

実験報告書様式(一般利用課題・成果公開利用)

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	承認日 Date of Approval 2015/6/3 承認者 Approver Takanori Hattori 提出日 Date of report 2015/6/3
課題番号 Project No. 2014A0158 実験課題名 Title of experiment Hydrogen position of Al-bearing hydrous phase D and superhydrous phase B 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 2014/11/13 21:00 - 11/19 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.

<p>1. 試料 Name of sample(s) and chemical formula, or compositions including physical form.</p> <p>We synthesized various Al-bearing hydrous phases in advance in Ehime University at 28 GPa and 1400°C, and 10 GPa and 900°C. In our 2014A0158 experiments, we performed the neutron diffraction experiments using these samples, which had the sintered forms with the dimension of $\phi \sim 2$ and ~ 2 mm length and the weight of ~ 30 mg, and the big sintered forms with $\phi \sim 6.5$ and ~ 3 mm length and the weight of ~ 200 mg.</p> <p>W2b & WH2a: Al-bearing deuterated & hydrous Mg-perovskite, $\sim \text{MgSi}_{0.9}\text{Al}_{0.1}\text{O}_3\text{D}_{0.1}$ & $\sim \text{MgSi}_{0.9}\text{Al}_{0.1}\text{O}_3\text{H}_{0.1}$</p> <p>DO1a & DO2a: Al-bearing Mg-perovskites, $\sim \text{MgSi}_{0.95}\text{Al}_{0.05}\text{O}_{2.975}$ & $\sim \text{MgSi}_{0.9}\text{Al}_{0.1}\text{O}_{2.95}$</p> <p>New phase D & H (Na, Nb, Nc, NHa, NHb, NHc): Al-bearing silicates, $\sim \text{Mg}_{5.5}\text{AlSi}_2\text{O}_8(\text{OD})_6$ & $\sim \text{Mg}_{5.5}\text{AlSi}_2\text{O}_8(\text{OH})_6$</p>
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<p>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。)</p> <p>Experimental method and results. If you failed to conduct experiment as planned, please describe reasons.</p> <p>The Al-bearing deuterated & hydrous Mg-perovskites and Al-bearing anhydrous Mg-perovskites were synthesized at ~ 28 GPa and $\sim 1600^\circ\text{C}$, and Al-bearing deuterated & hydrous new phases were synthesized at ~ 10 GPa and 900°C by Kawai-type high pressure apparatus in Ehime University. The recovered samples were characterized by X-ray powder diffraction, and confirmed that the Al-bearing deuterated & hydrous Mg-perovskite, Al-bearing anhydrous Mg-perovskites, and Al-bearing deuterated & hydrous new phases were the dominant phases in each run product.</p> <p>We conducted the neutron diffraction experiments in MLF on 2014/11/13 21:00 - 11/19 9:00. Before the experiments, we heard 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 of high pressure Al-bearing hydrous phases at ambient condition which we have already synthesized by high pressure apparatus in Ehime University.</p>
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2. 実験方法及び結果(つづき) Experimental method and results (continued)

Al-bearing deuterated & hydrous Mg-perovskites, Al-bearing anhydrous Mg-perovskites, and Al-bearing deuterated & hydrous new phases were successfully synthesized. The samples and the experimental details of each neutron diffraction experiments were summarized in Table.1. We measured 10 samples in this period. 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 Mg-perovskite (hy-Pv, D: deuterated, H: hydrous), oxygen-vacancy Mg-perovskite (O-Pv), and Al-bearing new hydrous phase (new phase, D: deuterated, H: hydrous). 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 Mg-perovskite of deuterated, hydrous and oxygen vacancy forms, and those of Al-bearing new hydrous phase at BL11 in MLF, J-PARC. 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 2014A.

Figure 1: The representative pictures of synthesized Al-bearing hydrous phases 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 Mg-perovskite and Al-bearing new hydrous phase at BL11 in MLF, J-PARC.

W2b: hy-Pv (aluminous deuterated Mg-perovskite), $\sim\text{MgSi}_{0.9}\text{Al}_{0.1}\text{O}_3\text{D}_{0.1}$

WH2a: hy-Pv (aluminous hydrous Mg-perovskite), $\sim\text{MgSi}_{0.9}\text{Al}_{0.1}\text{O}_3\text{H}_{0.1}$

DO1a: O-Pv (Oxygen vacancy Mg-perovskite), $\sim\text{MgSi}_{0.95}\text{Al}_{0.05}\text{O}_{2.975}$

DO2a: O-Pv (Oxygen vacancy Mg-perovskite), $\sim\text{MgSi}_{0.9}\text{Al}_{0.1}\text{O}_{2.95}$

Na: New phase_D (Al-bearing new deuterated phase), $\sim\text{Mg}_{5.5}\text{Si}_2\text{AlO}_8(\text{OD})_6$

NHa: New phase_H (Al-bearing new hydrous phase), $\sim\text{Mg}_{5.5}\text{Si}_2\text{AlO}_8(\text{OH})_6$