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# Effects of Few 1-Butene Defects on Isothermal Crystallization of Isotactic Polypropylene

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## 1 Introduction

The microstructure evolution of isotactic polypropylene-1-butene (iPPBu) and polypropylene-ethylene (iPPEt) random copolymers with 4 mol% comonomer studied by differential scanning calorimetry (DSC) and in-situ small angle X-ray scattering (SAXS) during the heating process was reported. The signal of melting enthalpy of iPPBu disappears slightly earlier than that of iPPEt, which retains steady with the decay trend of scattering intensity during the late period of melting process. Based on SAXS data during heating process, the crystal thickness of iPPBu is slightly larger than that of iPPEt. These results suggest that the melting behaviors of these copolymers rely on not only the lamellar thickness but also the crystal stability.

In this work, the effect of few 1-butene defects on isothermal crystallization kinetics of iPP were studied using differential scanning calorimetry (DSC) and in-situ small- and wide-angle X-ray scattering/diffraction (SAXS/WAXD) techniques. The results will show that iPP with few 1-butene defect crystallizes slower than neat iPP and reveal the comonomer confinement effect on iPP crystallization.

## 2 Experiment

Samples used in this study were MT12-s and MT18-s (the numbers stand for the melt flow rate (MFR) = 13.3 and 19.7 g/10 min at 230°C, respectively) and homopolymer isotactic polypropylene (V30G with MFR = 23.3 g/10 min). They were kindly provided as gifts from Beijing Research Institute of Chemistry and Industry, P. R. China. Two samples containing few 1-butene defects are denoted by MT12-s and MT18-s. The other sample (V30G) is the linear iPP homopolymer used for comparison. The specimens were prepared by compression molding at 180-190 °C to obtain thin films with 1 mm thickness. The number and weight averaged molecular weight (Mn and Mw) and polydispersity index (PDI) were characterized by gel permeable chromatography (GPC), Polymer Char GPC-IR5, using 1,2,4-Trichlorobenzene as the eluent.

The time-resolved SAXS/WAXD experiments were carried out at BL-15A2 beamline, Photon Factory, KEK (High-Energy Accelerator Research Organization) in Tsukuba, Japan. The measurements were performed for isothermal crystallization at 130°C with an exposure time of 5 s using PILATUS3 2M for SAXS and PILATUS 300KW for WAXD as a two-dimensional detector. The incident X-ray wavelength was 0.1204 nm. Polymer sample was placed in an aluminum holder (thickness = 1 mm and diameter = 4 mm). The samples were sandwiched by polyimide/Kapton film, (DuPont-Toray Co., Ltd., Tokyo, Japan). First, the samples were melted at 200°C for 5 min, and then quickly transferred to the other heater block at 130°C and then time-resolved experiments of SAXS/WAXD were performed. The raw data was corrected for background subtraction and the one-dimensional SAXS and WAXD profiles can be determined from the circular average and the sectorial average of the 2D-SAXS pattern and the 2D-WAXD data, respectively.

## 3 Results and Discussion

The content of 1-butene in MT12-s and MT18-s determined by C13-NMR are 6.43 and 5.20 wt%. The induction time is decreased and the crystallization is accelerated for iPP with 1-butene defects. Isothermal crystallization kinetics of these samples can be more studied by Avrami analysis and the results suggest that the dimension crystal growth (n) and the crystallization rate (k) tend to increase for iPP with 1-butene defects. For SAXS analysis, the electron density correlation function was used to estimate the long period (D) and the lamellar thickness (L). The D is increased for iPP with 1-butene defects (from 19 to 26 nm). In general, for iPP with 1-butene defects have higher L and D, but lower L/D ratio than L-iPP. Namely, the degree of crystallinity is lower for iPP with 1-butene defects, this result agrees with DSC, SAXS and WAXD. The diffraction peak of monoclinic γ forms can be detected in all PP samples, whereas the orthorhombic γ forms were found only for for iPP with 1-butene defects.

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