Depth-resolved GISAXS of Block Copolymer Films on Si substrates

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1 Introduction
Grazing-Incidence Small-angle X-ray scattering is a useful approach to examine three dimensional nanostructures of thin soft-matter films without destroying them. In principle, it is known that depth-sensitive measurements are possible by controlling penetration depth, which may be realized by controlling angle of incidence. However, soft matter films contain light elements, resulting in smaller critical angles. This sometimes makes a problem is controlling penetration depth by choosing appropriate incident angle because surface undulation is sometimes larger than the required angular resolution.

By using a softer X-ray photon energy, it is easier to control the depth owing to larger refractive indices, both in real and imaginary parts. We examined the change in GISAXS pattern with the incident angle at the Si K absorption edge.

2 Experiment
Toluene solution of the sample was spin-cast on a spinner with a rotation speed of 3000 rpm and then annealed at 413 K to form microphase separated structure.

The film thicknesses used in the present measurements were 400 nm for thick one and 50 nm for thin one. GISAS measurements were performed at BL11B. The photon energy was chosen as 1770 eV and 1830 eV in the present measurements. The GISAXS intensities were measured with Image Plates.

3 Results and Discussion
Figure 1 shows the change of the peak width (FWHM) of the 110 diffraction peak of microphase-separated structure that was reported to have bcc structures. Calculated penetration depth of the SEBS films are shown in the figure as a function of grazing angle, assuming densities taken from some references on bulk materials and the one obtained from reflectivity measurements. The penetration depth of the incident X-ray becomes smaller with decreasing nominal angle of incidence, and the resolution may have about a couple of ten nanometers. A simple kinematical calculation of FWHM which is determined by the penetration depth qualitatively agreed with the experimental results after correction concerning refractive indices.

The Bragg peaks observed in the GISAXS profile have two components, which have different FWHM, one with relatively wide and the other relatively small in the direction perpendicular to the substrate. For the smaller angle of incidence, the former becomes dominant, and very weak diffuse scattering from surface remains for large angle of incidence, which is eventually negligible at the larger angle of incidence above 0.8 degree. In contrast, the width of the peak is almost constant for the in-plane direction, suggesting that the degree of ordering does not change much in the in-plane direction. At present, the absolute value of FWHM in the in-plane direction is strongly affected by the beam shape which makes difficult to discuss the domain size in detail.

References
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