Effect of the High-Density Polyethylene Melt Index on the Microcellular Foaming of High-Density Polyethylene/Polypropylene Blends

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ABSTRACT: The effect of high-density polyethylene (HDPE)/polypropylene (PP) blending on the crystallinity as a function of the HDPE melt index was studied. The melting temperature and total amount of crystallinity in the HDPE/PP blends were lower than those of the pure polymers, regardless of the blend composition and melt index. The effects of the melt index, blending, and foaming conditions (foaming temperature and foaming time) on the void fractions of HDPEs of various melt indices and HDPE/PP blends were also investigated. The void fraction was strongly dependent on the foaming time, foaming temperature, and blend composition as well as the melt index of HDPE. The void fraction of the foamed 30:70 HDPE/PP blend was always higher than that of the foamed 50:50 HDPE/PP blend, regardless of the melt index. The microcellular structure could be greatly improved with a suitable ratio of HDPE to PP and with foaming above the melting temperature for long enough; however, using high-melt-index HDPE in the HDPE/PP blends had a deleterious effect on both the void fraction and cell morphology of the blends. © 2004 Wiley Periodicals, Inc. J Appl Polym Sci 93: 364–371, 2004

Key words: polyethylene (PE); poly(propylene) (PP); blends; foams; melt

INTRODUCTION

Currently, the recycling of plastic materials is an important issue because of limitations in landfill space. Most attention has been concentrated on postconsumer waste, especially plastic packaging materials. Polyolefins are among the most commonly used plastics of the packaging industry because of their good mechanical and processing properties and low cost. However, in municipal solid waste, the separation of various polyolefins, such as high-density polyethylene (HDPE) and polypropylene (PP), is difficult and rarely cost-effective. Furthermore, blends of HDPE and PP are immiscible and incompatible[1,2] and lead to the deterioration of some mechanical properties, such as the impact strength.[1,3,4]

In the last 3 decades, the blending of HDPE and PP has been extensively studied. Substantial research on HDPE/PP blends has concentrated on the rheological properties,[5–12] water vapor transmission,[5] crystallization,[3,13–24] structure and morphology,[3,6,15,16,18,20–27] mechanical properties,[1,3,4,12,15–21,27–33] viscoelastic behavior and interfacial tension,[27] surface modifica-

tions,[17,31,32] and thermodegradative properties.[33] Lovinger and Williams[20] studied the relationship between the morphology and tensile properties of HDPE/PP blends. They found that an increase in the stress at yield and ultimate stress was related to a size reduction of the spherulites, an increase in crystallinity, and the foaming of a permeating network. They also reported that the ultimate elongation of all the HDPE/PP blends was lower than that of the neat polymers because of the incompatibility of HDPE and PP. The tensile strength at yield increased gradually with increasing PP content.[20] Research on the melt rheology of polyolefins was reviewed by Gahleitner.[34] He reported that the melt-flow rate was related to the weight-average molecular weight (M_w) and molecular weight distribution [weight-average molecular weight/number-average molecular weight (M_w/M_n)], which also influence the Charpy impact strength.[34] When M_w and M_w/M_n increased, the melt-flow rate decreased and the Charpy impact strength increased. Kukaleva et al.[35] studied high-crystallinity isotactic PP and conventional PP blended with metallocene-catalyzed linear low-density polyethylene (LLDPE) blends. They found that the melt-flow rate decreased with increasing LLDPE content, but the melt density was independent of the blend composition and similar to the melt density of PP, regardless of the PP type. Moderated differential scanning calorimetry showed that the blends were miscible dur-

TABLE I
Typical Properties of HDPE and PP as Supplied by the Manufacturer

Physical property	HDPE1 (Dow HDPE 00452N)	HDPE2 (Dowlex IP 10262)	HDPE3 (Dowlex IP 40)	PP Insprie H704-04)
Density (g/cm^3)	0.9520	0.960	0.9520	0.90
Melt index (g/10 min)	4.0	9.0	40	4.0
DSC melting point (°C)	133	133	128	N/A

ing processing and that the phases separated during cooling, becoming immiscible in the solid state.[35] Furthermore, they reported that the level of crystallinity of PP in the blends was independent of the PP/LLDPE blend composition, and Young's modulus and the impact strength did not correlate with the level of crystallinity.[35] Liang[36] observed that crystallinity of HDPE increased with increasing melt density during processing. Nevertheless, the effect of the melt-flow index on the crystallization of HDPE/PP blends, in particular, has been little studied. A different melt-flow index might have an effect on the crystallinity of blends, which is related to the viscosity and stiffness of the polymer blend matrix. These properties might affect the foamability of HDPE/PP blends during the foaming process.

Our recent studies have shown that the deleterious effect of blending on the impact strength can be overcome by the creation of a microcellular structure in HDPE/PP blends. However, the successful production of a cellular structure in the blends strongly depended on the foaming conditions and the viscoelastic behavior of the blends, which controlled the cell growth and density reduction. For improved impact strength, the cell morphology had to consist of a well-developed, uniform microcellular structure, which was achieved via foaming at a relatively high temperature (175°C) for a longer time (30 s) with appropriate blend ratios (50:50 and 30:70 w/w HDPE/PP).[3] In contrast, the blend with the highest HDPE content, that is, 70:30 HDPE/PP, had poor morphology because the matrix was too soft, causing cell coalescence.[3] These results implied that the viscosity of the blends was one of the critical variables for proper foamability.

Postconsumer polymers contain a mixture of resins with differing properties (e.g., M_w, viscosity, and melt index) that can affect the processing and characteristics of microcellular foams. Therefore, it is imperative to examine the influence of the melt index on the foamability of HDPE/PP blends, and this was the goal of this study.

In this study, the influence of the HDPE melt-flow index on the crystallinity of the neat polymers and HDPE/PP blends as well as the melting temperature was investigated first. Second, the effects of the HDPE melt index and foaming conditions on the void fraction and cell morphology of HDPE/PP blends were examined.

EXPERIMENTAL

Sample preparation

The materials used in this study were PP (Inspire H704-04) and three HDPE grades differing in their melt indices: injection-molding-grade HDPE (Dow HDPE 00452N) and Dowlex IP 10262 and Dowlex IP 40 polyethylene resins from Dow Plastics (Midland, MI). These HDPEs are denoted HDPE1, HDPE2, and HDPE3, respectively, and their reported properties are summarized in Table I. Commercial-grade carbon dioxide was used as a blowing agent.

Samples of neat HDPE1, HDPE2, HDPE3, and PP and HDPE/PP blends (30:70 and 50:50 w/w) were manufactured with a Baker Perkins model ZSK-30 corotating twin-screw extruder (Werner & Pfleiderer Corp., Ramsey, NJ). The compounding conditions are shown in Table II.

It was not possible to set the same compounding conditions for all the polymers and their blends. For example, when the temperature was too high and the screw speed was too slow, HDPEs with a high melt-

TABLE II
Compounding Conditions

Samples	Temperature (°C)						Screw Speed (rpm)
	Port 1 (hopper)	Port 2	Port 3	Port 4	Port 5	Port 6 (die)	
PP and all HDPE/PP blends	180	180	155	155	155	155	100
Neat HDPE1	155	155	155	155	155	155	100
Neat HDPE2	155	155	135	135	135	135	100
Neat HDPE3	155	155	130	130	125	125	150

flow index lost their melt strength, and the extrudate could not be cut to the desired length. Therefore, the temperature profiles and screw speeds in the extruder were set differently for each polymer to obtain a continuous and consistent stream of flowing polymer. The extrudates were cut into six-in. lengths before they solidified at room temperature, and then they were compression-molded (Carver model M laboratory press, Wabash, IN) at 30,000 psi for 5 min.[16] The compression-molding temperatures were 160 and 185°C for the pure HDPE samples and the samples containing PP, respectively. Next, the system was cooled to room temperature with cooling water. The 2-mm-thick panels were cut to 0.5 in. × 1 in. (1.27 cm × 2.54 cm) test specimens.

Differential scanning calorimetry (DSC)

DSC was performed with a DSC 2010 (TA Instruments, New Castle, DE) to investigate the crystallinities of the HDPE1, HDPE2, HDPE3, PP, and HDPE/PP blends with 3–5-mg samples. The calibration for heat capacity was performed with an indium reference standard. Three to five replicates were heated from room temperature to 200°C at a heating rate of 10°C/min. Nitrogen was used as a purge gas at a flow rate of 50 mL/min. The crystallinities of HDPE and PP were calculated as follows.

For the pure polymers,

$$\chi_{\text{HDPE}}(\%) = \frac{\Delta H_{m,\text{HDPE}}}{\Delta H^o_{m,\text{HDPE}}} \times 100\% \tag{1}$$

$$\chi_{\text{PP}}(\%) = \frac{\Delta H_{m,\text{PP}}}{\Delta H^o_{m,\text{PP}}} \times 100\% \tag{2}$$

For each component in the blend,

$$\chi_{\text{HDPE}}(\%) = \frac{\Delta H_{m,\text{HDPE}}}{\Delta H^o_{m,\text{HDPE}}(1-x)} \times 100\% \tag{3}$$

$$\chi_{\text{PP}}(\%) = \frac{\Delta H_{m,\text{PP}}}{\Delta H^o_{m,\text{PP}}(x)} \times 100\% \tag{4}$$

For the total sample,

$$\chi_{\text{total}} = (1-x)(\chi_{\text{HDPE}}) + (x)(\chi_{\text{PP}}) \tag{5}$$

χ_{HDPE} and χ_{PP} are the crystallinity percentages of HDPE and PP, respectively, and χ_{total} is the crystallinity percentage of the total sample. The heats of fusion for HDPE ($\Delta H^o_{m,HDPE}$) and PP ($\Delta H^o_{m,PP}$) were 293 and 209 J/g, respectively.[37] The heats required for melting the HDPE phase ($\Delta H_{m,\text{HDPE}}$) and the PP phase ($\Delta H_{m,\text{PP}}$) were measured with DSC (J/g). x is the weight fraction of PP in the blend.

Microcellular foaming experiments and characterization of the foams

In batch microcellular foaming experiments, the samples were saturated with CO_2 [at room temperature (23–25°C) and 800 psi for 24 h]. The CO_2-saturated samples were microcellular-foamed by immersion in a hot glycerin bath[16,38−40] at different foaming temperatures (160 or 175°C) for foaming times of 20 or 30 s. The foamed samples were immediately quenched in cold water to prevent cell deterioration. The void fractions of the foamed samples were determined with the approach described in refs. 16 and 39−41. The sample morphology was investigated with environmental scanning electron microscopy (ESEM), as described previously.[16]

RESULTS AND DISCUSSION

Effects of blending on the crystallinity as a function of the melt index

Our previous study showed the effect of the HDPE/PP blend composition on the crystallinity: blending reduced the crystallinity of both HDPE and PP.[16] In this study, HDPEs with different melt indices were blended with PP to study the effects of blending on the heat of fusion, the crystallinity of the neat polymers and each component in the blends, and the total crystallinity in the samples, as well as the melting temperature, as a function of the melt index. DSC thermograms of pure HDPE1, pure PP, and the blends are presented in Figure 1. For clarity, the curves have been displaced from the baseline. The thermograms of pure HDPE2, pure HDPE3, and their blends were similar to those of HDPE1 and are not shown.

As shown in Figure 1, pure HDPE1 and pure PP showed single peaks. Two well-separated melting peaks were found for the blends (the first for HDPE1 and the second for PP), reflecting two crystalline phases in all the blends. The results agree well with our previous studies[3] investigating the crystallinity of HDPE/PP blends by optical microscopy, with which we also found phase separation.[3,16] The results also agree well with those published by Teh[14] and Finlay et al.[29] The blend composition strongly affected the heat of fusion (area under the peak). In all cases, the heat of fusion of HDPE and PP decreased in the blends. The HDPE peak decreased with increasing PP, and the peak of PP decreased with increasing HDPE content. The heat of fusion from the peaks was used to calculate the crystallinity of pure HDPE1 [eq. (1)], PP [eq. (2)], and each component in the blends [eqs. (3) and (4)], as well as the total crystallinity [eq. (5)]. The effects of blending on the crystalline fraction of HDPE and PP and the total amount of crystallinity in the HDPE/PP blends as a function of the HDPE melt-flow index are illustrated in Figures 2 and 3, and the crys-
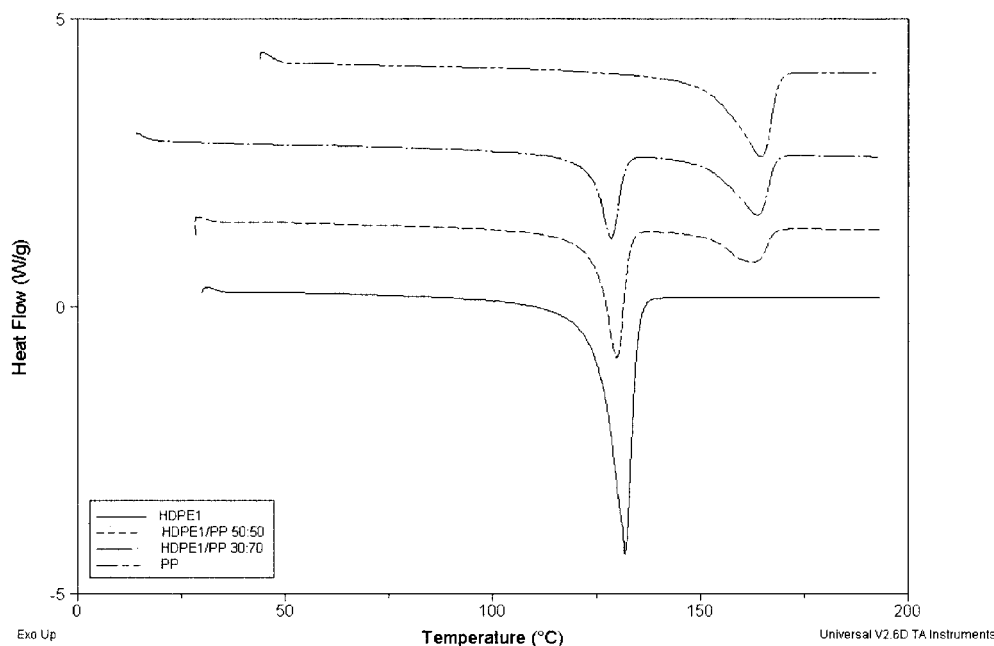
Figure 1 DSC thermograms of neat HDPE1, neat PP, and HDPE1/PP blends.

tallinity reduction percentage for each component and the melting temperature are presented in Table III.

Figure 2 shows that the crystalline fractions of both HDPE and PP in HDPE/PP blends tended to decrease as another component was added to the blends. As shown in Figure 2, pure HDPE with a lower melt index had higher crystallinity. The crystallinity of HDPE in blends decreased around 6–15% with added PP content (Table III). The crystallinity of PP in the blends also decreased with added HDPE content (Fig. 2). The crystallinity reduction percentage of PP was

around 10–20, 40, and 30% in PP blended with HDPE1 (melt index = 4 g/10 min), HDPE2 (melt index = 9 g/10 min), and HDPE3 (melt index = 40 g/10 min), respectively (Table III). However, there was no consistent pattern for the crystallinity reduction percentage as a function of the melt index; the effect of the melt index on the crystallinity reduction percentage appears to be complex.

There was a similar trend with the melting temperatures of HDPE1, HDPE2, HDPE3, and PP in the blends: the melting temperatures of the blends were
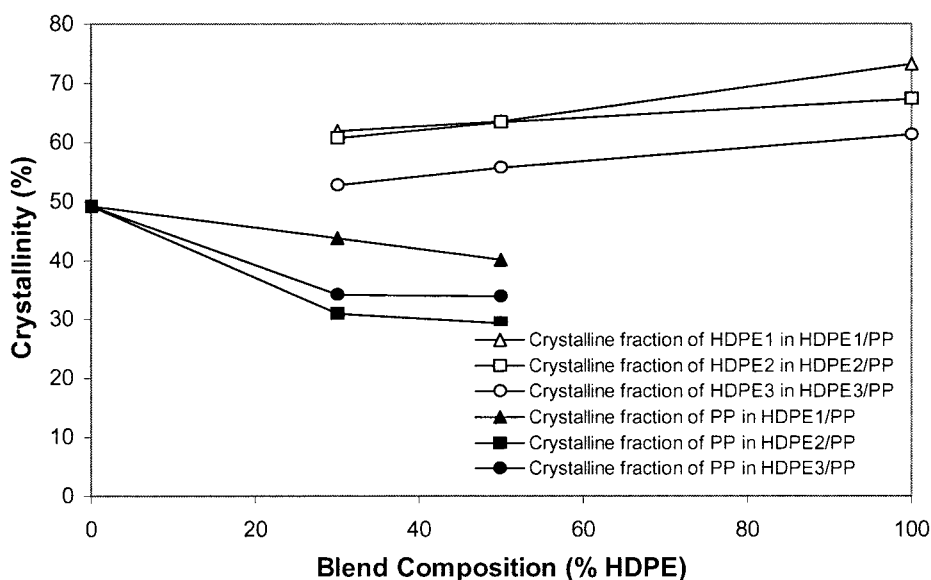

Figure 2 Effect of blending on the crystalline fractions of HDPE and PP in HDPE/PP blends as a function of the melt index.
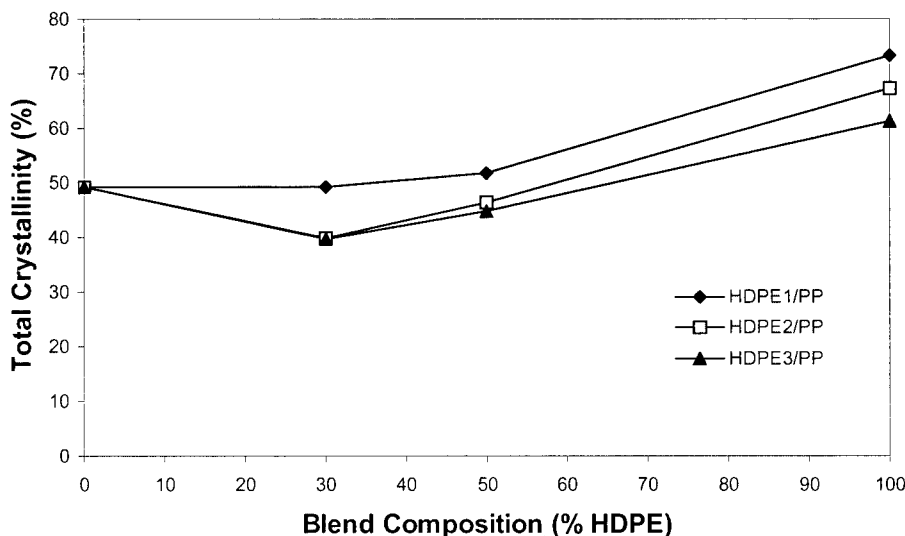
Figure 3 Effect of blending on the total amount of crystallinity in HDPE/PP blends as a function of the melt index.

generally lower than those of pure HDPE1, HDPE2, HDPE3, and PP (Table III). The total amount of the crystallinity of the blends also decreased with polymer blending, regardless of the melt index (Fig. 3).

Effects of the melt index, polymer blending, foaming time, and foaming temperature on the void fraction and cell morphology

The effects of the foaming time and temperature on the void fractions of pure HDPE1, pure HDPE2, pure HDPE3, pure PP, and their HDPE/PP blends were investigated, and the results are shown in Figures 4 and 5. The foaming times were 20 and 30 s, and the foaming temperatures were 160 and 175°C.

As shown in Figure 4, at 160°C, the void fraction is strongly dependent on the blend composition and HDPE melt index. The void fraction of PP was not high (~5%) because it was foamed below the melting

temperature of PP.[3,16] The effect of the melt-flow index is clear in neat HDPE: the higher the melt flow, the higher the void fraction. The void fraction increased with the foaming time, but the effect of differences in the melt-flow index on the void fraction decreased. The increased void fraction in foamed pure HDPE polymers resulted in large cells near the surface, as found in our previous study.[3,16]

Polymer blends of 30:70 HDPE/PP always resulted in higher void fractions than those of 50:50 HDPE/PP, as found in our previous work.[16] The reason for this is still not well understood, but it is likely related to the blend morphology.[3] As 160°C was below the melting temperature of PP, the void fractions of all the polymer blends were only around 10% at 160°C for 20 s. The void fractions of HDPE1 and HDPE2 blends increased as the foaming time increased from 20 to 30 s, but the HDPE3 blends did not improve with respect to

TABLE III
Effects of the Blend Composition on the Melting Temperature (T_m) and Crystallinity Reduction of Blend Samples as a Function of the Melt Index

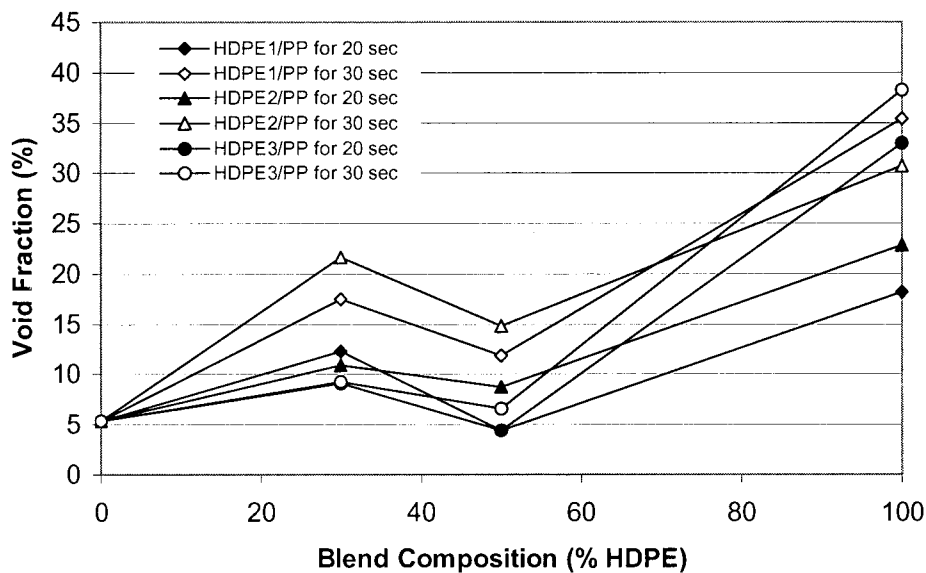|			Crystallinity reduction (%)	
Sample	$T_{m,\ \text{HDPE}}$ (°C)	$T_{m,\ \text{PP}}$ (°C)	HDPE	PP
HDPE1 (melt flow = 4 g/10 min)	132.1	—	—	—
50:50 HDPE1/PP	130.0	162.4	13.4	18.4
30:70 HDPE1/PP	128.6	163.8	15.5	11.0
HDPE2 (melt flow = 9 g/10 min)	134.1	—	—	—
50:50 HDPE2/PP	132.8	162.5	6.0	40.5
30:70 HDPE2/PP	132.2	162.0	9.9	37.2
HDPE3 (melt flow = 40 g/10 min)	129.3	—	—	—
50:50 HDPE3/PP	128.1	161.4	9.1	31.0
30:70 HDPE3/PP	128.5	164.5	14.0	30.5
PP	—	164.5	—	—

Figure 4 Effect of the melt-flow index, blending, and foaming time on the void fractions of samples foamed at 160°C.

the void fraction. The foaming temperature may be too high for HDPE3 blends, causing the matrix to be too soft and resulting in cell collapse. Therefore, for the foaming of blends with high-melt-flow-index HDPE, the foaming temperature should not be too high.

At the foaming temperature of 175°C (Fig. 5), the effect of the HDPE melt-flow index on the blends was more obvious. This foaming temperature was above the melting temperatures of HDPE and PP. It is known that the ability to use high foaming temperatures and long foaming times to achieve a high void fraction is limited by a rapid decrease in the strength of the polymer at temperatures above the melting point. This

results in substantial deformation of the polymer matrix, even though the softened polymer matrix is favorable to bubble growth.[16,42] The void fraction of pure HDPE did not increase significantly with the foaming time and a higher temperature (175°C), and it had large cells near the surface as discussed previously. The void fraction of foamed pure PP did increase with the foaming time, but it had a nonuniform structure.[3] Large cells developed close to the surfaces of the samples, whereas the centers of the samples were not foamed, and a microcellular structure developed in the subsurface.[3,16] The void fraction of the 30:70 HDPE1/PP blend increased with the foaming time, but the blends with higher melt index HDPE
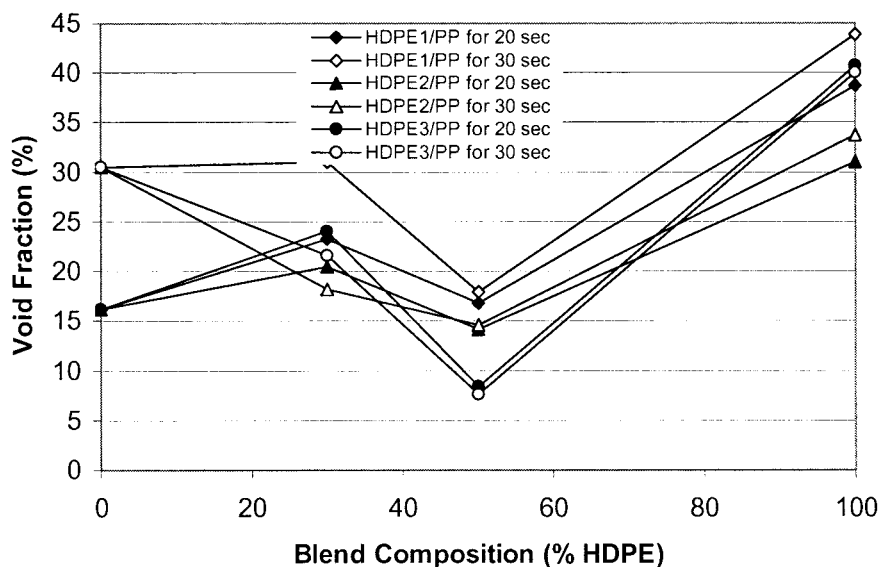

Figure 5 Effect of the melt-flow index, blending, and foaming time on the void fractions of samples foamed at 175°C.

resulted in a nearly unchanged or even lower void fraction when the foaming time increased from 20 to 30 s. For all the 50:50 HDPE/PP blends, increasing the foaming time resulted in nearly unchanged void fractions.

It is known that blending increases the foamability of HDPE/PP blends when the temperature and time are appropriate.[3,16] However, when HDPE with a higher melt-flow index was used in the blends, the crystallinity of PP in the blends decreased dramatically (Fig. 2 and Table III), and this made the matrix too soft to maintain the cellular structure. Therefore, to foam HDPE/PP blends containing high-melt-flow-index HDPE, it may be necessary to lower the foaming temperature or foaming time and use a suitable blend composition to achieve a high void fraction.

Electron microscopy of blends foamed at 175°C for 30 s revealed that the melt index of HDPE also had a significant impact on the cell morphology. For the HDPE1 resin, as shown in Figure 6, the microcellular structures in both the 30:70 and 50:50 HDPE1/PP blends were uniformly distributed, and the cells were fully grown. The void fraction of the 30:70 blend was higher than that of the 50:50 blend, whereas the cell size was smaller (see Fig. 6). The larger cell size of the 50:50 blend may be an indication of cell coalescence, perhaps due to the decreased viscosity at the higher HDPE content.

The 30:70 blends with the higher melt index HDPEs showed abundant cells, with the smallest cells in the intermediate HDPE2/PP blend, larger cells in the highest melt flow HDPE3/PP blend, and the largest cells in the low-melt-flow HDPE1/PP [Fig. 6(a,c,e)]. This corresponded to the void fractions (Fig. 5), but not to the crystallinity, and is not yet well understood.

For the 50:50 blends, little if any microcellular structure was evident. The intermediate-melt-flow HDPE2 showed evidence of a few large cells that appeared [Fig. 6(d)], from their irregular margins, to have collapsed. The high-melt-flow HDPE3 blend showed only a few isolated bubbles [Fig. 6(f)]. A possible explanation is that the coupling of a lower viscosity in the HDPE regions with the greatly increased crystallinity loss in the PP regions (40 and 31% for HDPE2 and HDPE3 blends, respectively, compared to 18% for HDPE1; Table III) resulted in a material that was simply too soft to maintain the microcellular structure, and this resulted in massive cell coalescence and collapse. Physical deformation of the foamed samples was also observed. Therefore, it should be concluded that the melt-flow index should be in a range that will not prevent nucleated cells from growing but that will still maintain their structure.
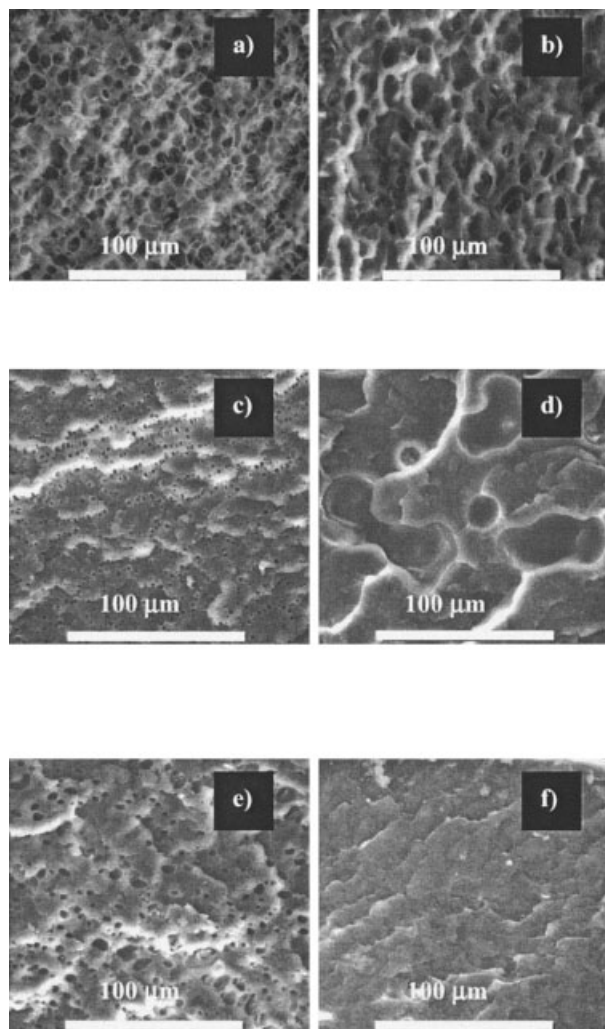

Figure 6   ESEM micrographs of polymer blends foamed for 30 s at 175°C (original magnification = 500×): (a) 30:70 HDPE1/PP, (b) 50:50 HDPE1/PP, (c) 30:70 HDPE2/PP, (d) 50:50 HDPE2/PP, (e) 30:70 HDPE3/PP, and (f) 50:50 HDPE3/PP.

CONCLUSIONS

The effects of melt index of HDPE on the microcellular foaming of HDPE/PP blends, the crystallinity reduction of HDPE and PP, the melting temperature, and the total amount of crystallinity as a function of the melt index were studied. Neat HDPE with a lower melt index had higher crystallinity. The crystallinity of HDPE and PP decreased in HDPE/PP blends, regardless of the blend composition and melt index. The total amount of crystallinity also decreased. The crystallinity reduction in HDPE was affected more by the melt index than the crystallinity reduction in the PP fraction. The melting temperature also tended to decrease in blends, regardless of the melt index and blending.

The effects of the blending, melt index, and processing conditions on the void fraction and cell morphology were also investigated. The foamability was

strongly dependent on the blend composition. The 30:70 HDPE/PP blend always provided a higher void fraction than the 50:50 HDPE/PP blend, regardless of the foaming conditions and melt index. At a foaming temperature of 160°C, the void fraction increased with the foaming time, regardless of the blend composition and melt index. Foamability was facilitated with a suitable HDPE/PP blend ratio at a high foaming temperature (175°C) and long enough foaming time (30 s); however, the use of HDPE with too high a melt index in the blend had a negative effect on the void fraction and cell morphology because the polymer matrix lost strength during the foaming process. The void fraction and cell morphology of the blends were dependent not only on the foaming conditions and blend composition but also on the stiffness or strength of the polymer matrix under the foaming conditions.

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