BL-10A/2014G081

Single crystal diffraction study on Al-bearing phase D

T. Kuribayashi[1,*], T. Inoue[2] and T. Nagase[3]
[1]Department of Earth Science, Graduate School of Science, Tohoku University, Sendai 980-8578
[2]GRC, Ehime University, Matsuyama 790-8577
[3]The Tohoku University Museum, Tohoku University, Sendai 980-8578

1. Introduction

Dense hydrous magnesium silicate (DHMS) minerals are important to consider the transportation of H into deep Earth's interior. DHMS phases are synthesized under cold slab conditions corresponding to the subduction zone. Several alphabetical phases belong to DHMS such as phase A, B, D, E and H. Phase D has the ideal chemical formula of $MgSi_2H_2O_6$, but generally has non-stoichiometric one. Two reports on the first synthesis of phase D was opened at the same time by two research groups [1, 2]. One of the group described the new phase as phase G, because the name of "phase D" was already used as another phase at that time.

Crystal structure of phase D belongs to the trigonal system with the space group $P\bar{3}1m$ (#162). One of the important properties of the structure is that Si occupies in six-coordinated site in the structure like stishovite [1, 3].

Recently, phase D including a larger amount of Al and H was synthesized at the conditions of 25-26 GPa and ~1600ºC coexisting with bridgmanite ($MgSiO_3$-perovskite) [4]. Therefore, Al in phase D structure is expected to play important role in its thermodynamic stability and H incorporation mechanism. However, the structural information on Al-bearing phase D is poor, and the incorporation mechanism of Al and H in phase D is unclear.

Single-crystal X-ray diffraction measurements on Al-bearing phase D was conducted to obtain its structural information in detail. In this report, we showed the brief results of X-ray diffraction experiments, and compared the lattice parameters of our sample with the previously reported values of the phase D structure.

2. Experimental Procedure

The sample used for this study was synthesized by [4] at 25-26 GPa and ~1600ºC. The run product included Al-bearing phase D, bridgemanite, stishovite and quenched dendritic phase, which was liquid under high pressure and temperature conditions. The Al content in Al-bearing phase D was briefly checked by [4] with EDS-SEM, and the content was measured less than 18 wt%Al_2O_3. After checking the quality of crystals by taking oscillation photographs, a single crystal of Al-bearing phase D was selected for synchrotron X-ray diffraction experiments. Single crystal X-ray diffraction experiments were performed using the automated four-circle X-ray diffractometer installed at the beam line BL-10A, Photon Factory, High Energy Accelerator Research Organization. The wavelength (λ = 0.7123 Å) of synchrotron radiation was calibrated by the unit cell volume of the NIST ruby standard crystal at ambient temperature. The unit cell parameters of Al-bearing phase D at room temperature were determined from 80 centered reflections in the 2θ range between 39° and 69°. The X-ray diffraction intensity data were collected up to $\sin\theta/\lambda < 1.16$ ($2\theta_{max}$ = 112º) by using ω-scan method.

3. Results and Discussion

The obtained lattice parameters of Al-bearing phase D are as follows: a = 4.7987(15) Å and c = 4.3106(4) Å. The lattice parameters of several phase D with minor components such as Al were summarized in Table 1.

The length of a-axis of our Al-bearing phase D has the largest value but the length of c-axis is the shortest except for the Mg-Free Al-phase D. However, since the space group of Mg-free Al-phase D is different from that of phase D, we need to check these properties carefully.

The axial lengths of phase D structure depend on its chemical formula, and especially, seem to be affected by the size of octahedron and the octahedral connection. These observations implied that the octahedral site is a key to understand the change of cell size and its incorporation mechanism. Now, the crystal structure of Al-bearing phase D is still in analysis to clarify Al distributions in its structure and Al replacement mechanism.

Table 1. Lattice parameters of phase D with various chemical formulae

Chemical Formula	a	c	Method	Reference
Al-bearing ph_D	4.7987 (15)	4.3106 (4)	SCXRD	this study
$Al_{1.54}Si_{0.98}H_{3.3}O_6$	4.7114 (6)	4.3039 (7)	SCXRD	Pamato et al. (2015)
$Mg_{1.11}Si_{1.89}H_{2.22}O_6$	4.7453 (4)	4.3450 (5)	SCXRD	Yang et al. (1997)
$Mg_{1.22}Si_{1.89}H_{2.33}O_6$	4.790 (3)	4.344 (3)	SCXRD	Kudoh et al. (1997)
$Mg_{1.11}Si_{1.60}H_{2.40}O_6$	4.7749 (6)	4.3389 (7)	PXRD	Frost and Fei (1999)
$Mg_{1.15}Si_{1.85}H_{2.30}O_6$	4.7590 (6)	4.3403 (4)	Neutron_PD	Suzuki et al. (2001)
$Mg_{1.12}Si_{1.88}D_{2.24}O_6$	4.7492 (4)	4.3520 (5)	Neutron_PD	Suzuki et al. (2001)

4. References

[1] Yang *et al*., *Am. Mineral*., **82**, 651-654 (1997) [2] Ohtani *et al*., *Geophys. Res. Lett*., **24**, 1047- 1050 (1997). [3] Kudoh *et al*., *Geophys. Res. Lett*., **24**, 1051- 1054 (1997). [4] Inoue *et al*., *in prep*. (2015). [5] Pamato *et al*., *.Nat. GeoSci*., (2015) DOI: 10.1038/NGEO2306. [6] Frost and Fei, *Phys. Chem. Mineral*., **26**, 415-418 (1999). [7] Suzuki *et al*., *Geophys. Res. Lett*., **28**, 3987-399 (2001).

* t-kuri@m.tohoku.ac.jp