were determined as functions of pressure and temperature (Fig. 2). The pressure dependence of the phase relations can be seen easily in a pseudobinary phase diagram in the MgSiO<sub>3</sub>-FeSiO<sub>3</sub> system (Fig. 3(a)). The univariant composition of Pv is also the maximum iron content of this phase at a given



Fig. 1. Pressures generated using SDMA. Two kinds of sintered diamond,  $Al_2O_3$  and MgO ceramics, were tested as the anvil and pressure medium, respectively.



Fig. 2. Results of chemical analyses plotted on ternary diagrams. Samples were synthesized at 22-43 GPa and  $2000^{\circ}C$  (**a**-**d**), and at 25-47 GPa and  $1500^{\circ}C$  (**e**-**h**). Solid symbols show the measured compositions using EPMA. ATEM analyses were carried out in the case of (**g**) and (**h**), and the results are shown as open symbols. Gray areas show univariant triangles indicating the coexistence of Pv, Mw, and St.

*P-T* condition, and the maximum iron solubility in Pv gradually increases with increasing pressure and temperature to be more than 30 mol% at 2000°C and pressures above 40 GPa. In addition to the pressure effect in the maximum iron solubility in Pv, a significant



Fig. 3. (a) Univariant compositions of Pv and Mw, and corresponding phase boundaries in the  $MgSiO_3$ -FeSiO<sub>3</sub> pseudobinary system. (b) Fe-Mg distribution coefficients between Pv and Mw as a function of pressure. Solid circles and solid lines, 1500°C; open circles and broken lines, 2000°C in each diagram.

pressure effect was observed in the composition of Mw. The iron content of Mw dramatically increases from 50 to greater than 90 mol% with increasing pressure. As a result of this iron enrichment in the univariant composition of Mw, a significant pressure effect was found in Fe-Mg partitioning between Pv and Mw at pressures between 22 and 35 GPa, and the Fe-Mg distribution coefficients between Pv and Mw,  $K_D = (X_{Fe}^{Pv}/X_{Mg}^{Pv})/(X_{Fe}^{Mw}/X_{Mg}^{Mw})$ , decrease to less than 0.05 (Fig. 3). This significant pressure effect in Fe-Mg partitioning is expected to cause a strong concentration of ferrous iron in Mw with increasing depth in the lower mantle. The presence of highly iron-rich Mw would have some influence on deep mantle properties such as electric conductivity and viscosity.

As briefly introduced here, the experimental technique using SDMA is a powerful tool to investigate phase equilibria under *P*-*T* conditions of the Earth's lower mantle. Therefore, it is expected that further systematic studies using SDMA will improve our understanding of lower mantle mineralogy.

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