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。 ）

CROSS TOKAI **Experimental Report** J-PARC	承認日 Date of Approval 2014/5/26 承認者 Approver Takashi Ohhara 提出日 Date of Report 2014/5/22
課題番号 Project No. 2013B0262 実験課題名 Title of experiment Crystal structure of Lithium ion conducting $Li_7La_3Zr_2O_{12}$ 実験責任者名 Name of principal investigator Kunimitsu Kataoka 所属 Affiliation Advanced Industrial Science and Technology (AIST)	装置責任者 Name of Instrument scientist Takashi Ohhara 装置名 Name of Instrument/(BL No.) SENJU (BL18) 実施日 Date of Experiment Feb. 26, 2014 – Mar. 3, 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.

1. 試料 Name of sample(s) and chemical formula, or compositions including physical form.

$Li_7La_3Zr_2O_{12}$ (A, small transparent and sphere-shaped single-crystal, diameter of 0.1mm)

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.

[Experiment]

A small single-crystal sample was adhered to the head of the aluminum wire of 0.1mm thickness with adhesive. Then we started the neutron diffraction measurements of a single-crystal sample. There is a stop of the accelerator unplanned, then, a single-crystal was exposed to about 96 hours. The obtained diffraction data was converted to crystal structure analysis data with various corrections. Crystal structure refinement was carried out using a computer program Jana2006 by multi data of X-ray and neutron.

[Result]

The cubic lattice parameter was determined by a least-squares refinement was a = 12.977(3) Å by single-crystal X-ray diffraction. This value was in good agreement with the reported values: a = 12.9827(4) Å. Space group was studied from extinction rule, the result, the space group was determined to be Ia-3d (No. 230). A small transparent and sphere-shaped crystal, diameter of 0.1 mm was used for the structure analysis by single-crystal X-ray and neutron diffraction.

The single crystal is too small to obtain the neutron diffraction data, but we have succeeded in this measurement. The structure refinement was initiated with the garnet framework structure finding that the La, Zr, and O atoms located at 24c, 16a, and 96h sites, respectively. Subsequently, two Li, Al and Hf sites were determined by the difference-Fourier map using X-ray and neutron diffraction data. The results, site of Li and Al were occupied 96g and 24d. Site of Hf was occupied 16a.

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

Figure 1 shows the refined crystal structure of cubic-type $Li_7La_3Zr_2O_{12}$ by single-crystal X-ray and neutron diffraction data. Future, we would carry out a further study of the crystal structure by present neutron data. We were able to refine crystal structure including Hf for the first time. In addition, it is the result of a single-crystal neutron diffraction measurements for the first time in this system.

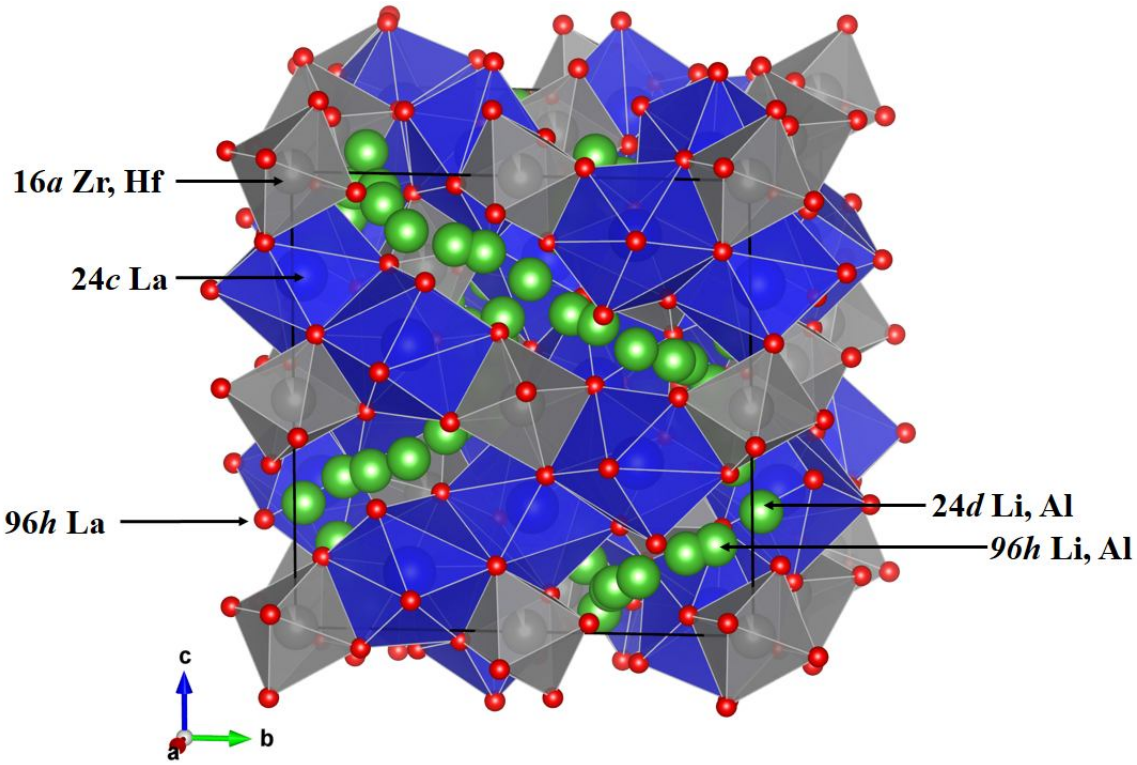

Figure 1. Crystal structure of $Li_7La_3Zr_2O_{12}$