

実験報告書様式(一般利用課題・成果公開利用)

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 Experimental Report 	承認日 Date of Approval 2016/10/11 承認者 Approver Takashi Ohhara 提出日 Date of Report 2016/10/05
課題番号 Project No. 2016A0235 実験課題名 Title of experiment Crystal structure of garnet-related type Li-ion conductor $\text{Li}_{7-x}\text{La}_3\text{Zr}_{(2-x-y)}\text{Ta}_x\text{Nb}_y\text{O}_{12}$ 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 May. 24, 2016 – May. 27, 2016

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.
$\text{Li}_{7-x}\text{La}_3\text{Zr}_{2-x}\text{Nb}_x\text{O}_{12}$ (A, small transparent and sphere-shaped single-crystal, diameter of 0.5mm)

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下さい。) 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. 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 $a = 12.9130(8) \text{ \AA}$ by single-crystal X-ray diffraction. 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 0.5 mm was used for the structure analysis by single-crystal X-ray and neutron diffraction.

The structure refinement was initiated with the garnet framework structure finding that the La, (Zr, Nb), and O atoms located at $24c$, $16a$, and $96h$ sites, respectively. Subsequently, two Li sites were determined by the difference-Fourier map using neutron diffraction data. The results, site of Li 1 were occupied $96g$ and $24d$. The refinement of site occupancy on $16a$ site used the data of X-ray diffraction. On the other hand, the refinement of site occupancy of lithium was used for neutron diffraction data. Finally, the chemical composition was determined to be $\text{Li}_{6.278}\text{La}_3\text{Zr}_{1.455}\text{Nb}_{0.545}\text{O}_{12}$.

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 $\text{Li}_{6.278}\text{La}_3\text{Zr}_{1.455}\text{Nb}_{0.545}\text{O}_{12}$ 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. It is the result of a single-crystal neutron diffraction measurements for the first time in this system.

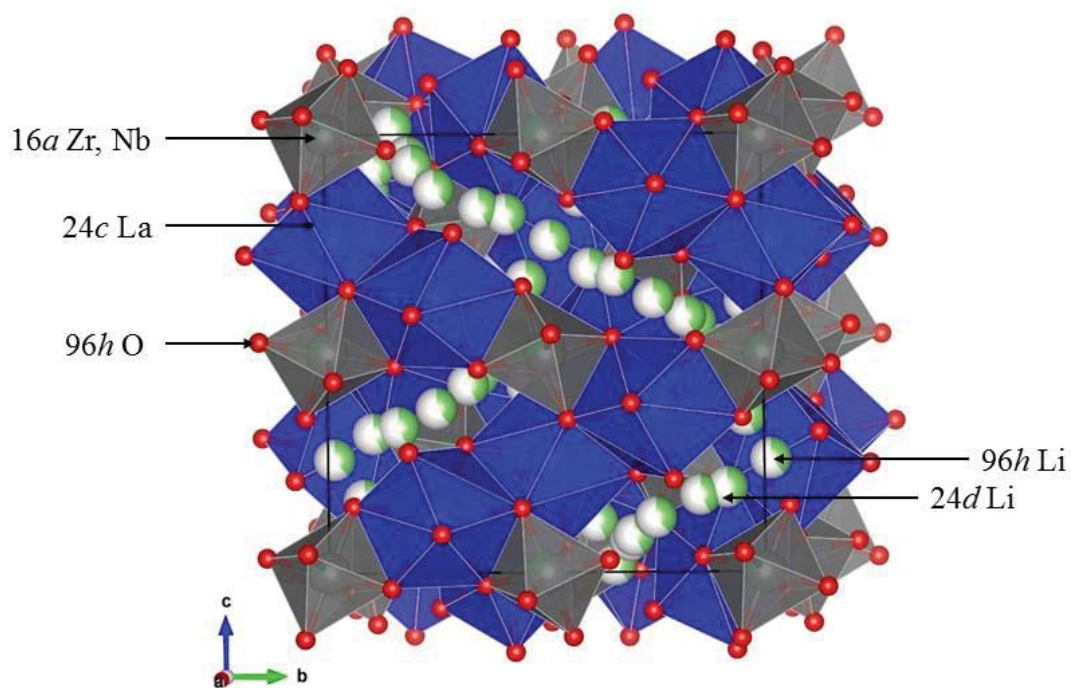


Figure 1. Crystal structure of $\text{Li}_{6.278}\text{La}_3\text{Zr}_{1.455}\text{Nb}_{0.545}\text{O}_{12}$