Materials Science

10C, 2004G066

# Study on Nano-Structure Formation of Organic Metal polymer by Small-Angle X-ray Scattering utilized with Anomalous X-ray dispersion Effect

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## Introduction

It is well-known that small-angle X-ray scattering (SAXS) is one of powerful tools to investigate structures with nano scale. The SAXS observes a spatial fluctuation of electron density of a sample. There are two kinds of the fluctuations: one is a density fluctuation and the other is a heterogeneous distribution of the atoms and/or molecules. However, it is difficult to distinguish between two electron density fluctuations only with a standard SAXS method. Therefore, our purpose is to find out a method to be able to distinguish these two fluctuations. Here, we focus attaint ion on X-ray anomalous dispersion effect to solve this problem: The anomalous atom changes its atomic form factor around the absorption edge. Using this feature, we could derive a distribution of anomalous atoms from the fluctuation of the electron density distribution, which is observed with the standard SAXS method. Along this line, we have made an attempt to utilize the X-ray anomalous dispersion effect in SAXS method by sweeping the incident X-ray energy around the absorption edge of the anomalous atom.

## Experimental

Dodecyl-ferrocenecarboxylic potassium (DFCP) was used for the sample with an anomalous atom, Fe. DFCP makes microphase separation between domains of alkyl chains and carboxylic potassium. The phase-separated structure is one-dimensional periodic one, lamella. It means that Fe atoms also periodically locate in the sample.

One of the features of A synchrotron light (SL) is X-ray source with wide wavelength range. Therefore, SL is a suitable X-ray source of our SAXS method with energy sweep. The SAXS measurements sweeping the energy around the Fe-*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 7.09-7.13 keV with an energy step of 20 eV. The energy of the incident X-ray was tuned with double monochromatores of Si with a resolution $\delta E/E=10^{-4}$. The scattered X-ray was detected with a one-dimensional detector and the 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

In most of the SAXS profiles of DFCP, a peak was observed around $2.0\times10^{-1}$ Å$^{-1}$, indicating that there exists a periodic structure with an interval of 31.9 Å. This value shows a good agreement with the published datum. The lamellar structure could be formed by the microphase separation between alkyl chains and carboxylic acid. Therefore, Fe atoms in ferrocene molecules also locate periodically with the same interval of the lamella.

Figure 1 shows the energy dependence of peak profiles by energy-sweep SAXS experiments. As you can see, the peak almost disappears at the Fe-*K* absorption edge (7.11 keV). It means that a negative anomalous atomic form factor f' does not ignore at the absorption edge. This result also indicates that we can control the contrast of sample for X-ray by sweeping the energy of an incident X-ray.

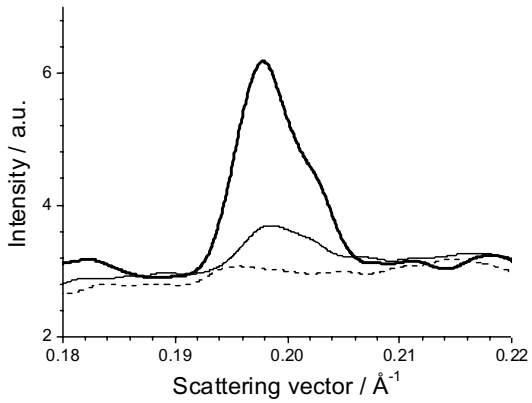


Figure 1. Energy dependence of of SAXS peak profiles of DFCP around the Fe-*K* absorption edge (7.1109 keV). The thick, thin and dot lines indicate the profiles of peaks observed with incident X-ray energies of 7.09, 7.11 and 7.13 keV, respectively.

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