Diffraction and Scattering



IN SITU X-RAY DIFFRACTION STUDY OF POSTSPINEL TRANSFORMATION KINETICS IN Mg₂SiO₄ AT HIGH-PRESSURE AND HIGH-TEMPERATURE

High pressure transformation kinetics of mantle minerals are very important for understanding the dynamics of mantle convection and the oceanic plate's descent into the Earth's deep interior [1]. In situ X-ray diffraction using synchrotron radiation is especially suitable for the study of transformation kinetics at high pressure [2,3], because it is possible to collect a large amount of kinetic data efficiently. Simultaneously, precise measurements of the overpressure from the equilibrium phase boundary can be recorded, which should significantly affect the transformation rate. Dissociation of Mg₂SiO₄ spinel into MgSiO₃ perovskite and MgO periclase (the postspinel transformation) is believed to be of utmost importance in the division of the Earth's mantle into upper and lower parts. Since the kinetics of this transformation have never been examined, we have addressed this issue in an in situ X-ray diffraction study at high-pressure and hightemperature.

High-pressure in situ X-ray diffraction experiments

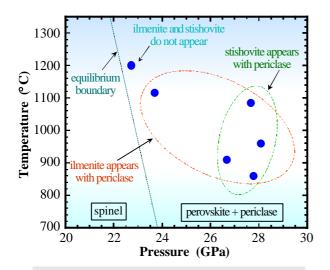


Fig. 1. Pressure - temperature diagram showing the experimental conditions. Conditions of metastable appearance of MgSiO₃ ilmenite and stishovite are also shown.

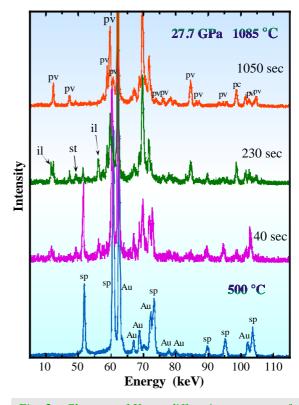


Fig. 2. Changes of X-ray diffraction patterns of the sample $(2\theta = 5.0^{\circ})$ during the postspinel transformation at 27.7 GPa and 1085 °C (sp: Mg_2SiO_4 spinel, pv: $MgSiO_3$ perovskite, pc: periclase, il: $MgSiO_3$ ilmenite, st: stishovite).

were performed using a SPEED1500 multi-anvil high pressure apparatus installed at beamline BL04B1 [4]. The starting material is a sintered mixture of Mg₂SiO₄ spinel and gold. Pressure was calculated from the lattice constants of gold [5]. The sample was compressed to the desired pressure at room temperature, and then heated to the desired temperature. The heating rate was maintained at about 500 °C/min. When the temperature reached the desired value, it was kept constant. Then, X-ray diffraction profiles were taken every 45 - 600 seconds. In this way, we were able to observe the postspinel transformation at 22.7 - 28.1 GPa and 860 - 1200 °C (Fig. 1). The equilibrium boundary of the postspinel transformation determined by SPEED1500 [6] was used in this study.

Figure 2 shows changes in diffraction patterns from Mg_2SiO_4 spinel to $MgSiO_3$ perovskite and periclase obtained at 27.7 GPa and 1085 °C.



Under these conditions, the transformation was completed in 1050 seconds. This figure indicates that, in addition to perovskite and periclase, MgSiO₃ ilmenite and SiO₂ stishovite were observed concurrently. Ilmenite and stishovite are thought to be metastable under these conditions, since these phases disappeared upon completion of transformation. The metastable appearance of ilmenite and stishovite was often observed at other pressure and temperature conditions (Fig. 1). The *in situ* X-ray data combined with SEM and TEM observations of the recovered sample indicate that spinel transforms into fine lamellae of stishovite and periclase, and ilmenite and periclase as intermediates in the postspinel transformation [7].

Figure 3 shows the time dependence of the transformed volume fraction estimated from the integrated intensities of the spinel diffraction lines. Based upon these kinetic data combined with previous results, we constructed transformation - temperature - time (t-t-t) diagrams showing the 5% transformation line at an overpressure of ~1 GPa and ~4 GPa (Fig. 4). Extrapolation of these lines to lower temperatures could be useful in evaluating whether or not the postspinel transformation occurs within the time scale for subduction (~10⁶ years) in the cold interior (~700 °C) of the descending oceanic plates.

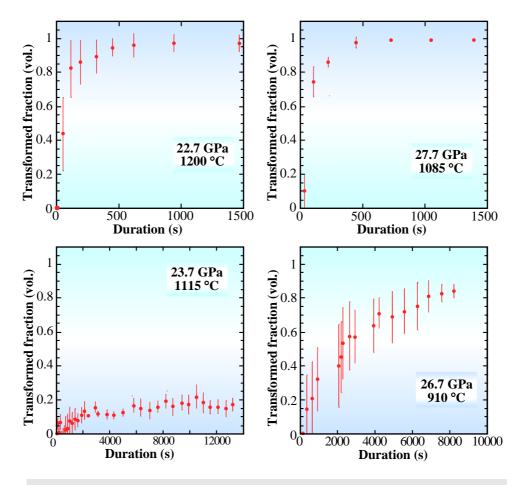


Fig. 3. Plots of transformed volume fraction with time in the postspinel transformation.



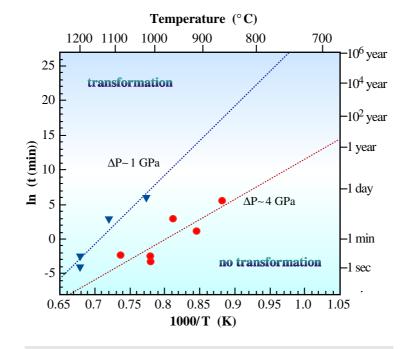


Fig. 4. Transformation - temperature - time diagrams for the postspinel transformation showing the 5% transformation with the overpressure of ~1 GPa (triangle) and ~4 GPa (circle).

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