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Melt-recrystallization Behavior of Highly Oriented α -iPP Fibers Embedded in HIPS Matrix

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Abstract

The melt-recrystallization behavior of α -iPP fibers embedded in amorphous HIPS matrix has been studied by means of optical microscopy. The amorphous HIPS served as supporter of iPP fibers does not involve in the nucleation and crystallization process of the molten highly oriented iPP fibers. It does also not provide any birefringence under optical microscope with crossed polarizers. This enables the study of orientation-induced β -iPP crystallization through control the melting status of the fibers. Through melting the fibers at different temperatures above 175 °C and subsequent recrystallization, some β -iPP crystals were always produced. The content of the β -iPP crystal depends strongly on the melting temperature and melting time of the iPP fibers. It was confirmed that melting the iPP fibers at relative lower temperature, e.g. 176 °C, less amount of β -iPP crystals were observed. The content of β -iPP crystal enhances first with increasing melting temperature and then decreases with further increase of the fiber melting temperature. The β -iPP crystallization is found to be most favorable by melting the fibers at 178 °C for 2 min. This demonstrates the requirement of certain chain or chain segment orientation for generating β -iPP crystallization on one hand, while higher orientation of the iPP chains or chain segments encourages the growth of iPP crystals in the α -form on the other hand. This has been further confirmed by varying the melting time of the fiber at different temperatures, since relaxation of the iPP molecular chains at a fixed temperature is time depended. Moreover, the complete transformation of α -iPP fibers in some local places into β -iPP crystals implies that the $\alpha\beta$ -transition may be not required for the orientation-induced β -iPP crystallization.

Introduction

Isotactic polypropylene (iPP) is a typical semicrystalline polymer with pronounced polymorphic crystalline modifications depending on processing condition and chemical structure of the macromolecules. At least three different crystalline forms, designated as monoclinic α -form, trigonal (or most frequently hexagonal) β -form and orthorhombic γ -form, have been reported in the literatures.¹⁻⁶ It was well documented that the monoclinic α -iPP, which is the thermodynamically most stable one, can be produced by normal melt or solution crystallization under varied conditions. The nucleation of the metastable β -iPP occurs, however, much more rarely in bulk and solution crystallization than that of the predominant α -modification. Therefore, it was most frequently obtained through the use of special nucleating agents during bulk crystallization.⁷⁻⁹ Crystallization of iPP in a thermal gradient and sheared or strained iPP melt was also found to encourage the formation of β -iPP.^{6,10} To understand the shear-induced β -iPP crystallization, fiber-pulling technique has been employed by many researchers to create and model the shear-induced crystallization of iPP under various thermal and mechanical conditions, since *in-situ* observation of crystallization under shear or elongation conditions has some technical difficulties.¹¹⁻¹⁶ On the basis of systematic studies, it was suggested that the stretched macromolecules in melt under shear can produce α -iPP row nuclei. On the growth front of the *in-situ* formed α -row nuclei, the nucleation of β -iPP is initiated via $\alpha\beta$ -bifurcation. This leads to the formation of cylindrites with mixed polymorphic composition. Varga and his co-workers¹⁶ reported that the polymorphous composition is not affected by the nucleation ability of the fibers but controlled by the temperature-dependent relative growth rate of the α - and β -iPP. A higher growth rate of the β -phase (G_β) than that of α -phase (G_α) is considered to be the requirement of the $\alpha\beta$ -growth transition.^{9, 14, 16-18} Otherwise,

the fast growing α -iPP crystals will embed the generated β -nuclei. Taking this into account, it is suggested that the content of the β -modification is highest when the fiber pulling and crystallization temperatures are closely matched within a temperature range of $T_{\alpha\beta}$ (α to β transition temperature) $\approx 100^\circ\text{C}$ and $T_{\beta\alpha}$ (β to α transition temperature) $\approx 140^\circ\text{C}$. The question remains difficult to be understood is why the $\alpha\beta$ -bifurcation occurs on the surface of the *in-situ* formed α -row nuclei rather than growth of α -iPP crystals. In other words, the mechanism of the $\alpha\beta$ -bifurcation is still not quite clear so far.

To get more insight of the orientation-induced β -iPP crystallization, our research group has utilized an experimental set-up with highly oriented iPP fibers being introduced into their homogeneity molten matrix.¹⁹⁻²² In this process, the molecular chain orientation of iPP in the melt was regulated by controlling the melting statues of the iPP fibers. It was found that solid iPP fiber induces α -iPP transcrystallization of the matrix due to the close matching between the lattice geometry.²³ Partial melting followed by recrystallization of the iPP fibers enables the formation of β -iPP transcrystals in the matrix. In this way, the non-molten α -iPP fiber cores plays a role for reinforcing the β -iPP matrix.²⁴ It was further demonstrated that the β -iPP crystallization is a nucleation controlled process. Chain segment orientation in the iPP melt plays a very important role in generating β -iPP nuclei. There seems to be an orientation window of iPP molecules in the molten state, which enables the β -nucleation of iPP.²⁵ It should be pointed out that, in our experimental procedure, there is some extent of shear on the iPP matrix caused by fiber introduction, even though it does not obviously influence the β -iPP crystallization. Moreover, it has been confirmed that in the used experimental set up, the matrix iPP is involved in the β -nucleation process through interdiffusion with the oriented chains of molten iPP fiber.²⁶ This makes it complicate for exploring

the orientation-induced β -iPP crystallization mechanism. To eliminate the influence of the iPP matrix, we have embedded the highly oriented iPP fibers into an amorphous matrix of atactic polystyrene and studied the melt recrystallization behavior of the oriented iPP fiber themselves.

The purpose of this paper is to present the experimental details and the obtained new results. The orientation-induced β -iPP crystallization mechanism is discussed accordingly.

Experimental section

The iPP fiber used in this work is the same as used in the previous work.¹⁹ Its average diameter is ca. 20 μm . To remove the sizing agents on the fiber surface, the fibers were treated before use for 4 hours with refluxing acetone and then dried in a vacuum oven at 40 $^{\circ}\text{C}$ for 5hs. The atactic polystyrene matrix material used in this work was commercial grade High Impact Polystyrene (HIPS), with M_w of ca. 5.0×10^4 g/mol.

The HIPS embedded iPP fiber samples were prepared by first immersing a bunch of iPP fiber with 2~4 fibers into the HIPS/chloroform solution and then waiting for the evaporation of the chloroform to obtain solid sheet sample of iPP/HIPS. The thus prepared iPP/HIPS sheets were dried subsequently over night under vacuum before further treatment.

The melt recrystallization of the iPP fibers was performed by heating the sample to different temperatures for varying times and then directly moved to another heat plate at 130 $^{\circ}\text{C}$ for isothermal crystallization till completion. Even though the iPP fibers show a peak melting temperature around 170 $^{\circ}\text{C}$, the end melting temperature of the iPP fibers was identified to be about 175 $^{\circ}\text{C}$. Since an insufficient melting of the polymer crystals will result in the self-seeding phenomenon during the recrystallization process, we select the heat-treatment temperature of the

iPP/HIPS fiber/matrix samples ranging from 176 °C to 185 °C to study the melt recrystallization behavior of iPP with different content of chain orientation.

For optical microscopy observation, an Axioskop 40A Pol Optical Microscope (Carl Zeiss) equipped with a Linkam LTS 350 hot stage was used in this study. The Linkam LTS 350 hot stage enabled holding steady temperature with accuracy of ± 0.1 °C. All optical micrographs presented in this paper were taken under crossed polarizers.

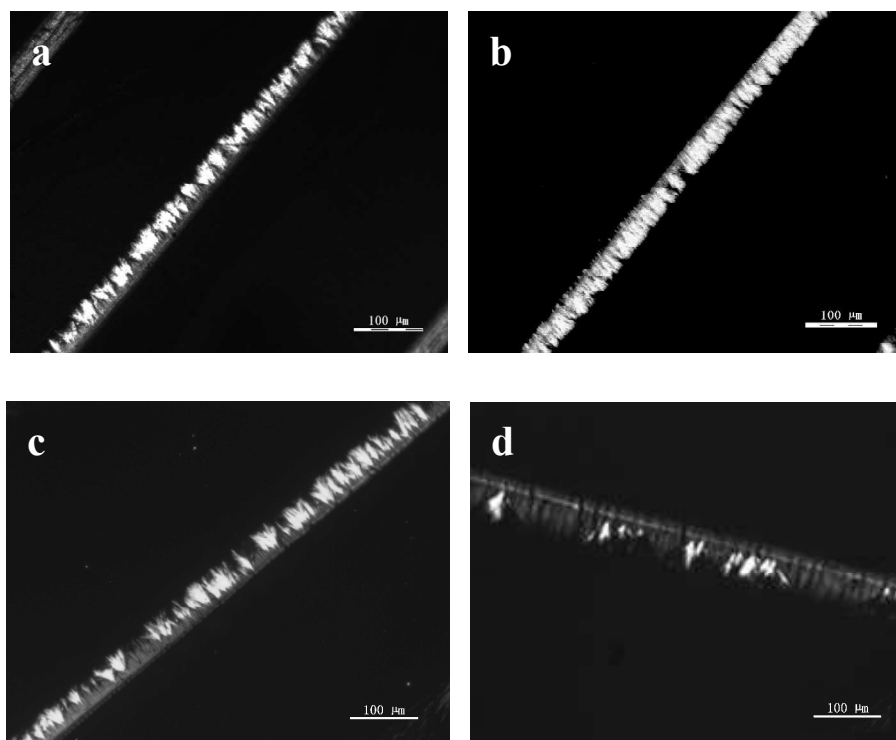


Figure 1 Optical micrographs of iPP/HIPS fiber/matrix composites prepared by melting iPP fibers at (a) 177 °C, (b) 178 °C, (c) 179 °C and (d) 185 °C for 2 min and then isothermally crystallized at 130 °C till completion.

Results and discussion

Figure 1 shows the representative optical micrographs of the iPP/HIPS fiber/matrix composites after heat-treated at (a) 177 °C, (b) 178 °C, (c) 179 °C and (d) 185 °C for 2 min and subsequently

crystallized isothermally at 130 °C. From Figure 1, one can clearly see two kinds of the morphologies with different birefringence. According to the results of previous study,¹⁸ the crystals with very strong birefringence should correspond to the β -form iPP crystals, while those with weak birefringence are the crystals of α -iPP. The selective melting test helps to confirm this. As shown in Figure 2, during the heating process of the sample shown in Figure 1b, no obvious changes were observed at temperature lower than 150 °C, compare Figures 2a and 1b. When the temperature reaches 160 °C, which is higher than the melting temperature of β -iPP crystals but lower than its α -iPP counterpart, the strong birefringent parts disappear completely, see Figure 2b. This unambiguously indicates the formation of β -iPP crystals with very strong birefringence.

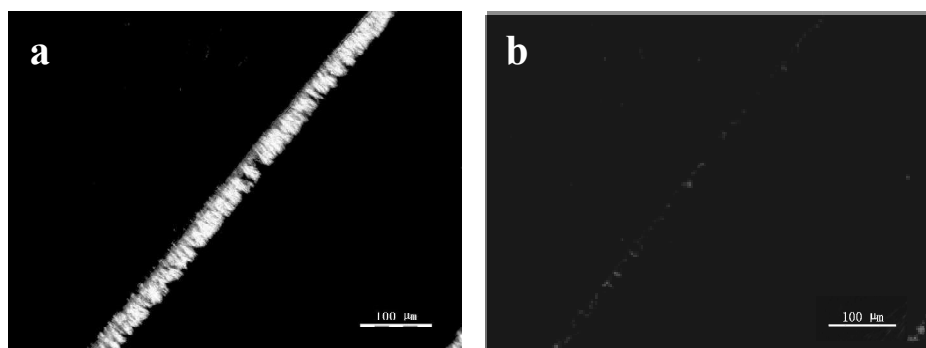


Figure 2 Optical micrographs of an iPP/HIPS fiber/matrix composite prepared by melting iPP fibers at 178 °C for 2 min and then isothermally crystallized at 130 °C. The pictures were taken during the heating process at temperatures of (a) 150 °C and (b) 160 °C.

According to the above-observed morphological features, two aspects should be discussed here. First of all, we can see from Figure 1 that the β -iPP crystallization is dependent on the melting temperature of the highly oriented iPP fibers. It is clear that melting the iPP fiber at 185 °C for 2 min and then crystallized isothermally at 130 °C leads to the formation of mainly α -iPP crystals

with only few randomly dispersed fan-shaped β -iPP crystals. With decreasing heat-treatment temperature, increasing β -iPP crystals were seen after the melt recrystallization of the fiber. This can be understood in the following way. Extensive studies on the relaxation and crystallization of sheared polymer melts indicated that metastable oriented structures in sheared polymer melts might resist relaxation upon cessation of shearing and could act as “prenuclei” from which development of fold chain lamellae with the chain axis highly oriented along the shear direction taken place.²⁷⁻³¹ A recent study on the melt-recrystallization behavior of a butene-1/ethylene random copolymer by Men et al.³² also confirms that surviving oriented polymer chain segments during melting can influence the crystallization kinetics and the polymorphic modifications. It is worth noting that the “prenuclei” cannot survive forever. They can remain active only for a certain period of time after cessation of shear flow at the temperatures above its melting point. Azzurri and Alfonso^{33,34} have reported that the lifetime of the nucleation precursors is a very sensitive function of relaxation temperature. In our systems, since the fibers are highly oriented, the melting process of the fibers is actually similar to the sheared polymer melts. By melting the iPP fibers at 185 °C, which is about 15 °C higher than the melting point of the used iPP material, for 2 min will lead to a high extent relaxation of the iPP molecular chains in molten fiber. This results in the recrystallization of molten iPP fiber preferentially in its α -form. On the other hand, when the heat-treatment temperature of the iPP fiber is just above its melting point, much slower relaxation rate of the iPP molecular chains in the molten fibers is expected. Therefore, less relaxation of the iPP molecular chains or chain segments in molten fiber could be attained before recooling of the samples. In other word, the molten iPP fiber may exist actually in the form of amorphous domains with relatively highly oriented or stretched macromolecular segments and/or chains as in the sheared polymer melts.

These oriented molecular segments or chains, in turn, serve as nucleation precursors and initiate the crystallization during cooling process. In this case, the formation of the β -iPP crystals is expected on the basis that shear-oriented iPP melt is in favor of the β -iPP crystallization as observed in the fiber-pulling experiments. These results demonstrate that the formation of β -crystals is associated with the oriented or stretched macromolecular chains survived in the molten iPP fiber. It should be pointed out that melting the iPP fibers at 178 °C is most favorable for triggering the β -iPP crystallization, compare Figures 1a~d. This implies that the β -iPP crystallization proceed most preferentially in a certain orientation window of the iPP molecular chain segments in the molten state. The β -iPP crystallization is suppressed when the chain orientation beyond this window, either with higher or lower orientation. This will be further discussed later on the basis of other experimental results.

Second, as mentioned in the introduction part, the occurrence of the $\alpha\beta$ -secondary nucleation on the surface of the *in situ* formed α row nuclei in the fiber pulling experiments rather than growth of α -iPP crystals is a hard understanding phenomenon. In our previous study on the iPP fiber/matrix homogeneity composite systems,²⁰ we have once occasionally observed the break of iPP fibers by selective melting at temperatures above the melting point of β -iPP but below that of α -iPP. Since highly oriented iPP fibers can only exist in their α -form as $\beta\alpha$ transition always takes place on mechanical stretching, the break of the iPP fibers unambiguously indicates the transformation of some α -iPP fiber parts into β -form through melt recrystallization. It is therefore suggested that the $\alpha\beta$ -transition is not necessary for the β -iPP crystallization. Here from Figure 2b, after melting of the β -iPP crystals, we see only few birefringent spots, corresponding to the remaining α -iPP crystals. This means that most of the α -iPP crystals included previously in the fiber have transformed into

their β -counterparts through melt-recrystallization. It should be pointed out that there exist nonbirefringent regions of tens of micrometers. From this point of view, it further suggests that the $\alpha\beta$ -transition may be not required for the β -iPP crystallization. It is the degree of local chain orientation of the molten iPP fiber just before nucleation taking place, or in other words, the melting status of the highly oriented iPP fibers, that plays a very important role in generating β -iPP crystallization.

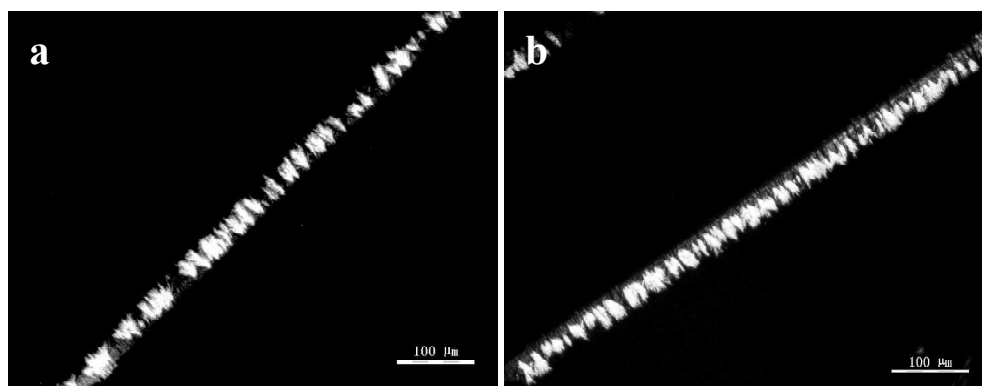


Figure 3 Optical micrographs of the iPP/HIPS fiber/matrix composites prepared by melting iPP fibers at 178 °C for (a) 1 min and (b) 3 min and then isothermally crystallized at 130 °C.

If the above hypothesis holds true, different relaxation time of the molten iPP fibers should cause different recrystallization behavior. To confirm this, the iPP fibers were molten at 178 °C for 1 min and 3 min before cooled to 130 °C for isothermal crystallization. As presented in Figure 3, in both case, less β -iPP crystals were generated comparing to the sample shown in Figure 1b. This helps to confirm that the β -iPP crystallization is governed by the orientation status of the iPP molecular chains before crystallization. For the sample heated to 178 °C for 1 min, less chain relaxation is achieved with respect to heat-treatment the sample at 178 °C for 2 min. The reduced amount of β -iPP crystals is then caused by relatively higher molecular chain orientation of the

molten iPP. On the contrary, heat-treating the sample at 178 °C for 3 min further relaxation of the iPP molecular chains produces a poorly oriented state of the molten iPP fiber. In this case, the decrease in the amount of β -iPP crystals can be correlated to the insufficient chain orientation of the iPP molecular chains or chain segments. This indicates again the existence of an orientation window of the iPP molecular chain segments in the molten state that enables the crystallization of iPP in its β -form.

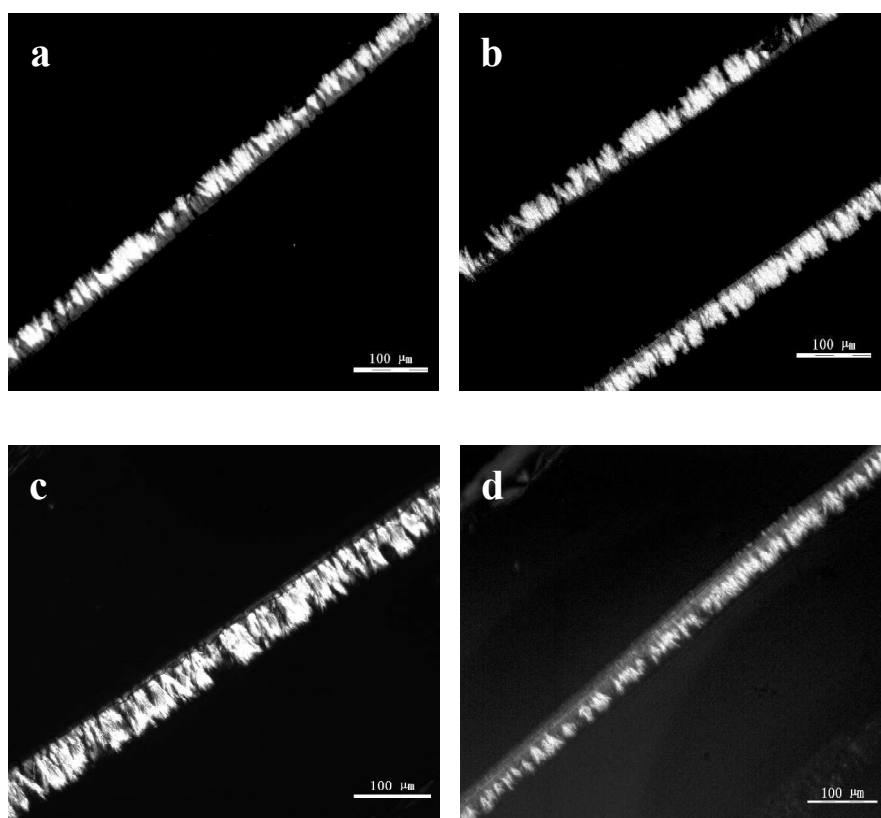


Figure 4 Optical micrographs of the iPP/HIPS fiber/matrix composites prepared by melting iPP fibers at 177 °C for (a) 2.5 min, (b) 3 min, (c) 4 min and (d) 4.5 min and then isothermally crystallized at 130 °C.

The above conclusion is further confirmed by molten the iPP/HIPS fiber/matrix samples at 177 °C for different time and subsequently moved to a preheated stage at 130 °C for isothermal

crystallization. Figure 4 shows the representative optical micrographs. As we can see from Figure 4a, similar crystalline morphology of the sample heated to 177 °C for 2.5 min and crystallized isothermally at 130 °C was obtained as that heat-treated for 2 min at 177 °C, compare Figure 4a with Figure 1a. This could be accepted under the consideration that the relaxation extent of iPP molecular chains at 177 °C for 2 min and 2.5 min should not be much different. When the iPP fibers were molten at 177 °C for 3 min and 4 min, see Figure 4b and c, somewhat increased amount of β -iPP crystals was seen, indicating an increased ability in generating β -iPP crystallization with decreased molecular chain orientation. The capacity of β -iPP crystallization is, however, decreased with further increase of the fiber melting time at 177 °C, see Figure 4d. This is associated to a more extent molecular chain relaxation of the molten iPP fibers, which reduces the molecular chain orientation and depresses the β -crystallization of iPP.

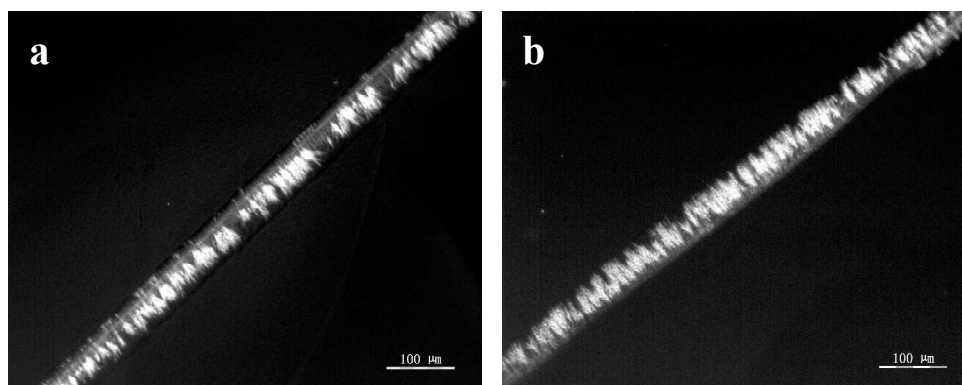


Figure 5 Optical micrographs of the iPP/HIPS fiber/matrix composites prepared by melting iPP fibers at 176 °C for (a) 3 min and (b) 4.5 min and then isothermally crystallized at 130 °C.

It is expected that melting the iPP at even lower temperature will further slow the molecular chain relaxation of the molten iPP fibers. Therefore, much more oriented iPP chains or chain segments can be attained after melting for the same time as at higher temperature. According to the

above racemization, the recrystallization should be less preferred for β -iPP crystallization. To check this, Figure 5 shows the optical micrographs of the iPP/HIPS fiber/matrix samples isothermally crystallized at 130 °C after molten at 176 °C for 3 min and 4.5 min, respectively. Figure 5a tells us that crystallization of iPP after melting at 176 °C for 3 min produces really less β -iPP crystals with respect to the sample heated to 177 °C for 3 min, compare Figure 5a with Figure 4c. Combining the fact that less relaxation achieved by holding the sample for 3 min at 176 °C than at 177 °C, it also indicates that highly oriented chain segments are not in favor of β -iPP crystallization. On the other hand, Figure 5b shows obviously more β -iPP crystals than Figure 5a, indicating further relaxation of the iPP molecular chains or chain segments provides a more favorable environment for β -iPP crystallization. Moreover, it is reasonable to say that the relaxation of iPP molecular chains at 176 °C for 4 min should be less proceeded than at 177 °C for 4 min. Keep this in mind, a comparison of Figures 5b and 4d leads to the conclusion that certain chain or chain segment orientation is nevertheless required for generating β -iPP crystallization.

Conclusion

The melt-recrystallization behavior of highly oriented iPP fibers originally in the α -form was studied through embedding the fibers into the amorphous HIPS matrix. It was found that melting the iPP fibers at temperatures ranging from 176~185 °C and subsequent recrystallization always leads to the formation of some β -iPP crystals. This is related to the orientation induced β -iPP crystallization as found in shear-induced crystallization of iPP, considering that a fully relaxation of the highly oriented iPP molecular chains or chain segments cannot be reached. The phenomenon itself is not new. But the dependence of the amount of β -iPP crystals on the melting

temperature and time helps us to understand the mechanism of orientation-induced β -iPP crystallization. It was found that melting the iPP fibers at relative lower temperature, e.g. 176 °C, less amount of β -iPP crystals were created. With increasing melting temperature, the content of β -iPP crystal enhances first and reaches its maximum at 178 °C. When the melting temperature is higher than 179 °C, the amount of the obtained β -iPP crystals decreases with further increase of the fiber melting temperature. This kind of dependence demonstrates that the β -iPP crystallization requires certain chain or chain segment orientation of iPP in the molten fiber on one hand, while higher orientation of the iPP chains or chain segments encourages the growth of iPP crystals in the α -form on the other hand. In other words, the β -iPP crystallization is most favored in a certain chain or chain segment orientation window. This has been further confirmed by varying the melting time of the fiber at different temperatures, since relaxation of the iPP molecular chains at a fixed temperature is time depended. Moreover, during the selective melting test, the α -iPP fiber was found to be completely transformed into β -iPP crystals in the some local places after melt recrystallization. This implies that the $\alpha\beta$ -transition should be not required for the orientation-induced β -iPP crystallization.

Acknowledgements

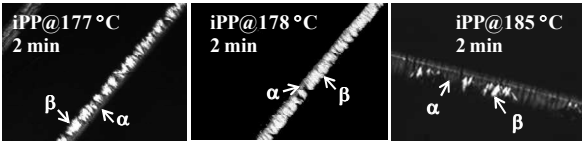
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