



A particular interfacial strategy in PVDF/OBC/MWCNT nanocomposites for high dielectric performance and electromagnetic interference shielding

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ABSTRACT

Multi-walled carbon nanotubes (MWCNTs) were easily dispersed at the interface of polyvinylidene difluoride (PVDF) and ethylene- α -octene block copolymer (OBC) blend by melt compounding and the nanocomposites exhibited enhanced dielectric performance, electromagnetic interference shielding effectiveness (EMI SE) and balanced mechanical performance. Through the simple, efficient and scalable interfacial strategy to disperse MWCNTs at the interface of PVDF and OBC phases, the accumulation of charge carriers at the interfaces and strong interfacial polarization effect can be achieved. At low frequency, PVDF/OBC/MWCNT nanocomposite exhibits high dielectric permittivity (753.8) and low dielectric loss tangent (0.8), offering great potential in energy storage applications. Simultaneously, in X-band range, PVDF/OBC/MWCNT nanocomposite shows high EMI SE of around 34 dB which is higher than the industrial requirement when the content of MWCNTs is as low as 2.7 vol.%. This study provides possibilities to realize high-performance polymer nanocomposites via the particular interfacial structure through one-step melt processing.

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1. Introduction

Carbon nanotube (CNT)-polymer nanocomposites (PNCs) have shown great promise for many advanced applications such as actuators, pressure sensors, electro-active materials, electrostatic discharge (ESD) coatings, charge-storage capacitors, antistatic materials, and electromagnetic interference (EMI) shielding materials and so on, due to their excellent performance such as low density, easy processing, and low cost [1–9].

To obtain materials with high dielectric and EMI performance, much attention has been devoted to improve the compatibility of multi-walled carbon nanotubes (MWCNTs) and polymer matrix, i.e. interfacial engineering [10–12]. Construction of special structures is another strategy to improve the conductive, dielectric and EMI performance [13,14]. Huang et al. synthesized a core-shell structure composed of fluoro-polymer@BaTiO₃ nanoparticles and the nanocomposite shows high dielectric constant and low dielectric loss. [15] Zhao et al. prepared graphene/polymer nanocomposites with a segregated network via in situ reduction of graphene oxide (GO), which efficiently improved the electrical

conductivity of the nanocomposites [16]. Song et al. fabricated multilayer graphene/polymer nanocomposite films with sandwich structures to improve the electromagnetic interference shielding performance [17].

Although the utilization of nanoparticles offers many advantages for designing multi-functional polymer nanocomposites, there are still great challenges in fabricating high performance nanocomposites with specifically designed structures with simple and scalable technologies. To achieve highly conductive composites, high loadings of conductive fillers are usually added into polymer matrix, leading to complex processing procedures, high cost and poor mechanical properties due to the aggregation of nanoparticles and poor compatibility between nanoparticles and polymer matrix.

Many studies have tried to disperse nanoparticles at the interfaces of binary polymer blends to achieve high performance composites with a low amount of fillers [19,20]. As we know, the distribution of conductive filler in polymer is mainly controlled by kinetic and thermodynamic factors. Based on classical thermodynamics, the interfacial free energy at the interface should be the minimum if the fillers prefer to concentrate at the interface of two polymer phases, which is commonly achieved by grafting block copolymers or random copolymers onto the fillers to make them affinitive to the two immiscible phases simultaneously. Zhu et al.

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[19] dispersed most of MWCNTs with carboxyl groups at the interface of PMMA and PS phases by a thermodynamic strategy, which leads to a very low percolation threshold. Guo et al. [20] reported thermoplastic polyurethane (TPU) composites with low percolation threshold through the introduction of polyamide copolymer (COPA) to concentrate carbon black at the interface of TPU and COPA. However, the interfacial distribution of pristine MWCNT is still very difficult due to their high specific area and poor affinity with polymer.

In our previous work, a particular ‘sositie’ morphology was constructed in the ternary nanocomposite of isotactic polypropylene (iPP), ethylene- α -octene block copolymer (OBC) and MWCNTs, which leads to a low electrical percolation threshold, remarkably high dielectric permittivity and low dielectric loss, greatly enhanced toughness and balanced mechanical properties of the nanocomposites [18]. It seems that it is possible to disperse pristine MWCNTs at the interface of polar polymer/OBC blends due to the affinity between OBC and pristine MWCNTs.

In this work, a simple, efficient and scalable interfacial strategy to disperse MWCNTs at the interface of PVDF and OBC phases is presented, which leads to the accumulation of charge carriers at the interfaces and strong interfacial polarization effect. The interfacial polarized PVDF/OBC/MWCNT (FO-x, with a PVDF/OBC ratio fixed at 78/22 v/v and x referring the volume content of MWCNTs in the whole nanocomposites) nanocomposites and the PVDF/MWCNT (F-x, x referring the volume content of MWCNTs in the whole nanocomposites) nanocomposites used for comparison were fabricated by one-step melt compounding of the polymers with virgin MWCNTs. It is observed that most MWCNTs are distributed at the interface of FO-x composites, which significantly enhances the electric, dielectric, EMI and mechanical property of the composites. This study provides possibilities to realize high-performance polymer nanocomposites via the particular interfacial structure through one-step melt processing.

2. Experimental section

2.1. Materials

PVDF with the trade name of 906 and the density of 1.77 g/cm³ was purchased from Shanghai 3F New Materials Co Ltd. OBC (infuse 9807) with the density of 0.877 g/cm³, consisting of nearly amorphous soft blocks and crystallizable hard blocks, was from DuPont and synthesized by the chain shuttling polymerization technology. MWCNTs (XFM16), with an average diameter of 10–20 nm and length of 0.5–2 μ m, were purchased from XFNANO Inc. (Nanjing, China) and used as-received without further treatment. The apparent density of MWCNT is 1.8 g/cm³.

2.2. Sample preparation

PVDF and OBC were compounded with MWCNTs in the mixer of a torque rheometer XSS300 (Shanghai Kechang Rubber Plastics Machinery Set Ltd., Shanghai, PR China) with a rotor speed of 50 r/min at 190 °C for 8 min (PVDF: OBC = 78: 22 vol.%). The resulted composites were named FO-x where x stands for the volume fraction of nanotubes in the composites. For comparison, PVDF/MWCNT composites, labelled as F-x, where x stands for the content of MWCNTs by volume, were also melt compounded in the same mixer with the rotor speed of 50 r/min at 190 °C for 8 min.

After compounding, all the composites were compression molded into sheets with a thickness of about 0.5 mm at 190 °C for 10 min under a pressure of 10 MPa. For the evaluation of electromagnetic properties, all the composites were compression molded into samples with 10 mm diameter and 2.0 mm thickness.

2.3. Characterization

2.3.1. Contact angle measurements

Contact angle measurements were conducted with a KRÜSS DSA100 (German). The measurements were performed in sessile drop mold with water and diiodomethane as the wetting solvents. PVDF and OBC samples were compression molded at 190 °C for 5 min, and then cooled to 25 °C under pressure for 2 min. Contact angles were measured on 3 μ l of wetting solvent at 25 °C, and the results reported were the average values of at least five replicates.

The surface tension, dispersion and polar components of the materials can be calculated from the contact angle data by using Eq. (1) for water and Eq. (2) for diiodomethane according to Margolina and Wu [21]:

$$(1 + \cos \theta_{H_2O})\gamma_{H_2O} = 4 \left(\frac{\gamma_{H_2O}^d \gamma^d}{\gamma_{H_2O}^d + \gamma^d} + \frac{\gamma_{H_2O}^p \gamma^p}{\gamma_{H_2O}^p + \gamma^p} \right) \quad (1)$$

$$(1 + \cos \theta_{CH_2I_2})\gamma_{CH_2I_2} = 4 \left(\frac{\gamma_{CH_2I_2}^d \gamma^d}{\gamma_{CH_2I_2}^d + \gamma^d} + \frac{\gamma_{CH_2I_2}^p \gamma^p}{\gamma_{CH_2I_2}^p + \gamma^p} \right) \quad (2)$$

in which, $\gamma = \gamma^d + \gamma^p$, $\gamma = \gamma_{H_2O}^d + \gamma_{H_2O}^p$, $\gamma = \gamma_{CH_2I_2}^d + \gamma_{CH_2I_2}^p$, γ is surface tension, d is dispersion component, p is polar component, and θ_{H_2O} and $\theta_{CH_2I_2}$ are contact angles of the polymer with water and diiodomethane, respectively. Other numerical values used are $\gamma_{H_2O}^d = 22.1$ dyn/cm, $\gamma_{H_2O}^p = 50.7$ dyn/cm, $\gamma_{CH_2I_2}^d = 44.1$ dyn/cm, $\gamma_{CH_2I_2}^p = 6.7$ dyn/cm [22].

2.3.2. Morphological observation

The phase morphology was characterized with a JEOL JSM-5900LV scanning electron microscope (SEM, Japan). The samples were immersed in liquid nitrogen for 1 h, and then quickly impact fractured. Before SEM observation, all the fractured surfaces were gold sputtered. The accelerating voltage was 20 kV. Furthermore, the dispersion state of MWCNTs at the microcosmic scale was examined with a high-resolution transmission electron microscope (TEM, FEI, Tecnai G2 F20, FEI Company, USA) equipped with a field emission gun operating at 200 kV. Before observation, the samples were cryomicrotomed into 100 nm-thick sections at –120 °C.

2.3.3. Electrical conductivity

For the characterization of room-temperature volume resistivity, compression molded composites were cut into rectangular sheets with dimensions of 0.5 \times 10 \times 30 mm (both sides of the samples were coated with silver paste to reduce the contact resistance) and 0.5 \times 150 \times 150 mm, respectively. Then the volume resistivity of the samples were measured consecutively by employing a digital multimeter (6517B, Keithley Instruments, Inc, Ohio, USA) (below 10⁶ Ω) and a high-resistivity meter (ZC36, Shanghai Precision Instruments Co., Ltd. China) (above 10⁶ Ω), respectively. At least five specimens were tested for each sample and the average results were reported. Before characterization, all the samples were stored at room temperature for one week.

2.3.4. Dielectric performance

The sheet samples with a thickness of about 0.5 mm for dielectric performance tests were compression molded at 190 °C for 10 min under a pressure of 10 MPa. The dielectric properties were determined by an impedance analyzer (German Novocontrol, Concept 50) at a voltage of 1 V and a frequencies range of 10²–10⁷ Hz.

2.3.5. Electromagnetic interference shielding performance

Electromagnetic interference shielding performance of the composites was measured with an Agilent N5230 vector network

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