（ ※本報告書は英語で記述してください。ただし、産業利用課題として採択されている方は日本語で記述していただいても結構です。 ）

CROSS TOKAI **Experimental Report** J-PARC	承認日Date of Approval 2015/7/21 承認者Approver Hattori Takanori 提出日Date of Report 2015/7/18
課題番号 Project No. 2014B0043 実験課題名 Title of experiment Hydrogen position of Al-bearing hydrous silicate perovskite (2) 実験責任者名 Name of principal investigator Toru Inoue 所属 Affiliation Ehime University	装置責任者 Name of responsible person Takanori Hattori 装置名 Name of Instrument/（BL No.） ATSUHIME (BL11, PLANET) 実施日 Date of Experiment 2015/4/8 21:00 - 4/15 9:00

試料、実験方法、利用の結果得られた主なデータ、考察、結論等を、記述して下さい。（適宜、図表添付のこと）
Please report your samples, experimental method and results, discussion and conclusions. Please add figures and tables for better explanation.

1. 試料 Name of sample(s) and chemical formula, or compositions including physical form.

We synthesized Al-bearing silicate perovskites with hydrous and anhydrous forms and Al-bearing new hydrous silicate phase in advance in Ehime University at 28 GPa and 1400℃, and 10 GPa and 950℃, respectively. In our 2014B0043 experiments, we performed the neutron diffraction experiments using the samples which had the sintered forms with the dimension of φ ~2 and ~2 mm length and the weight of ~30 mg, at ambient condition, and using the crashed sample from the big sintered form with φ ~6.5 and ~3 mm length and the weight of ~200 mg for high pressure experiment by Paris-Edimburgh (PE) cell.

For the details, see Table 1.

W2c and W1d:: Al-bearing deuterated Mg-perovskites, ~$MgSi_{0.9}Al_{0.1}O_3D_{0.1}$ and ~$MgSi_{0.95}Al_{0.05}O_3$ $D_{0.05}$

DO1b & DO2b: Al-bearing anhydrous silicate perovskites (oxygen vacancy type), ~$MgSi_{0.95}Al_{0.05}O_{2.975}$ & ~$MgSi_{0.9}Al_{0.1}O_{2.95}$

D1b: Al-bearing anhydrous silicate perovskite (coupled substitution type), ~$Mg_{0.95}Si_{0.95}Al_{0.1}O_3$

Na: Al-bearing new hydrous silicate phase, ~$Mg_{5.5}AlSi_2O_8(OD)_6$

2. 実験方法及び結果（実験がうまくいかなかった場合、その理由を記述してください。）
Experimental method and results. If you failed to conduct experiment as planned, please describe reasons.

The Al-bearing deuterated (hydrous) silicate perovskites and Al-bearing anhydrous silicate perovskites with oxygen vacancy type and coupled substitution type were synthesized at ~28 GPa and ~1600℃, and Al-bearing deuterated (hydrous) new phases were synthesized at ~10 GPa and 950℃ by Kawai-type high pressure apparatus in Ehime University. The recovered samples were characterized by X-ray powder diffraction, and confirmed that the silicate perovskite phase and Al-bearing new hydrous silicate phase were the dominant phases in each run product.

2. 実験方法及び結果（つづき） Experimental method and results (continued)

We conducted the neutron diffraction experiments in MLF on 2015/4/8 21:00 - 4/15 9:00. First, we collected the clear neutron diffraction pattern of high pressure Al-bearing deuterated silicate perovskite phases, Al-bearing anhydrous silicate perovskites (oxygen vacancy type), and Al-bearing anhydrous silicate perovskite (coupled substitution type) at ambient condition which we have already synthesized by high pressure apparatus in Ehime University. The experimental details of each neutron diffraction experiments were summarized in Table.1. We measured 5 samples. The V rod, V tube and air (empty) were also measured for the data correction.

Figure 1 shows the representative pictures of synthesized Al-bearing hydrous silicate perovskite (W2c and W1d), deficit oxygen (oxygen-vacancy) silicate perovskite (DO1b and DO2b), Al-bearing dry silicate perovskite (D1b) and new hydrous phase (Na). We designed and developed larger cell assembly to synthesize large Mg-perovskite sample at ~28 GPa and Al-bearing new hydrous phase at 10 GPa. The sample was sealed by Au capsule. As the results, we succeeded to synthesize the samples which have the dimension of φ ~2.2 and ~2.5 mm length and the weight of ~30 mg for various perovskite, and the dimension of φ ~6.5 and ~3 mm length and the weight of ~200 mg for Al-bearing new hydrous phase. These dimensions were large enough to collect the neutron diffraction data in less than ten hours.

Figure 2 shows the obtained neutron diffractions of Al-bearing silicate perovskite with hydrous and anhydrous forms at BL11 in MLF, J-PARC.

We also collected the neutron diffractions of Al-bearing new hydrous silicate phase at ~1, ~3, 6, 8, 10 and 15 GPa, which correspond to the oil pressures of 10, 30, 60, 80, 100 and 153 MPa, respectively from the previous pressure calibration (Fig. 3). Now we are conducting Rietveld analysis to determine the hydrogen position.

(Attached table and figure)

Table 1. The sample and experimental details of each neutron diffraction experiments in 2014B.

Figure 1: The representative pictures of synthesized Al-bearing hydrous perovskites and the related phases used for our neutron diffraction experiments. The ruler graduation is 1 mm.

Figure 2. The obtained neutron diffractions of Al-bearing silicate perovskite with hydrous and anhydrous forms at BL11 in MLF, J-PARC.

W2c: hy-Pv (aluminous deuterated silicate perovskite), ~$MgSi_{0.9}Al_{0.1}O_3D_{0.1}$

W1d: hy-Pv (aluminous deuterated silicate perovskite), ~$MgSi_{0.95}Al_{0.05}O_3D_{0.05}$

DO1b: O-Pv (Deficit Oxygen silicate perovskite), ~$MgSi_{0.95}Al_{0.05}O_{2.975}$

DO2b: O-Pv (Deficit Oxygen silicate perovskite), ~$MgSi_{0.9}Al_{0.1}O_{2.95}$

D1b: Dry-Pv (aluminous silicate perovskite), ~$Mg_{0.95}Si_{0.95}Al_{0.1}O_3$

Figure 3. The obtained neutron diffractions of Al-bearing new hydrous silicate phase (Na) under high pressure using PE press at BL11 in MLF, J-PARC. Based on the previous pressure calibration, the generated pressures should be ~1, ~3, 6, 8, 10 and 15 GPa at the oil pressures of 10, 30, 60, 80, 100 and 153 MPa, respectively.

Na: New phase (Al-bearing new hydrous silicate phase), ~$Mg_{5.5}Si_2AlO_8(OD)_6$