(※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。)

MLF Experimental Report	提出日 Date of Report 2011/06/30
課題番号 Project No. 2010B0040 実験課題名 Title of experiment BL16 High-Perfomance Neutron Reflectometer with a Hozizontal Sample Geometery 実験責任者名 Name of principal investigator Masao Yonemura 所属 Affiliation High Energy Accelarator Research Organization, Institute of Materials Structure Science	装置責任者 Name of responsible person Norifumi Yamada 装置名 Name of Instrument/(BL No.) BL16 High-Perfomance Neutron Reflectometer with a Hozizontal Sample Geometery 実施日 Date of Experiment 2011/03/08-2011/03/11

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.

Li_3PO_4(approx. 3nm)/$Li_4Ti_5O_{12}$(approx. 23nm)/STO111(Substrate)

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.

Epitaxial $Li_4Ti_5O_{12}$(LTO) thin films modified by Li_3PO_4(LPO) at the surface were synthesized on the plane (111) of a 0.5% Nb-doped $SrTiO_3$ substrate by pulsed laser deposition using a KrF excimer laser with a wavelength of 248nm and pulsed laser deposition system (PLAD131, AOV inc). The synthesis condition is skipped. An in-situ spectroelectrochemical cell designed for Neutron Reflectivity measurements provided continuous surface changes with electrochemical lithium intercalation and de-intercalation. An electrolyte solution of ethylene carbonate and propylene carbonate with a molar ratio of 1:1 and 1M -LiClO4 was injected into the cell. De-intercalation and intercalation were performed by the potentiostatic method with potentiostat/galvanostat (Invium Tech., Compactstat). Structural changes were observed by the potentiostatic method during electrochemical (de)intercalation. The reflectivity was measured as a function of the momentum transfer in a Q range of 0.07 to approx. 0.15$Å^{-1}$.

At 2010A period, the only LTO thin films were measured and observed concentration gradient at the electrolyte solution side. To investigate the more detail of structure changes at electrolyte solution, LTO thin films modified by LPO were measured. The concentration gradient was formed by the interaction between the electrode and electrolyte solution and that the modification by LPO will affect the electrolyte side structure changes.

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

Fig. 1 shows the results of in-situ neutron reflectivity measurements. Reflectivity data obtained during the electrochemical process were analyzed, but that is still not enough. The obtained value of scattering length density of LTO is 3.8x10-6. This is rather larger than $Li_4Ti_5O_{12}$'s one, $2.1x10^{-6}$. The non-modified LTO shows the scattering length density is $2.2x10^{-6}$, thus there is a possibility that the LPO affect properties of the bulk LTO. The interfacial structures are not clear. We carry forward the analysis with the other method, X-ray reflectivity and so on.

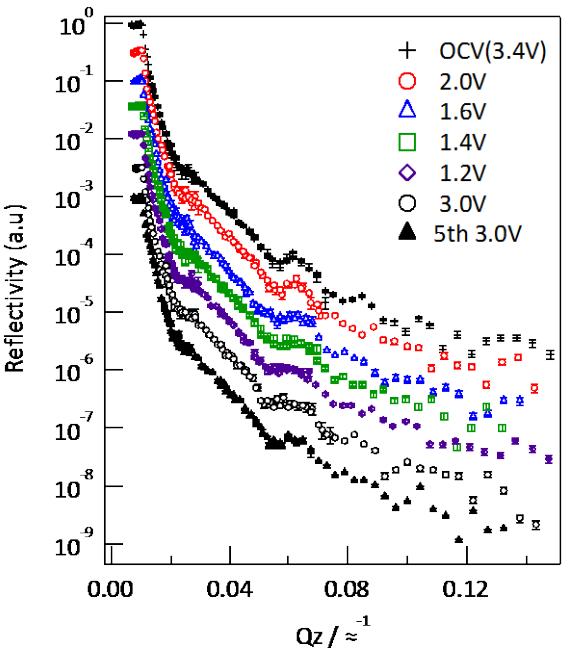

Fig.1 In-situ neutron reflectivity spectra of LTO thin films modified by LPO during the first charge/dis-chargeing ondictions.