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Structural changes of amphiphilic monomer and it's polymer observed by the simultaneous DSC-XRD

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Introduction

Amphiphilic monomer consisted with alkyl ammonium having two or three long alkyl chains and styrene unit forms high ordered self-assemble structure[1]. This self-assemble structure was fixed into polymer film by radiation of low energy electron beam or x-ray. The phase transition behavior and structural changes of amphiphilic monomer and its polymer were investigated by the simultaneous DSC-XRD and DSC-FTIR methods.

Experimental

Styrene monomer derivative which contains two long alkyl chains obtained by the reaction of chloromethyl styrene and N,N-di-n-octadecylmethylamine ($A_2$18) was used as the amphiphilic monomer ($M_2$18). $M_2$18 which consists of unsaturated double bond and self-assemble structure was self-aligned on various substrates and polymerized by irradiating low energy electron beam under the condition of 150 kV and 15 mA with the total dose of 300 kGr. ($P_2$18).

The phase transition behaviors of $A_2$18, $M_2$18, and $P_2$18 were investigated by DSC, WAXRD, the simultaneous DSC-FTIR, and DSC-XRD methods. The DSC-XRD instrument was setting on SAXS optics at BL-10C, PF, KEK. The wavelength of X-ray was 0.1488 nm monochromated by double Si crystals. The size of X-ray was 0.6 x 0.6 mm. The distance between sample and PSPC was 450 mm, which covered $1.5 < q = 4\pi \sin\theta / \lambda < 8.5$ nm^{-1}. DSC-XRD measurement was carried out at 2 Kmin^{-1}. The time resolution of a XRD profile was 30 sec.

Results and discussion

The obtained polymer film showed the highly ordered anisotropy composed of the stacked bilayer structure along film thickness. From ATR-IR observation, di-octadecyl chains existed on the film surface faced to the hydrophobic substrate, the other surface contained mainly polystyrene backbone structure. These results suggested that the self assemble monomer structure was fixed in the obtained polymer film. The anisotropic order of the obtained polymer depended on the substrate. The phase transition behaviors of $A_2$18, $M_2$18 and $P_2$18 were measured by the simultaneous DSC-XRD method.

Four types of orders, such as conformational and internal rotation of long alkyl chains, short range order and bilayer orders were observed for $A_2$18, $M_2$18 and $P_2$18.

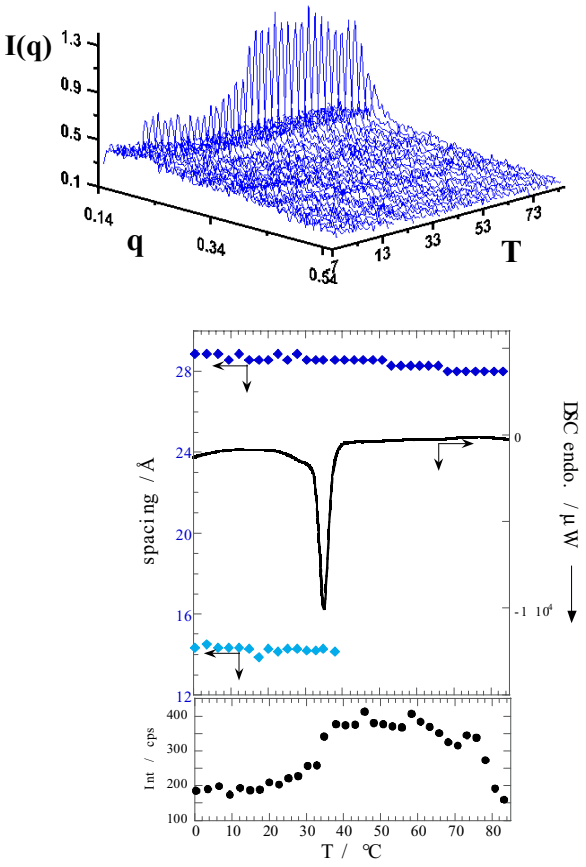

Fig.1 Stacked XRD profiles (upper), bilayer distance (middle) and diffraction intensity (lower) for $M_2$18 measured by DSC-XRD on heating

The DSC-XRD results for $M_2$18 heated at 2 Kmin-1 was shown in Fig.1. The diffraction peaks of bilayer structure (1, 1/2, 1/3) are observed and the high-order diffraction peaks are vanished at 35 °C corresponding to the melting of alkyl chains. However, the bilayer structure remained to 80 °C. For A218, the bilayer structure disappeared immediately after the melting of alkyl chains. The bilayer structure of $P_2$18 was remained until the thermal decomposition temperature.

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