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Influence of injection rate on injection-molded β-nucleated isotactic polypropylene

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Wide-angle x-ray diffraction and differential scanning calorimetry measurements, as well as simulations, were used to investigate the crystallization properties of layered parts.

Isotactic polypropylene (iPP) is one of the most widely used thermoplastic materials[1] and can take two different crystalline forms, namely, the α- or β-phase. Most commercial iPP grades consist mainly of the α-phase crystals, which are thermodynamically stable and have good mechanical strength. However, the low impact toughness of the material limits its extensive application, especially at low temperatures. In contrast, the β-form of iPP exhibits improved elongation-at-break properties and excellent impact strength (especially at low temperatures). Nonetheless, β-phase crystals are thermodynamically metastable and are difficult to obtain under normal processing conditions. Within industry, a β-nucleating agent is thus considered a viable choice for obtaining the high β-crystal content necessary for toughening iPP.[2,3] Processing methods, however, can strongly affect the final microstructure of the β-nucleated iPP.[4,5] Indeed, the thermal and flow stresses that develop during injection molding (a commonly used processing technique for molding thermoplastics) complicate the nucleation and crystallization process and influence the microstructure of the molded component.

It is thought that the development of a layered microstructure within injection-molded iPP parts can be used as a way to better engineer the fabrication process and the final product. To that end, flow-induced crystallization in β-nucleated iPP has previously been studied under precisely controlled conditions (i.e., with the use of a Linkam CSS450 shear cell).[4,5] It has been found[4] that when the melt is subjected to a shear rate of $60 s^{-1}$ and a shear time of 5s, the β-phase crystal content decreased from 42.11 to 1.96% (which is not conducive to toughening iPP). In addition, a potential relationship between a high shear rate with a short shearing time and a low shear rate with a long shearing time has previously been investigated.[6]

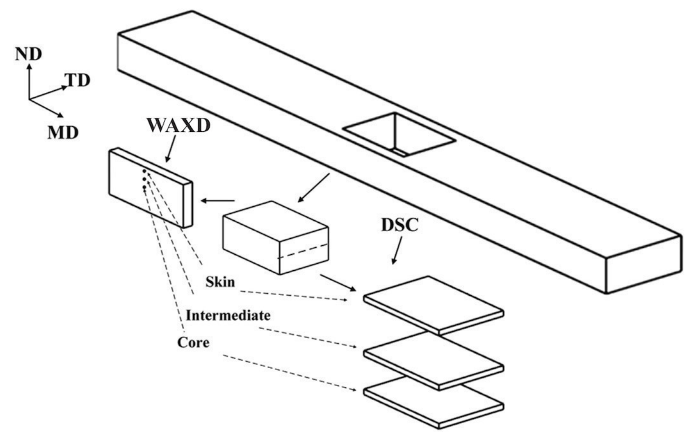

Figure 1. *Schematic diagram illustrating the different sections within the β-nucleated isotactic polypropylene (iPP) injection-molded samples, at which wide-angle x-ray diffraction (WAXD) and differential scanning calorimetry (DSC) measurements were obtained to quantify the crystallization. MD: Molding direction. TD: Transverse direction. ND: Normal direction to the MD–TD plane.*

In our work,[3] we have thus studied the effect of injection rate on the crystallization of β-nucleated iPP. For our study, we used the WBG-II β-nucleating agent (consisting of lanthanum and organic ligands), which was provided by Guangdong Winner Functional Materials Co. We characterized our samples through wide-angle x-ray diffraction (WAXD) and differential scanning calorimetry (DSC). We also used a commercial computer-aided engineering software package (Moldflow Plastics Insight 5.1 from Autodesk Inc., USA) to simulate the thermo-mechanical conditions during the filling stage. These simulations allowed us to identify the cases where extreme changes in temperature fields occurred simultaneously.

We prepared our samples with the use of a co-rotating twin-screw extruder, which allowed us to ensure a good dispersion of the

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Table 1. *Crystallinity (X) of α-phase and β-phase iPP at three positions within injection-molded bars produced at different injection rates, as measured through DSC.*

Injection rate (cm s^{-1})	40		70		85		100	
Crystallinity (%)	X_α	X_β	X_α	X_β	X_α	X_β	X_α	X_β
Skin layer	31.8	3.8	32.2	2.7	30.9	2.1	32.5	1.7
Intermediate layer	27.7	24.6	27.4	27.2	26.4	30.0	31.3	25.1
Core zone	27.8	26.0	27.7	26.1	26.98	26.3	29.0	25.1

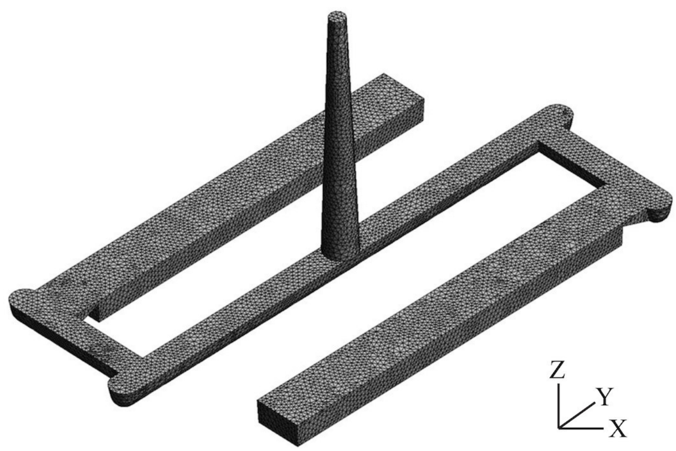

Figure 2. *Computer-aided design model and finite element method mesh used for the simulations of the rectangular impact bars.*

β-nucleating agent (0.25wt%) in the iPP. After drying the material at 90°C for three hours, we then formed pelletized granules via injection molding. We also used the injection-molding machine (Japan Steel Works J110AD-180H) to prepare standard tensile and impact bars. In addition, for our experiments, we varied the injection rates from 40 to 100cm s^{-1} (in increments of 15cm s^{-1}) and kept all other processing parameters constant.

To start, we inspected the mechanical properties of the β-nucleated iPP samples formed with different injection rates. We find that with increasing injection rate (in an appropriate range), the impact strength of the β-nucleated iPP increases, whereas the tensile strength decreases. These experimental results thus reveal that—unlike pure iPP in the same conditions—the injection rate has a significant influence on the β-nucleated iPP. Furthermore, we have quantified the crystallization of the β-nucleated iPP at different parts of the samples' layered structures (see Figure 1). Our clear crystal characterization results (see Table 1) show that both the β-nucleated and the α-nucleated iPP, in the core zone and in the skin layer, experience little change as the injection rate increases. The effect of injection rate on the intermediate layer, however, is complex. We observe that the crystallinity of the β-phase iPP increases to 30% as the injection rate increases to above 85cm s^{-1}, but then begins to decrease as the injection rate is raised further. We thus find that the shearing experienced during injection molding greatly influences the crystallization of β-nucleated iPP, and that our crystallization variation patterns are in agreement with results from similar studies.[4,5]

In the next part of our study, we used a mold-filling simulation for rectangular impact bars to model a shearing flow field and a temperature field at each layer. To improve the accuracy of these simulations, we used a dense mesh (see Figure 2). From our simulation results, we find that the temperature in each layer did not vary substantially with the different injection rates. The shear rate that the layers experience is therefore the key factor affecting the morphology and crystallization. Our simulations also indicate that the shear rate increases quickly in the intermediate and skin layers with increasing injection rates, but it varies little within the core zone. The results from these simulations therefore make it easier for us to understand the diversity of crystallization and morphology in the different layers of our molded parts.

In summary, we have used WAXD and DSC to investigate the influence of injection rate on the crystallization of injection-molded β-nucleated iPP. Specifically, we have examined the crystallization of the α- and β-phases within three layers of our molded parts. We find that with increasing injection rate, the β-phase crystal content (which is mainly responsible for the mechanical properties of β-nucleated iPP) exhibits different tendencies in the skin, intermediate, and core layers. Our results therefore provide valuable insight for controlling iPP toughness during injection molding. In the next stages of our work we plan to further study the morphology and mechanical properties of β-nucleated iPP during injection molding. We will also investigate the potential applications of our work in industrial manufacturing (e.g., for vehicles and electrical appliances).

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