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Tensile properties in the oriented blends of high-density polyethylene and isotactic polypropylene obtained by dynamic packing injection molding

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Abstract

In this article, tensile properties have been discussed in terms of phase morphology, crystallinity and molecular orientation in the HDPE/iPP blends, prepared via dynamic packing injection molding, with aid of scanning electron microscopy (SEM), differential scanning calorimetry (DSC) as well as two dimensional X-ray scattering (2D WAXS). For the un-oriented blends, the tensile properties (tensile strength and modulus) are mainly dominated by the phase morphology and interfacial adhesion related to the influenced crystallization between HDPE and iPP component. A maximum in tensile strength and modulus is found at iPP content in the range of 70–80 v/v%. As for the oriented blends, however, the presence of dispersed phase in the blends, independent of phase morphology and crystallinity, always makes tensile properties to be deteriorated through reducing molecular orientation of matrix. It is molecular orientation of matrix that determines the tensile properties of oriented blends. In the blends with HDPE as matrix, steep decreasing of tensile properties is related to the rapid reducing of molecular orientation of HDPE, whereas in the blends with iPP as a major component, slight decreasing of molecular orientation of iPP results in slight reducing of tensile properties. Other factors, such as interfacial properties and phase morphology, seem to be little contribution to the modulus and tensile strength.

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Keywords: Tensile properties; Orientation; HDPE/iPP blends

1. Introduction

Studies of high-density polyethylene (HDPE) and isotactic polypropylene (iPP) are motivated, in part, by their technological importance, especially in the form of a blend [1–3]. In general, in systems of two polymers that can crystallize the mechanical properties are strongly related to their crystallinity, crystalline morphology, interfacial adhesion and degree of dispersion [4,5]. The presence of dispersed particles might cause lager changes not only in the morphology of the continuous phase but also in the overall crystallization and crystal morphology [6–8]. Since, HDPE and iPP are generally immiscible and incompatible, their mixtures are expected to be poor in mechanical properties [9]. However, some literatures have documented that there exists a maximum in tensile strength and modulus at a certain composition [10,11]. The synergistic effects are related to the crystal morphology, especially the profusion of intercrystalline links introduced by HDPE. The presence of HDPE or iPP crystals can, serving as nucleation sites, influence the crystallization of the other, due to their approximate crystallization temperature range at absence of stress [8,12]. Therefore, the mechanical properties are strongly influenced by their thermal history. The modulus, that exceeds the upper bound calculated from the Voigt model, has been found in the rapidly quenched HDPE/iPP blends [13]. Also, transmission electron microscopy (TEM) revealed that the blends were immiscible and both matrix-droplet and co-continuous morphologies were observed. The unexpected modules in blends are related to the change of crystallization of each component in their blends during quench. On the other hand, fibrous morphology, obtained by drawing HDPE/iPP blends at solid state, can be significant to enhance the tensile strength and modulus. Moreover, the annealing, at a temperature intermediate the melting point of two components, can result in the epitaxial growth of

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lamellae of HDPE onto that of iPP [14]. Epitaxial growth is helpful for the improvement of modulus, due to the bridging of the amorphous interlamellar phase of one component by the crystalline lamellar component of the other phase [15–17].

To better understand the dependence of tensile properties of HDPE/iPP blends on the phase morphology, crystallinity, particular on the molecular orientation, HDPE/iPP blends in the whole composition range were firstly prepared by co-rotating twin-screw extruder, and then were dynamic packing injection molded to control the molecular orientation. The tensile properties of such HDPE/iPP blends were investigated in detail, based on the phase morphology, crystallinity and molecular orientation, with aid of scanning electron microscopy (SEM), differential scanning calorimetry (DSC), and two-dimensional wide angle X-ray scattering (2D WAXS).

2. Experimental

2.1. Materials and sample preparation

Isotactic polypropylene (iPP), supplied by the Duzisan Limited Company, had a melt flow index (MFI) of 3 g/10 min and a density of 0.91 g cm^{-3}. High-density polyethylene (HDPE), supplied by Yansan Petrochemical Corp., had a MFI of 15 g/10 min and a density of 0.968 g cm^{-3}. Melt blending was conducted using TSSJ-25 co-rotating twin-screw extruder with a barrel temperature of 160–190 °C. After pelletized and dried, blends were injected into a mold with aid of a SZ 100 g injection-molding machine with barrel temperature of 190 °C and injection pressure of 900 kg cm^{-2}. Then dynamic packing injection molding technology was applied. Its main feature was to introduce shear to the cooling melt during packing stage by two pistons that moved reversibly with the same frequency. Shear rate was about 10 s^{-1} calculated from the geometry of mold. Detail descriptions can be found elsewhere [18]. The injection molding under static packing was also carried out by using the same processing parameters but without shear for comparison purpose. The specimen obtained by dynamic packing injection molding is called dynamic sample, whereas that obtained without shear static one. In this case, blends are labeled by the weight percentage of iPP in blends and by prefix ‘d’ and ‘s’ for dynamic and static samples, respectively. For instance, d20PP represents dynamic samples consisting of 20 iPP and 80% HDPE by weight.

2.2. Scanning electron microscope (SEM)

The specimens were firstly etched chemically by 1% solution of potassium permanganate in a 10:4:1 (by volume) mixture, respectively, of concentrated sulphuric acid, 85% orthophosphoric acid and water [19]; and then the surface was coated with gold and subsequently examined by an X-650 Hitachi scanning electron microscope at 20 KV.

2.3. Differential scanning calorimetry (DSC)

The thermal analysis of the samples was conducted using a Perkin–Elmer DSC priys-1, indium calibrated. Melting endotherms were obtained at 10 °C/min with 4–5 mg of sample in a nitrogen atmosphere. For the purpose of comparison with plain polymer, the crystallinity X_c of component i in the blends can be normalized by the equation,

$$X_c = \frac{\Delta H_i}{\Delta H_i^m \phi_i} \tag{1}$$

where ΔH_i is the enthalpy of fusion of component i, directly obtained from DSC, and ϕ_i is the mass fraction of component i in the blends. The enthalpy of fusion ΔH_i^m of 100% crystalline polymer is 293 and 207 J/g for HDPE and iPP, respectively, [20].

2.4. 2D WAXS measurements

The two dimensional wide-angle X-ray scattering experiments (2D WAXS) were conducted using a Rigaku Denki RAD-B diffractometer. The wavelength of the monochromated X-ray from Cu Kα radiation was 0.154 nm and reflection mode was used. The samples were placed with the orientation (flow direction) perpendicular to the beams. The intensity was corrected by subtracting the background scattering. Azimuthal scan of 2D WAXS were made for (110) plane of HDPE and (040) plane of iPP at a step of 1° from 0 to 360 °. The orientation of chains could be calculated by the orientation parameter f,

$$f = \frac{3\langle\cos^2\varphi\rangle - 1}{2} \tag{2}$$

$$\langle\cos^2\varphi\rangle = \frac{\int_0^{\frac{\pi}{2}} I(\phi)\sin\phi\cos^2\phi\,\mathrm{d}\phi}{\int_0^{\frac{\pi}{2}} I(\phi)\sin\phi\,\mathrm{d}\phi} \tag{3}$$

where ϕ is the angel between the normal of a given (hkl) crystal plane and shear flow direction, and I is the intensity. Its limiting values of orientation parameter f, taking $\phi=0$ as the shear flow direction, are -0.5 for a perfect perpendicular orientation and $+1.0$ for a perfect parallel orientation. An un-oriented sample gives $f=0$.

2.5. Tensile experiments

The tensile experiments were carried out with aid of Shimadzu AG-10TA Universal Testing Machine. The moving speed of crosshead was 5 and 50 mm/min for modulus and tensile strength measurements, respectively.

The measure temperature was 23 °C. The tensile strength and modulus could be directly obtained from the stress–strain curves by the provided software. The values were calculated as averages over six specimens for each composition.

3. Results

3.1. Tensile properties

The selective stress–strain curves of dynamic samples are shown in Fig. 1, including static ones for comparison. As for dynamic samples, there is no obvious necking and the fracture mode is ductile, with ultimate elongation of about 10%, except for pure HDPE with brittle fracture, which is the characteristic of extreme molecular orientation for semi-crystalline polymers. The ultimate tensile strength and modulus of dynamic samples with compositions is shown in Fig. 2. It is evident that the change of tensile strength and modulus with compositions is very similar, with abrupt decreasing in the blends with HDPE matrix and slight reducing in the ones with iPP matrix. In the blends with HDPE matrix (iPP content is less than 40 wt%), the tensile strength and modulus decrease linearly, with slope of −188 and −3.2, respectively, with increasing of iPP content up to 30 v/v%. In the blends with iPP matrix (iPP content is more than 60 wt%), the tensile strength and modulus can be also linearly fitted, with increasing HDPE content up to 30 v/v%. However, slopes are much lower, −22 and −0.67 for tensile strength and modulus, respectively, as compared with that in HDPE matrix. As for the static samples, the fracture mode is similar to that of dynamic ones, with ultimate elongation of about 15%. However, obvious necking occurs in every composition. The tensile strength and modulus of static samples is also matrix dependent, though the relevancy is much weaker. Slightly negative deviation in both tensile strength and modulus occurs in the HDPE matrix, whereas positive deviation in the iPP matrix. A maximum in tensile strength and modulus is found at iPP content in the range of 70–80 v/v%, in a good agreement with literature [10,11].

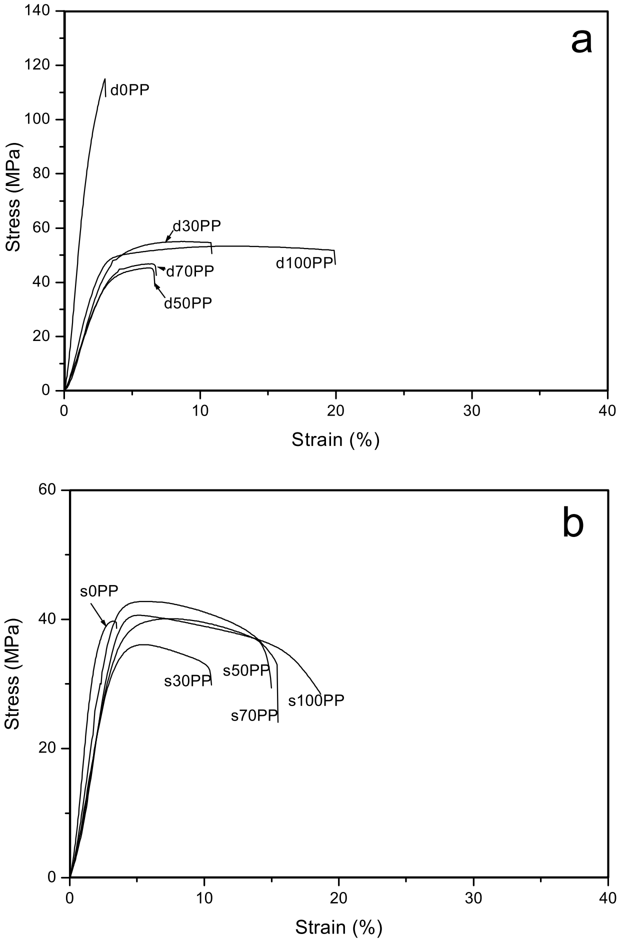

Fig. 1. Selective stress–strain curves of dynamic (a) and static (b) samples.

3.2. Phase morphology

The phase morphology of dynamic samples along the flow direction with respect to compositions is selectively shown in Fig. 3. Before SEM observation the 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 by the dark area in the pictures. One observes elongated domains of dispersed phase in all compositions, originated from the introducing of shear during packing stage. The extent to deformation of domains varies with compositions. The deformation of iPP droplets is less than that of HDPE droplets, as compared with Fig. 3(a) and Fig. 3(c). The difference of extent to deformation is related to the viscosity of matrix, since, higher viscous stress can make deformation more easily, according to Cox [21].

Fig. 4 is the SEM pattern of dynamic samples, viewed perpendicular to the flow direction, with respect to various compositions. Similar to the morphology shown in Fig. 3, typical droplet-matrix morphology is also observed, indicating that HDPE/iPP blends is immiscible. The difference is that the dispersed phase is sphere or ellipse, rather than elongated domains. The size of droplets varies with the compositions, which is related to the viscosity ratio between droplet and matrix, coarse in the HDPE matrix (i.e. d20PP), about 3 μ, and finer in the iPP matrix (i.e. d70PP), around 0.5 μ. An obvious co-continuous phase forms in the 50PP.

For static samples, one always observes a droplet-matrix or co-continuous morphology, viewed both in perpendicular and in parallel to the flow direction, no elongated domains at all (Fig. 5). In the composition of s20PP, droplets of iPP, with around 0.7 μ, are dispersed in the HDPE matrix. With increasing of iPP content, a typical co-continuous structure is formed in the composition of s50PP. While the iPP becomes the major component, droplets of HDPE form and the size is about 0.3 μ. Compared with the morphology of

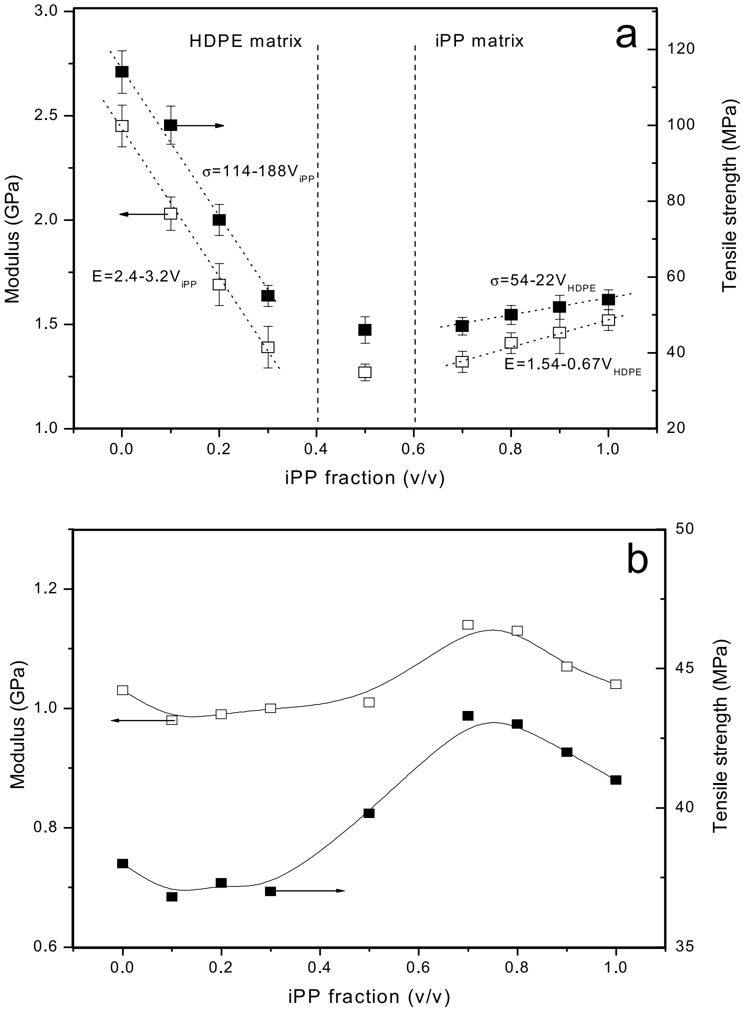

Fig. 2. Plots of modulus and tensile strength as a function of iPP fraction, including both dynamic (a) and static (b) samples. The functions in pictures are the linear fits for modulus and tensile strength with respect to fraction of dispersed phase (see text).

dynamic sample, not only the phase pattern is different, but also the degree of phase segregation is quite smaller in static sample. This can be explained by shear induced phase separation at low shear rate [22,23] and longer time for coalescence, while subjected to dynamic packing injection molding. The morphology along flow direction is similar to that perpendicular to the flow direction due to the break up and retraction of droplets in absence of shear stress (Fig. 5(d)).

3.3. Thermal analysis

Fig. 6 presents the melting trace of dynamic samples with various compositions at heating rate of 10 °C/min, and that of static samples is also included for comparison. Obviously, two separated melting peaks, corresponding to that of plain polymers, are presented. The melting peaks, around 133 and 165 °C, are attributed to the lamellar melting of HDPE and iPP, respectively. The melting point of HDPE in both dynamic and static samples is nearly constant in the its matrix, but decreased in its inclusions dispersed in the iPP matrix, by about 2.1 and 3.1 °C for dynamic (d90PP) and static (s80PP) samples, respectively. The depression of melting point of HDPE could be arisen from retarded crystallization in its inclusion, which may influence the interfacial properties between HDPE and iPP component, due to much finer size [24]. From Fig. 6(a), a higher melting peak around 137 °C, which is the characteristic of shish structure of HDPE caused by the shear-induced crystallization [25–28] via dynamic injection packing molding and absent in the static samples (Fig. 6(b)), can be also found in the HDPE-rich compositions. Moreover, the ratio of shish structure to lamellae decreases with the increasing of iPP content, originated from the decreasing of shear rate as a result of increasing viscosity, since, the formation of shish structure is dependent of the molecular extension, related to the shear rate, and its molecular weight dependent relaxation time [29–31].

The normalized crystallinity, calculated from the Eq. (1), of HDPE and iPP in the blends, are presented in Fig. 7. As

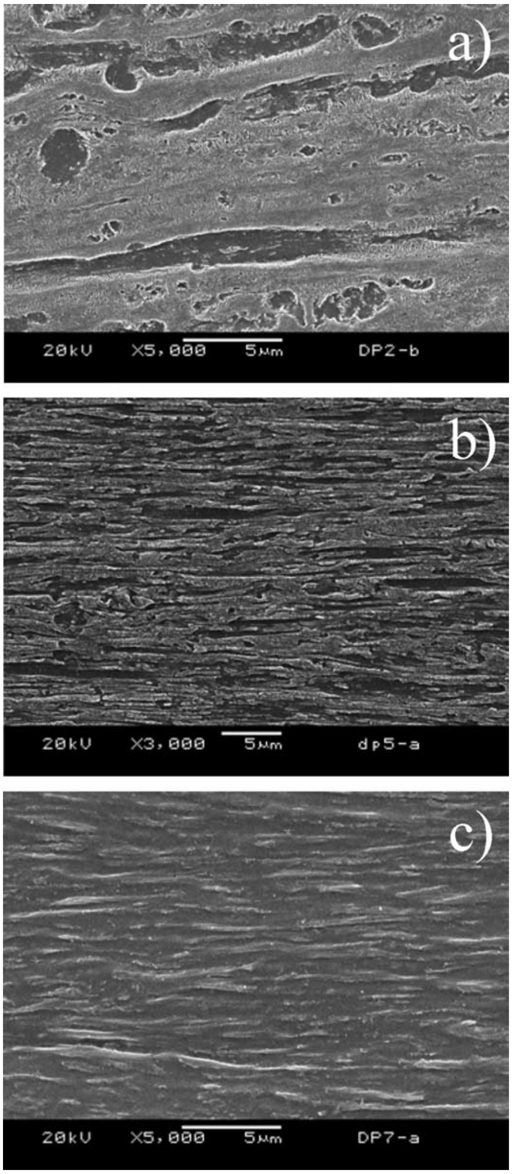

Fig. 3. SEM patterns of dynamic samples along flow direction with different composition, (a) d20PP, (b) d50PP and (c) d70PP.

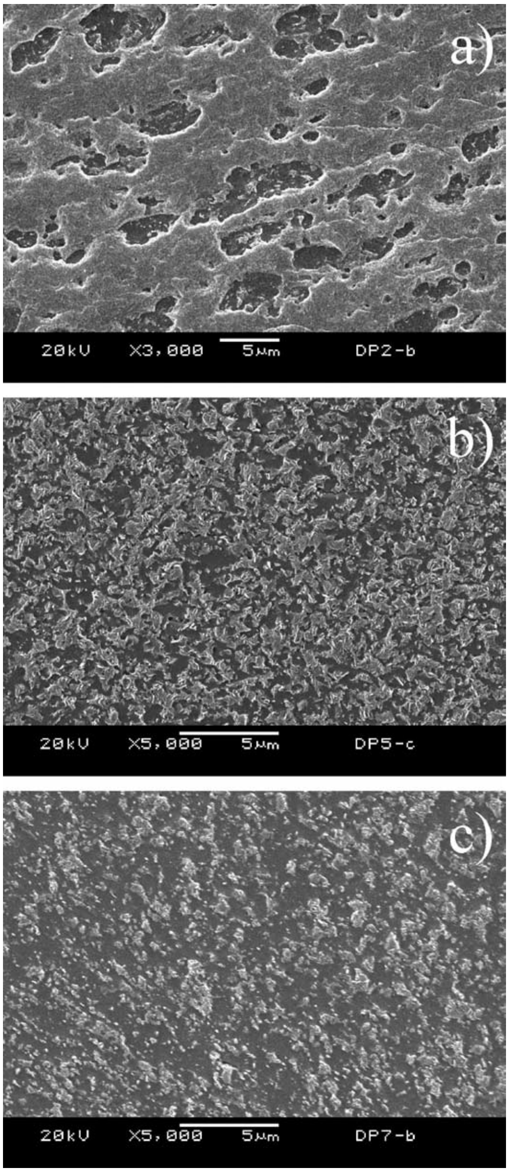

Fig. 4. SEM pictures of dynamic samples perpendicular to flow direction with different composition, (a) d20PP, (b) d50PP and (c) d70PP.

for HDPE component, Fig. 7(a), its normalized crystallinity changes with different tendency in its droplets and matrix for both dynamic and static samples. The normalized crystallinity increases linearly with the HDPE content in its droplets, whereas changes little in its matrix. As for iPP component, the normalized crystallinity changes little with respect to its content in both dynamic and static samples.

3.4. Molecular orientation

Fig. 8 is the azimuthal scans of dynamic samples, with respect to (110) plane of HDPE and (040) plane of iPP, respectively. The dotted lines in the pictures represent the position of equator, which is perpendicular to the flow direction. Chains of HDPE is preferentially oriented to the shear direction, while it forms matrix. However, a distinct orientation of HDPE chains is exhibited in its droplets, with about 50° apart from the shear direction. This special orientation is related to the epitaxial growth of HDPE lamellae onto that of iPP, which has been discussed elsewhere in detail [32]. As for iPP, its molecular orientation is always parallel to the shear direction, irrespective of compositions, though the intensity is much weaker in its droplets due to lower content.

The orientation parameter of the normal of given crystal planes, with respect to the shear direction, is shown in Fig. 9, including both dynamic and static samples. As for (110) plane of HDPE, the orientation parameter in dynamic samples varies with phase morphology. Its value is about 0 in its droplets, and decreases from about −0.2 to −0.4,

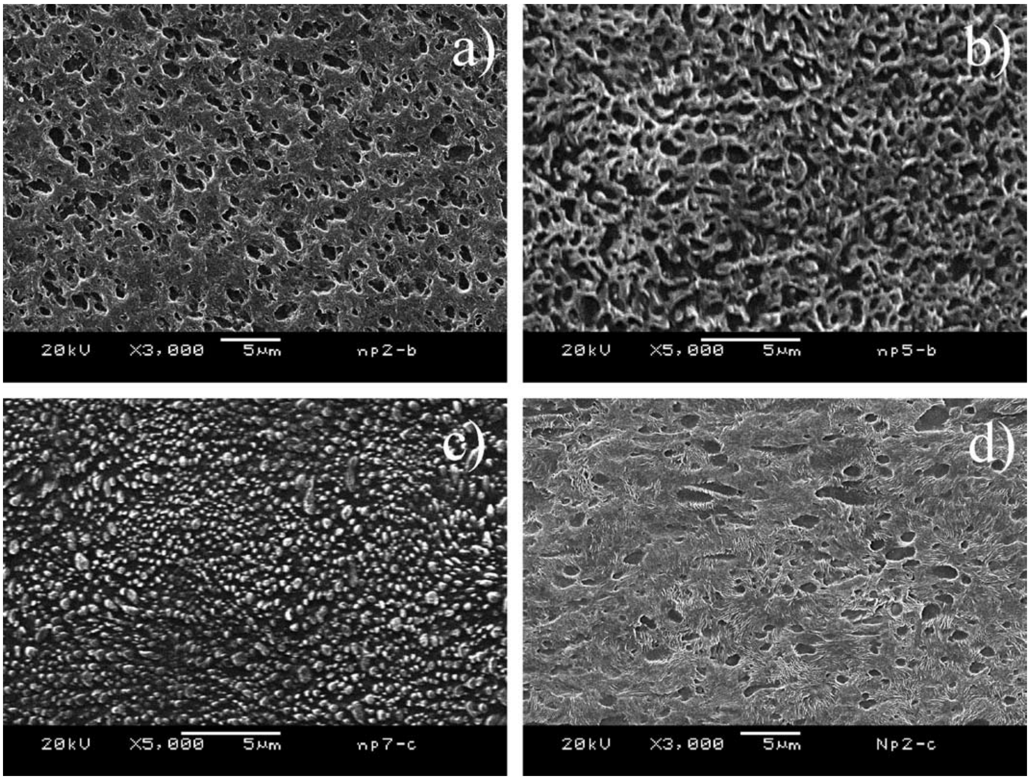

Fig. 5. SEM pictures of static samples perpendicular (a) s20PP, (b) s50PP and (c) s70PP, and parallel (d) s20PP, to the flow direction.

with reducing of iPP content, in its matrix, indication of higher molecular orientation along shear direction. The molecular orientation of HDPE in its matrix, with respect to the content of iPP, is consistent with the change of shish structure, demonstrated by DSC results. Note that $f\sim 0$ in HDPE droplets does not mean that random orientation, but originates from the special angle ($\sim 50°$) between the its normal and shear direction due to epitaxial growth. The orientation parameter of static samples is close to zero in all composition ranges for (110) plane of HDPE, indication of random orientation, though there may have some molecular orientation induced by flow during injection. Similar to that of HDPE, the orientation parameter of (040) plane of iPP in dynamic samples is also different in its matrix and droplets. While dispersed in the HDPE matrix, the value decreases slightly down to ~ -0.05, with increasing iPP content up to 50 v/v%. However, while iPP forms continuous phase, the orientation parameter reduces slightly with increasing of HDPE content. As for the static samples, the orientation parameter of (040) plane is decreased down to ~ -0.1, while increasing of iPP content up to 100%, different from that of HDPE. This much higher molecular orientation may be related to flow-induced crystallization [30,31]. From the above results, it can be concluded that the change of molecular orientation in their individual matrix, with respect to fraction of dispersed phase, is more obvious for HDPE component than that of iPP.

4. Discussion

Negative or positive deviation in tensile strength and modulus of HDPE/iPP blends, dominated by processing conditions, has been reported in many literatures [1,2,10,11]. Without orientation, the change of modulus and tensile strength, with respect to composition, can be related to the phase morphology, crystallinity and interfacial adhesion. In this case, for both tensile strength and modulus of static samples, positive deviation can be observed in the iPP matrix, consistent with others results. Apparently, positive deviation should arise from much better dispersion of HDPE inclusion in the iPP matrix (Fig. 5), since, the crystallinity of iPP matrix holds constant for various HDPE contents. Moreover, good tensile properties may be originated from the intercrystalline links between HDPE and iPP, since, in some extent the crystallization of HDPE or iPP can influence each other [8,10]. Combination with the DSC results, it is reasonable to speculate that the retarded crystallization of HDPE inclusion should be influenced by iPP matrix, due to much finer HDPE droplets dispersed in the iPP matrix, and there may have some interactions between HDPE and iPP component. Therefore, it can be concluded that, with no or less orientation, the tensile properties are dictated by the phase morphology and interfacial adhesion related to the influenced crystallization between HDPE and iPP component.

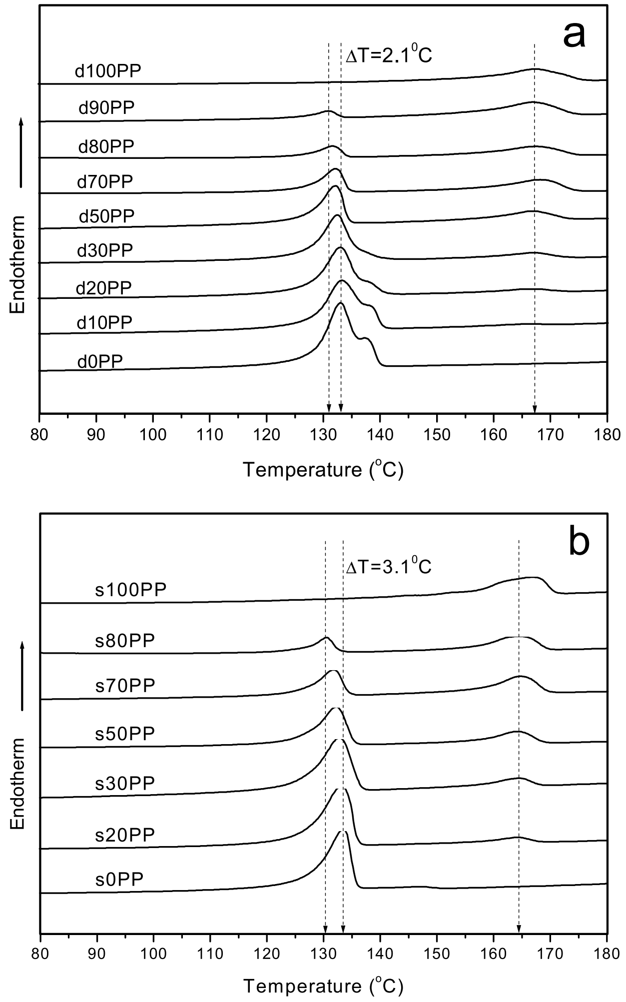

Fig. 6. DSC heating traces of HDPE/iPP blends with various compositions at a rate of 10 °C/min, (a) dynamic samples and (b) static samples. Curves have been shifted vertically for clarification.

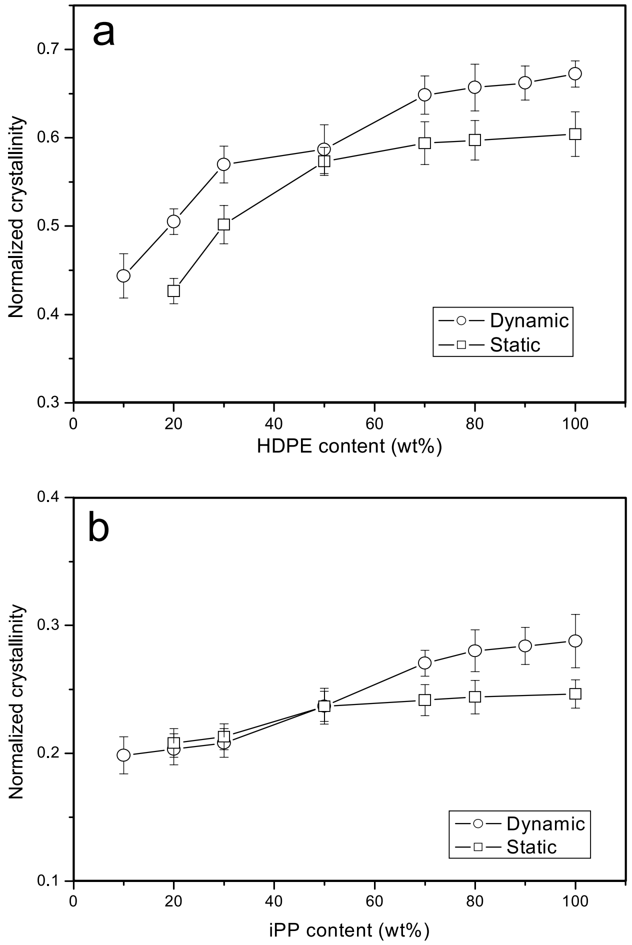

Fig. 7. Normalized crystallinity of individual component calculated from the DSC heating traces in Fig. 6, (a) HDPE and (b) iPP.

As for dynamic samples, however, the situation becomes more complicated due to orientation of both dispersed phase and matrix. From Fig. 2, it is evident that change of tensile strength and modulus is totally different, with abrupt decline of tensile strength and modulus in the blends with HDPE matrix and slight changes in the ones with iPP matrix. At first glance, it is expected that the tensile properties is dependent of degree of orientation. This is true in our case, while comparing the orientation parameter with tensile properties. The orientation parameter of major component, i.e. matrix, decrease with increasing the content of dispersed phase, in either HDPE or iPP matrix. Fig. 10 shows this case, for samples with HDPE matrix. Rapid decreasing of orientation parameter results in the deterioration of modulus and tensile strength in the HDPE matrix. In the iPP matrix, however, slight reducing of orientation parameter gives rise to slight decreasing of modulus and tensile strength. Therefore, it can be concluded that the molecular orientation of matrix is the determining factor for the tensile properties of oriented samples. It can be easily understood since, load is firstly borne by the matrix and then could be transferred to the dispersed phase, dependent of interfacial properties. In the second, other factors, such as interfacial properties and phase morphology, can also be responsible for mechanical properties of polymer blends. To our knowledge, it is immiscible in the whole composition range for HDPE/iPP blends. Therefore, it is expected that contribution to tensile properties mostly result from the matrix, much less from dispersed phase. In general, mixing rule can be used to predict the interfacial properties, as shown above for static samples. However, for dynamic samples with molecular orientation varying with compositions, as shown in Fig. 9, it is difficult to obtain true tensile properties for each component, and thus, detail analysis seems to be impossible, especially for samples with HDPE matrix. Due to its slight changes of molecular orientation and crystallinity for iPP component in its matrix, the true modulus and tensile strength in the blends can take the values of pure iPP. While the molecular orientation of iPP component in its matrix remains constant, the tensile properties will decrease with reducing of its content in blends. They can be written as $E = 1.54 - 1.54V_{\mathrm{HDPE}}$ and $\sigma = 54 - 54V_{\mathrm{HDPE}}$, for modulus and tensile strength of blends, respectively. Note that, with

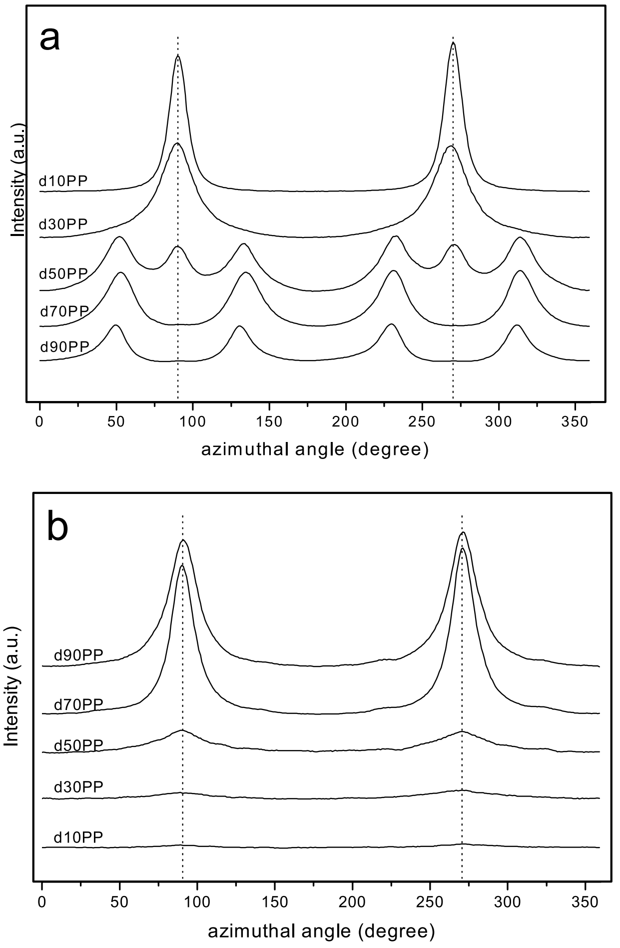

Fig. 8. Selective azimuthal scans of dynamic samples of (110) plane of HDPE (a) and (040) plane of iPP (b). Curves have been shifted vertically for clarification. The dotted lines in pictures are the position of equator, perpendicular to the shear direction.

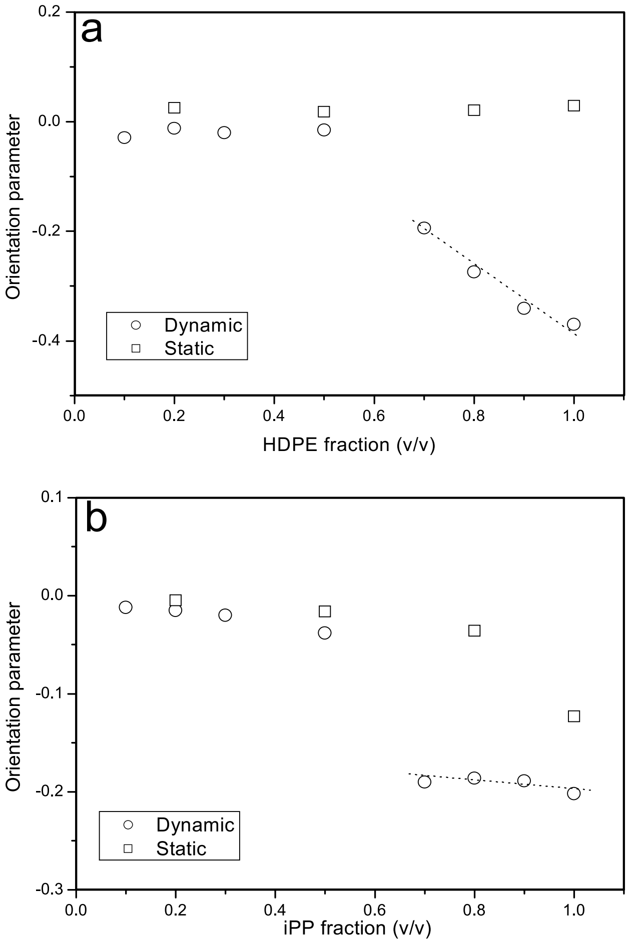

Fig. 9. Orientation parameter of (110) plane of HDPE (a) and (040) plane of iPP (b) for both dynamic and static samples, with respect to compositions.

larger values of pure iPP in above equations, the predicted modulus and tensile strength will be higher. Compared with experimental ones, $E = 1.54 - 0.67V_{\mathrm{HDPE}}$ and $\sigma = 54 - 22V_{\mathrm{HDPE}}$ for modulus and tensile strength, respectively, shown in Fig. 2, it can be concluded that, in the blends with iPP as a major component, the HDPE inclusion can partly contribute to the mechanical properties, with at least modulus of 0.87 GPa and tensile strength of 32 MPa, respectively. This contribution is speculated that, finer HDPE dispersed phase and epitaxial growth of HDPE component in the iPP matrix, demonstrated by SEM and 2D WAXS, could has some effects on the modulus and tensile strength.

As PP droplets are dispersed in the HDPE matrix, however, it is well known that the tensile strength and modulus is a function of matrix molecular orientation. Fig. 9(a) shows that the orientation of HDPE matrix decreases considerably with increasing of PP content. The tensile strength and modulus of matrix will reduce with increasing of PP content. Therefore, it is difficult at this moment to calculate the tensile strength and modulus of the blends. In this case, the equation: E=EHDPE−(EHDPE −Eipp)-Vipp holds also true. For a qualitative explanation, the value of (EHDPE −Eipp) should be always less than that of EHDPE in any case because Eipp can not be negative. However, from Fig. 2 it is found that the theoretical results are well agreement with the experimental data if $E = 2.4 -$ 3.2Vipp. Certainly, ‘3.2’ here is not reasonable and it should be smaller than 2.4. Thus, the theoretical result must be higher than that of experiment. This modulus difference between experiment and theory just results from the orientation difference for different Vipp. In return, this result proves again that the tensile modulus depends on matrix molecular orientation.

5. Conclusion

Tensile and structure characterization have been carried out for HDPE/iPP blends obtained by dynamic packing injection molding. Phase separated morphology is observed for both dynamic and static samples, with major component forming matrix and minor component dispersed phase, though elongated and larger domains can be found in the

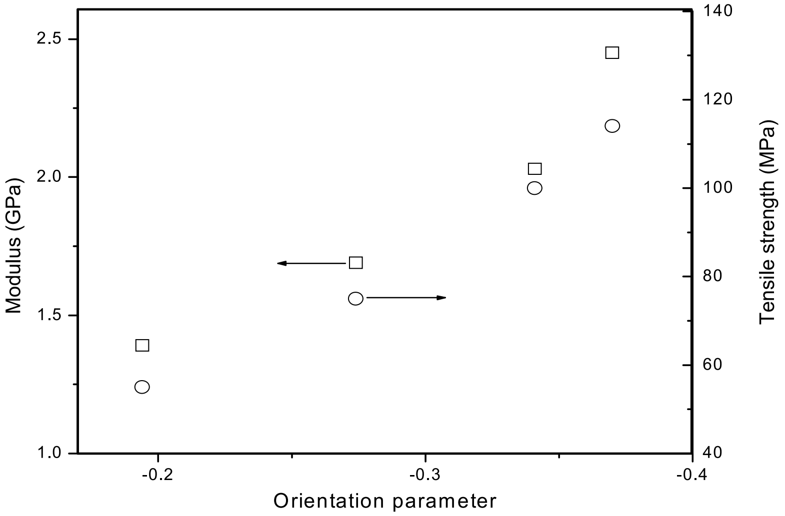

Fig. 10. Plots of modulus and tensile strength of dynamic samples with HDPE matrix as a function of orientation parameter of (110) plane of HDPE component.

former due to introduction of shear during packing stage. Molecular orientation and crystallization is related to phase morphology, i.e. droplet or matrix. Higher orientation and constant crystallinity is always found in its matrix for either HDPE or iPP component. Different from the static ones with almost no molecular orientation, whose tensile properties can be illustrated by the phase morphology and interfacial adhesion, the change of tensile properties of dynamic samples is matrix dependent, as a result of molecular orientation of corresponding matrix. Molecular orientation of matrix is the determining factor for the modulus and tensile strength of dynamic samples. Other factors, such as interfacial properties and phase morphology, seem to be little effect on the mechanical properties.

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