

The Impact Properties of Bulk Compatibilized PS/LDPE Blends

C. Kunyawut ^a and J. S. Higgins ^b

Abstract:

The impact properties of bulk compatibilized PS/LDPE blends have been studied. Polystyrene-*block*-poly(ethylene-butylene)-*block*-polystyrene (SEBS) triblock copolymer with number average molecular weights (M_n) of 250,000 and 55,000 were employed as compatibilizers. The PS/LDPE blends with various concentrations of SEBS were prepared by melt mixing using a mini twin screw extruder at a mixing temperature of 180°C and a screw speed of 60 rpm. The bulk morphology of the blends was studied using optical (OM), electron (SEM) and atomic force (AFM) microscopy. From these techniques the size and size distribution of the rubbery phase were obtained. Clear evidence of droplet type morphology was found in most samples. The SEBS copolymers have the effect of reducing the interfacial tension between the homopolymers, as indicated by the smaller dispersed phase dimensions when small amounts of the SEBS copolymers were added to the blends. The low molecular weight copolymer was found to be superior to the high molecular weight one in terms of emulsifying effect. The mechanical properties (impact strength) of the blends were studied using a standard Charpy impact instrument. Little increase in impact strengths of the blends was observed in the uncompatibilized blends. A further increase in impact strength was obtained in the SEBS compatibilized blends. However, the marginal increase in the impact strengths of the both the uncompatibilized and compatibilized blends

indicated that LDPE has low potential in toughening PS.

Keyword: PS, LDPE, SEBS, Rubber toughening, Impact strength

1. Introduction

Glassy polymers have properties which are adequate for many applications and are normally used in service below their glass transition temperatures, T_g . In general, they are prone to brittle fracture¹ particularly when notched or subjected to high strain rate impact loading at low temperatures. Because of such problems, toughened polymers have been developed in which a glassy polymer matrix is toughened through the incorporation of second phase consisting of particles which are usually spherical and composed of a rubbery polymer above its T_g .

¹ Brittle: In common usage; liable to break, fragile. It is often used to describe fracture in which sharp cracks propagate through bodies with small amount of local deformation around the tip. In general, this will lead to lower toughness than cases where there is larger deformation, but some brittle fractures involve much larger energies than other. For example, an inorganic glass and a high strength steel can both have brittle cracks but one is much tougher than the other (Williams, 1987). Practically, the expression *brittle fracture* means that the extent of yielding in the specimen is very limited, so that the radius of the plastic zone at the crack tip is small in relation to the crack length (Bucknall, 1977).

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This can lead to significant improvements in the mechanical behavior of the matrix polymer and is often referred to as "rubber toughening".

The initiation and propagation of cracks in glassy polymers is essentially a competition between crazing and shear yielding. If crazing dominates the deformation, the polymer is brittle whereas if the bulk shear yielding occurs preferentially the polymer is generally ductile. Glassy vinyl polymers such as PS and PMMA have relatively high shear yield stresses and low resistance to craze propagation and failure. Their low chain entanglement density and low surface free energy make them susceptible to crazing, while the large cross-sectional area of their molecules increase the probability of chain rupture under a given applied stress (Bucknall, 2000). In contrast, polymers having flexible main-chain linkages such as PC and poly(phenylene ether) (PPE) are relatively ductile and undergo extensively shear yielding at ambient temperature because of their high chain entanglement density and a smaller chain cross-sectional area which requires high stresses for crazing to take place via chain rupture or scission (Donald, 1997).

It is well agreed that in brittle polymers, as well as in ductile ones, most of the impact energy is absorbed by the matrix polymer (Sperling, 1997). For instance, in glassy polymers which are susceptible to crazing, multiple crazing is the desired toughening mechanism. It is the main mechanism of toughening in high-impact polystyrene (HIPS) and ABS. Multiple crazing is also important in rubber-toughened PMMA, especially at low temperature (Bucknall, 1997). Recently, it has been recognized that optimization of rubber toughening requires the rubber particles not only to act as stress

concentrators but also to undergo cavitation. The formation of holes, called cavitation, in the rubber phase has been found to be one of the most important ways in which toughened plastics can respond to tensile stress. While cavitation in itself cannot be regarded as an important energy absorbing process, it reduces the resistance of the particles to deformation, initiating yielding in the matrix at reduced stresses and allowing the cavitated particles to cold draw (strain softening) despite the constraints imposed at the crack tip. Then, at a later state of deformation, stretching of rubber fibrils within the cavitated particles introduces a significant degree of strain hardening. This combination of responses to applied stresses makes rubber cavitation an effective response to the triaxial tensile stresses generated at a crack tip, because it allows the surrounding matrix to deform by all available mechanisms. The matrix may respond by shear yielding, multiple crazing, or both, depending on its properties, and the strained rubber particles then stabilize the most highly strained regions of the polymer (Bucknall, 2000).

In most cases, blending of two or more immiscible homopolymers results in a multiphase system which normally exhibits coarse unstable phase morphology, thus making it difficult, or impossible, in practice to achieve improvement of mechanical properties. The interfaces between phases have been often addressed as one of sources of problems. The typical case is that immiscible blend systems have high interfacial tension and poor adhesion between phases. This interfacial tension contributes, along with high viscosity, to the inherent difficulty of imparting the desired degree of dispersion to random mixtures and to their

subsequent lack of stability to gross separation or stratification during later processing or end-use. Poor adhesion leads, in part, to the very weak and brittle mechanical behavior often observed in dispersed blends (Paul and Newman, 1978). When an A-B copolymer capable of phase segregation is added to an immiscible blend of homopolymers A and B it will try to reduce the number of unfavorable contacts between the homopolymer and the dissimilar copolymer blocks thus reducing the interaction energy. This results in localization of the copolymer at the interface between the two homopolymers. In so doing it causes a decrease in the interfacial tension between the homopolymers which leads to a reduction in the average size of the dispersed phase (Wu, 1978). Noolandi and Hong (1982, 1984) and Leibler (1988) have developed theories based on statistical thermodynamics that can be employed to predict the emulsifying effect of a copolymer on immiscible polymer blends. In this study, Polystyrene-*block*-poly(ethylene-butylene)-*block*-polystyrene (SEBS) triblock copolymer of various molecular weights were employed as compatibilizers.

The objective of this study is to evaluate any improvement in the toughness² of SEBS compatibilized PS/LDPE blends.

2. Materials and Experimental Methods

The PS used was a provided by BP Chemical Company ($M_w = 250,000$, polydispersity = 2.2). The LDPE (Stamylan LD2100 TN00) is a commercial product of DSM, The Netherlands ($M_w = 121,000$, polydispersity = 12.3). The molecular weights of the homopolymers were determined using GPC technique performed by RAPRA Technology Ltd. UK. All homopolymers were obtained in pellet form. The SEBS copolymers used are commercial

products of Shell Development Company: Kraton G1651 and Kraton G1652. The number average molecular weight (M_n) values of the SEBS were obtained from the supplier and are shown in Table 1.

Table 1 Molecular characteristics of SEBS

SEBS	Total M_n	Mid-Block M_n	End-Block M_n	PS (%)
G1651	240,000	160,000	39,000	33
G1652	55,000	39,000	8,000	29

Blends were prepared using a co-rotating twin-screw extruder (PRISM Co., UK). The barrel length (L) and diameter (D) are 224 mm and 16 mm respectively. The diameter of the capillary die is 1 mm. The concentration of the blends were 70/30wt% of PS/LDPE. The blend components were dry mixed before feeding into the hopper of the extruder. In order to minimize the effect of processing conditions, at the desired screw speed (60 rpm), the temperature of the barrel wall inside the extruder (the feed and mixing zone and at the die) was maintained constant at $180^\circ\text{C} \pm 5^\circ\text{C}$ using three automatic temperature controllers. To investigate the effect of compatibilizers on the morphology and impact strength of the blends, the concentration of the SEBS triblock copolymers (G1651 and G1652) which was varied from 5 to 15wt% of LDPE content were added into the blend. After extruding, the

² Toughness may be defined as the ability to resist fracture by absorbing energy. It is usually referred to the work done in forming a new unit area of fracture or crack surface.

strands were cooled in air to room temperature. The cooling time was about 10 minutes. The extruded strands about 10 cm in length were immersed in liquid nitrogen for 5 minutes and then kept in plastic bags. They were stored in a dry place until required for morphological investigation. The rest of the strands were cut into small pellets using a pelletizer (PRISM Co., UK). In order to minimize any possible contamination arising from the blend components remaining in the extruder, all parts of the extruder which were in contact with the material (the barrel, the screw and the die) were cleaned after every single preparation.

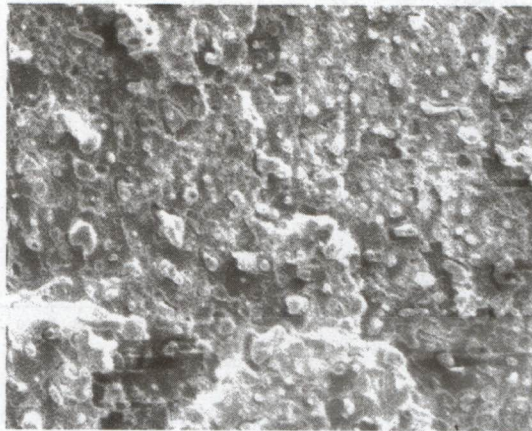
The morphology of the blends was obtained from their fracture surfaces using SEM (JEOL model JSM-5300, Japan Electric and Optic Laboratories, Co. Ltd. Tokyo). The PS can be identified as the matrix phase while the dispersed phase is LDPE using the tapping mode AFM (NanoScope IIIa MSP, Digital Instruments Co. Santa Barbara, California, USA). The LDPE droplets were identified by their lamellar structures using high-resolution images. Average droplet diameters of the blend samples were determined using OM. Pellets of blend samples were extracted using warm THF in which the PS phase was dissolved while droplets of the LDPE were suspended in THF. A drop of this solution was placed on a glass slide and the solvent was allowed to evaporate at room temperature. The glass slide was stored in an oven at room temperature for several days (normally a couple of weeks) before performing OM. This would allow the LDPE droplets to return to their original shape and size from swelling. An

optical microscope (Optiphot-2, Nikon Co. Ltd., Japan) set for phase contrast was used to determine an average phase size and size distribution of the minor phase. A video camera coupled with image frame grabber installed in a computer was mounted onto the microscope. The image analysis was performed using a commercial image analysis software. The blend morphology was characterized by its size distribution function $f(d)$, or by the corresponding cumulative distribution $F(d)$, where d is the droplet diameter. Once $f(d)$ is known, average quantities can be computed. In particular, use will be made of the number-average and volume-average diameters, d_n and d_v , defined as:

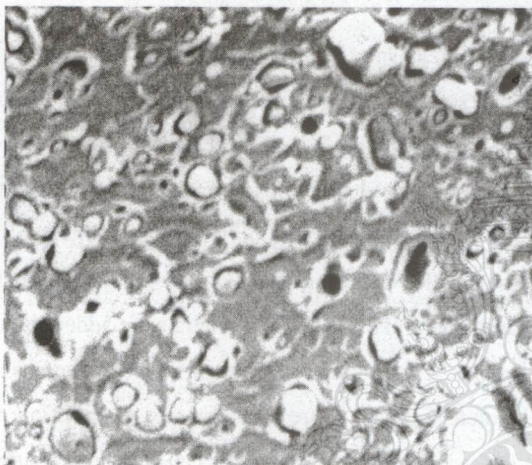
$$d_n = \frac{\sum_i f(d_i)d_i}{\sum_i f(d_i)} = \frac{\sum_i n_i d_i}{\sum_i n_i} \quad (1)$$

$$d_v = \frac{\sum_i f(d_i)V_i d_i}{\sum_i f(d_i)V_i} = \frac{\sum_i n_i d_i^4}{\sum_i n_i d_i^3} \quad (2)$$

Considering equations (1) and (2), d_i is the diameter of a generic drop and n_i is the total number of the droplets of diameter d_i . In each blend, a number of order 800 droplets were analyzed. The measurements of particle size diameter were repeated three times on several samples and it was found that d_n and d_v were reproducible to $\pm 15\%$.



PS/LDPE Blend without SEBE



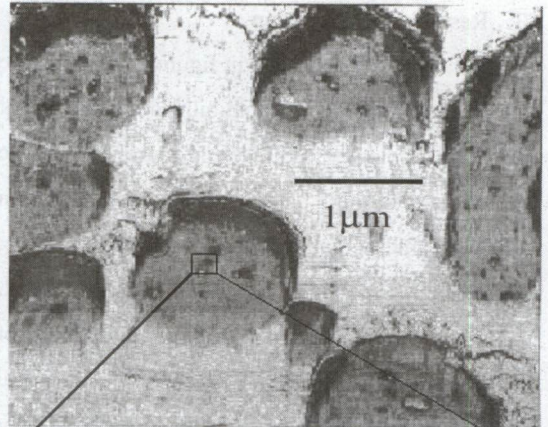
PS/LDPE Blend with SEBS(G1651)



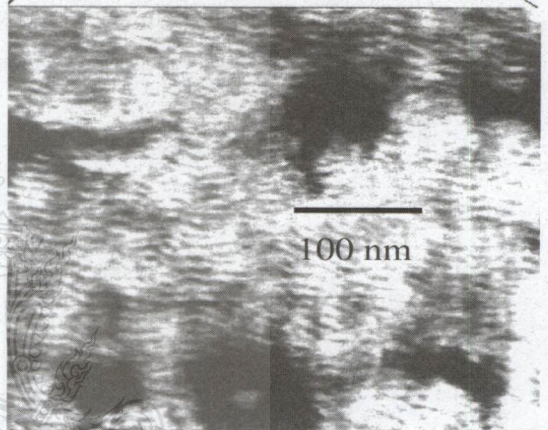
PS/LDPE Blend with SEBS(G1652)

10 μm

Figure 1 SEM micrograph for PS/LDPE (70/30 wt%) without and with SEBS(G1651) and SEBS (G1652) (5 wt% with respect to LDPE content)



1 μm



100 nm

Figure 2 Lamellar structures of the dispersed LDPE particles for the PS/LDPE (70/30 wt%) blend compatibilized with SEBS (G1651)

The impact strength of the blends was determined using the Charpy test and quoted in terms of energy absorbed per unit area of the broken cross-section. For each blend, test specimens were prepared from a molded plaque under identical condition in accordance with BS EN ISO 2818: 1994. The Charpy test was performed on a Hounsfield Plastic Impact Tester (Tensometer Ltd., Croydon, Surrey, England) in accordance with BS EN ISO 179: 1997. The machine satisfies the majority of the conditions specified in BS 4618: Section 1.2: 1972 (Impact) which lays emphasis on the multipoint tests. The number of repeat specimens used was at least ten.

3. Results and discussion

It can be clearly seen in figure 1 that both the uncompatibilized and compatibilized blends exhibit only the droplet-type dispersion morphology. The morphology of these blends was also obtained from TMAFM micrographs. It can be clearly seen that no co-continuous morphology was obtained. Although the contrast of the AFM micrograph of the uncompatibilized blends is low due to technical problems, the droplet-type dispersion morphology is still identified. By using the high-resolution image of AFM, the LDPE droplets were identified by their lamellar structure (see figure 2).

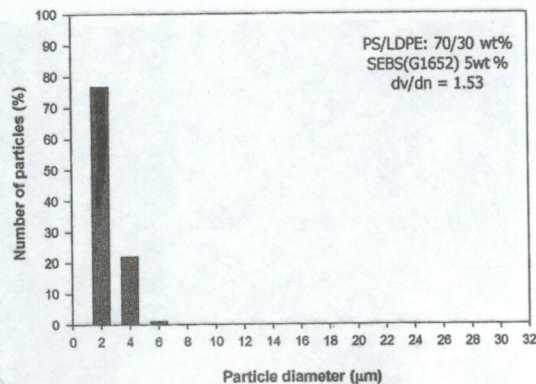
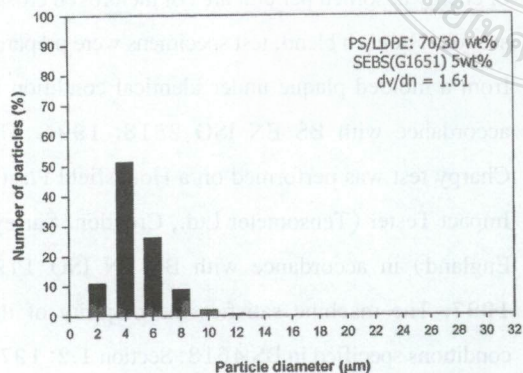
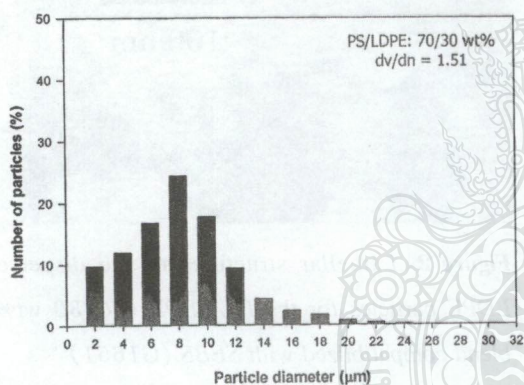


Figure 3 Frequency-distribution histograms for PS/LDPE with and without SEBS

Hemispherical embeddings and hollows representing removed particles are clearly visible on the fracture surface. The SEBS copolymers have the effect of reducing the interfacial tension between the PS and LDPE homopolymers, as indicated by the smaller dispersed phase dimensions when small amounts of the SEBS copolymers were added to the blends. By producing lower dispersed phase size dimensions and more uniform size distribution at the same concentration used as can be seen in figures 3 and 4, the SEBS(G1652) has been shown to be more effective at compatibilizing than the SEBS(G1651). This may be explained by the fact that there is more of the low molecular weight SEBS at the interface than the high molecular weight one. Since the high molecular weight SEBSs forms micelles more readily in the matrix phase than the small molecular weight ones (Char *et al.*, 1993) and together with the higher viscosity, one can expect that much of the high molecular weight SEBS does not reach the interface (Cigana *et al.*, 1997). A superior compatibilizing efficiency of the SEBS(G1652) over the SEBS G1651 has also been found in the PS/EBR blend systems reported by Cigana *et al.* (1997) and Hong and Jo (2000).

An increase in impact strength higher than the uncompatibilized blends was observed from the blends compatibilized with the SEBS(G1651) whereas this behavior was not found from those compatibilized with the SEBS(G1652) as can be seen in figure 5. The impact strength of the SEBS(G1652) compatibilized blends was even lower than that of the uncompatibilized blends. As seen in figure 5, the impact strengths of the SEBS(G1651) compatibilized blends were higher than that of the SEBS(G1652) compatibilized blends although the morphology of these blends is less fine than that of the SEBS(G1652) compatibilized blends. Regarding only the extent of anchoring of the block segment corresponding to the blend components, the SEBS(G1651) would be deeper than the case of SEBS(G1652) and this would contribute to higher impact strength. According to figure 5, the impact strength of the compatibilized blends is generally less influenced by the concentration of the block copolymer used. This indicates that a finer dispersion due to reduction of the interfacial tension (emulsifying effect) promoted by a block copolymer does not always lead to an improvement in the mechanical properties, i.e. the impact strength.

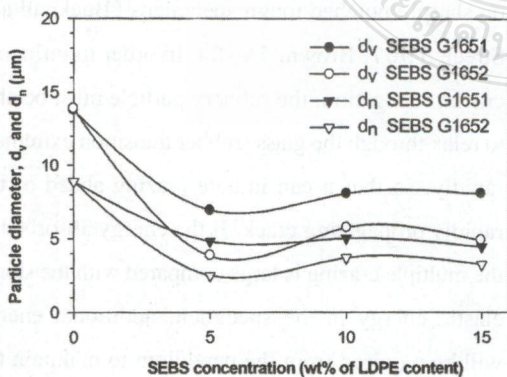


Figure 4 Emulsification curve for SEBS compatibilized PS/LDPE blends (70/30wt%).

Table 2 Properties of a typical PS and HIPS (ASTM test methods D 638-61 T, D 256-56 and D 648-56) (from Bucknall, 1977).

Properties	PS	HIPS
Tensile modulus (GPa)	3.5	1.6
Tensile strength at yield (MPa)	Does not yield	17.5
Tensile elongation at yield (%)	Does not yield	2
Tensile strength at break (MPa)	54	21
Tensile elongation at break (%)	2.1	40
Notched Izod impact strength (J/cm)	1.0	4.5

It was clear that the compatibilized blends have higher impact strengths than those of the uncompatibilized blends. However, the degree of improvement in this property is marginal as the highest impact strength obtained in the SEBS(G1651) compatibilized blends was still only about 40% higher than that of the uncompatibilized blends. Note that, the Izod impact strength of a HIPS is four and a half times higher than that of a PS (see Table 2).

A typical emulsification curve for these blends is shown in figure 4. It is interesting to note that the shape of the curves shown in figure 4 is similar to that of a classical emulsion. A rapid drop in particle size diameter especially d_v with increasing concentration of the SEBS was observed. These data are in agreement with the work of Matos and Favis (1995). It appears that 5wt% of both the SEBS (G1651) and the SEBS(G1652) added produces a maximum reduction of the LDPE phase size whereas no further reduction of the particle size is found from addition of 10 and 15wt%. Therefore, it could imply that a critical concentration for compatibilizing the PS/LDPE blend studies with the SEBS triblock copolymers is approximately 5wt%.

Since the critical concentration for the block copolymers to be saturated at the interface of PS/LDPE is about 5wt% of LDPE content, the rest of the block copolymers dispersed in the PS matrix phase is expected to also play a role in increasing the impact strength of the blends. These results indicated that the significant improvement in the impact strength in the HIPS is not solely a result of the strong particle-matrix adhesion. The physical and mechanical properties, and the morphology of the rubbery phase have also been recognized as important factors which are responsible for the considerably higher impact strength of HIPS.

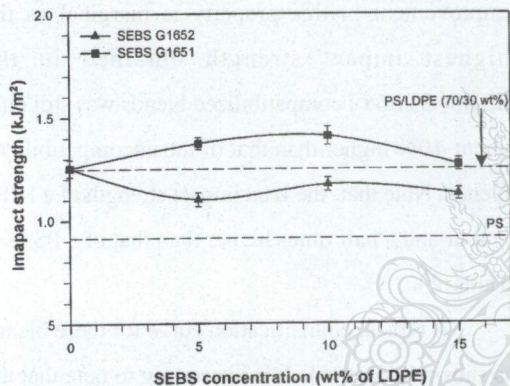


Figure 5 Impact strength versus SEBS concentration for compatibilized PS/LDPE blends (70/30wt%).

Tests on sharply notched specimens place a greater emphasis on the material's resistance to crack propagation. In a thick sharply notched specimen, the stresses at the root of the notch are triaxial. The reason for the state of triaxial tension is that the material in the tip of the notch is trying to contract in the plane perpendicular to the applied stress, in order to maintain constant volume, but is unable to do so because it is constrained by the surrounding material. The resulting change in the form of the stress tensor from uniaxial or biaxial tension to triaxial tension is sufficient to raise the yield stress above the crazing stress, with consequent reduction

in fracture resistance. Note that under impact loading, the sharp notch not only creates a locally highly triaxial stress concentration but also increasing the rate of strain at the notch tip. The high strain rate has a similar effect to low temperature (Williams, 1987). Therefore, the material normally reaches the critical tensile strain for craze initiation before the shear strain energy density is sufficient to cause shear yielding (Bucknall, 1977) as found in PS. As mentioned in section 1, multiple crazing is of importance in toughening PS.

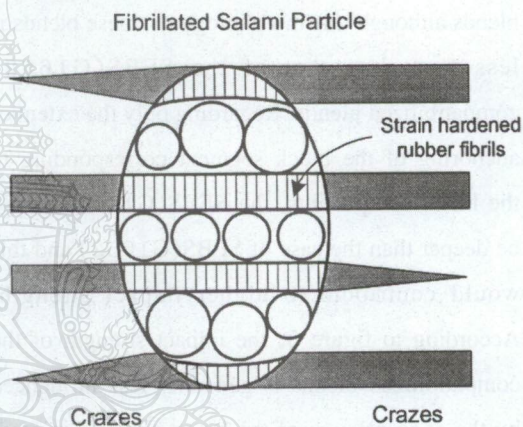


Figure 6 Schematic diagram showing mechanisms of deformation in HIPS (from O'Connor et al., 1997).

It was found experimentally that impact energy is controlled by crack propagation behavior, in tests on sharply notched tough specimens (Bucknall and Street, 1967; Brown, 1973). In order to influence crack propagation, the rubbery particle must be able to relax through the glass-rubber transition extremely rapidly, so that it can initiate crazing ahead of the rapidly propagating crack. If the energy absorbed in the multiple crazing is large compared with the stored elastic energy in the specimen, additional energy will be required from the pendulum to maintain the propagation, and the strength will consequently increase. However, in HIPS, it was found that there is extensive craze-like fibrillation of the rubber

membranes within the salami-like particles as illustrated in figure 6. Fibrillation occurring in the rubber particles indicates that the salami-like rubber particles in HIPS act not only as stress concentrators initiating crazes but also as load-bearing units. Load sharing between the rubber particles and the craze fibrils in the matrix can allow large strains (over 50%) to be reached (Bucknall, 2000). Bubeck *et al.* (1991) have shown that crazing occurred after the rubber particles started to cavitate, as well as quantifying the relative contributions of energy dissipated by each mechanism. The authors has also shown that crazing is responsible for less than 50% of dilatational strain developed during tensile impact testing of HIPS. Fibrillated rubber particles act to stabilize the crazes that they have initiated. In order to allow almost every rubber molecule in a cavitated particle to eventually contribute to craze stabilization, there must be as uniform as possible stresses applied within the cavitated particle. These conditions would be obtained only in a larger particle containing rigid inclusions as found in the salami-like particles in a HIPS. Therefore, it can be expected that an LDPE particle would have less ability to stabilize a craze even if it could cavitate. Morphology of rubber particles is one of the important parameters which are responsible for the considerably high impact strength of a HIPS.

Table 3 Typical values of Young’s modulus; shear modulus, thermal (volumetric) expansion coefficient, and glass transition temperature for PB, LDPE and PS.

(from Brydson, 1981).

Properties	PB	LDPE	PS
Young’s modulus (MPa)	14 - 17	102 - 310	3,200 - 3,400
Shear modulus (MPa)	2 - 10	40 - 120	1,200
Thermal expansion coefficient (K ⁻¹)	~7.5x10 ⁻⁴	~3.2x10 ⁻⁴	1.7 x10 ⁻⁴ - 2.1x10 ⁻⁴
Glass transition temperature (K)	~172	~150	~373

Since PB has relatively low shear modulus, as can be seen in table 3, when dispersed in PS, a PB particle is much better than LDPE not only in term of ability to generate stress concentrators but also to cavitate for the reasons given in section 1. Note that, as seen in table 7.2, the thermal expansion coefficient of PS is only about one-third that of PB. Thus, cooling a HIPS from the melt, which can be considered stress-free, produces stresses in the rubber particle and in the surrounding matrix as a result of differential thermal contraction. The large triaxial tensile stresses generated in the rubber particle cause a shift in the α -transition temperature T_{α} (corresponding to the glass transition temperature T_g) of the rubber particle to lower temperatures due to a decrease in density which reduces relaxation times. In other words, the PB particle will be able to relax rapidly because of the increased free volume due to constraints on thermal contraction. Together with relatively low shear modulus, the PB particle is likely to cavitate when a HIPS is subjected to mechanical loading in order to release stresses (Bucknall *et al.*, 2000). By contrast, cavitation is unlikely to occur in a LDPE particle owing to its relatively high shear modulus and little difference in the thermal expansion coefficient between the PS and LDPE. Furthermore, owing to its crystalline phases, LDPE is much stiffer than PB although the value of T_g of LDPE is of the same order to that of PB. Thus, the ability to respond to very high fracture crack speeds by relaxation during impact loading of the LDPE particle is expected to be lower than that of the PB particle. Together, these factors provide the reason why LDPE provided only little improvements in toughness of PS.

4. Conclusions

The uncompatibilized PS/LDPE blend prepared provided better (though small) improvements in impact strength. However, the degree of improvement in impact properties in the uncompatibilized blends was considerably lower than that of commercial high-impact polystyrene. Addition of the SEBS block copolymer did not provide much improvement over the uncompatibilized blends (70/30wt% PS/LDPE) although improvements in interfacial properties such as finer dispersion of the dispersed phase were observed. These results confirm that improvement in toughness of glassy polymers which are prone to crazing does not depend solely on the interfacial properties of the phases but are strongly affected by the properties of the included particles themselves.

Acknowledgements

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