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Sequential drawing behavior of isotactic polypropylene film by time-resolved WAXS/SAXS measurements

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Introduction

The sequential drawing is the deformation process of the highly oriented crystalline and amorphous chains in transverse direction perpendicular to the first drawing direction. This operation is often performed to produce isotactic polypropylene (iPP) film used in packaging and other applications. However, the structural evolution during the sequential drawing is not well understood.

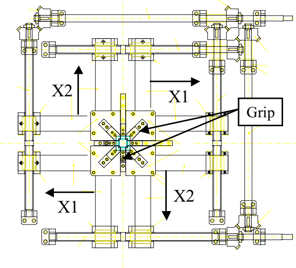
Recently, we have developed newly designed biaxial film drawing machine and have successfully applied it for *in-situ* observation of deformation behavior of iPP in a series of drawing from first draw to second draw direction.

Experimental

As a typical example, we used here a commercially available Zieglar-Natta iPP with Mw of 4.4 × 10^5 g/mol, Mw/Mn of 5.1, melt flow rate index of 2 g/10min, and a melting temperature of 160.9 °C.

The specimen (20mm×20mm×0.5mmt) was prepared by pressing at 230 °C followed by cooling to room temperature. In Figure 1, we briefly present the set up of newly designed biaxial drawing machine. Sequentially biaxial drawing tests were carried out at temperature close to melting point (160 °C) and draw rate of 1mm/sec. The specimen was at first stretched in the X1 direction and then in the X2 direction, respectively.

Wide- and small-angle X-ray scattering (WAXS and SAXS, respectively) measurements were conducted at the BL-15A beam line. For tracing the structural change during drawing, time-resolved two-dimensional (2D) WAXS and SAXS patterns were measured with 0.5 sec time slices by using a CCD X-ray detector [1].



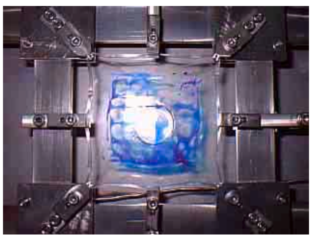


Figure 1 Newly designed biaxial film drawing machine

Results and Discussion

Figure 2 shows the time dependence of 2D WAXS and SAXS patterns observed for the sequential drawing process. The 2D WAXS pattern before drawing exhibited Debye-Scherrer rings. These rings started to change into arcs, and the reflections became more spot-like (50 sec), indicating an improvement of the *c*-axis crystallites (α-form) oriented along the first draw direction [2]. As times passed from first to second drawing process, the spot-like of the 2D WAXS started to change into the arcs (55 sec) at first. Then, the 2D WAXS pattern showed ring pattern (60 sec) followed by the arc pattern (80 sec), which indicated c-axis crystallites oriented towards the second draw direction in the second biaxial drawing process.

The 2D SAXS pattern before stretching consisted of rings. It transformed to the elliptical pattern and then showed spot pattern with the scattering maximum on the horizontal line and the streak-like pattern along the vertical line (50 sec) [2]. It is noted here that the spot and streak-like patterns behave in quite different manner in sequential drawing process. The spot was distorted into arc pattern (55 sec) in the early stage of biaxial drawing process followed by the diffuse spot (60 sec) towards the second draw direction, revealing immediate orientation of crystalline lamellae towards the second draw direction. On the other hand, the streak changed into circle (60 sec) by way of lozenge (55 sec) in the biaxial drawing process. In final stage of second biaxial drawing, weak streak along the horizontal line is recognized slightly.

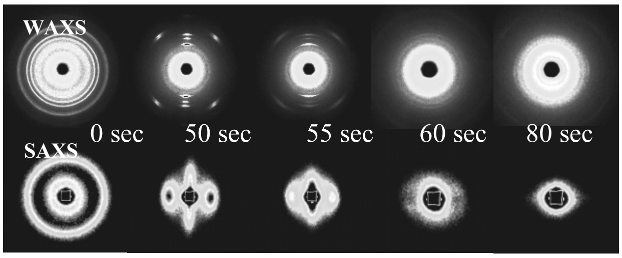


Figure 2 The time dependence of WAXS and SAXS patterns observed for the sequential drawing process. First drawing: 0–50 sec; Second drawing: 50-80sec.

References

[1] Y. Amemiya et al., *Rev. Sci. Instrum.* 66, 2290 (1995).
[2] T. Sakurai et al., *Polymer*, 46, 8846 (2005).

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