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Electromagnetic interference shielding effectiveness of nickel-plated MWCNTs/high-density polyethylene composites

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ABSTRACT

Multi-walled carbon nanotubes (MWCNTs) were nickel (Ni)-plated chemically to enhance the electromagnetic interference shielding effectiveness (EMI-SE) of Ni-MWCNTs/High-density polyethylene composites (Ni-MWCNTs/HDPE). The surface properties of the Ni-MWCNTs were characterized by scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS), and X-ray diffraction (XRD). The EMI-SE of the Ni-MWCNTs/HDPE was tested by a EMI shielding analyzer. The EMI-SE of 3-MWCNTs/HDPE and 3-MWCNTs (30-Ni) showed approximately 5 and 12 dB at 1.0 GHz, respectively. The EMI-SE of Ni-MWCNTs/HDPE was enhanced compared to that of as-received MWCNTs/HDPE. Our results indicate that the Ni-MWCNTs can lead to a EMI-SE improvement due to the EMI adsorption behavior of the nickel particles.

1. Introduction

Electromagnetic interference (EMI) has emerged as a serious issue with growth of electronic devices and components [1,2]. EMI not only can cause operational malfunction of electric devices but also has a bad impact of human health. Therefore, EMI shielding materials have recently been developed by many researchers [3–6].

Metal materials have been commonly used to reduce EMI despite various disadvantages such as heaviness, rapid corrosion, and poor processability [7,8]. Because of metal's disadvantages, polymer-based composites consisting of carbon fillers embedded in a polymer matrix have recently received much attention as EMI shielding materials. Polymer-based composites have light weight, corrosion resistance, and good processability [9–13]. EMI shielding effectiveness (EMI-SE) of these carbon/polymer composites depends on intrinsic conductivity and aspect ratio of the carbon fillers [14–17]. Among various carbon fillers, carbon nanotubes offer a polymer matrix electrical conductivity and mechanical properties due to their high aspect ratio (>1000) and nanoscopic dimensions [18,19].

In previous works [20], the effects of multi-walled carbon nanotubes (MWCNTs) on EMI SE were examined, and EMI SE of the composites were effectively enhanced according to the concentration of MWCNTs. Following our precious results, we prepared nickel (Ni)-plated MWCNTs to make a better result because some studies reported the EMI-SE of metal/CNT/polymer composites. The studies also reported metal helped to absorb the electromagnetic waves [3,21–26].

Herein, we prepared Ni-MWCNTs/HDPE composites for EMI shielding. The MWCNTs were Ni-plated chemically, and were characterized by SEM, XPS and XRD to confirm the morphology and elemental compositional changes. The EMI-SE of the composites were investigated according to Ni-plating time.

2. Experimental

2.1. Specimen preparation

The matrix resin was HDPE (2200 J, MFI: 5.0 g/10mim, density: 0.96 g/cm^3, T_m: 126–136 °C, Izod impact strength: 10 kg-cm/cm at 23 °C), supplied by Lotte Chemical Co. of Korea. The MWCNTs, produced by a chemical vapor deposition (CVD) process, were obtained from Nanosolution Co. of Korea (purity: >95 wt.%, diameter: ≤10 nm, and length: 10–20 μm).

To obtain Ni-MWCNTs, we used a electroless Ni-plating method. The schematic diagram of the electroless Ni-plating process is

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illustrated in Fig. 1. Before the electroless nickel-plating process, the MWCNTs were pretreated in 5M HNO_3 for 30 min in order to increase interfacial adhesion between Ni and MWCNTs. They were sequentially activated in tin chloride ($SnCl_2$) and palladium chloride ($PdCl_2$) solution for 30 min each. Sn^{2+} ions were initially deposited onto the MWCNT surfaces. Pd^{2+} ions were reduced to Pd element by Sn^{2+}, allowing catalytic centers (Sn/Pd nuclei) to form. The Sn/Pd nuclei adhered to the MWCNT surfaces, and accelerated the metal plating during the electroless Ni-plating process. During the Ni-plating process, Sn/Pd nuclei reduced nickel ions to neutral nickel atoms and caused deposition of the nickel or alloy. Sodium hypophosphite was used as a reducing agent. The reducing agent have two types of synchronous reactions [27,28]:

The first type is the cathodic reaction of Ni^{2+}, H^+, and $H_2PO_2^-$ or the deposition of Ni–P alloy and the production of hydrogen:

$$Ni^{2+} + 2e^- \rightarrow Ni \quad (1)$$

$$2H^+ + 2e^- \rightarrow H_2 \quad (2)$$

$$H_2PO_2^- + 2H^+ + e^- \rightarrow P + 2H_2O \quad (3)$$

The second type is the anodic oxidation of $H_2PO_2^-$:

$$H_2PO_2^- + H_2O \rightarrow H_2PO_3^- + H + H^+ + e^- \quad (4)$$

The composition of plating solution and reaction conditions are listed in Table 1. The Ni-MWCNTs were obtained by dipping the MWCNTs into the plating solution for 5, 15, and 30 min.

The Ni-MWCNTs/HDPE composites were obtained using an internal mixer at 180 °C for 1.5 h. The preparation process of MWCNTs/HDPE composites is illustrated in Fig. 2. Prior to mixing, the HDPE pellets were fed to the internal mixer and previously heated to 180 °C for 15 min. After the pellets were partially melted, the rotors were run for 15 min at 60 rpm to completely melt the pellets. Subsequently, the melted HDPE were mixed with MWCNTs for 60 min to disperse MWCNTs. To prepare a sheet with 3 mm thickness, the mixtures were hot-pressed at 180 °C for 5 min under 8 MPa. Finally, the composites were cut to the dimension for EMI-SE testing (ø134 mm × 3 mm). All sample names are listed in Table 2.

Table 1
Composition and conditions of electroless Ni-Plating bath.

Composition & condition	
$NiSO_4 \cdot 6H_2O$	280 g/L
$NiCl_2 \cdot 6H_2O$	40 g/L
$Na_3C_6H_5O_7 \cdot 1.5H_2O$	15 g/L
$NaH_2PO_2 \cdot 2H_2O$	100 g/L
NH_4Cl	100 g/L
$PbNO_3$	30 g/L
pH	8.25
Temperature (°C)	90 ± 1
Plating time (min)	5–30

2.2. Characterization

The morphologies of the Ni–MWCNTs were measured by a field emission-scanning electron microscope (FE-SEM, S-4300SE, Hitachi, Japan) equipped with an energy dispersive spectroscope (EDS). The X-ray photoelectron spectroscopy (XPS, K-Alpha, Thermo scientific, USA) spectra of the Ni-MWCNTs were collected using a AlKα X-ray source (hν = 1486.6 eV). The base pressure in the sample chamber was maintained within in the range of 10^{-8} to 10^{-9} torr during analysis. The wide-angle X-ray diffraction (XRD, D2 PHASER,

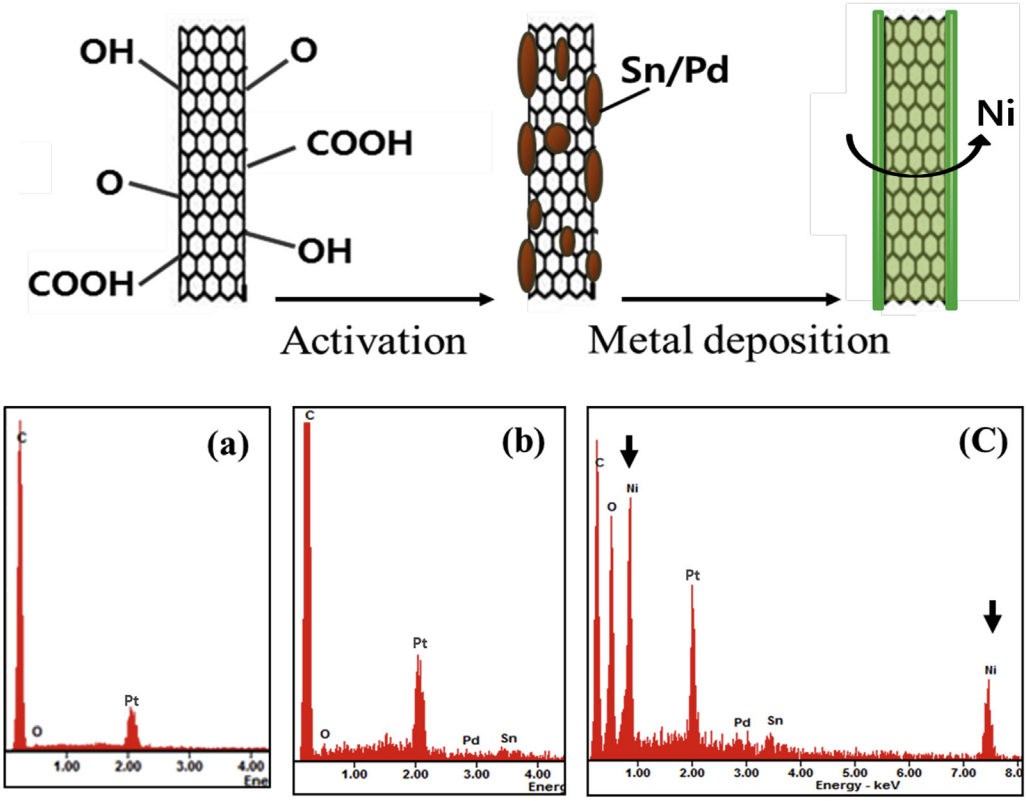

Fig. 1. Schematic diagram of the electroless Ni-plating processes and EDS spectra of Ni-MWCNTs; (a) as-received MWCNTs, (b) Sn/Pd MWCNTs, and (c) (30-Ni) MWCNTs.