Structural analysis of a thin film of an SEBS triblock copolymer forming spherical microdomains

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Introduction

Various different kinds of supermolecular structures have been found in microphase-separated structures of block copolymers. We report here an experimental result of the transformation of packing order of spherical microdomains in block copolymer thin film.

Experimental

The sample used is polystyrene-*block*-poly(ethylene-co-but-1-ene)-*block*-polystyrene triblock copolymer (SEBS) having Mn = 6.7×10^4, ϕ_{PS}=0.084 , M_w / Mn=1.04. Thick films were prepared by solution casting from a 5wt% w/v solution of the SEBS in toluene on silicon wafers. Thin films were prepared by spincoating from a 0.5wt% w/v solution of the SEBS in toluene on silicon wafers. All samples were annealed under vacuum at 140°C for 12h. Annealing is essential to obtain well-ordered spherical microdomain structures.

Results and Discussion

Atomic force microscopy (AFM) revealed that spherical microdomains ordered hexagonally on the free surface of the thin film from the 0.5wt% solution (Fig. 1) and the thick film from the 5wt% solution.

Fig. 2 shows the results of small-angle X-ray scattering (SAXS) experiments for thin films spin-cast on the cover glass. The SAXS profile for the thin film from 5 wt% solution exhibits the diffraction peaks at relative positions $1:\sqrt{2}:\sqrt{3}$, suggesting BCC (body-centered cubic) packed spheres in bulk. Thus, the free surface of the thick film from the 5wt% solution might be covered with a (111) plane of the BCC lattice. However, thus deduced lattice constant was just the half of the bulk one. It is rather reasonable to consider that the spherical microdomains at the free surface of the film are ordered in FCC lattice, giving rise to the hexagonal surface cleavage with a (111) plane of the FCC (face-centered cubic) lattice.

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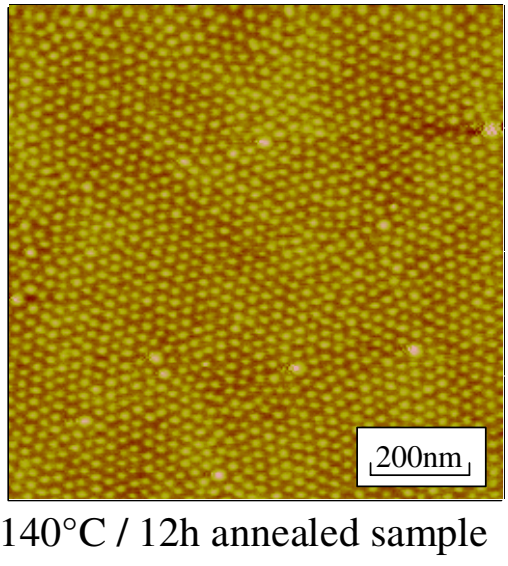

140°C / 12h annealed sample

Fig. 1 AFM tapping mode phase image.

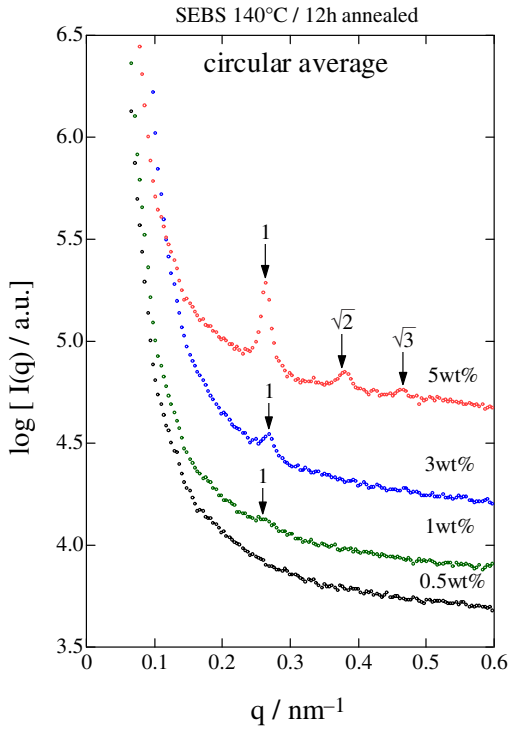

Fig. 2 SAXS profiles for thin films spin-cast on the cover glass.