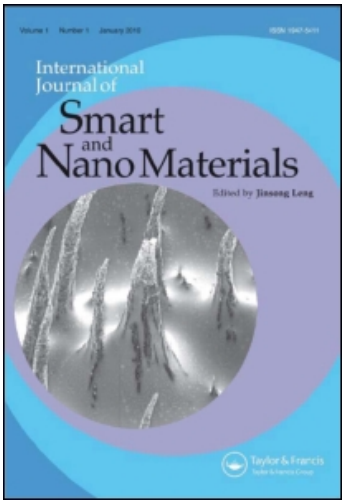

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Electroactive shape-memory polymer nanocomposites incorporating carbon nanofiber paper

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A conductive carbon nanofiber (CNF) paper is described for the actuation of shape recovery of a shape-memory polymer (SMP) by electrically resistive Joule heating. The CNF paper was manufactured using a traditional physical vapor deposition process and the microscale morphology and structure of the CNF paper were observed using a scanning electron microscope. The CNF paper was found to be porous, with the pore size determined by the weight concentration of CNF. The excellent electrical properties of pure CNF papers and their SMP composites were characterized by the four-point probe method. Shape recovery actuation of this type of SMP composite induced by electrically resistive heating was achieved with a 12 V voltage. Additionally, the thermomechanical properties of the SMP composite were studied with respect to the volume fraction of CNF paper.

Keywords: shape-memory polymer; resistive heating; electroactive; carbon nanofiber

1. Introduction

In recent years, the development and applications of shape-memory polymers (SMPs) have grown rapidly due to their outstanding advantages, such as ease in fabrication and manufacturing processes, high elastic deformation capability (up to more than 400%), and tailored recovery temperature in comparison with traditional shape-memory alloy materials [1–3]. These unique characteristics have led SMPs to be used in a myriad of fields, including clothing manufacture, automobile engineering, aerospace engineering, medical treatment, and many other applications [3–6]. SMPs are classified as thermally responsive materials that can undergo large deformation upon being heated above a particular transition temperature of the polymer or segment in the polymer. Such heat treatment makes the polymer easily deformable upon applying an external force. This deformed shape could be fixed when the polymer is cooled down below the transition temperature. Subsequently, SMPs can relax to and regain their original shape through additional external heating. Similar to other types of shape-memory materials, the application domain of SMPs is increasingly being explored. However, the actuation approach of shape recovery is seriously limited by traditional direct temperature heating procedures. Fortunately, many interesting and valuable studies have been done on the actuation of SMPs or SMP composites, for which shape recovery can not only be induced by

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external stimuli, such as heating, but can be manipulated by infrared light heating [5], laser light heating [6], electrical resistive heating [7–13], magnetic fields [14,15] or solvents [16–18].

Many significant developments have been achieved for SMP composites for which shape recovery actuation can be carried out by electrically resistive heating. However, for the fabrication of electroactive SMP composite, almost all previous works were focused on conductive fillers blended into pure polymers. This approach is not the best way to improve the conductive properties of polymers, as demonstrated in previous studies. With this as motivation, an attractive technique of making conductive carbon nanofibers (CNFs) or carbon nanotubes (CNTs) into paper form have been scaled up for functional materials [19,20]. This attempt was inspired by the successful manufacture of single-walled carbon nanotube (SWCNT) buckypaper [20]. However, owing to the high cost, CNF nanopaper has been widely used in practical applications to displace SWCNT buckypaper. Therefore, to utilize the characteristic properties of CNFs at the macroscopic scale, macroscopic assemblies of CNFs have been explored in the form of paper, fiber, pellet or film. They have been found to be useful for a huge range of potential applications, such as catalysts, sensors, actuators, capacitors, electrodes, and so on. These attempts were inspired by the successful manufacture of CNF nanocomposites, which can significantly improve the performance of a polymer, ceramic or metal matrix using a small amount of CNF. It is desirable to organize high-quality self-assembled CNF paper on an hydrophilic or hydrophobic membrane. Its structural characteristics, such as dispersion and orientation, imply that the electrical properties of scaled-up buckypaper will be superior to individual conductive particles blended into a polymer. Based on the excellent electrical properties of pure buckypaper, it is expected that composites incorporating CNF paper will possess outstanding electrical properties.

In this paper, the use is described of a CNF assembly paper integrated with a thermosetting SMP to fabricate electroactive SMP composites. The combination of outstanding electrical properties of CNF paper and shape memory effect of SMP will help this type of composite to be multifunctional. On the other hand, the actuation of the SMP composite can be carried out by electrical resistive heating.

2. Experimental

2.1. Raw materials

Vapor-grown carbon nanofiber (CNF) was supplied by Applied Sciences, Inc., Cedarville, OH. Table 1 presents the detailed physical properties of this type of CNF (Pyrograf®-III); this CNF is available with diameters ranging from 70 to 200 nm and with estimated lengths of 30–100 μm. Therefore, these CNFs are much smaller than milled carbon fibers (ranging from 5 to 10 μm) but significantly larger than single-walled carbon nanotube (SWCNTs) (1–10 nm). This type of CNF can simultaneously provide enhanced electrical conductivity and mechanical reinforcement of a polymer matrix. Another benefit provided by the nanofiber

Table 1. Physical property parameters of PR-25 Pyrograf®-III (CNF).

Property	Value
Bulk density	1.95 g/cm^3
CNF tensile strength	400–600 GPa
CNF modulus	2.7–7.0 GPa
Electrical conductivity	0.75 S/cm
Thermal conductivity	1950–2000 W/m K

includes improved heat distortion temperatures. Distilled water was used as a solvent. The non-ionic surfactant Triton X-100 ($C_{14}H_{22}O(C_2H_4O)_n$), which has a hydrophilic polyethylene oxide group and a hydrocarbon lipophilic group, was used to aid the dispersion of the CNFs. The styrene-base shape-memory resin Veriflex®S (VF 62), supplied from Cornerstone Research Group (CRG), Inc., was used as the matrix. For curing, the styrene resin was mixed with a dibenzoyl peroxide hardener at a weight ratio of 24:1.

2.2. *Fabrication of CNF paper*

The generally accepted method of making CNF paper (also called "buckypaper") involves the use of non-ionic surfactants such as Triton X-100 and sodium lauryl sulfate, which aids CNF dispersion into the aqueous or organic solvent. The CNF suspension needed to be sonicated for 30 min at room temperature. The suspension was then filtered through a 4.5 μm hydrophilic polycarbonate membrane filter with the aid of pressure to form paper. The suspensions were membrane filtered under positive pressure to yield uniform films. The individual CNF was bonded by van der Waals forces. After filtration, the remaining water in the CNF paper was dried in an oven at 120°C for 2 h. In this study, four weight concentrations of CNF specimens were prepared, with thicknesses of 1.5 mm, 3 mm, 4.5 mm and 6 mm for corresponding weights of 0.6 g, 1.2 g, 1.8 g and 2.4 g, respectively. Figure 1 shows the CNF paper specimen. The van der Waals forces between the nanofiber surface and the surfactant molecule are often mechanically strong and quite stable; therefore, it is not certain that all the surfactant was removed from the CNF paper after formation. Additionally, to avoid adverse side effects from the possible presence of surfactants, an alternative casting process can be used involving a frit compression method that does not require the use of surfactants or surface modification [21].

2.3. *CNF paper-enabled SMP composite*

The SMP composites were fabricated by coating CNF papers onto the surface of SMP sheets by hot-press modeling. In this process, the papers were first placed on the bottom of the metallic mold. The styrene-based shape-memory resin Veriflex®S (VF 62) was used as the matrix and mixed with dibenzoyl peroxide hardener at a weight ratio of 24:1. The resin must

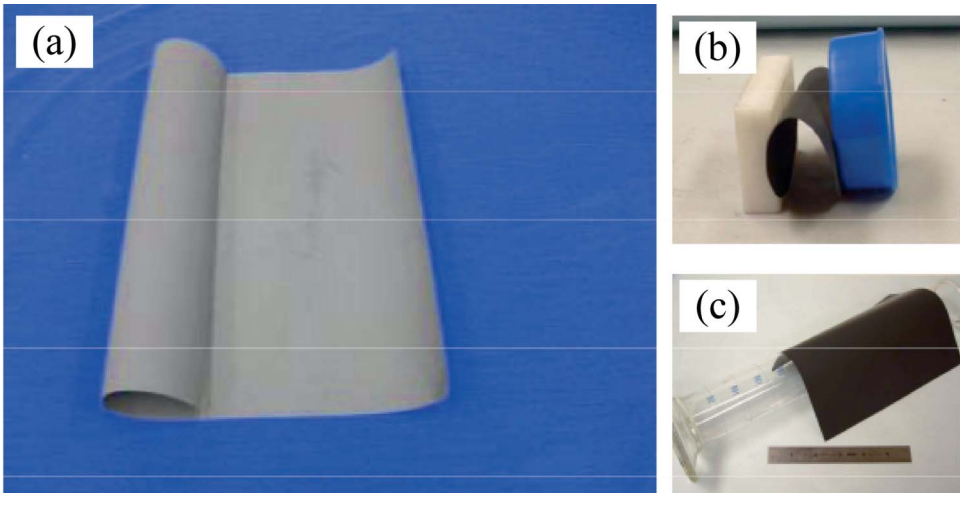


Figure 1. (a) Large-size carbon nanofiber paper (18″ × 18″). Carbon nanofiber paper sheet with (b) good strength and (b) flexibility to allow for handling like traditional fiber mat.

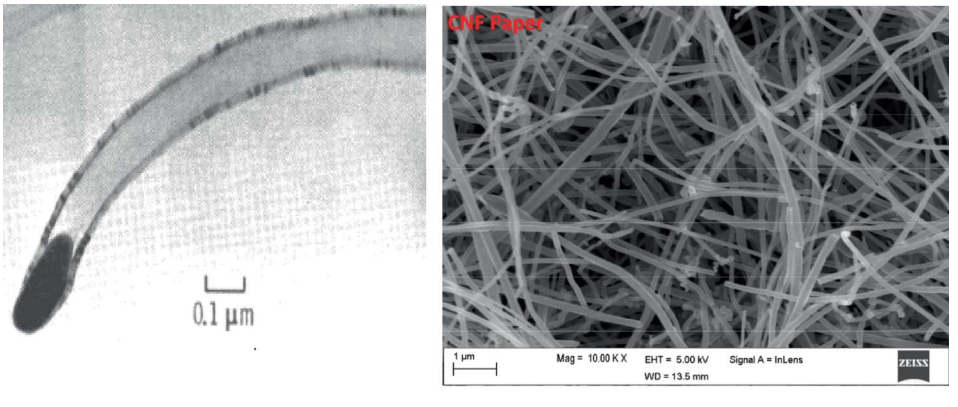
come into full contact with the curing agent in the mechanical mixing process. After the resin mixture filled in, the mold was cured in an oven at 75°C for 12 h. Five specimens were manufactured with various CNF paper weight concentrations and the same thickness using the procedure described. Sample A was pure SMP, sample B was SMP composite with 5 vol. % CNF paper, sample C was SMP composite with 10 vol. % CNF paper, sample D was SMP composite with 15 vol. % CNF paper, and sample E was SMP composite with 20 vol. % CNF paper.

3. Results and discussion

3.1. Characteristic morphologies of CNF paper

Carbon nanopaper is one-tenth the weight, yet potentially 500 times stronger than steel, and it can conduct electricity like copper or silicon. Therefore, there is a great deal of interest in determining its morphology to identify the reasons for its superior electrical properties. Scanning electron microscopy (SEM) images the sample surface by probing it with a high-energy beam of electrons in a raster scan pattern. When the primary electron beam interacts with the surface of the tested sample, the electrons lose energy by repeated random scattering and absorption within a teardrop-shaped volume of the specimen known as the interaction volume.

The morphology of the CNF array was characterized with SEM (ZIESS Ultra-55 at 10 kV), and Figure 2b shows a typical side view of raw CNF arrays on a 10 μm scale. The diameters of individual CNFs are seen to range from 50 to 100 nm. The CNF array was made up of well-dispersed individual nanofibers with very few aggregates due to the excellent dispersion in the solvent. Furthermore, no large aggregates of CNFs were observed, and individual nanofibers were closely packed within the network structure. Since individual CNFs are one of the most electrically conductive materials known, CNF paper is considered one of the best candidates for a conductive material that would allow an insulating polymer to behave like a conductor. This, in turn, could lead to even greater advances in enabling these continuous networks to act as conductive paths for electrons. On the other hand, the pores in the CNF paper are formed during the filtering process, and the pore size is determined by the quantity of CNFs and the applied pressure. Therefore, this porous character allows CNF paper to be used as a filter.



(a) (b)

Figure 2. (a) Catalytic lengthening and CVD thickening of Pyrograf®-III (reprinted from Applied Sciences Incorporated, http://www.apsci.com/ppi-pyro3.html). (b) SEM images of random CNF array form side view at the scale of 1 μm.

3.2. Dynamic mechanical analysis

The dynamic mechanical properties of the SMP composites were measured using a dynamic mechanical analysis (DMA) instrument. The TA Instruments Q800 DMA is a thermal analytical instrument used to test mechanical properties. In the measurement process, the test specimen is fixed on one of several clamps, all of which have been designed using finite element analysis to minimize mass and compliance. Basically, a deformation is imposed on the specimen in order to evaluate the intrinsic as well as extrinsic mechanical properties of the material. In this manner, the effect of the weight concentration of the CNF paper on the thermomechanical properties of the composite was studied and analyzed at different temperatures. All experiments were performed in the dual cantilever mode at an oscillation frequency of 1.0 Hz and a constant heating rate of 10.0°C min^{-1} from –20°C to 150°C. In the DMA measurements of composites with various weight fractions of CNF paper, the storage modulus, loss modulus and tangent delta were recorded with respect to temperature. The storage modulus is the modulus of the elastic portion of the material, while the loss modulus is the modulus of the viscous portion. The tangent delta, defined as the ratio of the loss modulus over the storage modulus, indicates the damping capability of a material.

Figure 3 shows the dynamic thermomechanical properties of pure SMP and SMP composites with CNF paper concentrations of 1.2 g and 2.4 g. These three samples were selected to characterize the effect of different CNF weight concentrations on the change of dynamic mechanical properties of SMP samples. At –20°C, the storage moduli of samples A, C and E were 2756 MPa, 2121 MPa and 1763 MPa, respectively. This result revealed that the storage modulus of the composite gradually decreased with increasing weight concentration of CNF paper. This phenomenon can be accounted for by the complex interaction between the polymer and CNF paper. As the SEM image shows, CNF paper is porous, especially at the microscopic level, with the result that resin penetrated into or even through the paper bulk during the polymer composite fabrication. When a thin paper (with a relatively low CNF mass) is incorporated with polymer, it is easier for the polymer to penetrate through the paper, resulting in a separation of the CNF arrays and polymer. Thus, the mechanical properties of this type of

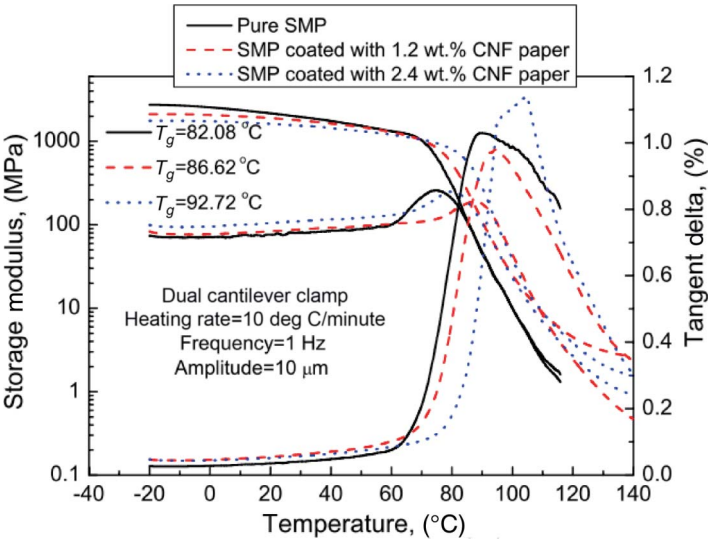


Figure 3. Storage modulus and tangent delta curves of composites as a function of temperature.

composite are sometimes decreased because there is no effective connection in the composite. On the other hand, the CNF paper is formed by individual CNFs that are held together by van der Waals forces. This molecular force, which is always lower than the chemical bonding force, leads the paper to have relatively weak mechanical properties on the macroscopic scale. Therefore, when the weight concentration of CNF paper in the composite increased, the mechanical properties of the composite were further weakened in comparison with the pure polymer. At the same time, the damping property of SMP integrated with CNF paper was improved, as shown in the tangent delta curves. The CNF paper is packed with individual CNFs, nanomaterials with a huge surface area. When an external force is applied, the CNF can help to disperse the force and enhance the damping performance of the composite.

Sometimes, DMA curves can also be used to determine the glass transition temperature (T_g) of polymeric materials. In this study, the T_g is defined as the point of intersection between the storage modulus and the tangent delta curves. The T_g values for the pure SMP specimen, SMP composite with 1.2 g CNF paper and SMP composite with 2.4 g CNF paper were found to be 82.08°C, 86.62°C and 92.72°C, respectively. This experimental result reveals that the T_g increased as the weight concentration of CNF paper increased. This again indicates that the presence of CNF paper has a positive effect on improving the damping properties of the polymer composite.

3.3. *Isothermal bending stress–strain tests*

The flexural strength of the SMP composites was measured using a three-point bending test on a Zwick/Z010 static materials testing machine. The static bending test was performed at a loading speed of 2 mm min^{-1}. The dependence of the CNF paper weight fraction on the flexural strength of the SMP composite was investigated at a testing temperature of 20°C.

Rectangular samples with dimensions of 49.5 × 12.1 × 3.0 mm were taken from the fabricated SMP composites. When the nanopaper was coated with SMP, the flexural modulus of the SMP composite was higher than that of the pure SMP. As shown in Figure 4, the flexural

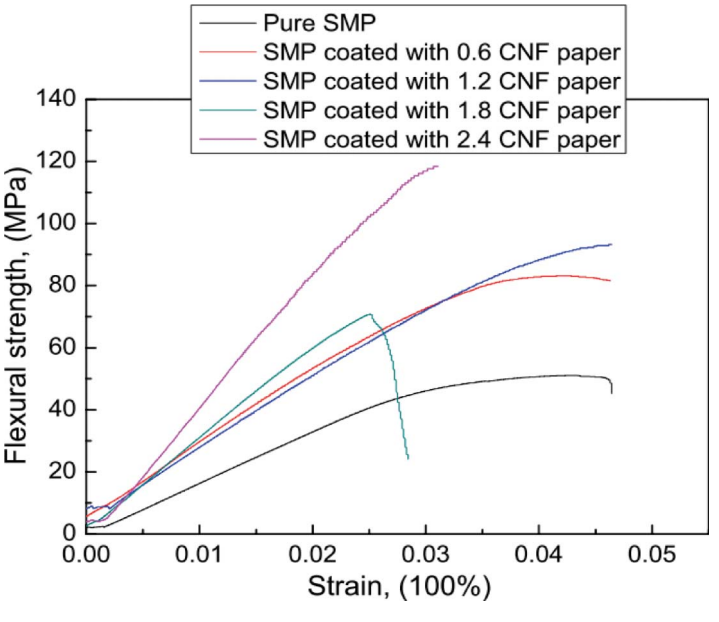


Figure 4. The flexural strength of composites vs. the weight fraction of CNF paper.

moduli of the SMP specimens with various weight concentrations of nanopaper were recorded as a function of flexural strain. When the weight fraction of the nanopaper increased from 0 g, 0.6 g, 1.2g, 1.8 g to 2.4 g, the flexural moduli of the SMP specimens were 70.36, 113.92, 126.08, 96.59 and 161.32 MPa, respectively. This phenomenon can be mainly attributed to the excellent mechanical properties of CNFs. Therefore, the mechanical properties of SMP composites depend on the weight fraction of CNF paper. However, as higher weight concentrations of CNF paper are introduced to SMP, the composite becomes more brittle and has a lower ultimate bending strain. In addition, because the CNFs are heterogeneously dispersed in the nanopaper, the flexural strength of the SMP composite will not show a regular pattern. The SMP composite specimens tested here have a higher flexural strength than pure SMP.

3.4. *Electrical property measurements*

The electrical resistivity of the pure CNF papers and SMP composites was measured with a four-point probe apparatus (Signatone Quadpro system), which consisted of a combination of four probes in a straight line with a constant inter-probe spacing (S) of 1.56 mm. Figure 5 shows a schematic representation of rectangular sample preparation for resistivity measurements.

When a Kelvin connection is used, electrical current is supplied with a pair of force connections. These connections generate a voltage drop across the impedance to be measured according to Ohm's law: $V = IR$. This current also generates a voltage drop across the force wires themselves. To avoid including that drop in the measurement, a pair of sensor connections are made immediately adjacent to the target impedance. The sense wires are

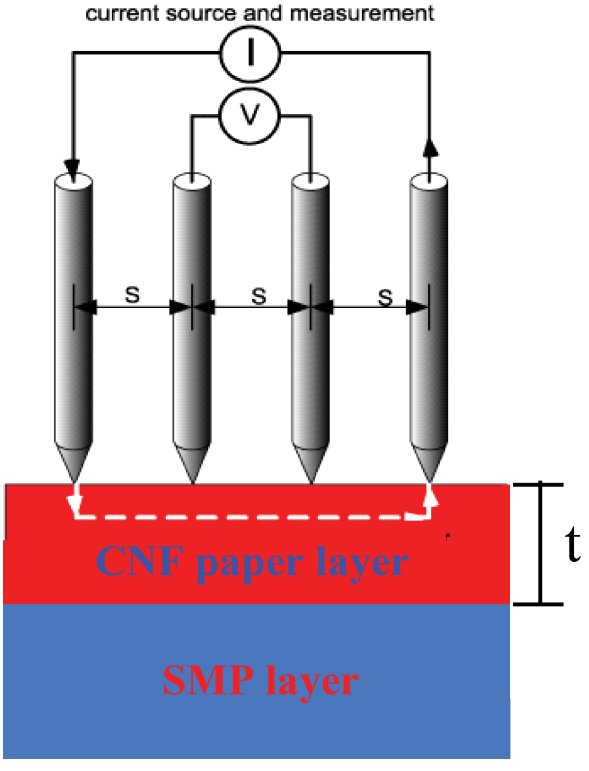


Figure 5. Schematic representation of resistivity measurement.

conventionally placed as the inside pair, whereas the force wires are the outside pair. The force and sense connections can theoretically be exchanged without affecting the accuracy of the technique, but this leads to uncertainty regarding exactly where within the force connection the resistance measurement begins, as the force connections are relatively large so that they can carry the necessary current, whereas the sense connections can be very small. The key difference between the four-probe measurement and the traditional two-probe method is that the separation of current and voltage electrodes in a four-point probe measurement allows the ohmmeter/impedance analyzer to eliminate the impedance contribution of the wiring and contact resistances, given that the voltage electrodes have sufficiently high input impedance.

The characteristic electrical resistivity was measured as a function of the weight of pure CNF papers and their composites at room temperature. Based on previous results, the electrical resistivity of sample A was considered to be 10^{16} Ω cm [8]. Figure 6 shows a comparison of the resistivities of pure CNF paper and of SMP composites with varying CNF paper weight concentrations. The electrical conductivity of SMP coated with 1.8 g CNF paper was measured as 1.77±0.08 Ω cm. This is lower than most previously reported conductive SMPs with comparable filler contents (see Table 2). This low electrical resistivity allows fast activation of shape recovery by applying a constant voltage. This occurs because more CNFs are involved in the conductive networks, leading to a greatly reduced probability that a site is occupied by a pore. This evolution also plays the same role in the electrical properties of SMP composites. However, comparing the two curves, the resistivity of composites with 0.6 g, 1.2 g and 1.8 g paper is higher than that of the corresponding pure paper, whereas the resistivity of composites with 2.4 g paper is slightly lower than that of the corresponding paper. Perhaps the interaction between polymer and paper should be considered to account for this phenomenon. As previously mentioned, the polymer penetrates into the paper bulk. When the resin penetrates through the paper, the pores in the conductive paper structure become filled with the insulating polymer, resulting in an increased electrical resistivity of the composites. However, when only part of the paper is filled with polymer,

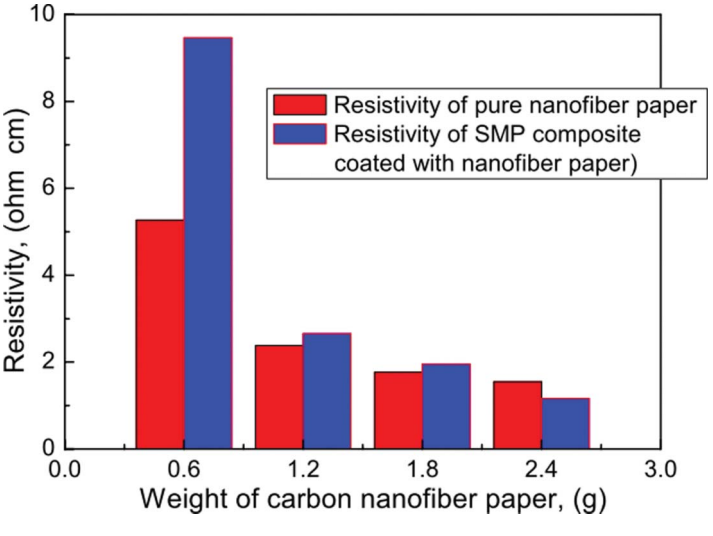


Figure 6. Comparison of the electrical resistivity of pure CNF paper and of SMP composites with varying CNF paper weight concentrations.

Table 2. Comparison of electrically conductive SMPs reported in the literature.

Reference	SMP composite system	Electrical resistivity (Ω m)
[7]	Polyurethane SMP filled with multi-walled carbon nanotubes	~10
[9]	Polyurethane SMP filled with Ni powders	0.1218
[10]	Polystyrene SMP filled with 5 wt % carbon black and 2 wt % short carbon fiber	0.0891
[11]	Polyurethane SMP filled with Ni powders and carbon black	~0.1
Current work	Polystyrene SMP coated with 15 vol. % CNF paper	0.017

even though the polymer provides strong bonding and acts as a bridge between conductive arrays, the other part of the paper has more closely packed conductive arrays, leading to a decrease in electrical resistivity. This is the reason that the electrical resistivities of SMP composites with high weight concentrations of CNF paper are lower than pure CNF paper.

3.5. Function of CNF paper in shape recovery actuation

Another active area of research is the use of electrical resistive Joule heating to trigger the shape memory effect of SMPs, which is desirable for engineering applications where it would not be possible to use heat. Current efforts to implement this technology use conducting SMP composites with carbon nanotubes [7], short carbon fibers (SCFs) [8,9,12], and metallic Ni powder [10,11]. The shape memory effect in these types of SMPs have been shown to depend on the filler content, and they exhibit good energy conversion efficiency and improved mechanical properties.

Another technique under investigation involves the use of surface-modified superparamagnetic nanoparticles. Introduction of these electromagnetic particles into the SMP matrix enables remote actuation of shape transitions by an electromagnetic field. An example of this approach involves the use of oligo-(ε-caprolactone) dimethacrylate/butyl acrylate composite with between 2–12% magnetite nanoparticles. Nickel and hybrid fibers have also been used with some degree of success [11,13,14].

Previous studies on the shape memory of SMPs driven by electrical resistive heating have shown some disadvantages, such as requirements for excessive filler content or voltage, as well as a severely inferior shape memory effect. Our study used a 1.2 g conductive CNF paper, an extraordinarily low applied voltage of 12 V, and an excellent recoverability approaching 100% of the SMP composite, demonstrating excellent electrically induced shape memory, as shown in Figure 7. A straight SMP (permanent shape) sheet was bent into an "n-like" shape at 80°C, and it retained this shape during cooling back to room temperature. No apparent recovery was observed after the deformed sample was kept in the absence of external forces, as shown in Figure 7 at 0 s. However, when a voltage of 12 V was applied for about 20 s to the composite specimen (sample C), conversion of the flexural shape was observed. With another 80 s of applied voltage, the specimen showed a relatively fast response to electrical resistive heating. A change in shape from the temporary shape to the permanent shape was completed within 140 s. The final shape was close to the original straight shape with some remaining flexion due to friction among the polymer chains. This experimental outcome demonstrates that the SMP composite with conductive CNF paper not only has an electroactive response, but also has a relatively better shape memory effect than the polymer matrix blended with conductive filler or fillers.

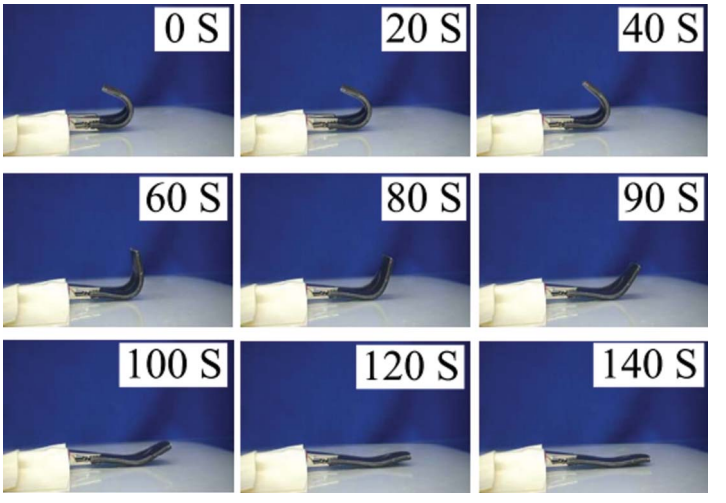


Figure 7. Demonstration of shape recovery driven by electrical resistive heating, as recorded by digital camera.

4. Concluding remarks

This study reports the development of SMP composites with incorporated CNF paper with actuation triggered by electrical resistive heating. The novel feature of this composite is the integration of a shape memory effect with outstanding electroactive responsive behavior. The following conclusions can be drawn from this study:

1. Owing to the excellent electrical properties of CNF paper, incorporation of this material enables production of an SMP composite with proper electrical conductivity.
2. The CNF paper is porous, with the result that the thermomechanical and electrical properties of the composite are determined by the interaction between the polymer and the CNF paper.
3. The electrical behavior of CNF paper with the proper weight concentration is suitable for SMP actuation by electrical resistive heating. This can be extended to achieve SMP material actuation driven with a low electrical voltage.
4. It is expected that the SMP recovery process can be carried out in a programmable and controllable manner by controlling the dispersion of individual CNFs in the paper.

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