Precise determination of the phase boundaries among ilmenite, garnet and perovskite structures in MgSiO₃ by in situ x-ray observations

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We attempted to determine the phase boundary between ilmenite and perovskite phases in MgSiO3 under high pressure and high temperature using a large multianvil high pressure device (SPEED-1500). We made two runs during this beam time, where some interesting results on the phase boundary were obtained as follows.

In the first run, we used a mixture of enstatite and gold powders as the starting material, while MgO+gold was also used in a separate sample charge to check the consistency of the pressure values in different media. Pressure was increased to about 25 GPa, and then temperature was gradually increased and X-ray diffraction data were acquired for about 5 minutes at each P/T condition.

We observed formation of metastable ilmenite at temperatures near 900°C, at GPa. about 25 which partially transformed to perovskite when temperature was increased to 1300°C. and at about 21 GPa. When the temperature was further increased, virtually no diffraction peaks from the ilmenite phase were identified at temperatures between 1400 and 1600°C and at pressures near 20 GPa, although the P/T condition apparently within the stability field of this We then guit the run and phase.

quenched to the room temperature, where some diffraction peaks of ilmenite were confirmed in the course of releasing pressure.

The above "amorphization" of MgSiO3 in the ilmenite phase region was interpreted as having been caused by the crystal growth of ilmenite and its preferred orientation, in addition to the overlapping of diffraction peaks with those of the mixed gold powder. Then, in order to identify the phases present unambiguously, we used a powder of pure MgSiO3 in the second run, and the pressure was determined from the other sample where the gold pressure marker was mixed with the MgO powder.

In this run, the formation of crystals of the ilmenite phase with significant preferred orientation was indeed confirmed, and further data on the phase boundary were obtained. However, the crystal growth and the resultant preferred orientation made it quite difficult to identify the phases present solely from the x-ray diffraction pattern.

In summary, the P/T condition of the phase boundary between ilmenite and perovskite phases was found to be close to that of the spinel to postspinel phase boundary of Mg2SiO4, but it seems likely that the former boundary locates at slightly lower pressures as compared to the postspinel phase boundary.