In Situ X-ray Diffraction Study of the Phase Transformations in (Mg,Fe)₂SiO₄ System at Low Temperatures

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Introduction

A knowledge of the kinetics and mechanisms of the transformation of olivine to its high-pressure polymorphs (wadsleyite and ringwoodite) under subduction zone conditions is important for understanding the dynamics of subduction, the mechanism of deep earthquakes, and the mechanical properties of subducting lithosphere. Although there have been many experimental investigations of these transformations, most studies were conducted using quenching technique on analogue materials or on olivine at high temperatures. The depth where the phase transition occurs in subducting slabs is not well constrained due to the large uncertainties in extrapolating laboratory experimental results to conditions in subducting lithosphere. We have initiated a research project studying the transformational characteristics of mantle olivine to its high-pressure polymorphs in the pressure and temperature regime of a subducting slab (i.e., high pressure and low temperature). We report a series of experimental studies directed to this effort. In the current experimental study, we investigate how low the temperature could be at which the olivine phase transformations could occur under high pressure.

Materials and Methods

Experiments were performed at beamline 13-BM. The starting material is San Carlos olivine. The olivine powder was loaded into the high-pressure cell together with powder NaCl as a pressure marker, each occupying about one half of the cylindrical cell. After the sample was compressed at room temperature to the target pressure (P) using the 250-ton large-volume press coupled with a T-Cup device, the temperature (T) was increased stepwise. The phase transformations were monitored by collecting timeresolved x-ray diffraction patterns while the sample remained at the desired P, T conditions. The pressure was calculated based on the Decker's EOS of NaCl, and the temperature was measured by a W-3%Re/W-25%Re thermocouple. This *in situ* x-ray technique has advantage over the quenching technique because it has the capability of tracing the P-T path of each run.

Results

For cold-compressed powder samples, the phase transformations were observed within minutes at 600°C and about 13-16 GPa. Figure 1 shows the sequential diffraction patterns from an experiment in which the sample was first compressed to 16.2 GPa at room temperature. Phase transformation (olivine to ringwoodite) was observed after 10 minuets at 15.6 GPa and 600°C. Olivine was completely transformed to a mixture of wadsleyite and ringwoodite after 5 minutes at 15.1 GPa and 850°C. In another experiment, olivine to wadsleyite transformation was observed at 12.8 GPa and 600°C (Fig. 2).

Discussion

Several important conclusions can be drawn from these observations. First, the phase transformations in the $(Mg,Fe)_2SiO_4$ system can proceed rapidly at temperature as low as 600°C, which is pertinent to the interior and cold region of subducting slab. The transformation at such low temperature has never been reported before. This result, if directly applied to subduction



FIG. 1. Sequential diffraction patterns for San Carlos olivine at different conditions. The dashed lines indicate the diffraction peaks of ringwoodite.



FIG. 2. Sequential diffraction patterns for an olivine sample compressed to 13.5 GPa at room temperature. The dotted lines indicate the diffraction peaks from wadsleyite.

zones, would indicate that no metastable olivine exists in subducting slab. On the other hand, one should realize that the transformations in current experiments were likely facilitated by the high differential stress and the defects, both resulted from compression at room temperature. Stress is manifested by peak broadening of the olivine diffraction. Cold compression could also generate a high density of defect and nanosized grains, both of which can catalyze the transformations by providing preferred sites for heterogeneous nucleation.

Second, the diffraction peaks of newly formed phases (wadsleyite and ringwoodite) are always sharp, suggesting that these phases are relatively free of stress. This observation rules out the proposition that the transformation could be slowed down by the high-pressure phases acting as pressure vessel over olivine grains.

Third, there is no significant difference in the kinetics of transformation at different pressures (12.8 and 15.6 GPa), sug-

gesting that activation volume for transformational process at current experimental conditions is trivial.

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