

 MLF Experimental Report	提出日 Date of Report 2011/7/28
課題番号 Project No. 2009B0039 実験課題名 Title of experiment Crystal structure analysis of Thio-LISICONs - Super ionic conductor for lithium batteries 実験責任者名 Name of principal investigator Ryoji Kanno 所属 Affiliation Tokyo Institute of Technology	装置責任者 Name of responsible person Takashi Kamiyama 装置名 Name of Instrument/(BL No.) SHRPD (BL No.8) 実施日 Date of Experiment 2010/6/24 - 6/25

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.

<p>1. 試料 Name of sample(s) and chemical formula, or compositions including physical form.</p> <p>Powdered $\text{Li}_{3.35}\text{Ge}_{0.35}\text{P}_{0.65}\text{S}_4$ was synthesized at 500-600 °C for 8 h in a quartz tube. $\text{Li}_{3.35}\text{Ge}_{0.35}\text{P}_{0.65}\text{S}_4$ has a new crystal structure. Peak indexing of the synchrotron XRD pattern revealed that the new phase has a tetragonal unit cell with cell parameters of $a = 8.71771(5)$ and $c = 12.63452(10)$ Å and with the extinction rule $hk0: h+k = 2n$, $hhl: l = 2n$, $00l: l = 2n$, and $h00: h = 2n$, which is characteristic of the space group $P4_2/nmc$ (137). An <i>ab initio</i> structure analysis determined the arrangement of PS_4 and GeS_4 tetrahedra in the unit cell. Synchrotron X-ray Rietveld refinements obtained using the structural model determined by the <i>ab initio</i> method revealed low agreement factors. In this proposal, we tried to determine the positions of lithium ions and the lithium content by neutron Rietveld analysis based on the structural model obtained by synchrotron XRD data analysis.</p>
<p>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。)</p> <p>Experimental method and results. If you failed to conduct experiment as planned, please describe reasons.</p> <p>Fig. 1 shows neutron diffraction patterns of $\text{Li}_{3.35}\text{Ge}_{0.35}\text{P}_{0.65}\text{S}_4$. The beamtime of 24 h was not enough to obtain the high quality diffraction data for $\text{Li}_{3.35}\text{Ge}_{0.35}\text{P}_{0.65}\text{S}_4$. We continued to measure the diffraction data using another beamtime (2010A0060) continuously set after this proposal. We will report Rietveld structural analysis results and a new crystal structure of $\text{Li}_{3.35}\text{Ge}_{0.35}\text{P}_{0.65}\text{S}_4$.</p>

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

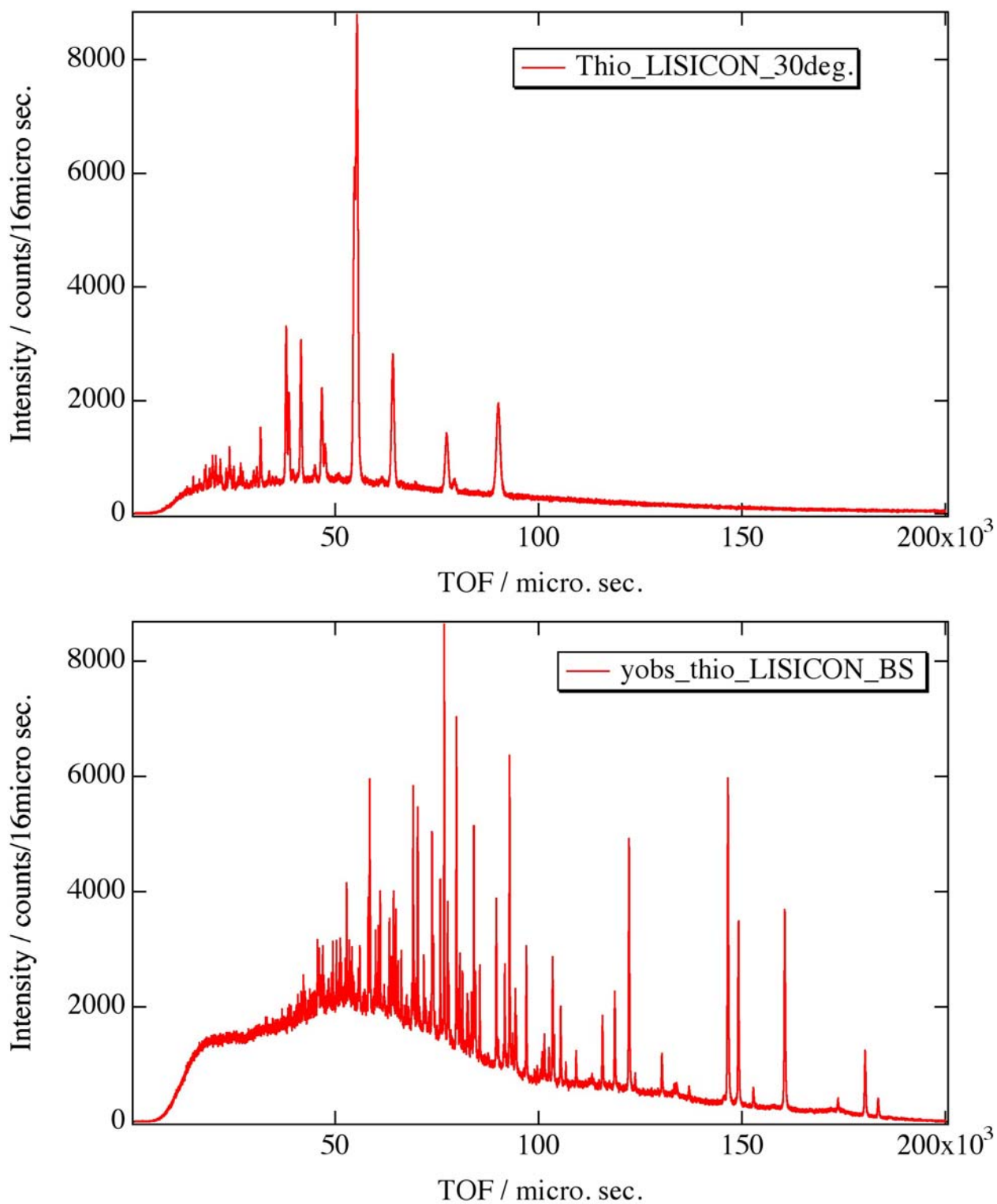


Fig. 1 shows neutron diffraction patterns of $\text{Li}_{3.35}\text{Ge}_{0.35}\text{P}_{0.65}\text{S}_4$.