MLF Experimental Report	提出日 Date of Report 17 June 2011
課題番号 Project No. 2010A0049 実験課題名 Title of experiment Research on conduction process of high ion conductors by neutron diffraction 実験責任者名 Name of principal investigator Dyah Sulistyanintyas 所属 Affiliation Ibaraki University Frontier Research Center for Applied Atomic Sciences	装置責任者 Name of responsible person Prof. Toru Ishigaki 装置名 Name of Instrument/(BL No.) BL-20 iMATERIA 実施日 Date of Experiment 17 December 2010 AM 10 ～ 18 December 2010 AM 10

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.

Copper Iodide (CuI), powder.
(γ phase at room-temperature, β phase at around 642 K～680 K, α phase at high temperature)

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.

For the sample measurement, 10.4 g powdered CuI was sealed into the vanadium tube and was placed on the cryo-furnace (CF) which is able for the measurement up to 800 K. In order to confirm the γ, β, α phase of CuI, the temperature controller of CF was set from room-temperature (RT) to high-temperature (HT). Measurements were carried out by performing the Ibaraki Powder Neutron Diffractometer (iMATERIA) of the J-PARC facility and the diffraction data were then analyzed by the Z-Rietveld program.

In previous experiment, though the temperature sensor of CF has been raised up to 728 K, the neutron diffraction pattern still indicated the γ-CuI. Such as shown in Fig. 1, there was a largely difference between the temperature on the sensor and the sample. When compared to the result on the lattice constant with temperature change [1], it revealed that the highest real temperature was up to around 560 K.

In order to reduce the temperature difference between the sensor and the sample, a radiation shield was placed in this experiment. Firstly, we applied a radiation shield made from vanadium (Fig. 2a). Due to the poor thermal conductivity, then we added the copper (Cu) wires around the shield (Fig. 2b). However, because insufficient conductivity reason, we finally fabricated a radiation shield made from thick copper (Fig. 2c). Fig. 3 shows the "off-beam" result to examine the performing of each radiation shield and the smallest difference is showed by the radiation shield made from thick Cu. Result on the "on-beam" test by applied the Cu radiation shield for the standard materials (CeO_2), revealed the temperature difference around 130 K at HT (Fig. 4).

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

Based on the "on beam" result of CeO_2, the Cu radiation shield was applied therefore in this experiment with focusing to examine the β −CuI. In spite of raising the CF temperature from RT to HT, the highest temperature reached only at 775K which correspond to the real temperature of 624K before the phase transition between γ to β of CuI.. In the next work, the conduction process of the β −, α −phase of CuI will be re−examined at iMATERIA by applying the vanadium furnace which is specialized for the HT measurement up to 1000°C.

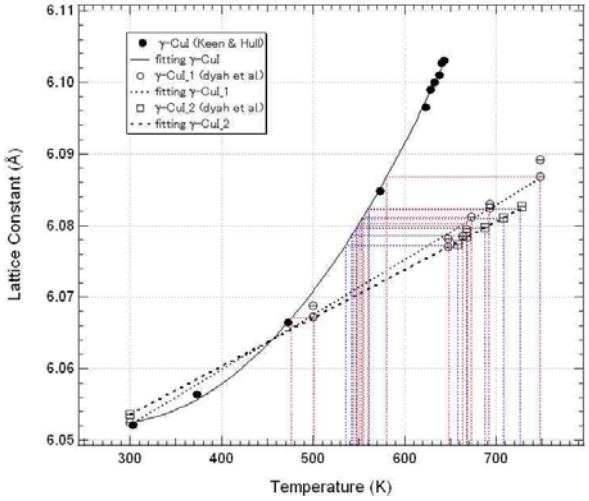

Fig. 1. The lattice constant at various temperatures of γ −CuI

[1] D. A. Keen and S. Hull, J. Phys.: Condens. Matter 7 (1995), 5793–5804.

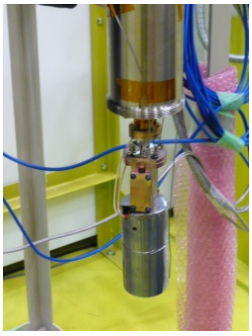

Fig. 2a

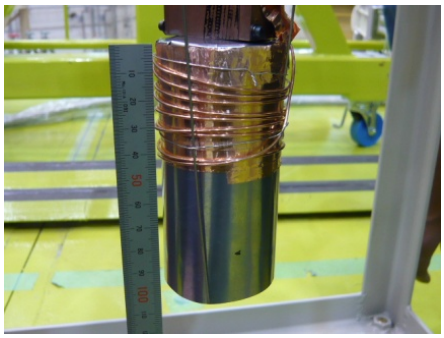

Fig. 2b

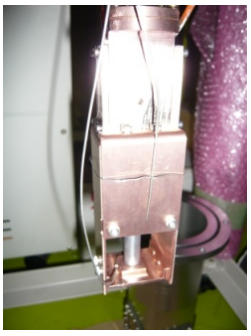

Fig. 2c

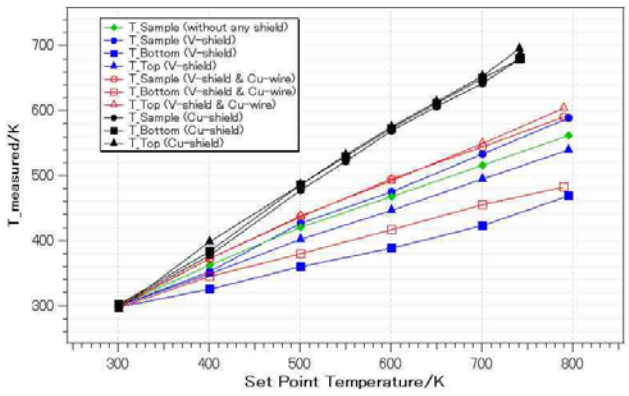

Fig. 3

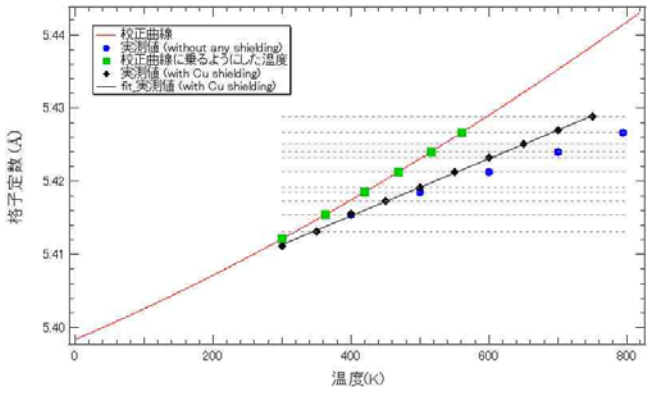

Fig. 4