



Effect of PA6T on morphology and electrical conductivity in PA66/PA6T/PPE/multiwalled carbon nanotube nanocomposites

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ABSTRACT

Nanocomposites made of blends of polyamide (PA66), polyphthalamide (PA6T), and poly(2,6-dimethyl-1,4-phenylene ether) (PPE) with multi-walled carbon nanotubes (CNTs) were investigated. At 1 wt% CNT loading, the electrical conductivities of PA66/PA6T/PPE/CNT nanocomposites were around four orders of magnitude larger than those of PA66/PPE/CNT nanocomposites without PA6T. Line mapping images and spot spectrum results from transmission electron microscopy/energy dispersive spectroscopy analysis showed that the continuous phase contained PA66 and PA6T with dispersed PPE domains. Phase inversion was observed as the CNT content of PA6T/PPE/CNT increased. Depending on the polymer composition, alignment of some of CNTs at the interface was observed. Using the wetting coefficient analysis and the Hansen solubility parameter, the morphologies of the nanocomposites and the resulting electrical conductivities were found to be affected by the compatibilizing effect of PA6T on PA66 and PPE as well as the higher affinity of CNTs for PA6T than for PPE or PA66. CNTs were observed to help maintain the integrity of the PA66/PA6T continuous phase by dynamic mechanical analysis.

1. Introduction

Polyamide (PA) is one of the most important engineering plastics. However, PA also has some disadvantages, such as poor dimensional stability and degradation of various properties owing to high water absorption [1]. In contrast, poly (2,6-dimethyl-1,4-phenylene ether) (PPE) exhibits excellent dimensional stability and heat resistance [2]. Accordingly, PA/PPE blends have been used in a variety of engineering [3]. Recently, many metallic components in automobile parts have been replaced with high-performance polymer blends, such as PA66/PPE blends. As PA66/PPE blends are immiscible blends, improved interfacial adhesion between the two immiscible phases is very important for obtaining good physical properties [2–4]. When PA66/PPE blends are used for the exterior panels of automobiles, they require electrical conductivity for electrostatic painting. Electrical conductivity is usually achieved by adding conductive fillers such as carbon black or carbon nanotubes (CNTs) [5–7].

Polyphthalamide (PPA) is a semi-aromatic PA composed of aliphatic chains and aromatic groups containing terephthalic or isophthalic acid

in the polymer backbone. There have been numerous investigations on aliphatic PAs, but there are only a few reports on PPAs and their blend systems [8,9]. Although some PA/PPE/CNT nanocomposites have been reported [10,11], PPA/PPE/CNTs nanocomposites have been little explored.

In the present study, nanocomposites of blends of PA66, PPA, and PPE with CNTs were investigated. Specifically, PA6T was used as a PPA. The influences of adding semi-aromatic PA6T to PA66/PPE/CNT nanocomposites on the phase behavior and electrical conductivity was investigated. The morphology changes caused by the addition of PA6T, as well as the location and dispersion state of CNTs in PA66/PA6T/PPE/CNT nanocomposites, was investigated.

2. Experimental

2.1. Materials

PA66 ($M_w = 75,000$ g/mol, Technyl 2110R, Rhodia Company), PA6T ($M_w = 48,000$ g/mol, Vestamid M1100, Evonic Degussa

Abbreviations: PA, Polyamide; PPE, Poly(2,6-dimethyl-1,4-phenylene ether); MA, Maleic anhydride; CNTs, Carbon nanotubes; PPA, Polyphthalamide; TEM/EDS, Transmission electron microscopy/Energy dispersive spectroscopy; DMA, Dynamic mechanical analysis; DSC, Differential scanning calorimetry; PS, Polystyrene; HSP, Hansen solubility parameter

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Table 1

Sample designation of PA66/PA6T/PPE/CNT nanocomposites.

Sample designation	Polymer and CNT composition (wt%)			
	PA66	PA6T	PPE	CNT
B1	60		40	-
B2	48	12	40	-
B3	30	30	40	-
B4		60	40	-
N1(X)	60		40	X
N2(X)	48	12	40	X
N3(X)	30	30	40	X
N4(X)		60	40	X

X: CNT loading (wt%).

Company), and PPE ($M_w = 29,300$ g/mol, Xyron S202A, Asahi Kasei Corp.) were used in the experiments. CNTs synthesized by chemical vapor deposition (purity = ~95 wt%, VGCF-X, Showa Denko) were used without further purification to prepare the nanocomposites.

2.2. Preparation of PA66/PA6T/PPE/CNT nanocomposites

Prior to melt compounding, PA66, PA6T, and PPE pellets were dried at 130 °C for 12 h and CNTs were dried in a vacuum oven at 80 °C for 12 h. The CNT loadings in all the nanocomposite samples were 0, 0.5, 1.0, and 2.0 wt%. The polymers and CNTs were fed into a rotary feeder simultaneously and melt mixing was conducted in a twin-screw extruder (BA-19ST, Bautek Corp.). The nanocomposites were processed with a screw speed of 150 rpm at temperatures in the range of 280–330 °C in the extruder. The extrudates were pelletized and subjected to hot-press molding at 280–330 °C for 5 min under a pressure of 5.0 MPa to obtain thin-film specimens.

The sample designation listed in Table 1 is used hereinafter. Blends of PA66, PA6T, and PPE without CNTs are indicated by a capital “B” and the following numbers (1–4) corresponds to the blend composition specified in Table 1. Nanocomposites containing both a blend and CNTs are indicated by a capital “N”. The following number again denotes the blend composition (Table 1), with another number in parentheses indicating the CNT loading in wt%. For example, B1 denotes the blend sample containing PA66/PA6T/PPE (60/0/40) and N2(2) denotes the nanocomposite sample containing PA66/PA6T/PPE/CNT (48/12/40/2).

2.3. Characterization and measurement

Electrical resistivity (surface resistivity) was measured using a digital ultra-high resistance meter (R8340, Advantest Corp., USA) at 2.5 V with a test fixture. TEM images and EDS mapping of the nanocomposite specimens were obtained at the Korea Basic Science Institute in field emission mode (JEM 2100-F, JEOL, Japan) at an operating voltage of 200 kV. EDS quantitation was performed by the Cliff–Lorimer thin ratio method. The thermal properties of the nanocomposites were determined by DSC (Q20, TA Instruments, USA) at a heating rate 20 °C/min in a nitrogen atmosphere. Samples were heated from 25 to 350 °C followed by quenching and the thermograms obtained during the second heating run were used for analysis. DMA was performed using a dynamic mechanical analyzer (SS6100, Seiko Instrument Inc.) at an amplitude of 4.5 μ m and a frequency of 1 Hz in a nitrogen atmosphere. The temperature was increased from 30 to 350 °C at a rate of 20 °C/min.

3. Results and discussion

3.1. Electrical conductivity of PA66/PA6T/PPE/CNT nanocomposites

Fig. 1 shows the electrical resistivities of various compositions of PA66/PA6T/PPE/CNT nanocomposites depending on CNT loading.

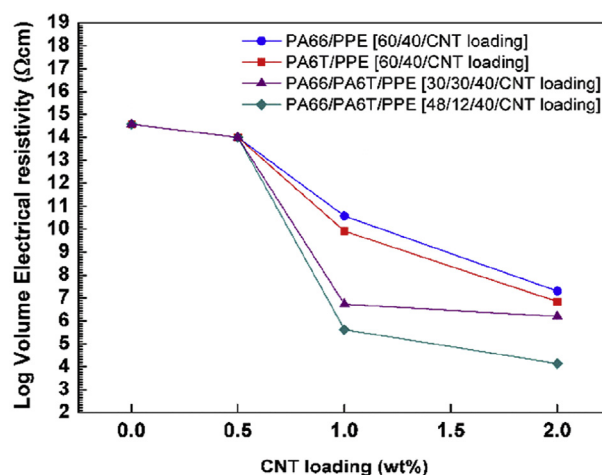


Fig. 1. Electrical Volume resistivity of various compositions of PA66/PA6T/PPE/CNT nanocomposites depending on CNT loading.

(Standard deviation bars are included in Fig. 1 to show the variation in electrical resistivity). Irrespective of blend composition, all PA66/PA6T/PPE blends without CNTs (B1 – B4) show low electrical conductivity (high electrical resistivity in Fig. 1). For improved clarity, the term electrical conductivity is used hereinafter instead of electrical resistivity. As expected, the electrical conductivity of PA66/PA6T/PPE nanocomposites with CNTs gradually increased with increasing CNT loadings. Fig. 1 shows that the electrical percolation threshold value is between 0.5 and 1 wt% CNTs. Notably, the electrical conductivities of N2(1) or N3(1), which are PA66/PA6T/PPE ternary blends with 1 wt% CNTs, were several orders of magnitude higher than those of N1(1) or N4(1), which are binary blends with CNTs.

In particular, N2(1), with a composition of PA66/PA6T/PPE/CNT (48/12/40/1), has the highest electrical conductivity. This is quite an interesting result because the properties of ternary blend nanocomposites (N2 or N3, PA66/PA6T/PPE/CNT) usually appear between those of two binary blend nanocomposites (N1, PA66/PPE/CNT and N4, PA6T/PPE/CNT). These results are thought to be due to the heterogeneous morphologies of the PA66/PA6T/PPE/CNT nanocomposites resulting from phase separation.

Double percolation structure resulting from the selective localization of CNTs or carbon black is reported to reduce the electrical percolation threshold values in the literature [12,13]. This double percolation phenomena is observed in the shape memory polymer composite containing biodegradable poly(propylene carbonate)/poly(lactic acid)/carbon nanotube [12] or in the carbon black filled immiscible polystyrene/polypropylene blend [13]. Addition of nanoclay to polypropylene/CNT nanocomposites is also reported to reduce the percolation threshold values [14]. Thus, the phase separation behavior, as well as the localization and dispersion state of CNTs, in PA66/PA6T/PPE/CNT nanocomposites has an important effect on the electrical conductivity.

3.2. Morphology of PA66/PA6T/PPE/CNT nanocomposites

3.2.1. Morphology of nanocomposites prepared by one step mixing

TEM images of the PA66/PA6T/PPE/CNT nanocomposites containing 1 wt% CNT are shown in Fig. 2.

The TEM samples were prepared by melt mixing all the ingredients simultaneously in the melt extruder. The morphologies in Fig. 2(a) – (d) correspond to those of N1(1) – N4(1). For N1(1), the dispersed phase is PPE and the continuous phase is PA66. Detailed identification of these phases is discussed in Section 3.3. Fig. S1(a) – (d) show higher magnification TEM images of N1(1) – N4(1), respectively. For N1(1), the CNTs are observed to be mainly located in dispersed PPE domains with

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