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Abstract

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Self-Monitoring and Self-Correcting Polymer Fibers coated with Carbon Nanotubes

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Abstract

Bandaging, which has been used either to support a medical device such as a dressing or splint, or on its own to provide support to the body or restrict the motion of a part of the body, is further studied in our work in the form of multifunctional materials for self-monitoring and self-correcting by integration with carbon nanotubes (CNTs). Here, modified polymer fibers for bandages can be fabricated from fibers of commercially available bandages with carbon nanotubes uniformly adhering to the fibers, via a simple solution process to produce tough, flexible, and electrically conducting fibers. The conductivity of the coated bandage fibers is reversibly sensitive to strain, and strain in the coated bandage fibers also can be generated by electrical heating, leading to proof-of-concept sensor and actuator demonstrations.

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1. Introduction

Driven by some major factors such as an increasing aging population with more chronic wounds and the unexpected sufferings of civilians from terrorist attacks, warfare, and frequent casualties from traffic accidents, there is an increasing demand for advanced wound care products.[1, 2] Many advanced materials and nanotechnology have been used to improve their functions, including drug delivery, biodegradable scaffolding, functional adhesion, etc.[3-5] Commercial bandages can be used either to support a medical device such as a dressing or splint, or on their own to provide support to the body or restrict the motion of a part of the body. When there is more severe damage, such as an acute ankle sprain, a supportive ankle brace has to be used to limit the motion of the broken ankle. From the viewpoint of the patients, owing to the additional appurtenance, their motion looks clumsy, and the dressings have an unacceptable cosmetic appearance. It would be ideal if the passive appurtenance could be replaced by some portable, beautiful, practical dressings for wound healing. Here, we introduce the concept of self-monitoring and self-correcting polymer composite fibers for developing advanced bandages.

Traditionally, wound dressings merely act as a cover for the wounded area so that the wound can be rehabilitated underneath. Today, wound dressing materials combine many functions in one. For example, interactive materials, including polymeric films or foams (such as hyaluronic acid, hydrogels, and foam), are used to prepare wound dressings due to their good barriers against permeation of bacteria.[6-9] Since the conventional wound dressing materials are not suitable to promote the rapid healing of an acute wound, bioactive materials, such as hydrocolloids, alginates, collagens, and chitosan have been added or used to produce the wound dressing.[10-12] Meanwhile, a series of bandages with various kinds of antibiotics incorporated have been reported, such as a bandage that releases nitric oxide, leading to enhanced wound healing.[7] As research continues, more and more functions, including hemostasis, absorbability, semi-permeability, and conformability, have been explored.[13] In existing bandage technology, however, not much work has been undertaken to study or monitor the deformation of the bandage and correct its

deformation, especially in motion.

Fortunately, some fundamental work has already been carried out by some pioneers. Ray H. Baughman and his coworker demonstrated that inexpensive high-strength polymer fibers can be easily transformed by twist insertion. After adding an additional heating element to the fiber, the extreme twisting generates 5.3 kilowatts of mechanical work per kilogram of muscle weight, similar to that produced by a jet engine.[14] Even so, a passive heating system would make its components more complicated and limit the further application of the scaffold. In addition, as carbon nanotube (CNT) networks are increasingly utilized as highly sensitive sensors for detecting the onset, nature, and evolution of damage in composites,[15] more and more CNT-based sensor materials have been proposed, such as carbon nanotube yarn strain sensors.[16] In the example of human-motion detection, a thin film of aligned single-walled carbon nanotubes were used in wearable and stretchable devices to monitor the deformation of clothing worn over the skin.[17] The CNT-based stretchable electronics can be fabricated, since they are based on the intertwining and curvilinear nature of CNTs, which makes CNTs intrinsically stretchable.[14, 18, 19] Meanwhile, CNT, as one of electrothermal materials could convert electric energy into heat which was already demonstrated in literature [20] Therefore, we propose a new design based on applying an active heated layer of CNT to simplify an otherwise complicated system.

In this study, we have designed and developed a novel CNT/bandage (CNT/B) composite fiber via a simple “brushing” method for multifunctional bandage materials. The assembled CNT/B composite fiber shows a strikingly strain-dependent resistance relation as a human motion detector. Further research also shows that the accumulated strain on the polymer fiber can be released following the generation of heat due to the heat effect of the CNT layer. Finally, a novel bandage composed of self-monitoring and self-correcting composite fibers enhanced by CNTs is demonstrated.

2. Experimental

2.1 Methods

The CNT/B composite fibers were prepared through a “brushing and drying” method. In a typical process, 200 mg CNTs and 30 mg sodium dodecyl benzene sulfonate (SDBS) were dispersed in 200 ml deionized water, and the resultant dispersion was probe-sonicated for 3 h at 200 W to form a CNT ink. Then, a commercially available bandage (First Aider’s Choice) was cleaned several times with water and ethanol, followed by complete drying and cutting into small pieces with width and length of $5 \times 20 \text{ cm}^2$, respectively. All bandage samples were brushed using a simple “brushing and drying” method, as shown in Figure S1 in the Supporting Information. After drying at 60°C for 12 h, the bandage samples were washed with abundant amounts of water to remove the surfactant. Then, the bandage samples were dried again under the same conditions to remove the water completely. The washing and drying was continued until the resistance of the conductive CNT/B composite fiber was slightly altered. Here, we denote the samples with CNT/B composite fibers that were twisted 50 times, 100 times, and 150 times as FC-50, FC-100, and FC-150, respectively.

2.2 Characterization

Scanning electron microscope (SEM) images were obtained by using a JEOL JSM-7500 scanning electron microscope. Transmission electron microscope (TEM) images were collected using a JEOL JEM-2010 microscope at 200 kV. The microstructure of the samples was investigated on a Raman system (Thermo Nicolet Almega XR Raman Microscope, with the excitation wavelength at 532 nm). Thermogravimetric analysis (TGA) measurements (Mettler-Toledo TG/DTA system) were conducted on the samples from 60°C to 1000°C at a heating rate of 10°C/min under argon atmosphere. Mechanical measurements of CNT/B composite fiber bundles were performed using a universal tensile machine (Instron 5900) equipped with a 1 kN load cell. Before testing, the number of fibers was determined using an optical microscope with dark field illumination. Four-probe resistivity measurements were carried out in the temperature range between 10 and 300 K with silver paste electrical contacts and a 14 T physical properties measurement system (PPMS). The

distance between the inner electrodes was 1 mm. The resistance was obtained by applying a fixed electric current of 10 μA through the two outer silver contacts and monitoring the voltage drop between the two inner contacts.

3. Results and discussion

Figure 1. (a) Fresh bandage. (b) The coated bandage samples after the final drying. (c) A bundle of fibers coated with the CNTs. (d) Comparison of the Raman spectra of the pure bandage and CNT/B composite fiber. (e) TGA curves of the pure bandage and CNT/B fibers. (f) SEM image of CNT/B composite fiber. Scale bar: 100 μm . (g) Magnified SEM image of a CNT/B composite fiber. The fiber has a tetrahedral cross-section, and its width is $\sim 15\mu\text{m}$. Scale bar: 10 μm . (h) High magnification SEM image of CNT/B composite fiber, indicating uniform covering by the CNT, Scale bar: 100nm. (i) Cross-section of CNT layer on the bandage fiber. Scale bar: 1 μm .

Commercially available bandages can be used for many purposes, such as support, dressing retention, and compression, due to their versatile performance. Figure 1(a) shows a typical conforming bandage composed of bundles of polymer fibers. After “brushing and drying” several times, the resultant composite shows a uniform black appearance in Figure 1(b). Here, we used a bundle of CNT/B fibers as our initial research material (Figure 1(c)).

Raman spectroscopy is powerful tool to determine the composition of composites. Raman spectra of the pure bandage and CNT/B fibers are shown in Figure 1(d).

Compared with Raman spectrum of the pure bandage, the CNT/B fibers show the typical carbon nanotube G-mode feature at 1590 cm^{-1} and D-mode feature at 1330 cm^{-1} , respectively, which indicates that the CNTs were coated on the surfaces of the polymer fiber.[21]

To further evaluate the amount of CNT in the CNT/B fibers, TGA was carried out to determine the thermal decomposition behavior of the samples, and the results are presented in Figure 1(e). Between 400°C and 450°C , the polymer fibers seem to decompose drastically at the maximum weight loss rate. Afterwards, the weight loss rate starts to flatten, which continues until the end. Depending on the weight reduction of the sample, the mass fraction of the CNTs on the polymer fibers was calculated to be 7.1 wt%.

Figure 1(f-i) shows SEM images of CNT/B fibers at different magnifications. Each individual fiber was uniform in cross-section, and the cross-section shows a tetrahedral shape with a width of $\sim 15\text{ }\mu\text{m}$. Most importantly, the CNT layer also uniformly covers the surfaces of the polymer fibers, forming an intercrossing and interconnected layer for the transport of electricity. Compared with the camber of the surface of a cylinder, the tetrahedral plane would provide less local stress and a more uniformly porous morphology.[22]

Figure 2. Mechanical performance of the samples: Tensile strain-stress curves (a) and elongation at the break point (b) of commercially available thread, polymer fiber bundles, and CNT/B composite fiber bundles.

Figure 2(a) shows the stress-strain curves of commercially available cotton thread, a polymer fiber bundle, and a CNT/B bundle, respectively. Here, the commercially available cotton thread was used as the reference. In the typical stress-strain curve, three regimes of deformation can be observed: the straightening, elastic, and plastic regimes.[23] Compared with the cotton thread, the polymer fiber and the CNT/B bundle show better elasticity, as can be seen in Figures 2(b) and S1(g-i). The strength of a CNT/B bundle (~ 37 MPa) is a little higher than that of a fiber bundle (~ 35 MPa). From its stress-strain curves, we can clearly see that the elastic regime would be longer than for the pure fiber bundle, which also shows different deformation behavior. The axial tension leads to an equal distribution of stresses, which are transferred mostly through the tension deformation of the fiber, whereas the deformation of fibers introduces stresses into the CNT layer on the fiber surface. The stress on the outer layer is further transferred among the CNTs by the combination of the shear and adhesive forces and results in gliding lamellae. The experimental results suggest that the interlocked CNTs will provide better adhesive performance and extend the elastic regime on the curves.

Figure 3 Conductive properties of the samples: (a) Temperature dependence of the resistivity of CNT/B bundle under zero-field. (b) Resistance changes after bending the CNT/B composite fiber bundle into different radii.

The temperature dependence of the resistivity of a CNT/B bundle is shown in the Figure 3(a)). As the temperature decreases from 300 to 15 K, the resistance of the fiber bundle shows a slight increasing trend, while between 15 K and 10 K, it exhibits a rapid drop, which is rather similar to the values measured for a reported carbon nanotube bundle.[24]

Figure 3(b) shows that the conductive fiber bunch can be bent down from a 6 mm radius to a 3 mm radius without any measurable change in electrical conductivity. This can be attributed to the uniform stress and flexible porous morphology of the carbon nanotubes on the tetrahedral plane. Therefore, its superior flexibility and high conductivity make the conductive fiber bundle promising for various flexible devices. Here, the flexible and conductive fiber bundles were studied for application in human motion sensors and strain actuators to further enlarge the functions of the bandages containing them.

Figure 4. Performance of fiber bundles in human motion application. (a-d) Demonstration of the strain-sensitive resistance as a human motion sensor for a straight and bent arm, using several CNT/B bundles. (e) Schematic illustration

showing the junctions among the CNTs on the surface of fiber in the elastic regime. (f) Resistance versus strain for the fiber bundles coated by different CNTs contents up to 70% strain. (g) The calculated gauge factors of CNT/B bundles at different CNT contents. (h-i) Cyclic resistance of fiber bundles under varying strain between 0 and 70%. (j-k) Cyclic resistance of fiber bundles as the arm in (a-d) unbends and bends, respectively.

Resistivity-strain data recorded for the CNT/B bundles in Figure 4(f) show a monotonic increase in the resistivity up to 70% strain (Strain speed, 2mm min⁻¹). The gauge factor, GF, of a strain gauge is defined as the ratio between the electrical resistance change and the deformation in the gauge:

$$GF = \frac{\Delta R/R_0}{\varepsilon}$$

Where $\Delta R/R_0$ is the relative change in the resistivity and ε is the strain in the gauge.[25] And the resistance versus strain curves is shown in Figure 4(f) and 4(g) and its GF of a CNT-coated fiber bundle is ~6.6. In comparison, CNT film has a GF of 0.82 (0 to 40% strain), and CNT fiber has a GF of 0.56 (0 to 200% strain);[15] To further prove its stability of CF, the CF of CNT/B bundles at the different CNT content (13.4wt% and 5.9wt%) shows an extreme slight variation. Here, the hybrid fiber bunch could deliver the high GF because of the excellent performance of the polymer substrate. Nevertheless, the GF value for our CNT-coated fibers is approximately 6 times larger than for a reported CNT based strain sensor.[16] In Figure 4(e), the different mechanisms that affect the changing resistivity are shown during its deformation. A CNT/B bundle consists of many fibers with a tetrahedral cross-section, and each fiber has a slight twist, resulted in its curly morphology, as is shown in the SEM images in Figure 1. The curly morphology will provide not only many “local connections” between the fibers, but also allow for the recovery from stress at low strain. Therefore, the reversible sliding will release more “local connections”, even at low strain, and alter the resistance of the CNT/B bundle. After that, with increasing strain, plastic deformation of the polymer fibers and elastic

deformation of CNT layer occur, and the resultant “sliding” of the CNTs further decreases their contact area. Finally, the resistivity of the CNT/B bundle will show the variation as the change of strain. Further testing was used to evaluate its durability and stability. Figure 4(h) and (i) show the relative change in resistivity under the strain testing of a fiber bundle at 20% and 70% strain, respectively. The R/R_0 shows almost no variation, even after 1000 cycles.

To demonstrate the potential of the CNT/B bundles as a self-monitoring material to detect human motion, we fabricated a piece of CNT/B bandage by connecting electrodes. To fix the CNT/B bandage, adhesive tape was used to enhance the connection between the detector and the body, as shown in Figure 4(a-c). The resistivity data, as shown in Figure 4(j) and (k), were recorded as the arm unbent and bent, with corresponding changes in the strain from 0 to ~60%, which already exceeds the limits (5%) for conventional strain sensors. Such a device could be designed for monitoring the motion of wounded joints, waists, arms, feet, legs, etc. to help the healing process.

Figure 5. Tensometer measurement data for the twisted CNT/B bundle: Photographs of the original (a) and the twisted CNT/B bundle (b), scale bar: 1 mm. (c) Schematic illustration of single fiber coated by CNT. (d) Mechanism of electrothermal contraction. (e) Contraction stress of CNT/B bundle after twisting under different operational voltages. Contraction stress (f) and speed of response (g) of samples after twisting for different numbers of twists. Contraction stress (h) and speed of response (i) of FC-150 under different operational temperatures.

As shown in Figure 5(a) and (b), the as-prepared CNT/B bundle consists of parallel fibers; after twisting, the resultant crimp indicates its high stress. Figure 5(c) shows the basic structure of CNT/B composite: the outer shell is the CNT layer, which not

only provides the pathways for the electrons, but is also the generator of heat. The inner core is the polymer fiber, which is responsible for the mechanical properties.

Based on this coaxial structure, an appropriate current will make the CNT layer generate heat and act as a local heater for the polymer fiber, as shown in Figure 5(d).

To investigate thermal contraction behavior, the generated contraction stress at the voltages of 5, 15, and 25 V was measured and recorded for FC-100 in Figure 5(e).

The generated contraction stress increases as the operational voltage rises. After applying an operational voltage of 5 V, there is almost no additional contraction force generated due to the slight thermal expansion. As the voltage further increases to 25 V,

the generated stress is close to 25 MPa, which is nearly 4 times that at 5 V.

Nevertheless, the stress generated is still lower compared with the mechanical strength of commercial polymer fiber (Figure 2(a)). We further studied the release of stress via changing other factors, including twisting and operational temperature, and the results are shown in Figure 5(f), (g), (h), and (i). As the twisting increases, the stress released in CNT/B at 25 V is enhanced. Meanwhile, the speed of response is also accelerated. Moreover, when we decrease the operational temperature, the stress released in CNT/B is further improved to 32 MPa, close to the mechanical limit for CNT/B, and its speed of response decreases. The same as with most materials, after twisting, the stress increases as the operational temperature decreases; and the stress is also easy to release, although a low operational temperature would play a negative role in terms of heating the CNT/B, so compared with the high operational temperature, its speed of response is decreased.

Here, we have demonstrated that the twisted CNT/B can generate stress driven by current. This suggests that after detecting a change in the strain, the subsequent current can make the bandage fiber release some contraction stress spontaneously to decrease the damage.

4. Conclusion

In conclusion, we have developed a simple “brushing” method of fabricating tough, self-monitoring, and self-correcting polymer fibers from a bandage coated by carbon

nanotubes. After a simple coating of CNTs, when acting as a simple sensor, the resultant CNT/polymer fiber shows high sensitivity and long stability against stress. More importantly, as the thermal effect from the electric heating element, the CNTs, sets in, the stress from the twisted CNT/polymer fiber can be released. Here, in the case of bandages, the innovative self-monitoring and self-correcting performance can be developed to widen the application of wound dressings. Moreover, the concept of self-monitoring and self-correcting would have various applications in detection-control systems, as well as energy storage and conversion materials. In actual use, however, bandages are almost inevitably in a wet environment, such as from soaking with blood. Therefore, the future work will focus on the design of a new structure for evaluating our CNT coated bandages in the actual situation.

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