

UNIVERZITET U NIŠU / UNIVERSITY OF NIS
Tehnološki fakultet, Leskovac / Faculty of Technology, Leskovac

ZBORNİK IZVODA RADOVA
X SIMPOZIJUM
“SAVREMENE TEHNOLOGIJE I PRIVREDNI RAZVOJ”

BOOK OF ABSTRACTS
10th SYMPOSIUM
“NOVEL TECHNOLOGIES AND ECONOMIC
DEVELOPMENT”

Leskovac, 22. i 23. Oktobar 2013.
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CRYSTALLIZATION OF β -NUCLEATED iPP IN MODEL COMPOSITES WITH GLASS FIBRES

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The aim of this work is to analyze the crystallization of β -nucleated iPP and its crystallization in model composites with different content (10-60 %) of glass fibers, known as α -nucleators (1, 2), in order to predict the behaviour of real glass fiber/iPP composites, since the two polymorphic modifications of iPP are known to have different mechanical properties .

Melting and crystallization behavior of the polymer was analyzed by Perkin-Elmer DSC-7, in dynamical (heating rate 10 K/min, cooling rate 1, 3, 5, 10, 15 and 20 K/min) and isothermal regime ($T_c=391-409$ K) in a nitrogen atmosphere.

β -nucleator, Ca pimelate was synthesized and mixed (01 %wt.), with iPP in Brabender PL 2000 at 460K.

High content of hexagonal crystal phase of iPP in model composites was determined by the wide-angle X-ray diffraction, PLM and DSC. The k_β values estimated from x-ray diffraction patterns by Turner-Jones method was 0.94.

From isothermal DSC investigations, thermodynamical parameters of crystallization were determined. The equilibrium melting temperature (T_m^0) was 446 K, and it rises with content of glass fibers up to 452 K. Crystal fold surface energy (σ_e) was $85.4 \times 10^{-3} \text{ Jm}^{-2}$ for a polymer, and $105 \times 10^{-3} \text{ Jm}^{-2}$ for the composites. The Avrami plots were linear both for iPP and glass fiber composites, and the determined coefficients suggested that heterogeneous nucleation took place during the crystallization process. This was expected, because Ca-pimelate crystals represent a substrate for hexagonal crystal phase growth.

An attempt was made to evaluate results of nonisothermal crystallization with respect of nucleation activity. We applied the approach of Dobrev et al. (3) for calculation of Θ parameter, which represents the ratio between the work of heterogeneous and homogenous nucleation, during the crystallization in polymer systems with different additives (substrates). For the neat polymer, $\Theta = 0.34$ because Ca-pimelate is a very strong nucleator, however for the composites values of 0.43-0.46 were calculated. The content of the α crystal phase increases, with a content of the glass fibers and the temperature of crystallization for about 7%. It should be noticed that despite the α -nucleation activity of glass fibers, our investigation has shown that the β -phase is predominant in model composites with glass fibers in which β -nucleated iPP is used as a matrix.

CRYSTALLIZATION OF β -NUCLEATED iPP IN MODEL COMPOSITES WITH GLASS FIBRES

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Introduction

Isotactic polypropylene (iPP) is one of the most important commodity thermoplastic widely used in many areas because of its versatility, good physical and mechanical properties, recyclability and low cost. iPP has been studied extensively for its polymorphic characteristics and crystallization behavior, since the formation of specific crystalline form (α , β , γ or smectic) can affect the macroscopic behavior quite dramatically. The trigonal β -form is metastable and can be obtained or can become predominant under specific crystallization conditions or in the presence of β -nucleating agents. β -nucleated iPP shows strong differences in mechanical properties as compared to common α -PP, namely higher toughness, ductility and drawability.

Objective

The aim of this work is to analyze the crystallization of β -nucleated iPP and its crystallization in model composites with different content (10-60 %) of glass fibers, known as α -nucleators, in order to predict the behaviour of real glass fiber/iPP composites, since the two polymorphic modifications of iPP are known to have different mechanical properties

Experimental and materials

Commercial grade Shell homo-iPP with a weight-average molecular weight, M_w , of 158 500, as determined by GPC, and a polydispersity index, M_w/M_n , of 6.36 was used. β -nucleated iPP was obtained by mixing 0.1 %wt. Ca pimelate with iPP in Brabender PL 2000 at 460 K. To study the influence of glass fibers on crystallization peculiarities and morphology of the polymer, model composites BNP/glass-fibers were prepared with 10, 20, 30, and 60 % wt. Isothermal and nonisothermal crystallizations of BNP was analyzed by DSC. In isothermal regime, the sample was rapidly heated to 478 K and held in the molten state for 5 min, to erase the thermal history of the polymer. Then the sample was cooled to a given crystallization temperature, T_c with a cooling rate of 80 K/min. Isothermal crystallization was carried out at T_c until crystallization was completed. The crystallization under nonisothermal conditions was performed by cooling at different cooling rates: 1, 3, 5, 10, 15, and 20 K/min.



Figure 2. Polarizing optical micrograph of BNP.

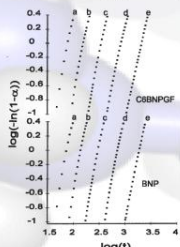


Figure 3. Avrami plots of BNP and C6BNPGF at different T_c (K): (a) 397; (b) 400; (c) 403; (d) 406; (e) 409.

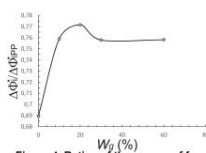


Figure 4. Ratios of the energy of formation of nucleus of critical dimension versus content of glass fibre (W_g , mas%) at $T_c=409$ K.

Surface Energy σ_e , Equilibrium Melting Temperature T_m , θ γ Constants Determined from DSC Data

	BNP	C1BNPGF	C2BNPGF	C3BNPGF	C6BNPGF	iPP
T_m^0 (K)	446.0	451.1	452.0	451.0	451.0	465.7
$\sigma_e \times 10^3$ (Jm ⁻²)	80.7	93.0	105.0	102.0	101.0	208
γ	2.20	1.84	1.81	1.86	1.87	2.37
θ	0.34	0.44	0.46	0.43	0.43	1.00

Assignment and content of the Samples

assignment	content
BNP	iPP+0.1 % Ca - pimelate
C1BNPGF	BNP /10% mas glass fibre
C2BNPGF	BNP + 20% mas glass fibre
C3BNPGF	BNP + 30% mas glass fibre
C6BNPGF	BNP + 60% mas glass fibre
iPP	iPP Shell

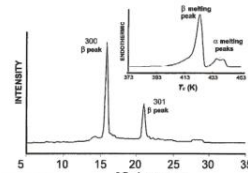


Figure 1. X-ray diffraction pattern and DSC melting thermogram of BNP.

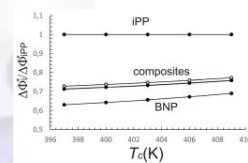


Figure 3. Ratios of the energy of formation of nucleus of critical dimensions for polymer (iPP), BNP and composite versus T_c .

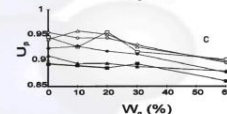
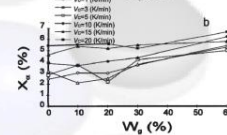
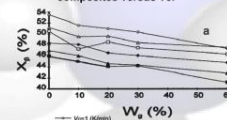


Figure 6. Dependence of the (a) amount of the β phase (X_b), (b) amount of the α phase (X_a) and (c) relative content of β phase (U_b) on content of glass fibre (W_g , mas%).

Conclusions

Glass fibers as a weak α -nucleator for iPP have insignificant effect on the morphology of β -nucleated iPP in glass fiber composites. Thermodynamic and kinetics parameters of crystallization of BNP in model composites are close to those obtained for the nucleated polymer. The relative content of β -crystalline phase is decreasing only for several percents by increasing glass fiber's content from 10 %wt to 60 %wt, due to appearance of α -crystallites. The relative content of β -phase depends mostly on the conditions of crystallization: the lowest value of U_b (for high fiber content) is 0.85.



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COMPOSITES WITH GLASS FIBRES**

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