

Neutron diffraction experiment of hydrous wadsleyite

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Neutron diffraction experiments have been succeeded in determining of hydrogen position in many hydrous minerals. By contrast, little application to nominally anhydrous minerals has been done, despite its importance in the earth's interior. Wadsleyite, a high pressure polymorph of olivine, is a main constituent at the depth of 410 - 520 km of upper part of the mantle transition zone. Ever since Smyth (1987) predicted that protonation of O1 site of wadsleyite results in significant amount of water content up to 3.3 wt%, hydration of wadsleyite and related change in physical properties has attracted much attention. However, previous studies are based on X-ray diffraction and spectroscopic methods, and the position of hydrogen in wadsleyite still remains controversial. The aim of this study is the precise determination of hydrogen position in wadsleyite by neutron diffraction experiment.

Hydrous and deuterated wadsleyite were synthesized at around 17 GPa and 1300 °C, with duration of 1 h using Kawai-type multi-anvil apparatus at Tohoku University. The starting materials for the synthesis experiments were a mixture of powdered forsterite (Mg_2SiO_4), brucite ($\text{Mg}(\text{OH})_2$ or $\text{Mg}(\text{OD})_2$), and clinoenstatite (MgSiO_3), with overall compositions of $\text{Mg}_{1.85}\text{SiH}_{0.3}\text{O}_4$ and $\text{Mg}_{1.85}\text{SiD}_{0.3}\text{O}_4$ (corresponding to 2 wt% H_2O or D_2O). The starting material was put into a gold or platinum capsule with 4 mm in outer diameter and 3.8 mm in height. To avoid chemical differentiation from the various synthesis runs, the sample synthesized by single run was used in neutron diffraction experiment. Neutron diffraction pattern was obtained at D20 at ILL (Grenoble, France). Neutron beam was monochromized to $\lambda = 1.87$ Angstrom by Ge 115 monochromator. The sample was hold in a vanadium can with a diameter of 5 mm. The sample stage was oscillated between -1.5° and $+1.5^\circ$ with a step of 0.2° , and in each step, the data was corrected for 180 sec. Rietveld refinement was carried out using GSAS package.

Synthesized H-wadsleyite and D-wadsleyite were not sintered, but polycrystalline with a grain size of ~ 50 nm. Total amount of each sample was about 40 mg. Using the correlation between axial ratio of b/a and water content proposed by Jacobsen et al. (2005), the water content of H-wadsleyite and D-wadsleyite were calculated to be 1.7 wt% and 1.4 wt%, respectively. The deuteration of the synthesized sample was confirmed by Raman spectroscopy. Raman band of D-wadsleyite was observed in O-D stretching region, around 2480 cm^{-1} and 2650 cm^{-1} , as expected from reduced mass ratio of O-H and O-D. The quality of the obtained diffraction data with exposure time of 135 min may be sufficient to refine the crystal structure. The result of refinement will be presented in the meeting.