Mechanical Reinforcement of Poly(1-butene) Using Polypropylene-Grafted Multiwalled Carbon Nanotubes

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ABSTRACT: The mechanical properties of poly(1-butene) reinforced by pristine multiwalled carbon nanotubes (MWNTs) and polypropylene-grafted MWNTs (PP-g-MWNTs) were evaluated. The incorporation of pristine MWNTs to PB led to an improvement in stiffness, but not in strength, ductility, and toughness. In comparison, PP-g-MWNTs were able to improve the stiffness, strength, and toughness of PB significantly, without compromising the ductility. The mechanical properties of PB improved with increasing amount of PP-g-MWNTs up to an effective MWNT content of 1.5 wt%. Further increase in the effective MWNT content led to a downturn in mechanical properties due to the existence of MWNTs bundles as observed by microscopy. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 113: 1165–1172, 2009

Key words: poly(1-butene); carbon nanotubes; mechanical properties

INTRODUCTION

The use of single-walled and multiwalled carbon nanotubes (SWNTs and MWNTs, respectively) as reinforcing fillers for polymers has received considerable interest in recent years.[1–7] The extremely high elastic modulus (about 1 TPa) and strength (about 200 GPa) of carbon nanotubes (CNTs) are envisaged to produce polymer composites with greatly improved mechanical properties. However, the incorporation of pristine CNTs generally enhances the stiffness and strength of polymers, but not the ductility and toughness. In many cases, the ductility of polymers is greatly reduced. The less than satisfactory mechanical performance of polymer/CNTs composites is mainly due to the tendency of CNTs to aggregate and the weak polymer-CNT interfacial adhesion. The aggregation of CNTs prevents the homogeneous dispersion of individual tubes, and thus stress cannot be evenly distributed in the polymer matrix. The weak polymer-CNT interfacial adhesion prevents an efficient load transfer from the matrix to the nanotubes. A number of recent studies have shown that polymer-grafted CNTs are highly effective reinforcing fillers for polymers.[8–19] The polymer chains grafted onto CNTs reduce the tendency of CNTs to aggregate. Load can be transferred more effectively from the matrix to CNTs through polymer chains grafted onto CNTs. As a result, the mechanical performances of polymers are greatly improved on the addition of polymer-grafted CNTs.

The mechanical properties of polyethylene (PE)/CNTs[10,20–25] and polypropylene (PP)/CNTs composites[15,26–33] have been studied extensively. On the other hand, poly(1-butene) (PB)/CNTs composites have received scant attention. Wanjale and Jog[34,35] studied the crystallization, viscoelastic and dielectric behavior of PB/multiwalled carbon nanotubes (MWNTs) composites, but not the mechanical properties. We have studied the mechanical properties of PB reinforced by pristine MWNTs and also by polypropylene-grafted MWNTs (PP-g-MWNTs). We have found that the addition of pristine MWNTs only improved the yield stress and Young's modulus of PB marginally. However, the ductility, strength, and toughness of PB were reduced substantially. In contrast, the addition of PP-g-MWNTs was able to improve the stiffness, strength, and toughness significantly, with only a marginal reduction in the ductility of PB.

EXPERIMENTAL

Materials and preparation of composites

MWNTs (purity > 95%, diameter within 10–20 nm) produced by chemical vapor deposition were obtained from Shenzhen Nanotech Port Co. Ltd., China. PB (isotactic, $M_w \sim 570,000$) from Sigma-

Aldrich Co Ltd. was used as the matrix. The preparation of PP-*g*-MWNTs was reported earlier.[15] Briefly, polypropylene-*graft*-maleic anhydride (PP-MA) (0.6 wt % MA) was melt blended with amine-functionalized MWNTs in an Atlas Minimax mixer at a speed of 80 rpm at 200°C for 30 min. The reaction between anhydride and amine groups led to the grafting of PP onto MWNTs.

PP-*g*-MWNTs (MWNT content of 29 wt %) thus prepared were then melt blended with PB to obtain composites of varying MWNT contents. The blending was also carried out in the Altas Minimax mixer at a speed of 100 rpm at 220°C for 1 h. The composites were then pressed into thin films of 0.2-mm thickness first at 220°C and then at room temperature using a hydraulic press. For comparison, PB/ pristine MWNTs composites of similar MWNT contents were also prepared under the same processing condition.

Characterization

DSC measurements were made using a TA Instruments 2920 differential scanning calorimeter in a nitrogen atmosphere (flow rate = 50 mL min^{-1}). To erase the thermal history of the samples, each sample was heated to 160°C at a heating rate of 10°C min^{-1} and kept at that temperature for 10 min, and the sample was then allowed to cool to 60°C at a cooling rate of 5°C min^{-1}. After the sample was kept at 60°C for 10 min, the second heating/cooling cycle was conducted to record the heating/cooling curves. The reported melting temperature, crystallization temperature, and degree of crystallization values were the average of at least three measurements. Transmission electron micrographs were obtained using a JOEL TEM 3010F (300 kV). Scanning electron micrographs of the fractured surface of composites were obtained using a JEOL JSM-5200 SEM.

The mechanical properties of PB and various composites (dimension $25 \times 5 \times 0.2$ mm^3) were measured using an Instron Model 3345 mechanical tester at room temperature. The strain rate was 40 mm min^{-1} under a load of 1 kN. At least five measurements were made for each composite. The dynamic mechanical properties of the composites (dimension $25 \times 5 \times 0.2$ mm^3) were measured using a TA Instruments 2980 dynamic mechanical analyzer under nitrogen using a heating rate of 3°C min^{-1} and a frequency of 1 Hz.

RESULT AND DISCUSSION

Dispersion of nanotubes in polymer matrix

To ensure an efficient load transfer from polymer matrix to CNTs, the polymer grafted onto CNTs and the polymer matrix are to be the same[8–10,15–17] or to be miscible with each other if the two polymers are different.[11–14,19] We have earlier grafted PE[10] and PP[15] onto MWNTs by melt blending polyethylene-*graft*-maleic anhydride (PE-MA) and PP-MA, respectively, with amine-functionalized MWNTs. We did not graft PB onto MWNTs because PB-*graft*-MA is not commercially available. Several studies showed that there was a certain degree of miscibility between PP and PB, particularly when the blend contained only a small amount of either polymer (< 20%).[36,37] It was then envisaged that PP-*g*-MWNTs could be effective in enhancing the mechanical properties of PB.

For a uniform stress distribution in the composite, it is essential that CNTs are homogeneously dispersed in the polymer matrix. The dispersions of PP-*g*-MWNTs and pristine MWNTs in the PB matrix were examined by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Figure 1 shows the cryofractured surfaces of PB/ MWNTs and PB/PP-*g*-MWNTs composites, both of which had an effective MWNT content of 1.5 wt %. The nanotubes were individually dispersed in the PB/PP-*g*-MWNTs composite, whereas big bundles of nanotubes were observed in the PB/MWNTs composite. The micrographs confirm that grafting of polymers onto MWNTs helps disperse nanotubes in polymer matrices. Our previous studies have shown that it is difficult to completely disperse polymer-grafted MWNTs in polymer matrices at higher CNTs loadings.[10,11,13,15] Figure 2 shows the transmission electron micrographs of two PB/PP-*g*-MWNTs composites. For the composite with an effective MWNT content of 1.5 wt %, the transmission electron micrograph also shows that the nanotubes were individually dispersed. In comparison, in addition to individually dispersed nanotubes, bundles of nanotubes can be seen for the composite with an effective MWNT content of 2.0 wt % [Fig. 2(b)]. As will be discussed in a later section, the aggregation of nanotubes causes a downturn in the mechanical performance of the composite.

Crystallization behavior

When PB is crystallized from the melt, it adopts a tetragonal form II (melting point around 110-115°C), which is transformed to a hexagonal form I (melting point around 125-130°C) upon standing at room temperature after several days.[34,38–40] The addition of MWNTs or clay accelerates the crystallization rate of PB.[34,38–40] The DSC melting and crystallization curves of PB and various composites are shown in Figure 3. The melting temperatures (T_m), crystallization temperatures (T_c), and the degree of
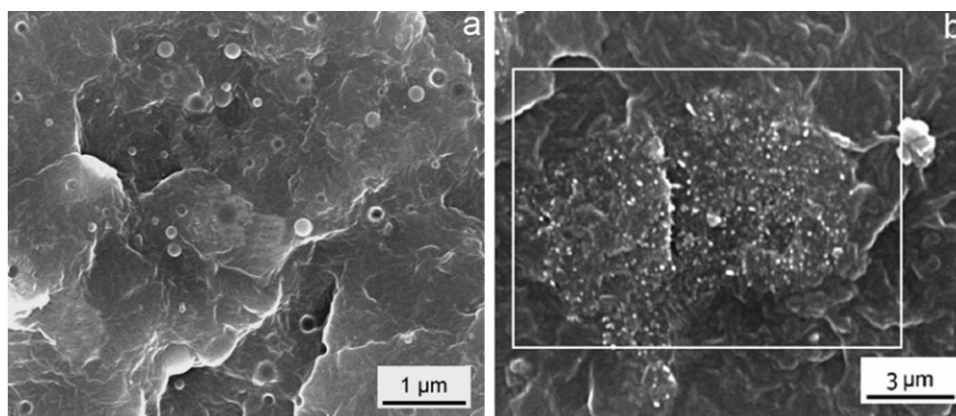
Figure 1   Scanning electron micrographs of composites. (a) PB/PP-g-MWNTs composite with an effective MWNT content of 1.5 wt %; (b) PB/MWNTs composite containing 1.5 wt % of pristine MWNTs; the presence of a large amount of nanotubes is indicated inside the rectangle.

crystallization (X_c) are summarized in Table I. X_c was calculated using the equation, viz.

$$X_c = (\Delta H / \Delta H_0) \times 100\%$$

where ΔH is the enthalpy of fusion of the sample (normalized by the weight fraction of PB in the sample) and ΔH_0 is the enthalpy of fusion of 100% crystalline PB (Form II), which is taken to be 75.2 J g^{-1}.[38,39] For PB thermally treated under the condition described earlier, a single melting peak at 110°C indicates that PB crystallized in Form II. The T_c value of PB increased significantly on the addition of pristine MWNTs or PP-g-MWNTs, and the value increased progressively with increasing MWNT or PP-g-MWNTs content. The results show that in the presence of MWNTs or PP-g-MWNTs, PB was able to crystallize at a higher temperature upon cooling from the melt, confirming that the nanotubes served

as nucleating agents. In addition, pristine MWNTs appeared to be more effective as nucleating agents than PP-g-MWNTs as shown by the higher T_c values of composites with the same MWNT contents. For example, the PB/MWNTs composite containing 2.0 wt % of MWNTs crystallized at 85.7°C, whereas the corresponding PB/PP-g-MWNTs composite crystallized at 80.0°C. In an earlier study,[12] we found that a poly(vinylidene fluoride) (PVDF)/MWNTs composite containing 1.60 wt % of MWNTs crystallized at 145.6°C, whereas a PVDF/poly(methyl methacrylate)-grafted MWNTs composite with an effective MWNT content of 1.66 wt % crystallized at 142.7°C. The results indicate that the nucleation ability of MWNTs is somehow reduced on the grafting of polymers. Other studies have shown that CNTs produced less significant effects on T_m and X_c than on T_c of semicrystalline polymers.[10,22,27] Similarly, the effects of MWNTs and PP-g-MWNTs on the T_m and X_c values of PB are less significant than those on T_c.
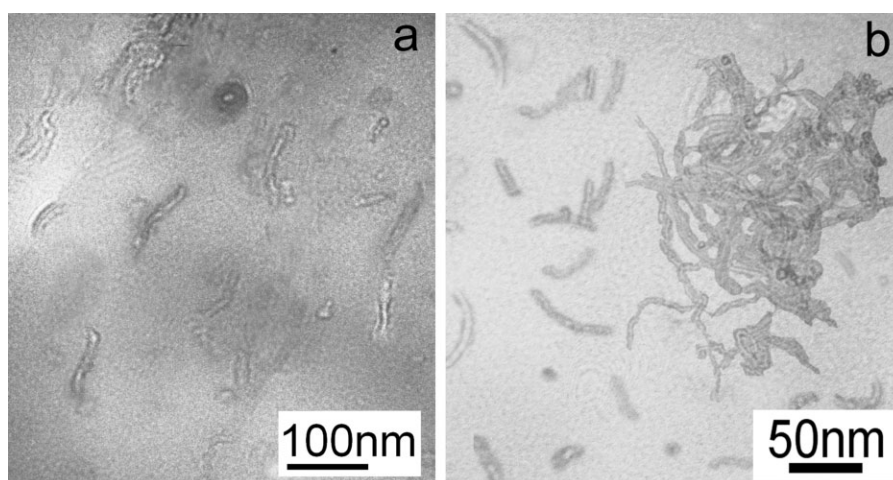

Figure 2   Transmission electron micrographs of PB/PP-g-MWNTs composites with effective MWNT contents of: (a) 1.5 wt % and (b) 2.0 wt %.
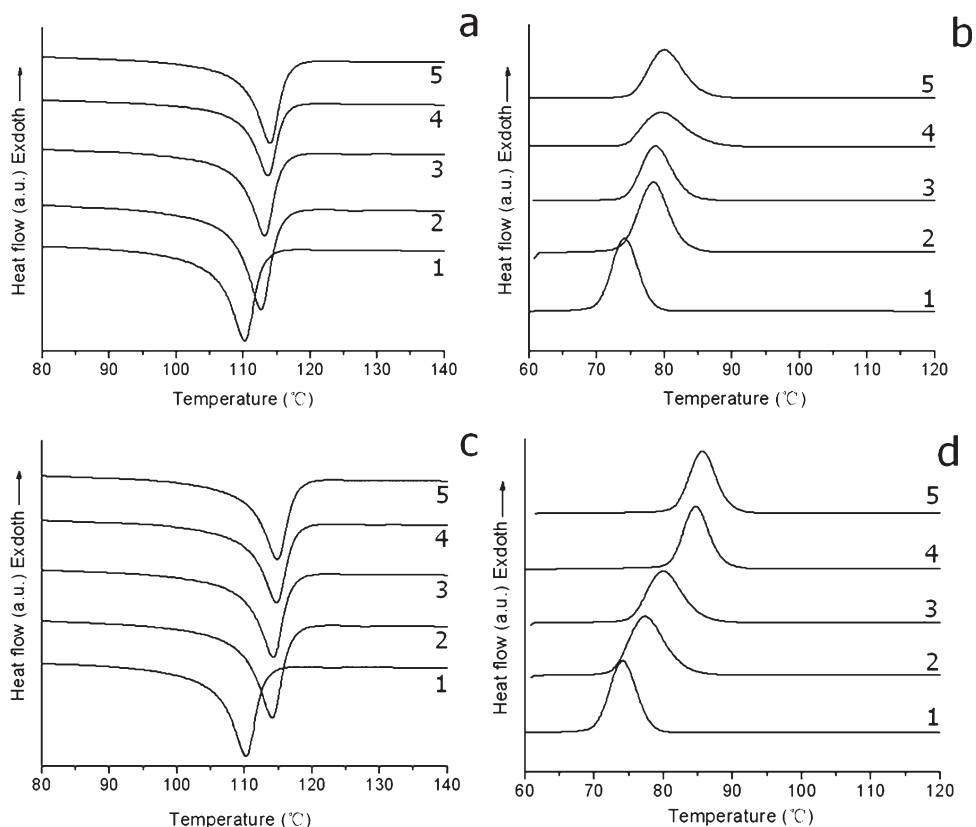
Figure 3 (a) Melting curves of PB/PP-*g*-MWNTs composites; (b) crystallization curves of PB/PP-*g*-MWNTs composites; (c) melting curves of PB/MWNTs composites; (d) crystallization curves of PB/MWNTs composites. MWNT content: (1) 0%; (2) 0.5%; (3) 1.0%; (4) 1.5%; and (5) 2.0%.

The T_m value increased slightly with increasing MWNT or PP-*g*-MWNTs content. On the other hand, the X_c value decreased with increasing MWNTs or PP-*g*-MWNTs contents. This is because a more rapid crystallization means less time for the polymer chains to attain a high degree of order. Therefore, MWNTs and PP-*g*-MWNTs accelerate the crystallization rate but reduce the degree of crystallization

slightly. The degree of crystallinity of PB was reduced more by PP-*g*-MWNTs than by MWNTs.

Mechanical properties of composites

The storage modulus (E') determined by dynamic mechanical analysis is a measure of energy stored elastically and also the stiffness of the polymer. Wanjale and Jog[35] reported that pristine MWNTs did not produce a significant effect on the E' value of PB in the glassy state, but the effect was more significant in the rubbery state. At 40°C, the storage modulus of PB was increased by 34% upon the incorporation of 3 wt % pristine MWNTs.[35] The E' versus temperature curves of PB and various composites are shown in Figure 4. The storage modulus of PB was improved upon the addition of MWNTs. The improvement in E' was 20% at -50°C for the composite containing 1.5 wt % MWNTs. For the PB/PP-*g*-MWNTs composite with an effective MWNT content of 1.5 wt %, the storage modulus of PB at −50°C was improved by 71%. The improvement in E' in the rubbery region is also impressive. For the same PB/PP-*g*-MWNTs composite, the E' value of PB at 40°C was raised by 94%. Figure 5 shows a

TABLE I
Melting Temperature (T_m), Crystallization Temperature (T_c) and Degree of Crystallization (X_c) of PB and Composites

MWNT content	T_m (°C)	T_c (°C)	X_c (%)
0%	110.2	74.1	47.6
0.5%_AR	114.2	77.5	47.3
1.0%_AR	114.3	80.0	46.4
1.5%_AR	114.8	84.7	45.2
2.0%_AR	114.9	85.7	45.1
0.5%_M	112.7	78.4	44.6
1.0%_M	113.3	78.7	43.9
1.5%_M	113.8	79.6	43.8
2.0%_M	114.0	80.0	43.7

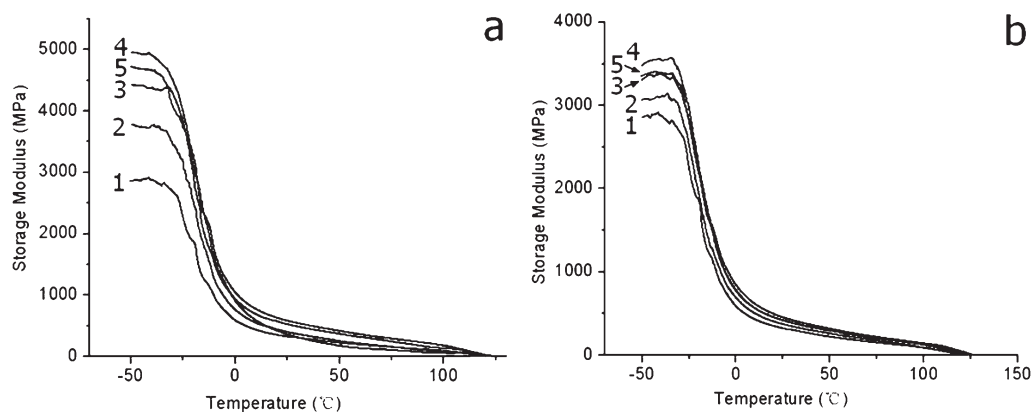AR denotes pristine MWNTs; M denotes PP-*g*-MWNTs.

Figure 4 Storage modulus-temperature curves: (a) PB/PP-*g*-MWNTs composites; (b) PB/MWNTs composites. MWNT content: (1) 0%; (2) 0.5%; (3) 1.0%; (4) 1.5%; and (5) 2.0%.

downturn in the E' value when the effective MWNT content of the PB/PP-*g*-MWNTs composite was further increased to 2.0 wt %. Such a downturn can be attributed by the inhomogeneous dispersion of nanotubes as shown by scanning and transmission electron micrographs as discussed in the earlier section. For PB reinforced by organoclay (montmorillonite ion-exchanged with octadecylammonium ions), its E' value at $-50°C$ was increased by 43, 51, and 140% on the incorporation of 3, 5, and 7 wt % of clay, respectively, and by 9, 20, and 31% at 50°C.[38] The ability of PP-*g*-MWNTs in improving the storage modulus of PB is comparable to that of the organoclay.

The stress–strain curves of PB and composites are shown in Figure 5, and the tensile properties are summarized in Table II. The incorporation of pristine MWNTs increased the Young's modulus and yield stress of PB, but not the tensile strength, ultimate strain, and toughness. The decreases in ultimate strain and toughness are particularly significant. As mentioned earlier, the addition of pristine MWNTs to polymers generally improves the stiffness and strength at the expense of ductility and

toughness. This is the common problem encountered in polymer composites containing CNTs or organoclay. In comparison, the mechanical properties of PB were significantly improved by the addition of PP-*g*-MWNTs. The Young's modulus, yield stress, tensile strength, and toughness of PB were improved, without compromising the ductility. In general, the mechanical properties improved with increasing PP-*g*-MWNTs content up to 1.5 wt %. At an effective MWNT content of 1.5 wt %, the yield stress, tensile strength, toughness, and Young's modulus of PB were increased by 83, 61, 52, and 72%, respectively, and the ultimate strain was reduced by only 3%. Further addition of PP-*g*-MWNTs to 2.0 wt % brought along a slight increase in Young's modulus, but decreases in the other mechanical properties. Interestingly, we have also observed downturns in mechanical properties of PE/PE-*g*-MWNTs,[10] PP/PP-*g*-MWNTs,[15] and poly(ethylene oxide)/phenoxy-grafted MWNTs composites[11] when the effective MWNT content of the composite was increased from 1.5 wt % to 2.0 wt %. For the three composite systems mentioned earlier, the downturns in the
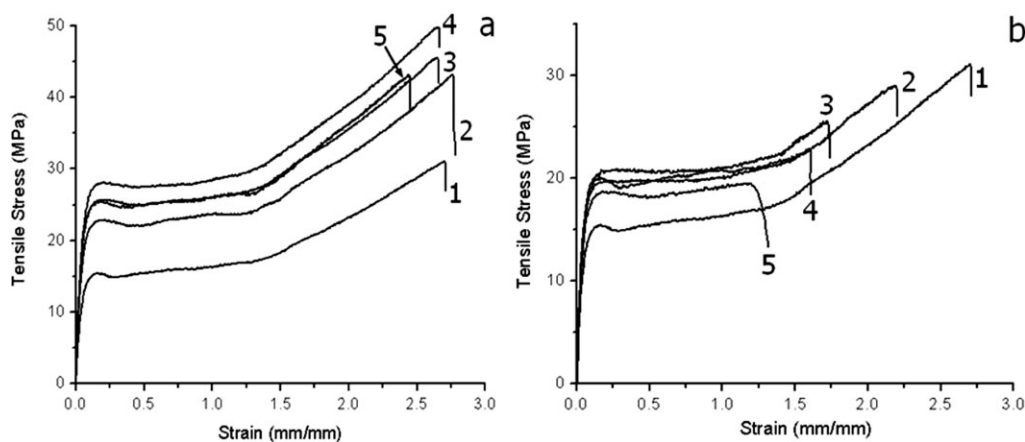

Figure 5 Stress–strain curves: (a) PB/PP-*g*-MWNTs composites; (b) PB/MWNTs composites. MWNT content: (1) 0%; (2) 0.5%; (3) 1.0%; (4) 1.5%; and (5) 2.0%.

TABLE II
Tensile Properties of PB and Composites

MWNT content	Yield stress (MPa)	Tensile strength (MPa)	Ultimate strain (mm/mm)	Toughness (MJ/m³)	Young's modulus (MPa)
0%	15.4 ± 1.3	31.1 ± 3.2	2.70 ± 0.28	53.3 ± 5.1	380 ± 36
0.5%_M	23.0 ± 1.7 (49)	43.4 ± 3.7 (40)	2.76 ± 0.23 (2)	77.9 ± 6.8 (46)	593 ± 48 (56)
1.0%_M	25.8 ± 1.7 (68)	45.5 ± 4.3 (46)	2.64 ± 0.27 (−2)	81.1 ± 7.3 (52)	653 ± 56 (72)
1.5%_M	28.2 ± 1.6 (83)	50.1 ± 4.6 (61)	2.65 ± 0.28 (−3)	88.9 ± 8.5 (67)	726 ± 75 (91)
2.0%_M	25.2 ± 1.8 (64)	43.1 ± 4.5 (39)	2.44 ± 0.25 (−10)	72.0 ± 7.6 (35)	777 ± 78 (104)
0.5%_AR	20.0 ± 2.1 (30)	29.0 ± 1.9 (−7)	2.18 ± 0.37 (−19)	47.8 ± 4.6 (−10)	397 ± 38 (4)
1.0%_AR	20.8 ± 1.8 (35)	25.6 ± 2.1 (−18)	1.73 ± 0.16 (−36)	36.7 ± 3.3 (−31)	422 ± 45 (11)
1.5%_AR	19.7 ± 2.3 (28)	22.8 ± 2.5 (−27)	1.61 ± 0.23 (−40)	32.0 ± 3.2 (−40)	447 ± 42 (18)
2.0%_AR	18.7 ± 1.8 (21)	19.5 ± 1.7 (−37)	1.19 ± 0.09 (−56)	23.8 ± 2.1 (−55)	431 ± 45 (13)

Value in parentheses represents percentage increase/decrease when compared to PB.
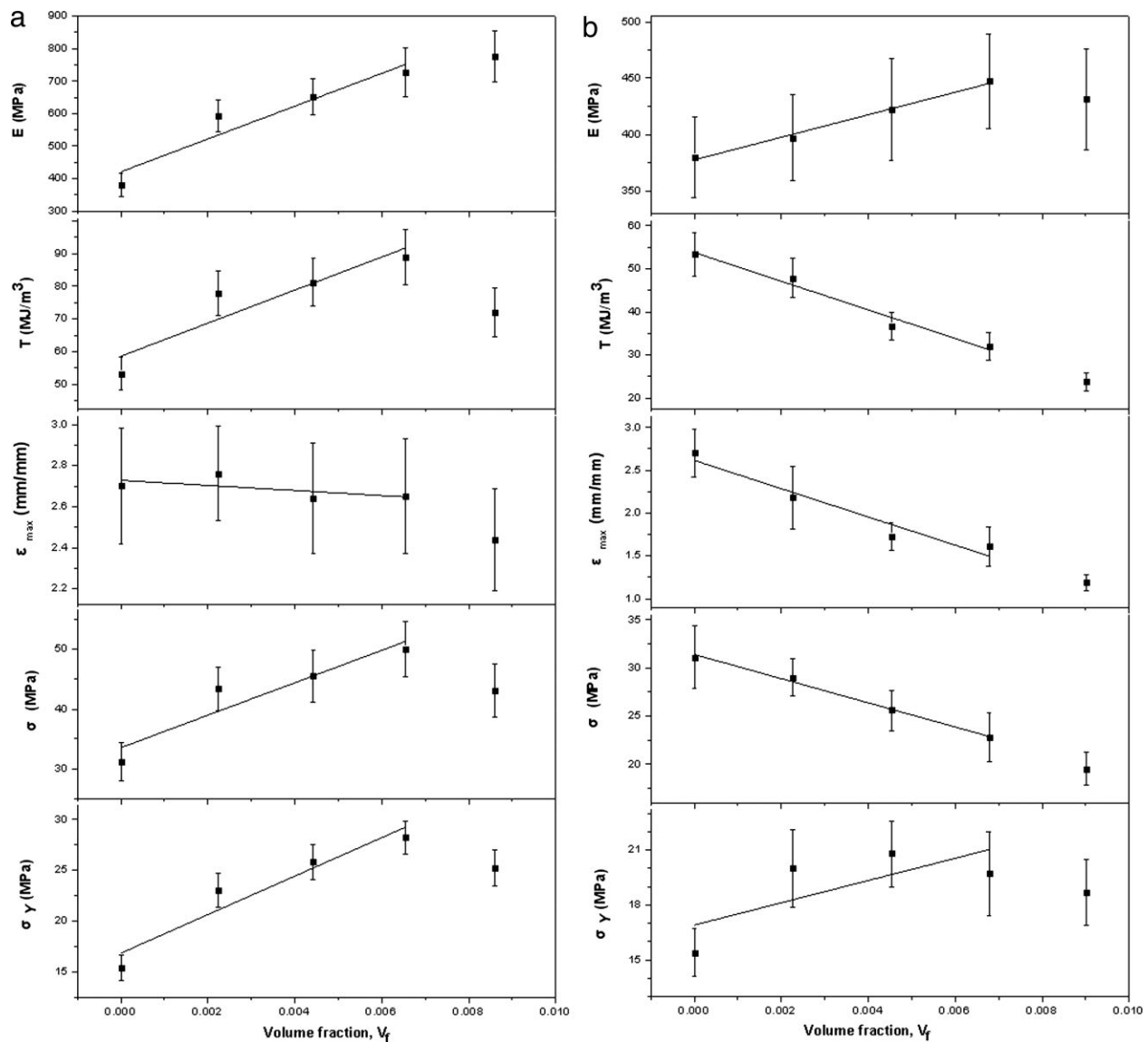AR: pristine MWNTs; M: PP-g-MWNTs.

Figure 6 Plots of tensile property as a function of volume fraction of MWNTs. (a) PB/PP-g-MWNTs composites; (b) PB/MWNTs composites.

TABLE III
Reinforcement Values of Composites

	PB/MWNTs	PB/PP-g-MWNTs
dE/dV_f (GPa)	10 ± 6	50 ± 11
$d\sigma_y/dV_f$ (GPa)	0.61 ± 0.4	1.9 ± 0.4
$d\sigma/dV_f$ (GPa)	-1.3 ± 0.1	2.7 ± 0.7
$d\varepsilon_b/dV_f$ (mm/mm)	-164 ± 29	-12 ± 10
dT/dV_f (GJ/m^3)	-3.3 ± 0.4	51 ± 11

E: Young's modulus; σ_y: yield stress; σ: tensile strength; ε_b: ultimate strain; T: toughness; densities of PB and MWNTs are taken to be 0.91 and 2.0 g cm^{-3}, respectively.

mechanical properties were accompanied by the observation of some degrees of aggregation of CNTs. As discussed earlier, bundles of CNTs were observed by TEM for the PB/PP-g-MWNTs composite with an effective MWNT content of 2.0 wt %. The present series of studies thus shows that polymer-grafted MWNTs are much more effective than pristine MWNTs in enhancing the mechanical properties of polymers. However, we and other researchers[17,18] have found that the addition of a large amount of polymer-grafted MWNTs does not necessarily bring along further improvements in mechanical properties due to the dispersion problem.

Figure 6 shows various plots of tensile property versus volume fraction of MWNTs in the composite. The slope of the initial portion of the plot, dX/dV_f, is the rate change of a particular tensile property X with volume fraction of MWNTs. The dX/dV_f values have been used to compare the effectiveness of reinforcement of MWNTs.[9,13,17,18] Table III summarizes the dX/dV_f values of PB/MWNTs and PB/PP-g-MWNTs composites. Once again, the dX/dV_f values show that the addition of pristine MWNTs only enhances the Young's modulus (E) and yield stress (σ_y) of PB, but the ductility, toughness, and strength are greatly reduced. Except for the ultimate strain, the other reinforcement values are positive for PB reinforced by PP-g-MWNTs. In terms of dE/dV_f and $d\sigma_y/dV_f$, PP-g-MWNTs are five and three times more effective than pristine MWNTs.

Causin et al.[40] have reported the effect of an organoclay, Cloisite 15A, on some mechanical properties of PB. For a clay content of 5 wt %, the flexural modulus of PB was increased by 83%, but the tensile strength and the ultimate strain were reduced by 15 and 59%, respectively. Therefore, the present study also shows that PP-g-MWNTs are more effective than organoclay in enhancing the mechanical properties of PB.

CONCLUSIONS

The mechanical properties of PB reinforced by pristine MWNTs and PP-g-MWNTs have been studied.

The incorporation of pristine MWNTs to PB led to an improvement in stiffness, but not in strength, ductility, and toughness. In comparison, PP-g-MWNTs were able to improve the stiffness, strength, and toughness of PB significantly, without compromising the ductility. The mechanical performance of PB improved with increasing amount of PP-g-MWNTs up to an effective MWNT content of 1.5 wt %. Further increase in the effective MWNT content led to a downturn in mechanical performance due to the existence of MWNTs bundles.

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