

## ***In-situ* observation of heat/draw deformation behavior of isotactic polypropylenes by SAXS measurements**

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### **Introduction**

It is well known that the drawability of *isotactic* polypropylene (iPP) is affected significantly by various factors such as drawing temperature, microtacticity and sample molecular weight (MW). Among these factors, the effect of the MW distribution (MWD) on the drawability has not yet been well understood.

The purpose of this study is to examine the effects of MWD and MW on the drawability during heating and drawing process. The structural changes during the heating/drawing process of iPP with different MWD have been investigated by using small-angle X-ray scattering (SAXS) technique.

### **Experimental**

Three types of samples were used, i.e., iPP polymerized by conventional Ziegler catalysts (zPP), the fraction insoluble in boiling heptane (BHIS) and the fraction insoluble in boiling octane (BOIS). They are different in MWD, MD and microtacticity.

The samples were cut from the sheet and shaped to the dimensions shown in Figure 1. The specimen was uniaxially stretched with a strain rate of 20 mm/min at 393K. The draw ratio (DR) was determined from the separation of marks put on the specimen.

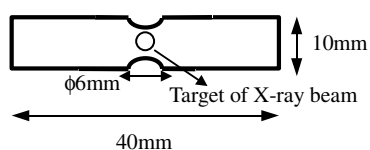


Fig.1 Dimensions of specimen

The SAXS measurements were conducted at the BL-15A beam line. For tracing the structural change in the specimen during drawing, time-resolved two-dimensional (2D) SAXS patterns were measured with 2-sec time slices by using a CCD X-ray detector [1,2].

### **Results and Discussion**

Figure 2 shows 2D SAXS patterns of zPP and BOIS at DR=6. The 2D SAXS pattern of the zPP exhibited a two-bar pattern with the scattering maximum on the meridian and the streak-like pattern on the equator. Different from the case of zPP, the equatorial scattering of BHIS was found to become weaker in intensity with increasing DR.

In case of BOIS, the equatorial scattering pattern was found to disappear with drawing.

Figure 3 shows the changes of the long period as a function of draw ratio. The long period increased linearly with the draw ratio, and this increasing rate was in order of zPP < BHIS < BOIS.

These experimental results suggest that the deformation behavior, which causes an increase in stress during the heating/drawing process, depends on MWD and microtacticity of the sample.

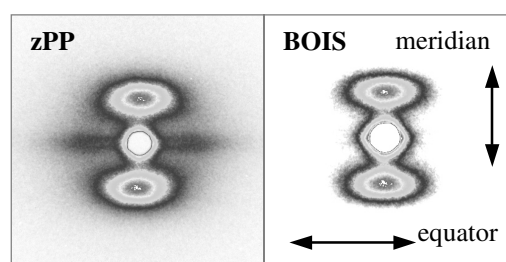


Fig.2 2D SAXS patterns of zPP and BOIS. Drawing rate =6.0

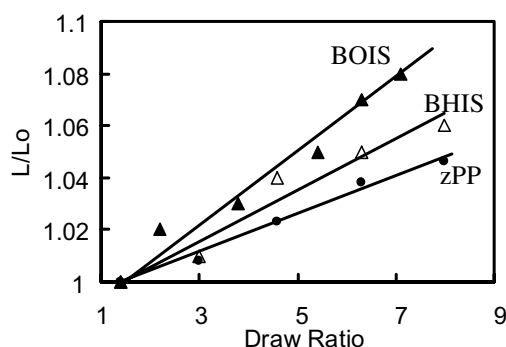


Fig.3 Changes of the long period as a function of draw ratio.

### **References**

- [1] Y. Amemiya et al., *Rev. Sci. Instrum.* **66**, 2290 (1995).
- [2] K. Ito et al., *Jpn. J. Synchrotron Rad.* Vol.13, No.5, 372 (2000).

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