

Effect of *N,N'*-Diphenyl Adipamide on the Formation of the β -Crystalline Form in Isotactic Polypropylene

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ABSTRACT: The polymorphic compositions and mechanical properties of isotactic polypropylene (iPP) samples nucleated by a selective β -nucleating agent [*N,N'*-diphenyl adipamide (DPA)] were investigated with wide-angle X-ray diffraction, polarized light microscopy, scanning electron microscopy, and mechanical tests. It was found that β -phase crystals emerged with the addition of DPA, and the relative proportion of the β -crystalline form reached the maximum value of 0.97 with the addition of 0.1 wt % DPA. The curved lamellae in the β spherulites were like flowers. The β spherulites were etched more easily than α spherulites because

amorphous regions were distributed inside the β spherulites. The Izod notched impact strength increased sharply with the addition of DPA and attained the maximum value of 7.30 kJ/m² (the value of blank iPP was 3.13 kJ/m²) with the addition of 0.1 wt % DPA. An analysis of the misfit factors between DPA and β -iPP showed that β -iPP could epitaxially crystallize on the DPA crystal well. © 2008 Wiley Periodicals, Inc. *J Appl Polym Sci* 111: 1738–1744, 2009

Key words: crystallization; nucleation; polymorphism; poly(propylene) (PP)

INTRODUCTION

As a polycrystalline polymer, isotactic polypropylene (iPP) has α , β , γ , and smectic forms. The α -crystalline form of iPP (α -iPP) is the most stable, and the other forms will transform into it under certain conditions such as heat treatment and extension. The β -crystalline form of iPP (β -iPP) has attracted many researchers' interest because of its unique properties, such as improved toughness and microvoids from β - α transformation, and it has been used in toughened plastic products, microporous membranes, fibers, and so forth. The most reliable method to prepare high-purity β -iPP involves the introduction of β -nucleating agents such as quinacridone pigment, a pimelic acid/calcium stearate mixture, calcium salts of suberic or pimelic acid, CaCO₃ modified with dimeric aluminates, *N,N'*-dicyclohexyl-terephthalamide, and *N,N'*-dicyclohexyl-2,6-naphthalene-dicarboxamide.¹

Among them, amide β -nucleating agents are important and have been studied by many researchers. The most frequently used one is NJStar NU100, the trade name of *N,N'*-dicyclohexyl-2,6-naphthalenedicarboxamide, which is produced by New Japan Chemical (Osaka, Japan). Chu et al.² investigated the microvoid formation process that takes place during

the plastic deformation of β -form polypropylene (polypropylene blended with 0.1 wt % NJStar NU100) at different crystallization and drawing temperatures. Varma-Nair and Agarwal³ compared nucleating efficiencies of the β nucleator (NJStar) in metallocene and Ziegler–Natta polypropylene. Nezbedova et al.⁴ examined the influence of the processing conditions and the distance from the gate on the dynamic fracture toughness and dynamic *J* integral value of samples of β -iPP with various weight contents of the β nucleant (NJStar NU100). Bohaty et al.⁵ also studied the toughness of α -iPP and β -iPP (the β nucleant NJStar NU100 was used). Kawai et al.⁶ carried out the melt crystallization of iPP containing crystallites of *N,N'*-dicyclohexyl-2,6-naphthalenedicarboxamide as a nucleating agent under a magnetic field (6 T) to obtain the alignment of the iPP crystal induced by the magnetic alignment of *N,N'*-dicyclohexyl-2,6-naphthalenedicarboxamide. Marco et al.⁷ found NJStar NU100 to be very efficient at inducing the crystallization of iPP into the β modification, with the relative content of the β -crystalline form (*k*) between 0.85 and 0.95, depending on the concentration of the additive and the cooling rate. Cho and coworkers^{8,9} prepared a β -iPP sample by adding 0.2 wt % NJStar to the iPP melt and studied the melting memory effect in the crystallization of β -iPP. Romankiewicz and coworkers,^{10–12} Raab and coworkers,^{13–15} Kotek and coworkers,^{16–20} Obadal and coworkers,^{21–28} and Výchopňová and coworkers^{29,30}

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also modified iPP with NJStar NU100, and they studied the morphology, molecular and supermolecular orientation, crystallization, mechanical behavior, and structural transformation of β -iPP induced by heating and ultraviolet light, composites of β -iPP with calcium carbonate, wood flour, and ethylene-propylene rubber, a synergy application of NJStar NU100, and high pressure during crystallization in the tailoring of multicomponent crystalline systems in iPP. Zhou et al.³¹ studied a β -form iPP spherulite induced by N,N' -dicyclohexyl-2,6-naphthalenedicarboxamide at 130°C with high-temperature atomic force microscopy *in situ* and in real time. Behrendt et al.³² studied the electret property of isotropic and biaxially oriented polypropylene films containing α -nucleating (NA11) and β -nucleating agents (NU100). Menyhárd and coworkers^{33,34} found NJStar NU100 to be an efficient nucleating agent for preparing β -iPP. However, NJS is not a selective β -nucleating agent because of the partial α -nucleating ability of the lateral surface of NJS needle crystals. Hou et al.³⁵ found N,N' -dicyclohexyl-2,6-naphthalenedicarboxamide to be a selective β -nucleating agent, whereas N,N' -dicyclohexyl-1,4-naphthalenedicarboxamide has only a very weak α -nucleating effect for iPP crystallization. Stocker et al.³⁶ and Mathieu et al.³⁷ suggested that nucleating agents with ≈ 6.5 -Å periodicity (dicyclohexylterephthalamide and γ -quinacridone) and an orthogonal geometry of the contact face are likely to induce the β -iPP polymorph. Zhang and Xin³⁸ studied the isothermal crystallization behaviors of iPP nucleated with the α nucleator 2,2'-methylene-bis(4,6-di-*tert*-butylphenyl) phosphate (NA40), the β nucleator N,N' -dicyclohexyl-terephthalamide (NABW), and NA40/NABW (weight ratio is 1 : 1) as a compound nucleating agent, respectively. Mohmeyer et al.³⁹ demonstrated efficient β -nucleating agents for iPP with a highly symmetric molecular structure, including two dicyclohexyl-substituted, 1,4-phenylene bisamides (N,N' -dicyclohexylterephthalamide derived from terephthalic acid chloride reacted with an excess of cyclohexylamine and an isomer synthesized from *p*-phenylenediamine and cyclohexane carboxylic acid chloride). An asymmetrical isomer, cyclohexane carboxylic acid-(4-cyclohexylaminophenyl) amide, resulted in a decreased K value (the relative content of β form). Tang et al.⁴⁰ found that the fraction of the β form of iPP increases in the presence of 1,3,5-benzene tricarboxylic-(*N*-2-methylcyclohexyl) triamine and demonstrated that the nonisothermal crystallization temperature of iPP increases by an additional 7°C when the triamine is efficiently distributed within the polymeric matrix via coating onto zinc oxide nanoparticles. Blomenhofer et al.⁴¹ presented a new family of 1,3,5-trisamides with the generic structure A-(X-R)₃ (where A is benzene, X is an amide, and R is an apolar substituent). They found that compounds based on 1,3,5-benzenetrisamides in which R is 2,3-dimethylcyclohexyl, cyclooctyl, 1,1,3,3-tetramethylbu-

tyl, cyclododecyl, or 3-methylcyclohexyl induce β -crystal modifications of iPP.

In the aforementioned research, two frequently used selective amide β nucleators are N,N' -dicyclohexyl-2,6-naphthalene-dicarboxamide and dicyclohexylterephthalamide. Studies on the effects of other amides on the polymorphs of polypropylene are seldom. In this study, N,N' -diphenyl adipamide (DPA) was found to be a selective β nucleator for iPP. Its effect on the formation of the β -crystalline form in iPP is examined in this article.

EXPERIMENTAL

Materials

iPP powder [produced by a Ziegler-Natta catalyst system; melt flow rate = 4.2 g/10 min (measured according to ISO 1133 : 1997 at 230°C and a 21.2-N load)] was obtained from Jinling Plastics and Rubber Chemical Co, Ltd. An industrial antioxidant (B215, Nanjing Hualim Co., Ltd., Nanjing, China) was commercially available. DPA was synthesized in the laboratory and was characterized with infrared technology (Nexus 670 Fourier transform infrared spectrometer, Nicolet Corp., Madison, WI) and ¹H-NMR (Avance 400D+ HR/MAS, Bruker, Fällanden, Switzerland). The Fourier transform infrared and ¹H-NMR data of DPA are given next.

Fourier transform infrared: 3245 (ν_{NH}), 3129 and 3074 ($\nu_{\text{Ar-H}}$), 2935 ($\nu_{\text{as-CH}_2}$), 2869 ($\nu_{\text{s-CH}_2}$), 1658 ($\nu_{\text{C=O}}$, amide I band), 1596 (ν_{NH} , amide II band), 1548, 1488, and 1444 cm^{-1} ($\nu_{\text{Ar-CH}}$). ¹H-NMR (deuterated dimethyl sulfoxide, 400 MHz, δ): 1.64 (m, 4H, -CH₂-), 2.34 (m, 4H, -CO-CH₂-), 7.02 (t, 2H, Ar-H), 7.28 (t, 4H, Ar-H), 7.58 (d, 4H, Ar-H), 9.87 ppm (s, 2H, -CONH-).

Preparation of nucleated iPP samples

iPP (500 g) was intensely mixed with 0.5 g of B215 and various amounts of DPA. The mixtures were compounded in a corotating twin-screw extruder [diameter = 20 mm, length/diameter = 32/1; TE-20, Coperion Keya (Nanjing) Machinery Co., Ltd., Nanjing, China]. The rates of the feeder screw and main screw were set at 20 and 200 rpm, and the temperature of the extruder from the hopper to the die was set at 200, 210, 210, and 200°C, respectively. The melt was cooled and pelletized. iPP doped without DPA was prepared in a similar way to produce a blank control sample. A granule of nucleated iPP was placed between two glass slides on a hot stage kept at 220 \pm 2°C for 10 min to allow the sample to melt completely and remove the thermal memory; the top slide was squeezed to form a film, and then it was cooled in air to room temperature. The thickness of

the samples for wide-angle X-ray diffraction (WAXD) characterization and scanning electron microscopy (SEM) observation was 0.5 mm, and for polarized light microscopy (PLM) observation, it was 20–40 μm .

WAXD characterization

WAXD diffraction patterns of the nucleated iPP samples were recorded in an X-ray diffractometer (ARL X'Tra, Thermo Electron Corp., Waltham, MA) with Cu K α radiation ($\lambda = 1.54 \text{ \AA}$). It was operated at a voltage of 40 kV and a filament current of 35 mA. Radial scans of the intensity versus the diffraction angle (2θ) were recorded in the range of 10–30°. The scanning rate was 4°/min.

The characteristic crystal planes for the monoclinic α phase of iPP are (110), (040), (130), (111), and (131) planes, which correspond to 2θ values of 14.2, 17, 18.8, 21.3, and 22°, respectively. The crystal planes (300) and (301) for the β form are characterized by 2θ values of 16.1 and 21.2°. k in the iPP specimen was measured with an empirical ratio proposed by Turner-Jones et al.⁴²

$$k = H_{\beta 1} / (H_{\beta 1} + H_{\alpha 1} + H_{\alpha 2} + H_{\alpha 3}) \quad (1)$$

where $H_{\beta 1}$, $H_{\alpha 1}$, $H_{\alpha 2}$, and $H_{\alpha 3}$ are the heights (from the top of the peak to the background curve) of $\beta(300)$, $\alpha(110)$, $\alpha(040)$, and $\alpha(130)$, respectively. k tends to zero when no β form is present and to unity when the α form is absent but is not, of course, an absolute measure of the proportion of β crystallinity.

DPA was also investigated with the same X-ray diffractometer. The scanning angle range was 2–50°, and the scanning rate was 2°/min.

PLM observation

The spherulitic morphology of the nucleated iPP sample was observed on a polarized light microscope (LW-200-4JS, Shanghai LW Scientific Co., Ltd., Shanghai, China) equipped with cross-polars and a charged coupling device camera. Images were captured and stored in a computer. The signs of birefringence of the spherulites were determined by means of a primary red filter (λ plate) located diagonally between cross-polars. In this way, the first and third quarters of the sight were yellow and the second and fourth were blue when the spherulites were negative, whereas a reversed arrangement of the quarters was observed for positive spherulites.

SEM observation

Samples were etched under 80°C for 2 h in a chromic anhydride solution (50 g of chromic anhydride dissolved in 80 mL of distilled water and 20 mL of concentrated sulfuric acid), and then the surfaces of

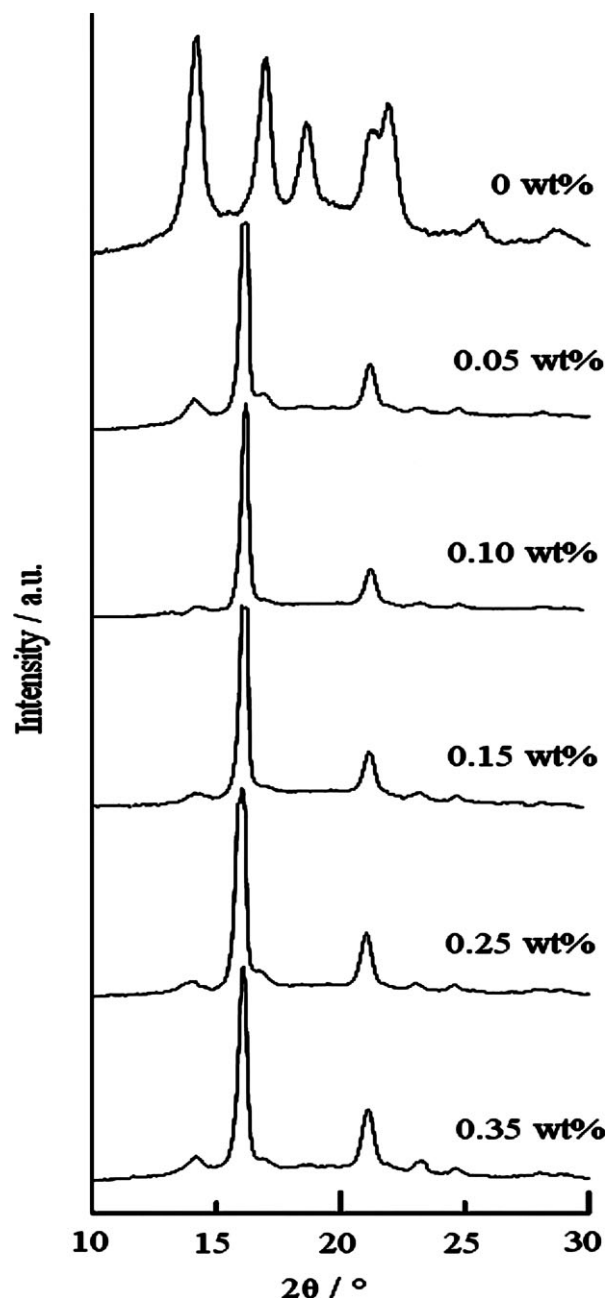


Figure 1 WAXD diffraction patterns of iPP samples doped with different amounts of DPA.

the samples were carefully washed with distilled water several times. The surfaces were sputtered with gold *in vacuo*, and SEM observations were performed on a JSM-5900 (JEOL, Tokyo, Japan).

Mechanical measurements

Standard test specimens for mechanical characterization were injection-molded with a reciprocating-screw injection-molding machine (CJ80M3V, Chen De Plastics Machinery Co., Ltd., Foshan, China) from 190 to 210°C at an injection pressure of 70 MPa. The measurements of the tensile strength, tensile strain at

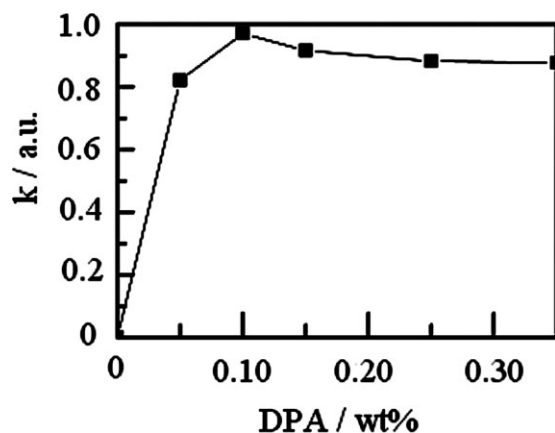


Figure 2 k values of iPP samples doped with different amounts of DPA.

break, and flexural modulus were carried out on a electromechanical universal testing machine (CMT5254, Shenzhen SANS Testing Machine Co., Ltd., Shenzhen, China) according to ISO527-2/1BA/50:1996 and ISO178:2003, respectively. The testing speeds for the tensile and flexural tests were 50 and 2 mm/min, respectively. The specimens for the measurement of the Izod notched impact strength were notched with a plastic specimen notcher (MZ2061, Jiangdu Mingzhu Testing Machine Factory, Jiangdu, China) and were measured on a pendulum impact testing machine (MZ2056, Jiangdu Mingzhu Testing Machine Factory) according to ISO180/A:2000.

RESULTS AND DISCUSSION

Polymorphic composition characterization

WAXD diffraction patterns and k values of iPP samples doped with different amounts of DPA are

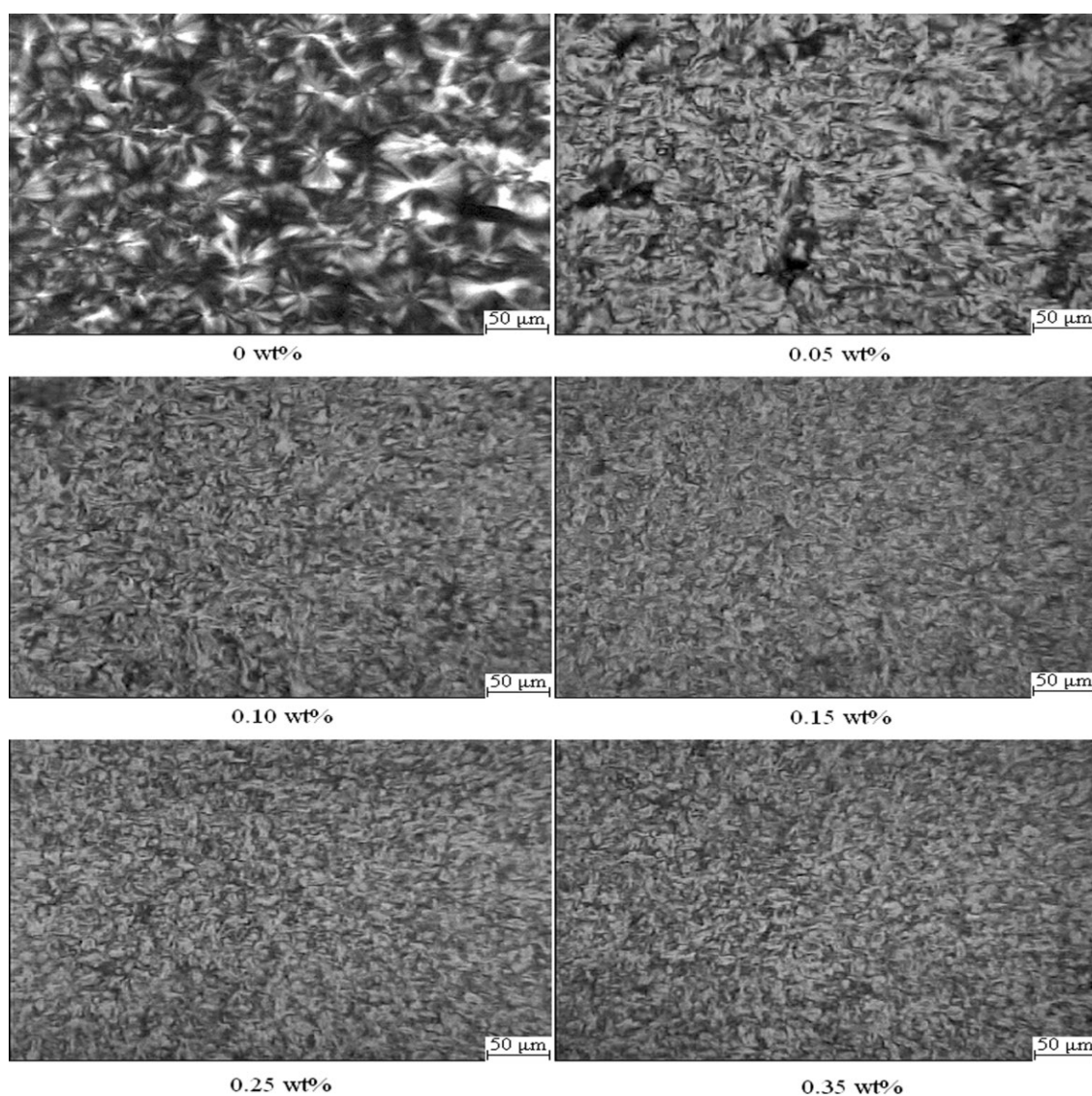


Figure 3 PLM photographs of iPP samples doped with different amounts of DPA.

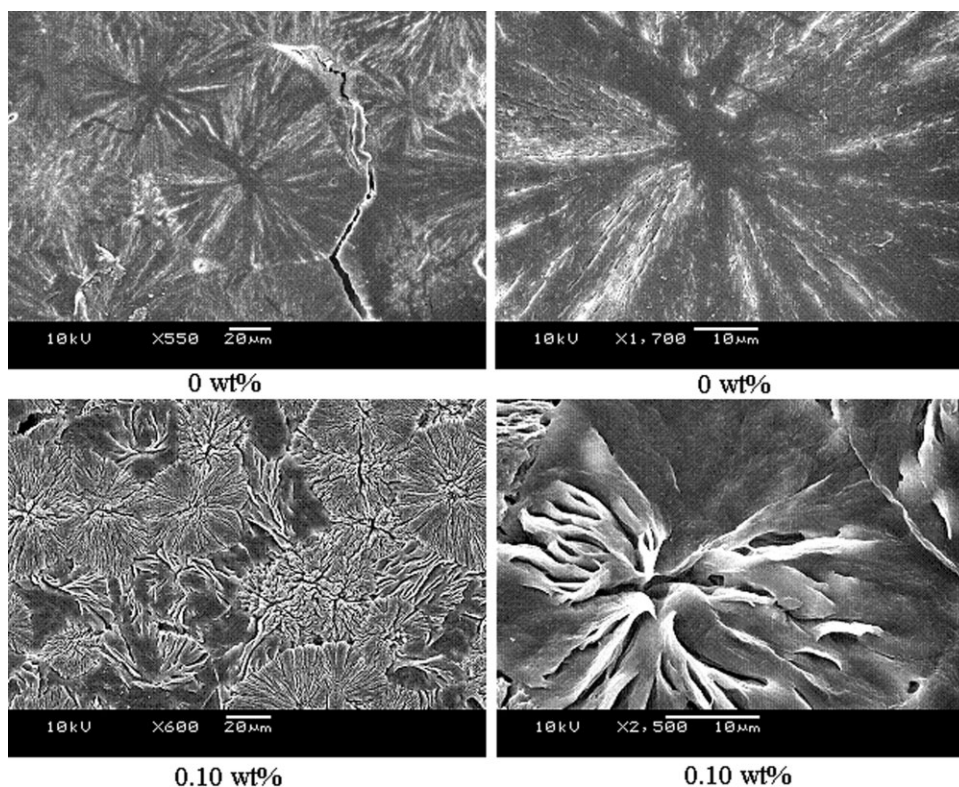


Figure 4 SEM photographs of blank iPP and iPP samples doped with 0.1 wt % DPA.

shown in Figures 1 and 2. k for the blank iPP is zero. The diffraction peaks of (300) and (301) for the β -crystalline form increase abruptly with the addition of DPA. The k value is 0.82 for iPP doped with 0.05 wt % DPA, and it reaches the maximum value of 0.97 at 0.1 wt % DPA. Then, the k value decreases somewhat with the concentration of DPA increasing.

PLM photographs of iPP samples doped with different amounts of DPA are shown in Figure 3, and they are similar to those reported in the literature.^{1,31,43} Black and white α spherulites can be seen in the photograph of the blank iPP sample. A large number of bright and colored β spherulites emerge in the nucleated samples with the addition of DPA, especially when the amount of DPA is greater than 0.1 wt %. The signs of birefringence of the α and β spherulites are positive and negative, respectively, and this was determined by means of a primary red filter (λ plate) located diagonally between cross-polars. The distinct optical differences between α and β spherulites arise from the different lamellar structures, as shown in Figure 4.

SEM photographs of the blank iPP and iPP samples doped with 0.1 wt % DPA are shown in Figure 4. Radial lamellar architectures are found in the α spherulites. The β spherulites are like flowers, and their lamellar arrangement is curved. The morphologies of the α and β spherulites are similar to those reported in the literature.^{1,14,17,34} The margins

between α spherulites that are amorphous regions are etched easily, but the α spherulites themselves are hard to etch. A sample rich in β spherulites is etched more easily than one rich in α spherulites because the amorphous regions are distributed inside the β spherulites.

Mechanical property measurements

In comparison with α -iPP, β -iPP has a lower modulus and yield stress but a higher ultimate tensile strength and strain. The improvement in the latter

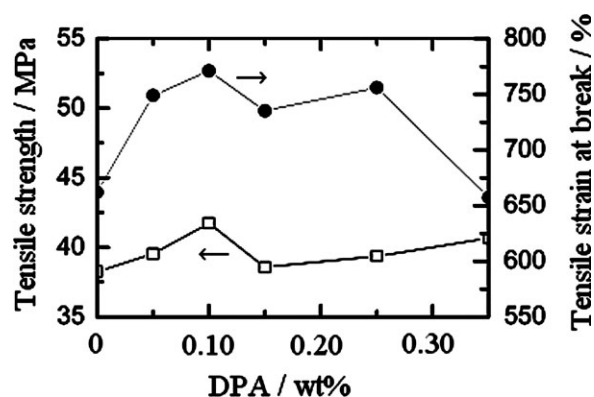


Figure 5 Tensile strength and tensile strain at break of iPP samples doped with different amounts of DPA.

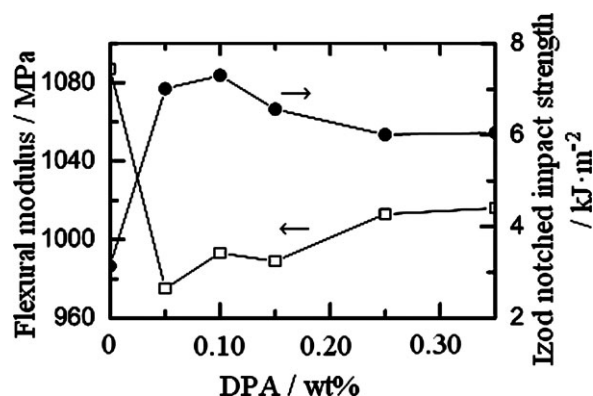


Figure 6 Flexural modulus and Izod notched impact strength of iPP samples doped with different amounts of DPA.

might be related to the β-to-α transition occurring during necking, which leads to the formation of an α phase of enhanced strength. The impact strength and toughness of β-iPP also exceed those of α-iPP.¹ The mechanical properties of iPP samples doped with different amounts of DPA are shown in Figures 5 and 6. Along with the increasing DPA content, the curve of the tensile strength varies and reaches the maximum value at 0.1 wt % DPA; the tensile strain at break increases, except for the concentration of 0.35 wt % DPA, and the maximum value is also attained at 0.1 wt % DPA. The flexural modulus decreases to a minimum value at 0.05 wt % DPA, and then it increases somewhat; the Izod notched impact strength increases sharply and reaches the maximum value of 7.30 kJ/m² (the value of a blank iPP sample is 3.13 kJ/m²) at 0.1 wt % DPA, and then it decreases somewhat. Some relationships exist between the mechanical properties and *k*. The *k* value reaches the maximum value of 0.97 at 0.1 wt % DPA, and so the tensile strength, tensile strain at break, and Izod notched impact strength maximize at 0.1 wt % DPA. The tensile strength and flexural modulus increase somewhat after the minimum values at 0.15 and 0.05 wt % DPA, respectively; the tensile strain at break and Izod notched impact strength decrease after the maximum value at 0.1 wt % DPA because the *k* value decreases somewhat after the maximum value at 0.1 wt % DPA; that is, the contents of the α-crystalline form increase and intensify their effects on the mechanical properties.

Analysis of the β-nucleation mechanism of DPA

According to the epitaxial crystallization mechanism, a lattice match between the polymer and the nucleating agent is required. The misfit factor (*f_m*) between the two crystal structures of iPP and the nucleating agent should be less than 15% empirically. *f_m* can be calculated as $f_m = 100 \times (PB - PA)/PA$, where PA and PB are the appropriate period lengths of the substrate and polymer, respectively.⁶ Stocker et al.³⁶ and Mathieu et al.³⁷ proposed that nucleating agents with ≈0.65-nm periodicity and an orthogonal geometry of the contact face are likely to induce the β-iPP polymorph.

On the basis of this discussion, the amide compound DPA is an active β-nucleating agent for iPP. When a molten nucleated iPP sample is cooled, the macromolecular chains epitaxially deposit on the surface of the nucleating agent, and then iPP forms the specific crystal structure, so there should be a connection between the crystal structures of DPA and β-iPP. The unit cell parameters of DPA, which were calculated from the WAXD data with the computer program DICVOL91,⁴⁴ and those of β-iPP, which were first revealed by Meille et al.,⁴⁵ are summarized in Table I. The *b*-axis parameter of DPA is almost equal to the *c*-axis periodicity of β-iPP. *f_m* between the lengths of *c*_{β-iPP} and *b*_{DPA} is about 0.2%, which shows a good matching relationship between them. It also suggests that the *c* axis of β-iPP parallels the *b* axis of DPA. In addition to the matching along *c*_{β-iPP}, lateral matching also should be checked. The interchain spacing on the *ab* plane of β-iPP is 11.03 Å, whereas the length of the *a* axis of DPA is 11.77 Å. *f_m* between them is 6.3%, and this shows that one helical period of β-iPP can be packed in one span of the *a* axis of DPA. As discussed previously, the crystal structures of DPA and β-iPP satisfy the lattice matching relationship. Thus, the β-nucleation ability of DPA is good.

CONCLUSIONS

β-iPP is produced by the addition of a highly effective β-nucleating agent (DPA). WAXD has shown that *k* increases sharply with the addition of DPA and attains the maximum value of 0.97 at 0.1 wt % DPA. It has been revealed by PLM that a large number of bright and colored β spherulites can be seen

TABLE I
Unit Cell Parameters of β-iPP and DPA

	System	<i>a</i> (Å)	<i>b</i> (Å)	<i>c</i> (Å)	α (°)	β (°)	γ (°)
β-iPP	Trigonal	11.03	11.03	6.49	90	90	60
DPA	Triclinic	11.77	6.48	8.26	95.07	90.64	100.68

with the addition of DPA, especially when the amount of DPA is greater than 0.1 wt %. It has been shown by SEM that the β spherulites are like flowers and that their lamellar arrangement is curved. The toughness of β -iPP is superior to that of α -iPP, and this may be attributed to the special morphology of β spherulites. The crystal structures of DPA and β -iPP satisfy the lattice matching relationship. Thus, the β -nucleation ability of DPA is good.

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