

Thermal and Mechanical Properties of Poly(butylene terephthalate)/Epoxy Blends

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ABSTRACT: In this study, melt blends of poly(butylene terephthalate) (PBT) with epoxy resin were characterized by dynamic mechanical analysis, differential scanning calorimetry, tensile testing, Fourier transform infrared spectroscopy, and wide-angle X-ray diffraction. The results indicate that the presence of epoxy resin influenced either the mechanical properties of the PBT/epoxy blends or the crystallization of PBT. The epoxy resin was completely miscible with the PBT matrix. This was beneficial to the

improvement of the impact performance of the PBT/epoxy blends. The modification of the PBT/epoxy blends were achieved at epoxy resin contents from 1 to 7%. The maximum increase of the notched Izod impact strength ($\approx 20\%$) of the PBT/epoxy blends was obtained at 1 wt % epoxy resin content. © 2008 Wiley Periodicals, Inc. *J Appl Polym Sci* 109: 4082–4088, 2008

Key words: mechanical properties; polyesters; miscibility

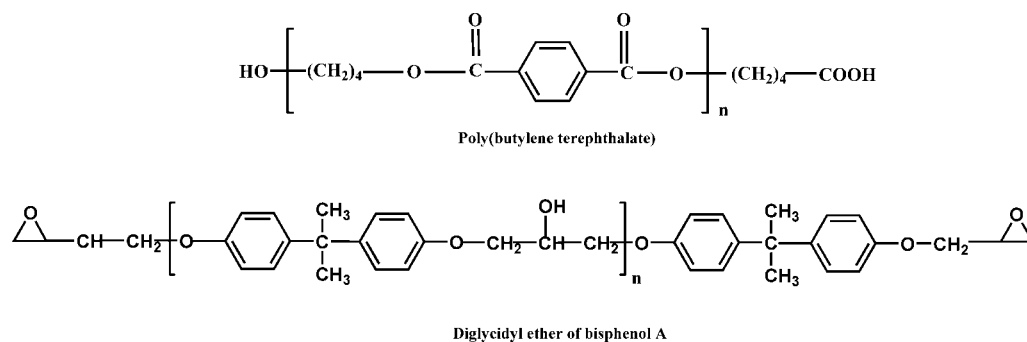
INTRODUCTION

Poly(butylene terephthalate) (PBT) is a polyester that is rapidly gaining importance as an engineering thermoplastic because of its attractive mechanical properties, rapid crystallization rate, and good moldability. However, neat PBT materials fail in a brittle manner under certain loading conditions, such as notched Izod impact. This indicates that PBT has a high resistance to crack initiation but a low resistance to crack propagation. To overcome this problem, many studies of the incorporation of impact modifiers into PBT have been reported. PBT has been blended with several thermoplastics^{1–5} to modify its properties, but relatively little work has been done on PBT–thermoset blends. Epoxies are often used as a reactive solvent in structural composites because of their high stiffness, high strength, good chemical resistance, and excellent dimensional stability. Kulshreshtha et al.^{6,7} studied the crystallization and equilibrium melting behavior of PBT/epoxy blends. They concluded that epoxy resin was compatible with PBT and significantly affected the crystallization behavior of PBT.

The mechanical properties of PBT/epoxy blends have not been studied up to this point. The blending of PBT, in particular, with a low-cost solid epoxy resin, would be of both technical and commercial interest. For this reason, the purpose of this study was to increase the notched impact resistance of PBT with a solid diglycidyl ether of bisphenol A high-molecular-weight epoxy resin blended with PBT. In this study, a curing agent for the epoxy resin was not used. This was because of the fact that after curing, the epoxy phase separated out and was not part of the spherulites, as in the uncured case. The network formed by the epoxy also hindered the crystallization of PBT and thus lowered the degree of crystallinity.⁷ Reactions between carboxylic acid and epoxy groups have been exploited in many blends of thermoplastic polyesters.⁸ The objective of this study was to investigate the effect on the enhancement of the mechanical properties and the crystallinity of PBT by the addition of the high-molecular-weight epoxy resin E03. Epoxy resin E03 had a high stiffness, high strength, good chemical resistance, and excellent dimensional stability; furthermore, the solid epoxy resin had good processability with PBT. In this study, we investigated the impact strength influence on the PBT/epoxy blends from the variation of the amount of epoxy resin. Through X-ray diffraction analysis of the PBT/epoxy blends and tensile testing, we found that the 99/1 PBT/epoxy had a better impact strength and elongation at break than the pure PBT.

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Scheme 1

EXPERIMENTAL

Material and blend preparation

The materials used were polybutylene terephthalate (PBT) S3122 (intrinsic viscosity $[\eta] = 1.22$ dL/g), measured in a 1/1 w/w phenol/tetrachloroethane solvent at 25°C, from the Engineering Plastics Plant of Yihua Group Corp., Yizheng, China, and epoxy resin EPO 1691-410 (E-03 609, diglycidyl ether of bisphenol A) with an epoxy equivalent weight of 2400–4000 g/eq, supplied by Diandong Branch Plant of Shanghai Resin Plant, Shanghai, China (Scheme 1).

PBT and epoxy resin were dried at 80°C for 24 h in a vacuum oven; PBT/epoxy resin blends with an epoxy content of 1–30 wt % were prepared in a miniature corotating twin-screw extruder at 230°C and 150 rpm. The extruded strands were cooled in a water bath and then pelletized.

Characterization

Notched Izod impact tests were performed at $23 \pm 2^\circ\text{C}$ according to ASTM D 256 on a XJU-22 apparatus (Chengde, China). Samples with dimensions $63.5 \times 12.7 \times 6.35$ cm³ were obtained from injection-molded specimens. The notch was milled with a depth of 2.54 mm, an angle of 45°, and a notch radius of 0.25 mm. The uniaxial tensile tests were carried out at $23 \pm 2^\circ\text{C}$ on a Shimadzu AGS-H tensile tester (Kyoto, Japan) at a crosshead speed of 50 mm/min according to ASTM D 638.

Thermal analysis was carried out with a PerkinElmer DSC-7 differential scanning calorimeter (Waltham, MA) calibrated with indium. The heating and cooling rates were 10°C/min with a nitrogen purge, and the sample weights were between 4.5 and 5.5 mg. The temperature range was 50–250°C.

Melt flow rate determination was carried out with a $\mu\text{PXRZ-400C}$ Instruments apparatus (Changchun, China) at 235°C with a load of 2.16 kg.

Dynamic mechanical analysis (DMA) was performed on a Netzsch DMA 242C (Selb, Germany), which provided plots of the loss tangent ($\tan \delta$) and

the storage modulus (E') against temperature. The scans were carried out in tension mode at a constant heating rate of 3°C/min and a frequency of 3.33 Hz from 0 to 150°C.

Possible reactions between PBT and the epoxy resin were studied by Fourier transform infrared (FTIR) spectroscopy with a Bruker Vertex 70 FTIR spectrophotometer (Karlsruhe, Germany).

Wide-angle X-ray diffraction (WAXD) patterns were taken with a Rigaku D/max 2500PC type X-ray diffractometer (Tokyo, Japan) with a 18-kW rotating anode generated at 40 kV and 200 mA and curved graphite crystal monochromator filtered Cu K α radiation. Before measurement, the samples were melted and crystallized isothermally at 180°C for 1 h. The melt sample of PBT was quenched rapidly in liquid nitrogen to obtain the amorphous sample.

RESULTS AND DISCUSSION

Miscibility

DMA is one of the most sensitive techniques available for characterizing and interpreting the mechanical behaviors of materials. Figures 1 and 2 show the dependence of $\tan \delta$ and E' on the temperature for

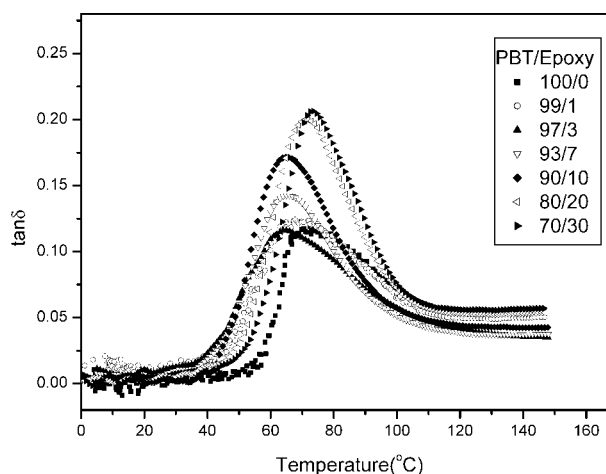


Figure 1 DMA plots of $\tan \delta$ versus temperature for the PBT/epoxy blends.

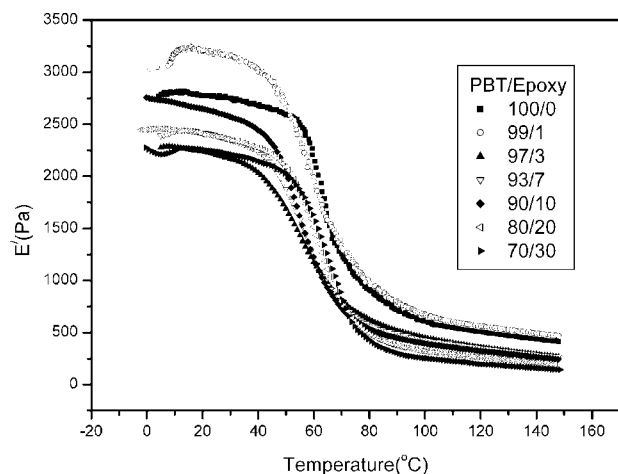


Figure 2 DMA plots of E' versus temperature for the PBT/epoxy blends.

PBT modified with different contents of epoxy resin, respectively. The single glass transition confirmed that the blend components were miscible. At the temperatures of 74, 68, and 62, the 100/0, 99/1, and 97/3 PBT/epoxy blends had single glass-transition temperature (T_g) peaks, respectively. The 97/3 PBT/epoxy blend had the lowest T_g . T_g for the PBT/epoxy blends shifted to a higher temperature with increasing epoxy resin content when the epoxy resin content was above 7%. Compared with the pure 100/0 PBT curve, the T_g peak of the 93/7 PBT/epoxy blend curve became narrower, which indicated that the PBT amorphous region was affected by the epoxy resin. The height of the $\tan \delta$ peak is generally a measure of the damping nature of a compound. From the $\tan \delta$ curve, we observed that the height of the peak increased with epoxy resin addition. The higher damping indicated reduced strength. This indicated that higher epoxy resin addition may have acted as a solvent to reduce the interaction between

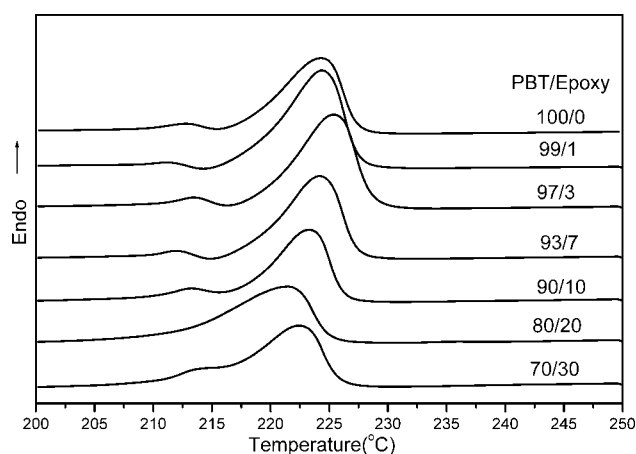


Figure 3 Differential scanning calorimetry plots for the PBT/epoxy blends reheated from 50 to 250°C.

TABLE I
Mechanical Properties of the PBT/epoxy Blends

PBT/epoxy	Impact strength (J/m)	Tensile strength (MPa)	Elongation at break (%)	Energy (J)
100/0	47	48.8	335	240
99/1	57	49.4	425	315
97/3	56	50.7	378	278
93/7	50	52.9	366	273
90/10	48	53.9	315	252
80/20	44	57.1	132	110
70/30	42	59.8	34	32

the PBT and epoxy resin.⁷ The E' values for the 99/1 PBT/epoxy blend changed more than those of the pure PBT over the temperature range. This implied that the toughness of PBT was reinforced by the addition of 1% epoxy resin.

Thermal properties

The melting behavior was studied by differential scanning calorimetry; the samples were first heated from 50 to 250°C at 10°C/min, then cooled at the same rate, and finally heated again. Figure 3 shows that the position of the melting peak of the PBT/epoxy blends shifted to higher temperatures when the content of epoxy resin was 3%. This suggested a nucleation effect of the 3% epoxy resin. On the contrary, when the content of the epoxy resin was above 10%, the melting temperature decreased. It seemed that the amount of PBT was not enough to completely react with the large amount of epoxy resin. The excessive epoxy resin may have acted as a solvent to restrict crystallization in confined regions.⁷ The 80/20 PBT/epoxy blend showed a single crystallization peak; this indicated that the 20% epoxy resin had greatly interacted with the PBT matrix. However, the other PBT/epoxy blends showed dou-

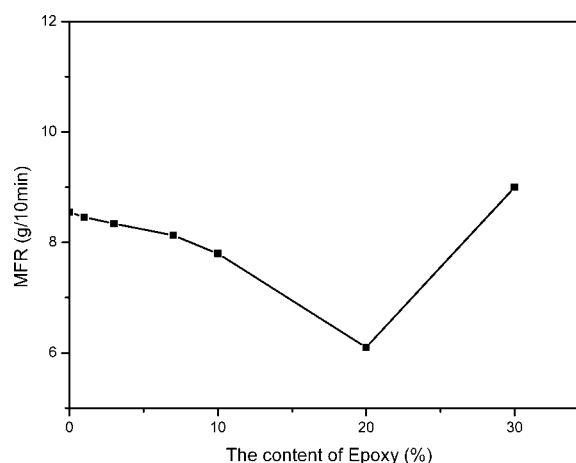
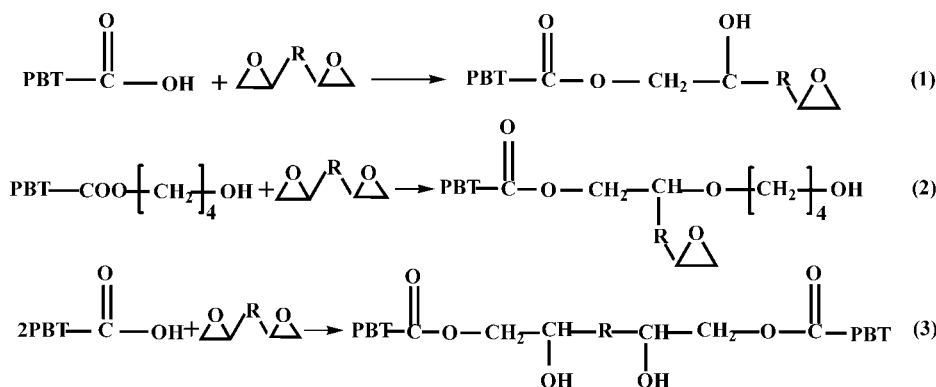


Figure 4 Plots of melt flow rate (MFR) versus the content of epoxy resin for the PBT/epoxy blends.



Scheme 2

ble melting behavior; this may have been due to the recrystallization process during heating.

These results led us to conclude that the increased interactions between the phases modified the crystallization behavior of the blend components. In particular, the crystallization of the PBT/epoxy blends was affected by the addition of epoxy resins.

Mechanical properties

The ultimate goal of the functionalization of epoxy resins was the improvement of the mechanical properties of the blends, especially impact strength. The mechanical properties of the polymer blends were greatly influenced by their miscibility. The notched Izod impact strengths and tensile properties at various epoxy resin E03 contents for the PBT/epoxy blends are given in Table I. The mechanical properties of the PBT/epoxy blends increased to some extent with the addition of a small amount of epoxy resin E03. The Izod impact strength of the 99/1 PBT/epoxy was 57 J/m, which was higher than that of pure PBT (47 J/m), but when we continued to

increase the epoxy resin content, the impact strength slightly decreased to 42 J/m at 30% epoxy in the blend. The tensile strength for the blends increased with increasing epoxy resin. The elongation at break increased 26.8%, whereas the epoxy resin content was 1% and decreased gradually with increasing epoxy resin weight percentage. The increase in the elongation at break was evidence for the improvement of the impact strength in the blends. Interestingly, the values for elongation at break and Izod impact strength showed the same trend. It was obvious that 1% was the optimum content of epoxy resin for the improvement of the mechanical properties of the PBT/epoxy blends. These results may have been caused by the existence of a probable ideal interfacial action between the PBT and epoxy resins phases in the PBT/epoxy blends.

Rheological properties

The melt flow rate for PBT and the blends at 235°C is shown in Figure 4; as the epoxy resins content increased, the melt flow rate decreased, and PBT had lower viscosity than that of the PBT/epoxy

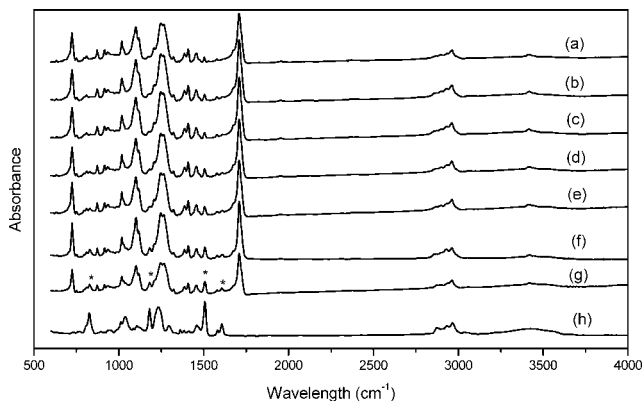


Figure 5 FTIR spectra of the PBT/epoxy blends (the bands marked with asterisks were sensitive to the relative intensities of epoxy) at 180°C for 1 h: PBT/epoxy (w/w) (a) 100/0, (b) 99/1, (c) 97/3, (d) 93/7, (e) 90/10, (f) 80/20, (g) and 70/30 and (h) epoxy resin.

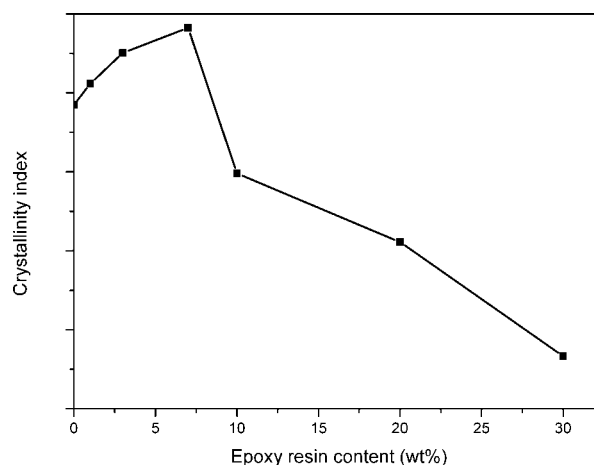


Figure 6 CIs of the PBT/epoxy blends as determined by FTIR measurements.

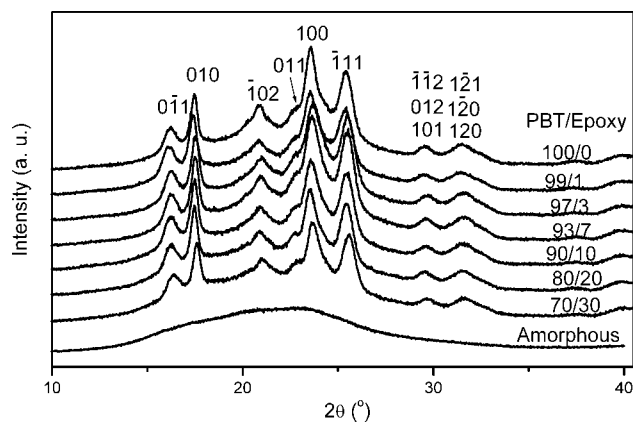


Figure 7 WAXD patterns of the PBT/epoxy melt-crystallized at different compositions.

blends when the epoxy resin content was below 20%. It is known that an increase in the complex viscosity of a blend generally occurs when there is either a specific interaction between the phases^{9,10} or chemical bonding between the blend components.¹¹ Such a chemical bonding will also induce a strong interaction between the phases. Consequently, the matrix will give more resistance to flow, which will result in a high viscosity. When there is no interaction or chemical bonding between the phases, however, low viscosity will be found because the domain can be elongated easily in the matrix. Therefore, from the higher complex viscosity of the PBT/epoxy blends over pure PBT, we deduced that a chemical reaction took place between the epoxy resins and the carboxylic acid end groups of PBT.

Because of epoxidation, epoxy resins, which bore epoxy groups, provided the functionalized sites with which the terminal PBT hydroxide or carboxylic acid groups could react. An et al.¹² studied how epoxy resin E-44 reacted with the carboxylic acid end groups of PBT, as shown in eqs. (1)–(3) in Scheme 2.

When the epoxy resins was melt-mixed with the PBT matrix, according to the reactions shown in eqs. (1)–(3) in Scheme 2, a graft copolymer (epoxy-g-PBT) was presumably formed, which could have improve the toughness of the PBT/epoxy blends. When the epoxy resin content was below 20%, the epoxy resin may have acted as a reactant to increase viscosity of the PBT/epoxy blends. Therefore, the melt flow rate decreased. Contrarily, when the epoxy resins content was over 20%, because of excess epoxy, the epoxy resins may have acted as a solvent to decrease the viscosity of the PBT/epoxy blends, and the melt flow rate increased.¹³

FTIR

To obtain more information on the miscibility, the crystallization behavior of the PBT/epoxy blends

was studied by FTIR spectroscopic measurements (Fig. 5). As shown in Figure 5(a), the spectrum of PBT was characterized by the following peaks: $\nu(\text{O}-\text{CH}_2)$ at 2961 cm^{-1} , $\nu(\text{CH}_2-\text{CH}_2)$ at 2925 and 2854 cm^{-1} , $\nu(\text{C}=\text{O})$ at 1711 cm^{-1} , $\nu[\text{C}(\text{O})-\text{O}]$ at 1270 cm^{-1} , $\nu(\text{O}-\text{CH}_2)$ at 1102 cm^{-1} , $\nu(\text{ring})$ at 1017 cm^{-1} , and $\lambda(\text{CH aromatic})$ at 728 cm^{-1} .¹⁴ As shown in Figure 5(h), the spectrum of the epoxy resin was characterized by the following peaks: $\nu(\text{O}-\text{CH}_2)$ at 2961 cm^{-1} ; $\nu(\text{CH}_2-\text{CH}_2)$ at 2925 and 2854 cm^{-1} ; $\nu(\text{O}-\text{CH}_2)$ at 1102 cm^{-1} ; $\nu(\text{aromatic ring})$ at 1607 , 1582 , and 1506 cm^{-1} ; $\nu(\text{COC})$ at 1236 and 1182 cm^{-1} ; $\lambda(\text{CH})$ at 827 cm^{-1} ; and $\nu(\text{CH}-\text{CH}_2)$ at 769 cm^{-1} .¹⁵ As shown in Figure 5(b–g), with increasing composition of epoxy, the relative intensities of the bands at 1607 , 1506 , 1181 , and 827 cm^{-1} became correspondingly stronger. However, the band at 1711 cm^{-1} was sensitive to the degree of crystallinity of PBT.¹⁶ Thus, we were particularly interested in this region. The band at 938 cm^{-1} corresponded to the CH_2 rocking vibration of the amorphous hard segments,¹⁷ which was insensitive to the composition and degree of crystallinity, so it was chosen as a normalized peak. The degree of crystallinity was obtained by normalization of the absorbance at 1711 cm^{-1} to that of the 938-cm^{-1} band. Hence, crystallinity index (CI)^{18,19} was defined as the ratio of the intensity of the band at 1711 cm^{-1} to that at 938 cm^{-1} (Fig. 6). This CI is not to be confused with the absolute degree of crystallinity. When the epoxy content was below 10%, the CI was higher than that of pure PBT. Contrarily, when the epoxy content was over 10%, the CI was lower than that of pure PBT. This result indicates that the epoxy resin portion interfered with the crystalline formation of PBT; a small amount ($<10\%$) of epoxy resin improved the crystallinity degree of the PBT/epoxy blends.

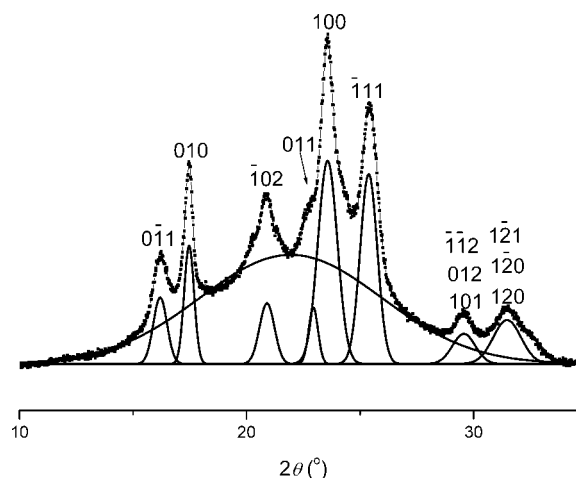


Figure 8 Resolution of the WAXD curve of PBT into crystalline and amorphous portions.

TABLE II
X-Ray Diffraction Data of Pure PBT

hkl	A	011	010	102	011	100	111	112, 012, 101	121, 120, 120
2θ	22.02	16.22	17.45	20.90	22.83	23.59	25.42	29.59	31.47
$I_a(\theta)$ or $I_{i,hkl}(\theta)$	21,223	1080	1394	726	579	3057	2364	610	1042
I	45,880	1080	1709	1495	1578	9165	9145	4008	8880
T	0.86	0.92	0.91	0.87	0.85	0.84	0.82	0.76	0.73
$f^2 = 12f_H^2 + 12f_C^2 + 12f_O^2$	466	555	536	483	452	442	414	358	331
LP	52.3	98.4	84.3	58.1	48.1	45.2	38.5	28.1	24.3
C(θ)	2.40	1.00	1.23	2.06	2.73	2.99	3.87	6.57	8.52
K	2.16								

A, amorphous peak; C (θ) is correction factor of amorphous or crystalline peak of polymer.

Analysis by WAXD

Figure 7 shows the WAXD patterns for the PBT/epoxy blends. By means of the graphic multipeak resolution method,²⁰ the degree of crystallinity of PBT and the PBT/epoxy blends determined by WAXD was calculated as follows:^{21,22}

$$W_{c,x} = \frac{\sum_i C_{i,hkl}(\theta)I_{i,hkl}(\theta)}{\sum_i C_{i,hkl}(\theta)I_{i,hkl}(\theta) + k_i C_a(\theta)I_a(\theta)} \times 100\% \quad (4)$$

where $W_{c,x}$ is the degree of crystallinity, $I_{i,hkl}(\theta)$ and $I_a(\theta)$ are the relative intensities of the crystalline and amorphous peaks, respectively, and $C_{i,hkl}(\theta)$ and $C_a(\theta)$ are the correction factors of the crystalline and amorphous peaks, respectively. On the basis of the X-ray diffraction intensity theory, K is the total correction factor, the calibration constant [$K = C_a(\theta) \times k_i$, where k_i is the relative scattering coefficient, which is a ratio of calculated diffraction intensity ($\sum I_{i,cal}$) to total scattering intensity ($\sum I_{i,total}$) for unit weight of crystalline polymer, and $k_i = \sum I_{i,cal} / \sum I_{i,total}$ ($k_i \leq 1$)]. Here, $C_{i,hkl}(\theta)$ or $C_a(\theta)$ can be calculated by the following equation:

$$C_{i,hkl}^{-1}(\theta) \text{ or } C_a^{-1}(\theta) = f^2 \cdot \frac{1 + \cos^2 2\theta}{\sin^2 \theta \cdot \cos \theta} \cdot e^{-2B(\sin \theta / \lambda)^2} = \sum_i N_i f_i^2 \cdot \frac{1 + \cos^2 2\theta}{\sin^2 \theta \cdot \cos \theta} \cdot e^{-2B(\sin \theta / \lambda)^2} \quad (5)$$

where f is the atomic scattering factor for one crystallographic structural repeating unit, f_i is the scattering factors of the i th atom, N_i is the number of i th atoms in a repeating unit, 2θ is the Bragg angle, the angle factor (LP) = $(1 + \cos^2 2\theta) / \sin^2 \theta \cos \theta$, the temperature factor (T) = $e^{-2B(\sin \theta / \lambda)^2}$, and $2B = 10$.

f_i can be expressed approximately by

$$f_i(\sin \theta / \lambda) = \sum_{i=1}^4 a_i \cdot e^{-b_i(\sin \theta / \lambda)^2} + C \quad (6)$$

where the values of a , b , and C are given in ref. 23.

The WAXD curves of the PBT/epoxy blends showed eight crystalline peaks at $2\theta_{011} = 16.22^\circ$, $2\theta_{010} = 17.45^\circ$, $2\theta_{102} = 20.90^\circ$, $2\theta_{011} = 22.83^\circ$, $2\theta_{100} = 23.59^\circ$, $2\theta_{111} = 25.42^\circ$, $2\theta_{112, 012, 101} = 29.59^\circ$, and $2\theta_{121, 120, 120} = 31.47^\circ$ and one amorphous peak at $2\theta_a = 22.02^\circ$. The total WAXD curve of pure PBT was resolved into crystalline and amorphous portions (Fig. 8). From eqs. (4)–(6), the X-ray diffraction data of the PBT/epoxy blends are shown in Table II. $I = C_{i,hkl}(\theta)I_{i,hkl}(\theta)$ or $C_a(\theta)I_a(\theta)$ is the integrating intensity scattered over a suitable angular interval by the crystalline and the amorphous phases, respectively. There are 12 atoms of carbon, 12 atoms of hydrogen, and 4 atoms of oxygen in a repeating unit of PBT; the total atomic scattering factor was $f_{hkl}^2 = 12f_C^2 + 12f_H^2 + 12f_O^2$, where $k_i = 0.9$, $C_a(\theta) = 2.4$, and $K = k_i C_a(\theta) = 2.16$. With the data of Table II, eq. (4) can be reduced to give eq. (7).

$$W_{c,x} = \frac{I_{011} + 1.23I_{010} + 2.06I_{102} + 2.73I_{011} + 2.99I_{100} + 3.87I_{111} + 6.57I_{112,012,101} + 8.52I_{121,120,120}}{I_{011} + 1.23I_{010} + 2.06I_{102} + 2.73I_{011} + 2.99I_{100} + 3.87I_{111} + 6.57I_{112,012,101} + 8.52I_{121,120,120} + 2.40I_a} \quad (7)$$

where I_a is the intensity of amorphous peak. With eq. (7), the degrees of crystallinity of PBT and the PBT/epoxy blends were determined by WAXD and are shown in Table III. The degree of crystallinity of the 93/7 PBT/epoxy blend reached 50.1%, whereas that of pure PBT was 44.6%. The degree

of crystallinity was closely related to the impact strength, elongation at break, and energy for the PBT/epoxy blends. To improve the impact strength, the optimal epoxy resin content was 1%, and the degree of crystallinity was 46.8% for the PBT/epoxy blends.

TABLE III
X-Ray Degree of Crystallinity for the PBT/Epoxy Blends from Melting–Crystallization

PBT/epoxy	100/0	99/1	97/3	93/7	90/10	80/20	70/30
$W_{c,x}$ (%)	44.6	46.8	48.5	50.1	43.6	40.0	38.5

CONCLUSIONS

The mechanical properties of the PBT/epoxy blends were improved by the addition of a small amount of epoxy resin E03. In mechanical properties, the PBT/epoxy blends showed improved notched Izod impact strengths over the pure PBT when the epoxy resin content was lower 10%. From the FTIR analysis, the epoxy resin interacted with PBT. On the basis of this study, we concluded that epoxy resin E03 could be effectively used as a filler in PBT, and to obtain the best properties, an optimum loading of 1 wt % epoxy resin E03 should be used.

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