



Effect of epoxy-graft-polyoxyethylene octyl phenyl ether on preparation, mechanical properties and triple-shape memory effect of carbon nanotube/water-borne epoxy nanocomposites



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ABSTRACT

In the present study, the effect of epoxy-graft-polyoxyethylene octyl phenyl ether (EP-g-TX100) on the processing and properties of the novel carbon nanotube (CNT)/water-borne epoxy (WEP) triple-shape memory nanocomposites was investigated. Generally, common epoxy or CNT/epoxy nanocomposites only possess dual-shape memory effect (DSME) that can remember only one temporary shape. In addition, CNTs aggregation and chemically inactive surface have remained a vexing problem that limits CNTs in composite application. In order to obtain excellent CNT/epoxy triple-shape memory effect (TSME) nanocomposites prepared via environmental friendly approach, EP-g-TX100 was synthesized and introduced to the CNT-epoxy system as a reactive emulsifier for epoxy emulsion, a non-covalent dispersant for CNTs, and a new reversible phase for TSME epoxy system. The multifunction self-made TX100-dangled epoxy surfactant for preparing CNT/epoxy TSME nanocomposites was first reported. The experimental results show that EP-g-TX100 had a good emulsifying ability to emulsify epoxy in water. CNTs were homogeneously dispersed and well incorporated into WEP matrix and significantly improved the mechanical properties of the CNT/WEP nanocomposites. The final nanocomposites exhibited excellent TSME as we expected.

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1. Introduction

Shape memory polymers (SMPs), which have attracted considerable attention in recent years and will come to play a significant role in all areas of human life because of their scientific and technological significance, are a new class of stimuli-responsive materials that can maintain a temporary shape and subsequently recover their original shape by external stimuli. These stimuli include heat, water, pH, light, electric field, and magnetic field [1–5]. As smart materials, SMPs can be potential applied in aerospace structures, biomedical devices, sensors, textiles, dry adhesives, self-healing applications [1,2], and so on.

Recently, triple-SMPs (TSMPs) and multi-SMPs have attracted more attention due to the shape changes are not limited to be unidirectional rather than one could potentially offer unique

opportunities in many applications ranging from morphing aircrafts and fasteners, to medical devices [6–8]. Different from conventional dual-SMPs which can remember only one temporary shape and recovery from a temporary shape to a permanent shape, TSMPs are capable of remembering two temporary shapes and recovering sequentially from one temporary shape to the other, and eventually to the permanent shape. TSMPs, most of them either have more than one switching thermal transition [6] or have a single switching transition with a broad thermal transition range [7]. The triple-shape memory effect (TSME) can be achieved through many ways, such as, polymers blends, grafting and blocking copolymers, SMP hybrids, and polymer laminates [8]. Mather et al. [9] incorporated non-woven thermoplastic polycaprolactone fibers of a semicrystalline polymer into epoxy matrix. The resulting composites show excellent TSME. Xie et al. [7] revealed TSME via bilayers structure from epoxy of different glass transition temperatures. Kim et al. [10] reported that cohesive bilayers of two films fabricated from an interpenetrating polymer network (IPN) and exhibited TSME.

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Epoxy, exhibits good thermal stability and chemical resistance, is one important member in SMPs world [1,11]. The shape recovery ratio and elastic modulus of epoxies range from 98% to 100% and from 2 GPa to 4.5 GPa, respectively [1]. However, the vast majority of these products are still formulated with organic solvents. As environmental regulation becomes stricter, the requirement for the industries to switch to more ecological and safer systems is constantly increasing. Water-borne epoxy (WEP), as a novel environmentally-friendly materials, which is being attached great importance in engineering field and mainly used in concrete coatings, metal primers, epoxy cement concrete, glass fiber sizing, and wood adhesives [12]. Our previous research found that WEP has excellent dual-shape memory effect (DSME) [3,4], while possesses an inferior mechanical strength and a lower SM fixity ratio compared with solvent-based epoxy [1].

Improvements in the mechanical and SM properties of SMPs through incorporation of inorganic particles have been achieved. The most popular fillers for SMP matrices are carbon nanotubes (CNTs) [13,14]. For example, Ni et al. [15] found that the shape recovery ratio and recovery force of SM polyurethanes were significantly improved by incorporating a small amount of CNT. The Fe₃O₄-loaded CNT/polycaprolactone composite exhibited excellent shape memory effect (SME) triggered by an alternating magnetic field [16]. Jung et al. [17] modified CNTs using acid so that the CNTs could act as a cross-linking agent during the in-situ polymerization step of the SMP. Unfortunately, CNTs invariably exist in the aggregated form, especially at high load, due to strong van der Waals forces and a large aspect ratio [18]. In addition, the unique chemically inactive graphite sheet surface, as well as lower of surface functional group concentrations, and poor infusion with matrix such as polymers [18]. However, the effective utilization of CNTs in composite applications strongly depends on their homogeneously dispersion as well as having an excellent interfacial adhesion. Therefore, it is essential to modify the surface structure of CNT to enhance the dispersibility of CNTs into raw materials. Non-covalent functionalization by surfactants or polymers has been widely used in the preparation of both organic and aqueous solutions to obtain high weight fraction of individually dispersed CNTs [19–21]. Non-covalent functionalization is particularly attractive because of the possibility of adsorbing various groups onto the CNT surface without disturbing the π electron system of the graphene sheets. It is a useful method in tuning the interfacial properties of nanotubes. The non-covalent functionalization of CNTs lowers the CNT surface tension, effectively preventing the formation of aggregates. Moreover, surfactant-treated CNTs overcome the van der Waals attraction though electro static and steric repulsive forces [22].

As forementioned, a common epoxy polymer system only possesses DSME. In addition, CNTs aggregation and poor CNT-polymer interfacial adhesion are the key challenges in the processing or engineering of nanocomposites. In the present study, we attempt to develop novel CNT/epoxy nanocomposites with uniform CNTs dispersion, TSME, and prepared via environmental friendly method. For this purpose, a copolymer diglycidyl ether of bisphenol A-toluene diisocyanate-Triton X100 (EP-g-TX100) was introduced and used as a reactive emulsifier for epoxy emulsion, a noncovalent dispersant for CNTs, and a new reversible phase for TSME CNT-epoxy system. The performances of EP-g-TX100 in the triple-shape memory CNT/WEP nanocomposites were assessed and analyzed.

2. Material and methods

2.1. Materials

CNTs were obtained from Showa Denko K. K., (Japan) with an

average diameter about 80 nm [23]. The commercially available epoxy (EP, supplied by Wuxi Resin Factory, China), 2,4-toluene diisocyanate (TDI, supplied by Bayer, Germany) and curing agent (AB-HGF[®], Zhejiang Anbang New Material Development Co., Ltd., China) were obtained from commercial sources and used as received. WEP emulsifier EP-g-TX100 and WEP were synthesized in our previous research [4,24]. All dispersion experiments were performed with deionized water.

2.2. Techniques

2.2.1. Preparation of CNT/EP-g-TX100 solutions

CNTs were dispersed in water by mixing with a calculated content of EP-g-TX100 followed by intensive stirring with the water bath temperature maintained at room temperature. After stirring for 6 h, cold DI water was dropwisely added to the flask and the mixture then was stirred for another 18 h. The weight ratios of EP-g-TX100 to CNT ranged from 0.25 to 2.

2.2.2. Preparation of CNT/WEP nanocomposites

A three-step procedure was used to prepare the CNT/WEP nanocomposites. First, CNTs were added to a stainless steel cup with diluted WEP (5% solid weight) and dispersed to homogeneity using an intensive mixer at 3000 rpm for 6 h at room temperature. The curing agent was then sequentially added to the mixture. 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 -55°C for 7 d. Finally, the resulting composite powder was compressed into films at 120°C for 2 h under a pressure of 10 MPa.

2.2.3. Morphology characterization

CNT/WEP nanocomposites were examined using a transmission electron microscope (TEM, JEM-2100F, JEOL, Japan) operated at 200 kV. TEM images of the nanocomposites were obtained from ultrathin sections that were cut with a glass knife on the Leica EM UC7 ultramicrotome (Leica, Germany). Field emission scanning electron microscope (FE-SEM, Ultra 55, Zeiss, Germany) was applied to investigate the specimens at an operating voltage of 3 kV. Prior to testing, the samples were sputter-coated with gold to impart electrical conductivity and reduce charging artefacts. High vacuum conditions were applied and a secondary electron detector was used for image acquisition.

2.2.4. Tensile test and dynamic mechanical analysis (DMA)

Tensile test of the CNT/WEP nanocomposites were conducted using a testing machine (INSTRON 3367, USA) at a crosshead speed of 5 mm/min on dumbbell-shape specimens at room temperature. At least five effective specimens were tested for each sample. The nanocomposites were determined via a DMA Q800 (TA Instrument, USA) at a frequency of 1 Hz, and then heated from 0 to 100°C at a rate of $5^{\circ}\text{C}/\text{min}$. The test was conducted under engineering strain control, with a strain of 0.1% and a preload of 0.01 N.

2.2.5. Shape memory properties characterization

The TSME of the samples were conducted via a DMA Q800, using a strip specimen and strain mode. Firstly, straining the specimen at a stress rate of 0.1 MPa/min to 0.2 MPa yielded a temporary shape II at 65°C (original shape is I), marked as ϵ_{FII} ; and then the specimen was cooled to 40°C to release the stress, marked as ϵ_{II} . Secondly, the specimen was strained at a stress rate 0.5 MPa/min to 2.5 MPa to obtain another temporary shape III, marked as ϵ_{FIII} ; and then the specimen was cooled to 0°C to release the stress, marked as ϵ_{III} . Finally, the temporary shape III was heated to 40°C marked as ϵ_{RII} ; and the specimen was heated to 65°C marked as ϵ_{RI} . The heating or

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