

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。)

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|  Experimental Report  | 承認日 Date of Approval 2014/7/17 承認者 Approver Takanori Hattori 提出日 Date of Report 2014/7/14 |
| 課題番号 Project No. 2014A0050 実験課題名 Title of experiment Structural variations of $\text{Ca}(\text{OD})_2$ at high pressure and high temperature 実験責任者名 Name of principal investigator Takaya Nagai 所属 Affiliation Faculty of Science, Hokkaido University, Japan | 装置責任者 Name of Instrument scientist Takanori Hattori 装置名 Name of Instrument/(BL No.) BL11 実施日 Date of Experiment May 13, 2014 ~ May 18, 2014 |

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.

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| 1. 試料 Name of sample(s) and chemical formula, or compositions including physical form. deuterated portlandite, $\text{Ca}(\text{OD})_2$: 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. Experimental TOF neutron powder diffraction measurements of synthetic powder of deuterated portlandite, $\text{Ca}(\text{OD})_2$ were carried out from 300 to 773 K at about 4.5 GPa at the PLANET beamline. $\text{Ca}(\text{OD})_2$ belongs to a trigonal crystal system and the cell dimensions are $a=3.5826(6)$ Å and $c=4.885(2)$ Å at ambient conditions. Generation of high pressure and high temperature (high P-T) can be performed by using the 6-ram big press “Atsuhime” installed at the PLANET. 6 WC anvils truncated edge length of 10 mm, Al anvil guide, 17 mm cube ZrO_2 pressure medium and $4 \text{ mm}\phi \times 4 \text{ mm}$ graphite tube heater were used for high P-T experiments. Incident beams were cut by slits of $W 2 \text{ mm} \times H 4 \text{ mm}$ and radial collimators were set before detectors. Experimental P-T path was as follows. At first, pressure was increased to 4.5 GPa at room temperature and then, temperature was increased to 773 K. Data acquisition was carried out at each temperature condition (773, 673, 573, 473, 373 and 300 K) on the way to a room temperature. Duration of each measurement was for 720000 kickers (about 10 hours). |
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2. 実験方法及び結果(つづき) Experimental method and results (continued)

Results

TOF neutron powder diffraction data could be obtained at about 4 GPa and at 773, 673, 573, 473, 373 and 300 K, as shown in Figure 1. Diffraction peaks are indexed as $\text{Ca}(\text{OD})_2$ and the unit cell parameters could be reasonably refined. Variation of the unit cell dimensions are plotted at about 4 GPa as a function of temperature in Figure 2. It is quite interesting that the change of the c/a values is very small and the increment of c/a is less than 0.002 from room temperature to 773 K. This means that the behavior of the thermal expansion of $\text{Ca}(\text{OD})_2$ at about 4 GPa is rather isotropic, although Xu et al.(2007) reported that the thermal expansion of $\text{Ca}(\text{OD})_2$ is anisotropic and the increment of c/a at ambient pressure is about 0.02 from room temperature to 643 K. This evidence can be explained by the behavior of deuterium atoms located between CaO_6 octahedral sheets in the $\text{Ca}(\text{OD})_2$ structure.

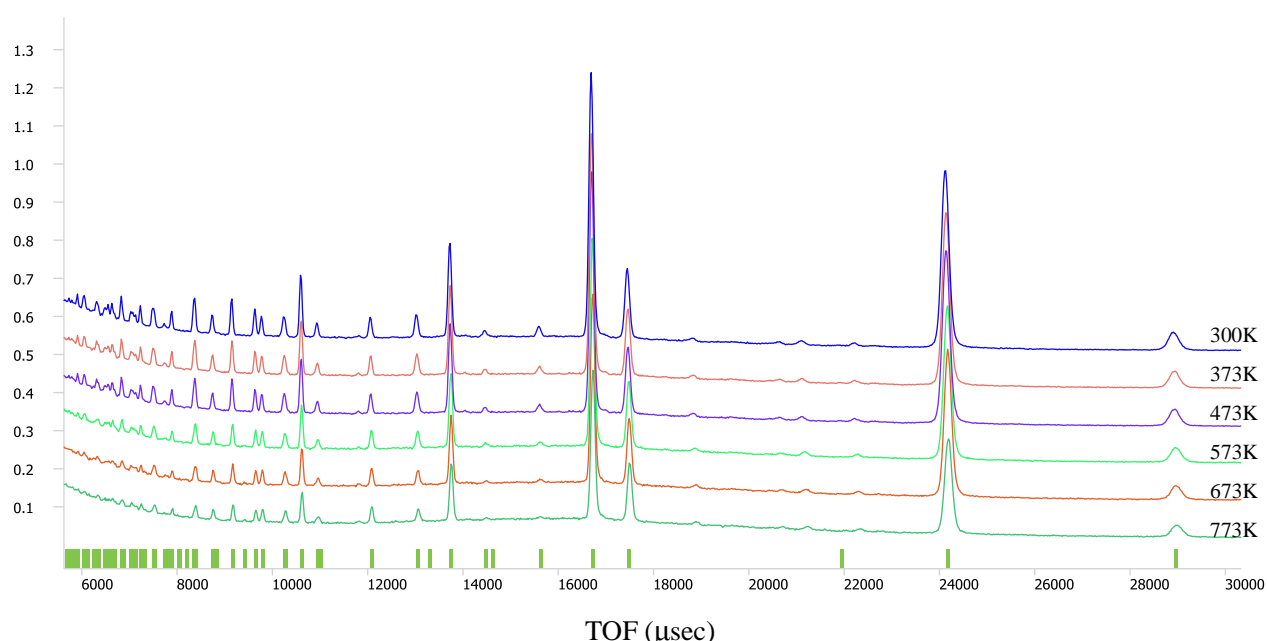


Figure 1: Raw data of TOF neutron powder diffraction measured at about 4 GPa and at several temperatures.

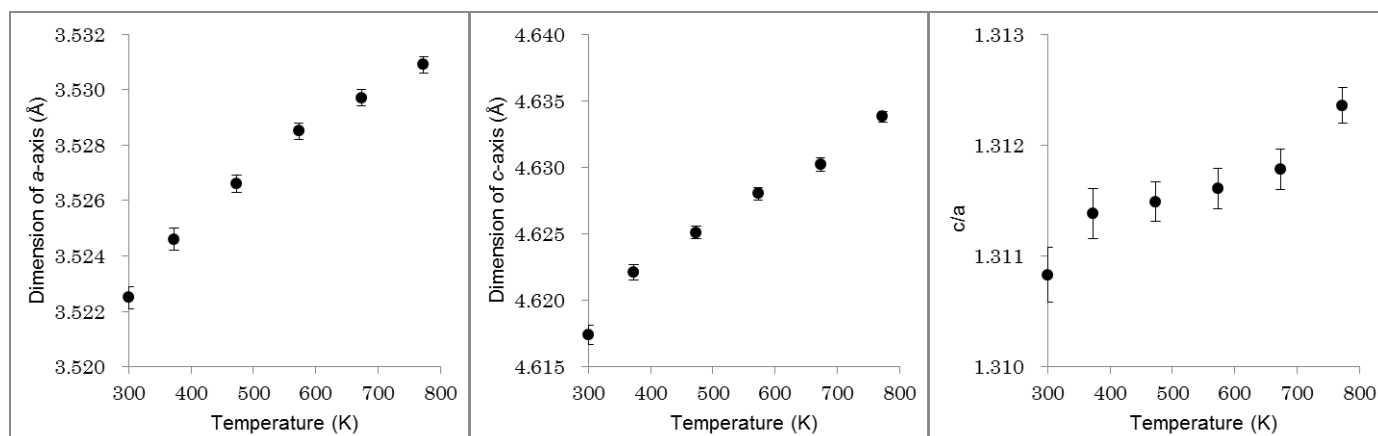


Figure 2: Variation of the unit cell dimensions as a function of temperature at about 4 GPa.

References: Xu et al. (2007) *J. Solid State Chem.*, 180, 1519-1525.