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Nucleation of crystallization of isotactic polypropylene under high pressure

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Abstract in English

Within the frame of the doctoral thesis, the influence of nucleating agents (NA) on the nucleation of crystallization of isotactic polypropylene (iPP) under high pressure, in the orthorhombic γ-form was investigated. Three NAs, which nucleate under atmospheric pressure the crystallization of iPP in the monoclinic α-form were used, two commercial ones, Hyperform HPN-20E containing calcium salt of cis-cyclohexane-1,2-dicarboxylic acid, ADK Stab NA-11UH, containing sodium 2,2'-methylene-bis-(4,6-di-t-butylphenyl) phosphate, and the third one - poly(tetrafluoroethylene) (PTFE) particles with sizes of 200-300 nm. For comparison also the ability of calcium pimelate to nucleate high pressure crystallization of iPP was studied; calcium pimelate under atmospheric pressure nucleates crystallization of iPP in the trigonal β-form. iPP with the addition of Hyperform HPN-20E, PTFE and calcium pimelate and, for comparison, neat iPP were crystallized under elevated pressure, up to 300 MPa, under various thermal conditions. Crystallized iPPs, neat and nucleated, were examined using differential scanning calorimetry, X-ray diffraction as well as polarized light microscopy. In addition, during cooling under high pressure the temperature of maximum crystallization rate was determined. It was shown that the NAs, nucleating the α-form under atmospheric pressure, under high pressure nucleated crystallization in the γ-form, which was reflected in an increased content of this polymorph in the crystalline phase, a reduction of the size of polycrystalline aggregates, and in the case of nonisothermal crystallization, an increase of the temperature of maximum crystallization rate. The increase of the content of γ-form was observed for those crystallization conditions that resulted in a significant content of the α-form in neat iPP without the NAs. On the other hand, calcium pimelate, nucleating under atmospheric pressure the trigonal β-form, under high pressure was ineffective in the nucleation of crystallization of iPP in the γ-form.

The next stage of the work was focused on explanation of the mechanism of nucleation of iPP crystallization in the γ-form under high pressure by the NAs. For this purpose, iPP nucleated with Hyperform HPN-20E, PTFE and ADK Stab NA-11UH, as well as neat iPP, were crystallized under pressure of 200 and 300 MPa at different temperatures. The crystallized materials were etched and then analyzed by scanning electron microscopy. In addition, examination by X-ray diffraction as well as polarized light microscopy was carried out.

These studies evidenced that nucleated iPP crystallized in the γ-phase in the form of polycrystalline aggregates much smaller than those in neat iPP. Analysis of the lamellar structure of the nucleated iPP showed, that regardless of the nucleating agent used, first nucleation of the α-lamellae occurred, on which γ-lamellae grew epitaxially.