Crystallization of syndiotactic polystyrene from the melt observed by the simultaneous DSC-XRD method

Gongzheng ZHANG, Takamasa KITAMURA, Hirohisa YOSHIDA*
Graduate School of Engineering, Tokyo Metropolitan University,
Hachiouji, Tokyo 192-0397, Japan

Introduction

Syndiotactic Polystyrene (SPS) forms two types of crystal modification, α-, and β-form depending on the cooling rate from the molten state. The β-form intend to crystallize in the temperature range close to the melting temperature, therefore, the β-form is obtained by slow cooling from the molten state.

In the previous work [1], we have reported that the crystallization rate was influenced by mixing with miscible polymer. In this study, the effect of mixing with atactic polystyrene (APS) on the crystallization behavior of SPS from the molten state was investigated using the simultaneous DSC-XRD method.

Experimental

Sample

SPS (Mn = 1.7 x 10^5, Mn/Mw = 2.3, 90% tacticity) supplied from Asahi Chemical Co. Ltd on July 14, 1997 was used in this study. APS (Mn = 1.1 x 10^5, Mn/Mw = 2.6) was mixed with SPS in *o*-dichrolo benzene solution. The blend content of SPS/APS was 8/2 in weight fraction.

Method

The simultaneous DSC [2] was setting on the SAXS optics at BL10C. The wavelength of X-ray was 0.1488 nm. The distance between the sample and the detector was 680 mm, which covered 0.16 nm < $Q^{-1} = (4\pi\sin\theta/\lambda)^{-1}$ < 10 nm. DSC-XRD measurement was carried out at 5 $Kmin^{-1}$, and XRD profiles were stored each 2 K.

Results

Figure 1 shows the time resolved XRD profiles of SPS/APS blend (8/2) during cooling from the molten state (300 ^{o}C) at 2 $Kmin^{-1}$ obtained by DSC-XRD. The scattering peak at Q=0.29 A^{-1} was due to the background. Two small diffraction peaks at 0.42 and 0.46 A^{-1} were observed during the crystallization at 248 ^{o}C, these peaks were assigned to the diffraction from β(020) and α(110) , respectively. Under this cooling condition, SPS formed only the β form crystal, the α form crystal appeared when SPS was cooled from the molten state at 10 $Kmin^{-1}$. In the case of SPS, the crystal growth rate of β-form at around 245 ^{o}C is expected to be faster than that of α-form. However, the crystal growth rate of β-form decreased and closed to that of α-form with the mixing of APS.

The β(020) peak appeared faster than the α(110) peak during cooling for SPS/APS blend, however, the temperature difference between the appearance of β- and α- form was only 2 K. The effect of blend content of APS on the crystal growth rate of α- and β-form are investigating using the DSC-XRD method.

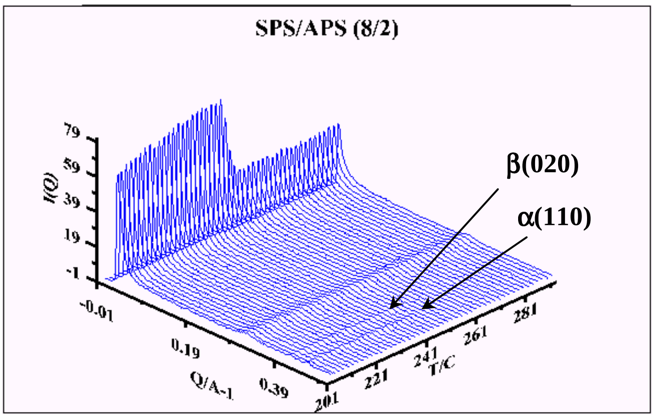

Fig. 1 Time resolved XRD profiles of SPS/APS blend (8/2) during cooling from the molten state at 2 $Kmin^{-1}$.

Not only for the diffraction peak of β(020) and α(110) but also the scattering peak from the long range ordering was observed at low Q range. The scatting peak at 0.009 A^{-1} was observed at the crystallization during cooling. The scattering maximum (Qmax) was out of scale in Fig.1, however, the Qmax shifted slightly to high Q region with decreasing temperature after the crystallization, and changed to the diffraction peak due to the long period, the regular alternating repeating of crystal and amorphous layers.

References

[1] H. Yoshida, J. Thermal Analysis, 49, 101 (1997).
[2] H. Yoshida et al., Thermochimica Acta. 264, 173 (1995).

* yoshida-hirohisa@c.metro-u.ac.jp