



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。)

 Experimental Report 	承認日 Date of Approval 2017/5/8 承認者 Approver Soyama Kazuhiko 提出日 Date of Report 2017/5/8
課題番号 Project No. 2016B0081 実験課題名 Title of experiment Study of the effect of halide ions on 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 Instrument scientist Soyama Kazuhiko 装置名 Name of Instrument/(BL No.) SHARAKU (BL-17) 実施日 Date of Experiment 2017/3/2-3/6

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.
A highly deuterated 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)amide (α -[BMIM]TFSA) was synthesized for this study and it was used as an electrolyte. Prior to experiments, α -[BMIM]TFSA was dehydrated under vacuum at 80°C. 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 Kel-F and tightly sealed. The electrochemical cell was stored in a box filled with Ar gas during measurements and placed at the center of a goniometer under the potential controlled condition. Reflectivity were measured between $Q_z = 0.005$ and 0.34 \AA^{-1} at between $E = -1.0$ and $+0.5 \text{ V}$. In the previous NR experiment, reflectivity profiles indicated that the layer structure of the [BMIM]TFSA molecules exists and it depends on the electrode potential. However, [BMIM]TFSA was only partially deuterated; therefore, the detail of the layer structure was not able to be analyzed. In this study, highly deuterated [BMIM]TFSA was used to reveal the detail structure of [BMIM]TFSA molecules on the electrode surface.

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

Figure 1 shows a NR profile measured at $E = -1.1$ V. At this electrode potential, the electrode surface is negatively charged; therefore $[\text{BMP}]^+$ molecules are assumed to adsorb on the electrode surface and a stacking structure is formed. The NR profile showed a weak oscillation. We carried out a fitting analysis and results are shown in Figure 2. A calculated profile is also shown in Figure 1. Figure 2 indicate that at $E = -1.1$ V, distinct two $[\text{BMIM}]^+\text{TFSA}^-$ pair layers exist on the electrode surface; however, the second layer is rougher than the first layer. In this study, the potential dependence of the $[\text{BMIM}]\text{TFSA}$ molecule layer structure was also observed. Detail analysis of the NR profiles is in progress.

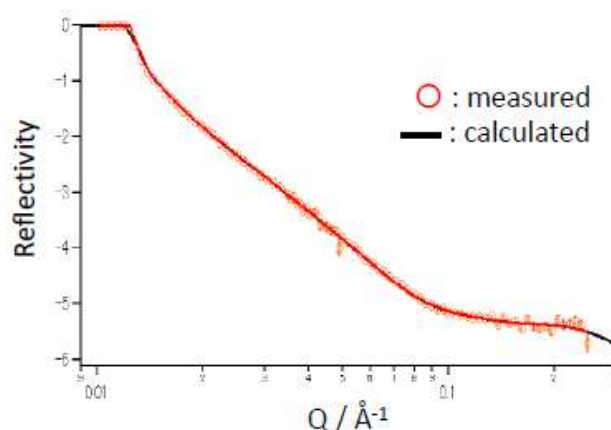


Figure 1 NR profile measured at $E = -1.1$ V

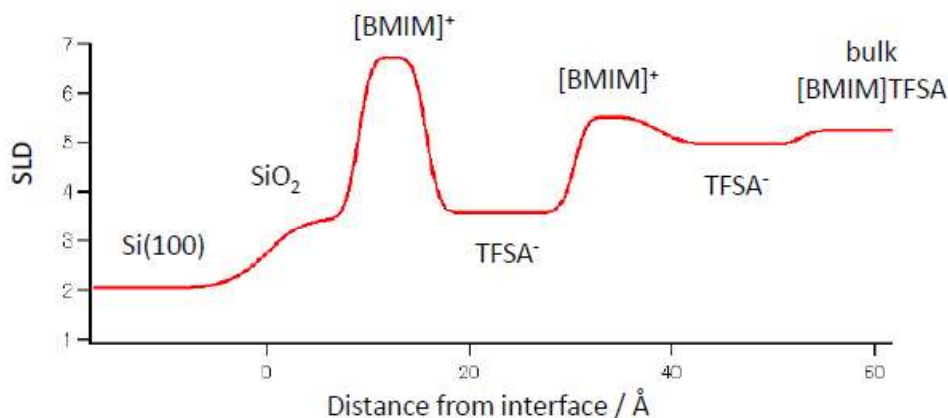


Figure 2 SLD profile measured at $E = -1.1$ V