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Dynamics of the Lamella-*Fddd* transition in SI diblock copolymer melts

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Introduction

Block copolymers that are composed of chemically different polymers connected by a covalent bond can self-assemble into various microstructures via microphase separation. Typically, the microstructures of block copolymer depend on several parameters such as the copolymer composition (f), the Flory-Huggins interaction parameter (χ), and the total number of the statistical segments (N). The phase behavior in block copolymers has also been studied extensively, both theoretically and experimentally [1]. Recently we reported a new bicontinuous microdomain morphology with the symmetry of *Fddd* space group found in a polystyrene-*block*-polyisoprene (SI) diblock copolymer with number–average molecular weight M_n = 2.67 x 10^4 g/mol, polydispersity index M_w/M_n =1.02, and volume fraction of polyisoprene f_{PI} = 0.629 [2]. In this study, we performed time-resolved SAXS experiments on the order-order transition (lamella-*Fddd*-gyroid) induced by temperature jump in SI diblock copolymer to investigate the ordering mechanism.

Experimental

We synthesized SI diblock copolymer with M_n = 2.68 x 10^4 g/mol, M_w/M_n=1.02 and f_{PI} = 0.641 via living anionic polymerization in benzene at 50˚C using *sec*-butyl lithium (Kanto Chemical Co.) as initiator. SI diblock copolymer were prepared by casting 5wt% toluene solution with 0.2 wt% Irganox and obtained by solvent casting at room temperature. Figure 1 shows the phase diagram of the IS diblock copolymer. Time-resolved SAXS experiments were performed at BL-06A in KEK, Japan by temperature jump of the sample. The X-ray wavelength and the sample-to-detector distance were, respectively, 0.154nm and 2000mm. Imaging plate was used as the detector.

Experimental

The temperature jump measurements were conducted by rapid heating SI diblock copolymer from 120˚C where we observed lamellar structure to 155˚C where we observed gyroid structures. Figure 2 shows the time-evolution of SAXS profiles after the T-jump from 140˚C to 150˚C. Before the T-jump to 150˚C, we found the peaks at q/q_m = 1, 2, and 3, where q_m is q at the first order peak, indicating that lamellar structure was formed. After the T-jump in 2000sec we found the peak at q/q_m =1, 1.22, thus, SI diblock copolymer transformed into *Fddd* structure. The time change in the peaks at 1.22 indicates that the L to *Fddd* transition may occurs via spinodal decomposition. Since there is no incubation time after temperature jump.

References

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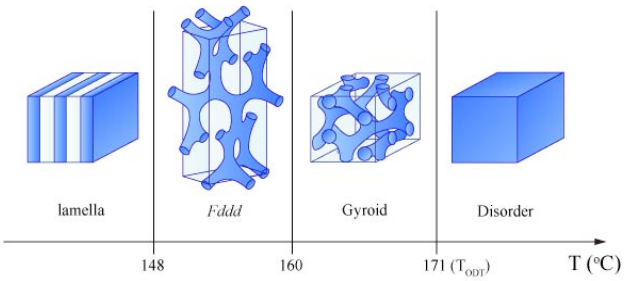

Figure 1. Phase diagram of the SI diblock copolymer.

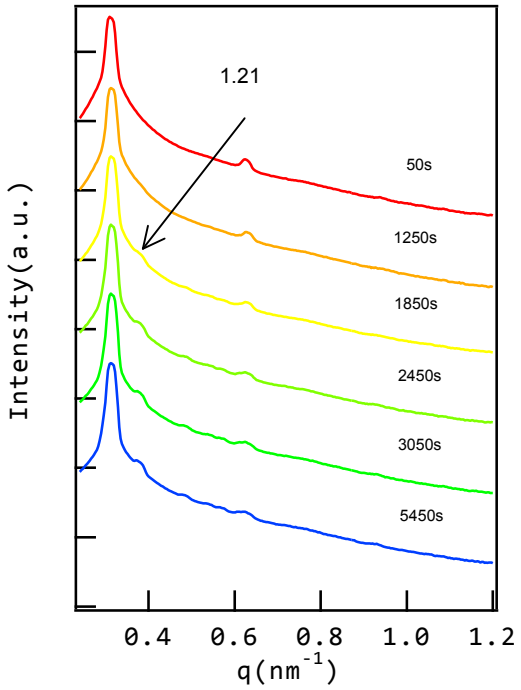

Figure 2 Time changes in the scattered intensity plotted as a function of q_m after T-jump from 140℃ to 150℃.