MLF Experimental Report	提出日 Date of Report
課題番号 Project No. 2013B0104 実験課題名 Title of experiment Neutron reflectivity measurements on the ionic multilayer structures at ionic liquid|electrode interface 実験責任者名 Name of principal investigator Naoya Nishi 所属 Affiliation Kyoto University	装置責任者 Name of responsible person Norifumi Yamada 装置名 Name of Instrument/（BL No.） BL16 実施日 Date of Experiment 2014. 03/19-03/22

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.

Two ionic liquids were used as samples:

DEMEC1C1N(C10H10F6N2O5S2, liquid)

TOMAC4C4N(C33H54F18N2O4S2, liquid)

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.

Al-Ti film on a polished silicon substrate was used as conductive electrode film. The surface of the electrode film was covered by an ionic liquid (IL). The IL was sandwiched by the electrode film and another electrode film that was made on as-sliced silicon and acts as a counter electrode. Neutron beams were irradiated from the edge of the polished silicon to the Si/electrode/ionic liquid interface and the reflected neutron beams from the interface were measured.

For TOMAC4C4N|electrode interface, reflectivity profiles showed fringes whose period reflects the thickness of Al-Ti film (41 nm). The amplitude of the fringes is smaller with increasing the momentum transfer, Q, due to surface roughness on the electrode. When negative potentials was applied to the Al-Ti electrode, the amplitude of the fringes became further smaller at high Q compared with that in open circuit condition or at positive applied potentials. The same trend was observed for neutron reflectivity measurements at the DEMEC1C1N|electrode interface.

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

To explain the results described above, we used a scattering length density (SLD) model that includes Al–Ti layer and also the first ionic layer on the electrode surface between silicon and IL bulk. The thickness of the first ionic layer was fixed to be ion size (16 angstrom) and the SLD was variable. A lower SLD of the first ionic layer than that in IL bulk can qualitatively explain the small amplitude of the fringes at high Q at negative potentials. Since both of the ILs used in the present study are composed of low–SLD cations and high–SLD anions, the low SLD at the first ionic layer means accumulation of cation and depletion of anion on the electrode surface at negative potentials due to electrostatic interaction between ions and the surface negative charge on the electrode. To quantitatively analyze experimental data and to reveal further the interfacial structure of ILs at electrode surface, we will continue neutron reflectivity measurements with higher precision at the next beam time.