Influence of Different Functionalized Multiwall Carbon Nanotubes on the Mechanical Properties of Poly(ethylene terephthalate) Fibers

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ABSTRACT: Master batches with four different kinds of functionalized multiwall carbon nanotubes (MWCTs) were prepared through the mixing of MWCTs with poly(ethylene terephthalate) (PET) (0.01 : 0.99 w/w) in trifluoroacetic acid/dichloromethane mixed solvents (0.7 : 0.3 v/v) followed by the removal of the solvents in the mixture by flocculation. The results of scanning electron microscopy showed that a good dispersion of MWCTs in PET was achieved. The reinforced fibers were fabricated by the melt spinning of PET chips with small amounts of the master batch and then further postdrawing. The optimal spinning conditions for the reinforcement of fibers were a 0.6-mm

INTRODUCTION

Carbon nanotubes (CNTs), according to the number of graphitic sheets, are classified as multiwall carbon nanotubes (MWCTs) and single-wall CNTs. Many investigators have endeavored to fabricate advanced CNT polymer materials.^{1–13} Among them, the reinforcement of polymer fibers through the incorporation of CNTs has been reported. For example, polypropylene,¹ poly(methyl methacrylate),³ polyamide 6,9 polyamide 12,10 and polyimide11 have been fabricated by melt spinning. At the same time, polyaniline,⁴ ultrahigh-molecular-weight polyethylene,¹² and polyacrylonitrile¹³ have been fabricated by wet spinning. Poly(ethylene terephthalate) (PET) fiber has been widely used in the textile industry and in other industrial fields because of its excellent properties. However, reinforced PET fibers doped by CNTs have not yet been reported.

In the fabrication of CNT-reinforced composites, the CNT dispersion in the matrix is one of the most critical issues for the strength of CNTs. Ultrasound treatment, as the simplest and most convenient method, produces a uniform dispersion of CNTs in a polymer matrix dissolved in a certain kind of

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spinneret hole and a 250 m/min wind-up speed. Among the four master batches, the fibers obtained from PET/ master batch B made by acid-treatment had the highest enhancement of mechanical properties. For a 0.02 wt % loading of acid-treated MWCT, the breaking strength of the PET/master batch B composite fibers increased by 36.9% (from 4.45 to 6.09 cN/dtex), and the initial modulus increased by 41.2% (from 80.7 to 113.9 cN/dtex). © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 108: 2865–2871, 2008

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organic solvent, which can be removed rapidly to keep CNTs from reaggregation in the matrix. Functionalization of the CNT surface can enhance the dispersibility and sometimes even lead to substantial solubility of CNTs in various organic solvents. When the solvent is chosen properly, the CNTs and polymer matrix can be dissolved simultaneously so that a great dispersion of CNTs in the matrix will be achieved. In addition to the enhancement of dispersibility, the functionalization of CNTs can increase the strength of the interface between the CNTs and polymer matrix, and this will promote the reinforcement effect also.

As reported earlier,^{14–16} among all the solvents of PET, the mixture of trifluoroacetic acid (TFA) and dichloromethane (DCM) has the advantages of rapid dissolution at room temperature and very low degradation. Hence, this mixture was chosen as the cosolvent in this study. One of the typical CNT functionalization methods is to capitalize on the carboxylic acids that are formed on the surface of CNTs via an acid treatment.^{17–19} To prepare amide- or ester-modified CNTs, the carboxylic acids usually need to be converted into acid chlorides^{19–22} to increase the reactivity of CNTs or even directly react with long-chain amines.^{23,24} During the process, inert gas, anhydrous solvents, and a long reaction time (12–96 h) are necessary. Qin et al.²⁵ developed a concise route for fabricating functionalized CNTs that

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were soluble in many organic solvents via the esterification reaction of the carboxylate salt of CNTs and alkyl halides in the presence of a phase-transfer reagent in water. In this work, we report the preparation of master batches for fibers through the dissolution of PET and MWCTs in the cosolvent and the melt spinning of neat PET chips with master batches to make reinforced PET fibers.

EXPERIMENTAL

Materials

MWCTs with diameters of 10–30 nm and lengths of 0.5–40 μ m were purchased from Nanotech Port Co., Ltd. (Shenzhen, China). PET chips were supplied by China Luoyang Petrochemical Co. (Luoyang, China) TFA was purchased from Tianjin Fuchen Chemicals Co. (Tianjin, China) DCM, tetrabutylammonium bromide (TBAB), and 1-bromohexadecane (C₁₆H₃₃Br) were purchased from Beihua Chemicals Co., Ltd. (Beijing, China).

Preparation of the MWCT master batch

Fifty milligrams of pristine MWCTs was introduced into 20 mL of the cosolvents TFA and DCM (0.7 : 0.3 v/v), and the mixture was sonicated for 3 h until a suspension was formed. PET (4.95 g) was then added, and the suspension was stirred with a magnetic stirrer until the PET was totally dissolved (ca. 1 h). The suspension was sonicated for another 3 h, after which the mixed solution of PET and MWCTs was poured into a large amount of ethanol (ca. 200 mL) to remove the cosolvent. The resulting spongelike materials were cut into small pieces and dried in a vacuum oven, which was set at 70°C and 0.01 MPa for 12 h. The product was called master batch A, and it contained 1 wt % MWCTs in PET.

Two grams of pristine MWCTs was added to a 120-mL mixture of concentrated sulfuric acid and concentrated nitric acid (0.75 : 0.25 v/v) and sonicated for 2 h. The treated MWCTs were filtered to remove acid, washed thoroughly with distilled water, ground into powders, and dried in a vacuum oven set at 70°C and 0.01 MPa for 12 h. The product thus obtained was called MWCT-1. Master batch B was formed by the doping of 1 wt % MWCT-1 into PET with the method described for the preparation of master batch A.

MWCT-1 (0.1 g) was sonicated in a 100-mL aqueous solution of NaOH (10 mM) for 5 min to convert the carboxylic acids into sodium salts. To this black homogeneous suspension were added 0.06 g of TBAB and 2 g of $C_{16}H_{33}Br$, and the mixture was then divided into two equal portions. Both portions were refluxed under vigorous stirring, one for 12 h and the other for 36 h. The precipitates were filtered and washed thoroughly with ethanol to remove the excess C₁₆H₃₃Br, and they were dried in a vacuum oven set at 70°C and 0.01 MPa for 12 h. The products thus obtained were called MWCT-2a (refluxing time = 12 h) and MWCT-2b (refluxing time = 36 h), respectively. The MWCTs in both MWCT-2a and MWCT-2b were grafted with alkyl chains, and their only difference was the degree of esterification. Master batch C and master batch D were prepared by the doping of 1 wt % MWCT-2a and MWCT-2b to the PET following the previous method of master batch A, except that both MWCT-2a and MWCT-2b were first treated with DCM before being added to the cosolvent. The procedure of MWCT grafting with linear alkyl chains is shown in Scheme 1.

Preparation of the PET/MWCT fibers

PET chips and a certain amount (0.6–10 wt %) of a master batch were mixed and dried in a vacuum oven set at 105°C and 0.01 MPa for 6 h. The melt spinning of the mixtures was carried out at 274°C with a UPXRZ-400A melt-speedometer (Jida Kejiao Apparatus Co., Changchun, China) with a single spinneret hole (diameter = 0.6 mm/1.0 mm). The extruded fine stream was air-cooled, and it was formed into a fiber by being wound up at speeds of 150, 250, and 350 m/min. The take-up fibers subsequently were postdrawn at the maximum draw ratio on a homemade setup at 70°C. Heat setting was performed by the placement of the drawn fibers under tension in an oven at 170°C for 10 s.

Analytical methods

Fourier transform infrared (FTIR) spectra were measured on a Nicolet Nexus spectrometer (Madison, WI). Thermogravimetric analysis (TGA) was performed at a heating rate of 10° C/min under an N₂ atmosphere on a Seiko Exstar 6000 thermogravimetric analyzer (Tokyo, Japan). Scanning electron microscopy (SEM) was carried out on a JEOL 6360LV scanning electron microscope (Tokyo, Japan) at 10 kV after the coating of the samples with gold. The mechanical properties of the fibers were tested on a YG001D tensile testing machine (Taicang Appa-



Scheme 1 Procedure of MWCT grafting with linear alkyl chains.

ratus Co., Jiangsu, China). The crystallite size and degree of crystallinity of the fibers were obtained with a Rigaku DMAX/B wide-angle X-ray diffraction (WAXD) apparatus (Tokyo, Japan). The birefringence (Δn) was obtained with an XP-1 double-refracting interference microscope (Xintian Optical Instruments Co., Guiyang, China).

RESULTS AND DISCUSSION

Infrared (IR) spectra

The IR spectra of composites of functionalized MWCTs and PET are shown in Figure 1. As shown in Figure 1(a), the IR spectra of MWCT-1 exhibit a peak at 1768 cm⁻¹, indicating the existence of the nomadic carboxylic acid groups rather than the carboxylic acid groups under the hydrogen bonding, for which a peak at 1720 cm⁻¹ was expected.¹⁷⁻¹⁹ The result suggests that in our system the carboxylic acid groups were not in dimeric form through hydrogen bonding. The absorption at 3347 cm⁻¹ belongs to the hydroxyl group associated with hydrogen bonding, and it suggests that acid treatment might have introduced the hydroxyl groups into MWCTs in addition to the existing carboxylic acid groups. Figure 1(b) (MWCT-2b) shows a characteristic peak of carbonyl of ester at 1749 cm⁻¹, C-O-C stretching at 1087 and 1042 cm^{-1,26} and C-H stretching of the alkyl chains at 2918 and 2847 cm^{-1.25} The IR results confirm the formation of carboxylate ester. An additional peak at 2879 cm⁻¹, observed in Figure 1(d), might have arisen from the formation of a new bond between PET and MWCT-1.

TGA



As shown in Figure 2(a), the weight percentage of pristine MWCTs began to decrease at about 580°C,

Figure 1 FTIR of the functionalized MWCTs and PET/ MWCT composites: (a) MWCT-1, (b) MWCT-2b, (c) neat PET, (d) master batch B (PET/MWCT-1), and (e) master batch D (PET/MWCT-2b).



Figure 2 TGA curves of (a) MWCTs, (b) MWCT-1, and (c) MWCT-2b.

and this indicated that the nanotubes began to decompose at this temperature. Because the pristine MWCTs remained rather stable before 580°C, we are inclined to believe that the decreases in the weight percentage before 580°C in Figure 2(b) resulted from the decomposition of carboxylate groups in MWCT-1 rather than that of the pristine MWCTs. The two steps of decreases in the weight percentage in Figure 2(c) might correspond to the decomposition of alkyl and carboxylate groups in the sample.

SEM

An SEM image of master batch D is shown in Figure 3. As shown in the figure, a great dispersion of MWCT-2b embedded in PET was indicated. There was no entanglement of MWCTs observed, and all the MWCTs existed individually, as shown in the micrograph. In addition to the PET matrix and MWCT-2b, there were a number of grapelike materials attached to the matrix and tubes, which might have been due to the chemical modification of MWCTs.

Mechanical properties

The mechanical properties of PET/MWCT melt-spun fibers made of different master batches and with different process parameters are summarized in Tables I–VIII. The results of neat PET fibers are also shown for reference.

Tables I–IV show the results of spinning PET/ master batch A fibers with different spinneret holes and different wind-up speeds. For the neat PET fibers, there were no significant changes in the mechanical properties when the process parameters were altered. This indicates that the change in the process parameters does not affect the supermolecular structure of neat PET. In other words, if the me-

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Figure 3 SEM micrographs of the fracture surface of the PET/MWCT composites: (a) master batch D and (b) amplified image of the circled part in part a.

chanical properties of fibers are improved when MWCTs are loaded, they come from the addition of MWCTs.

Among the four processing conditions, the reinforcement of fibers was significant only with a 0.6-mm spinneret hole and 250 m/min wind-up speed, as evidenced by the improved mechanical properties shown in Table II. Under this condition, when the loading level of MWCTs was 0.04 wt %, the breaking strength was 5.25 cN/dtex and the initial modulus was 119.7 cN/dtex for the PET/master batch A fibers; they increased by 21.0 and 37.6%, respectively, versus those of the neat PET fibers. The alignment¹¹ and waviness^{27,28} of MWCTs embedded in the matrix may play very important roles in the enhancement of the mechanical properties when MWCTs serve as fillers to reinforce composites. When the pressure on molten PET is constant, the shear rate of the molten PET will increase as the spinneret hole diameter decreases, and this results in a better alignment of the embedded MWCTs in PET and thus favors the reinforcement of PET fibers. The alignment of MWCTs embedded in the PET fibers was basically stable because the molten PET was cooled and solidified immediately after extrusion from the spinneret. The waviness of the MWCTs embedded in PET will attenuate the effective strength of the MWCTs. In the wind-up process, the waviness of the MWCTs was expected to decrease, and so the reinforcement of PET fibers was expected to increase. When the wind-up speed went too fast (e.g., 350 m/min), the MWCTs were unable to respond fast enough, so the waviness of the tubes was not affected. On the other hand, when the wind-up speed was too slow (e.g., 150 m/min), the stretching stress on the MWCTs was not strong enough to affect the waviness. Only when the spinneret hole diameter was 0.6 mm and the wind-up speed was 250 m/min did it provide enough alignment and straightening of MWCTs to reinforce the PET fibers significantly.

In Tables V and VI are shown the results for the mechanical properties of PET/master batch B fibers spun with different spinneret holes. Similar to that of PET/master batch A, the reinforcement of MWCT-doped fibers was better for the 0.6-mm spinneret hole. In comparison with master batch A, the improvement of the mechanical properties was more favorable for master batch B. When 0.02 wt % MWCT-1 was doped, the PET/master batch B fibers yielded a breaking strength of 6.09 cN/dtex and an initial modulus of 113.9 cN/dtex, which were increased by 36.9% and 41.2%, respectively, in comparison with those of neat PET fibers (see Table VI).

TABLE I Mechanical Properties of PET/Master Batch A Fibers Produced Under the Conditions of 1.0 mm and 250 m/min

MWCT content (wt %)	Breaking elongation (%)	Breaking strength (cN/dtex)	Initial modulus (cN/dtex)		
0.00	50.4	4.15	79.4		
0.02	50.0	4.38	92.8		
0.04	47.4	4.40	81.3		
0.06	45.9	4.20	79.3		
0.08	43.4	4.38	89.3		
0.10	34.7	4.12	75.7		

TABLE II Mechanical Properties of PET/Master Batch A Fibers Produced Under the Conditions of 0.6 mm and 250 m/min

MWCT content (wt %)	Breaking elongation (%)	Breaking strength (cN/dtex)	Initial modulus (cN/dtex)	
0.00	42.2	4.34	87.0	
0.02	39.6	4.86	90.9	
0.04	34.4	5.25	119.7	
0.06	32.0	4.81	106.8	
0.08	33.8	3.94	88.2	
0.10	28.4	3.84	64.6	

TABLE III
Mechanical Properties of PET/Master Batch A
Fibers Produced Under the Conditions of 0.6 mm
and 150 m/min

MWCT content (wt %)	Breaking elongation (%)	Breaking strength (cN/dtex)	Initial modulus (cN/dtex)	
0.00	41.2	4.36	87.0	
0.02	41.1	4.54	93.1	
0.04	38.8	4.37	76.0	
0.06	34.9	4.24	70.3	
0.08	28.2	4.01	69.1	
0.10	29.3	3.75	65.5	

The improvement may have arisen from two factors. First, the carboxyl groups located on the MWCT surface could undergo esterification with the functional groups at the end of the PET macromolecule, which resulted in the formation of covalent bonds in the interface and significantly enhanced the interfacial adhesion between PET and MWCTs. As a result, the load transfer was favored. Moreover, the tubes were cut into shorter pieces after the acid treatment, and the entanglement and aggregation were relieved to such an extent that the MWCTs were more easily dispersed in the PET matrix.

The mechanical properties of the PET/master batch C and PET/master batch D fibers spun with the 0.6-mm spinneret hole and 250 m/min wind-up speed are shown in Table VII and VIII. The improvement of the mechanical properties was better for master batch C than master batch D. As mentioned in the Experimental section, master batches C and D used MWCT-2 as a filler, which was made of the ester formed by the reaction of sodium salts of MWCT-1 carboxylate and $C_{16}H_{33}Br$. The only difference was that the reaction time for MWCT-2a (for master batch C) was 12 h, whereas that for MWCT-2b (for master batch D) was 36 h. The longer reaction time introduced more alkyl chains into the MWCTs and led to better solubility (or dispersibility), which favored the reinforcement. However, the flexibility of alkyl chains in MWCT-2 disfavored the

TABLE IV Mechanical Properties of PET/Master Batch A Fibers Produced Under the Conditions of 0.6 mm and 350 m/min

MWCT content (wt %)	Breaking elongation (%)	Breaking strength (cN/dtex)	Initial modulus (cN/dtex)		
0.00	34.8	4.37	85.5		
0.02	32.5	4.60	91.0		
0.04	32.0	4.47	81.5		
0.06	31.7	4.14	79.6		
0.08	30.7	4.09	67.1		
0.10	26.9	4.08	56.5		

TABLE V Mechanical Properties of PET/Master Batch B Fibers Produced Under the Conditions of 1.0 mm and 250 m/min

MWCT-1 content (wt %)	Breaking elongation (%)	Breaking strength (cN/dtex)	Initial modulus (cN/dtex)	
0.00	44.7	4.20	77.4	
0.01	45.5	4.40	79.3	
0.02	43.4	4.42	80.2	
0.04	42.7	4.48	81.5	
0.06	33.9	4.88	88.0	
0.08	30.5	4.75	71.2	
0.10	30.0	4.73	73.9	

reinforcement. The bigger improvement of the mechanical properties of PET/master batch C fibers implied that the latter factor dominated over the former one.

As can be seen in Table I–VIII, among these four master batches under investigation, the maximum improvement of the mechanical properties was observed when master batch B was used because of the good dispersion of MWCTs in the PET matrix and significant enhancement of interfacial adhesion. Although a great dispersion of MWCT-2 in PET was obtained by the dissolution of both of them in a mixed solvent of TFA and DCM in the preparation of master batch C and master batch D, the alkyl chains in MWCT-2, which imparted the solubility, also weakened the reinforcement of fibers because of their flexibility.

Tables I–VIII also show that for master batches A– D, the mechanical properties of MWCT-doped fibers increased to a maximum value as the weight percentage of MWCTs increased. However, it began to decrease upon further doping and finally became weaker than that of the neat PET fibers. This indicates that the doping of MWCTs in PET enhances the mechanical properties of PET fibers, and in the meantime, this enhancement will decline when too much master batch is added, probably because of

 TABLE VI

 Mechanical Properties of PET/Master Batch B

 Fibers Produced Under the Conditions of 0.6 mm

 and 250 m/min

MWCT-1 content (wt %)	Breaking elongation (%)	Breaking strength (cN/dtex)	Initial modulus (cN/dtex)	
0.00	43.9	4.45	80.7	
0.01	41.3	5.05	90.9	
0.02	36.5	6.09	113.9	
0.04	36.0	6.08	97.6	
0.06	34.9	5.07	82.0	
0.08	32.3	5.04	81.0	
0.10	31.9	3.94	64.6	

TABLE VII Mechanical Properties of PET/Master Batch C Fibers Produced Under the Conditions of 0.6 mm and 250 m/min

MWCT-2a content (wt %)	Breaking elongation (%)	Breaking strength (cN/dtex)	Initial modulus (cN/dtex)	
0.000	45.8	4.34	80.8	
0.006	45.6	4.80	82.6	
0.008	44.0	4.88	86.4	
0.010	35.0	4.97	92.7	
0.020	31.5	5.76	109.1	
0.040	34.7	5.19	95.2	
0.060	34.5	4.41	88.4	
0.080	30.2	4.15	77.3	

the reaggregation of MWCTs and the flexibility of the alkyl chains.

It is noteworthy that for PET/master batch D fibers, the mechanical properties decreased with respect to those of the neat PET when 0.006 wt % (as shown in Table VIII) MWCT-2b was added. This behavior could be explained as follows. With this tiny amount of functionalized MWCTs added, the reinforcement due to the strength of MWCTs was not strong enough to overcome the reversed effect that arose from the flexibility of the alkyl chains grafted onto the MWCT surface.

The breaking elongations of the fibers were reduced when MWCTs were incorporated into the PET fiber, whether the significant reinforcement was observed or not, as shown in Tables I–VII. These lower values were undoubtedly due to the lower ductility of the fibers caused by MWCTs. The plasticization by the excess alkyl chains in master batch D neutralized the effect of MWCTs and thereby caused the steady, even slightly increased elongation with the loading of MWCT-2b (see Table VIII).

Table IX shows the results of WAXD and Δn of the maximum-reinforced fibers spun with different master batches. On the basis of Table IX, we found that both the crystallinity and Δn of the PET/master batch reinforced fibers decreased slightly in compari-

TABLE VIII
Mechanical Properties of PET/Master Batch D
Fibers Produced Under the Conditions of 0.6 mm
and 250 m/min

MWCT-2b content (wt %)	Breaking elongation (%)	Breaking strength (cN/dtex)	Initial modulus (cN/dtex)	
0.000	31.9	4.30	85.6	
0.006	32.8	3.77	75.5	
0.008	31.0	4.67	101.0	
0.010	30.1	5.14	109.5	
0.020	36.4	4.87	89.4	
0.040	35.2	4.29	82.1	

TABLE IX Supermolecular Structure of the PET/MWCT Fibers

	Crystallite size (Å)		Degree of		
Fiber type	010	110	100	crystallinity (%)	Δn
Neat PET PET/MWCT	58.34	37.03	31.83	48.6	0.195
(0.04 wt %) PET/MWCT-1	55.31	39.95	32.70	42.0	0.145
(0.02 wt %) PET/MWCT-2a	55.70	38.64	34.02	42.6	0.160
(0.02 wt %)	71.51	37.12	31.12	44.0	0.186

son with those of the neat PET fibers (not to mention any improvement). Apparently, the enhancement of the mechanical properties of the PET/master batch fibers arose predominantly from the intrinsic properties of MWCTs rather than the supermolecular structure of PET fibers.

Further work is underway with the focus on the preparation of a master batch that will give high dispersibility of MWCTs in the matrix and in the meantime maximize the improvement of the reinforcement of fibers through the control of both the degree of esterification of carboxylate on MWCTs and the proper choice of the length of linear alkyl chains.

CONCLUSIONS

Reinforced fibers obtained by melt spinning with PET and master batches were studied and compared with those obtained from neat PET. Among the four master batches under investigation, the fibers obtained from PET/master batch B had the highest improvement of mechanical properties. Their breaking strength and initial modulus increased 36.9 and 41.2%, respectively, in comparison with those of the neat PET fibers. Good dispersion and stronger interfacial adhesion due to the formation of covalent bonds between PET and MWCT-1 account for the improvement.

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