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Effect of Multi-walled Carbon Nanotube Dispersion on the Electrical, Morphological and Rheological Properties of Polycarbonate/Multi-walled Carbon Nanotube Composites

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Abstract: The effect of a multiwalled carbon nanotube (MWCNT) dispersion on the electrical, morphological and rheological properties of polycarbonate (PC)/MWCNT composites was investigated, with and without pretreating the MWCNTs with hydrogen peroxide oxidation and lyophilization. The resulting PC/treated MWCNT composites showed higher electrical conductivity than the PC/untreated MWCNT composites. The morphological behavior indicated the treated composites to have greater dispersion of MWCNTs in the PC matrix. In addition, the electromagnetic interference shielding efficiency (EMI SE) of the treated composites was higher than that of the untreated ones. Rheological studies of the composites showed that the complex viscosity of the treated composites was higher than the untreated ones due to increased dispersion of the MWCNTs in the PC matrix, which is consistent with the electrical conductivity, EMI SE and morphological studies of the treated composites. The latter results suggested that the increased electrical conductivity and EMI SE of the treated composites were mainly due to the increased dispersion of MWCNTs in the PC matrix.

Keywords: polycarbonate/carbon nanotube composites, dispersion, electrical property, rheology, morphology.

Introduction

Carbon nanotubes (CNTs) and polymer/CNT composites have been extensively studied.[1-9] The properties of polymer/MWCNT composites are strongly dependent on the extent of CNT dispersion, and CNT dispersion within the polymer matrix can be improved with enhancing the interaction between the polymer and CNT components through the solution mixing technique, including ultrasonication, surfactant-assisted mixing via a colloidal intermediate, noncovalent polymer wrapping, and covalent carbon nanotube functionalization with the polymer matrix.[10-19] In mixing simple solutions, a solvent dissolves the polymer, CNTs are added during high-energy ultrasonication, and the CNT/polymer composites formed by solvent evaporation.

The electrical, rheological, and mechanical properties of polymer/CNT composites can be altered due to their dependency on the CNT dispersion in the polymer matrix.[20-26] Electrical conductivity and electromagnetic interference shielding efficiency (EMI SE) of polymer/CNT composites can be produced with extremely low CNT content because of high aspect ratio of CNT and a wide range of electrical property values has been reported for polymer/CNT composites with variations in the carbon nanotube type and the mixing methods. The polymer matrix is usually electrically insulating and does not contribute to shielding, but it can affect the

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connectivity of the conductive filler and enhance the existing shielding effectiveness. CNT-containing polymer composites are attractive as shielding materials due to their process ability, which helps to reduce or eliminate the seams in the housing that is the shield. The seams are commonly encountered in the case of metal sheets as the shield and they tend to cause leakage of the radiation and diminish the effectiveness of the shield.[9-12]

The degree of carbon nanotube dispersion in the polymer matrix at, below, and above the electrical percolation threshold has been well described.[1-5] The rheological properties of polycarbonate (PC)/MWCNT composites in terms of PC-MWCNT interactions have been reported[1-3] and the strong influence of the alignment and dispersion of CNTs in the polymer matrix on the rheological and electrical behavior of polymer/CNT composites has been studied.[4-9]

In this study, PC/MWCNT composites with MWCNT content produced from tetrahydrofuran (THF) solutions were investigated regarding their electrical percolation thresholds and EMI SE through measurements with a digital multimeter and vector network analyzer, respectively. The morphological and rheological properties of these composites are also reported from scanning and transmission electron microscopy (SEM and TEM) and rotational rheometer examinations, respectively.

Experimental

Materials. Polycarbonate PC 201 15 was supplied by LG Chemical Ltd. and the number and average molecular weights of the PC were 11,000 and 30,000, respectively. MWCNTs were synthesized by the chemical vapor-grown method, supplied by Jeio Ltd., and had diameters ranging from 9 to 12 nm with lengths from 10 and 15 μm.

Composite Preparations. MWCNTs were concurrently treated with H_2O_2 and sonication and not purified before acid treatment. A 1.5 g sample of MWCNTs was sonicated with 500 mL of 28% H_2O_2 at 50 °C for 90 min, the mixture lyophilized at -60 °C for 72 h, rinsed with water up to pH 7, effectively removing the H_2O_2. For PC/MWCNT composite preparation, 10 g of PC/MWCNT mixture with 0.3, 0.5, 0.7, 1, 2, 3, 5, or 7 wt% MWCNTs was sonicated in 300 mL of tetrahydrofuran at 60 °C for 6 h, dried for at least 12 h at 120 °C, and the resulting composite particles were hot pressed at 260 °C and 20 MPa for 3 min.

Electrical Properties. The electrical conductivity of composites was measured by the four-probe method using a digital multimeter (Keithley, Model 2000 multimeter) to eliminate contact resistance. Four thin, gold wires (0.05 mm thick and 99% gold) were attached in parallel to the samples with conducting graphite paint.[10] The Fourier transformation Infrared spectra (FTIR) of the MWCNT were obtained with Perkin-Elmer FTIR. The FTIR samples were prepared by pressing the MWCNT with potassium bromide (KBr). Scans (400-4000 cm^{-1}) of 64 were averaged with a scan rate of 2 cm^{-1}. EMI SE measurements were made of composites connected to a vector network analyzer (Agilent-HP 8719ES-400 Network Analyzer) with a 2-port, flanged, coaxial line holder and monitoring in the far-field region for magnetic composite films with a frequency range of 1-12 GHz using the ASTM D4935-99 method.[12]

Rheology. Dynamic measurements of rheological properties were carried out using the Advanced Rheometric Expansion System (ARES) with frequency sweeps from 0.1-100 rad/s at 260 °C under dry nitrogen. All samples were tested within the linear viscoelastic strain range.

Morphology. The morphologies of the PC/MWCNT composites were investigated using transmission electron microscopy (JEOL, JEM-2000EX/T) using unstained specimens embedded in epoxy and cut with a microtome at ambient temperature. In addition, the morphologies of the composites were recorded using an atomic force microscope (AFM, MFP3D, Asylum Research) at room temperature in tapping mode with 10 nm standard cantilevers (OMCL-AC160TS, Olympus). The areas of the MWCNT dispersions in the PC matrices were calculated using the Image J program and analysis of particle method by the NIH, USA.

Results and Discussion

Electrical Properties. Electrical conductivities (σ) of PC/MWCNT composites with MWCNT content are shown in Figure 1(a). For the PC/MWCNT composites without H_2O_2 treatment in Figure 1(a), the percolation threshold of the electrical conductivity is observed at about 5 wt% MWCNT. For the composites of PC with freeze-dried MWCNT after H_2O_2 treatment, the percolation threshold of the electrical conductivity is shown to be about 0.5 wt% MWCNT. From Figure 1(a), it is known that the electrical conductivity of the PC/freeze-dried MWCNT composites is higher than that of the PC/MWCNT composites without H_2O_2 treatment. The electrical conductivity of the PC/MWCNT composites was 2.06×10^{-4} S/cm (H_2O_2-treated, freeze-dried) at 0.5 wt% MWCNT. For the PC/MWCNT composites without H_2O_2 treatment, the electrical conductivity of the composites containing below 3 wt% MWCNT was not able to be detected because of the low magnitude of the electrical conductivity.

Figure 1(b) shows the electrical conductivity (σ) of the PC/freeze-dried MWCNT composites as a function of MWCNT mass fraction (p). The percolation behavior of the PC/MWCNT composites can be explained by the power law equation,[12] and the correlation between the electrical conductivity and power law is shown in Figure 1(b). At the onset of the percolative network, the σ of composites with conductive filler concentration (p) can be described by the power law shown in eq. (1) as follow[12]:

$$\sigma = \sigma_0(p-p_c)^t \quad \text{for } p > p_c \tag{1}$$

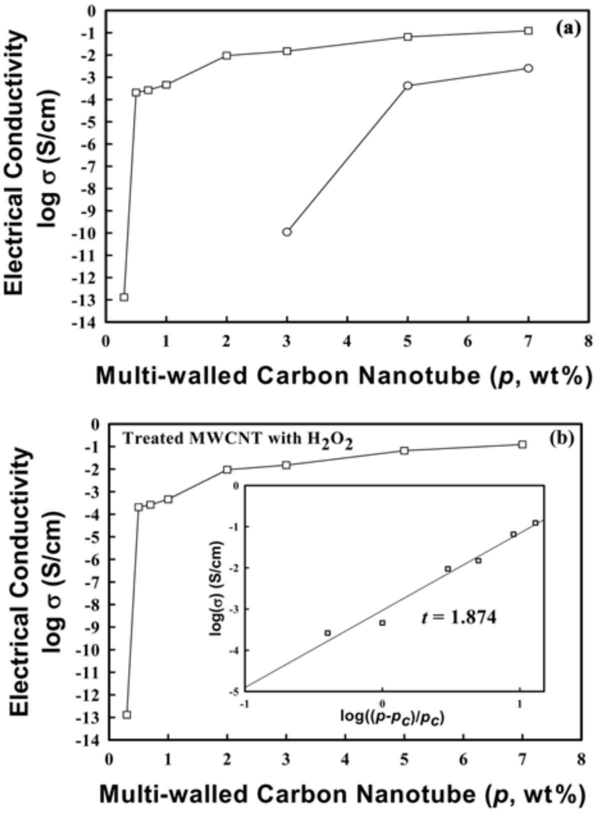


Figure 1. (a) Electrical conductivity of PC/MWCNT composites with MWCNT content: (○) MWCNT was untreated; (☐) MWCNT was freeze-dried after treatment with H_2O_2. (b) The inset shows the log-log plot of electrical conductivity (σ) with $(p-p_c)/p_c$ for $p>p_c$.

where σ is the electrical conductivity of the composite, σ_0 the fitting constant, p_c the percolation threshold, and t the critical exponent. The best result of nonlinear curve fitting is also shown in Figure 1(b) as the solid line. The best-fitted constant was $\sigma_0=2.06\times10^{-4}$ S/cm, t=1.874, and P_c=0.5 wt%. The exponent, t, was determined from the slope of the least-square on a log-log scale as shown in the inset of Figure 1(b).

As shown in the inset of Figure 1(b) for the log(σ) versus log($(p-p_c)/p_c$) plot, the electrical conductivity of the PC/freeze-dried MWCNT composite agrees very well with the percolation behavior predicted by eq. (1). From the results of Figure 1(b), it is shown that the electrical percolation threshold in this system is obtained at 0.5 wt% MWCNT content, and this result is in good agreement with the result obtained from Figure 1(a).

Figure 2 shows FTIR spectra of the untreated and treated MWCNTs with H_2O_2. From Figure 2(a), the MWCNTs without H_2O_2 treatment show the C=C peak at about 1650 cm^{-1}. From Figure 2(b), the freeze-dried MWCNTs with H_2O_2 treatment shows the C-C peak at 1220 cm^{-1} and the C=O stretching peak at about 1730 cm^{-1}, which are not shown in Figure 2(a). In Figure 2(b), the appearance of the C-C peak at 1220 cm^{-1} and C=O peak at 1730 cm^{-1} suggests that the MWCNTs are functionalized with the functional groups (-COOH) by the H_2O_2 oxidation. From Figures 1 and 2, therefore, the observation of percolation threshold at 0.5 wt% MWCNT of the PC/freeze-dried MWCNT composites may be due to the less entanglement of the MWCNTs.

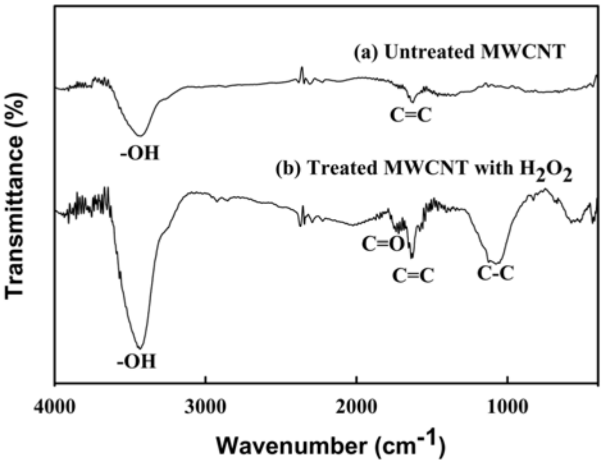


Figure 2. FTIR spectra of the treated and untreated MWCNTs: (a) MWCNT was untreated; (b) MWCNT was freeze-dried after treatment with H_2O_2.

The higher electrical conductivity of the PC/freeze-dried MWCNT composites with H_2O_2 treatment could be explained by the morphological and rheological properties of the PC/MWCNT composites such that the increased MWCNTs connectivity is related to the increase of the percolated MWCNT-MWCNT network structure. A detailed explanation of the effect of morphology on the electrical properties of PC/MWCNT composites will be provided in the morphology section of this paper.

Figure 3(a) and (b) show the EMI SE of PC/MWCNT composites with MWCNT content of 1, 2, 3, 5 and 7 wt% over the frequency range of 1-12 GHz. From Figure 3(a), it is shown that the EMI SE of PC/MWCNT composite without H_2O_2 treatment ranged from 1-7 dB with MWCNT content. EMI SE was not observed for the PC/MWCNT composites of 0.3, 0.5 and 0.7 wt% MWCNT because of the low MWCNT contents. Figure 3(b) shows the EMI SE of the composites of PC/freeze-dried MWCNT after H_2O_2 treatment. From Figure 3(b), the EMI SE of the PC/MWCNT (93/7 wt%) composites is observed to be 11-15 dB for the H_2O_2-treated, freeze-dried MWCNT. From Figure 3(a) and (b), the higher EMI SE of the PC/freeze-dried MWCNT composites may suggest homogenous dispersion of the MWCNT in the PC matrix compared the PC/MWCNT composites without H_2O_2 treatment.

Many researchers[11-15] have studied EMI SE of the polymer/carbon nanotube composites, and Table I shows the comparison of EMI SE for the various polymer/carbon nano-

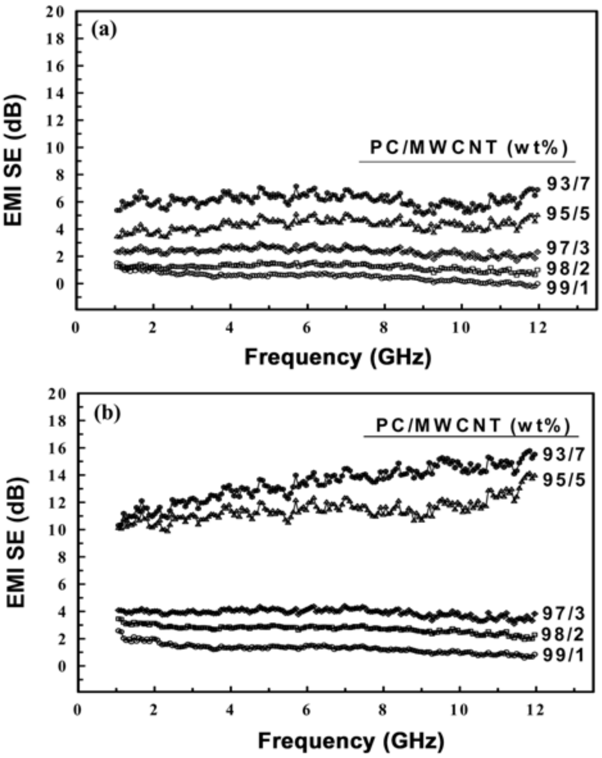


Figure 3. Electromagnetic interference shielding efficiency (EMI SE) of PC/MWCNT composites by MWCNT content: (a) MWCNT was untreated; (b) MWCNT was freeze-dried after treatment with H_2O_2: (○) PC/MWCNT = 99/1; (□) 98/2; (◇) 97/3; (△) 95/5; (⊕) 93/7.

tube composites. For the EMI results of PU/MWCNT composites[12] and PMMA/MWCNT composites,[14] the testing method (ASTM D4935-99) and equipment (HP vector network analyzer) were identical to those used in our experiments to measure the EMI results. Values of EMI SE of the various polymer/CNT composites are summarized in Table I. As shown in Table I, by comparing the EMI SE of PC/MWCNT composites with the results reported by other researchers,[11-15] the increased values of EMI SE are observed when the amount of CNT are increased in the composites. In Figure 3(b), the values of EMI SE of PC/freeze-dried MWCNT composites with H_2O_2 treatment is about 10-13 and 11-16 dB at 5 and 7 wt% MWCNT in the frequency range of 1-12 GHz, respectively. For the 2 and 3 wt% MWCNT, the EMI SE is about 3 and 4 dB, respectively. By comparing our results of EMI SE with the results reported by others,[11-15] it is suggested that the level of EMI SE of the composites shown in this study is comparable with others by considering the weight fraction of MWCNT in the composites.

The EMI SE increases as electrical conductivity (σ) of shielding material increases based on the EMI shielding theory(far-field shielding) by eq. (2) as follow:[14,19]

$$\text{EMI SE} = 20\log\left(1+\frac{1}{2}\sigma d Z_0\right) \tag{2}$$

where σ is the electrical conductivity, d the sample thickness, and Z_0 the free space impedance (constant: 377 S^{-1}). Table II shows the estimated and experimental values of EMI SE of PC/MWCNT composites at 6 and 12 GHz, respectively. The estimated values of EMI SE shown in Table II

Table II. Estimated and Experimentally Obtained EMI Shielding Efficiency of the PC/MWCNT Composites with MWCNT Treatment at the Frequency of 6 GHz and 12 GHz

	Values of Estimated/Experimentally Obtained EMI Shielding Efficiency (dB)	
	At 6 GHz	
MWCNT Content (wt%)	H_2O_2-Untreated	H_2O_2-Treated, Freeze-dried
1.0	0.00/0.02	1.54/1.05
2.0	0.00/1.00	3.19/2.29
3.0	3.67×10^{-6}/2.33	5.20/3.93
5.0	5.53/5.01	12.65/13.95
7.0	7.08/6.90	14.26/15.52
	At 12 GHz	
1.0	0.00/0.67	1.54/1.46
2.0	0.00/1.51	3.19/2.88
3.0	3.67×10^{-6}/2.77	5.20/4.19
5.0	5.53/4.94	12.65/11.96
7.0	7.08/6.82	14.26/13.80

Table I. Comparison of EMI Shielding Efficiency for the Various Polymer/CNT Composites

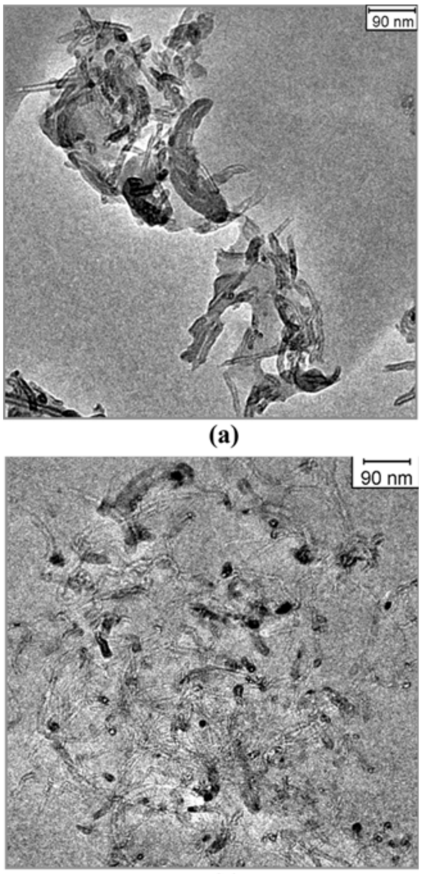
Composites	CNT Content (wt%)	EMI Shielding Efficiency (dB)	Frequency Range (GHz)	Ref.
PU/SWCNT	20	16-17	8.2-12.4	(11)
PU/MWCNT[a]/Ag[b]	0.2	80-100	0.01-1	(12)
PS/MWCNT	7	18	8.2-12.4	(13)
PMMA/MWCNT	40	27	2-11	(14)
Epoxy/SWCNT	15	15-20	0.5-1.5	(15)
PC/MWCNT	7	16	1-12	This work

[a]0.2 vol% of MWCNT was added. [b]10 vol% of Ag was added.

were calculated using eq. (2) and the electrical conductivity from Figure 1(a). The EMI shielding mechanisms should include reflection, absorption and internal refraction in shielding materials. But, eq. (2) is only based upon reflection mechanism. From the studies by Colaneri and Shacklette,[19] they have proposed eq. (2) to measure the EMI SE when there is a skin-depth effect for EMI SE, especially, for the electrically thin samples. In this study, we have used electrically thin sample of PC/MWCNT composites, therefore, eq. (2) was applied to calculate the EMI SE in order to compare with the experimentally obtained values of EMI SE.

From Table II, the estimated values using eq. (2) of the EMI SE of PC/freeze-dried MWCNT composites are in good agreement with the experimentally obtained values with MWCNT content. The good agreement of the EMI SE between estimated and experimentally obtained values of the PC/freeze-dried MWCNT composites suggests the increased dispersion of MWCNT in the PC matrix.

Morphology. TEM morphologies of the PC/MWCNT (95/5 wt%) composites with sample preparation methods are shown in Figure 4(a) and (b) show the PC/MWCNT composites without H_2O_2 treatment and PC/freeze-dried MWCNT composites with H_2O_2 treatment, respectively. From the result of Figure 4(a), it is seen that the MWCNTs form an entangled structure in the PC matrix. In Figure 4(b), the entanglement of the MWCNTs loosens after H_2O_2 treatment and freeze-drying. The freeze-dried MWCNTs after H_2O_2 oxidation improved the MWCNT dispersion in the PC matrix. The increased dispersion of the MWCNT in the PC/freeze-dried MWCNT composite may result in the higher electrical conductivity of the PC/freeze-dried MWCNT composite as can be seen in Figure 1(a).

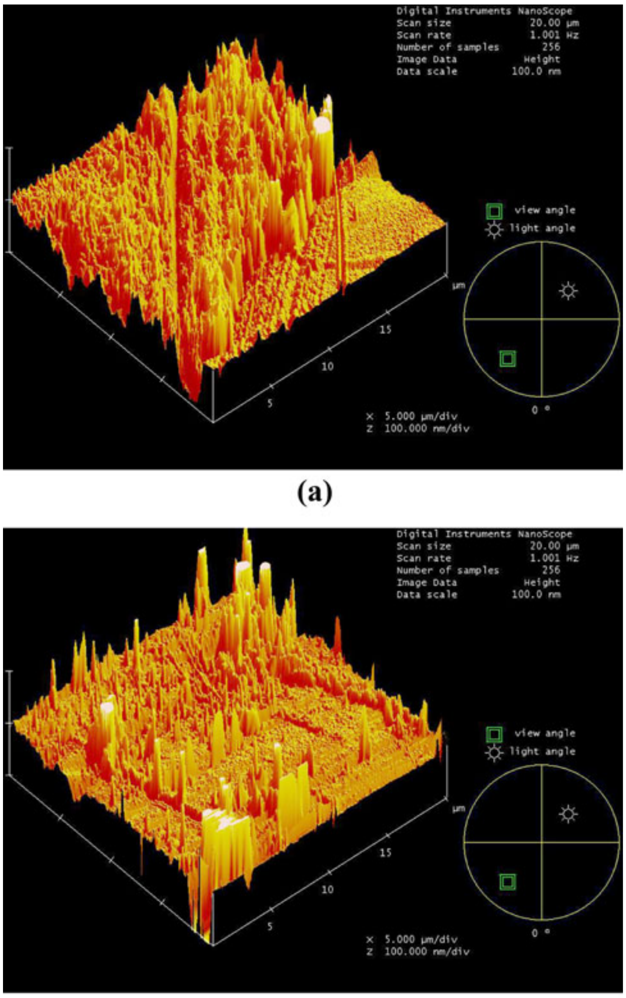


(a)

(b)

Figure 4. Transmission electron micrographs of PC/MWCNT (95/5 wt%) composites obtained by solution mixing: (a) MWCNT was untreated; (b) MWCNT was freeze-dried after treatment with H_2O_2.

The AFM morphologies of the PC/MWCNT (95/5 wt%) composites are shown in Figure 5(a) and (b) which represent PC/MWCNT composites without H_2O_2 treatment and freeze-dried PC/MWCNT composites with H_2O_2 treatment, respectively. From AFM morphologies of Figure 5, the area of dispersed MWCNT was obtained from the total area



(a)

(b)

Figure 5. Atomic force micrographs of PC/MWCNT (95/5 wt%) composites obtained by solution mixing: (a) MWCNT was untreated; (b) MWCNT was freeze-dried after treatment with H_2O_2.

(pixel 1020×1020) of PC/MWCNT (95/5 wt%) composites. From Figure 5(a) and (b), the values of dispersed area of MWCNT are found to be 1,957 and 13,825 units, respectively. The area of the MWCNT dispersed in PC matrix of the PC/freeze-dried MWCNT composites is found to be about 7 times compared that of the PC/MWCNT composites without H_2O_2 treatment. From the results of morphological behaviors shown in Figure 5(a) and (b), it is suggested that the dispersion of the MWCNT in the PC matrix has been improved for the PC/freeze-dried MWCNT composites with H_2O_2 treatment.

From the results of TEM and AFM of the PC/MWCNT composites, it is suggested that the oxidation of MWCNTs with H_2O_2 and subsequent freeze drying are an important sample preparation method to improve the dispersion of MWCNT in the PC matrix. The results of electrical conductivity, EMI SE and morphological studies of PC/MWCNT composites suggest that the electrical conductivity and EMI SE are strongly dependent on MWCNT pre-treatment and degree of MWCNT dispersion in the PC matrix.

Rheology. Figure 6(a) and (b) shows the complex viscosity (η^*) of the PC/MWCNT composites without H_2O_2 treatment and PC/freeze-dried MWCNT composites with H_2O_2 treatment, respectively. Figure 6 shows that the complex viscosity (η^*) of the PC/MWCNT composites increases with increasing MWCNT content. The increase of the complex viscosity is more significant in the PC/freeze-dried MWCNT composites than in the PC/MWCNT composites without H_2O_2 treatment. Also, for the PC/freeze-dried MWCNT composites with H_2O_2 treatment, the complex viscosity increased more significantly at low frequencies than at high frequencies. The rheological properties of polymer composites at low frequencies reflect the percolated filler-filler network structure.[16] Therefore, the increased complex viscosity of PC/freeze-dried MNCWT composites at low frequency is thought to be related with the increased MWCNT-MWCNT network structure.

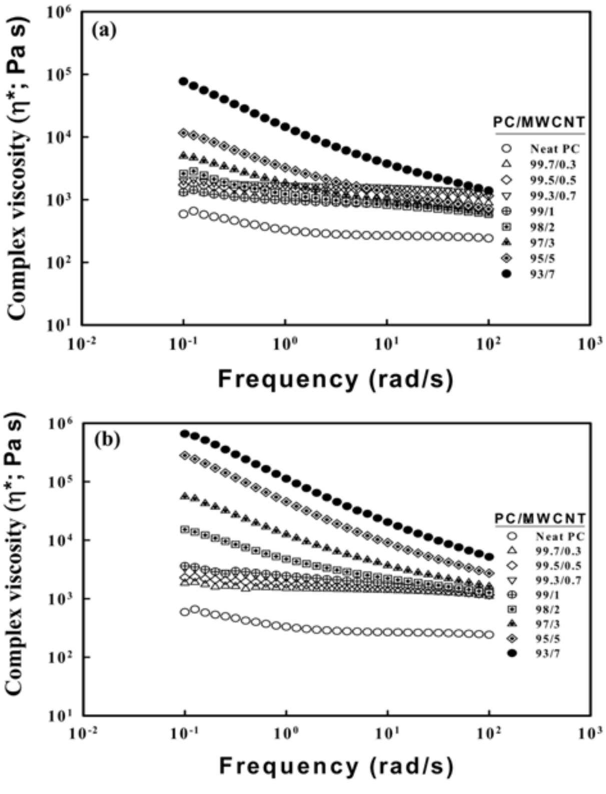


Figure 6. Complex viscosity (η^*) of PC/MWCNT composites with MWCNT content at 260 °C: (a) MWCNT was untreated; (b) MWCNT was freeze-dried after treatment with H_2O_2.

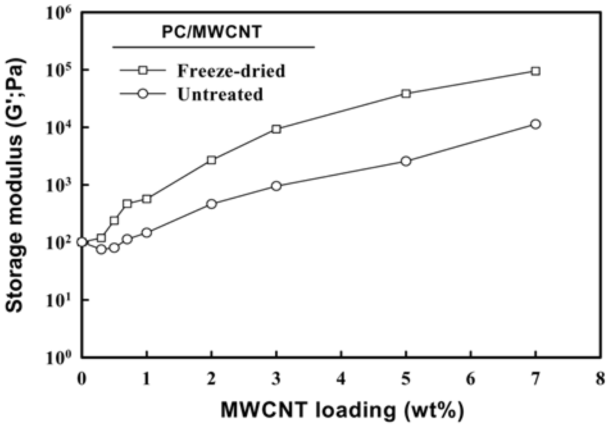


Figure 7. Storage modulus (G') of PC/MWCNT composites at 260 °C and frequency of 1 rad/s with MWCNT content: (○) MWCNT was untreated; (□) MWCNT was freeze-dried after treatment with H_2O_2.

Figure 7 shows the storage modulus of the PC/MWCNT without H_2O_2 treatment and PC/freeze-dried MWCNT composites with H_2O_2 treatment. From Figure 7, it is shown that the storage modulus of the PC/freeze-dried MWCNT composites is increased with MWCNT contents compared the PC/MWCNT composites without H_2O_2 treatment. An increase in storage modulus may occur when the dispersion of the MWCNT in the matrix is increased than that of the composites without H_2O_2 treatment. In the studies of single-walled carbon nanotube/poly(methyl methacrylate) nanocomposites, Du *et al.*[5] have reported that higher values of G' at low frequency can associated with better nanodispersion, when all other factors are constant. Therefore, from the results of rheological properties shown in Figures 6 and 7, the increased complex viscosity of the PC/freeze-dried MWCNT composites maybe resulted from the increase of MWCNT dispersion in the PC matrix. This result is also consistent with the results of electrical conductivity and EMI SE for the PC/freeze-dried MWCNT composites with H_2O_2 treatment.

Conclusions

In this study, the electrical, morphological and rheological properties of PC/MWCNT composites were investigated with and without pre-treatment of MWCNT with H_2O_2. The PC/freeze-dried MWCNT composites with H_2O_2 treatment showed higher electrical conductivity than PC/MWCNT composites without H_2O_2 treatment. From morphological behavior of the PC/MWCNT composites by TEM and AFM, the improved dispersion of MWCNT in the PC matrix was observed for the PC/freeze-dried MWCNT composites with H_2O_2 treatment.

The EMI SE of the PC/freeze-dried MWCNT composites with H_2O_2 treatment was increased compared the composites without H_2O_2 treatment because of the increased dispersion of the MWCNT in the PC matrix. The estimated values of EMI SE are in good agreement with the experimentally obtained values of the PC/freeze-dried MWCNT composites with MWCNT content, and this result suggests the increased dispersion of MWCNT in the PC matrix. From the results of electrical conductivity, EMI SE and morphological properties of PC/MWCNT composites, it is concluded that the increased electrical conductivity and EMI SE of the PC/freeze-dried MWCNT composites are mainly due to the increased dispersion of the MWCNT in the PC matrix.

From the results of rheological properties of the composites, complex viscosity of the PC/freeze-dried MWCNT composites has increased compared that of the PC/MWCNT composites without H_2O_2 treatment because of the increase of MWCNT dispersion in the PC matrix. This result is consistent with the results of electrical conductivity, EMI SE and morphological behavior of the PC/freeze-dried MWCNT composites with H_2O_2 treatment.

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