

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。)

 Experimental Report 	承認日 Date of Approval 2014/10/27 承認者 Approver Takanori HATTORI 提出日 Date of Report 2014/10/15
課題番号 Project No. 2014A0120 実験課題名 Title of experiment In-situ observation by neutron diffraction on the formation of dense ice containing MgCl ₂ in the structure 実験責任者名 Name of principal investigator Hiroyuki Kagi 所属 Affiliation Graduate School of Science, The Univ. of Tokyo	装置責任者 Name of Instrument scientist Takanori Hattori 装置名 Name of Instrument/(BL No.) PLANET 実施日 Date of Experiment 2014/4/20-2014/4/26

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. MgCl ₂ -25D ₂ O solution
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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. The sample solution was loaded into the 'Mito system' (Komatsu et al., High Press. Res. 2013), and load and temperature were controlled as follows. ① Compress up to 7 tonf (~0.3 GPa) at room temperature ② Cool down to 100 K with ~5 K/min ③ Compress up to 28 tonf (~3 GPa) at 100 K ④ Heat up to 300 K without controlling pressure The sample was amorphized in the path ①-②, and sustained in ③ as well. The representative neutron diffraction patterns are shown in Fig. 1. The amorphous sustained until at least up to 226 K, but some broad peaks are appeared from 246 K to 279 K. This phase was also observed in our previous test experiments in synchrotron x-ray diffraction, and considered to be a nano crystalline hydrate.

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

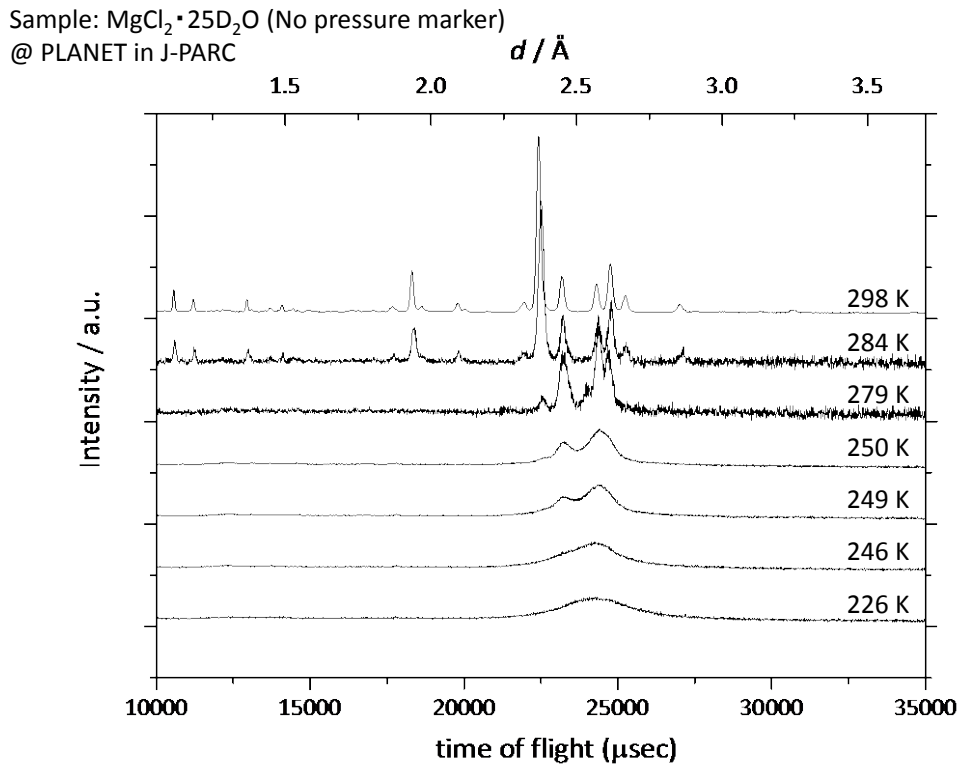


Fig. 1. Representative neutron diffraction patterns obtained at high pressure and elevated temperature. All patterns were normalized by V/Empty intensities.

Further heating up to 284 K made a new unknown crystalline phase (Fig. 1), which was also observed in our previous x-ray diffraction, and already solved its crystal structure without H-positons. We analysed this pattern using the structure model and extracted the D positions by using the difference Fourier method. Details are shown in our original paper in near future.

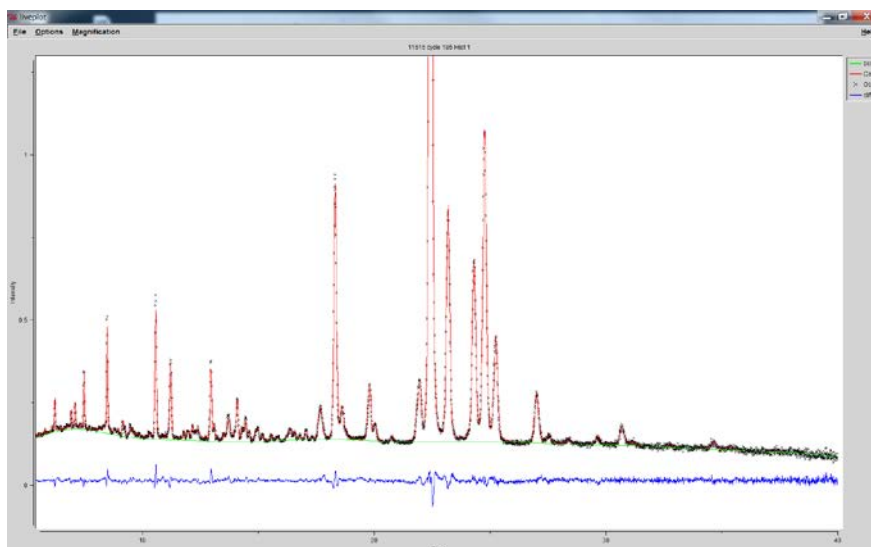


Fig. 2. Neutron diffraction pattern taken at 2.5 GPa, 298 K, and the result of Rietveld refinement.