


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。)

 Experimental Report 	承認日 Date of Approval 2014/6/9 承認者 Approver Takashi Ohhara 提出日 Date of Report 2014/6/8
課題番号 Project No. 2013B0100 実験課題名 Title of experiment Precise Crystal Structure Analysis of Apatite-Type Interstitial-Oxide-Ion Conductors 実験責任者名 Name of principal investigator Masatomo Yashima 所属 Affiliation Department of Chemistry and Materials Science, Graduate School of Science and Engineering, Tokyo Institute of Technology	装置責任者 Name of Instrument scientist Takashi Ohara 装置名 Name of Instrument/(BL No.) SENJU (BL-18) 実施日 Date of Experiment April 2-6, 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.
Lanthanum silicon oxide, $\text{La}_{9.7}\text{Si}_6\text{O}_{26.55}$, single crystal with size of 2.4 mm × 1.8 mm × 1.2 mm Lanthanum silicon oxide, $\text{La}_{9.33}\text{Si}_6\text{O}_{26}$, single crystal with size of 2.0 mm × 1.0 mm × 1.0 mm

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.																																																					
Time-of-Flight neutron single crystal diffraction experiments of lanthanum silicon oxides ($\text{La}_{9.7}\text{Si}_6\text{O}_{26.55}$ and $\text{La}_{9.33}\text{Si}_6\text{O}_{26}$) were carried out on the diffractometer SENJU. The single crystals were attached to an aluminum stick and mounted on the goniometer. The quality of the mounted single crystal was evaluated from a diffraction image by indexing and examination of rocking curves of the diffraction spots. Finally, the single crystals of $\text{La}_{9.7}\text{Si}_6\text{O}_{26.55}$ with size of 2.4 mm × 1.8 mm × 1.2 mm and $\text{La}_{9.33}\text{Si}_6\text{O}_{26}$ with size of 2.0 mm × 1.0 mm × 1.0 mm were selected for the measurements. The diffraction measurements were performed at the 12 different orientations without oscillation of the single crystal. Exposure times (total number of events) for each set were determined as	Table 1 : Measurement condition of $\text{La}_{9.7}\text{Si}_6\text{O}_{26.55}$ <table border="1" data-bbox="943 1576 1409 2045"> <thead> <tr> <th>No.</th> <th>$\varphi / ^\circ$</th> <th>$\omega / ^\circ$</th> <th>Total events</th> </tr> </thead> <tbody> <tr><td>1</td><td>260</td><td>260</td><td>153000</td></tr> <tr><td>2</td><td>260</td><td>265.5</td><td>153000</td></tr> <tr><td>3</td><td>260</td><td>140</td><td>153000</td></tr> <tr><td>4</td><td>260</td><td>145.5</td><td>153000</td></tr> <tr><td>5</td><td>80</td><td>140</td><td>153000</td></tr> <tr><td>6</td><td>80</td><td>145.5</td><td>153000</td></tr> <tr><td>7</td><td>80</td><td>260</td><td>153000</td></tr> <tr><td>8</td><td>80</td><td>265.5</td><td>153000</td></tr> <tr><td>9</td><td>170</td><td>260</td><td>153000</td></tr> <tr><td>10</td><td>170</td><td>265.5</td><td>153000</td></tr> <tr><td>11</td><td>170</td><td>80</td><td>153000</td></tr> <tr><td>12</td><td>170</td><td>85.5</td><td>153000</td></tr> </tbody> </table>	No.	$\varphi / ^\circ$	$\omega / ^\circ$	Total events	1	260	260	153000	2	260	265.5	153000	3	260	140	153000	4	260	145.5	153000	5	80	140	153000	6	80	145.5	153000	7	80	260	153000	8	80	265.5	153000	9	170	260	153000	10	170	265.5	153000	11	170	80	153000	12	170	85.5	153000
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2. 実験方法及び結果(つづき) Experimental method and results (continued)

they gave sufficient S/N ratio for structural analysis. The measurement condition for the single crystal of $\text{La}_{9.7}\text{Si}_6\text{O}_{26.55}$ is shown in Table 1.

Some of measured diffraction images for $\text{La}_{9.7}\text{Si}_6\text{O}_{26.55}$ were shown in Figure 1. The single crystals of both $\text{La}_{9.7}\text{Si}_6\text{O}_{26.55}$ and $\text{La}_{9.33}\text{Si}_6\text{O}_{26}$ gave good quality of spots. Indexing (determination of UB matrix) and integration of diffraction intensities were carried out using the data processing program STARGazer package. Least-squares structure refinements based on the obtained integrated intensities using the program JANA2006 gave good reliability factors ($R_{\text{int}} = 16.12\%$, $R1 = 8.60\%$) for $\text{La}_{9.33}\text{Si}_6\text{O}_{26}$. We are now carrying out further structure refinements for both data sets of $\text{La}_{9.7}\text{Si}_6\text{O}_{26.55}$ and $\text{La}_{9.33}\text{Si}_6\text{O}_{26}$, in order to discuss the structural difference between these two materials in detail. In particular, we aim to determine the precise positions, occupancy factors and atomic displacement parameters of oxygen atoms which are usually difficult to determine from X-ray diffraction data.

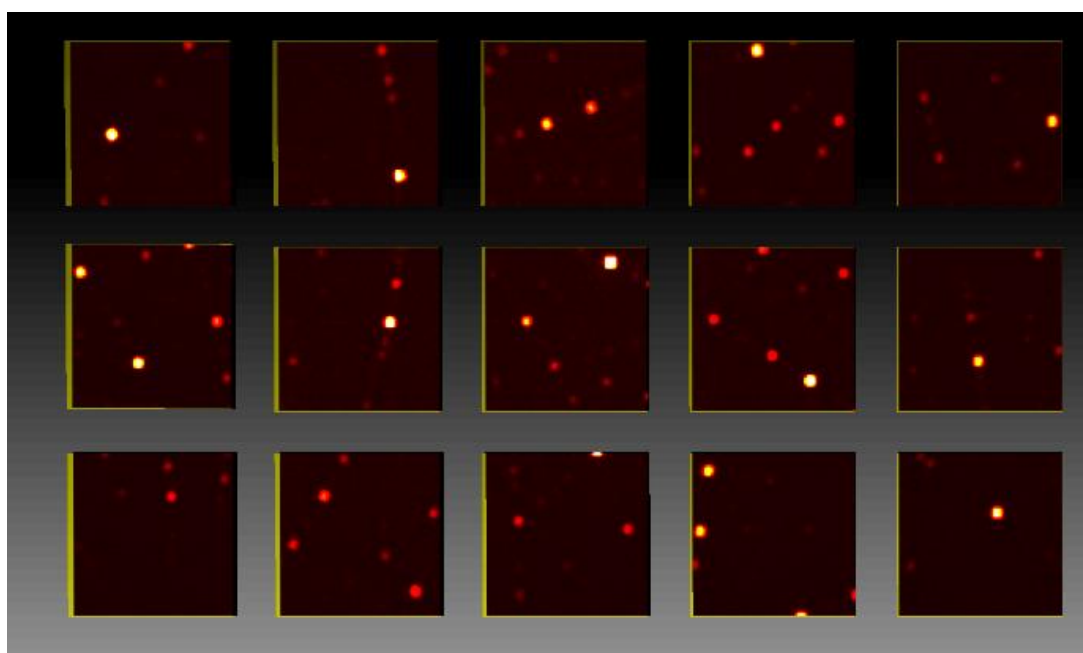


Figure 1: Measured single crystal diffraction images for $\text{La}_{9.7}\text{Si}_6\text{O}_{26.55}$.