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Structural analysis of the interface between polypropylene and rubber by microbeam wide-angle x-ray scattering

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Introduction

Polypropylene (PP) is often mixed with other polymers to improve its properties such as elasticity. In such cases, the interface is one of the critical factors that control the properties, and it is important to understand the interface structure and its formation process.

In this research, we focused on a heat-sealed film of PP and ethylene-octene-rubber (EOR). The two polymers are thermodynamically immiscible, but recent research showed that they make a 30 μm-thick interface by heat-sealing. By analyzing the interface structure of such immiscible polymers, we expect to get clues for developing a new material with improved property. In this report, we present the results of scanning microbeam wide-angle X-ray scattering (WAXS).

Sample Preparation

We used heat-sealed films of PP and EOR as the samples. We put a pellet of EOR on a sheet of PP film and heat-sealed it at 120˚C for 4 hours. The heat-sealed film was sectioned perpendicularly to the interface with a microtome. The sample was 30 μm thick.

Experimental

Microbeam WAXS was performed at BL-4A. A microbeam, which has a size of about 4 x 5 μm, was made with Kirkpatrick-Baez mirrors. The X-ray energy and the sample-to-detector distance were set to 14.3 keV and 160 mm, respectively. An X-ray CCD detector coupled with an X-ray Image Intensifier was used as WAXS detector. We scanned the x-ray beam position from PP to EOR (Fig. 1 (upper)), and observed the change of WAXS image. The μ-beam was scanned for 50 μm with a step of 1 μm.

Result and Discussion

Figure 1 (lower) shows the typical WAXS images. At PP region, a broad amorphous peak and sharp diffraction peaks are observed, while only a broad amorphous peak is observed at EOR region. The position of the amorphous peak is different in each region. At the interface, smooth shift of the position, from 1.18 $Å^{-1}$ to 1.38 $Å^{-1}$, is observed as shown in Fig. 2. By deconvoluting the beamsize of microbeam from the profile, we estimated that the thickness of the interface layer is 9 μm.

As for the diffraction peak of crystals, the center position and the FWHM of PP diffraction peaks are analyzed, which correspond to *d*-spacing and crystal size, respectively. We did not find any significant change either in d-spacing or in crystal size near the interface.

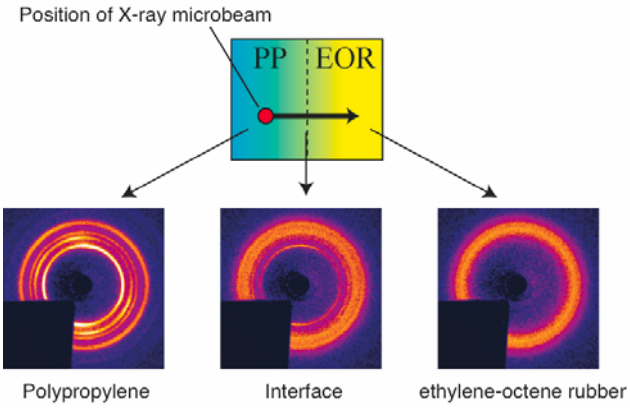

Fig. 1: Schematic view of sample (upper) and typical WAXS images (lower).

Conclusion

From the microbeam WAXS measurement, we conclude that the heat-sealed film of PP and EOR forms a 9 μm-thick interface layer. However, neither the d-spacing nor the crystal size of PP changed near the interface. This suggests that EOR diffuse not into the crystal part but into the amorphous part of PP.

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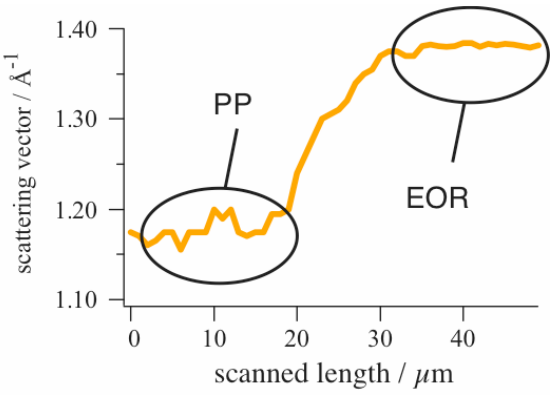

Fig. 2: The change of amorphous peak position.