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CROSS TOKAI Experimental Report J-PARC	承認日 Date of Approval 2014/6/9 承認者 Approver Takanori Hattori 提出日 Date of Report 2014/6/9
課題番号 Project No. 2013B0095 実験課題名 Title of experiment Hydrogen position of Al-bearing hydrous silicate perovskite 実験責任者名 Name of principal investigator Trou 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 3/27 21:00 - 3/31 21:00

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 aluminous Mg-perovskites in dry, hydrous and deuterated forms at ~28 GPa and ~1600℃, in advance, in Ehime University. In our 2013B0095 experiments, we performed the neutron diffraction experiments using these samples, which have sintered forms with the dimension of φ ~2.2 and ~2.5 mm length and the weight of ~30 mg.

W1: Al-de Pv (aluminous deuterated Mg-perovskite), ~$MgSi_{0.95}Al_{0.05}O_3D_{0.05}$

W2: Al-de Pv (aluminous deuterated Mg-perovskite), ~$MgSi_{0.9}Al_{0.1}O_3D_{0.1}$

D1: Al Pv (aluminous Mg-perovskite), ~$Mg_{0.95}Si_{0.95}Al_{0.1}O_3$

D2: Al Pv (aluminous Mg-perovskite), ~$Mg_{0.9}Si_{0.9}Al_{0.2}O_3$

WH1: Al-hy Pv (aluminous hydrous Mg-perovskite), ~$MgSi_{0.95}Al_{0.05}O_3H_{0.05}$

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 aluminous Mg-perovskites in dry, hydrous and deuterated forms were synthesized at ~28 GPa and ~1600℃ by Kawai-type high pressure apparatus in Ehime University. The recovered samples were characterized by X-ray powder diffraction, and confirmed that aluminous Mg-perovskites were the dominant phase coexisting with a very small amount of stishovite (SiO_2) or phase D, which depends on the synthesis conditions.

We conducted the neutron diffraction experiments in MLF on 3/27 21:00 - 3/31 21:00, 2014. At that time, we knew that the condition of ATSUHIME press was not so good because of terrible oil release from the plunger pump. Because we are worried about the further damage of the piston scratch in the plunger pump system, we decided to collect the clear neutron diffraction pattern at ambient condition using the various kind of aluminous Mg-perovskite in anhydrous, hydrous and deuterated forms. The sample and experimental details of each neutron diffraction experiments were summarized in Table.1. We measured 7 samples (W1a, W1b, W1c, W2, D1, D2 and WH1) in this period. The V rod, V tube and air (empty) were also measured for the data correction.

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

Figure 1 shows the representative pictures of synthesized aluminous Mg-perovskite (a: D2) and the deuterated form (b: W2). We designed and developed larger cell assembly to synthesize large Mg-perovskite sample at ~28 GPa conditions. The sample was sealed by Au capsule (Figure 1c). 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. These dimensions were large enough to collect the neutron diffraction data in less than ten hours.

Figure 2 shows the obtained neutron diffractions of aluminous Mg-perovskite of anhydrous, hydrous and deuterated forms at BL11 in MLF, J-PARC. Preliminary results show that the lattice parameters and unit cell volume of D2 and W2 are a = 4.77656(4) Å, b = 4.94206(5) Å, c = 6.93226(6) Å, V = 163.643(2) Å^{3,}, and a = 4.78011(5) Å, b = 4.93827(6) Å, c = 6.92090(8) Å, V = 163.371(2) Å^3, respectively. Kubo and Akaogi (2000) shows that the b- and c-axes and the volume of aluminous Mg-perovskite expand, whereas the a-axis does not change so much with increasing Al_2O_3 content. Our anhydrous data shows that trend. Whereas in hydrous and deuterated samples, all lattice constants and the volume expand with increasing Al_2O_3 content (Figure 3). These phenomena must come from hydrogen in aluminous Mg-perovskite.

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

Figure 1: The representative pictures of synthesized aluminous Mg-perovskite (a: D2) and the deuterated form (b: W2). c: Sample was sealed by Au capsule.

Figure 2. The obtained neutron diffractions of aluminous Mg-perovskite of anhydrous, hydrous and deuterated forms at BL11 in MLF, J-PARC.

W1: Al-de Pv (aluminous deuterated Mg-perovskite), ~$MgSi_{0.95}Al_{0.05}O_3D_{0.05}$

W2: Al-de Pv (aluminous deuterated Mg-perovskite), ~$MgSi_{0.9}Al_{0.1}O_3D_{0.1}$

D1: Al Pv (aluminous Mg-perovskite), ~$Mg_{0.95}Si_{0.95}Al_{0.1}O_3$

D2: Al Pv (aluminous Mg-perovskite), ~$Mg_{0.9}Si_{0.9}Al_{0.2}O_3$

WH1: Al-hy Pv (aluminous hydrous Mg-perovskite), ~$MgSi_{0.95}Al_{0.05}O_3H_{0.05}$

Figure 3. The lattice parameters and unit cell volume of aluminous Mg-perovskite as a function of Al_2O_3 content (mol%). The following large marks are our present data, and small blue diamonds are from Kubo and Akaogi (2000).

- W2: Al-de Pv (aluminous deuterated Mg-perovskite), ~$MgSi_{0.9}Al_{0.1}O_3D_{0.1}$
- WH1: Al-hy Pv (aluminous hydrous Mg-perovskite), ~$MgSi_{0.95}Al_{0.05}O_3H_{0.05}$
- D2: Al Pv (aluminous Mg-perovskite), ~$Mg_{0.9}Si_{0.9}Al_{0.2}O_3$