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。 ）

CROSS TOKAI **Experimental Report** J-PARC	承認日 Date of Approval 2017/1/13 承認者 Approver Kazuhiko Soyama 提出日 Date of Report 2017/1/10
課題番号 Project No. 2016A0040 実験課題名 Title of experiment Study of the electric double layer structure formed at the ionic liquid/electrode interface using neutron reflectivity 実験責任者名 Name of principal investigator Kazuhisa Tamura 所属 Affiliation Japan Atomic Energy Agency	装置責任者 Name of responsible person Dai Yamazaki 装置名 Name of Instrument/(BL No.) SHARAKU (BL-17) 実施日 Date of Experiment 2016/12/01-12/04

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.

D-1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)amide (*d*-[BMIM]TFSA) was used as an electrolyte. Prior to experiments, *d*-[BMIM]TFSA was dehydrated under vacuum. A Si(100) wafer was used as the working electrode. The reference and the counter electrodes were Pt wires.

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.

The electrochemical cell was assembled in a glovebox filled with Ar gas. The electrochemical cell was made from quartz and tightly sealed. The electrochemical cell was placed at the center of a goniometer under the potential controlled condition. Reflectivity were measured between Q_Z = 0.005 and 0.34 $Å^{-1}$ at E = −0.6, −1, and +1 V. The potential value of −0.6 V was the open circuit voltage (OCV). At E = −1 and +1 V, the electrode surface is negatively and positively charged, respectively. Previous studies show that cations adsorb on the negatively charged surface and vice versa. Further, ionic liquid (IL) molecules form layer structure. In this study, at E = −1 V, $[BMIM]^+$ molecules are assumed to adsorb on the Si(100) electrode surface and at E = +1 V, $TFSA^-$ molecules adsorb on the surface. At E = OCV, [BMIM]TFSA molecules may not form layer structures or may have weakly layered structure.

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

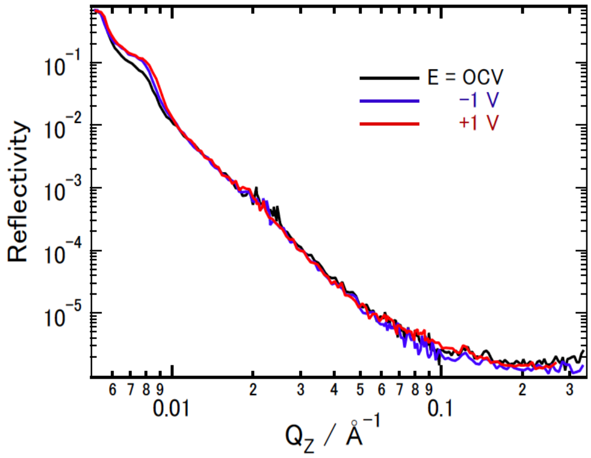

Figure 1 Reflectivity profiles measured at E = OCV, −1, and +1 V.

Figure 1 shows reflectivity profiles measured at E = OCV, −1, and +1 V. In the profiles, there observed distict oscillations at 0.006 ＜ Q_Z ＜ 0.01 Å^{-1}. They also showed the potential dependence. The fringe spacing was about 0.005 Å^{-1}, indicating that the 1300 Å −thick layer is formed at the Si(100)/[BMIM]TFSA interface. Analysis revealed that the distribution of [BMIM]TFSA molecules at the interface depends on the electrode potential. At any electrode potential, [BMIM]TFSA forms multilayer, so called electric double layer. Further, at E = −1 V, the first layer mainly consist of $[BMIM]^+$ molecules, on the other hand, at E = +1 V, it mainly consist of $TFSA^-$ molecules. It was also figure out that the larger overpotential (E − E_{OCV}) leads the thicker layered structure. The detail of the electrode potential dependence of layer structures will be studied in the future.