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Effect of PBT melt viscosity on the morphology and mechanical properties of compatibilized and uncompatibilized blends with ABS

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Abstract

The toughness and morphology of poly(butylene terephthalate), PBT, blends with acrylonitrile–butadiene–styrene, ABS, materials are very sensitive to the PBT molecular weight or melt viscosity. High PBT melt viscosity is shown to lead to improved ABS (and styrene–acrylonitrile copolymer, SAN) dispersion which improves the low temperature fracture toughness of PBT/ABS blends. Compatibilization by methyl methacrylate, glycidyl methacrylate, ethyl acrylate terpolymers, or MGE, was shown to further improve ABS and SAN dispersion and reduce the ductile–brittle transition temperature. Addition of the compatibilizer broadens the window of acceptable processing temperatures. The minimum quantity of ABS required to toughen PBT is lower the higher the PBT melt viscosity. The tensile properties of PBT/ABS/MGE blends were shown to be relatively insensitive to PBT melt viscosity or reactive compatibilization. © 1999 Elsevier Science Ltd. All rights reserved.

Keywords: Methyl methacrylate–glycidyl methacrylate; Poly(butylene terephthalate); Styrene–acrylonitrile

1. Introduction

Poly(butylene terephthalate), PBT, is an important engineering polymer because of its excellent tensile properties, abrasion and chemical resistance, as well as its uses for electrical insulation [1–15]. PBT has very high unnotched Izod impact strength; however, notched specimens of PBT fail in a brittle manner. This indicates that PBT has a high resistance to crack initiation but low resistance to crack propagation. In order to overcome this problem, many studies of incorporation of impact modifiers into PBT have been reported [1–38].

The current work is part of a series of studies on the impact modification of PBT by acrylonitrile–butadiene–styrene materials, ABS, using methyl methacrylate (MMA), glycidyl methacrylate (GMA), ethyl acrylate (EA) terpolymers, or MGE, as a reactive compatibilizer [2–4]. It has been shown that an MGE-g-PBT graft copolymer is formed at the PBT/ABS interface by reactions between the carboxyl endgroups of PBT and the epoxide groups along the MGE chain; the copolymer successfully reduces the scale of dispersion, provides morphological stability, gives a broader processing window, and improves low temperature impact toughness of PBT/ABS blends. However, undesirable crosslinking reactions also occur involving the epoxide functionality of MGE catalyzed by residual acid impurities in emulsion-made ABS materials which result in a reduction of impact strength in toughened PBT blends [3]. By altering the order of mixing of the blend components this problem can be reduced to a large extent; e.g. using a two-pass extrusion method where PBT and MGE are melt-mixed together prior to extrusion with ABS. This allows for the formation of the desired graft copolymer first and reduces the competition for available epoxide units for crosslinking reactions.

This paper addresses the question of how the molecular weight, or melt viscosity, of the PBT matrix affects blend properties. Previous work on blends of nylon 6 or polycarbonate materials has shown that the molecular weight of these engineering plastics has a strong influence on the morphology and impact properties of blends with maleated elastomers, maleated polyolefins, and core-shell type impact modifiers [39–45]. Typically, a high matrix viscosity produces a finer dispersion of the impact modifiers; this usually leads to increased blend toughness and reduced ductile–brittle transition temperature. In addition, the inherent ability of the matrix to be toughened may increase with molecular weight. Most of the changes in properties observed here appear to correlate with blend morphology which suggests that the effect of PBT molecular weight on

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Table 1
Materials used

Designation here	Supplier designation	Brabender torque (N m)[a]	Apparent viscosity (Pa s)[b]	Izod impact strength (J/m)[c]	Source
PBT-6131	Crastin 6131	1.4	178	50	DuPont
PBT-6130	Crastin 6130	3.7	301	50	DuPont
PBT-6129	Crastin 6129	5.7	375	50	DuPont
PBT-310	Valox 310	3.0	260	50	General Electric
PBT-315	Valox 315	5.8	387	50	General Electric
ABS	SAN-g	16.5	—	550	Cheil Industries
SAN	Tyril 1000	6.5	—	30	Dow Chemical

[a] Values taken after 10 min at 250°C and 50 rpm.
[b] Values taken at 230°C and 1000 s^{-1} using a capillary viscometer.
[c] Room temperature notched Izod impact.

the melt viscosity of this phase is the dominant issue rather than any intrinsic effect of molecular weight on properties.

The current work will focus on the effects of PBT melt viscosity on the morphology of blends with a styrene–acrylonitrile copolymer, SAN, and the morphology and mechanical properties of blends with an ABS material compatibilized by MGE terpolymer. Evidence will be presented that high PBT melt viscosity is beneficial for developing a finer SAN or ABS dispersion during processing which improves low temperature toughness.

2. Experimental

The materials used in this study are described in Table 1. Five PBT resins of different melt viscosities were obtained from DuPont Engineering Polymers and General Electric Co.; they are designated here by their supplier reference number. Determination of the absolute PBT molecular weight was beyond the scope of this work; our laboratory is not equipped to make such measurements on PBT. The apparent viscosities were measured by capillary viscometer at 230°C and are summarized in Table 2 as a function of shear rate.

An ABS material obtained from Cheil, an emulsion-made grafted rubber concentrate containing 45% rubber with an average particle size of 0.3 mm, was selected for this study since it leads to superior properties of blends with PBT compared to other ABS materials considered in this work [1,4]. The styrene–acrylonitrile copolymer, SAN, designated as Tyril 100 was obtained from the Dow Chemical Co. A more detailed characterization of the ABS and SAN materials is given elsewhere [1,2]. A Brabender Plasticorder with a 50 ml mixing head (operated at 250°C and 50 rpm) was also used for rheological characterization of these materials. Fig. 1 relates the viscosities of the PBT resins obtained from Brabender torque rheometry to those obtained from the capillary rheometer. There is a linear relationship between the results obtained by the two methods; hence, the following sections will represent the PBT melt viscosity in terms of Brabender torque. The reactive compatibilizer used in this work is a mass-made terpolymer containing 88 wt% MMA (for miscibility with SAN), 10 wt% GMA (for reactive functionality) and 2 wt% EA (to prevent unzipping) and is designated as MGE-10. The Brabender torque of MGE-10 is 6.1 N m after 10 min of mixing. The details of the synthesis and characterization of this material are found elsewhere [2].

Table 2
Melt viscosity of neat PBT resins for different shear rates at 230°C

PBT type	Apparent viscosity (Pa s) at 116 s^{-1}	350 s^{-1}	1160 s^{-1}
PBT-6131	260	240	166
PBT-6130	650	413	295
PBT-6129	579	495	354
PBT-310	469	319	240
PBT-315	662	524	360

Pellets of PBT were cryogenically ground to a powder and, along with reactive compatibilizer powder, dried for 16 h in a vacuum oven at 65°C. Cryogenically ground SAN and as-received ABS powders were dried in a convection oven for 16 h at 70°C. All blend components were thoroughly mixed prior to extrusion. Blends were processed in a twin screw extruder and injection molded into test specimens; a detailed description of the processing equipment used here is described elsewhere [1,2].

A JEOL JEM 200cx transmission electron microscope (TEM), operated at an acceleration rate of 120 kV, was

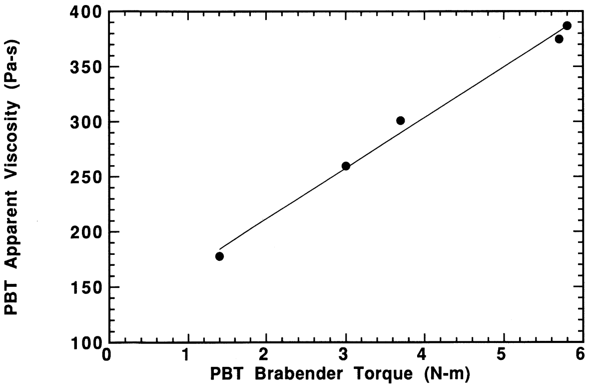

Fig. 1. Capillary melt viscosity at 230°C and 1000 s^{-1} versus Brabender torque after 10 min of mixing at 250°C for neat PBT materials.

used to characterize the morphology of selected blend compositions. Ultrathin sections were obtained from the center of injection molded Izod bars perpendicular to the direction of flow by cryomicrotoming with a Riechert-Jung Ultracut E microtome at − 45°C. The sections of PBT/SAN blends were stained over RuO_4 vapor for 20 min, whereas blends of PBT with ABS were stained with OsO_4 for 24 h. RuO_4 preferentially stains the SAN phase while OsO_4 stains the rubber particles of ABS. The weight average SAN particle diameters of PBT/SAN blends were determined using NIH Image software. Non-round particles are assigned the diameter of a circle with equivalent area. No attempt was made to account for the fact that the microtome does not cut each particle at its equator.

Notched Izod impact strength was measured according to ASTM D256 as a function of temperature, and the ductile–brittle transition temperature of each blend was determined. At least five samples each from the gate- and far-end of the injection molded bars were tested at room temperature and in the region of the ductile–brittle transition. At other temperatures fewer samples were used, the exact number being dictated by the consistency observed. Only gate-end information is presented here since differences between gate- and far-end values were typically insignificant. An Instron was used for tensile testing according to ASTM D638 at a crosshead speed of 5.08 cm/min. An extensometer strain gauge with a 5.08 cm gap was used to obtain modulus and yield stress values.

3. Morphology of PBT/SAN blends

The properties of immiscible polymer blends are a strong function of dispersed phase particle size or interparticle distance [46–48]. The particle size is governed by a competition between particle break-up and coalescence during melt processing [49–51]. The size of the particles of the dispersed phase is expected to depend on the melt viscosities of the dispersed and matrix phases, the shear rate applied during processing, and interfacial tension as described by the Taylor theory of drop break-up and by various correlations [40,52–54].

The effects of PBT melt viscosity and reactive compatibilization on the morphology of PBT/SAN blends are described in the following section as a simplified model for the PBT/ABS system. The SAN particle size of binary PBT/SAN blends can be manipulated by changing the shear rate during processing or the PBT to SAN viscosity ratio [54–56]. In general, a higher matrix viscosity should yield a finer dispersion of particles during melt processing. In the absence of a compatibilizer, the morphology generated may be unstable and coarsening can occur under certain conditions [2,57].

The TEM photomicrographs shown in Fig. 2 illustrate the effects of increasing PBT melt viscosity and reactive compatibilization on the morphology of PBT/SAN/MGE-10 blends containing 20% dispersed phase for three selected PBT types. The SAN phase is stained dark by RuO_4. By increasing the melt viscosity of the PBT, the stresses applied to the SAN phase during processing are increased and the SAN domain size is decreased for both compatibilized and uncompatibilized blends. The presence of compatibilizer decreases the SAN particle size below that seen for the corresponding uncompatibilized blend.

Fig. 3 shows that the weight average particle size, $\bar{d}_w$, of the SAN domains in PBT/SAN/MGE-10 blends decreases as the melt viscosity (expressed as Brabender torque) of the neat PBT is increased for both compatibilized and uncompatibilized blends. The addition of MGE-10 decreases $\bar{d}_w$ by only a small amount; the reason for such a small effect from adding compatibilizer, relative to observations for nylon 6 [58], is possibly a result of the difunctional nature of PBT. Some PBT chains are expected to have two carboxyl groups, both of which can react with epoxide groups, causing a crosslinking type effect that limits the degree of dispersion possible [2]. Similar behavior has been observed before for PBT/SAN/MGE blends where the epoxide content of the MGE terpolymer is increased in an attempt to further decrease the SAN particle size; it was found that relatively small GMA contents in the MGE terpolymer were required to reduce the $\bar{d}_w$ to a minimum, and further increases in epoxide content resulted in little change in dispersed phase particle size [2]. It was also observed that processing in a single screw extruder instead of the twin screw extruder used here resulted in a bimodal particle size distribution, i.e. a population of very large particles and a population of very small particles.

4. Impact properties of PBT/ABS blends

As mentioned earlier, a high matrix melt viscosity is generally beneficial for producing blends of engineering thermoplastics with improved fracture toughness. Fig. 4 shows the effect of matrix melt viscosity on the notched Izod impact properties of PBT blends containing 30% ABS. Two different mixing protocols were used to prepare these blends: one was a single-pass extrusion method where all components are melt-mixed together at once; while in the other, PBT and MGE-10 were compounded together prior to adding ABS in a second extrusion step.

As PBT melt viscosity is increased, the room temperature impact strength and the ductile–brittle transition temperature are improved for both compatibilized and uncompatibilized blends prepared by the single-pass extrusion method; however, with the exception of the lowest melt viscosity PBT, blends containing 5% MGE-10 have a lower impact strength than their uncompatibilized counterparts. Such a loss in fracture toughness upon compatibilization has been observed in prior work and has been attributed to crosslinking via reactions of the epoxide groups of the MGE terpolymer catalyzed by residual acid contaminants commonly present in emulsion-made ABS materials [3]. There is a significant decrease in the ductile–brittle transition temperature caused by addition of the reactive compatibilizer over the entire range of PBT melt viscosities. By melt mixing PBT and MGE-10 together prior to adding ABS, the potential for MGE-g-PBT graft copolymer formation is increased relative to crosslink formation within the ABS phase. For selected compositions prepared using this two-pass extrusion method, the impact strength is higher for the compatibilized blends than the uncompatibilized blends; however, there is little improvement in the ductile–brittle transition temperature relative to blends prepared by a single-pass extrusion.

The low viscosity material designated as PBT-6131 is an injection molding grade, whereas all other PBT types used are extrusion grade materials. Binary PBT-6131/ABS blends fracture in a brittle manner at room temperature; however, addition of 5% MGE-10 causes ductile failure. The benefit of compatibilization is seen by the increase in room temperature impact toughness and a substantial reduction of the ductile–brittle transition temperature for this blend; the lower melt viscosity PBT-6131/ABS/MGE-10 alloy is much easier to process (30% shorter injection times during the molding cycle) than blends with more viscous PBT resins. Thus, this compatibilized blend is an attractive material for injection molding applications.

The TEM photomicrographs in Fig. 5 show the effect of matrix melt viscosity and reactive compatibilization on PBT/ABS blend morphology made clear by the OsO_4 staining of rubber particles in ABS which appear dark in these

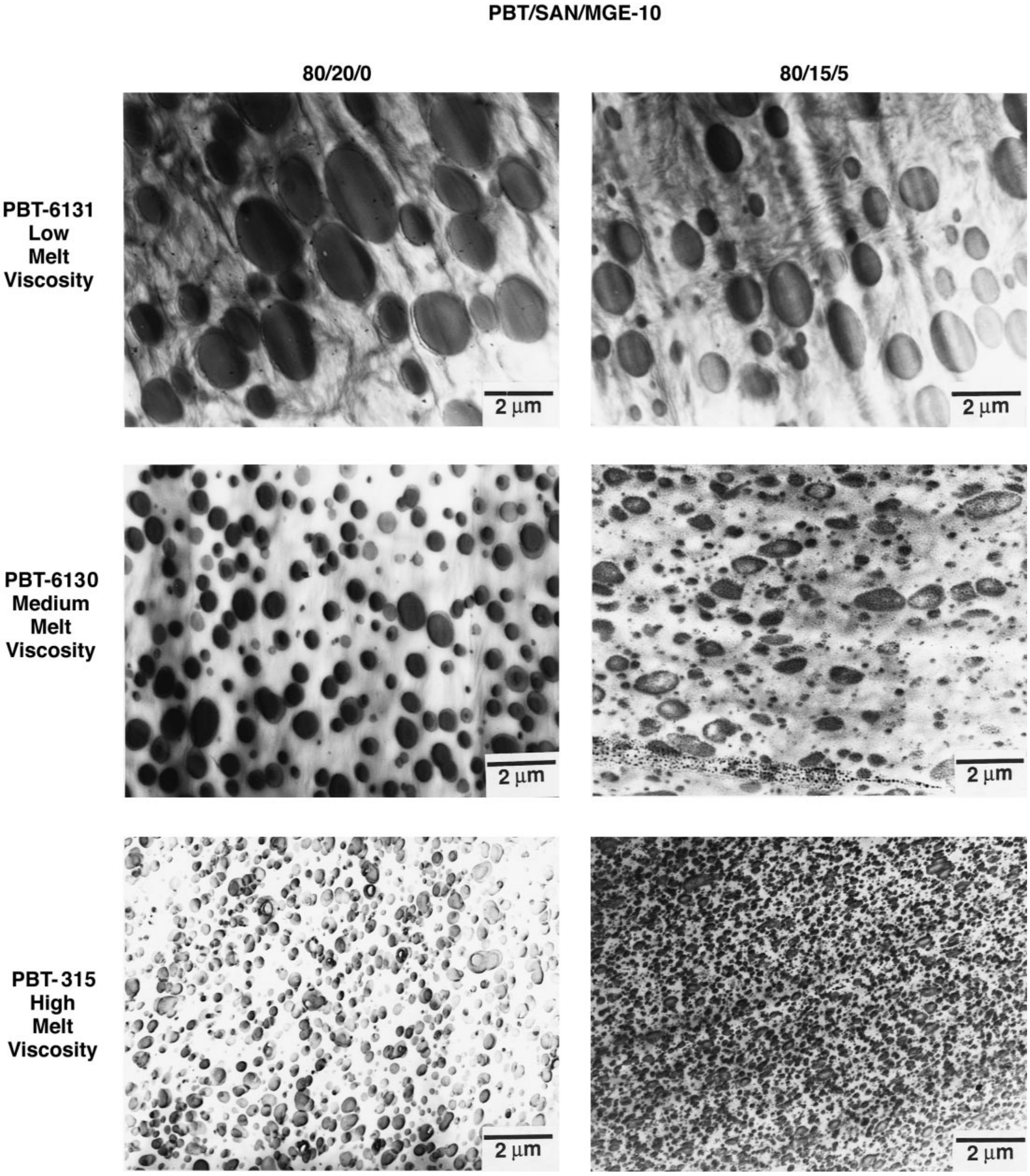

Fig. 2. TEM photomicrographs of PBT/SAN/MGE-10 $(80 - X)/20/X$ blends containing high, medium and low melt viscosity PBT materials extruded at 220°C and molded at 240°C. The SAN phase is stained dark by RuO_4.

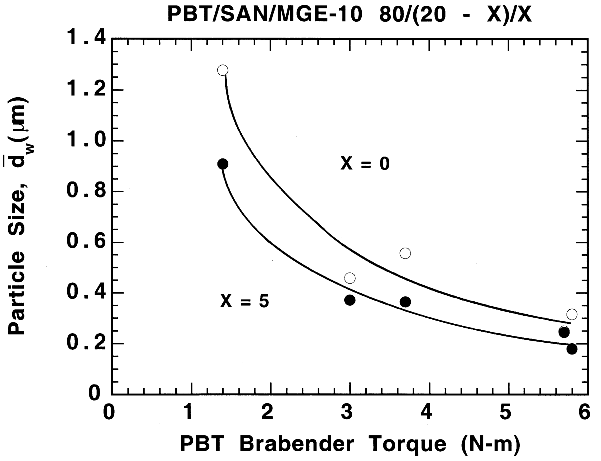

Fig. 3. Dispersed phase domain size of PBT/SAN/MGE-10 (80 − *X*)/20/*X* blends as a function of neat PBT melt viscosity for blends extruded at 220°C and molded at 240°C.

photomicrographs. As PBT viscosity increases, ABS dispersion in binary blends is improved. Because of the low melt viscosity of the PBT-6131 material, its binary blend with ABS has relatively poor dispersion which is probably the cause for the brittle failure of this blend. Addition of the MGE-10 terpolymer improves ABS dispersion for all PBT materials, which is no doubt responsible for the large improvement in the low temperature impact properties observed.

Previous work has shown that the fracture properties of uncompatibilized PBT-315 (high melt viscosity) blends are only slightly sensitive to extrusion temperature when compounded in a twin screw extruder; however, significant coarsening of the ABS domain size, resulting in an increase in the ductile–brittle transition temperature, was observed when these blends were injection molding at higher temperatures [1]. It was also shown that the addition of MGE-10 to these blends broadens the window of acceptable processing temperatures for molding which is attributed to the creation of the MGE-g-PBT graft copolymer at the PBT/ABS interface, thereby sterically hindering coalescence of the ABS domains [2].

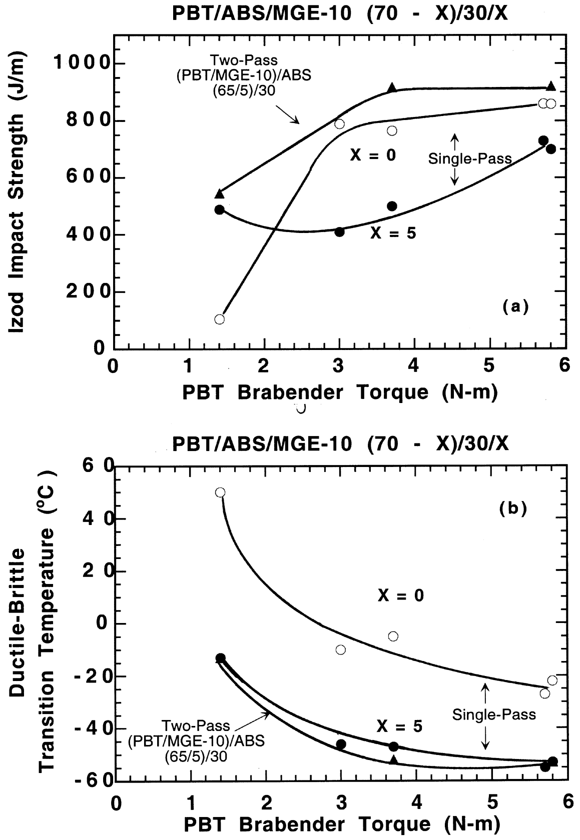

Fig. 4. Effect of PBT melt viscosity on the room temperature notched Izod impact strength (a) and the ductile–brittle transition temperature (b) of PBT/ABS/MGE-10 (70 − *X*)/30/*X* blends extruded at 220°C by two mixing protocols and molded at 240°C.

Table 3 compares the effect of processing temperature on impact properties for two extrusion grade PBT materials, PBT-315 (high melt viscosity) and PBT-6130 (medium melt viscosity). Blends based on PBT-315 have superior impact properties to those containing the lower molecular weight PBT-6130 material. Trends described above are the same for blends based on both PBT materials. When molded at a constant temperature of 240°C, different extrusion temperatures (220 or 260°C) have little effect on the impact properties of binary PBT/ABS blends containing both PBT types shown here; however, the ductile–brittle transition temperature is nearly 20°C higher for the uncompatibilized blend based on both PBT resins when molded at 260°C versus 240°C.

Table 3
Effect of processing temperature on notched Izod impact properties of PBT/ABS/MGE-10(70 − *X*)/30/*X* blends

Process temperature extrusion/ molding (°C)	PBT-315 (high viscosity)				PBT-6130 (medium viscosity)			
	$X = 0$		$X = 5$		$X = 0$		$X = 5$	
	Notched Izod impact strength (J/m)	Ductile–brittle transition temperature (°C)	Notched Izod impact strength (J/m)	Ductile–brittle transition temperature (°C)	Notched Izod impact strength (J/m)	Ductile–brittle transition temperature (°C)	Notched Izod impact strength (J/m)	Ductile–brittle transition temperature (°C)
220/240	859	− 22	720	− 52	790	− 5	480	− 47
260/240	887	− 18	652	− 65	692	− 2	425	− 45
220/260	926	2	580	− 55	359	19	520	− 45

Addition of the MGE-10 terpolymer improves the low temperature impact properties and the reduced ductile–brittle transition temperature is relatively insensitive to processing temperature.

The effect of ABS content on the impact properties of PBT/ABS/MGE-10 blends for the same two injection molding grade PBT resins described above is shown in Table 4. To achieve high toughness at room temperature, a minimum ABS content is required in these blends, 20% for PBT-315 and 30% for PBT-6130. Above this critical ABS content the presence of compatibilizer significantly reduces fracture toughness for both PBT types beyond their uncompatibilized counterparts. However, it is apparent that a high matrix melt viscosity during processing is beneficial for producing blends with superior impact strength for all compositions shown. Increasing the ABS content beyond this critical point has only a minor effect on the ductile–brittle transition temperature of uncompatibilized blends based on both PBT materials; however, the ductile–brittle transition temperature is reduced to as low as −75°C when MGE-10 terpolymer is added, which approaches that of the neat ABS material (−80°C).

5. Tensile properties of PBT/ABS blends

Previous papers have reported the tensile properties of PBT-315/ABS blends as a function of the content and characteristics of the ABS and MGE components [1,2]. As the ABS content is increased, tensile modulus and yield strength generally decrease as expected from a rule of

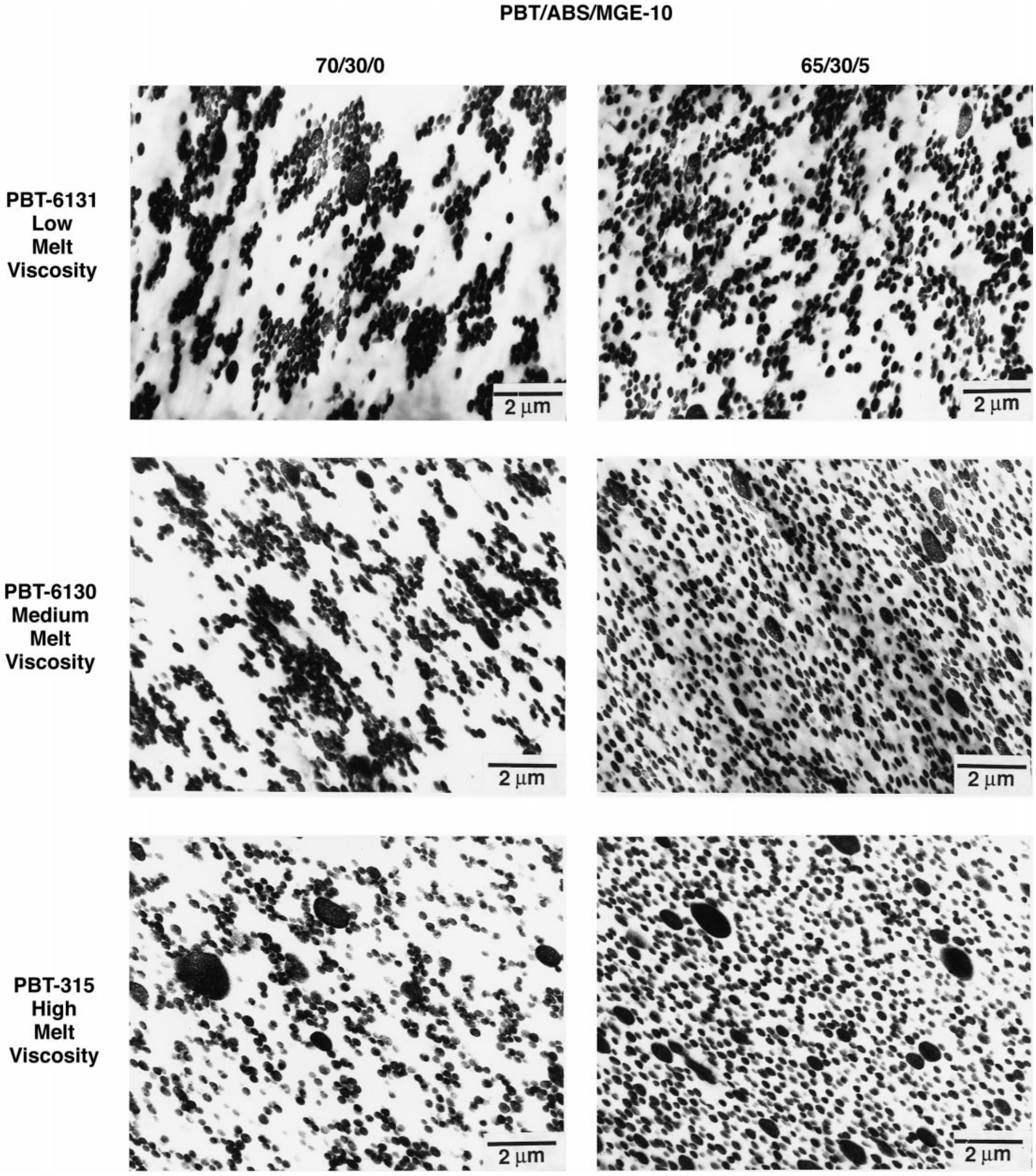

Fig. 5. TEM photomicrographs of PBT/ABS/MGE-10 (70 − X)/30/X blends containing high, medium and low melt viscosity PBT materials extruded at 220°C and molded at 240°C. The rubber in the ABS phase is stained dark by OsO_4.

Table 4
Effect of ABS content on notched Izod impact strength of PBT/ABS/MGE-10 blends

Blend composition PBT/ABS/MGE-10	PBT-315 (high viscosity)				PBT-6130 (medium viscosity)			
	$X = 0$		$X = 5$		$X = 0$		$X = 5$	
	Notched Izod impact strength (J/m)	Ductile–brittle transition temperature (°C)	Notched Izod impact strength (J/m)	Ductile–brittle transition temperature (°C)	Notched Izod impact strength (J/m)	Ductile–brittle transition temperature (°C)	Notched Izod impact strength (J/m)	Ductile–brittle transition temperature (°C)
$90 - X/10/X$	110	46	75	42	94	48	73	47
$80 - X/20/X$	640	8	558	− 10	351	26	260	26
$70 - X/30/X$	859	− 22	720	− 49	790	− 5	480	− 47
$60 - X/40/X$	920	− 22	880	− 52	911	− 18	647	− 62
$50 - X/50/X$	914	− 30	785	− 75	870	− 20	777	− 70

mixtures. The presence of compatibilizer has only a minor effect on these properties.

Table 5 shows the tensile properties of the neat PBT resins and their corresponding blends containing 30% ABS with and without 5% MGE-10. It is clear that the tensile properties of the neat PBT resins show little sensitivity to PBT type; however, the elongation at break of PBT-6131 is slightly lower than for the higher melt viscosity materials. The presence of 30% ABS reduces the tensile properties nearly equally for all PBT materials. Incorporation of 5% MGE-10 results in only a small increase in tensile modulus relative to binary PBT/ABS blends. Table 6 compares the effect of ABS content on binary PBT/ABS blends for two PBT types: PBT-315 (high viscosity) and PBT-6130 (medium viscosity). The tensile modulus and yield strength decrease equally, according to a rule of mixtures, for both PBT types as the ABS content is increased. The elongation at break does not follow the same trend as modulus and yield strength. Elongation at break is decreased by the presence of a small amount of ABS but increases substantially at higher ABS contents, e.g. 40% ABS for PBT-315 blends and 50% for PBT-6130 blends.

6. Conclusions

The morphology and fracture properties of PBT/ABS blends have been shown to be very sensitive to PBT melt viscosity. In general, high PBT viscosity results in improved SAN or ABS dispersion and, hence, improved low temperature impact toughness. Addition of MGE-10 terpolymer greatly improves blend properties by producing a finer dispersion of SAN or ABS; this leads to a reduction in the ductile–brittle transition temperature far beyond that of the corresponding uncompatibilized blends. The reduction in impact strength, which occurs as a result of addition of

Table 5
Tensile properties of PBT and PBT/ABS blends[a]

Blend composition (wt%)	Tensile modulus (GPa)[b]	Yield strength (MPa)[b]	Elongation at break (%)[b]
Neat PBT			
PBT-6131	2.4 ± 0.02	53 ± 0.5	100 ± 20
PBT-6130	2.4 ± 0.02	53 ± 0.6	250 ± 15
PBT-6129	2.3 ± 0.02	52 ± 0.5	249 ± 30
PBT-310	2.3 ± 0.03	53 ± 0.5	247 ± 30
PBT-315	2.4 ± 0.02	51 ± 0.4	240 ± 40
PBT/ABS 70/30			
PBT-6131	1.7 ± 0.02	38 ± 0.5	22 ± 5
PBT-6130	1.7 ± 0.01	38 ± 0.4	90 ± 30
PBT-6129	1.7 ± 0.02	39 ± 0.3	65 ± 30
PBT-310	1.7 ± 0.02	38 ± 0.4	67 ± 20
PBT-315	1.7 ± 0.01	39 ± 0.4	75 ± 30
PBT/ABS/MGE-10 65/30/5			
PBT-6131	1.7 ± 0.02	41 ± 0.1	21 ± 5
PBT-6130	1.7 ± 0.02	41 ± 0.4	80 ± 22
PBT-6129	1.7 ± 0.01	42 ± 0.5	78 ± 25
PBT-310	1.7 ± 0.01	42 ± 0.3	74 ± 20
PBT-315	1.7 ± 0.02	42 ± 0.4	60 ± 20

[a] Blends were extruded at 220°C and molded at 240°C.

[b] Five specimens of each sample were measured.

Table 6
Effect of ABS content on tensile properties of PBT/ABS blends[a]

Blend composition % ABS	PBT-315 (high melt viscosity)			PBT-6130 (medium melt viscosity)		
	Tensile modulus (GPa)[b]	Yield strength (MPa)[b]	Elongation at break (%)[b]	Tensile modulus (GPa)[b]	Yield strength (MPa)[b]	Elongation at break (%)[b]
0	2.4 ± 0.02	51 ± 0.4	240 ± 40	2.4 ± 0.02	53 ± 0.6	250 ± 15
20	1.9 ± 0.09	41 ± 0.3	76 ± 35	2.0 ± 0.07	41 ± 0.3	31 ± 14
30	1.7 ± 0.01	39 ± 0.4	75 ± 30	1.7 ± 0.01	38 ± 0.4	90 ± 30
40	1.5 ± 0.09	31 ± 0.1	300 ± 97	1.5 ± 0.06	31 ± 0.3	96 ± 16
$X = 50$	1.3 ± 0.06	28 ± 0.2	248 ± 69	1.3 ± 0.03	27 ± 0.1	190 ± 79

[a] Blends were extruded at 220°C and molded at 240°C.
[b] Five specimens of each sample were measured.

the compatibilizer, was overcome by altering the order of mixing the blend components. The deleterious effect on low temperature toughness, caused by ABS phase coarsening when molding these blends at high temperatures, is essentially eliminated for these PBT materials by adding MGE-10. The critical ABS content required for toughening these blends is lower the higher the PBT melt viscosity.

Tensile properties of these blends were shown to be relatively insensitive to PBT melt viscosity. The presence of ABS reduces the tensile modulus and yield strength. Quite small increases in yield strength occur as a result of incorporating MGE-10 into these blends for all PBT types used. No changes in tensile modulus or elongation at break were observed as a result of compatibilization.

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