Crystallization process of poly(ε-caprolactone)-*b*-polybutadiene in a spherical microdomain structure

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Introduction

From recent studies, it was clear that the crystallization in the spherical microdomain structures occurs when the microdomain structure was stables enough [1, 2]. In these cases, the crystal mechanism, the crystallinity, and the crystal morphology should be different from those for the case of crystalline homopolymer because the spherical microdomain structures have disadvantages for the crystallization especially in the vicinity of the interface between the crystalline spherical domain and the amorphous matrix.

In this study, we observed the geometrical changes of the crystallized spherical microdomain structure as a function of the crystallization temperature.

Experimental

The crystalline-amorphous diblock copolymer used in this study was poly(ε-caprolactone)-*block*-polybutadiene diblock copolymer (PCL-*b*-PB) and was synthesized by a successive living anionic polymerization with n-BuLi as an initiator in vacuum (Table 1). The commercially available PB homopolymer (Mw: c.a. 3000) was blended with the PCL-*b*-PB to control the fraction of the crystalline PCL chain in the samples. The percent of PCL-*b*-PB/PB blend was fixed on 20/80 in weight.

Table 1 Characterization of samples.

Notation	Mw[1)]	PCL : PB (vol.%)[2)]
PCL-*b*-PB	8300	40 : 60
PB	c.a. 3000	—

1) Determined by VPO. 2) Calculated from ^{1}H-NMR.

The morphology of the sample was observed by SAXS with synchrotron radiation (SR-SAXS) and conventional SAXS at various temperatures. The SR-SAXS measurement was performed at BL-10C.

Results

Figure 1 shows the typical SAXS intensity profiles for the PCL-*b*-PB/PB blend sample at several temperatures. Each profile shows the scattering peak from the microdomain structure and the broad maxima originated from the form factor. The repeating period in the blend sample was determined from the peak position. And the radius of the spherical structure was evaluated from the curve fitting to the form factor of the isolated sphere.

It was confirmed that after crystallization the intensity maxima assigned to the form factor of the isolated sphere changed in the shape and position as a function of the crystallization temperature. It suggests that the shape of the spherical domain was deformed by the crystallization.

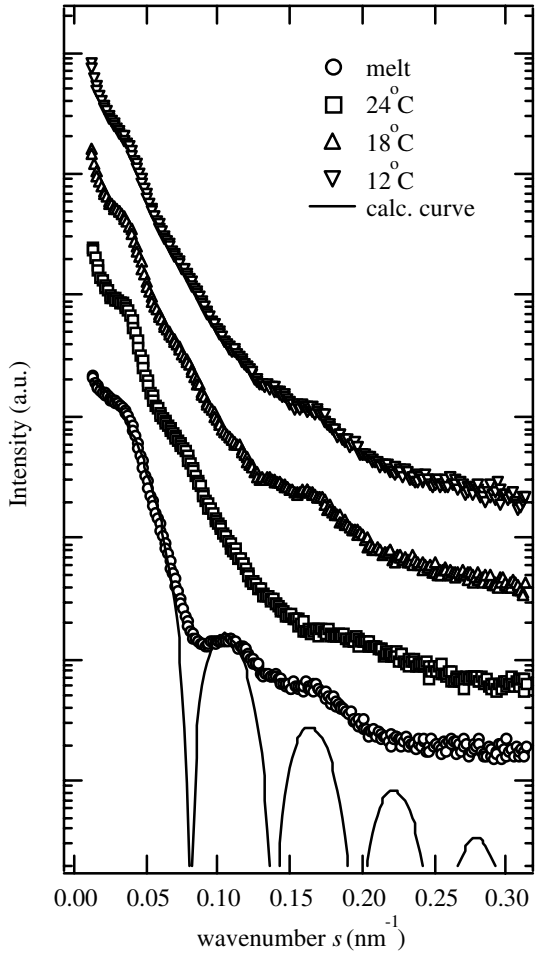

Figure 1. SAXS profiles for PCL-*b*-PB/PB blend at several temperatures. The solid curve is a calculated profile from the form factor of the isolated sphere.

References

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