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iso-ポリプロピレンの結晶化過程の観察

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1. Background and Objective

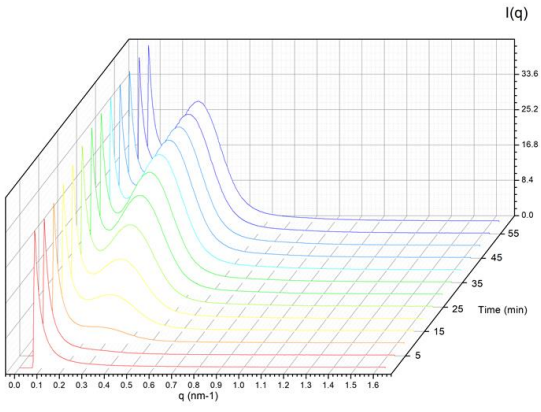
Isotactic polypropylene (iPP) has been commercially for several decades after the discovery of Ziegler-Natta (ZN) catalysts. iPP is one of the most widely used industrial plastics, as it possesses excellent thermal and mechanical properties, as well as thermal resistance. The physical properties of iPP products can be tailored by manipulating their crystal structure through the control of tacticity, molecular weight (MW), molecular-weight distribution (MWD) and copolymerization with different monomer. The main objective of this work is to study the effect of ethylene defect on the crystallization characteristics of iPP with small fraction of ethylene content using synchrotron X-Ray techniques. The crystallization kinetics and crystal morphology were investigated by *in-situ* isothermal crystallization using simultaneous synchrotron small-angle X-ray scattering (SAXS) and wide-angle X-ray diffraction (WAXD). Several quantitative parameters were extracted and analyzed in details based on the two-phase model of stacked lamellae.

2. Experiments

Five isotactics polypropylene samples with different amount of ethylene defects were used for SAXS and WAXD measurement with X-Ray of 0.15 nm wavelength. The samples were heated from the room temperature to 180°C and held for 2 min to erase thermal history. Then, they were rapidly cooled down (at the rate = 100°C/min) to crystallization temperature at 130°C untill the crystallization process were done. The isothermal measurements were started at 130 °C using 1 second explosure time with 2 seconds interval (1 measurement per every 3 second). The sample to detector distance is 2.1 m and 64.1 mm for SAXS and WAXD, respectively.

3. Results and Discussion

Preliminary results of SAXS profiles for isothermal crystallization of polypropylene samples are depicted in the Figure (the sample in this report is RPP05 containing 0.59% of ethylene defect). The results show that the induction period for the crystallization process takes about 10 min. For the sample with lower ethylene defect content (0.09% for RPP01), the induction period is decreased to only 3 min. These results suggest that the higher ethylene defect, the lower the crystalization kinetics. In addition, the crystal structure parameters were investigated using the analysis of 1-D electron density correlation function. For RPP05 sample, the average long period distance (L_p) and the average crystall lamellar thickness (L_c) are 18.36 nm. and 6.35 nm, respectively. Compared to the sample with lower ethylene content (RPP01), these parameters become larger *i.e.* L_p = 18.46 nm and L_c = 6.76 nm. These results suggest that the higher ethylene content, the smaller crystal lamellae are formed. The on-going data analysis is about the WAXD measurement which we plan to study later.



4. References

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