MLF Experimental Report	提出日 Date of Report 2009/8/19
課題番号 Project No. 2008A0065 実験課題名 Title of experiment Crystal structure change during charge-discharge process in Li(Mn, Ni)O_2 as a cathode active material for lithium secondary battery 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 Takashi Kamiyama 装置名 Name of Instrument/（BL No.） SuperHRPD/BL08 実施日 Date of Experiment 2009/1/27-1/28

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.

$LiMn_{1/3}Ni_{1/3}Co_{2/9}Al_{1/9}O_2$, powder

$Bi_{3.15}Nd_{0.85}Ti_{2.88}Mo_{0.12}O_{12}$, powder

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

$LiMO_2$ was prepared with a solution method using each metal hydroxide or metal acetate aqueous solution as starting materials. In the synthetic processes, the final sintering was carried out at 950 ^oC for 15 hour in air. The obtained sample was identified by powder X-ray diffraction and the metal composition was evaluated with Inductively Coupled Plasma emission spectrometer. In order to study the crystal structure in detail, we measured a neutron diffraction pattern of the sample by SuperHRPD. Powder of the sample was loaded in a vanadium can and then mounted in the apparatus. The measurement was performed in vacuum at room temperature.

Results

Figure 1 shows a neutron diffraction pattern of $LiMn_{1/3}Ni_{1/3}Co_{2/9}Al_{1/9}O_2$ measured with SuperHRPD. From this figure, it was confirmed that sufficient data to discuss the crystal structure was obtained by the measurement

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

for ca 15 hours despite the sample weight of ca 1 g. This figure also presents neutron diffraction patterns of $LiMn_{1/3}Ni_{1/3}Co_{2/9}Al_{1/9}O_2$ and $LiMn_{1/3}Ni_{1/3}Co_{1/3}O_2$, which were measured by back-scattering banks of GPPD at IPNS and HIPPO at LANSCE, respectively. Compared with these apparatuses, SuperHRPD had higher resolution, and the FWHM of the peak by SuperHRPD depended on the diffraction plane. These results demonstrate that SuperHRPD was preferable for the structural analysis of the samples.

We also carried out a preliminary measurement for $Bi_{3.15}Nd_{0.85}Ti_{2.88}Mo_{0.12}O_{12}$ ferroelectric material which obtained by solid state method, and Fig. 2 show its neutron diffraction pattern. This figure also gives a neutron diffraction pattern, which was collected by GPPD installed at IPNS, of $Bi_{3.29}La_{0.71}Ti_{2.85}Si_{0.15}O_{12}$ with the same crystal structure as $Bi_{3.15}Nd_{0.85}Ti_{2.88}Mo_{0.12}O_{12}$. The measurement time of SuperHRPD is not sufficient for detailed structure analysis. On the other hand, it is almost same resolution obtained by SuperHRPD. From the results, it is indicated that SuperHRPD is more suitable for the structural analysis of the ferroelectric material.

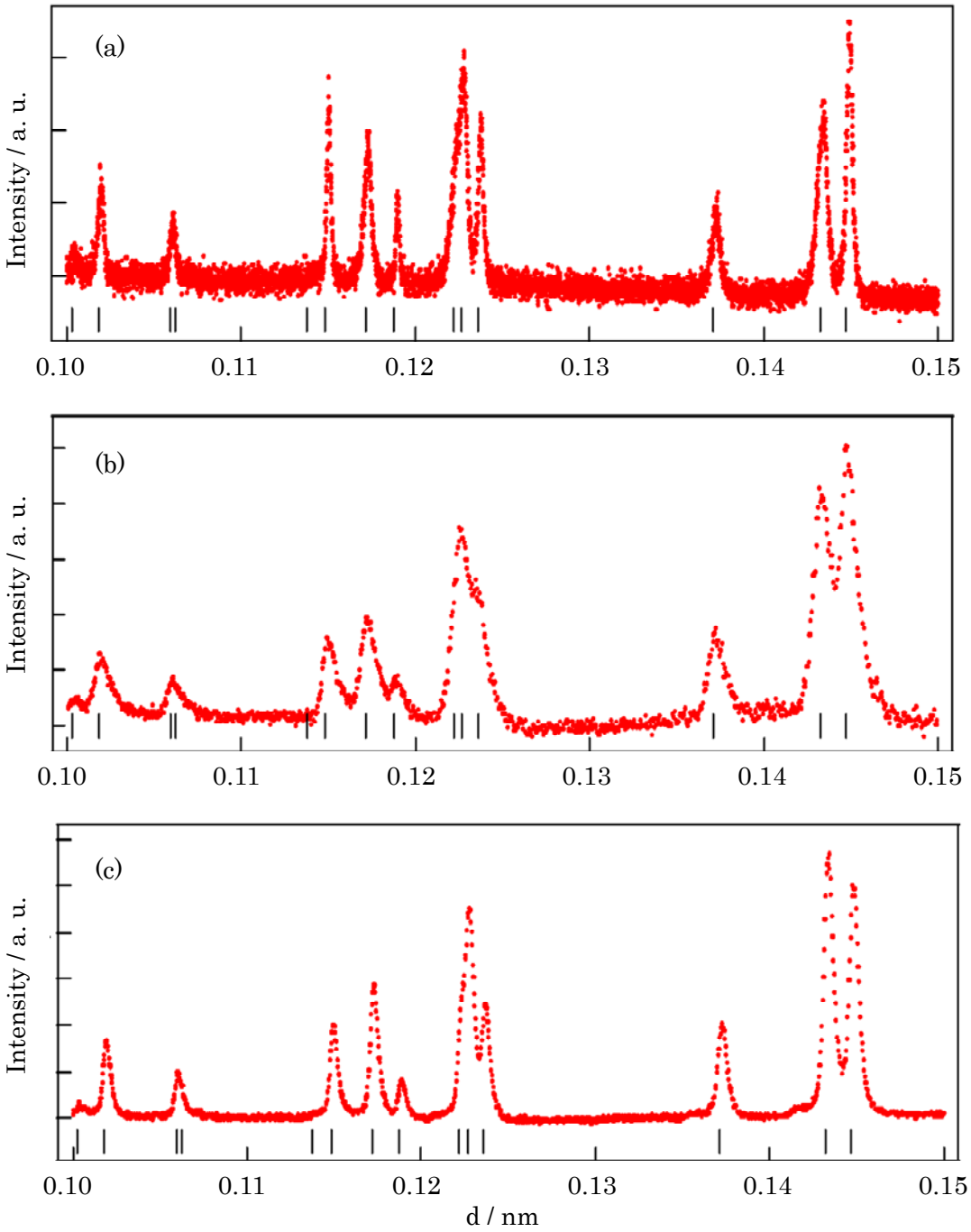

Fig. 1 Neutron diffraction patterns of (a) $LiMn_{1/3}Ni_{1/3}Co_{2/9}Al_{1/9}O_2$ [SuperHRPD], (b) $LiMn_{1/3}Ni_{1/3}Co_{2/9}Al_{1/9}O_2$ [HIPPO] and (c) $LiMn_{1/3}Ni_{1/3}Co_{1/3}O_2$ [GPPD].

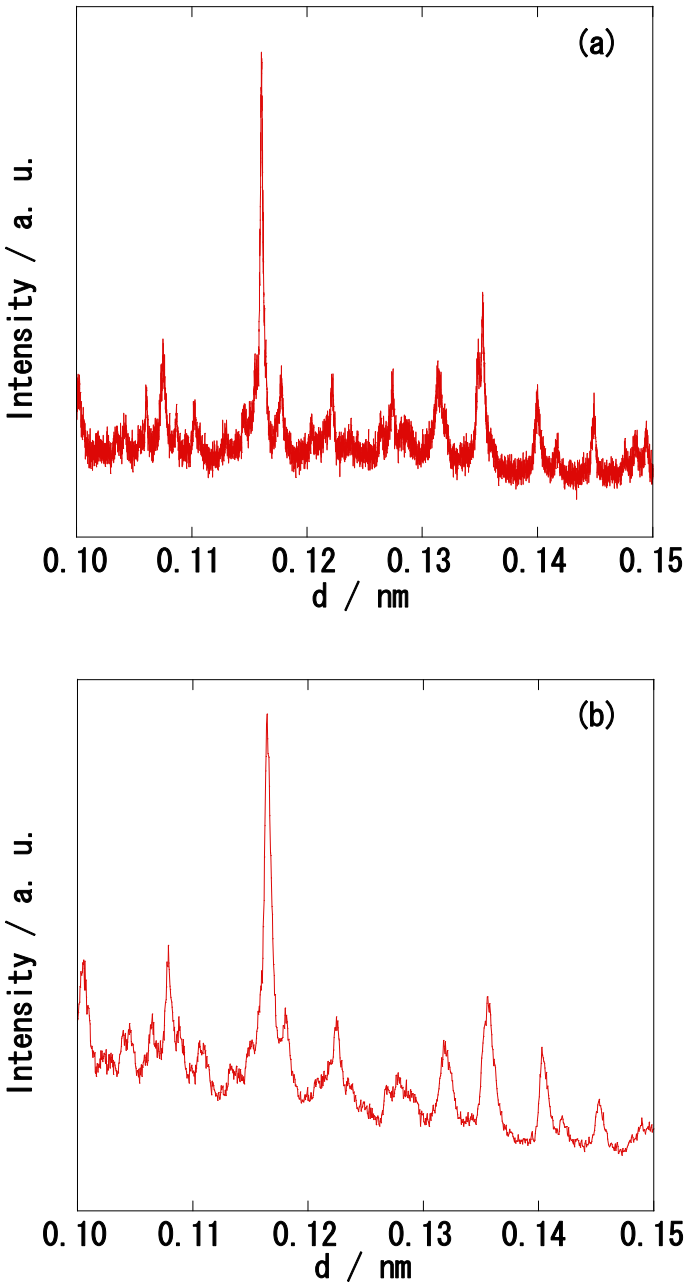

Fig.2 Neutron diffraction patterns of (a) $Bi_{3.15}Nd_{0.85}Ti_{2.88}Mo_{0.12}O_{12}$ [SuperHRPD] and (b) $Bi_{3.29}La_{0.71}Ti_{2.85}Si_{0.15}O_{12}$ [GPPD].