MLF Experimental Report	提出日 Date of Report 2014/09/26
課題番号 Project No. 2009A0039 実験課題名 Title of experiment Incorporated hydride in nanoporous material C12A7-H and its reaction to UV irradiation 実験責任者名 Name of principal investigator Ryoji Kiyanagi 所属 Affiliation Tohoku University	装置責任者 Name of responsible person Toru Ishigaki 装置名 Name of Instrument/(BL No.) iMateria(BL20) 実施日 Date of Experiment 2009/12/12-2009/12/13

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.

Maynite (Ca12Al14O33) powder

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.

A nanoporous material $12CaO \cdot 7Al_2O_3$ is known as maynite and has attracted much attention because of its fascinating properties. C12A7 can incorporate various ions such as O^{2-}, OH^- and even e^- and depending on the kind of the ions taken in, C12A7 exhibits different characteristics. For example, when O^{2-} is incorporated, C12A7 exhibits high oxygen ion conductivity at high temperature, and with some special treatment, C12A7 can be a superconductor. These properties are owing to the unique structure of C12A7. The crystal structure of C12A7 consists of small cages with free space inside the cages. The ions incorporated in C12A7 are captured in the free inner space weakly bonding with the atoms in the cage wall. This incorporation slightly modifies the electron band structure and drastically changes its properties.

C12A7 can incorporate H^-, as well (C12A7-H). C12A7-H basically is a transparent wide gap insulator, but can be transformed into a conductor by the irradiation of UV light. Although this characteristic is expected to be applied to actual devices, the mechanism of this phenomenon has been barely understood, including the position of the H^- ions. Therefor, neutron powder diffraction

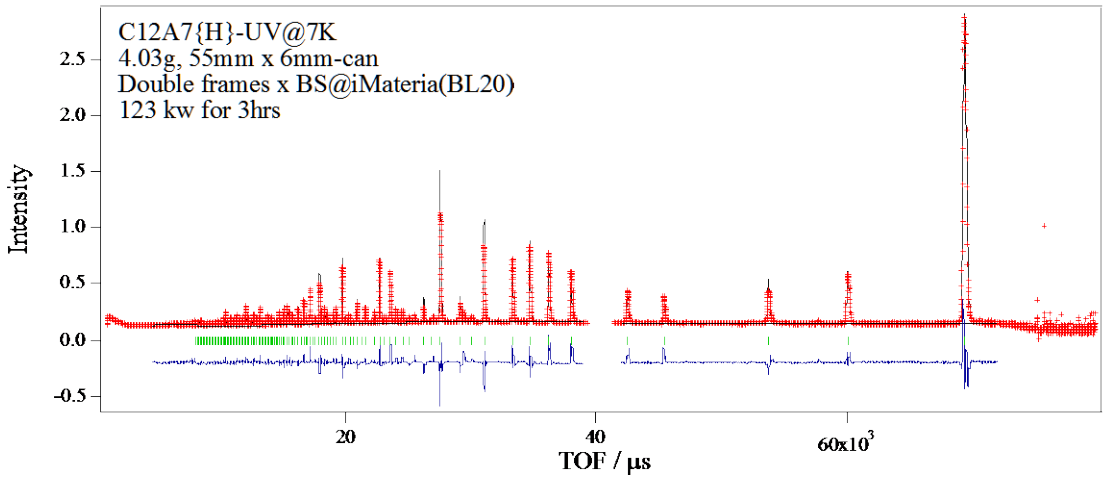

Fig. 1 Obtained powder diffraction pattern and the result of the Rietveld analysis.

measurements were performed in order to investigate the precise crystal structure of C12A7-H and its UV-irradiated one.

The samples were prepared by the solid state reaction of CaO and Al_2O_3. The obtained material in a powder form was treated in H_2 atmosphere at high temperature to make C12A7-H, which was afterward UV irradiated for several days.

The neutron powder diffraction experiments were performed on iMateria (BL20) at MLF. The samples were sealed in vanadium cans and they were attached onto a cryostat. The diffraction data were collected at several temperatures from 7 K to 300 K.

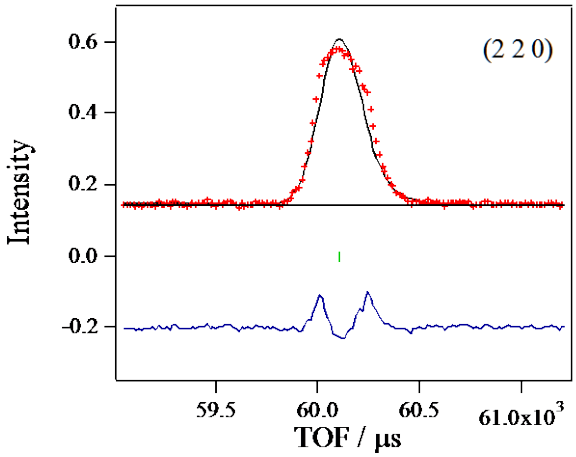

Fig. 2 Blowup of (2 2 0) reflection profile. Red cross: observed data, black curve: calculation, blue curve: desiduals

Figure 1 shows the obtained powder diffraction pattern of UV-irradiated C12A7-H and its Rietveld fitting. All the peaks were indexed by the same unitcell as C12A7, indicating no contamination of impurities. However, the Rietveld analysis could not fit the peaks well. By a close look of the profiles, it was found that the peaks were slightly split into two peaks. As is seen in Fig. 2, the peak shape is a bit broader compared with a standard profile, and the residuals between the observed data and the calculation curve show a two-peaks feature. This most probably is due to inhomogeneity of UV irradiation over the sample. Considering the lattice parameter of C12A7, the peaks with larger d values are the peaks from the UV-irradiated C12A7-H, implying some sort of relaxation of the framework structure of C12A7 due to the release of an electron from H^- ion.