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Determination of lamellar twisting manner in PCL/PVB with microbeam WAXD

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Introduction

Microbeam wide-angle X-ray diffraction (WAXD) is a powerful method to investigate the micron-scale inhomogeneity of nano-structures. In polymer sciences, many polymer blend show such micron-scale inhomogeneity. In the present study, we investigate the micron-scale inhogemensity in the blend of poly- (-caprolactone) (PCL) and poly- (vinyl butyral) (PVB), which forms banded spherulite [2] as shown in Fig. 1. Previous studies with microbeam WAXD suggested that the lamellar twisting manner in PCL/PVB largely depends on the crystallization temperature [3, 4]. In this report, we show a novel method to investigate the detailed twisting manner with microbeam WAXD [5].

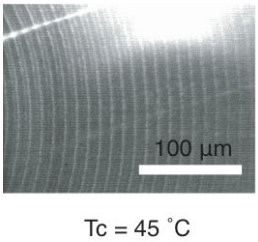

Figure 1: Polarized optical microscope image of PCL/PVB.

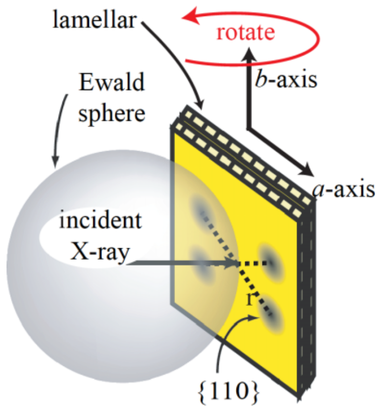

Figure 2: Schematic view of Ewald sphere and Laue spots in reciprocal space. The cross section of the sphere shell having with Ewald sphere shell yields the azimuthal distribution of 110 reflection. The twisting of lamellar results in the rotation of reciprocal space accompanied with the scanning.

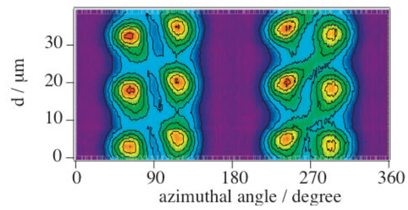

Figure 3: Intensity contour map of 110 reflection for PCL/PVB crystallized at 39˚C.

Experiment

Two-dimensional spherulite of PCL/PVB blend (95/5) was prepared. PCL (Mw: 40,000) and PVB (Mw: 100,000) supplied by Wako Chemicals ltd. was used. The sample was isothermally crystallized at 39˚C and 48˚C. The sample thickness was 70-90 µm. Detailed sample preparation scheme is shown in elsewhere [3, 5].

Scanning microbeam WAXD measurement was performed at BL-4A. The X-ray beam was focused to 4.5 µm (H) × 3.5 µm (V) at the sample postion with K-B mirrors. WAXD was measured with an X-ray CCD detector coupled with an X-ray Image Intensifier (Hamamatsu Photonics Ltd.). The x-ray wavelength was 0.83 Å and the distance between the sample and the detector was around 180 mm. We scanned the spherulite with the X-ray microbeam, along its radial direction from the center of the spherulite with a step of 1 µm. In order to investigate the detailed twisting manner, we rotated the sample around an axis perpendicular to the X-ray beam with a goniometer. By rotating the sample. The distribution of diffraction spots in three-dimensional reciprocal space is available as shown in Fig. 2.

Results & Discussion

Figure 3 shows a typical intensity contour map of 110 reflection of PCL/PVB crystallized at 39˚C. By comparing the experimental results and calculated 110 reflection intensity contour map, detailed information about lamellar twisting manner was obtained. By using this technique, we have confirmed the following facts [5]: the twisting manner in PCL/PVB banded spherulite depends on the crystallization temperature, and the lamellar which grows at high crystallization temperature shows twisting at a step-wise rate. These results will be a key to understand the origin of twisting in PCL/PVB. Furthermore the technique mentioned above is a powerful tool for the structural analysis of polymer that has micron-sacle spatial inhomogeneity.

References

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