

Reactive Compatibilization of PP/PBT Blends by a Mixture of PP-*g*-MA and Epoxy Resin

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ABSTRACT: In this study, dual compatibilizers composed of the commercially available maleic anhydride-grafted polypropylene (PP-MA) and a multifunctional epoxy resin were demonstrated to effectively compatibilize the immiscible and incompatible blends of PP and poly(butylene terephthalate) (PBT). The PP-MA with a low MA content is totally miscible with PP to make the PP phase quasi-functionalized, so that the multifunctional epoxy has the chance to react with PBT and PP-MA simultaneously to form PP-MA-*co*-epoxy-*co*-PBT copolymers at the interface. These desired copolymers are able to anchor along the interface and serve as efficient compatibilizers. The compatibilized blends, depending on the quantity of dual compatibilizers employed, exhibit higher viscosity, finer phase domain, and improved mechanical properties. Epoxy does not show compatibilization effects for the PP/PBT blends without the presence of PP-MA in the blends. © 2001 John Wiley & Sons, Inc. *J Appl Polym Sci* 79: 2272–2285, 2001

Key words: polyblends; reactive compatibilizer; PP; PBT; PP-*g*-MA; epoxy

INTRODUCTION

Polyolefins and poly(alkyl terephthalate) are considered to be immiscible and incompatible. Only a very limited number of studies on these blends have been reported; however, they have received considerable interest lately.^{1–12} Polypropylene (PP) is neither miscible nor compatible with poly(butylene terephthalate) (PBT) due to a great difference in polarity. In addition, PP does not contain the necessary functional group to react with PBT. Adding a functionalized polymer miscible with PP to react with PBT may improve the compatibilization of the PP/PBT blends. In our previous report,¹³ ethylene-glycidyl methacrylate (EGMA) was demon-

strated to be a good compatibilizer for the PP/PBT blends. EGMA is a reactive copolymer where the ethylene component is compatible with PP, while the glycidyl groups are able to react with hydroxyl or carboxylic terminal groups of the PBT.

In this article, a combination of the commercial available maleic anhydride-grafted polypropylene (denoted as PP-MA hereinafter) and a multifunctional epoxy resin (i.e., tetraglycidyl ether of diphenyl diaminomethane) was used as dual reactive compatibilizers for the PP/PBT blends. The PP-MA with a low MA content is totally miscible with PP to make the PP phase quasi-functionalized, so that the multifunctional epoxy has the chance to contact and react with PBT and PP-MA simultaneously to form PP-MA-*co*-epoxy-*co*-PBT copolymers at the interface. These desired copolymers are able to anchor along the interface and are expected to serve as efficient compatibilizers.

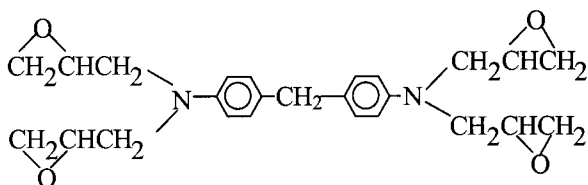
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A series of continuous investigations on *in situ* compatibilization of various blends using epoxy-containing copolymers or polymers as compatibilizers were carried out in our laboratories.^{14–21} In this article, the multifunctional epoxy serves as a coupling agent to combine PP–MA and PBT during the process of melt blending. We intend to report their specific compatibility and correlation with their resultant morphological, thermal, and mechanical properties.

EXPERIMENTAL

Polypropylene (PP-PT100) was obtained from the Taiwan Polypropylene Co. (Taiwan) with a melt-flow rate (MFR) of 1.8 g/10 min. A natural-grade PBT (D-201) was the product of the Sinkong Synthetic Fibers Corp. (Taiwan). PP grafted with MA of 0.15 wt % (Polybond-3001), 0.3 wt % (Polybond-3002), and 0.8 wt % (Polybond-3150) were obtained from the Welantech International Co., Ltd. (Taiwan). The epoxy resin, tetraglycidyl ether of diphenyl diaminomethane (TGDDM), with the trademark of NPEH-434, was received from the Nan Ya Plastics Co. (Taiwan). TGDDM has the structure shown below:



MFRs were determined according to ASTM D1238 at 240°C and 2.16 kg of loading. Melt blending was carried out using a corotating intermeshing twin-extruder with an L/D of 36 and barrel temperatures ranging from 200°C at the feed zone to 245°C at the metering zone and the die zone. Prior to melt blending, PP, PBT, and PP–MA were dried at least 8 h at 80, 120, and 70°C, respectively. After blending, the materials were dried again and injection-molded into standard $\frac{1}{8}$ -in. ASTM tensile and impact-testing specimens using an Arburg 3-oz injection-molding machine.

Capillary rheological measurements were performed at 240°C on a capillary rheometer ($L/D = 40$, orifice radius 0.02 in., orifice length 0.8 in.) from the Kayeness Co. (USA), Model Galaxy X. Unnotched Izod impact tests were carried out at ambient conditions according to ASTM D256 by

an impact tester from TMI Co. (USA), Model 43-1. Tensile and flexural tests were performed at ambient conditions using an Instron universal testing machine (USA), Model 4201, according to ASTM D638 and D790, respectively. The impact critical strain energy release rates (G_c) were determined by varying the depth of the notch (5 mil) according to the previously developed method.²²

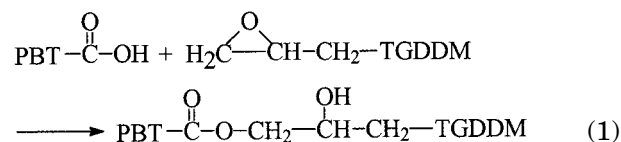
Thermal properties of the blends were studied by differential scanning calorimetry (DSC) with a sample weight of about 10 mg on a DSC model 910s from TA Instruments (USA). The samples were heated from 30 to 280°C at a rate of 10°C/min, maintained at 280°C for 5 min, and cooled to 30°C at the same rate. The temperatures and heats of crystallization of the samples were determined from the cooling traces.

Morphologies of the cryogenically fractured surfaces were etched with a mixed solvent of phenol/chloroform (3/2) to remove the PBT phase of the blends, then examined by scanning electron microscopy (SEM, Model S-570, Hitachi Co., Japan). All SEM morphologies were taken in a region midpoint between the central line and skin of the injection-molded specimens on the plane perpendicular to the injection-flow direction.

RESULTS AND DISCUSSION

Chemistry

The major chemical reactions involved in this reactive compatibilized blend system are from the epoxy groups of TGDDM with terminal carboxylic acid and/or hydroxyl groups of PBT and with anhydride groups of PP–MA. The reactions between epoxy groups and PBT terminal groups are well established²³ and can be expressed by the simplified eqs. (1) and (2). The reactions between epoxy and anhydride groups of PP–MA can be initiated by a hydroxyl-containing compound to proceed ring-opening reaction, as illustrated in eq. (3). A hydroxyl-containing compound can be obtained from the reaction between TGDDM and PBT or from the terminal hydroxyl groups of PBT. The ring-opened anhydride groups can then react with epoxy as illustrated in eq. (4):



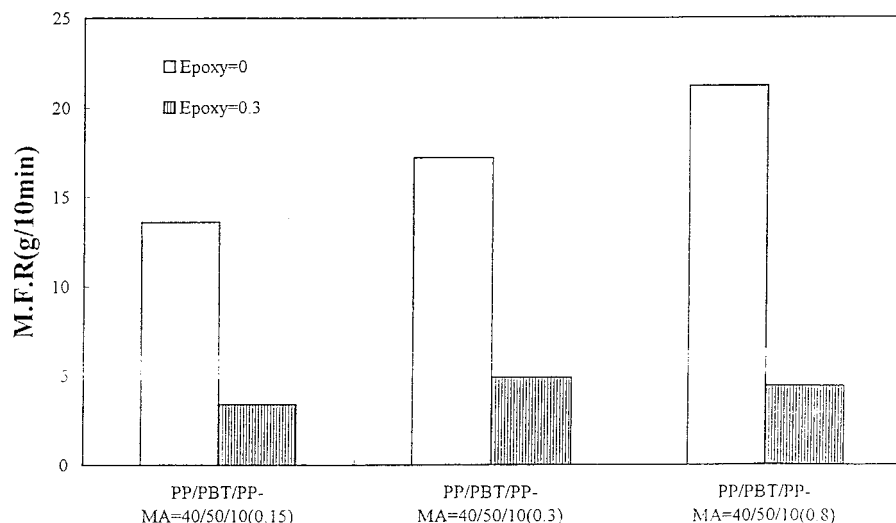
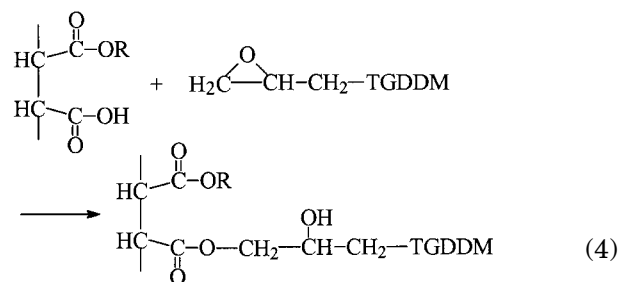
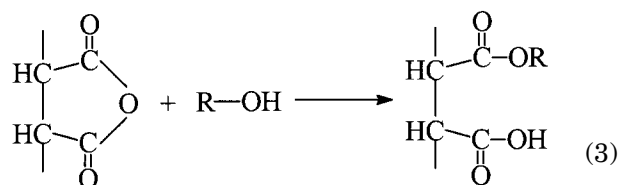
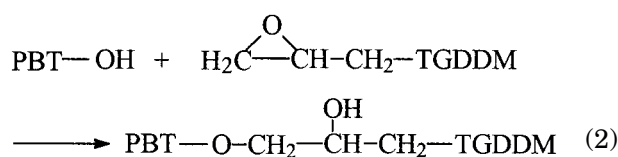


Figure 1 Effect of the epoxy on the MFR of the PP/PBT/PP-MA = 40/50/10 blend. The numbers in parentheses are the MA contents of the PP-MA.



MFRs

Figure 1 shows the effect of epoxy on the resulting MFRs of PP/PBT/PP-MA = 40/50/10 blends. The

MA contents of the PP-MA including 0.15, 0.3, and 0.8 wt % are shown in the parentheses in the figure. The MFR of the uncompatibilized PP/PBT = 50/50 blend is 17.0 g/10 min. The MFRs for all three PP/PBT/PP-MA blends prior to the incorporation of the epoxy range from 13.6 to 21.2 g/10 min as shown in Figure 1. This result demonstrates that the viscosity of the PP/PBT blend does not change significantly after adding the PP-MA, an indication that no reaction occurred. By adding 0.3 phr of the epoxy, viscosities of PP/PBT/PP-MA blends significantly increase, with MFRs varying from 3.4 to 4.9 g/10 min, an indication that reactions occurred between the epoxy and PBT and PP-MA.

Capillary Rheometry

Figure 2 gives plots of the shear viscosity versus the shear rate for PP and PBT measured at 240°C. Due to the more rigid chain structure of PBT than that of PP, the PBT shows significantly less shear thinning than that of PP. Figure 3 shows shear thinning behavior for the blends of PP/PBT = 50/50 before and after the incorporation of 0.3 phr of epoxy. The increase in viscosity due to the presence of the epoxy indicates that the epoxy in the blend is able to react with PBT and leads to a higher PBT molecular weight. The existence of reactions between PBT and the epoxy is also found in Figures 4 and 5 where the viscosities of PP/PBT/PP-MA blends were increased with increasing incorporation of the epoxy compatibilizer. The blend containing the epoxy compatibi-

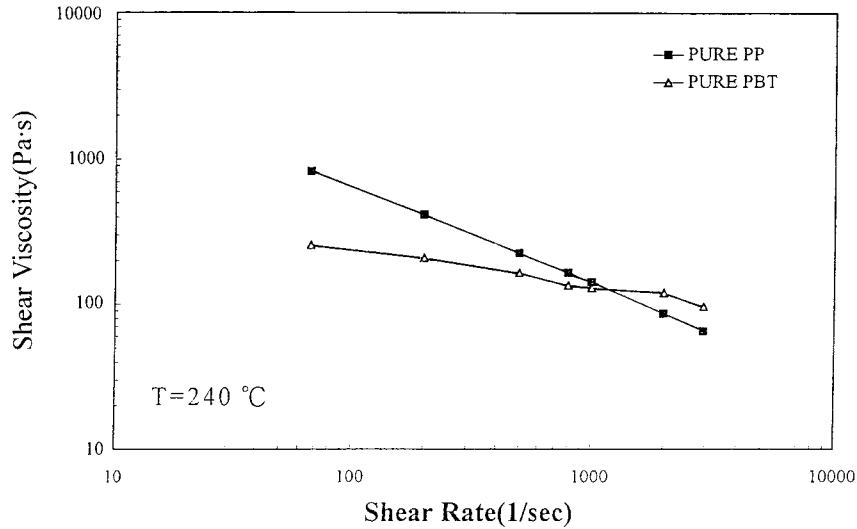


Figure 2 Shear viscosities measured at 240°C as a function of shear rate for PP and PBT.

lizer greater than 0.3 phr does not, however, show a further viscosity increase for the PP-rich blend (Fig. 5) due to shortage of PBT for reactions with an additional amount of epoxy. The general trend from this capillary rheometrical study is very similar to that from previous MFR data.

Mechanical Properties

Mechanical properties including unnotched Izod impact strengths, tensile properties, flexural strengths, and strain energy release rates (G_c) of

the blends investigated are summarized in Table I. Since both PBT and PP are brittle under standard notched Izod impact testing, all PBT/PP blends, uncompatibilized or compatibilized, are also brittle with nearly identical impact strength as would be expected (data not shown here). Unnotched impact strength is a more appropriate way to differentiate the toughness change of the notch-sensitive blends through compatibilization. The unnotched impact strengths for PP/PBT/PP-MA = 40/50/10 and 20/70/10 blends increase

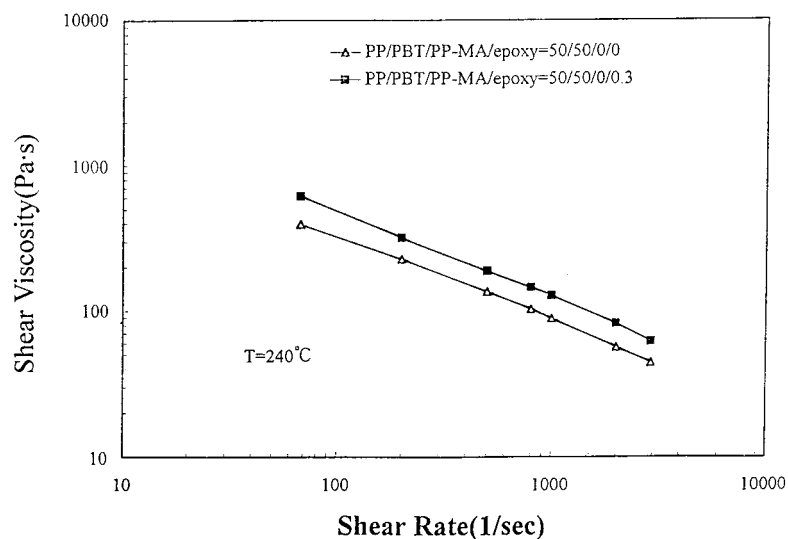


Figure 3 Shear viscosities measured at 240°C as a function of shear rate for the PP/PBT/PP-MA/epoxy = 50/50/0/0 and 50/50/0/0.3 blends.

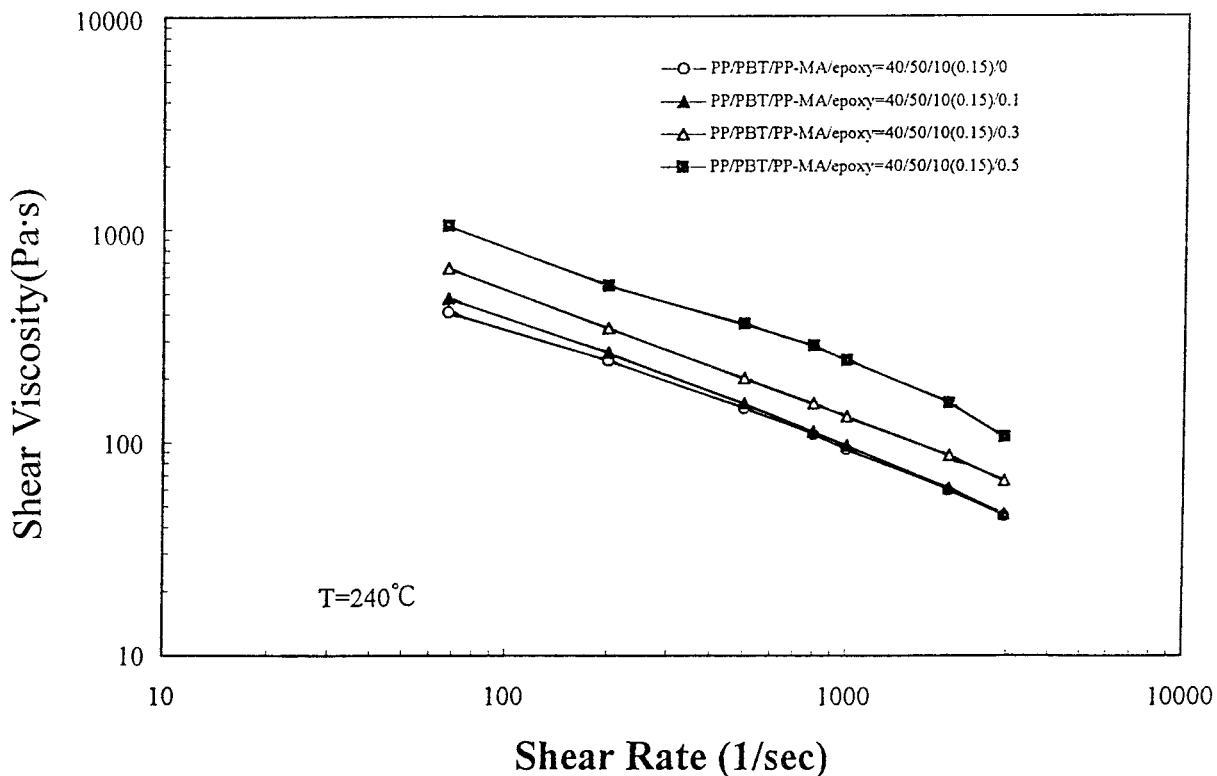


Figure 4 Shear viscosities measured at 240°C as a function of shear rate for the PP/PBT/PP-MA/epoxy = 40/50/10/0, 40/50/10/0.1, 40/50/10/0.3, and 40/50/10/0.5 blends.

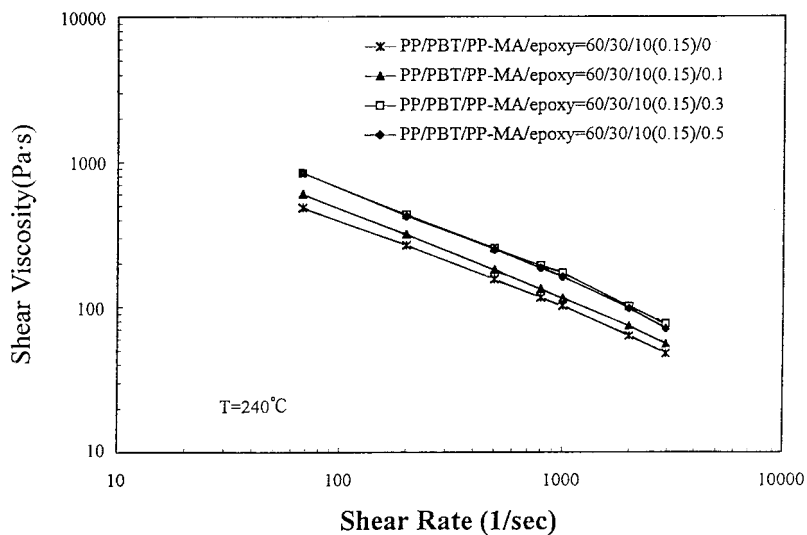


Figure 5 Shear viscosities measured at 240°C as a function of shear rate for the PP/PBT/PP-MA/epoxy = 60/30/10/0, 60/30/10/0.1, 60/30/10/0.3, and 60/30/10/0.5 blends.

Table I Mechanical Properties of the PBT/PP/PP-MA/Epoxy Blends

PP/PBT/PP-MA ^{a,b/} Epoxy	Unnotched Impact ^c (J/m)		Tensile Strength ^d (MPa)		Tensile Elongation ^e (%)		Flexural Strength ^f (MPa)		G_c^g (kJ/m ²)	
	MA1 ^a	MA2 ^b	MA1 ^a	MA2 ^b	MA1 ^a	MA2 ^b	MA1 ^a	MA2 ^b	MA1 ^a	MA2 ^b
60/30/10/0	160.4	120.5	33.9	33.7	8.3	9.3	60.0	55.7	—	8634
60/30/10/0.1	187.0	145.1	33.9	34.5	11.0	11.0	59.8	57.3	—	5650
60/30/10/0.3	154.5	185.0	28.9	32.7	10.8	9.5	51.4	56.3	—	4814
60/30/10/0.5	199.3	207.0	31.2	32.6	11.7	8.3	51.7	55.6	—	4434
40/50/10/0	97.6	185.6	30.3	33.5	5.1	7.1	48.0	50.0	5414	6119
40/50/10/0.1	230.6	285.1	34.7	38.3	8.8	13.2	59.6	56.5	7562	8989
40/50/10/0.3	258.1	367.8	36.6	37.7	11.6	14.9	61.0	55.8	9215	12,106
40/50/10/0.5	97.2	116.8	27.2	28.1	5.3	6.1	57.9	45.1	2228	1965
20/70/10/0	95.6	143.7	28.2	33.1	3.7	5.4	59.5	64.0	—	6583
20/70/10/0.1	183.1	176.4	35.8	35.9	4.8	5.9	67.3	67.7	—	7003
20/70/10/0.3	419.2	394.0	42.0	42.0	17.4	16.8	72.1	72.2	—	12,744
20/70/10/0.5	119.2	117.5	35.1	35.7	5.3	5.7	60.8	59.1	—	1296

^a The MA content of the PP-MA is 0.15 wt %.

^b The MA content of the PP-MA is 0.3 wt %.

^c The standard deviations are in the range of 4–6% of the data.

^d The standard deviations are in the range of 2–5% of the data.

^e The standard deviations are in the range of 2–5% of the data.

^f The standard deviations are in the range of 3–6% of the data.

^g The standard deviations are in the range of 3–6% of the data.

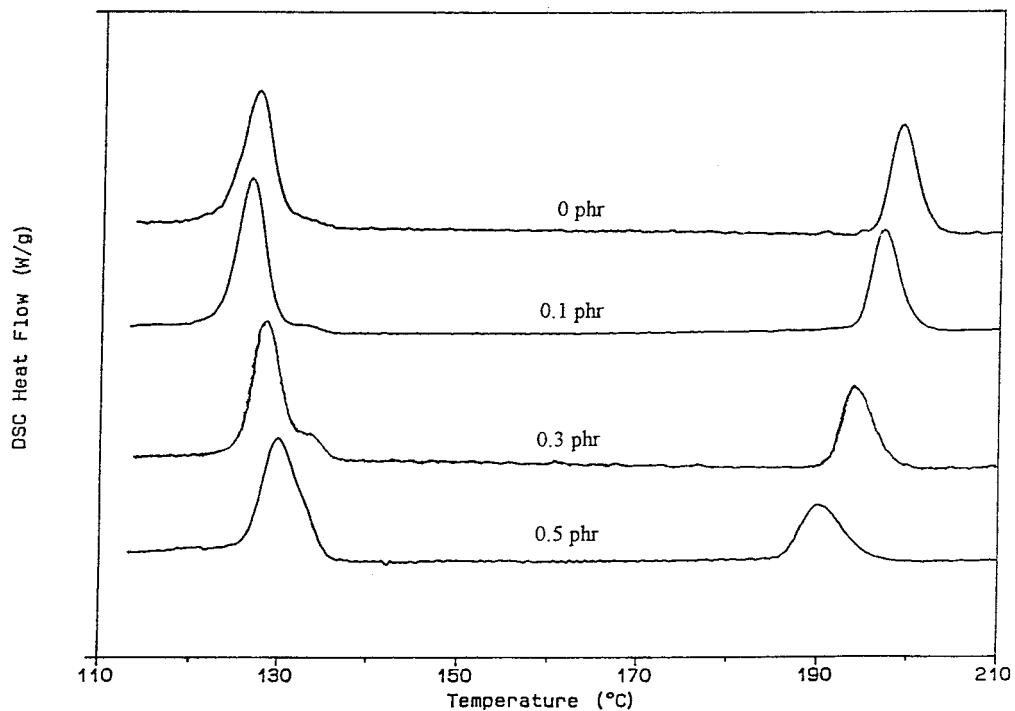


Figure 6 DSC cooling traces of the PP/PBT/PP-MA/epoxy = 40/50/10/x blends containing various amounts of epoxy.

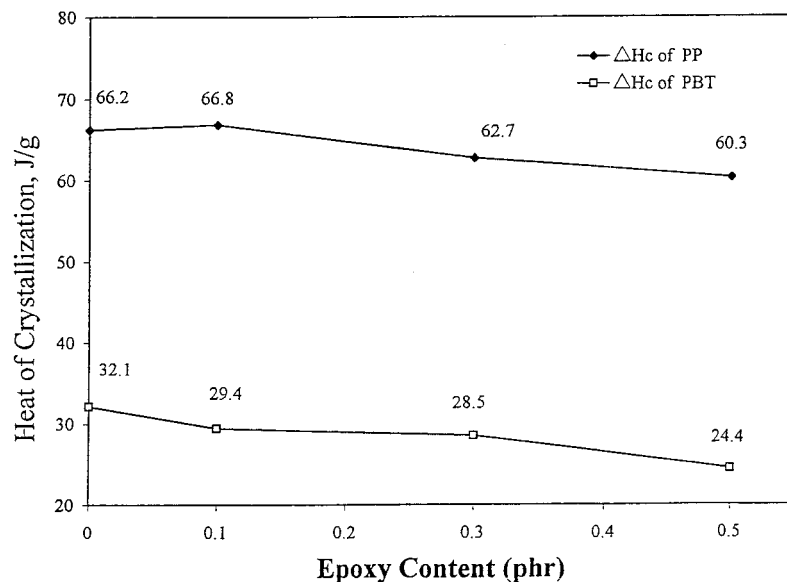


Figure 7 Exothermic heats of crystallization of the PP and PBT phases in the PP/PBT/PP-MA/epoxy = 40/50/10/*x* blends as a function of the epoxy content.

with increasing epoxy content up to 0.3 phr but decrease at 0.5 phr of epoxy as shown in Table I. This behavior was not found for the PP-rich PP/PBT/PP-MA = 60/30/10 blends. As demonstrated in Figure 4, the shear viscosity, suggesting compatibilization, increases with increasing epoxy content for the PBT-rich PP/PBT/PP-MA blends. The epoxy content of greater than 0.3 phr may, however, result in significant formation of PBT-epoxy copolymers, or light crosslinking,^{24,25} distributed in either phase and is thus unable to further improve the compatibility of the PBT-rich blend and lead to a decrease in the unnotched impact strength. This epoxy content (i.e., 0.5 phr) giving light crosslinking in the PBT phase of the PP-rich blends apparently is relatively insignificant and thus gives rise to an increase in the impact strength of the blends.

Tensile elongation represents the tensile toughness of a material. The observed trend of the tensile elongation improvement due to compatibilization by the epoxy in PP/PBT/PP-MA = 40/50/10 and 20/70/10 blends is similar to that of the unnotched impact as shown in Table I. The improvements in tensile elongation by the epoxy are not clear for the PP-rich PP/PBT/PP-MA = 60/30/10 blends.

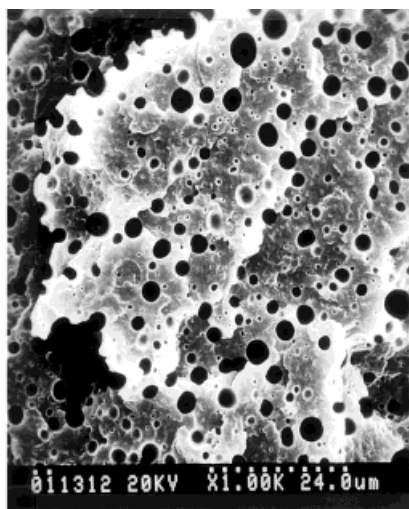
The effect of the epoxy content on the flexural strength for PP/PBT/PP-MA = 60/30/10, 40/50/10, and 20/70/10 blends are shown in Table I as well. The effect of the epoxy on the flexural

strengths is less clear for the PP-rich blends, whereas the flexural strengths are improved for the PBT-rich blends with increasing addition of epoxy up to 0.3 phr. Decreases in the flexural strengths are found for the PBT-rich blends if 0.5 phr of epoxy is added. The cause of these decreases has been explained previously.

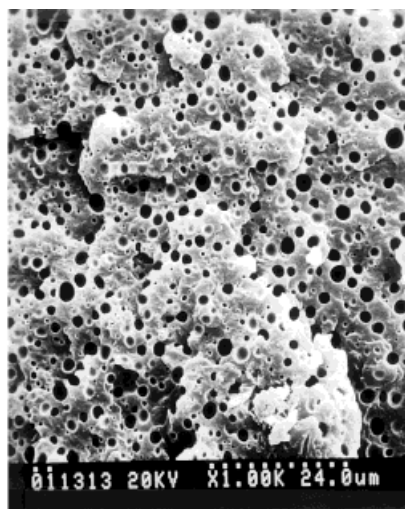
The strain energy release rates (G_c) using the Izod impact approach by varying the samples' notch depths are listed in the last column of Table I for selected blends. The optimized epoxy content is 0.3 phr to achieve the highest G_c for the PBT-rich blends. The epoxy content of 0.5 phr actually results in a reduced G_c due to a similar reason as described above. G_c is decreased, however, as the content of epoxy is increased for the PP-rich blends. This is apparently due to insignificant compatibilization for the PP-rich blends by the low molecular weight of the epoxy that has acted as a detrimental impurity in the blends.

Thermal Properties

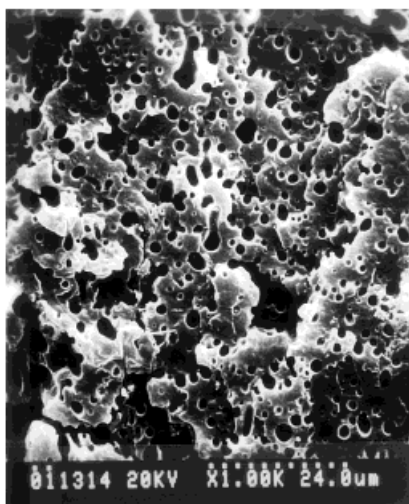
The glass transition temperature (T_g) can be used to indicate the compatibility of a binary polymer blend system. For semicrystalline PP and PBT, however, their T_g 's are not apparent from the DSC measurements. Temperatures and exothermic heats of crystallization are thus used to study the compatibility of the PP/PBT blend. Figure 6 demonstrates the effect of the epoxy content on



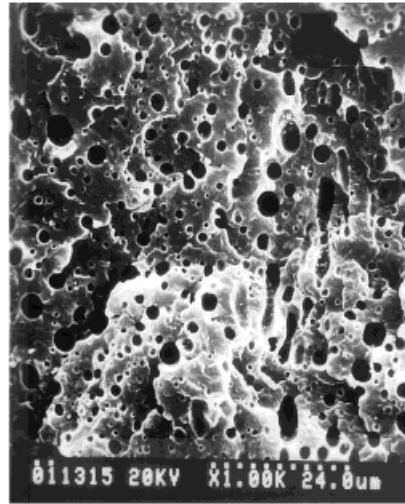
A PP/PBT/PP-MA(0.3)/Epoxy
= 60/30/10/0 phr



B PP/PBT/PP-MA(0.3)/Epoxy
= 60/30/10/0.1 phr

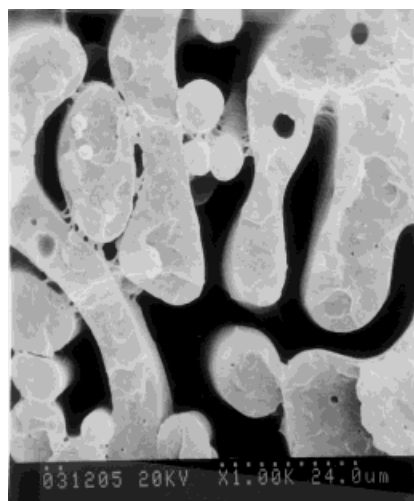


C PP/PBT/PP-MA(0.3)/Epoxy
= 60/30/10/0.3 phr

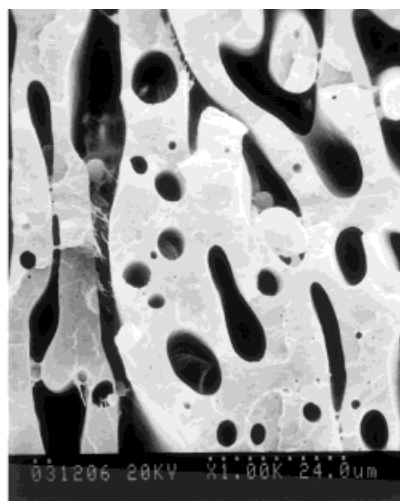


D PP/PBT/PP-MA(0.3)/Epoxy
= 60/30/10/0.5 phr

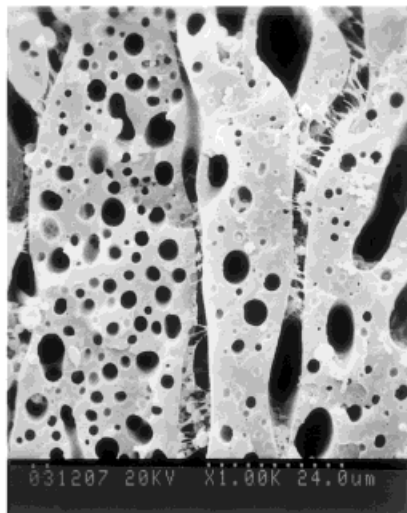
Figure 8 SEM micrographs of the etched surfaces of the PP/PBT/PP-MA/epoxy = 60/30/10/x blends containing various amounts of epoxy.



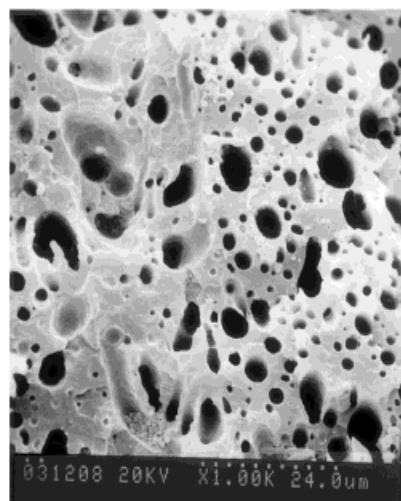
A PP/PBT/PP-MA(0.3)/Epoxy
= 40/50/10/0 phr



B PP/PBT/PP-MA(0.3)/Epoxy
= 40/50/10/0.1 phr

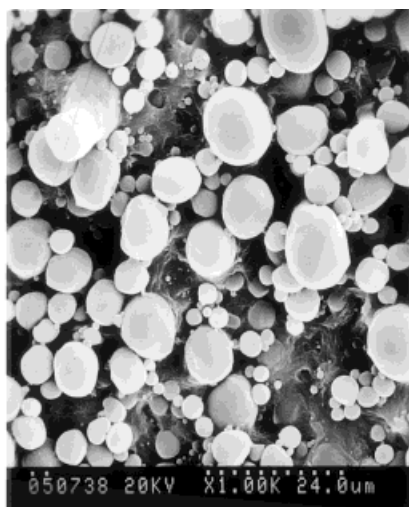


C PP/PBT/PP-MA(0.3)/Epoxy
= 40/50/10/0.3 phr



D PP/PBT/PP-MA(0.3)/Epoxy
= 40/50/10/0.5 phr

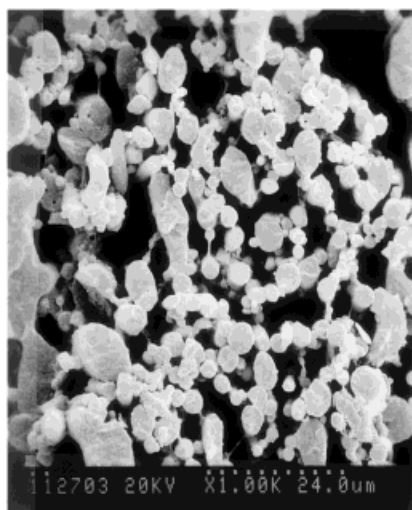
Figure 9 SEM micrographs of the etched surfaces of the PP/PBT/PP-MA/epoxy = 40/50/10/x blends containing various amounts of epoxy.



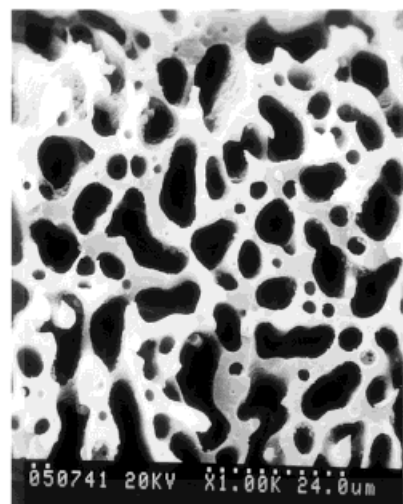
A PP/PBT/PP-MA(0.3)/Epoxy
= 20/70/10/0 phr



B PP/PBT/PP-MA(0.3)/Epoxy
= 20/70/10/0.1 phr

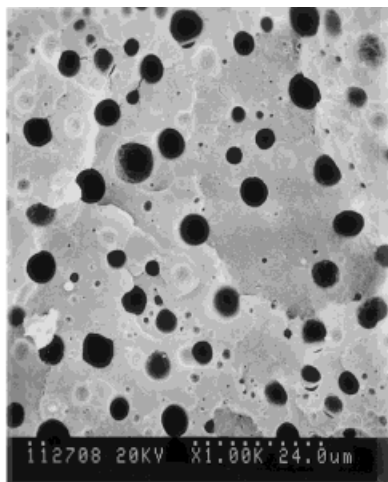


C PP/PBT/PP-MA(0.3)/Epoxy
= 20/70/10/0.3 phr

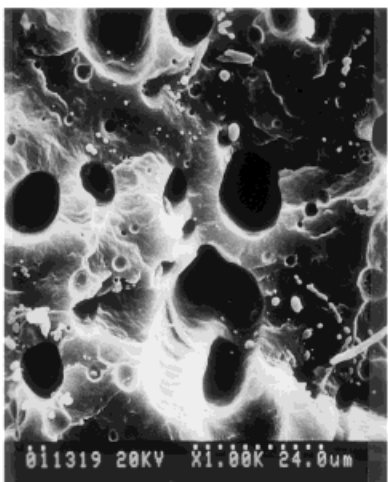


D PP/PBT/PP-MA(0.3)/Epoxy
= 20/70/10/0.5 phr

Figure 10 SEM micrographs of the etched surfaces of the PP/PBT/PP-MA/epoxy = 20/70/10/x blends containing various amounts of epoxy.



A PP/PBT/PP-MA/Epoxy = 70/30/0/0 phr



B PP/PBT/PP-MA/Epoxy = 70/30/0/0.3 phr

Figure 11 SEM micrographs of the etched surfaces of the PP/PBT/PP-MA/epoxy = 70/30/0/0 and 70/30/0/0.3 blends.

the crystallization temperature of the PP/PBT/PP-MA = 40/50/10 blend. Figure 7 shows the exothermic heats of crystallization of PP and PBT in the blend as a function of the epoxy content based on the DSC cooling curves from Figure 6. The heats of crystallization of the PP and PBT

phases are both decreased with increasing epoxy content, an indication of an increased compatibilization of the blend by increasing epoxy content. This suggests that the epoxy reacts with both PP and PBT phases, reduces their interfacial tension, and, as a result, suppresses their degrees of crystallizations.

As shown in Figure 6, during cooling of the blend, the crystallization of PBT occurs before that of PP. With increasing epoxy content, the crystallization peak temperature of the PP phase is increased whereas that of the PBT phase is decreased. The decreasing in the crystallization peak temperature of the PBT with increasing epoxy content is apparently caused by the increasingly compatibilized PP phase dispersed in the PBT phase. On the other hand, the dispersed crystallized-PBT phase in the PP matrix during cooling of the blend acts as a heterogeneous nucleating agent and, thus, facilitates the crystallization of PP. Due to an increasing extent of the PBT crystal dispersion in the blend with increasing compatibilization by the epoxy, the crystallization peak temperature of the PP phase is, thus, increased by increasing the epoxy content.

SEM Morphologies

The ultimate goal of compatibilization is to achieve stable phase morphology and improved mechanical performance. Mechanical properties of a heterogeneous polymer blend are directly related to its microstructure, especially the size and shape of the dispersed phase. Figures 8–10 show the SEM micrographs of the uncompatibilized and compatibilized by epoxy for PP/PBT/PP-MA = 60/30/10, 40/50/10, and 20/70/10 blends, respectively. The PBT component in these blends has been removed by solvent etchings. These figures show apparent decreases in the domain size of the PBT dispersed phases as the content of epoxy is increased to 0.3 phr. There is no apparently further decrease in the domain size of the PBT phase in the PBT-rich blends with a 0.5 phr of epoxy content. The finding from these SEM micrographs is consistent with the result of mechanical properties as discussed previously.

It has been known for many years that the larger volume phase tended to be more continuous in space. It was also known that the lower viscosity phase tended to be more continuous in space.²⁶ These concepts were formulated quantitatively by Paul and Barlow²⁷ and Jordhamo et al.²⁸:

Table II Effects of Amount of PP-MA on MFRs and Mechanical Properties of the PBT/PP/PP-MA/Epoxy Blends

PP/PBT/PP-MA ^a /Epoxy	MFR (g/ 10 min)	Unnotched Impact ^b (J/m)	Tensile Strength ^c (MPa)	Tensile Elongation ^d (%)	Flexural Strength ^e (MPa)	Gc^f (kJ/m ²)
50/50/0/0.3	5.1	182.6	32.9	8.0	51.0	4919
48/50/2/0.3	4.0	106.7	30.4	5.5	56.9	4534
45/50/5/0.3	3.3	159.5	31.0	6.1	58.7	7754
40/50/10/0.3	3.4	258.1	36.6	11.6	61.0	9215
30/50/20/0.3	3.7	192.1	37.4	9.2	62.6	8634

^a The MA content of the PP-MA is 0.15 wt %.

^b The standard deviations are in the range of 4–6% of the data.

^c The standard deviations are in the range of 2–5% of the data.

^d The standard deviations are in the range of 2–5% of the data.

^e The standard deviations are in the range of 3–6% of the data.

^f The standard deviations are in the range of 3–6% of the data.

$$\frac{V_I}{V_{II}} \cdot \frac{\eta_{II}}{\eta_I} = C \quad (5)$$

where if $C > 1$, phase I is continuous; if $C \sim 1$, both phases are cocontinuous; and if $C < 1$, phase II is continuous.

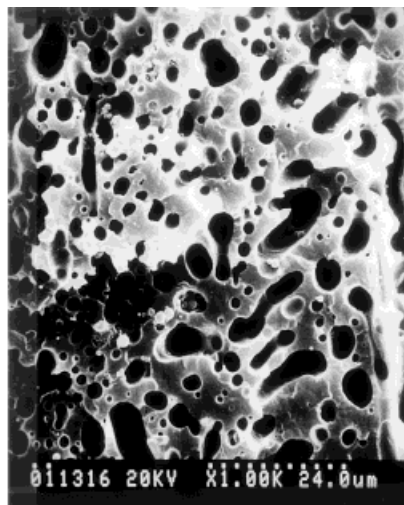
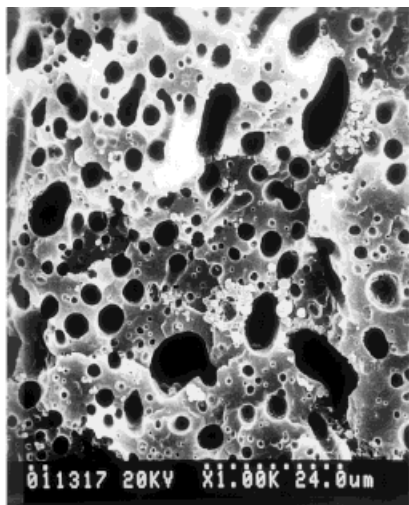
In eq. (5), V represents the volume fraction and η is the melt viscosity of phase I or phase II, as per subscript. As can be seen in Figure 9(A), phase separation is observed and the separated phases are cocontinuous before epoxy is added. The continuity of the PBT phase decreases with increasing epoxy content as observed in Figure 9(B,C). The continuity of the PBT phase disappears when 0.5 phr of epoxy is added [Fig. 9(D)]. If the PBT phase is designated as phase I in eq. (5), phase I is thus the less continuous phase due to the addition of epoxy, suggesting that epoxy reacts more significantly with PBT than with PP-MA and these reactions lead to much higher viscosity of the PBT phase than that of the PP phase, that is, smaller viscosity ratio of η_{II}/η_I . This decreased viscosity ratio of η_{II}/η_I in eq. (5) leads to a decrease of C and, thus, to a decrease of continuity of phase I. For the PP/PBT/PP-MA/epoxy = 40/50/10/0.5 blend as shown in Figure 9(D), the C value of eq. (5) is apparently less than unity because the PP phase (i.e., phase II) is continuous, but the PBT phase (phase I) is not.

Figure 11 shows the effect of epoxy on the compatibilization for the blend of PP/PBT = 70/30. It appears in Figure 11(B) that the epoxy does not give rise to an apparent compatibilization for the blend without the presence of PP-MA although the epoxy is able to react with PBT and a larger etched PBT

phase resulted. Table II shows the effects of the PP-MA content on the MFRs and the mechanical properties of the PP/PBT/PP-MA/epoxy blend system. As can be seen in Table II, the blend with 10% of PP-MA seems to give the optimum properties. SEM evidence as shown in Figure 12 demonstrates that the best compatibilization is achieved by using 10% of PP-MA for the blend because the blend gives the finest etched PBT domain among the four blends studied.

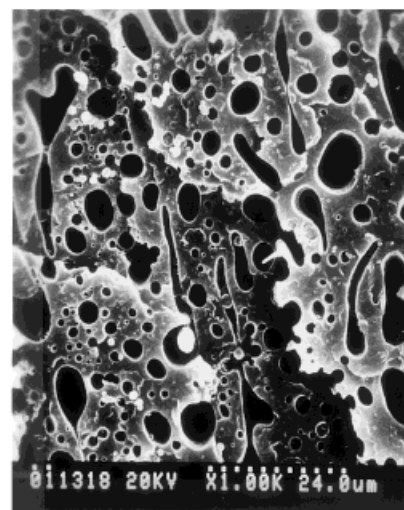
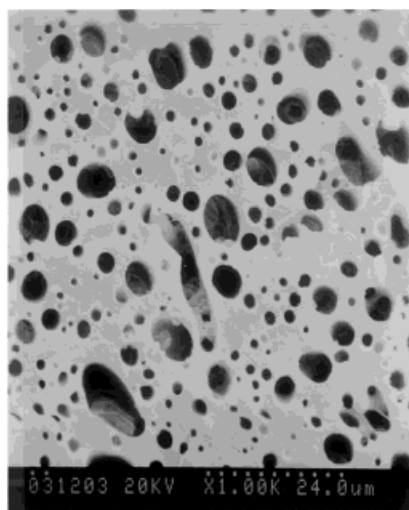
CONCLUSIONS

The mixture of PP-MA and an epoxy resin was demonstrated to be an efficient dual reactive compatibilizer for immiscible and incompatible PP/PBT blends. The PP-MA with a low MA content is miscible with PP to make it quasi-functionalized, while the multifunctional epoxy has the chance to react with PBT and PP-MA at the interface simultaneously. Thus, the *in situ*-formed PP-MA-co-epoxy-co-PBT copolymers are able to anchor along the interface and serve as efficient compatibilizers. The mechanical properties of the PBT-rich blends are improved by increasing the epoxy content to 0.3 phr. An epoxy content of 0.5 phr can lead to a light crosslinking in the PBT phase of the PBT-rich blends and result in a lack of a compatibilization effect. Epoxy does not demonstrate compatibilization effects for the PP/PBT blends without the presence of PP-MA in the blends. For PP/PBT/PP-MA/epoxy = (50 - x)/50/ x /0.3 blends, with x in the range of 2–20, the



A PP/PBT/PP-MA(0.15)/Epoxy
= 48/50/2/0.3 phr

B PP/PBT/PP-MA(0.15)/Epoxy
= 45/50/5/0.3 phr



C PP/PBT/PP-MA(0.15)/Epoxy
= 40/50/10/0.3 phr

D PP/PBT/PP-MA(0.15)/Epoxy
= 30/50/20/0.3 phr

Figure 12 SEM micrographs of the etched surfaces of the PP/PBT/PP-MA/epoxy = (50 - x)/50/x/0.3 blends containing various amounts of PP-MA.

blend with 10 wt % of PP-MA content gives the optimum properties.

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