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Small-Angle X-ray Scattering Measurement of Ionic Liquid-CO_2 Systems

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1 Introduction

Despite being salts, ionic liquids (ILs) that show liquid phase at room temperature have high CO_2 solubility and selectivity compared with gases included in the exhaust gas such as N_2, O_2, and H_2. Fluctuations, inhomogeneity of molecular distributions of the systems, provide mesoscopic structural information and are estimated by small - angle scattering methods for X-rays and neutrons. Containing heavy atoms such as fluorine, sulfur, some ionic liquids have higher X-ray absorbing power than some molecular liquids. Furthermore, in order to perform the measurement of IL-CO_2 systems at phase equilibrium without agitation, shorter exposure time is needed, which requires much higher intensity of X-ray source to perform scattering experiment.

2 Experiment

A high-pressure sample holder made entirely of titanium and a titanium alloy was designed to vary the path length precisely and perform stable operation at high temperature and pressure, while retaining a constant path length[1]. Imidazolium-based ionic liquid, 1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl) amide [C_2mim][NTf_2] was used as a sample. The measurement for IL-CO_2 mixtures was carried out at 314 K with pressures of 0.1, 1, 5, 10, 15, and 20 MPa under the process of pressure application using the apparatus at the BL-6A. Also, using samples which has different alkyl-chain length ([$C_n$mim][NTf_2] (n = 2, 4, 6, 8)) at ambient condition, operation was performed.

An X-ray beam was monochromatized to λ = 1.50 Å and the observable *s*-region was 0.02 $Å^{-1}$ ~ 0.20 $Å^{-1}$, where the scattering parameter, *s*, is defined as $4\pi\sin\theta / \lambda$ (2θ : scattering angle, λ : wavelength). *I* (0), scattering intensities at *s* = 0, directly relate to the mesoscale structural fluctuation, are evaluated from the obtained SAXS intensities.

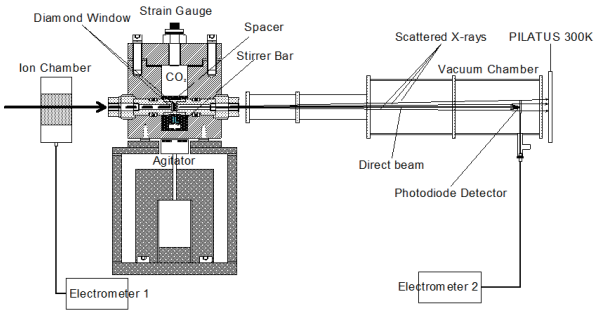

Fig.1: Experimental layout of the SAXS measurements

3 Results and Discussion

Fig.2 and 3 shows the intensities of [C_2mim][NTf_2]-CO_2 system and of [$C_n$mim][NTf_2], respectively. *I* (0), shown in Fig.4, was obtained from the extrapolation of the each plot. At the region over 10 MPa, *I* (0) got two or three times as *I* (0) under 10 MPa, implying the structural change of IL at higher pressure region. And as alkyl-chain becomes longer, *I* (0) at ambient condition showed higher value.

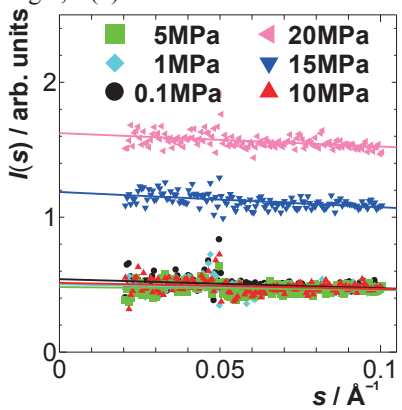

Fig.2: SAXS intensities for [C_2mim][NTf_2]-CO_2 system

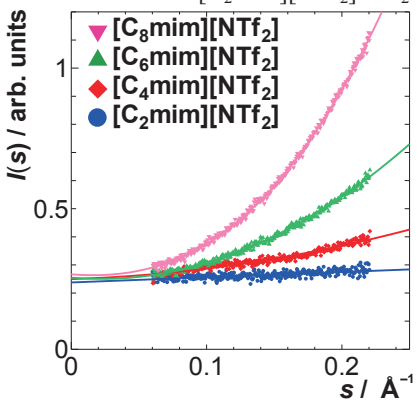

Fig.3: SAXS intensities for [$C_n$mim][NTf_2] at ambient condition

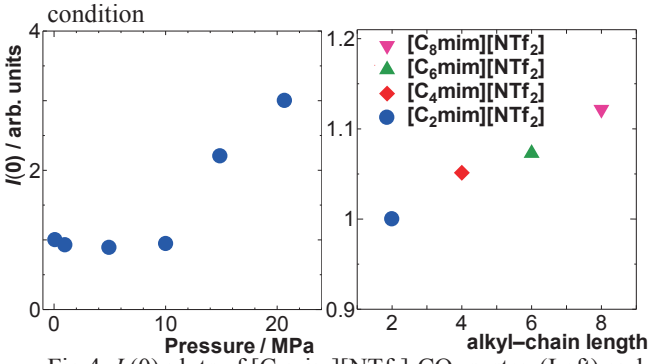

Fig.4: *I* (0) plots of [C_2mim][NTf_2]-CO_2 system(Left) and [$C_n$mim][NTf_2] at ambient condition(Right)

References

[1] T. Morita, *et al*., *Japanese Journal of Applied Physics*, **51**, 076703 (2012).

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