Measurement of Characteristics of PU/MWNT Films Made of Modified MWNT by Dry Heat Treatment

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Abstract: This study examines the electrical conductivity of multi-walled carbon nanotube (MWNT) /polyurethane (PU) nanocomposites film according to the dispersion conditions. For this purpose, various kinds of polyurethane block copolymer (PU)/multi-wall carbon nanotube (MWNT) with different MWNT contents were mixed and dispersed, and their characteristics related to the dispersion were investigated. Multi-walled carbon nanotubes (MWNT) are of interest in many areas of nanotechnology and used in a number of novel applications. However effective dispersion remains a problem and one solution is to functionalize the nanotubes. In this work, MWNTs are functionalized using an oxidative technique, including dry thermal treatment. The dispersion variation of the PU/MWNT nanocomposite films with MWNT content ranging from 0.1~5.0 wt% was examined. The various characteristics of the dispersed PU/MWNT nanocomposite film were characterized using thermo gravimetric analyser (TGA), scanning electron microscope (SEM) and measuring instrument of electrical conductivity. The degree of dispersion of PU/MWNT nanocomposite films according to the dispersion conditions was shown in various situations. The bad dispersion of PU/MWNT film was revealed during the phase separation between PU matrix and MWNT. Also PU/MWNT nanocomposite film shows good electro-conductivity on its surface because MWNT was collected on the surface of composite film due to its low density.

Keywords: Multi-walled carbon nanotube, polyurethane, dispersion, DMF, PU/MWNT film, electrostatic dissipation.

1. Introduction

Since the discovery of carbon nanotube (CNT) by Iijima [1], it has been attracting tremendous interest because of their unique combination of electronic, mechanical, chemical and thermal properties, which promises a wide range of potential applications [2-4]. For example, in electronic application, CNT are used to dissipate unwanted static charge build-up [2]. In the automotive industry, CNT are used as conducting agents to aid in electrostatic painting [3]. There are two main types of CNT most commonly in use, single-walled carbon nanotube (SWNT) and multi-walled carbon nanotube (MWNT). The walls of SWNT are lattices constructed from hexagons of carbon, whilst MWNT consist of a number of nested SWNT with strong van der waals forces between them. However, CNT can hardly disperse in polymer matrix because of its nonreactive surface. So, the CNT usually requires treatment before it can be applied, so as to obtain a better dispersion. With the aid of ultrasound, which helps the particles to overcome the van der waals forces of attraction among CNT, stable homogeneous aqueous dispersions were prepared with sodium dodecyl sulfate (SDS) as dispersing agent [5]. The use of ultrasound, however, has been shown to damage CNT [6]. In fact, appropriate chemical oxidation of CNT's surfaces can dramatically raise the stability of aqueous suspensions and also introduce the carboxylate groups onto the surface of CNT [7]. However, CNT often tend to aggregate up to a few tens of micrometers because of intrinsic van der waals forces of attraction among tubes and their high surface area and high aspect ratio, when they disperse in polymer or other matrix. Multi-walled carbon nanotube (MWNT) are of interest in many areas of nanotechnology and used in a number of novel applications. However effective dispersion remains a problem and one solution is to functionalize the nanotubes. In this work, MWNT are functionalized using oxidative techniques, including dry thermal treatment. This study surveys the electrical conductivity of multi-walled carbon nanotube (MWNT)/polyurethane (PU) nanocomposites film according to the dispersion conditions. This study surveys the functionalization of MWNT by chemically modifying it to improve its dispersion. The functionalised MWNT forms high molecule matrix with polyurethane to produce a film that has the characteristic of electrostatic dissipation. Finally the electrical characteristics according to dispersion conditions were analysed and discussed.

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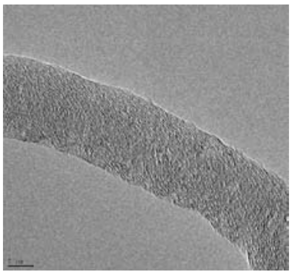
2. Experimental

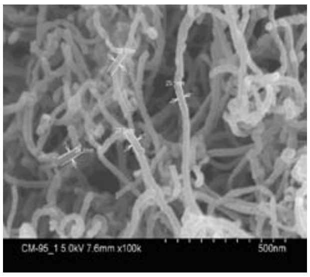
2.1 Materials

CM-95 multi-walled carbon nanotube, Hanwha Nanotech, Incheon, Korea was prepared. MWNT are produced via the catalytic carbon vapor deposition process. The synthesis process uses ethylene gas with an alumina supporting cobalt and iron as catalysts. The size and shape of MWNT was characterized using transmission electron microscopy (TEM) and scanning electron microscopy (SEM). The characteristics of MWNT are listed in Table 1. And, Figure 1 shows the images of TEM and SEM of MWNT.

Table 1 Specification of MWNT

Type	Diameter (nm)	Length (μm)	Purity (%)	Remark
CM-95	10~15	10~20	95	Hanwha Nanotech (KOREA)





(a) TEM (b) SEM

Figure 1 TEM and SEM images of MWNT.

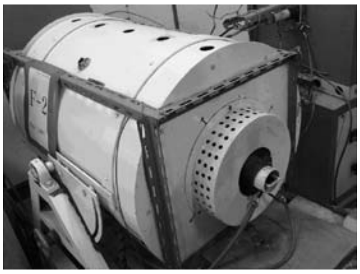
The polyurethane was used as the material for the formation of MWNT/high molecule matrix. DMF (dimethylformamide) was used as dispersion solvent of MWNT. The characteristics of polyurethane are listed in Table 2.

Table 2 Specification of polyurethane

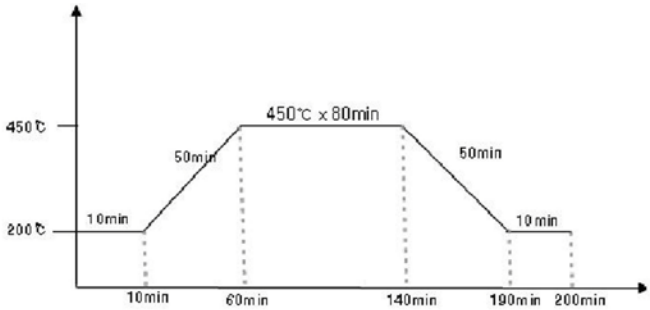
Type	Molecular weight	Viscosity	Remark
1 liquid (972DF)	100,000 ~200,000	85,000 ~110,000	Cytec Industries Inc.

2.2 Dry heat treatment of MWNT

When performing dry heat treatment on water, organic matter, catalyst and other impurities during refining process of MWNT, it improves the dispersion efficiency. So dry heat treatment was performed for 80 minutes in the air furnace with the temperature of 450℃ for the refinement of MWNT. Figure 2 shows the air furnace of dry heat treatment (a), the boat that includes carbon-nanotube (b) and heating graphs of air furnace (c).



(a) dry heater (b) boat



(c) dry heater processing profile

Figure 2 The dry heat treatment processing of MWNT.

2.3 Chemical modification of MWNT

MWNT includes impurities such as metal catalyst, cobalt and ion. So the acid solution was used to improve the purity by removing impurities such as metal catalyst that is included in MWNT. The mixed solution of HNO_3 and H_2SO_4 in the ratio 3:1 was made to improve interfacial adhesion when producing high molecule matrix using MWNT and was treated with ultrasonic wave for 60 minutes under normal temperature. The chemically revised carbon-nanotube was washed with distilled water for more than five times to reach pH 7 and to remove impurities on its surface. Its neutralization was checked with pH measurement paper. The washed carbon nanotube was dried in vacuum dryer at 60℃ for more than 12 hours.