



# Effects of reactive compatibilizer on the core–shell structured modifiers toughening of poly(trimethylene terephthalate)

Kunyan Wang, Yanmo Chen\*, Yu Zhang

State Key Laboratory for Modification of Chemical Fibers and Polymer Materials, College of Material Science and Engineering, Donghua University, Shanghai 201620, PR China

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## ABSTRACT

Poly(trimethylene terephthalate)/polybutadiene grafted polymethyl methacrylate (PB-g-PMMA, MB) blends were prepared by melt processing with varying weight ratios (0–5 wt%) of diglycidyl ether of bisphenol-A (DGEBA) epoxy resin as a reactive compatibilizer. DMA result showed PTT was partially miscible with MB particles in the presence of the compatibilizer. Fourier transform infrared (FTIR) and rheological measurements further identified the reactions between PTT and DGEBA epoxy resin. Scanning electron microscopy (SEM) displayed that the core–shell structured modifiers exhibit a smaller dispersed domain size with the addition of DGEBA epoxy resin. Mechanical tests showed the impact and tensile properties of PTT blends are improved by the introduction of DGEBA epoxy resin to the blends. SEM and TEM results showed shear yielding of PTT matrix and cavitation of rubber particles were the major toughening mechanisms.

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## 1. Introduction

Poly(trimethylene terephthalate) (PTT) is one member of the terephthalic polyesters, the most common substances used in textile industry. More recently, PTTs have drawn attention for their applications in engineering thermoplastic due to a great reduction in the manufacturing cost of 1,3-propanediol, the monomer used for PTT synthesis [1,2]. PTT is a tough and ductile material in the unnotched state. The presence of sharp notches or curvature in the material leads to failure in a brittle manner. The notched impact strength of PTT can be greatly enhanced by the incorporation of rubber filler [3–5]. However, this toughening technique often requires a substantial concentration in filler of the order of 10–20 wt% which, in turn, implies a significant loss in elastic modulus. Using core–shell impact modifiers instead of pure rubber is an interesting way to solve this issue [6–10].

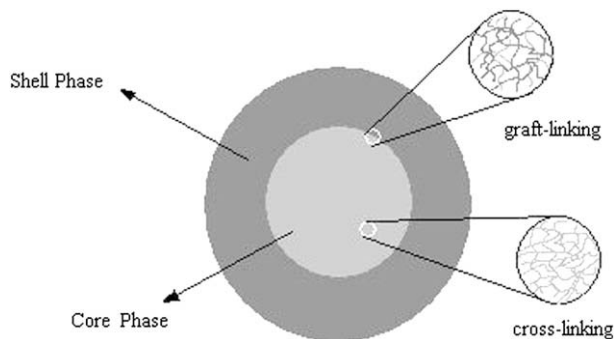
Core–shell modifiers were firstly commercially introduced as PVC impact modified in 1958 [11]. The particle size of the core–shell modifier, which is set during the synthesis process, can remain after they are dispersed in a host matrix. Core–shell modifiers are now widely used in variety of polymers, such as PC [12], PMMA [13], PBT [14] and PVC [15]. MB is a typical core–shell rubber modifier as described in Scheme 1, in which methyl methacrylate is graft

polymerized onto polybutadiene (PB) particles. The addition of MB can improve the impact resistance of polymer without sacrifice its modulus.

It is generally believed that the interfacial adhesion between the dispersed rubber particles and the matrix plays an important role in the toughening of polymers. The effect of interfacial adhesion on the impact strength has long been of great interest. In order to increase interfacial adhesion and improve the miscibility of polymer blends between matrix and disperse phase, the method of reactive compatibilization is very often used to obtain blends with desirable properties [16,17]. Since most polymer blends do not have the appropriate functional group, functionalization of the components is very often required. In some cases, it is possible to add a third polymer into the blend, which is miscible with one of the blend components and reactive with the other blend component [18,19].

It is reported that PMMA shell of MB is miscible with the DGEBA epoxy resin which is due to the specific interaction (hydrogen bonding) formed between this two polymers [20]. Since the epoxy group can react with hydroxyl and carboxyl group, as a reactive compatibilizer, DGEBA epoxy resin might react with PTT through reacting with the functional groups. So, in this work, DGEBA epoxy resin as a reactive compatibilizer was added to the PTT/MB blends by melt mixing. DMA was used to study the miscibility of PTT/MB blends with varying weight ratios (0–5 wt%) of DGEBA epoxy resin. The effect of DGEBA epoxy resin content on the morphological and mechanical properties of PTT blends was studied. SEM and TEM

\* Corresponding author. Tel.: +86 21 67792857; fax: +86 21 67792855.  
E-mail address: [yanmochen@tom.com](mailto:yanmochen@tom.com) (Y. Chen).



**Scheme 1.** Diagram of the typical polybutadiene grafted polymethyl methacrylate (PB-g-PMMA, MB) core-shell modifier particle.

were used to observe the fracture morphology and the structure inside the deformed zone of the toughened PTT, and the toughening mechanisms were then proposed.

## 2. Experimental section

### 2.1. Materials

PTT was purchased from the DuPont Company (USA) with  $T_m = 225.6^\circ\text{C}$ . Intrinsic viscosity of the PTT chip is 0.935 dl/g and the number average molecular weight is 38,000. The core-shell structured grafted copolymer particles of polybutadiene grafted polymethyl methacrylate (PB-g-PMMA, MB) were prepared by emulsion polymerization [21]. The weight average molecular weight of MB is 320,000. The epoxy resin used was E44 resin, which is diglycidyl ether of bisphenol-A (DGEBA) epoxy resin (Shanghai Epoxy Co. Ltd, equivalent epoxy weight:  $227 \pm 5$ ).

### 2.2. Specimens preparation

One blend composites 90/10 (W/W) PTT/MB with various amounts of the DGEBA epoxy resin (0–5 wt%) were prepared by melt mixing in a Haake Rheocorder having a capacity of about  $50\text{ cm}^3$  at  $235^\circ\text{C}$  and 100 rpm for 8 min. Before melt mixing, all polymers were completely dried in a vacuum oven at  $80^\circ\text{C}$  for 12 h. The ternary polymer composites will be named by their DGEBA epoxy resin content; i.e. PMD1 indicates a 90/10/1 PTT/MB/DGEBA blend, where P stands for PTT, M stands for MB, and D stands for DGEBA.

### 2.3. Characterizations

Dynamic mechanical analysis (DMA) was performed on the samples of  $18 \times 7 \times 1\text{ mm}^3$  in size using a dynamic mechanical analyzer from TA Instruments Q800 under single cantilever mode in a temperature range from  $-120^\circ\text{C}$  to  $150^\circ\text{C}$  at a constant heating rate of  $3^\circ\text{C}/\text{min}$ , and at a frequency of 10 Hz.

The specimens were cryogenically fractured in liquid nitrogen, and then were etched in boiling toluene for 1 h to selectively dissolve the core-shell structured modifiers. The PTT/MB blend was etched in toluene for 30 min at  $75^\circ\text{C}$ . And the phase morphology was observed in a JSM-5600LV SEM instrument operating at an accelerating voltage of 10 kV after the surface was coated with gold powder.

The internal damaged zone of blends was examined on SEM and transmission electron microscopy (TEM H-800) using an acceleration voltage of 100 kV. The samples were ultrathin-sectioned at 60–80 nm with a Reicher ultracut cryo microtome.

Ultramicrotomed samples were exposed to osmium tetroxide ( $\text{OsO}_4$ ) and stained for 20 min so that the MB particles appear to be dark in TEM pictures.

Fourier transform infrared (FTIR) spectroscopy has been used to monitor the chemical reactions between PTT and DGEBA epoxy resin. For the infrared measurement a small portion of the samples was grind to a fine powder, mixed with potassium bromide (KBr) powder and pressed into a pellet by hand press. FTIR were recorded by Nicolet NEXUS-670 IR spectrometer in the wavelength range of  $4000\text{--}400\text{ cm}^{-1}$ .

The torque measurements of PTT blends were performed on a Thermo Haake mixer. The rotating speed was set at 50 rpm and the temperature was set at  $240^\circ\text{C}$ .

The tensile tests were performed in a universal testing machine according to ASTM D-638. The dog-bone-shaped specimens with gauge lengths of 7.62 mm were prepared by micro-injector made in USA cross-head speed was set up at 10 mm/min. Young's modulus was calculated by linear regression of the stress data versus strain data from the initial strain to the data before the maximum strain. For each sample, the data reported are the average of five to seven specimens.

The impact experiment was carried out on an RESIL impact tester according to ASTM D-256. The notches (depth 2.54 mm and radius 0.25 mm) were machined after injection molding. A minimum of ten impact specimens were tested for each reported value.

The relative volume strain ( $\Delta V/V$ ) is calculated from

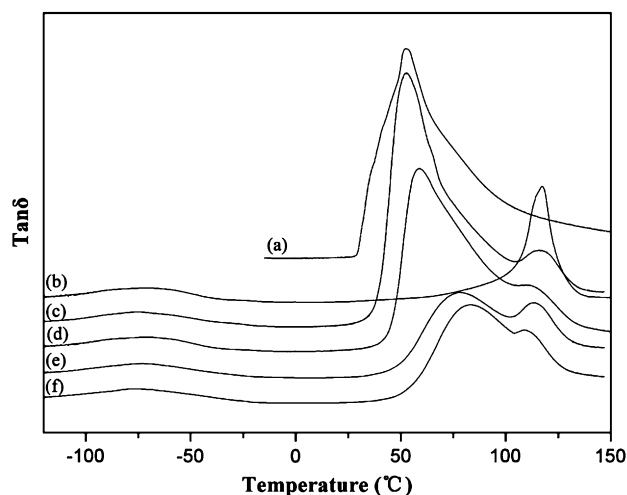
$$\Delta V/V = (\rho_b - \rho_w)/\rho_w$$

where  $\rho_b$  is the density of the blend outside the deformed zone;  $\rho_w$  is the density of the same blend in the deformed zone. These density values were measured by using a gradient column filled with NaBr aqueous solution.

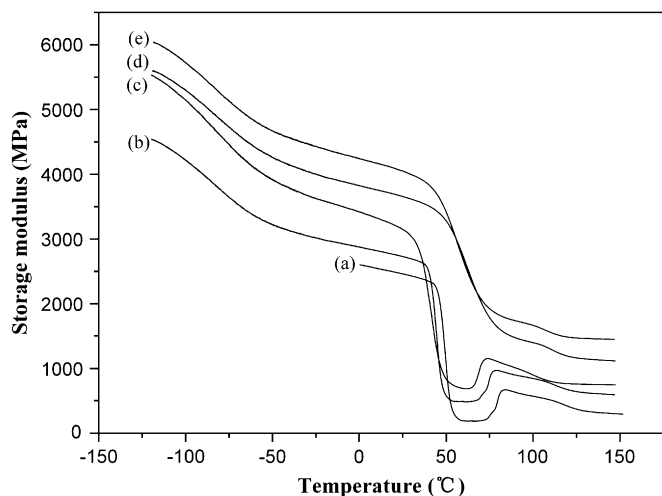
## 3. Results and discussion

### 3.1. Dynamic mechanical properties of composites

The miscibility property between PTT/MB blends with and without DGEBA epoxy resin could be studied by DMA. As can be seen in Fig. 1, the peak at  $50.7^\circ\text{C}$  is the  $T_g$  of PTT amorphous phase, and the pure MB exhibits a  $\tan \delta$  peak at  $117^\circ\text{C}$  due to the glass



**Fig. 1.** Temperature dependence of  $\tan \delta$  for (a) pure PTT and 90/10 (w/w) PTT/MB with various amounts of the DGEBA. The amount of the DGEBA (wt%) is (b) 0; (c) 1; (d) 3 and (e) 5.



**Fig. 2.** Temperature dependence of storage modulus for (a) pure PTT and 90/10 (w/w) PTT/MB with various amounts of the DGEBA epoxy resin. The amount of the DGEBA epoxy resin (wt%) is (b) 0; (c) 1; (d) 3 and (e) 5.

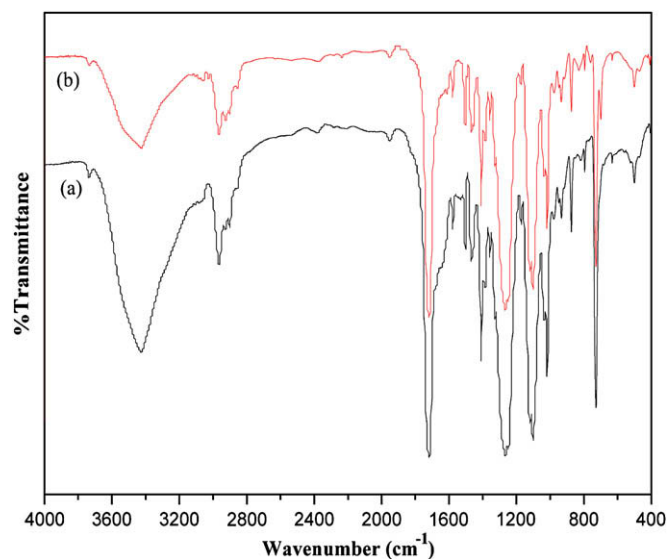
transition of the PMMA shell phase in MB and a peak at  $-72^{\circ}\text{C}$  due to the  $T_g$  of the PB phase. Compared with  $T_g$  of pure PTT and MB, the  $T_g$  of PTT shift to high temperature and the  $T_g$  of PMMA phase shift to low temperature for PTT/MB blends with DGEBA epoxy resin. For the PTT/MB blend without DGEBA epoxy resin, the peaks of both PTT and DGEBA are almost not changeable. As we know [22], if the blend displays two  $T_g$ , at or near the two components, then it is immiscible. On the other hand, if it shows a single transition or two transitions at temperature intermediate between those of the pure components, then the blend is miscible or partially miscible. So Fig. 1 shows PTT is immiscible with MB, but partially miscible with MB when DGEBA epoxy resin was added to the blends.

The storage modulus versus temperature curves are shown in Fig. 2. These results show that the storage modulus of PTT increased upon addition of MB and also increased with increasing DGEBA epoxy resin content. As the temperature increases, there is a sharp decline in the modulus for PTT, PMD, and PMD1 corresponding to the glass transition temperature of the PTT. Following this the modulus then increases with temperature as the more mobile molecules may reorganize and crystallize. It is believed that crystallization has the largest contribution to the increase in modulus. For PMD3 and PMD5 with the DGEBA epoxy resin content increased, it is not found this phenomenon. This result indicates that after addition of more DGEBA epoxy resin, the molecular mobility of the composites decreased and mechanical loss to overcome inter-friction between molecular chains reduced, which might be attributed to the interaction occurred between DGEBA epoxy resin and PTT.

Soh [20] studied the miscibility of PMMA shell of MB with DGEBA epoxy resin. The result is that PMMA is miscible with the DGEBA epoxy resin which is due to the specific interaction (hydrogen bonding) was formed between these two polymers. Since the epoxy group can react with hydroxyl and carboxyl group, as a reactive compatibilizer, DGEBA epoxy resin might react with PTT through reacting with the functional groups.

### 3.2. Fourier transform infrared spectroscopic analysis

In order to investigate whether DGEBA epoxy resin reacts with PTT, we first considered using FTIR to analyze. Fig. 3 showed the FTIR spectrum of 90/10 (w/w) PTT/MB blends with and without 5 wt% DGEBA epoxy resin,  $3423\text{ cm}^{-1}$  stretching band representing



**Fig. 3.** Infrared spectrums of the blends of (a) PM and (b) PMD5.

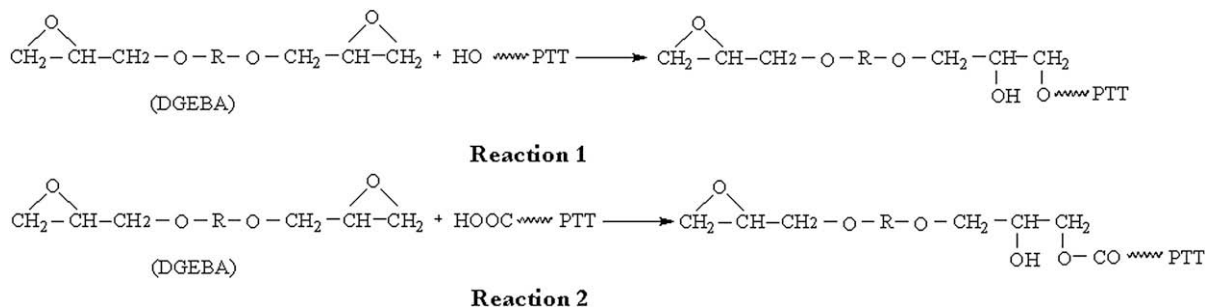
the hydroxyl band of PTT was observed in PTT/MB blend. By comparison, it is seen that there is no shifting of peak positions related to hydroxyl group of PTT ( $3423\text{ cm}^{-1}$ ) in PTT/MB blend with 5 wt% DGEBA epoxy resin. As 5 wt% DGEBA epoxy resin was added the blend sample, the peak intensity of hydroxyl group decreased which might suggested that there was certain number of hydroxyl groups of the PTT might react with epoxy group of DGEBA epoxy resin. For carboxyl band of PTT ( $1719\text{ cm}^{-1}$ ), the two blend of this peak almost has the same position and slightly change for intensity. So it might be considered that the reaction between carboxyl group of PTT and epoxy group of DGEBA epoxy resin was very weak.

Basis on FTIR analysis, two types of reactions are identified and shown to take place simultaneously during the active blending of PTT and DGEBA epoxy resin (Scheme 2). Some papers have reported on similar reactions involving the epoxy functional groups, namely for rubber modified PBT and PET [23–25]. In a recent publication [26], Martin reported on an analytical study of the compatibilization and cross-linking reactions between epoxy group and PBT in the PBT/E-GMA blends, and some of their study can be used to explain our experimental results.

Reactions 1 and 2 belong to compatibilization reactions that involve reactions between epoxy groups of DGEBA epoxy resin. Reactions 1 and 2 postulate the formation of PTT-co-DGEBA epoxy resin copolymers at the blend interface. PTT-DGEBA epoxy resin copolymer, acting as compatibilizer, can increase interfacial strengths. On the other hand, according to Soh's report as mentioned above, the specific interaction (hydrogen bonding) was formed between PMMA and DGEBA epoxy resin. So, the miscibility between the PTT and impact modifier MB could be enhanced.

### 3.3. Rheological properties

The reaction between polymers might be not clarified adequately by FTIR due to the data from FTIR was influenced by some factors (such as water). Rheological measurement was also used to obtain qualitative information concerning the chemical reactivity during blend processing. Compared to pure polymer and the blend formed without any reaction, chemical reaction between reactive components would lead to increasing the blend viscosity. The torque value is related to the viscosity of the blend. Fig. 4 illustrates the evolution of the torque as a function of the mixing



Scheme 2. Reaction between PTT and DGEBA epoxy resin at the blend interface.

time for PTT/MB and PTT/MB blends with DGEBA epoxy resin. Compared to PTT/MB blends with DGEBA epoxy resin, PTT/MB has the lowest torque value since there is no chemical reaction between PTT and MB. For the PTT/MB blends with DGEBA epoxy resin, with the increase in DGEBA epoxy resin content the torque value of the blends increased too, which further identified the reaction between the epoxy groups of DGEBA epoxy resin and the end functional groups of PTT.

### 3.4. Morphological properties

To understand the change of mechanical properties, particularly, the toughening mechanism in these composites, it is necessary to ascertain dispersion and phase morphology of rubber particles in PTT matrix. Fig. 5 presents the morphology of PTT/MB blend. The sample was etched in toluene for 40 min at 75 °C. It is observed that the MB phase is not completely removed. Compared to PMMA shell of MB, PB core of MB is easily dissolved by toluene. Most PB core of MB particles was removed by toluene, leaving PMMA shell on the surface of the PTT matrix. The MB particles show the typical core-shell structure. It is also observed that when the MB is mixed with PTT without DGEBA epoxy resin; a poor dispersion of MB particles is obtained. Some MB cluster together, which will influence the mechanical properties of PTT/MB blend.

Fig. 6 shows the SEM images of PTT/MB blends with various amounts of the DGEBA epoxy resin. All samples were etched with boiling toluene for 1 h to selectively dissolve the MB phase. The holes left on the surface of the PTT matrix reflect the morphology of

the dispersed phase. It is clear that the average diameters of these particles decrease with increasing content of DGEBA epoxy resin. The presence of DGEBA epoxy resin increases the interface adhesion between PTT and MB phase.

### 3.5. Mechanical properties

Generally, polymer blend composites with selectively located filler show poor mechanical properties and low ductility because of the weak interface between the two phases [27–29]. Fig. 7 exhibits a comparison of the stress–strain curves for pure PTT and the PTT/MB blend. At least five samples were used for each measurement. Only reproducible results are given in Fig. 7 for the comparison. A pronounced yield and postyield drop are observed for pure PTT and PTT/MB blend. Pure PTT and PTT/MB almost have the same modulus. The pure PTT possesses a tensile strength of about 48 MPa and an elongation at break of 242%. The overall effect is a decrease in the value of tensile strength and elongation at break when added MB to PTT. The poor interfacial adhesion between PTT and MB might be an important factor for this result.

The tensile properties of PTT/MB blends with different content of DGEBA epoxy resin are shown in Fig. 8. It is seen that the tensile strength, modulus and elongation at break are improved by the introduction of DGEBA epoxy resin and with the increase in DGEBA epoxy resin content the tensile properties of PTT/MB blend become much better. It is reported that the tensile properties of polymer blends are very sensitive to the state of the interface [30–32], that is, interfacial adhesion. According to Section 3.2 as mentioned

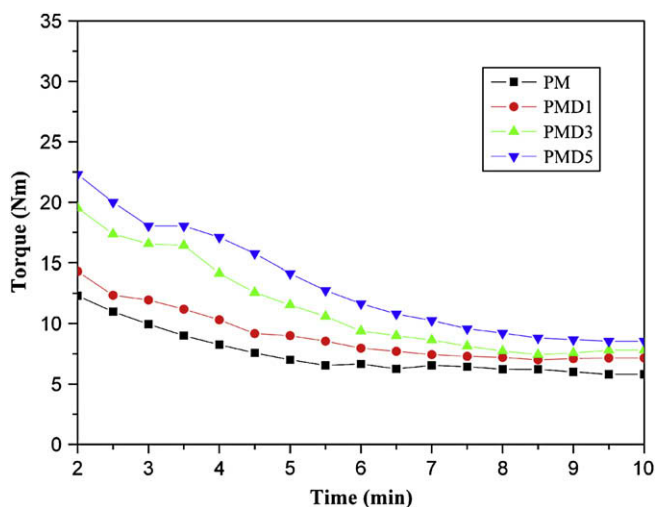


Fig. 4. Evolution of torque with time in PTT/MB and PTT/MB blends with DGEBA epoxy resin.

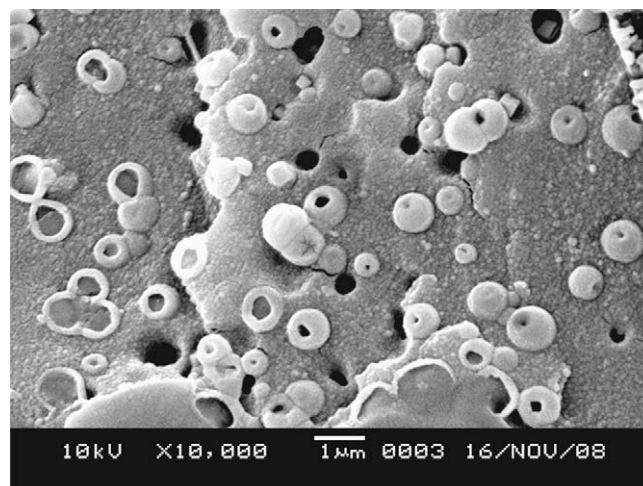
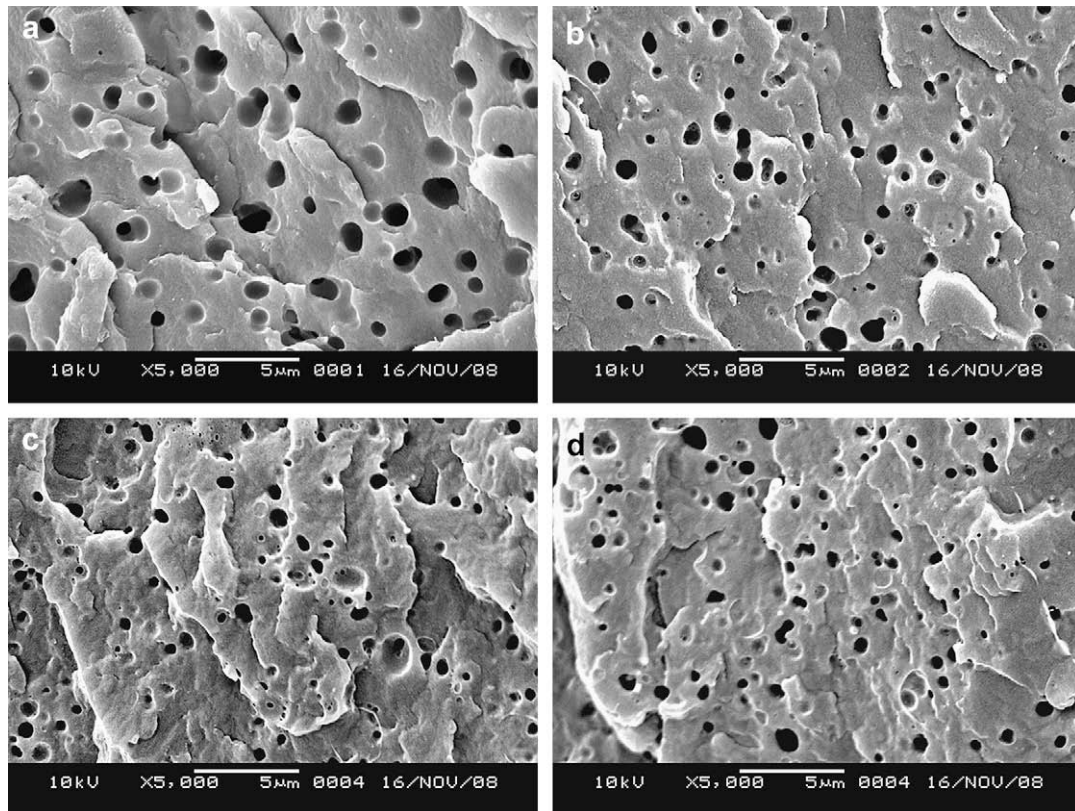


Fig. 5. Morphology of PTT/MB blend. The sample was etched in toluene for 40 min at 75 °C.



**Fig. 6.** Morphology of PTT/MB blends with and without DGEBA epoxy resin. The amount of the DGEBA epoxy resin (wt%) is (a) 0; (b) 1; (c) 3 and (d) 5. All samples were etched with in boiling toluene for 1 h.

above, the reactions could happen between PTT and DGEBA epoxy resin at the interface in Scheme 1; on the other hand, the specific interaction (hydrogen bonding) was formed between PMMA shell of MB and DGEBA epoxy resin. So, the marked improvement in the tensile properties of the composites is believed to be related to the improved interfacial adhesion between the PTT and impact modifier MB induced by DGEBA epoxy resin.

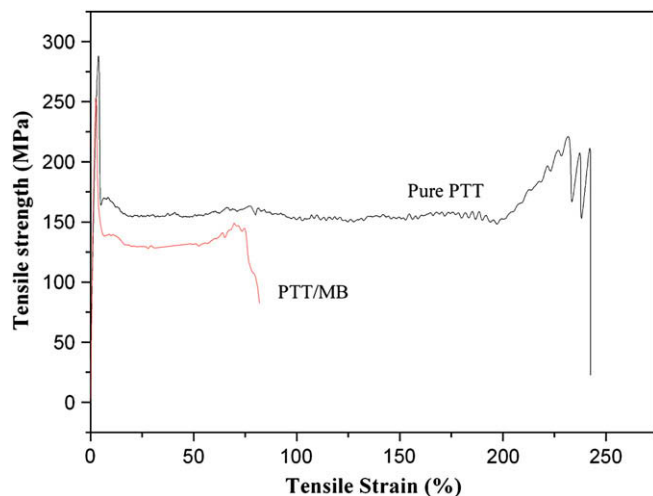
The impact strength of a material describes the energy required to break the specimen. The magnitude of impact strength reflects the ability of material to resist impact. Notch Izod impact strength

emphasizes the energy to propagate a crack under impact load. Impact strength of polymeric composites is complex because of the role of the filler and the filler/matrix interface in addition to the polymer [33]. The impact strength of PTT/MB blends with different DGEBA epoxy resin contents is shown in Fig. 9. Notched impact strength of pure PTT is about 42 J/m, while the PTT/MB blend possesses notched impact strength of about 45 J/m, no obviously increasing which is attributed to poor interfacial adhesion between PTT and MB. However, the toughness of PTT is improved by the introduction of DGEBA epoxy resin to the blends. Because of the DGEBA epoxy resin acted as compatibilizer, the MB can disperse in PTT matrix uniformly and the particle size of MB decreases. Wu [34] has suggested that the toughness of rubber modified thermoplastics increased as the ligament size is reduced. The interparticle distance or ligament size can be reduced either by increasing the rubber concentration or by decreasing the rubber particle size. The increase in the impact strength at 5 wt% DGEBA epoxy resin content could be attributed to the particle size and interparticles distance.

It should be noted that as the modified PTT maintains quite high tensile modulus and tensile strength. The impact strength for the PTT/MB blend with 5 wt% DGEBA epoxy resin is 112 J/m, which is about 167% higher than the value for pure PTT. The results are very significant in obtaining a material having remarkable stiffness–toughness balance.

### 3.6. Fracture mechanism

In toughened thermoplastics, the toughening mechanism is characterized by the fracture surface and stress-whitening zone of the sample, according to the impact condition. In our work, therefore, SEM was used to observe the fracture surface and stress-



**Fig. 7.** Tensile behavior of PTT and PTT/MB blends.

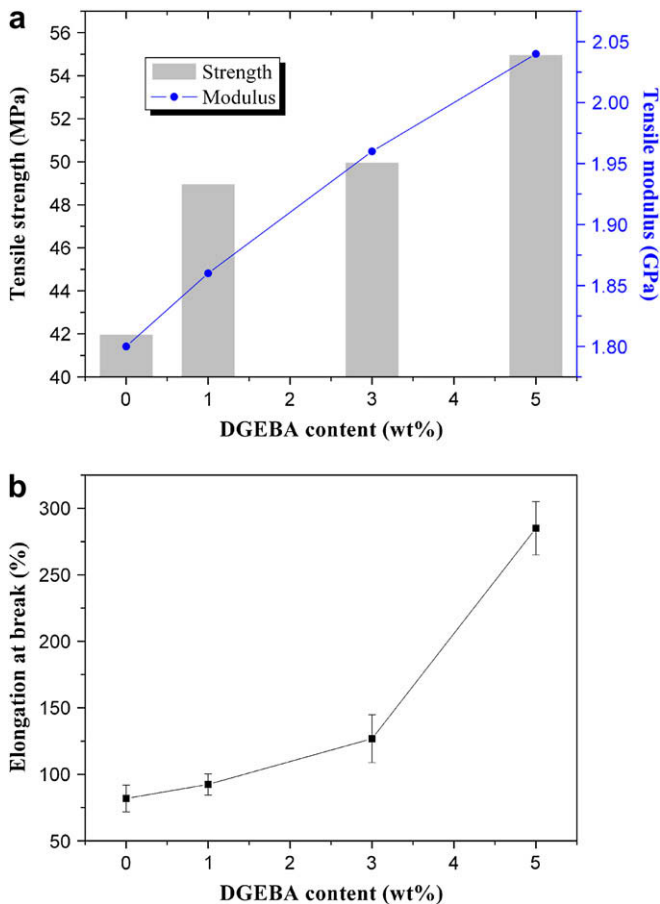


Fig. 8. Tensile properties of PTT/MB blends with different DGEBA epoxy resin content. (a) Tensile strength and tensile modulus. (b) Elongation at break.

whitening zone of a representative sample (PTT/MB blend with 5 wt% DGEBA epoxy resin content) in the notched impact strength test.

Fig. 10 shows the fractured surface of PTT/MB blend with 5 wt% DGEBA epoxy resin content, which was formed during Izod impact tests at room temperature. As expected, the fracture surface of this blend shows the characteristics of ductile fracture. A large number of fibrils and voids are observed on the Izod impact fractured

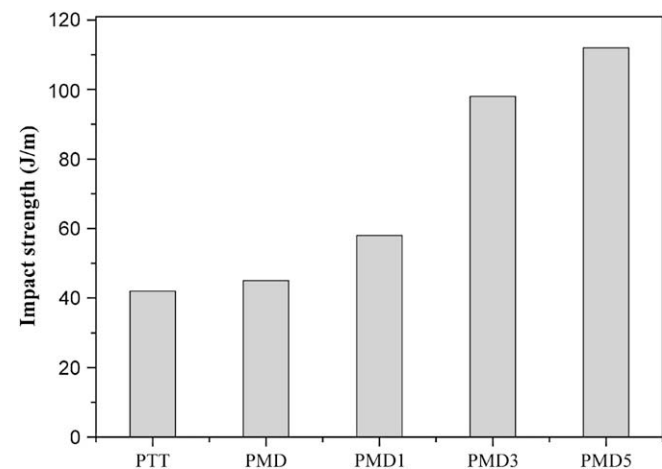


Fig. 9. Impact strength of PTT/MB blends with different DGEBA epoxy resin content.

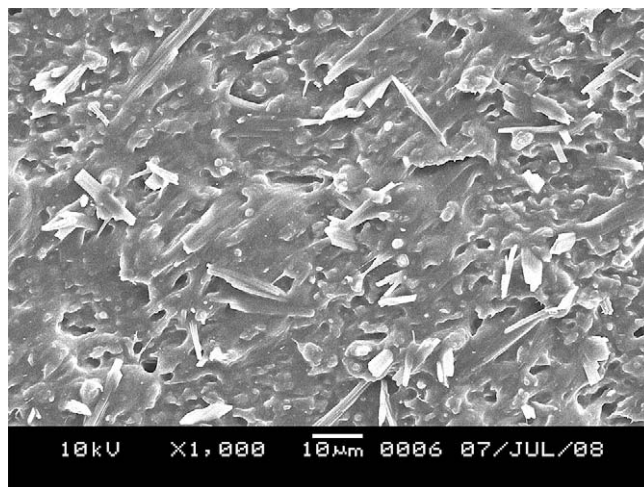


Fig. 10. SEM micrographs of the Izod impact fractured surfaces for PTT/MB blend with 5 wt% DGEBA epoxy resin content.

surface. This type of extensive plastic deformation implies that shear yielding of the PTT matrix has taken place.

In order to make sure that voids were formed within the deformed zone. We measured the volume strain  $\Delta V/V$  for the PTT/MB blend with 5 wt% DGEBA epoxy resin within the deformation region. The volume strain  $\Delta V/V$  for the PTT/MB blend with 5 wt% DGEBA epoxy resin is 0.052, indicating that there are voids in this sample. Likewise, Liu et al. [35] reported that there are voids at the PVC–NBR interface in tough PVC–NBR blends.

The preparation and observation locations of samples used for examinations of deformation mechanisms are illustrated in Fig. 11. PTT/MB blend with 5 wt% DGEBA epoxy resin content was still chosen for SEM analysis. The dotted areas denote the deformation region in the PTT/MB blend with 5 wt% DGEBA epoxy resin content. A sharp notch of about 0.5 mm in depth was made on the sample by using a razor blade after an Izod impact test. The sample having the sharp notch was cooled in liquid nitrogen, and then quickly fractured. The cryo-fractured surface was coated with gold for SEM observations.

Fig. 12 shows the SEM observations on the cryo-fractured surface of the blend. A–D is located along the symmetrical axis of the cryo-fractured surface. Fig. 12(a) was taken at location A, which is far away from the deformation region. A few holes are seen, which indicated that the MB particles are well bonded to the PTT matrix and are not easily detached during the cryo-fracture process. Location B is close to but inside the deformation region. Shear yielding of matrix is not obvious, and only some holes of rubber particles can be seen. Locations C and D are in the deformation region and about 100  $\mu\text{m}$  and 50  $\mu\text{m}$  away from the Izod

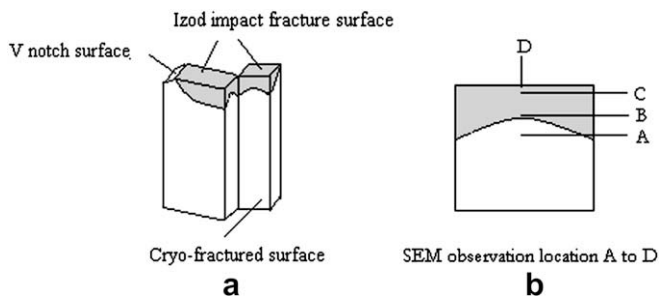


Fig. 11. Schematic illustration of the preparation of samples used for examination of deformation mechanisms in PTT/MB blends with 5 wt% DGEBA epoxy resin content by SEM. The dotted areas represent the deformation region.

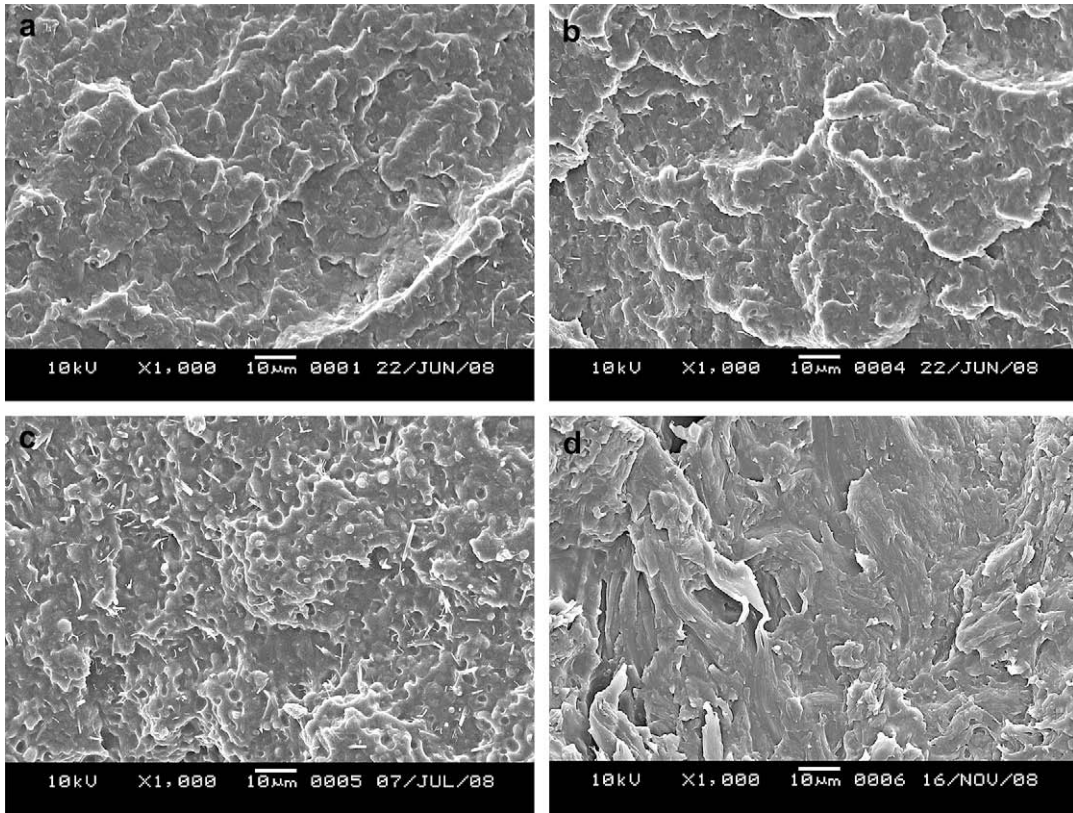


Fig. 12. SEM micrographs of the cryo-fractured surface of PTT/MB blend with 5 wt% DGEBA content. The location of the observed surface is illustrated in Fig. 11.

impact fractured surface, respectively. A great number of holes are seen in Fig. 12c. These holes might arise not only from the debonding at the PTT–MB interface but also from the cavities of rubber particles. Fig. 13 shows the TEM micrographs of the blend for which the SEM micrographs have been shown in Fig. 12c. Many voids inside one rubber particle were observed. Strongly deformed cavities can be seen from Fig. 12d. These cavitated rubber particles are so highly deformed that they appear to have closed up together.

Theoretical [36–38] and experimental [39,40] studies show that the role of rubber on toughening of semicrystalline polymers is mostly related to the cavitation of the filler. The high dilative stresses produced in front of a growing crack induce the formation

of voids in or around the rubber particles. As a result, hydrostatic pressure is relieved near the voids, and stress is redistributed in a cellular-like material. Cavitated particles act then as stress concentrators around which the matrix can deform plastically. It is also apparent that the matrix material has yielded, causing substantial irreversible deformation of the rubber particles. It is clearly shown that shear yielding of the matrix is the major toughening mechanism in these impact modified PTT.

Of course, shear yielding of the matrix is the major toughening mechanism in these impact modified PTT. There might be other minor toughening mechanisms existing in these impact modified PTT, which it could not be observed from SEM. They interact and combine each other as show in Fig. 14.

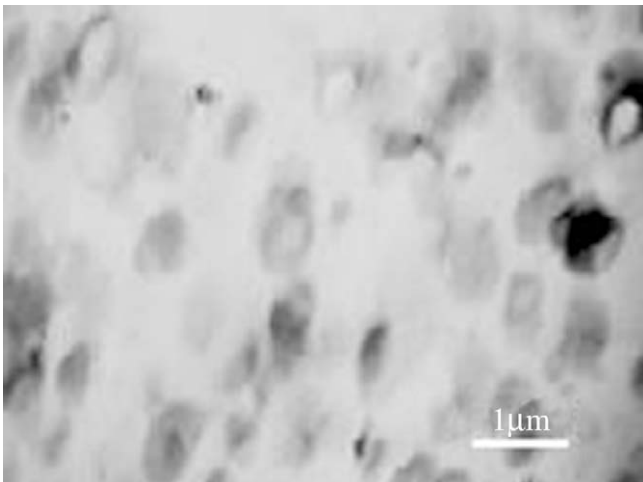


Fig. 13. TEM micrograph of the cryo-fractured surface of PTT/MB blend with 5 wt% DGEBA content taken at location C.

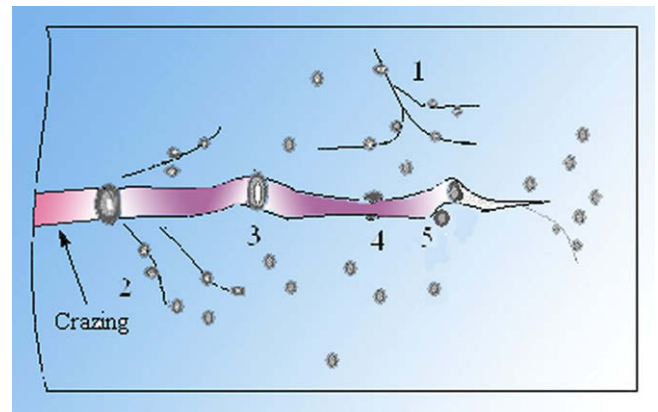


Fig. 14. Toughening mechanisms existing in impact modified PTT. (1. Shear yielding/craze interaction 2. Shear band formation near rubber particles 3. Cavitated particles 4. Transparticle failure 5. Debonding of particles).

#### 4. Conclusion

In this work, diglycidyl ether of bisphenol-A (DGEBA) epoxy resin as a reactive compatibilizer was added to the PTT/MB blends by melt mixing. Fourier transform infrared (FTIR) and rheological measurements identified the epoxy group of DGEBA epoxy resin can react with hydroxyl and carboxyl group of PTT. As the DGEBA epoxy resin content increases from 1 to 5 wt%, the interfacial adhesion between PTT and MB increases, and the size of rubber particles decreases. Mechanical tests showed the impact and tensile properties of PTT blends are improved by the introduction of DGEBA epoxy resin to the blends. The results are very significant in obtaining a material having remarkable stiffness–toughness balance. SEM and TEM were used to observe the fracture morphology and the structure inside the deformed zone of the toughened PTT in order to observe the toughening mechanism. The volume strain  $\Delta V/V$  for the PTT/MB blend with 5 wt% DGEBA epoxy resin is 0.052, indicating that there are voids in samples. Shear yielding of the matrix is the major toughening mechanism in these impact modified PTT.

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