Materials Science

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Structural Study of Nafion Film with Cu Ion by Small-Angle X-ray Scattering utilized with Anomalous X-ray dispersion Effect

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Introduction

Nafion film is used for a separator between electrodes in a fuel cell. In the long term operation, the Nafion film absorbs metal ions from an environment and its output voltage deteriorates. In order to overcome this difficulty and improve the electric property, we should know the distribution of the metal ion in Nafon film.

Small-angle X-ray scattering (SAXS) is one of powerful tools to observe a spatial fluctuation of electron density with nano scale. However, it is difficult to find out the distribution of only a kind of atoms, such as Cu ion, with a standard SAXS method. Here, we focus attention on X-ray anomalous dispersion effect to solve this problem: The anomalous atom drastically changes its atomic form factor around the absorption edge. Therefore, it is considered that the distribution of anomalous atoms could be derived from the SAXS intensity using this effect. Based on this idea, we observed the change of the SAXS intensity of Nafion film with Cu ion by sweeping the incident X-ray energy around the Cu-*K* absorption edge.

Experimental

Nafion 117 was used for the sample. Firstly, the Nafion film was washed with HCl and ultra pure water to remove remaining impurities. Then, the film was immersed into the metal ion solution: we adopted two solutions, 0.1 M $CuCl_2$ aqueous solution (sample A) and 0.1 M $CuCl_2$ ethanol solution (sample B). Finally, the samples were encapsulated into the cell to prevent from the evaporation of the absorbed solvent.

The SAXS measurements sweeping the energy around the Cu-*K* absorption edge were performed at BL10C of Photon Factory, synchrotron light facility in High Energy Accelerator Research Organization (KEK), Tsukuba Japan. The SAXS intensities were measured in the sweep range of X-ray energy between 8.93-9.03 keV with an energy step of 10 eV. The energy of the incident X-ray was tuned with double monochromators of Si with a resolution $\delta E/E=10^{-4}$. The scattering intensity was accumulated for 600 sec at each energy scanning step: the corrections were made for absorption by the specimen and for the decay of the orbit current of the synchrotron.

Results and discussion

As shown in Fig. 1, the scattering intensities of both samples show enhancement just below the Cu-*K* absorption edge (8.98 keV). It means that the scattering contrast of the region including Cu ion becomes higher because anomalous term of atomic form factor, f', shows negative divergence at the absorption edge. It is considered that the difference of the SAXS intensities and their enhancement between two samples originates from the difference of distributions of Cu ions in the samples. The detailed analysis is now in progress.

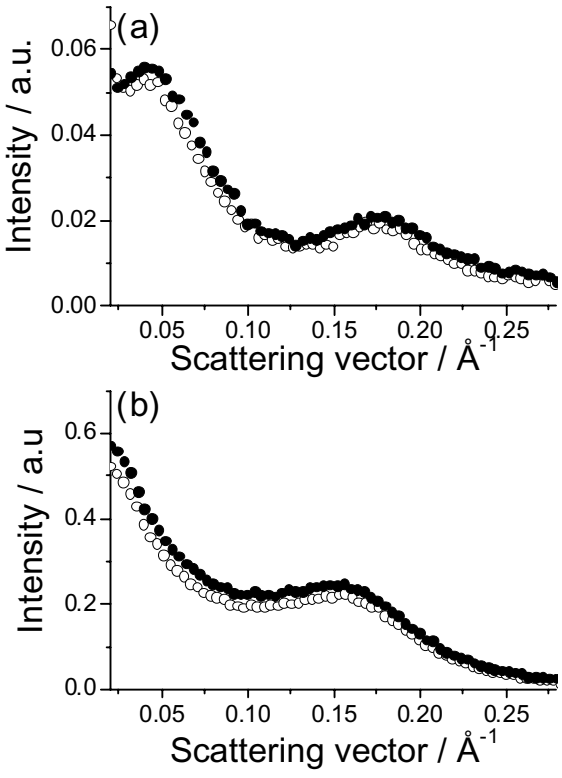

Figure 1. SAXS intensities of samples A (a) and B (b). Open and closed circle indicate the SAXS intensities at 8.96 keV and 8.98 keV, respectively. Cu-*K* absorption edge is 8.98 keV.

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