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Novel vapor-grown carbon nanofiber/epoxy shape memory nanocomposites prepared via latex technology

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A B S T R A C T

In this study, novel vapor-grown carbon nanofiber (VGCNF)/epoxy shape memory (SM) nanocomposites were prepared via latex technology. The prepared nanocomposites showed excellent SM functionality. The mechanical and SM properties of the nanocomposites were significantly improved by adding VGCNF. © 2014 Elsevier B.V. All rights reserved.

1. Introduction

Shape memory polymers (SMPs), which have attracted considerable attention in recent years because of their scientific and technological significance, are a new class of stimuliresponsive materials that can maintain a temporary shape and subsequently recover their original shape by external stimuli, such as heat, light, magnetic field, and chemicals [1–4]. As novel smart materials, SMP composites can be potentially applied in microelectromechanical systems, biomedical engineering, and space deployable structures [5].

Epoxies, which are widely used in many non-SM applications, such as coatings, adhesives, construction, and manufacturing, exhibit high strength, good thermal stability, and chemical resistance. Many recent studies have proven the good SM properties of epoxies [6–8]. The shape recovery ratio and elastic modulus of epoxies range from 98% to 100% and from 2 GPa to 4.5 GPa, respectively [1]. Unfortunately, the processes of epoxies are very environmentally unfriendly, complex, expensive, and the breakage elongation of epoxies are usually very low.

In the current study, latex technology was used to prepare VGCNF/epoxy SM nanocomposites. The mechanical and SM properties of the prepared nanocomposites were also valuated. The

http://dx.doi.org/10.1016/j.matlet.2014.06.084 0167-577X/© 2014 Elsevier B.V. All rights reserved. advantages of this technique include simplicity, versatility, reproducibility, and reliability. This study is the first to prepare VGCNF/ epoxy SM nanocomposites from water-borne epoxy resin using latex technology.

2. Material and methods

Preparation of VGCNF/epoxy SM nanocomposites: A three-step procedure was used to prepare VGCNF/epoxy SM nanocomposites, as shown in Fig. 1. First, VGCNFs (VGNF[®], Showa Denko K.K., Japan) were added to water-borne epoxy resin (synthesized by phase-inversion technique in our previous research [8,9]) with particles ranging from 50 nm to 300 nm and dispersed to homogeneity using an intensive mixer in a beaker at room temperature. A curing agent (AB-HGA[®], Zhejiang Anbang New Material Development Co., Ltd., China) was then added to the mixture. The weight ratio of the water-borne epoxy resin to the curing agent was 4:1. Second, the mixture was frozen in liquid nitrogen for 5 min and the aqueous solvent was removed using a Labconco Free Zone freeze-dryer operated at 0.1 mbar and -20 °C for 48 h. The resulting composite powder was compressed into films at 120 °C for 2 h between polypropylene sheets under a pressure of 10 MPa.

Experimental procedure: The images of the water-borne epoxy resin and VGCNFs were examined under a transmission electron microscope (JEM-2100F, JEOL, Japan) operated at 200 kV. The morphology of the nanocomposites was conducted under a field





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Fig. 1. Schematic of the three-step process for preparing of VGCNF/epoxy nanocomposites via latex technology. (A) TEM micrograph of the water-borne epoxy resin particles; (B) TEM image of pristine VGCNFs; and (C) SEM image of the fracture surfaces of the VGCNF/epoxy nanocomposites (fractured under liquid nitrogen). The inset of Fig. 1(C) is a sample of the VGCNF/epoxy nanocomposites. The VGCNF concentration is 1.0 wt%.



Fig. 2. (A) Recovery procedure of 1.0 wt% VGCNF/epoxy nanocomposites by pouring hot water (65 °C) in a Petri dish; and (B) storage moduli and tan delta values of the pristine epoxy resin, and 1.0 wt% VGCNF epoxy nanocomposites obtained from DMA testing.

emission scanning electron microscope (Ultra 55, Zeiss, Germany) operated at voltage of 3 kV. The storage modulus and tan delta of the nanocomposites were determined via a dynamic mechanical analysis (DMA) Q800 (TA Instrument, America) at a frequency of 1 Hz; the temperature was increased from 0 °C to 100 °C at a rate of 5 °C/min. The static tensile test of the pristine epoxy resin and the 1.0 wt% VGCNF/epoxy nanocomposites were carried out via a testing machine (INSTRON 3367) at a crosshead speed of 5 mm/ min using dumbbell-shape specimens at room temperature. At least five effective specimens were tested. The shape memory properties of the pristine epoxy resin and the developed nano-composites were examined by the fold-deploy shape memory test

[7]. The dumbbell-shape specimens with a square base (approximately $150 \times 10 \text{ mm}^2$) and a thickness of 3.5 mm were heated to 65 °C above glass transition temperature (T_g) in the oven, and then bent into a "U" shape circling a central axis with a diameter of 54 mm. The specimens were cooled to 25 °C below T_g at 25 °C in the air and held with a constant external force for 10 min. The specimens were unloaded at 25 °C, completing the shape fixity process. The specimens were heated from 25 °C to 65 °C under no load to examine their shape recovery capacity. Shape fixity and recovery ratios are important parameters for evaluating SMP characteristics [7,10]. These parameters are defined in Eq. (1) [7]. The maximum bending angle, bending angle, and bending angle at

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