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 MLF Experimental Report	提出日 Date of Report 2017.8.6
課題番号 Project No. 2017A0166 実験課題名 Title of experiment Transportation of hydrogen at high temperatures in the deep Earth by quasielastic neutron scattering II 実験責任者名 Name of principal investigator Takuo OKUCHI 所属 Affiliation Institute for Planetary Materials, Okayama University	装置責任者 Name of responsible person Kaoru SHIBATA 装置名 Name of Instrument/(BL No.) DNA / BL02 実施日 Date of Experiment May 29-June 6, 2017

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.

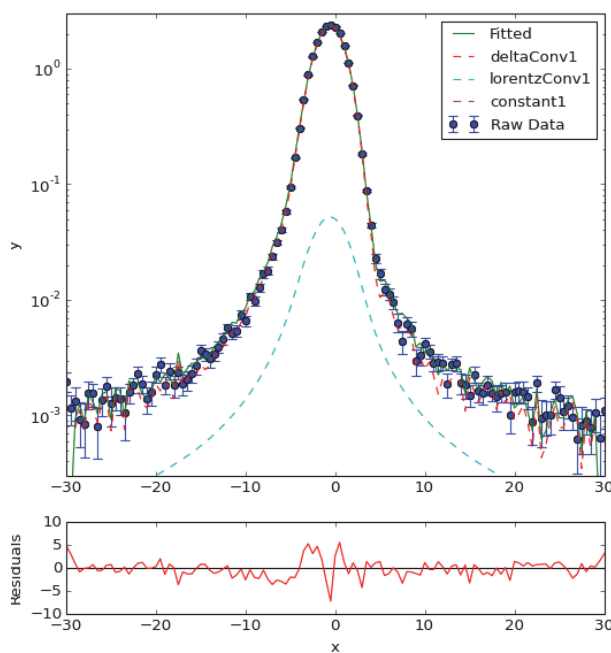
1. 試料 Name of sample(s) and chemical formula, or compositions including physical form. Dense Hydrous Magnesium Silicate Phase E [$Mg_{2+x}Si_{1+y}H_{4-2x-4y}O_6$], powder Hydrous wadsleyite [$(Mg,Fe,H)_2SiO_4$], powder
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2. 実験方法及び結果 (実験がうまくいかなかった場合、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. <p>As in the case for our previous proposal (2016B0159), this continuing proposal was technically supported by our pre-established scheme of mass-production of hydrous deep-Earth minerals of excellent quality and quantity (Okuchi et al., Am. Mineral. 2015). A single-phase powder of about 250 mg of dense hydrous magnesium silicate (DHMS) phase E was already synthesized by accumulating the products of five independent experimental runs. In addition, we has newly mass-produced hydrous wadsleyite sample powder for the current (and also for the related future) machine time, which is the most significant advance for the current machine time period. For all these synthesis experiments, we used a scaled-up Kawai-type cell along with 46 mm-sized cubic carbide anvils and the 18/10 cell assembly. The reagents were mixed and separately sealed into a gold tube capsule of 5 mm outside diameter, which yielded 90 ± 10 mg of the product after reacting at 15 GPa pressure. We heated the capsule at temperature around 1600 K to synthesize a homogeneous hydrous wadsleyite of that quantity, by applying electronic current</p>
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2. 実験方法及び結果(つづき) Experimental method and results (continued)

to a cylindrical ceramic (LaCrO_3) furnace installed inside the cell surrounding the gold capsule. Each product recovered from each sample capsule was separately evaluated by powder x-ray diffraction to confirm whether the product was single-phase, chemically homogeneous, as well as highly crystalline. Among all these recovered ones, we have selected four highest-quality products, which were measured together to analyze its quasi-elastic neutron scattering (QENS) at DNA, MLF, J-PARC.

The DNA spectrometer has been designed not only for providing the highest energy resolution but also for high neutron beam intensity. After previous efforts at DNA as the first practice for QENS of hydrous minerals, we found that both features were quite essential for revealing the slow dynamics of chemically-bonded hydrogen in these minerals. The extremely-low background feature of DNA was also proved to be indispensable for analyzing such dynamics in mass-limited synthetic deep-Earth minerals. In the current machine time proposed for hydrous wadsleyite, in spite of the severely reduced neutron beam intensity, we have obtained two $S(Q,E)$ datasets of this very important deep-Earth mineral (see figure below as an example, “Raw Data” points taken at 550 K temperature for $Q = 1.45 \text{ \AA}^{-1}$).



We observed that the dynamics of hydrogen in hydrous wadsleyite was not very significant up to 550 K temperature. However, in very near future after the scheduled increase of proton beam power, we will find more active dynamics in hydrous wadsleyite at even higher temperature conditions, which was already predicted by the occurrence of dehydration observed in thermogravimetric result of the wadsleyite sample. These results for wadsleyite will provide important implication for transportation dynamics of hydrogen in wet mantle transition zone in the deep Earth (Purevjav et al., Sci. Rep. 2016). We also obtained additional $S(Q,E)$ dataset of DHMS phase E using a part of the current machine time.