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Polymer 46 (2005) 5258–5267

polymer

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Epitaxy growth and directed crystallization of high-density polyethylene in the oriented blends with isotactic polypropylene

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Received 3 November 2004; received in revised form 17 April 2005; accepted 21 April 2005

Available online 10 May 2005

Abstract

Various lamellar orientations of high-density polyethylene (HDPE), due to competition between bulk nucleation and interfacial nucleation, have been realized in its melt drawn blends with isotactic polypropylene (iPP) upon cooling after subjected to 160 °C for 30 min. Directed crystallization, with heterogeneous nucleation in the bulk (within domains), is defined as lamellar growth along boundary of anisotropic domains and is favored in larger domains at higher temperature (slow cooling), since overgrowth of lamellae can feel the interface rather than impingement with neighbor ones as a result of scare nuclei at higher temperature. Moreover, lamellar growth caused by directed crystallization is dependent of dimension of confinement. Due to 2D confinement of cylindrical domains, lamellae can only grow along the axis of cylinder and thus *b*-axis orientation is formed. While in the layered domains with 1D confinement, however, lamellae grow with the normal of (110) plane along the melt drawn direction. On the other hand, epitaxial growth of HDPE chains onto iPP lamellae is related to the surface-induced crystallization and dominated by the interfacial nucleation. Only interfacial nucleation is preferred can epitaxial growth occur. Therefore, retarded crystallization, realized by either strong confinement in finer domains or rapid cooling or both, is favorable for it.

Keywords: Directed crystallization; Epitaxy; HDPE/iPP blends

1. Introduction

In general, crystallization of polymers proceeds via initial nucleation and subsequent growth. Due to building up of stress among amorphous regions, radially growing lamella splay and branching, and thus usually a spherulite with random lamellae forms [1]. While a polymer or a segment is dispersed in the other specie with finer domain size, however, specific lamellar growth can usually be encountered, especially for anisotropic domains [2–4]. Lamellar growth can be templated by the anisotropic phase boundary of the cylindrical or lamellar structure, since it can feel the constraint of boundary and then grow along it, especially at higher temperature due to lack of nuclei. Polyethylene segment in PE-*b*-aPP diblock copolymer can form *b*-axis orientation, i.e. growth of lamellae along the boundary, while it crystallizes at 95 °C, since the reflection of (110) plane of polyethylene is about 34° apart from meridian and meanwhile that of (200) remains at equator [5]. Same lamellar orientation of polyethylene can be formed in its blends with isotactic polypropylene [6]. In addition to polyethylene, specific lamellar growth of other polymers can also be found in the their blends, such as poly(ethylene oxide) in its blends with poly (butylenes succinate) [7] and poly(vinylidene fluoride) in its blends with nylon 11 [8]. In the above-mentioned cases, the effect of boundary is stressed to obstacle the growth of lamellae, rather than induce the crystallization at their interface. Moreover, lamellar orientation is related to the direction of lamellar growth along anisotropic domains.

Epitaxial growth, as a specific case of surface-induced crystallization, is originated from the interactions between

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doi:10.1016/j.polymer.2005.04.052

two polymers at their interface. Polymer chains can only be deposited on the substrate along specific direction due to lattice matching. A well-established epitaxial growth is found in many systems, especially for polyethylene/isotactic polypropylene pair [9–13]. The mechanism is that epitaxially grown high-density polyethylene chains interact with rows of methyl groups that populate the (010) planes of the isotactic polypropylene alpha crystal, since polyethylene chains fit exactly into the valleys formed by the methyl groups. Epitaxy is dependent on confined space and twisted lamellae can appear with reducing confinement. Moreover, epitaxy is can only be realized through secondary nucleation at interfaces, and thus is related to crystallization temperature and cooling rate [14].

So far, morphological and structural investigations for HDPE/iPP blends have been conducted in many groups, due to its technical importance [15–17]. Moreover, it is expected to enhance the mechanical properties through improvement of interfacial adhesion, since HDPE/iPP blends are thermodynamically immiscible. Epitaxial growth has been proven to be an effective way to strengthen the interface due to lamellar interaction [18,19]. Epitaxial growth of polyethylene lamellae onto that of isotactic polypropylene can be obtained through annealing of drawn blends of HDPE/iPP, which has been identified by X-ray scattering, with maximum reflection of (110) plane of HDPE about 50° apart from the drawing direction and (200) plane still in the equator [20,21]. In addition to epitaxial growth, *b*-axis orientation of HDPE in its annealed drawn blends with iPP has been reported, with maximum reflection of (110) plane of HDPE about 34° apart from the drawing direction and (200) plane still in the equator [6]. In above two cases, either epitaxy or *b*-axis orientation can be obtained in respective experiments. In recent, as for the drawn HDPE/iPP = 75/25 blend, after annealed even up to 180 °C, the orientation of HDPE remains along drawing direction, with reflections of both (110) and (200) plane of HDPE in the equator [22]. This special orientation is expected to arise from that HDPE chains near the interfaces cannot relax and can keep their original orientation. Up to now, the mechanism of various lamellar orientations of HDPE component in annealed drawn HDPE/iPP blends still remains unclear, let alone the relationship between structure and mechanical properties.

In our previous paper [23], lamellar orientation of high-density polyethylene (HDPE) in its blends with isotactic polypropylene (iPP), subjected to the dynamic packing injection molding, has been investigated with aid of 2D small-angel/wide-angle X-ray scattering (SAXS/WAXS). In iPP-rich compositions, dispersed HDPE component crystallizes to form peculiar lamellar orientation, with its normal about ±50° apart from flow direction. This special lamellar orientation is resulted from the epitaxial growth of HDPE onto iPP lamella with preferred interfacial nucleation due to depression of bulk nucleation in the fine domains. However, the factors affecting epitaxial growth in the HDPE/iPP blends remain unclear. In this case, therefore, our attention will be focused on the effect of composition, cooling rate and molecular weight on the lamellar orientation of HDPE in its melt drawn blends with iPP upon cooling after subjected to 160 °C for 30 min, with aid of 2D WAXS and scanning electron microscopy (SEM).

2. Experimental

2.1. Materials and sample preparation

Isotactic polypropylene (iPP), supplied by the Dusanzi Limited Company, had a melt flow index (MFI) of 1 g/10 min and a density of 0.910 g cm^{-3}. High-density polyethylene (HDPE) with higher molecular weight, supplied by Daqing Petrochemical Corp., had a MFI of 0.3 g/10 min and a density of 0.961 g cm^{-3}, and that with lower molecular weight, supplied by Yansan Petrochemical Corp., had a MFI of 5 g/10 min and a density of 0.968 g cm^{-3}. All compositions of HDPE/iPP blends were labeled by the percentage of iPP and a prefix with 'H' and 'L' to present HDPE with higher and lower molecular weight, respectively. For instance, H30PP represents 30 wt% of iPP in its blend with higher molecular weight HDPE. Tapes with thickness of 1 mm were firstly extruded using Haake counter-rotating twin-screw extruder with a barrel temperature of 160–200 °C, then followed by the die drawing with aid of rollers. After drawing, the thickness of tapes was reduced to about 0.3 mm and the drawing ratio was estimated to be eight (defined by the ratio of original cross sectional area to the drawn one). Thereafter, the drawn tapes were kept at 160 °C for 30 min in a hot press with a pressure of 5 MPa in order to prevent shrinkage, followed by cooled down to room temperature with a rate of 0.3 °C/min (slow cooling) or several 100°/min (quenching). At 160 °C, only HDPE component was melted and iPP component remain its original oriented structure caused by melt drawing.

2.2. Two-dimensional wide-angle X-ray scattering (2D WAXS)

Two-dimensional WAXS experiments were conducted using a Rigaku Denki RAD-B diffractometer. The wavelength of the monochromated X-ray from Cu Kα radiation was 0.154 nm. Samples were placed with the drawing direction perpendicular to the beam. Background scattering was subtracted from all 2D WAXS patterns. Azimuthal scans of 2D WAXS pattern were made for (110) and (200) plane of HDPE at a step of 0.1° from 0 to 360°.

2.3. Scanning electron microscope (SEM)

Specimens were firstly etched chemically by 1% solution of potassium permanganate in a 10:4:1 (by volume) mixture, of concentrated sulphuric acid, 85%

orthophosphoric acid and water [24], respectively; and then the surface was coated with gold and subsequently examined by an X-650 Hitachi scanning electron microscope at 20 kV.

3. Results

3.1. Crystallographic orientations

Two-dimensional WAXS patterns of samples with higher molecular weight HDPE upon slow cooling are shown in Fig. 1. The drawing direction is vertical. From inner outward, the reflections are originated from (110), (040), (130) planes of alpha modification of iPP and (110), (200) planes of orthorhombic modification of HDPE. Note that the reflection of (111) and (−131) planes of iPP can be overlapped with that of (110) plane of HDPE, due to similar diffraction angle. Evidently, for these three samples, named by H15PP, H30PP and H70PP, respectively, strong reflections of (*hk*0) plane for iPP component at the equator indicate that molecular chains of iPP are preferentially oriented along drawing direction. Four reflections around the meridian also emerge for the (110) plane of iPP, especially for H70PP, indication of lamellar branching through homoepitaxy between alpha crystals themselves [25,26]. The highly oriented structure should arise from the die drawing and remains after subjected to 160 °C. At 160 °C, however, HDPE component can be melted completely and then crystallizes upon cooling. In other words, the structure formed in the die drawing can be disintegrated and new structure is formed upon cooling. Therefore, the crystallographic orientation of HDPE component should be related to its crystallization in the presence of solid iPP component and can be used to investigate the effect of interface and space on the lamellar orientation. The reflections of (110) and (200) planes of HDPE are carefully picked out and their azimuthal scans are also included in Fig. 1. As for the reflection of (110) plane of above three samples, a common peak appears at an angle about 50° apart from the meridian, drawing direction. Combination with the permanent equatorial reflection of (200) plane, this special reflection can be ascribed to the epitaxial growth of HDPE lamella onto that of iPP, since HDPE chains are inclined about 50° to the iPP chain axis and the contact planes are established to be (100) HDPE and (010) iPP [14]. However, the reflection of (110) plane differs from each other while the angle is lower than 50°, with respect to drawing

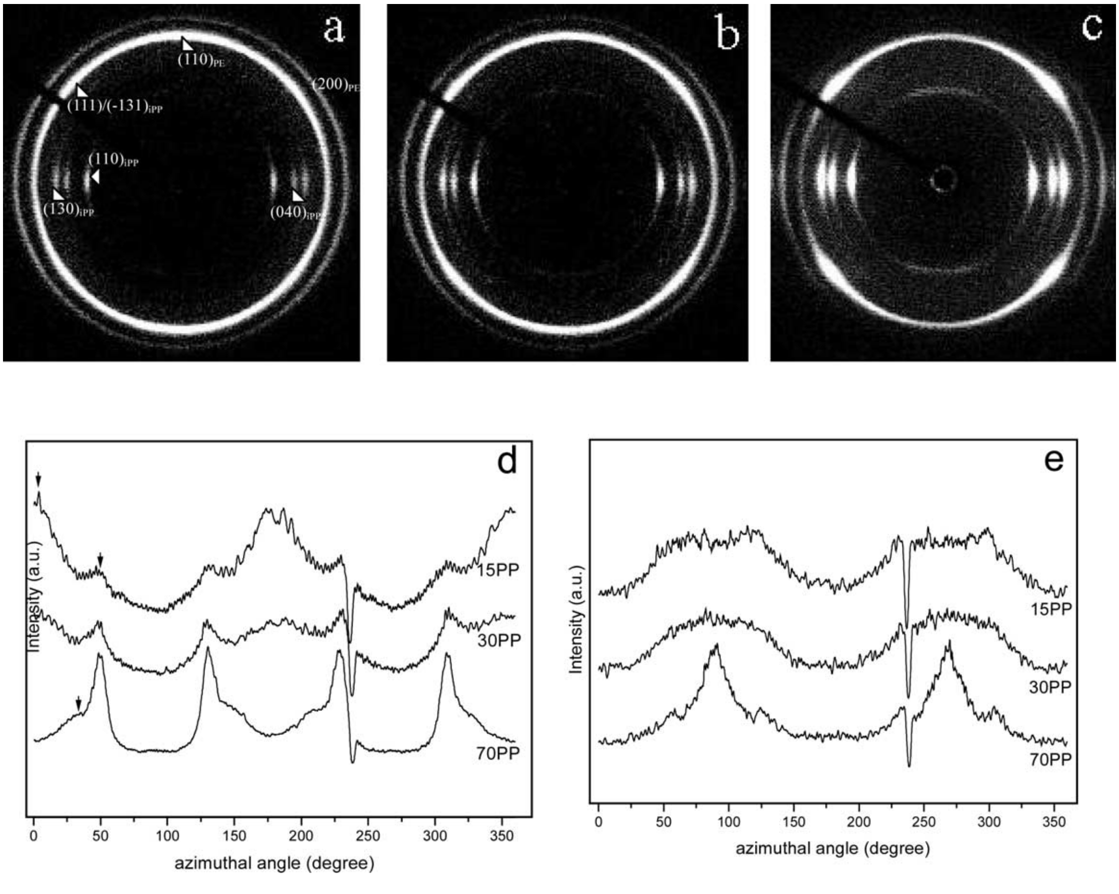

Fig. 1. Two-dimensional WAXS patterns of samples with higher molecular weight HDPE upon slow cooling to room temperature after subjected to 160 °C for 30 min, (a) H15PP, (b) H30PP, and (c) H70PP. Azimuthal scans of both (110) (d) and (200) (e) plane of HDPE for above three samples are also included. The drawing direction is vertical.

direction. As for H15PP and H30PP, with HDPE as a major component, the maximum reflection occurs at approximate to the meridian. According to the diffraction law, while the normal of (110) plane is along the meridian, *a** should be about 57° apart from the meridian, based on the parameter of unit cell. That is the fact in this case, with that the reflection of (200) manifests itself by a gradual peak at about 57° apart from meridian in both H15PP and H30PP. Note that the reflection of (200) plane at 90° is originated from epitaxy due to *a*-axis perpendicular to drawing direction. That is to say, in H15PP and H30PP, the *ab* plane is parallel to the drawing direction and *c*-axis is perpendicular to it. However, a clear-cut peak, about 34° apart from the meridian, appears in the H70PP, with HDPE as a minor component. Moreover, a relative sharp peak of (200) plane with a maximum at 90° apart from the meridian, which means that *a** is perpendicular to drawing direction, can be found for H70PP. Based on the parameter of unit cell, it can be concluded that in H70PP the *b*-axis is oriented along drawing direction, with *a*- and *c*-axis is perpendicular to it. Note that the small peak of (200) plane at about 57° in H70PP is same to that of H15PP and H30PP, indication of somewhat normal of (110) plane along drawing direction. Lamellar orientation via epitaxial growth and directed crystallization (*b*-axis orientation) is schematically shown in Fig. 2. Various molecular orientations via epixatial growth, with respect to the arrangement of side methyl (right-, left-hand and up, down) in the contact plane, are also included in Fig. 2.

While samples with higher molecular weight HDPE are subjected to quenching to room temperature, as expected, the orientation of iPP component remains, with *c*-axis along drawing direction (Fig. 3). However, as for the orientation of HDPE (Fig. 2), only the peak at about 50° apart from the meridian emerges, except for H15PP with additional relative weak reflection in the meridian. Based on the above diffraction analysis, therefore, upon rapid cooling, epitaxial growth prevails in three samples, almost independent of iPP content.

Two-dimensional WAXS patterns of samples with lower molecular weight HDPE subjected to slow cooling are shown in Fig. 4. Evidently, the crystal modification for HDPE and iPP component is also orthorhombic and monoclinic, respectively, and molecular orientation of iPP component is along melt drawing direction. Note that a weaker molecular orientation of iPP component appears in L30PP, caused by lower matrix viscosity. As for azimuthal scans of (110) and (200) planes of HDPE component (Fig. 4), only one peak at about 34° apart from the meridian appears for L30PP, whereas for L70PP a strong peak at about 50° and a weaker one at about 34° apart from the meridian show up. Combination with respective scattering of (200) plane, it can be concluded that in L30PP only *b*-axis orientation is formed and in L70PP both epitaxy and *b*-axis orientation occur. Upon quenching, the orientation of HDPE component is different from the slow cooling ones, shown in Fig. 5. There is almost no preferred orientation for both (110) and (200) planes of HDPE in L30PP, presented by isotropic scattering. Moreover, as for L70PP, only epitaxy shows up, with disappearance of *b*-axis orientation.

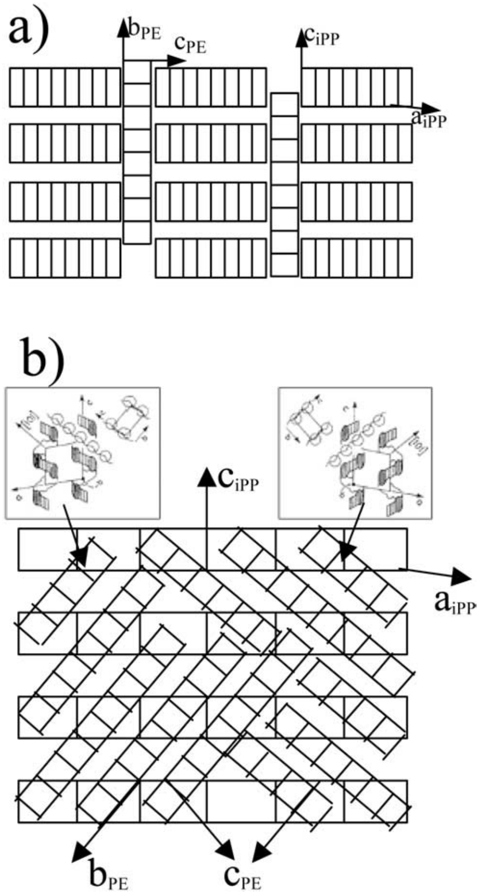

Fig. 2. Schematic diagram of lamellar growth of HDPE via directed crystallization (a) and epitaxial growth (b). Various molecular orientations via epitaxial growth, with respect to the arrangement of side methyl (right-, left-hand and up, down) in the contact plane, are also included. Note that lamellar thickness is abitary.

3.2. Phase morphology

Before SEM observation, samples were treated by the chemical etchant to improve the contrast. Due to its weaker resistant to the etchant, iPP component was extracted from the samples, represented itself by the dark areas in the pictures, consistent with other results [27,28]. Both phase morphology parallel and perpendicular to the melt drawing direction are obtained, since the die drawing can induce anisotropic deformation of dispersed phase and thus different morphologies. Figs. 6 and 7 are SEM pictures of samples with higher molecular weight HDPE and lower molecular weight one, respectively. Clearly, while HDPE as a major component, layered and irregular cylindrical structure show up for higher molecular weight HDPE and lower molecular weight one, respectively. Moreover, as for H15PP and H30PP, with increasing of iPP content from 15

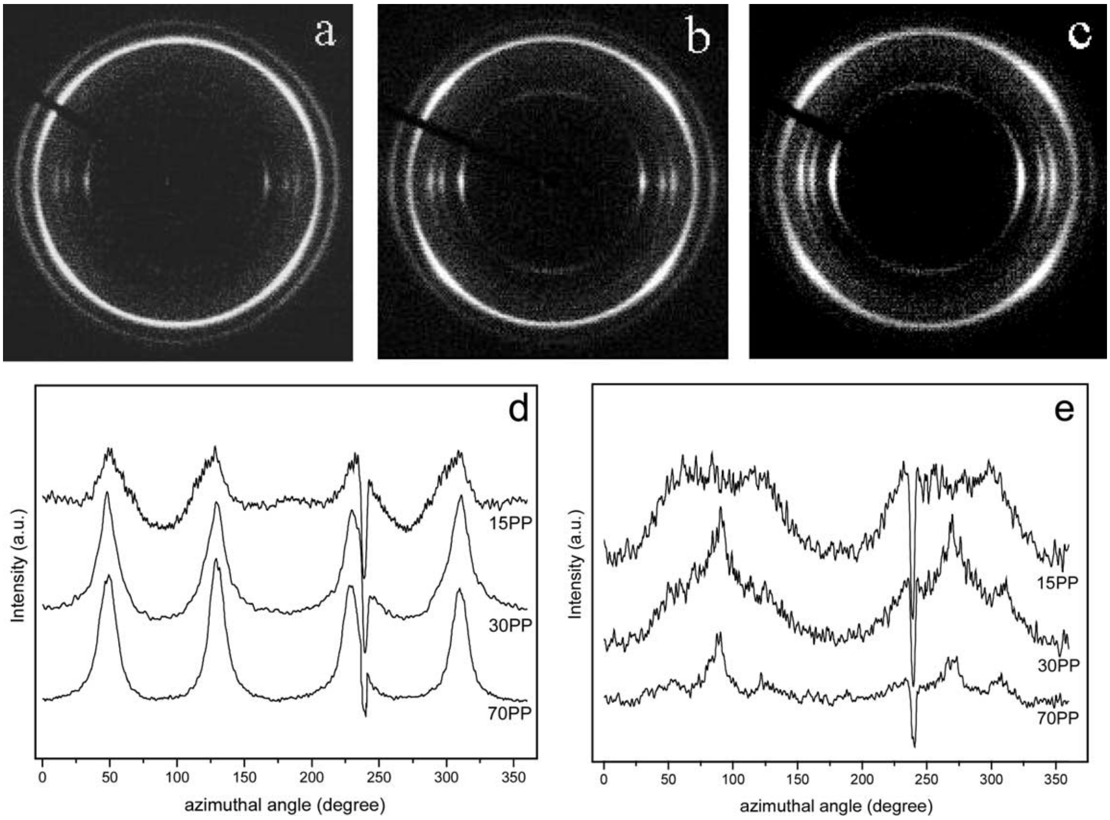

Fig. 3. Two-dimensional WAXS patterns of samples with higher molecular weight HDPE upon quenching to room temperature after subjected to 160 °C for 30 min, (a) H15PP, (b) H30PP, and (c) H70PP. Azimuthal scans of both (110) (d) and (200) (e) plane of HDPE for above three samples are also included. The drawing direction is vertical.

up to 30 wt%, the thickness of HDPE layer decreases, from about 1.5 down to 0.5 μm. While HDPE is dispersed in the iPP matrix, however, a cylindrical structure forms in H70PP, with a diameter of about 0.4 μm, whereas a separated layered structure shows up in L70PP with a thickness of about 0.4 μm. Note that the domain size is not uniform, with a broad distribution, since mechanical blending can lead to local inhomogeneity in the flow field [29]. Therefore, it can be concluded that the anisotropic phase morphology always shows up, for both higher molecular weight HDPE and lower molecular weight one, independent of compositions. The domain size of HDPE component in the blends is related to the viscosity ratio and elongational flow field encountered in the die drawing [30].

4. Discussion

4.1. Directed crystallization and epitaxy

Specific lamellar growth via nucleation in the bulk, namely directed crystallization, arises from the constraint of boundary in the anisotropic domains, especially at higher temperature, since at higher temperature sufficiently grown lamellae can feel the interface, rather than impinge with neighbor ones due to lack of nuclei. Therefore, it can be expected that slow cooling or isothermal condition is favorable for directed crystallization. Moreover, relative larger anisotropic domain is preferred, since it will take more time to realize directed crystallization in finer domains. On the other hand, as for epitaxial growth, due to its origin of interfacial nucleation, the grown HDPE lamellae must be smaller than that of iPP in the matching direction. Therefore, lower crystallization temperature or rapid cooling is good to epitaxial growth. Moreover, of importance is retarded crystallization caused by finer domain. The straightforward interpretation is that crystallization in finer domains can be significantly inhibited due to lack of nucleation sites in the bulk and thus favor nucleation at interfaces upon cooling. Based on above arguments, it is evident that directed crystallization and epitaxy can be modulated, with changing of cooling rate and domain size. Note that, while the domain size exceeds a critical value, it is unfavorable for either directed crystallization or epitaxial growth and lamellar growth will be random, resulting in an isotropic scattering in 2D WAXS patterns.

Specific molecular orientation will lead to anisotropic scatterings in the 2D WAXS patterns. To our knowledge, anisotropic scattering of (110) plane of polyethylene can be separated by subtraction of isotropic scattering (contributed by random crystal and amorphous part) from total scattering (contributed by oriented and random crystal and amorphous

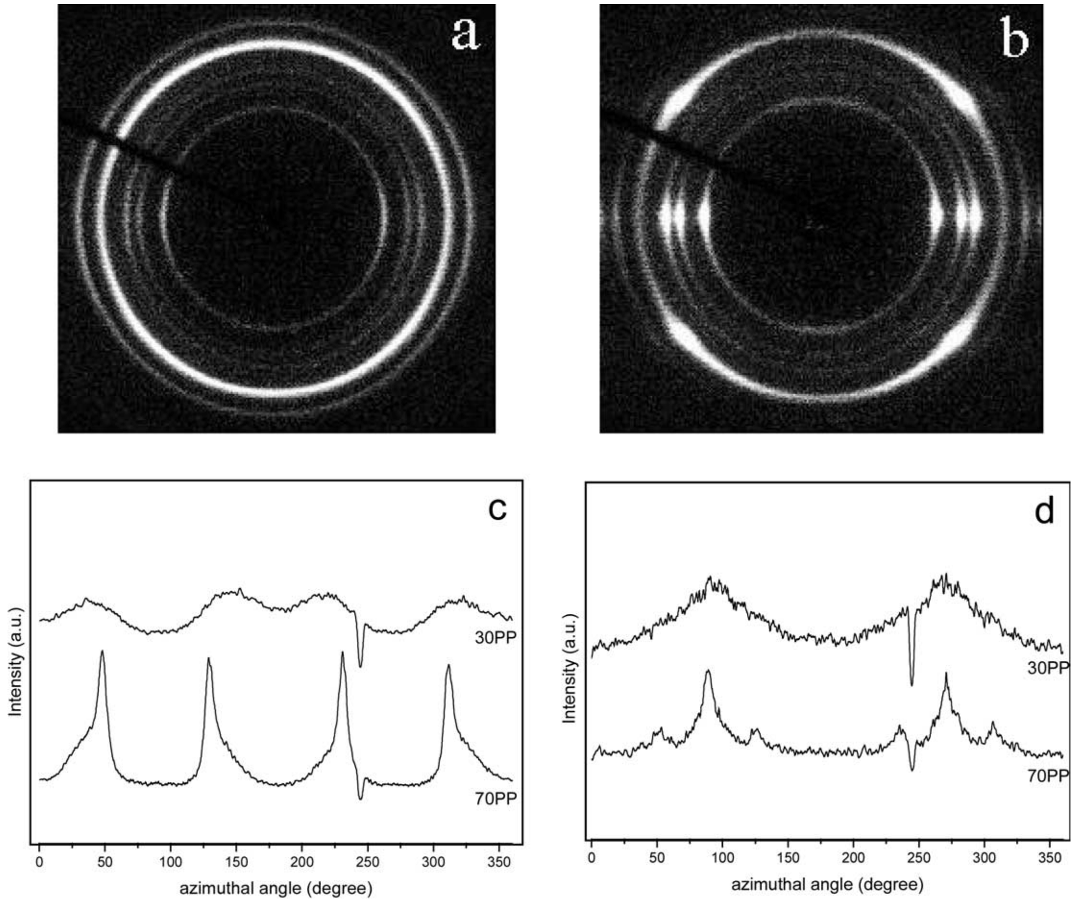

Fig. 4. Two-dimensional WAXS patterns of samples with lower molecular weight HDPE upon slow cooling to room temperature after subjected to 160 °C for 30 min, (a) L30PP, and (b) L70PP. Azimuthal scans of both (110) (c) and (200) (d) plane of HDPE for above two samples are also included. The drawing direction is vertical.

part), and thus orientation fraction f_{orient} (contributed by both directed crystallization and epitaxy) can be obtained through Eq. (1).

$$f_{\text{orient}} = \frac{\int_0^{\pi/2}(I_{\text{total}} - I_{\text{isotropic}})\mathrm{d}\theta}{\int_0^{\pi/2} I_{\text{total}}\mathrm{d}\theta} \tag{1}$$

where I_{total} is the total diffraction intensity, $I_{\text{isotropic}}$ is the isotropic diffraction intensity (base line) and θ is azimuthal angle.

Moreover, with consideration of symmetry of reflection peak, the epitaxy fraction f_{epitaxy} can also be calculated through Eq. (2)

$$f_{\text{epitaxy}} = \frac{A_1}{Af_{\text{orient}}} \tag{2}$$

where

$$A = \int_0^{\pi/2}(I_{\text{total}} - I_{\text{isotropic}})\mathrm{d}\theta$$

and A_1 is the integration intensity from epitaxial growth, shown by the shadow parts (one half) in Fig. 8. The calculated orientation fraction and epitaxy fraction are shown in Table 1. Note that we acknowledge that the calculation is somewhat rough but informative.

4.2. Effect of composition

From Table 1, the orientation fraction is dependent on composition, with larger value in blends with HDPE as a minor component and smaller one in blends with HDPE as a major component. Combination with the phase morphology, increasing orientation fraction with iPP content is related to

Table 1
The orientation fraction, epitaxy fraction of HDPE/iPP blends with different molecular weight HDPE upon either slow cooling at a rate of 0.3 °C/min or quenching to room temperature after subjected to 160 °C for 30 min

	Slow cooling		Quenching	
	Orientation fraction	Epitaxy fraction	Orientation fraction	Epitaxy fraction
H15PP	0.52	0.17	0.36	0.28
H30PP	0.54	0.22	0.42	0.37
H70PP	0.61	0.37	0.51	0.47
L30PP	0.36	0	0.08	0
L70PP	0.55	0.30	0.43	0.40

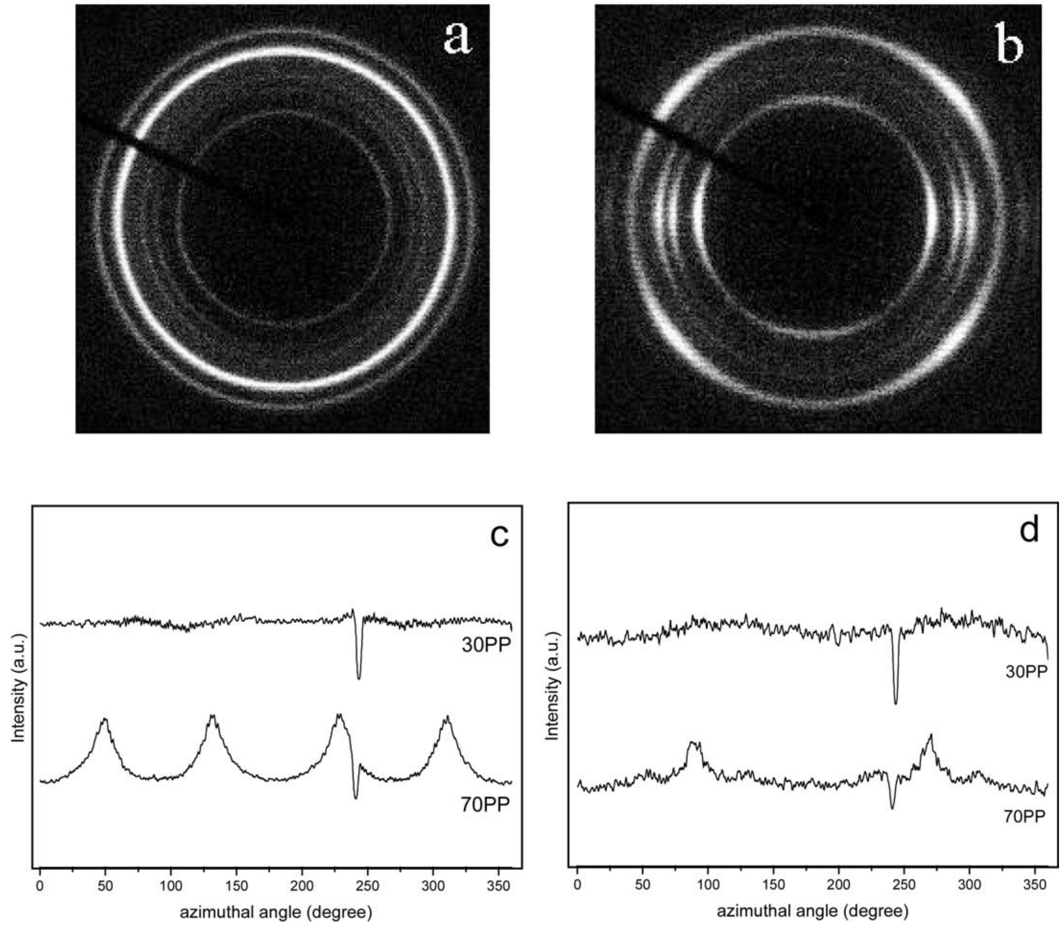

Fig. 5. Two-dimensional WAXS patterns of samples with lower molecular weight HDPE upon quenching to room temperature after subjected to 160 °C for 30 min, (a) L30PP, and (b) L70PP. Azimuthal scans of both (110) (c) and (200) (d) plane of HDPE for above two samples are also included. The drawing direction is vertical.

the reducing of domain size, independent on molecular weight. It can be expected that lamellar orientation is random in the larger domains due to numerous nuclei formed simultaneously (such as L30PP upon quenching). Moreover, epitaxy fraction increases significantly with reducing of domain size, indicating that smaller domain size is good to epitaxial growth, especially for HDPE with lower molecular weight (no epitaxy occurs at all in L30PP with larger domain size). Therefore, it can be concluded that epitaxial growth is more sensitive to domain size, as compared with directed crystallization. The mechanism is that only in narrow space can nucleation in its bulk be effectively inhibited and thus interfacial nucleation is favored. On the other hand, as for directed crystallization nucleated in the bulk, the specific lamellar growth only arises from the constraint of interface. Various lamellar growths via directed crystallization can be occurred with changing of composition. It has been demonstrated that *b*-axis is oriented along melt drawing direction in the cylindrical domains, such as H70PP, whereas the normal of (110) plane parallel to the melt drawing direction show up in the layered structure, such as H15PP and H30PP. The different lamellar orientation via directed crystallization is related to the dimension of confinement. Due to 2D confinement in the cylinder, lamellae can only grow along the axis of cylinder and thus *b*-axis orientation forms, since *b*-axis is the rapidest growth direction for polyethylene [6]. In the layered structure with 1D confinement, however, lamellae grow along at an angle apart from the drawing direction, which may be related to the longest growing path in case of impingement with neighbor ones. The detail mechanism needs to be further investigated. Therefore, it can be concluded that whether nucleation in its bulk or at the interface is related to the domain size, dependent of composition. Smaller domain size is preferred for epitaxial growth due to depression of nucleation in its bulk.

4.3. Effect of cooling rate

Upon slow cooling, it is evident that orientation fraction increases but epitaxy fraction decreases, as compared with that upon quenching. Note that in this case we choose slow cooling and quenching as two extreme conditions to study the effect of cooling rate on the oriented crystallization.

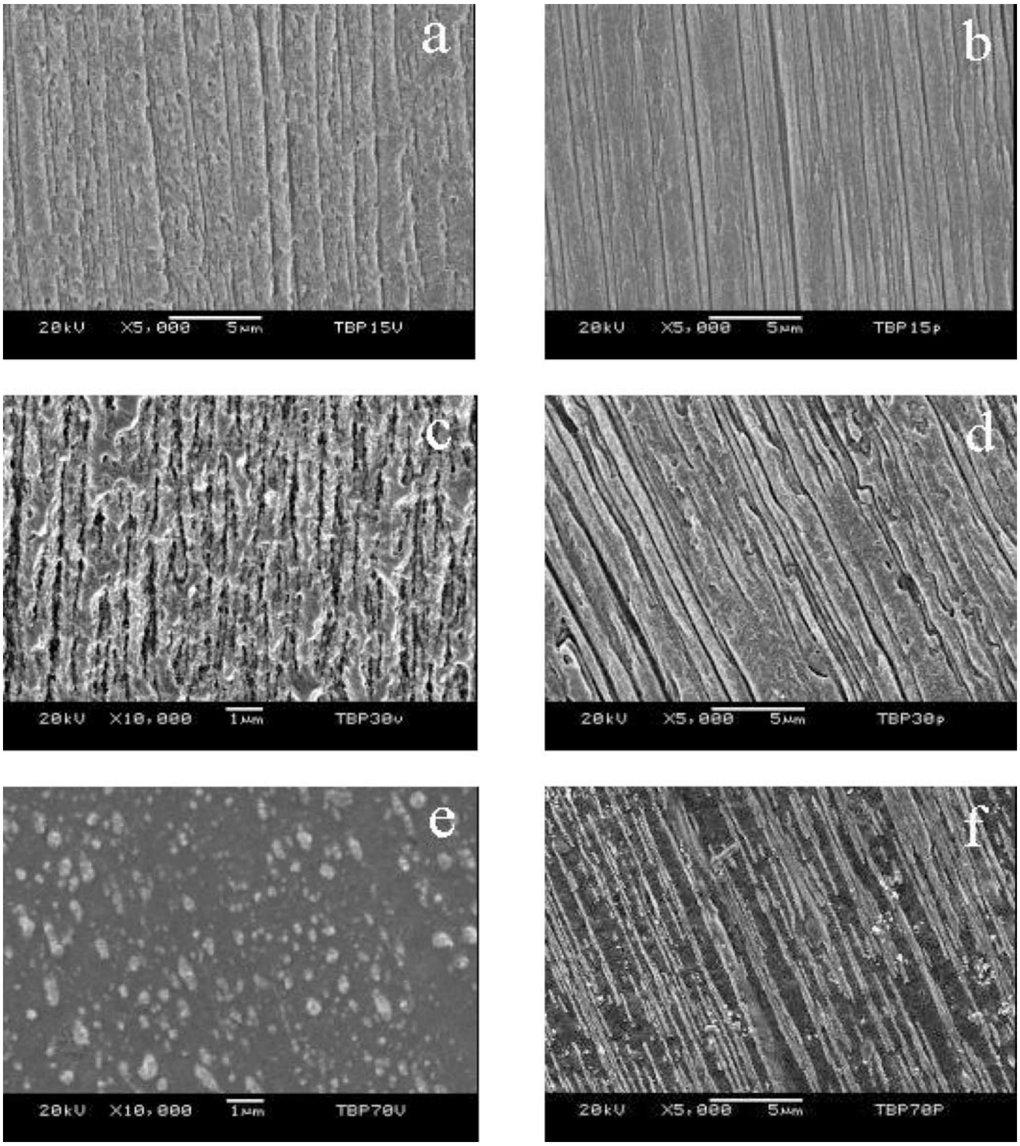

Fig. 6. SEM pictures of samples with higher molecular weight HDPE parallel (right column) and perpendicular (left column) to the drawing direction, respectively, (a) (b) H15PP, (c) (d) H30PP, and (e) (f) H70PP.

Upon slow cooling, most of chains can crystallize via directed crystallization in the domains rather than epitaxial growth at interface, since more time available at higher temperature. Therefore, chains available for epitaxy decrease and so does the epitaxy fraction. Just mentioned above, due to significantly retarded crystallization in much finer domain, chains in these domains can only crystallize through epitaxial growth at interfaces, even upon slow cooling. If the slower cooling rate or isothermal crystallization at higher temperature can be realized, it can be expected that only directed crystallization occurs and epitaxial growth disappears entirely. From above results, it has been demonstrated that cooling rate can also affect the lamellar orientation due to different temperature ranges favorable for directed crystallization and epitaxial growth, respectively.

4.4. *Effect of molecular weight*

At the same composition, the orientation fraction in the sample with higher molecular weight is higher than that in the sample with lower molecular weight, especially while HDPE as major component. Evidently, the higher orientation fraction is related to the smaller domain size in H15PP and H30PP. The same rule can also be suitable for epitaxy fraction. On the other hand, isothermal crystallization indicates that the rate of crystallization is higher for HDPE with lower molecular weight, as a result of lower density of entanglement. Rapid nucleation and growth in its bulk is less benefited for directed crystallization and even for epitaxial growth. It can be demonstrated by the result of L30PP upon quenching, with random lamellar orientation due to rapid nucleation in the bulk.

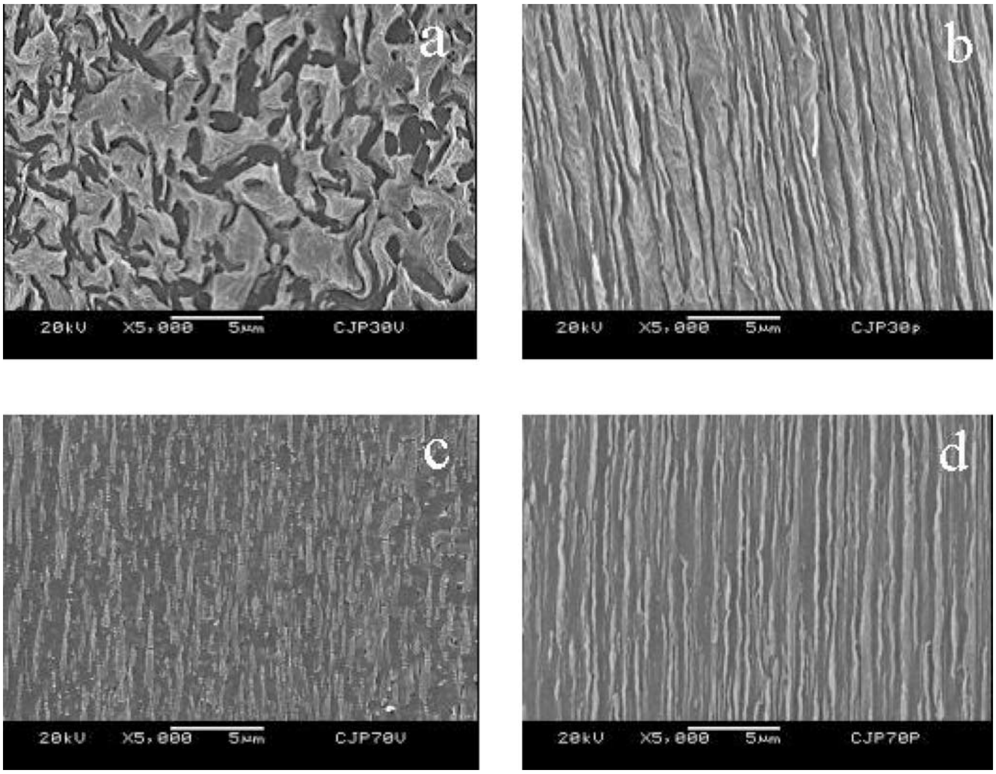

Fig. 7. SEM pictures of samples with lower molecular weight HDPE parallel (right column) and perpendicular (left column) to the drawing direction, respectively, (a) (b) L30PP, and (c) (d) L70PP.

5. Conclusion

With aid of 2D WAXS and SEM, specific lamellar growth of HDPE component, via directed crystallization or epitaxy or both, has been well understood in the melt drawn HDPE/iPP blends upon cooling after subjected to 160 °C for 30 min. Retarded bulk crystallization is favorable for epitaxial growth at interface, whereas directed crystallization is realized in the bulk under the effect of boundary. Therefore, factors, such as composition, molecular weight and cooling rate, can favor either directed crystallization or epitaxy by affecting the competition between bulk nucleation and interfacial nucleation. As for directed crystallization, due to its sensitive to the boundary, however, two different lamellar orientations have been found in layered and cylindrical domains, receptively.

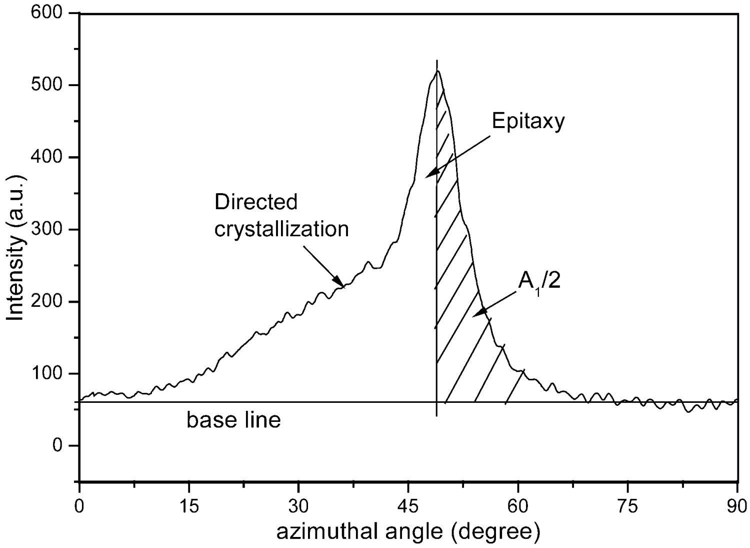

Fig. 8. Schematic diagram of orientation fraction and epitaxy fraction calculated from the azimuthal scans of (110) plane of polyethylene. The shadow is the half integration intensity from epitaxial growth.

Lamellae can only grow along the axis of cylinder, and thus *b*-axis orientation forms, as a result of 2D confinement in the cylindrical domains. However, lamellar growth, with the normal of (110) plane parallel to the drawing direction, is realized in the layered domains with 1D confinement.

Acknowledgements

We would like to express our great thanks to the National Natural Science Foundation of China (20274028, 50373030 and 20490220) for Financial Support. This work is also partly supported by Ministry of Education of China for Doctoral Degree (20020610004).

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