

(※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。)

 <p>Experimental Report</p>	承認日 Date of Approval 2015/4/28 承認者 Approver 服部 高典 提出日 Date of Report 2015/4/28
課題番号 Project No. 2014B0094 実験課題名 Title of experiment Investigation of hydrogen bond symmetrization in δ -AlOOH at high pressure 実験責任者名 Name of principal investigator Asami Sano 所属 Affiliation Japan Atomic Energy Agency	装置責任者 Name of Instrument scientist Takanori Hattori 装置名 Name of Instrument/(BL No.) PLANET / BL11 実施日 Date of Experiment 2015/3/25 21:00 – 2015/4/1 7:00

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>Samples:</p> <p>(1) Aluminous hydroxide, AlOOH, powder (2) Vanadium, V, solid</p> <p>Pressure medium: Deuterated methanol-ethanol mixture, CD₃OD-C₂D₅OD, liquid</p> <p>Gasket (sample cell): Titan zirconium alloy, TiZr, solid</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 aim of this study was to investigate the phase transition of δ-AlOOH at high pressure by in-situ neutron diffraction experiment. A Paris-Edinburgh press with double-toroid sintered diamond anvils was used to generate high pressure. The sample synthesized at 18 GPa and 900 °C was contained by null-scattering TiZr alloy encapsulating gaskets, together with a pressure medium of deuterated methanol-ethanol mixture. Time-of flight neutron powder diffraction data were collected at the press loads of 10, 30, 60, 70, 80, 90, 100, 120, 130 and 153 ton. The pressures were determined from the lattice volume of δ-AlOOH and an equation of state determined by previous X-ray diffraction study. The data of vanadium at high pressure and empty gaskets were also collected to correct the intensity of the sample data.</p>

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

The diffraction patterns were successfully collected up to 15.5 GPa. The phase transition from $P2_1nm$ to $Pnmm$ was observed at ~ 6 GPa, as indicated by the disappearance of weak reflection of 021 at around 1.69 \AA with extinction conditions of $0kl: k+1 \neq 2n$ (Fig. 1). This result is consistent with our previous single crystal X-ray diffraction study. Detail refinement is now under way to investigate whether another phase transition exists or not at higher pressure, which is accompanied by the slight change of hydrogen position. The result will provide an understanding about the sequence of pressure-induced hydrogen bond symmetrization and its relation with the change of compression behavior.

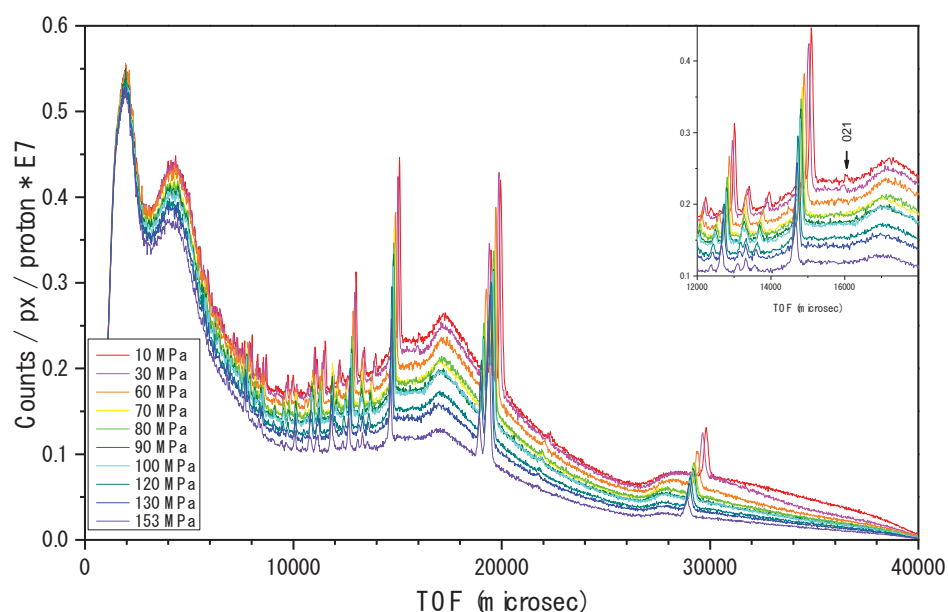


Figure 1. Diffraction patterns of the δ -AIOOH.

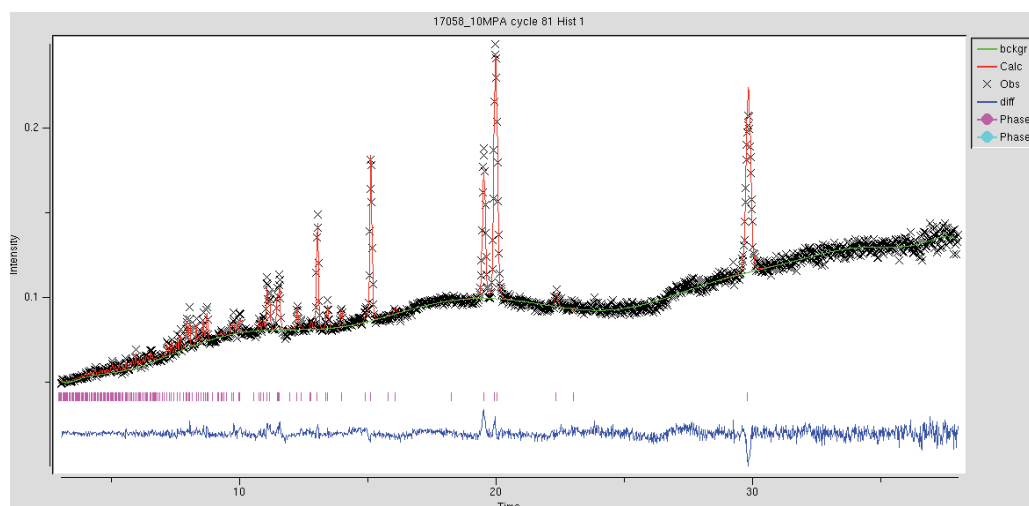


Figure 2. Preliminary Rietveld refinement for the data taken at 10 MPa.