



Functionally graded and self-assembled carbon nanofiber and boron nitride in nanopaper for electrical actuation of shape memory nanocomposites



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ABSTRACT

The present work studies the synergistic effect of self-assembled carbon nanofiber (CNF) and boron nitride (BN) nanopaper on the electrical and thermal properties, and electro-activated shape memory effect (SME) of polymeric shape memory nanocomposites. CNF and BN are first self-assembled on carbon fibers by means of deposition at a high pressure to form functionally graded nanopaper with high electrical and thermal conductivity. The resultant nanopaper is then coated on the surface of shape memory polymer (SMP) to enable actuation by means of Joule heating via passing an electrical current through CNFs and carbon fiber. Here, BN is introduced to improve the thermal conductivity and large dissimilarity of the nanocomposite for enhanced heat transfer and electric-activated shape recovery.

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1. Introduction

Shape memory polymers (SMPs) are featured by their ability to return the permanent shape after undergoing a process, known as programming, to introduce severe and quasi-plastic pre-deformation [1–4]. A specific stimulus is required to trigger the recovery [5,6]. Typical types of stimulus, include light [7,8], heat [9–12], water [13,14], and solvent [15–19], etc. Heating-responsive SMP composites loaded with functional fillers can be triggered by means of indirect heating by electric current (Joule heating) [20–29], or magnetic field (inductive heating) [30,31]. To achieve Joule heating, a small amount of conductive fillers, such as carbon nanotube [20–22], carbon nanofiber (CNF) [23,24], carbon black [25], electromagnetic particle [26,27], electrically conductive hybrid fiber [28], and continuous conductive carbon fiber [29], have been applied. However, in order to achieve high enough electrical conductivity the required amount of filler is higher, which results in high viscosity during processing and thus prevents not only uniform dispersion of fillers, but also efficient transfer of the properties of the fillers to SMP matrix [32].

The concept of electrically conductive composites based on nanopaper made of CNF has been previously explored [33], in which Joule heat is produced within the CNF nanopaper. However, the interface between nanopaper and SMP could be easily dam-

aged (or even burned out) upon heating due to remarkable dissimilarity in their thermal properties [34,35]. Consequently, the efficiency of heat transfer from nanopaper to SMP is reduced. In this work, we study the synergistic effect of self-assembled CNF and boron nitride (BN) nanopaper on the electrical and thermal properties, and electro-activated shape memory effect (SME) of SMP nanocomposites. CNF and BN are first self-assembled on carbon fibers by means of deposition at a high pressure to form functionally graded nanopaper with high electrical and thermal conductivity. The nanopaper is then coated on the surface of SMP for Joule heating induced thermo-responsive SME. Here, BN is introduced to improve the thermal conductivity and large dissimilarity of the composites to heat transfer and electrically activated shape recovery. A schematic illustration of the working mechanism of the functionally graded self-assembled CNF and BN in nanopaper for electrical actuation of shape memory nanocomposite is shown in Fig. 1.

2. Experimental details

CNFs were received in powder form and then heat treated at 2200 °C for 180 min to obtain complete graphitic structures and purify themselves to a high level of >99 wt%. The nanofibers have a diameter of 50–100 nm and length of 30–100 μm. BN was obtained by reacting boron trioxide (B₂O₃) with urea (CO(NH₂)₂) in a nitrogen atmosphere. The resulting disordered (amorphous)

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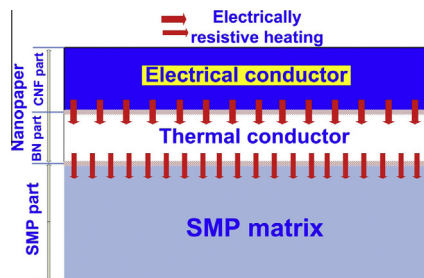


Fig. 1. Schematic illustration of the functionally graded and self-assembled CNF and BN in nanopaper for electrical actuation of SMP nanocomposite.

boron nitride contained 92–95% of BN and 5–8% of B_2O_3 . The amorphous BN was converted into crystalline structure and the remaining B_2O_3 was evaporated when heated to $1600\text{ }^\circ\text{C}$ for 12 h in nitrogen flow to achieve a BN concentration of >98%. BN is isoelectronic to a similarly structured carbon lattice. CNFs were firstly dispersed in distilled water with a non-ionic surfactant $C_{14}H_{22}O(C_2H_4O)_n$ (Triton X-100). The surfactant had a hydrophilic polyethylene oxide group and a hydrocarbon lipophilic group. The CNF suspension was sonicated with a high-intensity sonicator at $25\text{ }^\circ\text{C}$ for 40 min with an ultrasound power level of 300 W. Two duty cycles were applied for the dispersion of CNFs. After an initial 20 min sonication, both suspension and probe were cooled back to room temperature. The sonication was carried out again for another 20 min under the same condition. Subsequently, the CNF suspension was filtrated with a piece of hydrophilic polycarbonate membrane (with a diameter of 55 mm and $0.4\text{ }\mu\text{m}$ in gap) under a positive pressure. After this, BN was treated and prepared following the same approach as that of CNFs. In the next step, BNs were self-assembled and deposited on the surface of CNFs to form the final nanopaper. Finally, CNF and BN nanopaper was dried in an oven at $120\text{ }^\circ\text{C}$ for 2 h to remove remaining water and surfactant.

The SMP used in this study is an epoxy-based fully formable thermoset SMP resin, which is a two-part system. After mixing and curing, this polymer has the heating-responsive SME, in addition to high toughness and strength. Its glass transition temperature is $110\text{ }^\circ\text{C}$. Six pieces of nanopaper with different total weight and weight ratio of CNF:BN (namely, 0.10 g (0.08:0.02); 0.12 g (0.08:0.04); 0.14 g (0.08:0.06); 0.16 g (0.08:0.08); 0.18 g (0.08:0.10) and 0.20 g (0.08:0.12)) were used to prepare composite samples. They were individually placed on the bottom of a metallic mold. Resin transfer molding technique was used to make the SMP nanocomposite. The relative pressure of the resin transfer molding was kept constant around 6 bar. After filling the mold, the mixture was cured at a ramp of approximately $1\text{ }^\circ\text{C}/\text{min}$ from room temperature to $100\text{ }^\circ\text{C}$ and kept for 5 h before being ramped to $120\text{ }^\circ\text{C}$ at $20\text{ }^\circ\text{C}$ per 180 min. Finally, it was ramped to $150\text{ }^\circ\text{C}$ at $30\text{ }^\circ\text{C}$ per 120 min to produce the final SMP nanocomposites.

3. Results and discussion

3.1. Morphology and structure of CNF and BN in nanopaper

The morphology and structure of CNF and BN nanopaper were characterized by a field emission scanning electron microscopy (FESEM) at an accelerating voltage of 20.00 keV. As shown in Fig. 2(a), BN is hexagonal as graphite has a plate like morphology with layered lattice structure. Meanwhile, Fig. 2(b) reveals that a network structure was formed via molecular interaction and mechanical interlocking among individual nanofibers and BN in the nanopaper. Such a continuous network, in which CNFs work

as conductive path for electrons to move easily, makes the nanopaper electrically conductive. Fig. 2(c) is a typical image taken by a charge-coupled device (CCD) camera. The maximum magnifications of the ocular and objective are $10\times$ and $100\times$, respectively, and thus then final magnification is $1000\times$.

3.2. Fourier transform infrared (FT-IR) spectroscopy

Fig. 3 compares the Fourier transform infrared (FTIR) spectra (Nicolet AVATAR 360) of nanocomposites with 0.10, 0.12, 0.14 and 0.16 g of nanopaper, in the range from 4000 to 400 cm^{-1} . Ill-organized graphitic and aminated-CNF lead to the appearance of a series of bands with the maximum transmittance at 1392 cm^{-1} as D-band and 800 cm^{-1} as aminated-CNF, respectively. The D bonding of C=C ranges from 1378 to 1392 cm^{-1} with the concentration of CNFs from 0.08 to 0.14 g, which reveals the interaction force between CNF and BN.

3.3. Electrical resistivity measurement

The electrical resistivity of nanocomposites was obtained using a van der Pauw four-point probe, that is based on electrical impedance measuring technique which uses separate pairs of current-carrying and voltage-sensing electrodes to achieve higher accuracy than the traditional two-terminal sensing technique. The apparatus is incorporated with a Keithley 2400 resistivity tester and a Keithley 2000 picoammeter/voltage source and has four probes. A constant current passing through two outer probes and the output voltage was measured across the inner probes with a voltmeter. Fig. 4(a) presents the experimental results of each tested sample with twenty times of measurement by the mode I. Fig. 4(b) compares the resistivity at different weight concentration ratio of CNF–BN. As we can see, with the increase in BN contents in the nanopaper, electrical resistivity gradually decreases and then gradually increases. The average electrical resistivity decreases from 0.095, 0.090, 0.078 to $0.069\text{ }\Omega\text{ cm}$, as the weight of BN increases from 0.02 to 0.08 g. After that it increases from 0.069, 0.076 to $0.084\text{ }\Omega\text{ cm}$, as the weight of BN further being increased from 0.08 to 0.12 g. Nanopaper is a porous structure [36]. BN particles which permeate into the CNF nanopaper could provide a positive force to compact the individual nanofiber. Here the electrical resistivity of nanopaper is lowered due to the compacted CNFs. With the increase in BN content in the nanopaper, the particles permeate through the CNF nanopaper, which results in rearrangement of nanofibers. Hence, the continuous conductive CNF network is loosened, and thus, the electrical resistivity of the nanopaper decreases. It should be pointed out that the electrical properties of nanopaper are highly influenced by the positive pressure in the fabrication process.

3.4. Electrically induced shape recovery

All SMP nanocomposites were coated with one layer of functionally graded nanopaper incorporating with CNF and BN. The function and effectiveness of nanopaper in actuation of SMP nanocomposites by means of Joule heating were experimentally explored. Fig. 5 shows the shape recovery sequence of a nanocomposite specimen with 0.16 g nanopaper (0.08 g CNF and 0.08 g BN). The flat (permanent shape) specimen with a dimension of $60 \times 4.5 \times 1\text{ mm}^3$ and a small cut in the middle was bent into a “n” shape (temporary shape) at $160\text{ }^\circ\text{C}$ and then cooled back to room temperature with the deformed shape maintained. No apparent shape recovery was observed in the freestanding pre-deformed specimen within 60 min in air. A constant 4.8 V and 0.48 A direct current was applied through the tested pre-deformed specimen, so that the applied electric power was approximately 2.3 W. The

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