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Effects of μm-Size Particle on Microphase-Separated Structure of Block Copolymer

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1 Introduction

A block copolymer with incompatible components forms well-ordered microphase-separated structure on a scale of nm. There have been many studies on block copolymers with organic or inorganic nanoparticles for controlling distribution of the particles in the microdomain. However, the larger particle is expelled from the microdomain due to the loss of conformational entropy of block chain.

In this study, the effect of μm-size large particle during solvent cast process on phase-separated structure of block copolymer was investigated by using small-angle X-ray scattering (SAXS).

2 Experiment

The block copolymer used was poly(styrene-*b*-isoprene), SI, purchased from Polymer Source Inc., with number-averaged molecular weight of 223×10^3 and polydispersity index of 1.07. Its volume fraction of polystyrene block chain is 0.61 in a molecule. The polymer particle synthesized by cross-linking divinylbenzene (Sekisui Chemical Co., Ltd.) was used. Its diameter and density are 3 μm and 1.19 g/cm^3, respectively.

The dilute solution of the block copolymer with different particle content was prepared in toluene. The film specimens were prepared by solvent casting, and then annealed at 150˚C for 24 hours in a vacuum. The dispersion of particles in the solution are not stable due to the difference in density from toluene, so that most of the particles aggregated in the lower part of the film.

SAXS measurement was conducted on BL-6A. The wavelength, λ, of X-ray was 0.154 nm, and the camera length was set at about 2.6 m. PILATUS 300K (Dectris) was used as a detector. SAXS was observed in the edge-view geometry, in which X-ray is irradiated along the direction parallel to the film surface.

3 Results and Discussion

Fig. 1 shows a two-dimensional SAXS pattern from SI with the particle content of 5 wt% in the edge view. All the samples exhibited anisotopic scattering pattern like the one shown in Fig. 1 irrespective of the particle content. Thus, the scattering intensity profile, $I(q)$, as a function of the scattering vector, q, was obtained by sector-averaging the scattering pattern at ±5˚ around the specific azimuthal angle, ϕ. Fig. 2 compares $I(q)$ profiles obtained along the direction of ϕ=0˚ for the samples with different particle content. All the profile showed several Bragg peaks at the position of nq_1, in which n is the integer and q_1 is the position of the first Bragg peak, reflecting lamellar structure. The lamellar domain spacing, evaluated from the Bragg peak position, for SI with the particle is basically higher than that for the pure SI. Also, SI with 5 wt% particle showed the lowest degree of lamellar orientation, $\langle\cos\phi\rangle$, along the direction parallel to the film surface, evaluated according to the previous study [1], though its domain spacing was the highest. This may be attributed to the particles at the lower part of the film.

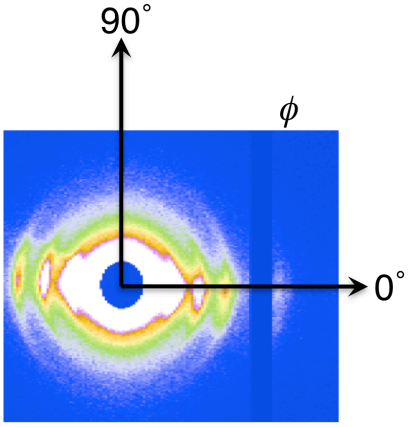

Fig. 1: A 2D-SAXS pattern from the block copolymer with the particle content of 5 wt%.

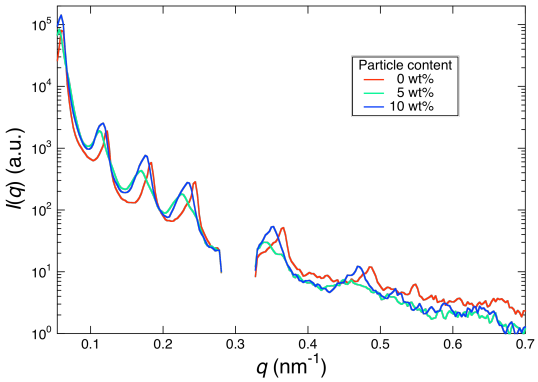

Fig. 2: SAXS profiles for the block copolymer with different particle contents along ϕ=0˚.

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Reference

[1] Y. Matsushita *et al.*, *Macromolecules* **23**, 4317 (1990).

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