Melting and Re-crystallization Process of Shish-kebab Structure

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When polymer are crystallized under elongational or shear flows, the so-called shish-kebab structure could be observed. The shish-kebab structure consists of long central fiber core (shish-structure) and lamellar crystal (kebab structure) periodically attached along the shish-structure and surrounded by the shish-structure. We studied the hierarchic structure of the shish-kebab structure using three neutron spectrometers [1].

In the present experiment, we carried out the time-resolved small angle neutron scattering (SANS) measurements on melting and re-crystallization processes of shish-kebab structure with hand-made temperature cell [2]. The initial shish-kebab structure was made from drawing blends of deuterated low molecular weight components and protonated ultra-high molecular weight polyethylene (PE) at 150 C. The molecular weight of low molecular weight deuterated and protonated ultra-high molecular weight PE (dPE/hPE) is 56,500 and 2,000,000. The SANS measurements were performed on SANS-U spectrometer on JRR-3M, JAEA, Tokai, Japan. We observed temperature change of the shish-kebab structure during the heating process from room temperature to above the melting temperature and then quenching it to room temperature.

Figure 1 shows that 2D-SANS images at various temperatures during the heating process and after quenching. In the heating process, the position of spot-like scattering parallel to the drawing direction shifts to lower angle. This result suggests the spacing of kebab structure grows larger. The kebab structure melts at 133 C. When the sample was heated up 180 C above the melting temperature, the shish-kebab structure was not observed after quenching to room temperature, but the isotropic crystal morphology was shown as in Figure 1(a). However, as seen in Figure 1(b), when the sample was molten at 136 C and then quenched to room temperature, the shish-kebab structure appeared again. These results suggest that the melt of PE blends on 180 C is isotropic, on the other hand, some " oriented structure " in melt is reserved at 136 C. For detailed analysis, we evaluated the scattering profiles normal to the drawing direction in order to clarify the shish-structure melting process. The scattering intensity in lower q-range below 0.15 Aˆ-1 vanished at about 138 C, while that of higher q-range above 0.15Aˆ-1 still remained at141 C.

References

[1] T. Kanaya et al., Macromolecules, 40, 3650, 2007.
[2] K. Nishida et al., Activity Reports on Neutron Scattering Research, ISSP, U of Tokyo, 13, 2006.

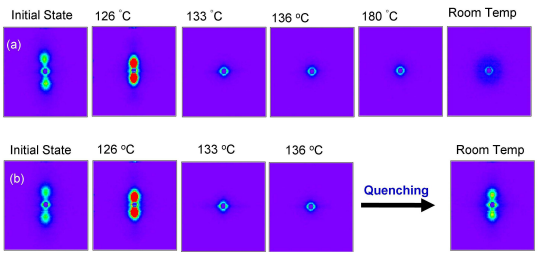

Fig. 1. Temperature dependence of 2D SANS profiles for melting and recrystallization process of shish-kebab.