


実験報告書様式(一般利用課題・成果公開利用)

(※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。)

 MLF Experimental Report	提出日 Date of Report
課題番号 Project No. 2016B0049 実験課題名 Title of experiment Pressure response of deuterated boehmite (AIOOD) to observe hydrogen bond symmetrization 実験責任者名 Name of principal investigator Kazuki Komatsu 所属 Affiliation Graduate School of Science, The University of Tokyo	装置責任者 Name of responsible person Takanori Hattori 装置名 Name of Instrument/(BL No.) PLANET (BL 11) 実施日 Date of Experiment 2016/11/28-2016/11/30

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. deuterated boehmite, AIOOD
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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. Deuterated sample of boehmite was synthesized by hydrothermal treatment for the hydrogenated boehmite with D ₂ O. Powder of Al(OH) ₃ (99.99%, High Purity chemical. inc.) with 10 ml mill-Q water was inserted into a PTFE-lined autoclave, and kept at temperature of 513 K for 48 h. The same treatment was conducted with deuterated water twice. In-situ neutron diffraction measurements of deuterated boehmite under pressure were also performed on the PLANET beamline. The synthesized deuterated samples and deuterated methanol-ethanol 4:1 mixture were loaded into a pair of encapsulating TiZr gaskets fitted with a tapered aluminum ring, sandwiched with WC anvils (MF10, Fujillo) and compressed by using the Paris-Edinburgh press (type V4). Sample pressures were estimated from the equation-of-state (EoS) obtained from the in-situ x-ray diffraction separately conducted at PF-18C, KEK.
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2. 実験方法及び結果(つづき) Experimental method and results (continued)

The hydrogen bonding geometric parameters, O-D, D...O and O...O distances, obtained from the Rietveld refinements for neutron diffraction patterns up to 9.48 GPa, are shown in Figure 1. Typical compression behavior for hydrogen bond is found; O...O and D...O distances linearly decreases with increasing pressure, whereas O-D distance linearly increases, i.e., O-D and D...O distances approach each other and their linear extrapolation will merge at around 26 GPa (Fig. 2).

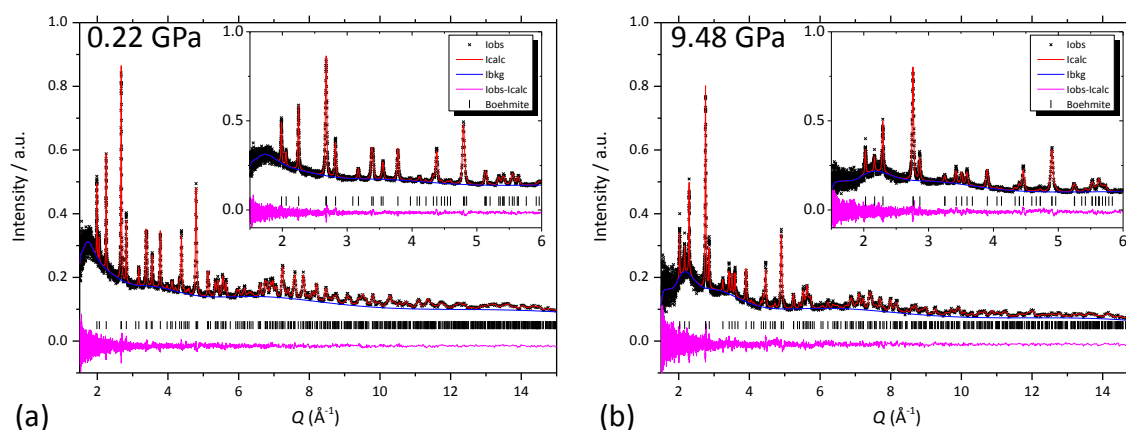


Fig. 1. Results of Rietveld refinements for neutron diffraction patterns acquired at (a) 0.22 GPa and (b) 9.48 GPa. Insets show the enlarged plot for low- Q region. Broad scattering particularly seen in low- Q is derived from the methanol-ethanol mixture used as pressure transmitting medium.

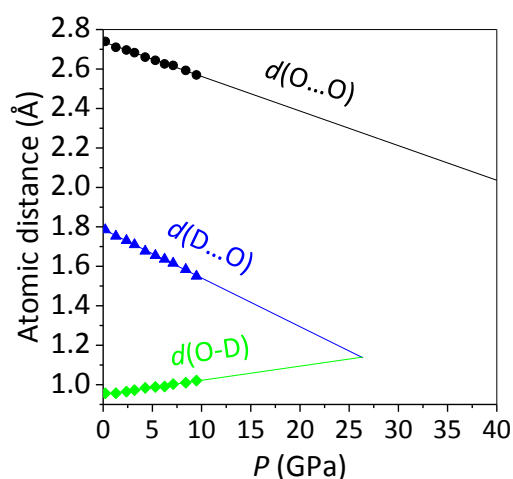


Fig. 2 Selective atomic distances, $d(\text{O-D})$, $d(\text{D}\dots\text{O})$ and $d(\text{O}\dots\text{O})$ in O-D...O H-bond, with increasing pressure. Lines describe linear extrapolations for respective distances. Errors are within the size of symbols.