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 MLF Experimental Report	提出日 Date of Report 2018/3/22
課題番号 Project No. 2017A0130 実験課題名 Title of experiment Pair distribution function analysis on lithium-rich transition-metal oxide after delithiation 実験責任者名 Name of principal investigator Yasushi Idemoto 所属 Affiliation Tokyo University of Science	装置責任者 Name of responsible person Toshiya Otomo 装置名 Name of Instrument/(BL No.) NOVA/BL21 実施日 Date of Experiment 2017/5/19 - 2017/5/21 2017/10/29 - 2017/10/30

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. Compositions: $\text{Li}_{1.2-x}\text{Mn}_{0.54}\text{Ni}_{0.13}\text{Co}_{0.13}\text{O}_2$ Physical form: Powder
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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. Experimental method By means of a coprecipitation method, lithium-rich transition-metal oxides with a layered structure, such as $\text{Li}_{1.2}\text{Mn}_{0.54}\text{Ni}_{0.13}\text{Co}_{0.13}\text{O}_2$, were synthesized. Phases of the samples were identified preliminarily by laboratorial X-ray diffraction measurements, and the metal compositions are determined by the inductively-coupled plasma (ICP) spectroscopy. Samples with different Li contents were also prepared. For these samples, neutron total scattering data were measured with NOVA installed at J-PARC. Powder of each sample was loaded in a V-Ni alloy container with 6 mm diameter and its total scattering pattern was measured for 12 h under ambient condition. Additional data for calibration (background, empty container and V rod) were also collected. By using the data, the obtained spectra of the samples were normalized into structure factors $S(Q)$ and then converted to reduced pair distribution functions $G(r)$. The $G(r)$ were analyzed by the PDF fitting (PDFgui).

Results

From the Rietveld analysis using Bragg profiles, it was found that the synthesized samples had a single phase of a layered structure with a space group of $C2/m$. It was also confirmed that the samples could deliver higher discharge capacities than 200 mAh/g at room temperature.

For a sample with a composition of $\text{Li}_{0.85}\text{Mn}_{0.51}\text{Ni}_{0.13}\text{Co}_{0.12}\text{O}_2$, a neutron total scattering pattern was measured. Figure 1 (a) shows $S(Q)$ derived from the pattern recorded at 45° bank. As shown in the figure, $S(Q)$ could be normalized successfully. Figure 1 (b) shows $G(r)$ obtained by the Fourier transform of $S(Q)$ with the Q range from 0.4 to 22 \AA^{-1} . The Q range was determined by considering data quality at high Q region. Although the Q range was a little bit narrow, clear atomic correlations could be observed in $G(r)$ spectrum. A peak around 1.6 \AA^{-1} seems to reflect C-C correlation in binder and/or conductive material which were used in a delithiation process, and large negative peaks around 2.0 \AA^{-1} could be attributed to Li-O and Mn-O. PDF fitting with synchrotron X-ray PDF in addition to the neutron PDF is in progress.

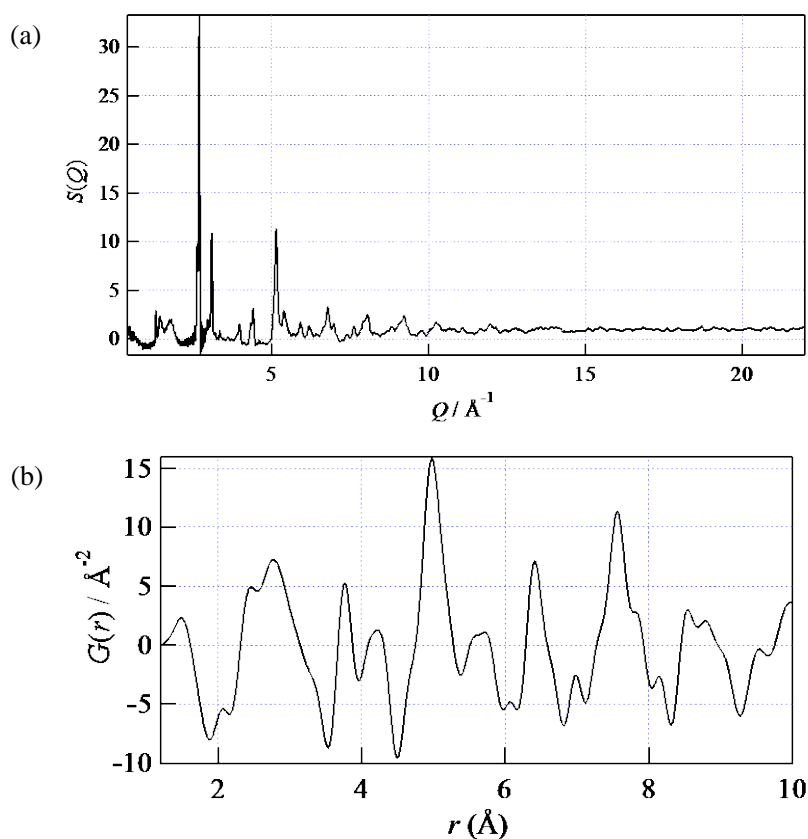


Fig. 1 (a) structure factor $S(Q)$ and (b) reduced pair distribution function $G(r)$ of $\text{Li}_{0.85}\text{Mn}_{0.51}\text{Ni}_{0.13}\text{Co}_{0.12}\text{O}_2$.