

# Study on *In Situ* Reinforcing and Toughening of a Semiflexible Thermotropic Copolyesteramide in PBT/PA66 Blends

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Received 4 June 1999; accepted 10 September 1999

**ABSTRACT:** Ternary *in situ* composites based on poly(butylene terephthalate) (PBT), polyamide 66 (PA66), and semiflexible liquid crystalline polymer (LCP) were systematically investigated. The LCP used was an ABA30/PET liquid crystalline copolyesteramide based on 30 mol % of *p*-aminobenzoic acid (ABA) and 70 mol % of poly(ethylene terephthalate) (PET). The specimens for thermal and rheological measurements were prepared by batch mixing, while samples for mechanical tests were prepared by injection molding. The results showed that the melting temperatures of the PBT and PA66 phases tend to decrease with increasing LCP addition. They also shifted toward each other due to the compatibilization of the LCP. The torque measurements showed that the ternary blends exhibited an apparent maximum near 2.5–5 wt % LCP. Thereafter, the viscosity of the blends decreased dramatically at higher LCP concentrations. Furthermore, the torque curves versus the PA66 composition showed that the binary PBT/PA66 blends can be classified as negative deviation blends (NDBs). The PBT/PA66/LCP blends containing up to 15 wt % LCP were termed as positive deviation blends (PDBs), while the blends with the LCP  $\geq 25$  wt % exhibited an NDB behavior. Finally, the tensile tests showed that the stiffness and tensile strength of ternary *in situ* composites were generally improved with increasing LCP content. The impact strength of ternary composites initially increased by the LCP addition, then deteriorated when the LCP content was higher than 10 wt %. The correlation between the mechanical properties and morphology of the blends is discussed. © 2000 John Wiley & Sons, Inc. *J Appl Polym Sci* 77: 1975–1988, 2000

**Key words:** poly(butylene terephthalate); polyamide 66; semiflexible liquid crystalline polymer; toughening

## INTRODUCTION

Poly(butylene terephthalate) (PBT) and polyamide 66 (PA66) are semicrystalline polymers

and are used extensively as structural materials in the automotive, electrical, and electronic industries and textile manufacture sectors owing to their high mechanical strength, stiffness, and good processability. The blending of thermoplastic polyester with polyamide has received considerable attention in recent years. Utracki and coworkers<sup>1,2</sup> reported that poly(ethylene terephthalate) (PET) and PA66 exhibit a small degree of compatibility in the molten state, and the blends show brittle failure in both tensile and impact

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Contract grant sponsor: City University of Hong Kong; contract grant number: 7000607.

*Journal of Applied Polymer Science*, Vol. 77, 1975–1988 (2000)  
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tests. The compatibility between PET and PA66 can be further improved by the addition of a catalyst, which promotes the ester–amide interchange reaction effectively. The products of the interchange reaction act as compatibilizers, thereby modifying the morphology of the PET/PA66 blends.<sup>3,4</sup> More recently, Huang and Chang<sup>5</sup> reported that solid epoxy resin is an effective compatibilizer for the incompatible PBT/PA66 blends.

*In situ* polymer composites based on blends of liquid crystalline polymers (LCPs) with thermoplastics have received considerable attention in the past decades. This is because LCPs can act as reinforcing agents and processing aids for thermoplastic polymers. In the former case, the LCP component can deform into fine fibrils under appropriate processing conditions, leading to the formation of LCP fibrils in the polymer matrix. In most cases, LCPs and thermoplastics are incompatible owing to their fundamentally different structures. Therefore, the LCP and the blend component of *in situ* composites exhibit a low interfacial adhesion, thereby resulting in poor mechanical properties, particularly the tensile strength. The adhesion can be improved by increasing the interactions between the phases, physically or chemically, via the addition of compatibilizers or the modification of the LCP structure. Compatibilizers based on block or graft copolymers are commonly used to improve the adhesion of the components for LCP/thermoplastic blends.<sup>6–8</sup> Compatibilization can also take place by specific interactions between the LCP and the matrix polymer, for example, ion–dipole interaction,<sup>9,10</sup> chemical interaction,<sup>11,12</sup> and hydrogen bonding.<sup>13</sup> In the case of modifying the LCP structure for enhancing the compatibility, several workers reported that the introduction of a long flexible spacer in the main chain of the LCP enhances the adhesion between the LCP and the polymer matrix as well as improving their processability.<sup>14–19</sup> More recently, Tjong and coworkers<sup>20,21</sup> studied the structure of the blends of semiflexible LCP with polyamide or with polyester. Semiflexible LCP is a copolyesteramide based on *p*-aminobenzoic acid (ABA) and PET. They reported that semiflexible ABA30/PET LCP containing 30 mol % of ABA units is partially miscible with PBT or PA66 in the solid state, but LCPs and homopolymers are miscible in the molten state due to the presence of the flexible PET segment chain in an LCP. Moreover, some specific

interactions between the LCP and the aliphatic polyamide or polyester matrix take place in the blends during blending. It is worth mentioning that the studies carried out by these workers were focused on the morphology, rheology, and mechanical properties of binary LCP/thermoplastic blends. Little information is available on the properties of *in situ* composites in which their matrices are polymer blends consisting of two thermoplastics.<sup>22–24</sup>

In the present work, we attempted to prepare ternary blends of PBT/PA66 with ABA30/PET liquid crystalline copolyesteramide, and to study their morphological, rheological, and mechanical properties. As the ABA30/PET molecules consist of ester and amide segments, it is expected that semiflexible LCP can act as a compatibilizer for the PBT/PA66 blends as well as a reinforcing agent.

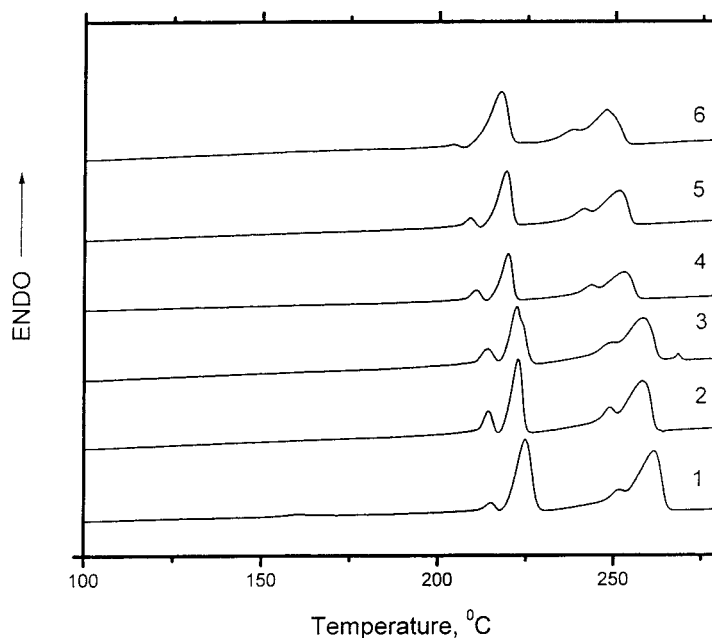
## EXPERIMENTAL

### Materials

The ABA30/PET liquid crystalline copolyesteramide (LCP) used in this work was synthesized from 30 mol % of ABA and 70 mol % of PET according to the procedures reported by Jackson and Kuhfuss<sup>25</sup> and Xie et al.<sup>26,27</sup> Its intrinsic viscosity was 0.57 dL/g. The glass transition and melting temperatures were 100 and 256°C, respectively. PBT (Lumicon 1401) and PA66 (Novamid) pellets were supplied by Toray Industries Inc. (Japan) and the Mitsubishi Engineering–Plastics Corp. (Taiwan), respectively. All materials were dried in an oven at 120°C for 48 h before mixing.

### Sample Preparation

Ternary PBT/PA66/LCP blends containing 0, 2.5, 5, 10, 25, and 35 wt % LCP were blended. The weight ratios of PBT/PA66 in the ternary blends were set at 75/25, 50/50, and 25/75. The blending was performed in a Brabender Plasticorder equipped with a mixing chamber of 50 cm<sup>3</sup> and operated at 275°C and 75 rpm for 5 min. In the process, PBT, PA66, and LCP pellets were simultaneously introduced into the Brabender batch mixer. The torques during blending were determined on-line. The blends produced were then compression-molded into plates under a pressure of 10 MPa at 270°C. These molded plates were finally cut into small granules for thermal measurement.



**Figure 1** Typical DSC heating scanning curves of PBT/PA66/LCP blends: (1) PBT/PA66 50/50; (2) PBT/PA66/LCP 48.75/48.75/2.5; (3) PBT/PA66/LCP 47.5/47.5/5; (4) PBT/PA66/LCP 45/45/10; (5) PBT/PA66/LCP 42.5/42.5/15; (6) PBT/PA66/LCP 37.5/37.5/25.

Samples for tensile and impact measurements were fabricated by injection molding. The binary PBT/PA66 50/50 blend (weight fraction)

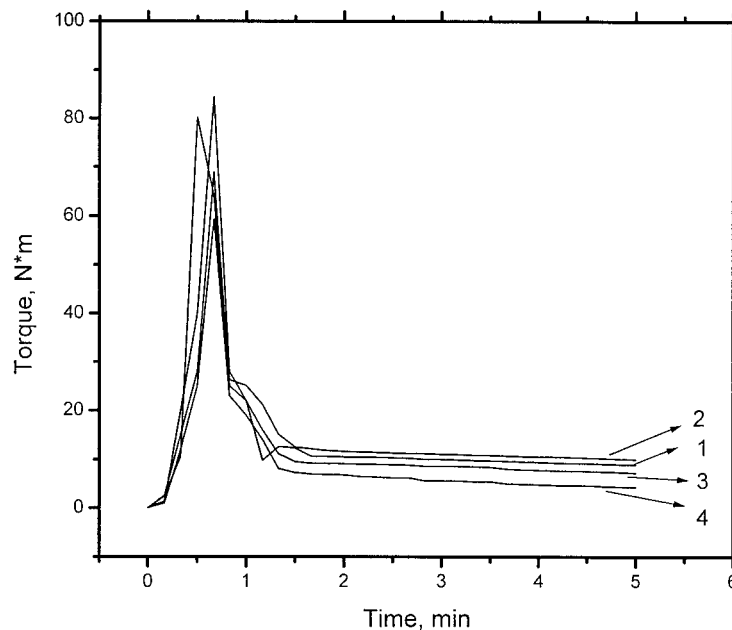
**Table I** Thermal Properties of PBT/PA66/LCP Blends

PBT/PA66/LCP	$T_{m1}$ (°C)	$T_{m2}$ (°C)	$T_{m2} - T_{m1}$ (°C)
75/25/0	219.94	250.98	31.04
73.125/24.375/2.5	219.58	249.46	29.86
71.25/23.75/5	218.23	244.65	26.4
67.5/22.5/10	216.77	241.66	24.89
63.75/21.25/15	215.34	239.66	24.22
58.75/16.25/25	211.9	225.97	14.07
50/50/0	219.72	252.07	32.35
48.25/48.25/2.5	218.64	248.95	30.31
47.5/47.5/5	218.40	248.43	29.65
45/45/10	215.69	243.04	27.34
42.5/42.5/15	214.39	241.16	26.75
37.5/37.5/25	211.66	238.76	27.70
25/75/0	219.88	252.77	31.89
24.375/73.125/2.5	218.14	249.69	31.55
23.75/71.25/5	217.1	247.94	29.84
22.5/67.5/10	214.39	245.84	31.24
21.25/63.75/15	212.36	242.24	29.88
16.25/58.75/25	208.65	237.54	28.89

was prepared as the matrix of ternary PBT/PA66/LCP *in situ* composites. The ternary blends consisting of 2.5, 5, 10, 15, 25, and 35 wt % LCP were also prepared by a twin-screw Brabender Plasticorder at 275°C and 75 rpm. The extrudates exiting from the Brabender were cut into pellets by a pelletizer. The plaques with dimensions of 200 × 80 × 3.2 mm were injection-molded from these pellets. The barrel-zone temperatures of the injection molder were set at 275, 280, and 275°C. These plaques were cut into dog-bone-shaped tensile bars. Notched Izod impact specimens were also prepared from the plaques. Both longitudinal and transverse specimens were used for the tensile and impact tests. For the longitudinal specimens, the length direction was parallel to the flow direction, while it was perpendicular to the flow direction for the transverse specimens.

**Table II** Values of the Interaction Parameter ( $\chi_{12}$ ) for LCP-PA66 and LCP-PBT Blends at 270°C<sup>20,21</sup>

LCP Concentration	$\chi_{12(\text{LCP-PA66})}$	$\chi_{12(\text{LCP-PBT})}$
<10 wt %	-0.21	$-7.01 \times 10^{-3}$
>10 wt %	-0.014	$-1.01 \times 10^{-3}$



**Figure 2** Torque curves of PBT/PA66 and some PBT/PA66/LCP blends: (1) PBT/PA66 50/50; (2) PBT/PA66/LCP 47.5/47.5/5; (3) PBT/PA66/LCP 42.5/42.5/15; (4) PBT/PA66/LCP 37.5/37.5/25.

### Differential Scanning Calorimetry (DSC)

DSC measurements were conducted using a Perkin-Elmer DSC-7 instrument at a heating rate of  $10^{\circ}\text{C min}^{-1}$  under a dry nitrogen atmosphere. Prior to the DSC recording, all samples were heated to  $280^{\circ}\text{C}$  and then kept at this temperature for 3 min to eliminate the influence of their previous thermal histories. They were finally quenched to ambient temperature.

### Dynamic Mechanical Analysis

Dynamic mechanical analysis (DMA) was conducted with a DuPont dynamic mechanical analyzer (Model 983) at a fixed frequency of 1 Hz and an oscillation amplitude of 0.15 mm. The samples with dimensions of  $50 \times 15 \times 3.2$  mm were prepared from compression-molded plates. The temperature range studied was from  $-10$  to  $170^{\circ}\text{C}$  with a heating rate of  $2^{\circ}\text{C min}^{-1}$ .

### Mechanical Properties

The tensile behavior of the injection-molded composites was determined using an Instron tester (Model 4206) at room temperature under a cross-head speed of  $1 \text{ mm min}^{-1}$ . Izod impact specimens with dimensions of  $65 \times 13 \times 3.2$  mm were cut

from the plaques and tested by a Ceast impact pendulum tester. These specimens were sharply notched with a Ceast cutter with a notch tip radius of 0.25 mm.

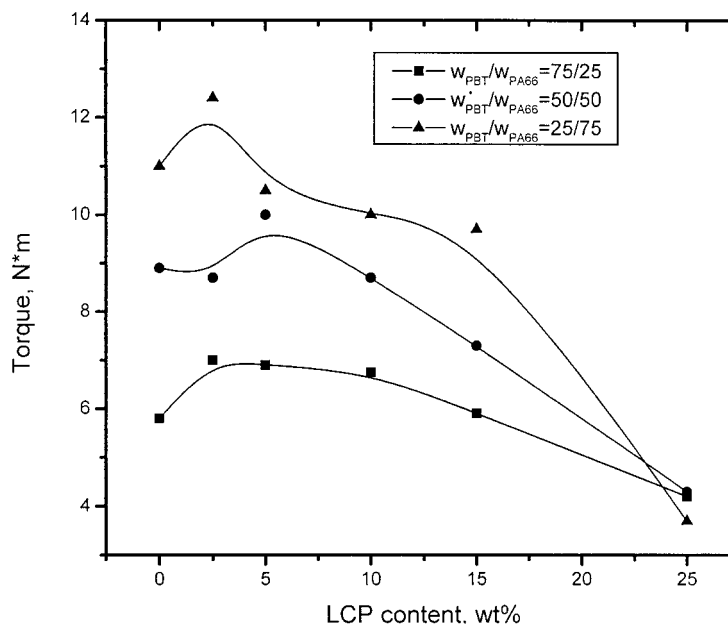
### Morphology Observations

The morphologies of the fracture surfaces of the ternary blends were observed in a scanning electron microscope (SEM, Model JEOL JSM 820). The specimens used for phase-dispersion observations were taken from the strands extruded from the twin-screw Brabender Plasticorder. These specimens were fractured in liquid nitrogen and then etched in 88 wt % formic acid to remove selectively PA66 phases in the blends from the surface of the fracture samples. The specimens used for microfibril observation were cut from the injection-molding plaques along the flow direction. They were also fractured in liquid nitrogen. All fractured surfaces were coated with a thin layer of gold prior to SEM examination.

## RESULTS AND DISCUSSION

### Thermal Properties

Figure 1(a-c) shows some representative DSC heating thermograms for ternary PBT/PA66/LCP



**Figure 3** Relationship between the steady torque value and the LCP content.

blends. The melting temperatures of PA66 ( $T_{m1}$ ) and PBT ( $T_{m2}$ ) are reported to be 252 and 220°C, respectively.<sup>20,21</sup> Table I lists the  $T_m$ 's of the samples investigated determined from the DSC curves. Apparently,  $T_{m1}$  and  $T_{m2}$  tend to decrease with increasing LCP content. The difference in the melting temperatures of the PA66 and PBT phases appears to become smaller with increasing LCP content. This indicates that the LCP addition enhances the interaction between the PBT and PA66 phases. In previous studies, Tjong and coworkers<sup>20,21</sup> indicated that the melting temperatures of the PBT phase in the PBT/LCP blends and the PA66 phase in the PA66/LCP *in situ* composites tend to decrease with increasing LCP concentration.

According to the Flory–Huggins theory,<sup>28</sup> the interaction parameter ( $\chi_{12}$ ) can be used to characterize the compatibility between the polymer components of a blend. In general, the more negative the value of  $\chi_{12}$  is, the more miscible/compatible the blend components are. From the Nishi and Wang theory,<sup>28</sup> the following equation can be used to determine the melting-point depression of PA66 and PBT in the LCP/PA66 and LCP/PBT blends:

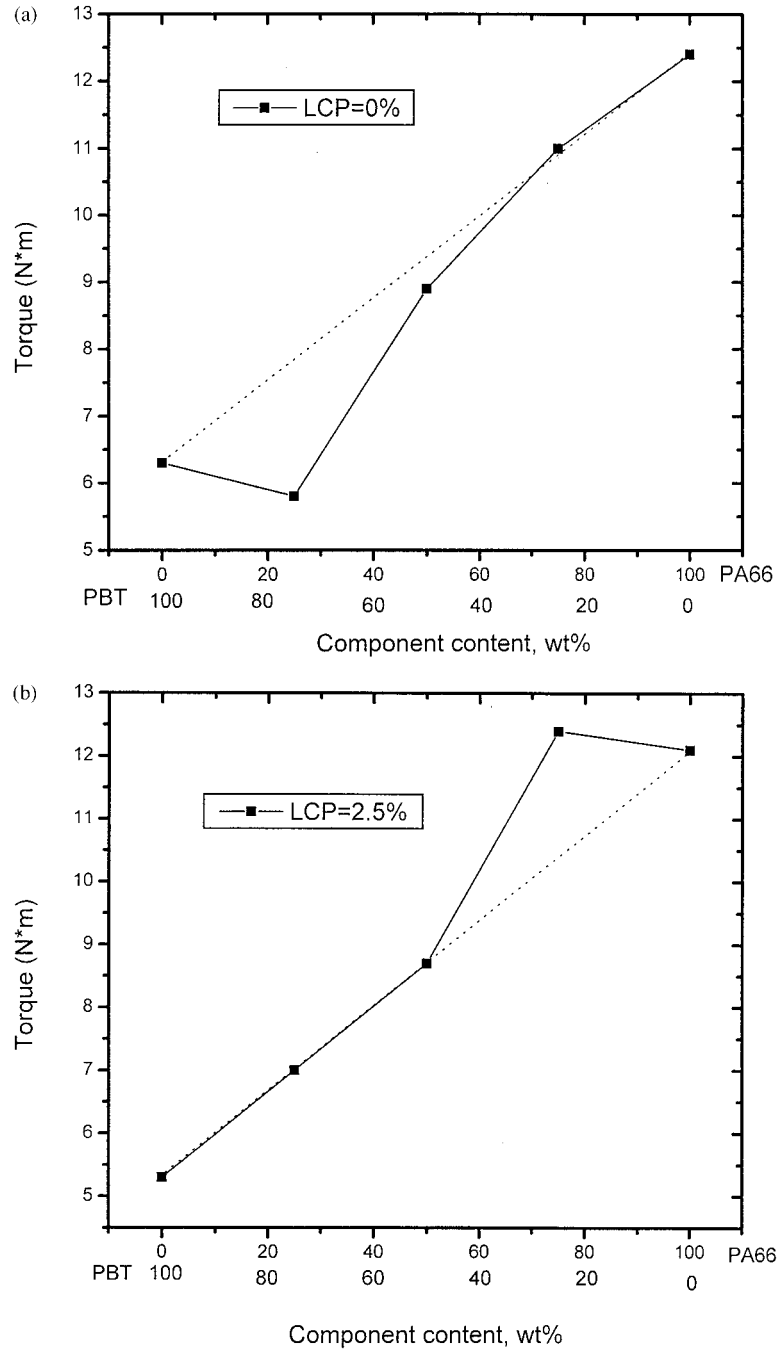
$$T_m = T_m^0 + T_m^0 \left( \frac{V_2}{\Delta H_2} \right) \left( \frac{RT}{V_1} \right) \chi_{12} \phi_1^2 \quad (1)$$

where  $T_m^0$  and  $T_m$  are the melting temperatures of pure PA66 or PBT and of PA66 or PBT in the blends, respectively;  $\Delta H_2/V_2$ , the heat of fusion of 100% crystalline PA66 or PBT per unit volume; and  $V_1$  and  $\phi_1$ , the molar volume and volume fraction of LCP, respectively.

From the above-mentioned melting-point depression equation, the interaction parameters  $\chi_{12}$  for the LCP–PBT and LCP–PA66 blends were determined previously, and their values are negative (Table II). This indicates that LCP is miscible with PBT and PA66 in the molten state due to the interaction of the amide and the PET flexible chain segments of the LCP with the PBT or PA66 chains via hydrogen bond and physical entanglements, respectively. When the LCP content reaches 10 wt % and above, the LCP–PBT and LCP–PA66 interactions become weaker owing to that more interlocked LCP domains link themselves via hydrogen bonding.<sup>20,21</sup>

### Torque Behavior

It is well known that the torque of a melting polymer is associated with the melt viscosity. From the torque curves versus mixing time for PBT/PA66/LCP blends, the torque curves generally show an apparent maximum during the initial loading of the pellets, and they then gradually approach a steady-state value after 5 min. Figure



**Figure 4** Relationship between the steady torque value and the PA66 content for (a) LCP = 0%, (b) LCP = 2.5%, (c) LCP = 5%, (d) LCP = 10%, (e) LCP = 15%, and (f) LCP = 25%.

2 shows representative torque curves of some of the PBT/PA66/LCP blends. The steady torque values at 5 min versus LCP content for the PBT/PA66/LCP blends are illustrated in Figure 3. An apparent maximum is observed at about 2.5–5 wt % LCP content. At higher LCP concentrations,

the viscosity of the blends decreases dramatically. La Mantia et al.<sup>29,30</sup> also observed an apparent maximum in the viscosity–concentration plots for Vectra B950/PA6.

From the literature,<sup>31</sup> polymer blends can be classified into four categories according to their

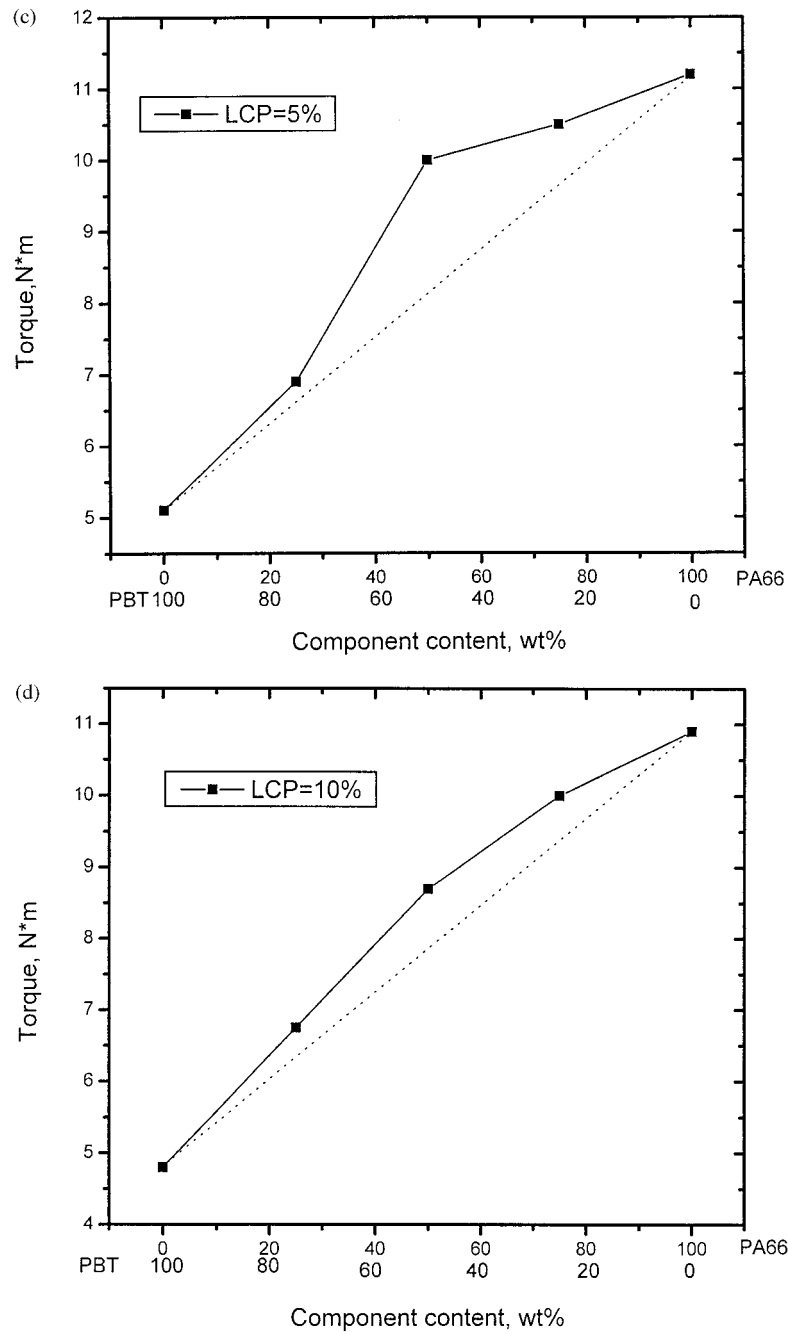


Figure 4. (Continued from the previous page.)

viscosity–composition dependence, that is, the log-additivity rule. Mathematically, it can be expressed as follows:

$$\log \eta = \sum_i C_i \log \eta_i \quad (2)$$

where  $C_i$  and  $\eta_i$  are the weight fraction and the viscosity of component  $i$ , respectively. The four classes of polyblends are

1. Additive blends (ABs), whose melt viscosities follow eq. (2);
2. Positive deviation blends (PDBs), whose



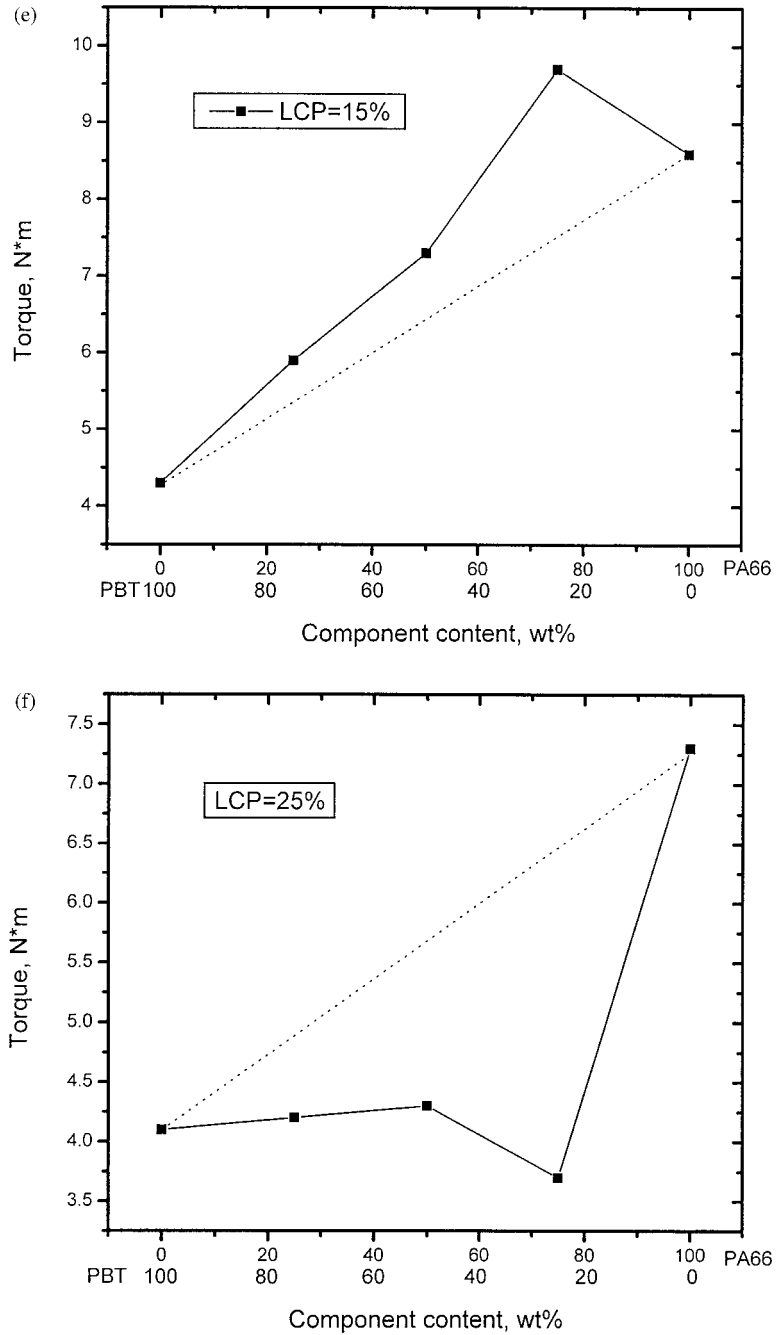


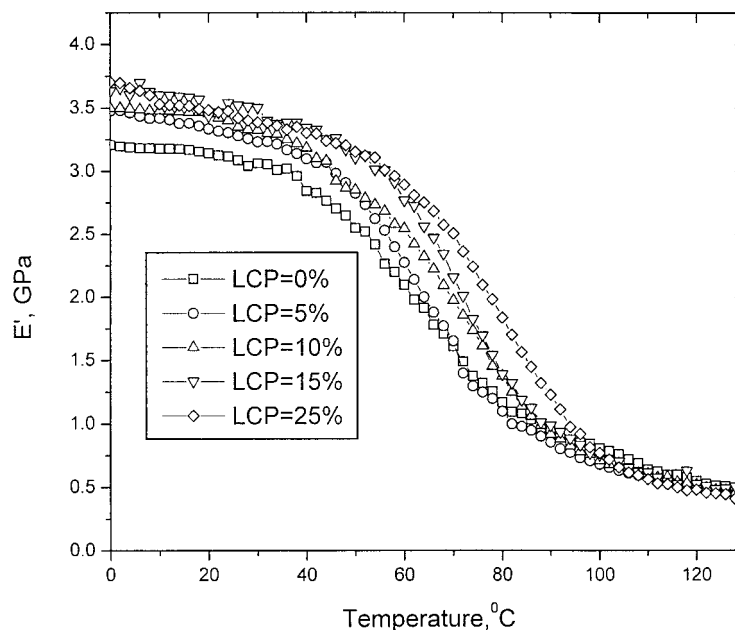
Figure 4. (Continued from the previous page.)

- viscosities are higher than the values predicted by eq. (2);
- 3. Negative deviation blends (NDBs), whose viscosities are lower than the values predicted by eq. (2); and
- 4. Positive-negative deviation blends (PNDBs), whose viscosities can be smaller

and higher than the values of eq. (2), dependent on blend compositions.

In the present study, the torque versus composition curves are shown in Figure 4(a-f). The interesting feature is that PBT/PA66 blends satisfy the NDB criterion [Fig. 4(a)], while the PBT/





**Figure 5** Variation of the storage modulus with temperature for PBT/PA66 50/50 and ternary PBT/PA66/LCP blends containing various LCP contents.

PA66/LCP blends containing LCP up to 15 wt % are PDB [Fig. 4(b–e)]. When the LCP content reaches 25 wt %, the PBT/PA66/LCP blends reverse to NDB. The PDB behavior observed in ternary PBT/PA66/LCP blends is suggested to be associated with the interactions between the ABA segment of the LCP and PA66 or PBT molecules via hydrogen bonding and physical entanglements. In this respect, the usual chain slip between the LCP and matrix is replaced by these specific interactions, leading to an increase in melt viscosity for blends containing lower LCP contents. As the LCP content is increased above 25 wt %, the number of LCP domains increases accordingly, and such domains act as processing aids for the PBT/PA66 matrix.

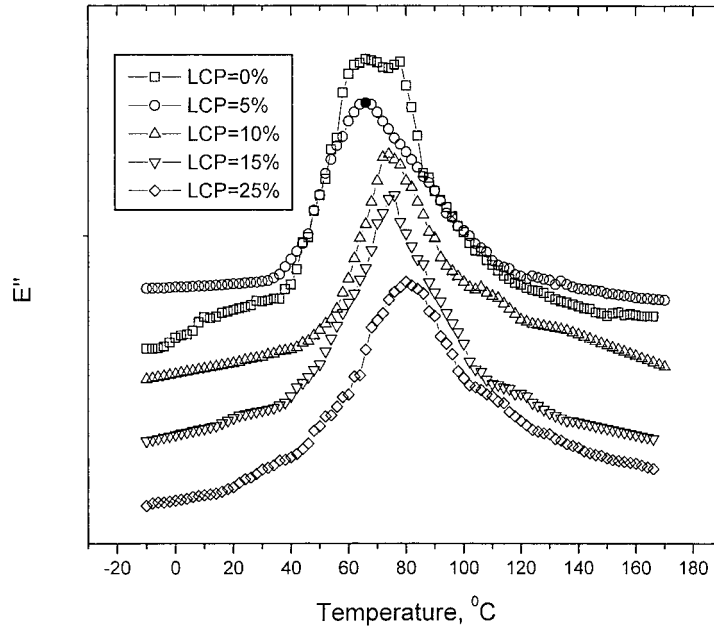
#### Dynamic Mechanical Properties

Figure 5 shows the variation of the storage modulus with temperature for some PBT/PA66/LCP blends where the PBT/PA66 ratio is maintained at equal weight fractions. Apparently, the storage modulus of the binary PBT/PA66 50/50 specimen is considerably lower than that of the ternary PBT/PA66/LCP blends. Moreover, the storage modulus of the blends appears to increase slightly with increasing LCP concentration. Figure 6 shows the loss modulus versus temperature for

these specimens. It can be seen that the PBT/PA66 50/50 blend exhibits a broad peak due to the overlapping of two peaks. These two peaks are related to the glass transition temperatures ( $T_g$ 's) of PA66 and PBT and are located at about 60 and 80°C, respectively. When the LCP is added to the PBT/PA66 blend, a weak and broad peak appears at ~100°C and is assigned as the glass transition of LCP. The  $T_g$  peaks of the PBT/PA66/LCP blends are sharper than those of the binary blends. They tend to shift to the high-temperature region with increasing LCP content. This indicates that the compatibility between the PBT and PA66 phases is improved due to the LCP addition.

#### Mechanical Properties

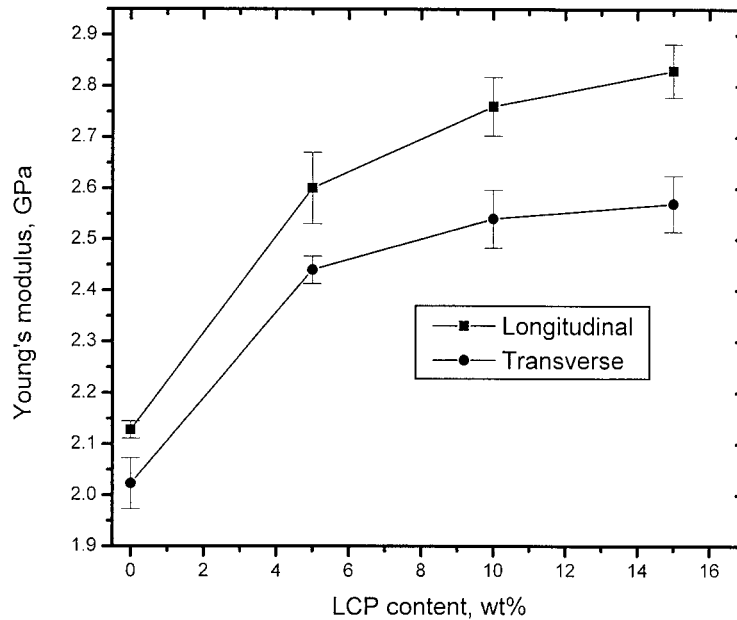
The variation of the Young's modulus and tensile strength with the LCP content for the ternary PBT/PA66/LCP samples in both longitudinal and transverse directions are shown in Figures 7 and 8. Apparently, the Young's modulus and tensile strength of the longitudinal blends are much higher than those of the transverse samples. This is typical behavior of the composites reinforced with LCP. It can also be seen from these figures that the addition of only 5 wt % LCP to the binary PBT/PA66 blend results in an increase in both the



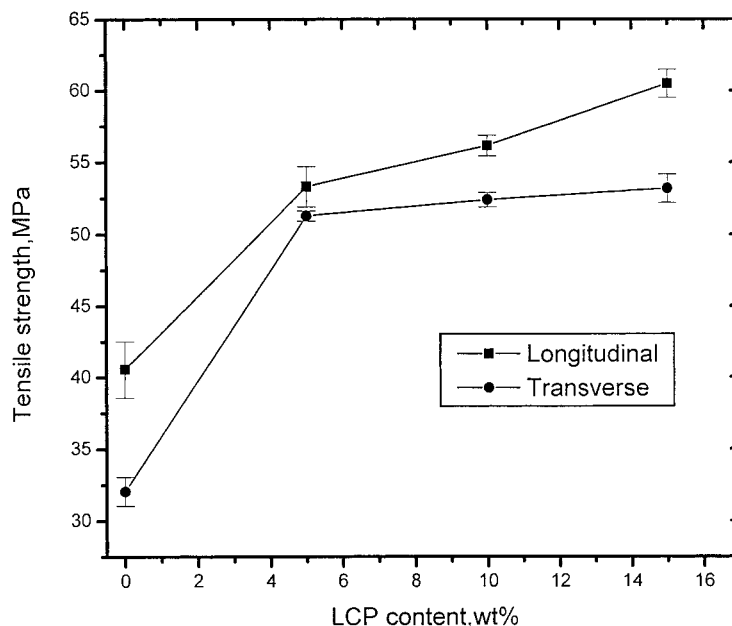
**Figure 6** Loss modulus versus temperature for PBT/PA66 50/50 and ternary PBT/PA66/LCP blends containing various LCP contents.

Young's modulus and tensile strength of the longitudinal and transverse samples. When the LCP content is >5 wt %, the stiffness and tensile strength of the longitudinal and transverse samples tend to increase more slowly with increasing

LCP content. Figure 9 shows the impact strength versus the LCP content for both longitudinal and transverse specimens of PBT/PA66/LCP blends. This figure indicates that the impact strengths of these composites increase with increasing LCP



**Figure 7** Young's modulus versus LCP content for PBT/PA66 50/50 and ternary PBT/PA66/LCP blends.



**Figure 8** Tensile strength versus LCP content for PBT/PA66 50/50 and ternary PBT/PA66/LCP blends.

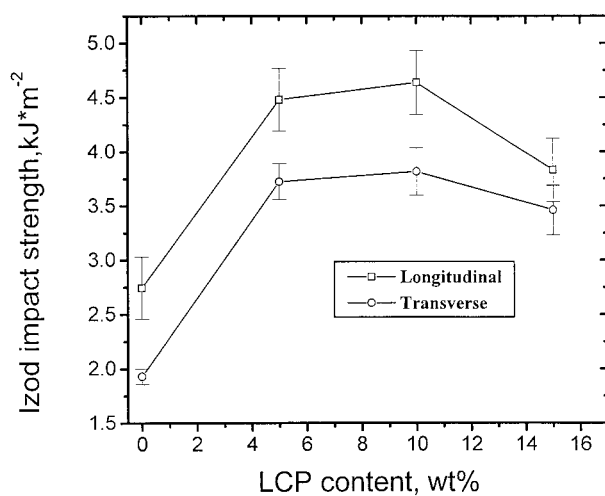
content up to 10 wt %. Thereafter, they decrease with increasing LCP content. From these results, it is clear that the addition of 5 wt % LCP enhances the miscibility and interfacial adhesion between the PBT and PA66 phases, thereby greatly improving the mechanical stiffness, strength, and impact toughness of the PBT/PA66/

LCP composites. As the LCP content further increases to 15 wt %, the PBT–LCP and PA66–LCP interactions become weaker as discussed above, leading to a decrease in the impact strength of the ternary *in situ* composites.

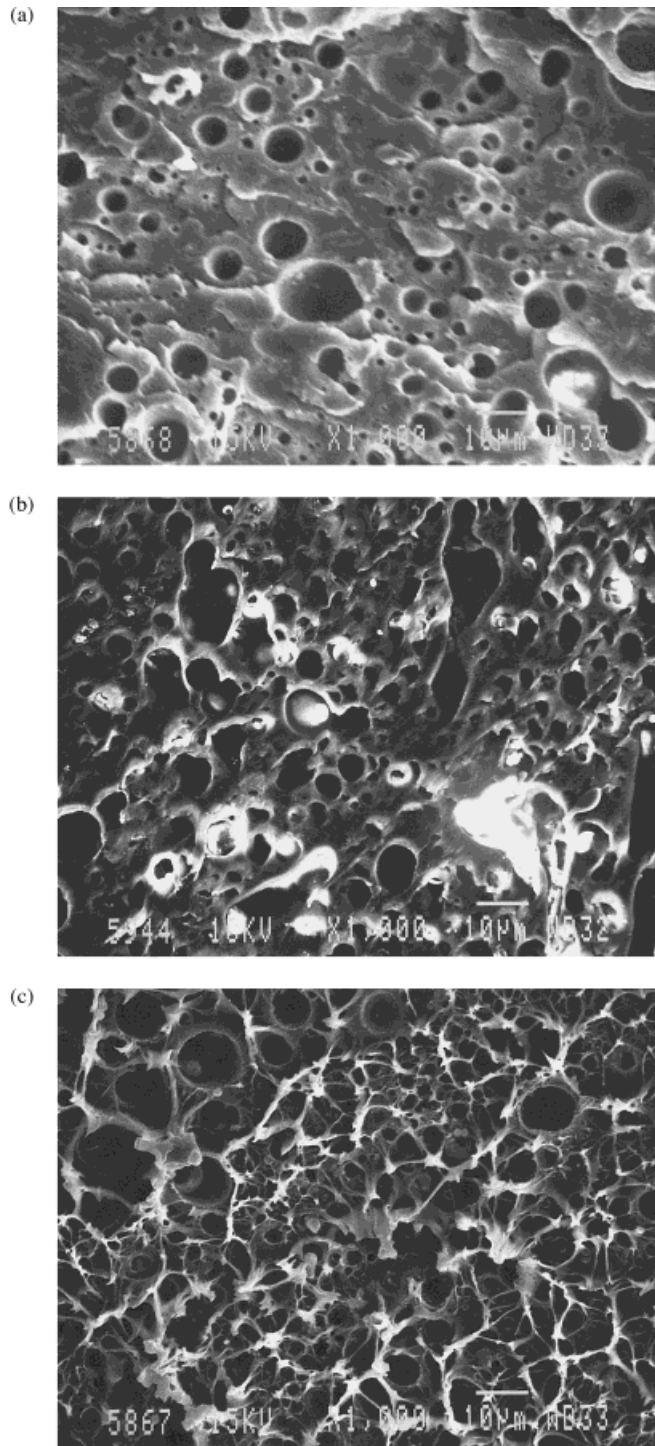
### Morphology

Figure 10(a–c) shows SEM morphologies of the etched cryo-fractured surfaces of the extruded PBT/PA66 50/50 blend and its ternary blend samples containing different LCP content. It can be seen that the PA66 phase is dispersed in the PBT matrix as spherical domains. The sizes of the PA66 spherical domains ranges between 1 and 20  $\mu\text{m}$  [Fig. 10(a)]. When 5 wt % LCP is added, the PA66 domains are more uniformly distributed in the PBT matrix, and some LCP ellipsoids can be observed in the micrograph [Fig. 10(b)]. When the LCP content is increased to 15 wt % [Fig. 10(c)], the morphological structure of the ternary PBT/PA66/LCP blend is characterized by a network structure.

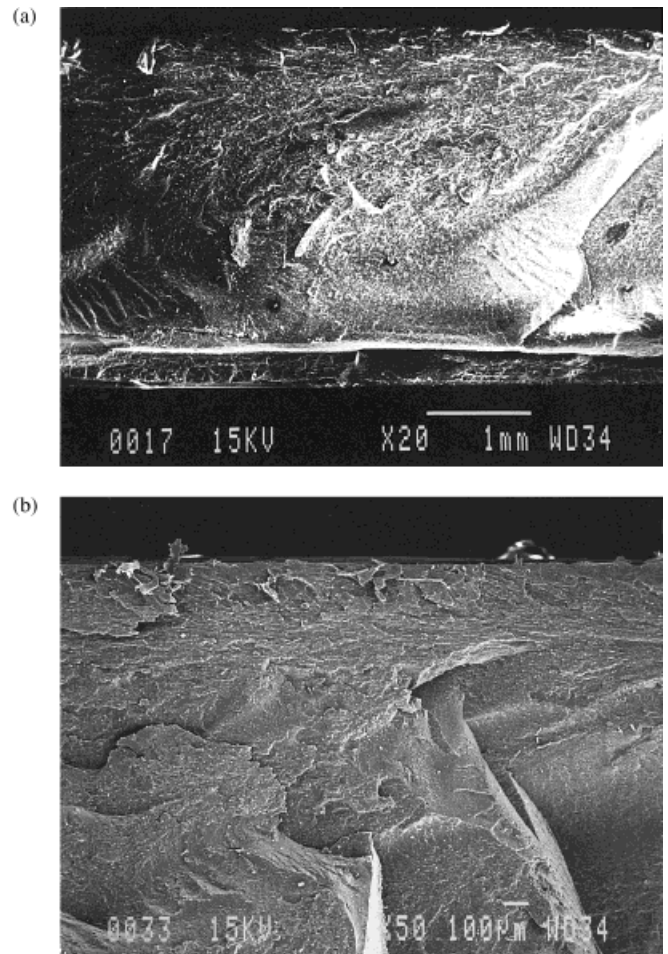
Figure 11(a,b) shows low-magnification SEM fractographs of the injection-molded PBT/PA66/LCP composite specimens. From these figures, it can be seen that the ternary blend containing 5 wt % LCP exhibits a typical skin–core structure,



**Figure 9** Izod impact strength versus LCP content for PBT/PA66 50/50 and ternary PBT/PA66/LCP blends.



**Figure 10** SEM micrographs showing the fractured surfaces of (a) PBT/PA66 50/50, (b) PBT/PA66/LCP 47.5/47.5/5, and (c) PBT/PA66/LCP 42.5/42.5/15 blends after being etched in formic acid.



**Figure 11** Low-magnification SEM fractographs of the injection-molded (a) PBT/PA66/LCP 47.5/47.5/5 and (b) PBT/PA66/LCP 42.5/42.5/15 composites.

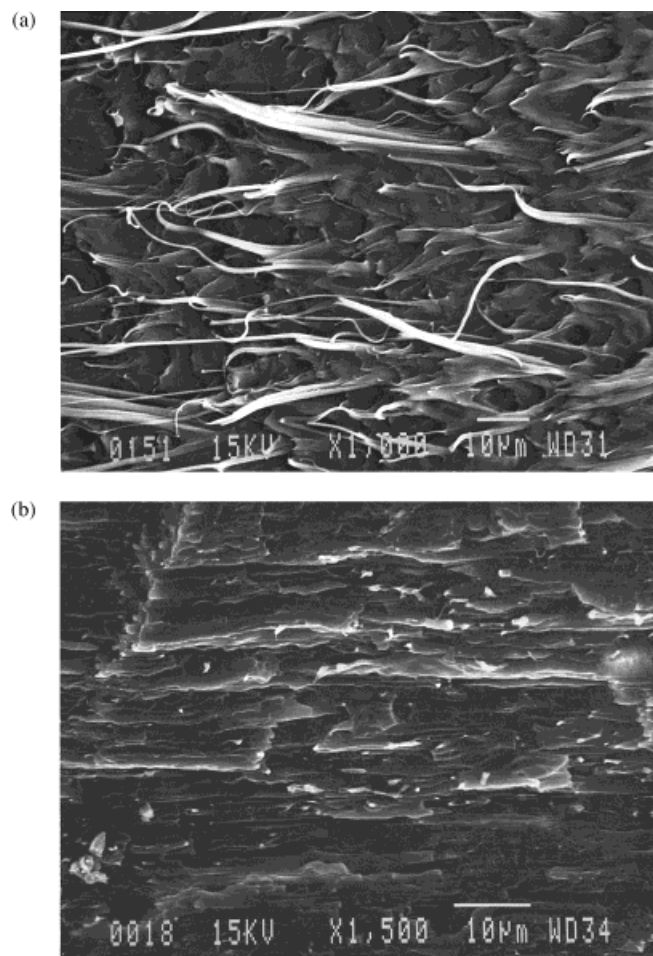
and such a structure disappears with the addition of 15 wt % LCP. Figure 12(a,b) shows higher-magnification views of the PBT/PA66/LCP 47.5/47.5/5 blend. It is apparent that fine and elongated LCP fibrils are well developed in the skin region of the composite with a low LCP content, that is, 5 wt % [Fig. 12 (a)]. Moreover, LCP domains begin to deform into fibrils in the core region. Such fibril formation is responsible for the high mechanical strength, stiffness, and impact toughness of this ternary specimen compared to binary PBT/PA66 blend as discussed above. Figure 13(a,b) shows SEM micrographs of the skin and core sections of the injection-molded PBT/PA66/LCP 45/45/10 composites. Apparently, fine LCP fibrils are developed in the both the skin and core sections of this specimen. In this respect, the



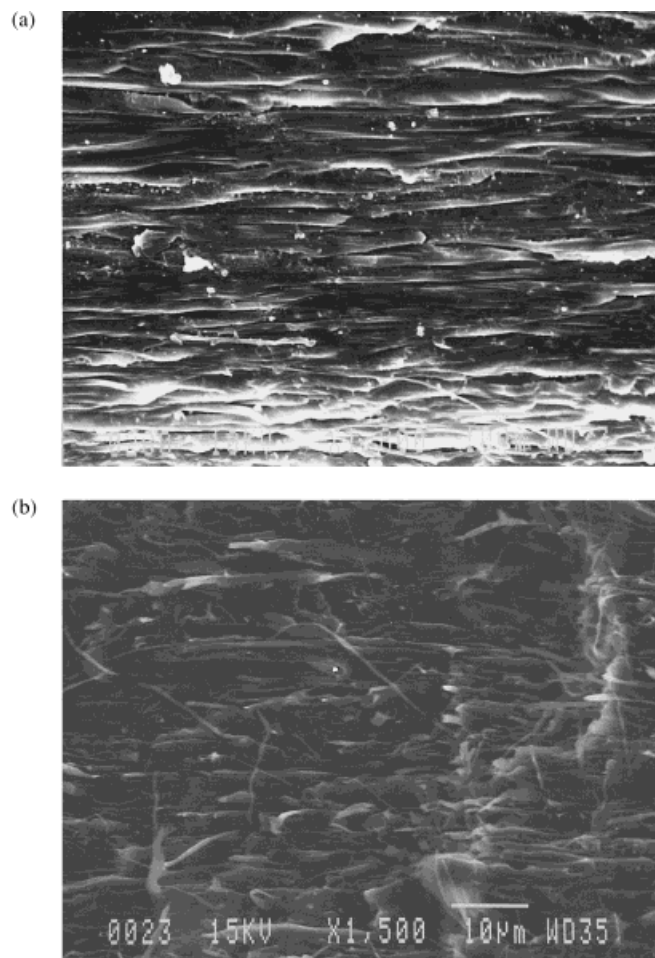
mechanical properties of this specimen are further improved (Figs. 7–9). Figure 14 shows an SEM micrograph of the core section of the composite containing 15 wt % LCP. Some LCP fibrils are detached from the matrix as evidenced by the grooved traces in the micrograph. This indicates that the adhesion between the LCP and the matrix becomes poorer with the incorporation of 15 wt % LCP.

## CONCLUSIONS

Ternary PBT/PA66/LCP blends reinforced with ABA30/PET liquid crystalline copolyesteramide were prepared, and their morphology, rheology,



**Figure 12** Higher-magnification SEM fractographs for (a) the skin and (b) core sections of injection-molded PBT/PA66/LCP47.5/47.5/5 blend.



**Figure 13** SEM fractographs of (a) the skin and (b) core sections of injection-molded PBT/PA66 /LCP 45/45/10 composites.

and mechanical properties were systematically investigated. The results show that the melting temperatures of the PBT and PA66 phases tend to decrease with increasing LCP concentrations. The torque curves versus PA66 composition show that the binary PBT/PA66 blends can be termed as NDBs. The ternary PBT/PA66/LCP blends are classified as PDBs, while the blends with LCP  $\geq 25$  wt % exhibit NDB behavior. The tensile tests show that the stiffness and tensile strength of ternary *in situ* composites are generally improved with increasing LCP content, illustrating the reinforcement of the LCP. The impact strength of the ternary composites initially increases owing to the toughening by the LCP. Then, it deteriorated when the LCP content was higher than 10 wt %.



**Figure 14** SEM fractographs of injection-molded PBT/PA66/LCP42.5/42.5/15 composite.

This work was supported by a Strategic Grant (No. 7000607), City University of Hong Kong.

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