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## Novel Wearable Energy Devices Based on Aligned Carbon Nanotube Fiber Textiles

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Portable and wearable energy devices are critically important to the mainstream direction in modern electronics.<sup>[1–5]</sup> To this end, it is a key to develop energy devices that are thin, lightweight, transparent, and flexible. Supercapacitors have been widely studied as promising candidates in energy storage because of their high power densities<sup>[6-8]</sup> and their availability to be made into all-solid-state devices. Flexible supercapacitors are mostly explored to achieve high electrochemical performances by sandwiching an active layer between two electrodes<sup>[9-12]</sup> but cannot effectively meet the wearable and lightweight requirements. As a result, interest in developing miniature wire-shaped supercapacitors that could be woven into clothes with light weights has increased.<sup>[13-16]</sup> Although much effort has been made to enhance the electrochemical performance of wire-shaped supercapacitors, weaving them into applicable energy storage clothes has not been achieved. The thin, wire-shaped supercapacitors with diameters on the order of micrometers are easily damaged and form short circuits during the weaving process.

Recently, some attempts have been made to fabricate flexible supercapacitors based on textile electrodes<sup>[17]</sup> that have been prepared by depositing electrically conducting materials on conventional fabrics. For instance, high performance supercapacitors have been fabricated by coating various activated carbon materials such as carbon nanotubes and graphene onto cotton or polyester fabrics as electrodes using a solution process;<sup>[18,19]</sup> the cotton textile could also be directly carbonized into the required electrode for the fabrication of hybrid supercapacitor where pseudocapacitive MnO<sub>2</sub> particles have been further incorporated for high specific capacitances.<sup>[20]</sup> Although these supercapacitors derived from commercial fabrics can be produced with low cost, they are relatively heavy due to the formation of thick film electrodes in relation to the wearable application. An ideal wearable energy device is expected to be thin, lightweight, and even transparent in addition to flexible.

Here, a thin, lightweight, transparent and flexible supercapacitor textile is developed by using aligned multiwalled carbon nanotube (CNT)/polyaniline (PANI) composite fiber-based

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textiles as electrodes. In particular, this supercapacitor has been further integrated with a photoelectric conversion function to form a novel, self-powering energy textile that can convert solar energy to electric energy and simultaneously store it by designing a stacked structure mimicking multilayered clothes.

Figure 1 schematically shows the fabrication process for the supercapacitor starting from a spinnable CNT array that has been synthesized by chemical vapor deposition. Aligned CNT sheets are dry-drawn from the array and stacked into a thicker film along the length direction, followed by twist into an aligned CNT fiber. The aligned CNT fibers are then woven into textiles and further electrodeposited with PANI to obtain the designed electrode. The CNT/PANI composite fiber-based textile is first coated with a layer of PVA-H<sub>3</sub>PO<sub>4</sub> gel electrolyte, and two textile electrodes are then stacked to produce the supercapacitor. Due to the high mechanical strength and electrical conductivity from the aligned CNT and remarkable electrochemical activity from PANI, the resulting supercapacitor textile demonstrates a specific capacitance of 272.7 F g<sup>-1</sup> that can maintain 96.4% after bending for 200 cycles.

The prepared CNT fiber can be controlled from several to tens of micrometers in diameter with a density of 1 g cm<sup>-3</sup>. In particular, the linear density of the CNT fiber was calculated on level of 10  $\mu$ g m<sup>-1</sup>, several orders of magnitude lower than 10 and 20–100 mg m<sup>-1</sup> for cotton and wool yarns, respectively.<sup>[21]</sup> Supporting Information Figure S1 shows a scanning electron microscopy (SEM) image of a CNT fiber with uniform diameter. The CNTs showed a multiwalled structure with a diameter of 13 nm (Supporting Information Figure S2). The CNT fiber is flexible and can be easily made into a knot without damages in structure (Supporting Information Figure S3). Therefore, these CNT fibers can be woven into textiles for a large-scale application. Figure 2a shows a typical SEM image of the CNT fiberbased textile, and the CNTs remain highly aligned (Figure 2b) to enable excellent mechanical strengths and conductivities. The voids among aligned CNTs are varied from tens of to hundreds of nanometers.

The CNT fiber-based textile can be further electrodeposited with PANI through a three-electrode system where platinum foil and Ag/AgCl serve as counter and reference electrodes (Supporting Information Figure S4). The weight percentage of PANI is determined by the electron transfer numbers and controlled by the electrodepositing time during synthesis. Figures 2c–f and Supporting Information Figure S5 show SEM images of the CNT/PANI composite fiber-based textiles with increasing PANI weight percentages. Obviously, the PANI is uniformly deposited on the surfaces of aligned CNTs; the size of voids among aligned CNTs decrease with the increasing

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Figure 1. Schematic illustration to fabrication of a supercapacitor textile based on the CNT/ PANI composite fiber.



PANI weight percentage as the voids have been filled with more PANI. At a critical weight percentage of 50%, the voids among CNTs are full of the PANI. With the further increase in weight percentage, the additional PANI mainly aggregates into particles (Supporting Information Figure S5). Supporting Information Figure S6 shows Raman spectra of bare CNT fiber-based and CNT/PANI composite fiber-based textiles. As expected, the C-H bending of the quinoid ring at 1159 cm<sup>-1</sup>, C-H bending of the benzenoid ring at 1245 cm<sup>-1</sup>, C-N stretching at 1331 cm<sup>-1</sup>, and C-C stretching of the benzene ring at 1475 and 1619 cm<sup>-1</sup> are observed in the CNT/PANI composite fiber. Fourier transform infrared spectroscopy was also used to analyze the CNT/PANI composite (Supporting Information Figure S7). The two peaks at 1558 and 1489 cm<sup>-1</sup> were attributed to the C=C stretching vibrations of quinoid and the benzenoid rings; the peak at 1303 cm<sup>-1</sup> was attributed to the C-N stretching vibration of the benzene ring; the peak at 1113 cm<sup>-1</sup> was attributed to the stretching vibration of C=N. These characteristic peaks demonstrated the formation of CNT/PANI composite as expected.



**Figure 2.** SEM images. a) A textile woven from aligned CNT fibers. b) High magnification of an aligned CNT fiber. c–f) Aligned CNT/PANI composite fibers with PANI weight percentages of 15%, 30%, 40%, and 50%, respectively. g) A Ti wire textile grown with perpendicularly aligned TiO<sub>2</sub> nanotubes. h) Cross-sectional view of a modified Ti wire grown with the TiO<sub>2</sub> nanotubes on the surface. i) Aligned TiO<sub>2</sub> nanotubes.





**Figure 3.** a) CV curves of bare CNT fiber-based textile and CNT/PANI composite fiber-based textile with PANI weight percentage of 50% as electrodes at a scan rate of 20 mV s<sup>-1</sup> and potential range from -0.2 to 0.8 V. b) Galvanostatic charge/discharge profiles of supercapacitor textiles with different PANI weight percentages at a current density of 1 A g<sup>-1</sup>. c) Dependence of specific capacitance on PANI weight percentage at a current density of 1 A g<sup>-1</sup>. d) Dependence of specific capacitance on current density.

The CNT/PANI textile was coated with PVA-H<sub>3</sub>PO<sub>4</sub> gel electrolyte and two textile electrodes were stacked into the supercapacitor. Cyclic voltammetry (CV) was used to study the electrochemical properties of supercapacitors. Figure 3a compares the bare CNT fiber-based textile and CNT/PANI fiber-based textile with PANI weight percentage of 50% at a scan rate of 20 mV s<sup>-1</sup>. Obviously, a typical rectangular shape corresponding to an electrochemical double layer capacitor is found for the bare CNT textile, while redox peaks stemmed from different oxidation states of PANI indicate a pseudocapacitance in the CNT/PANI composite textile. As expected, the current density for the CNT/PANI composite textile is much higher than the bare CNT textile. Figure 3b further compares galvanostatic charge-discharge curves of the bare CNT and CNT/PANI composite textiles with increasing PANI weight percentages at a current density of 1 A g<sup>-1</sup>. All curves are nearly symmetric during charge and discharge processes, indicating a high reversibility. When the PANI weight percentage reached 60%, a bend was observed in the galvanostatic charge-discharge curve. This phenomenon is ascribed to the pseudocapacitance property of PANI at high loads, and the same phenomena had been previously observed.<sup>[22,23]</sup> The specific capacitances are first increased with the increasing PANI weight percentage and then slightly decreased with the further increase in weight percentage (Figure 3c). More specifically, the specific capacitances are increased from 7.7, 108.5, 152.8 and 201.8 to 272.7 F  $g^{-1}$ with the increasing weight percentages from 0, 15%, 30%, 40% and 50% at a current density of 1 A  $g^{-1}$ , respectively. When the PANI weight percentage reaches 60%, the specific capacitance is slightly decreased to 240.6 F g<sup>-1</sup>. The high pseudocapacitance

of PANI contributes to the increased specific capacitance below 60%. However, the PANI aggregates into particles beyond the critical point, which decreases the contact with aligned CNTs with a reduced charge transport. The dependence of specific capacitance on current density is also investigated (Figure 3d). No obvious decreases are observed for the supercapacitor textiles at increasing current densities, showing that the supercapacitor can effectively work at a wide range of current density. CNT/PANI composite materials had been widely investigated for electrochemical supercapacitors,<sup>[24]</sup> and the specific capacitances were typically ranged from 200 to 500 F g<sup>-1</sup>. For instance, a PANI-coated CNT fabric composite electrode showed a specific capacitance of 200 F g<sup>-1</sup> in 1M NaCl electrolyte;<sup>[25]</sup> a PANI/CNT paper-like electrode displayed a specific capacitance of 332 F g<sup>-1</sup> in a gel electrolyte.<sup>[26]</sup> Here, the wearable supercapacitor textile was comparable with the previous reports.<sup>[24,25]</sup>

The supercapacitor textile with the PANI weight percentage of 50% had been carefully investigated. **Figure 4**a shows CV curves with increasing scan rates; the curve shape is well maintained. A typical triangular shape under increasing current densities that corresponds to a nearly ideal capacitive behavior has been maintained for the galvanostatic charge/discharge curve (Figure 4b), so the supercapacitor textile can be stably performed also at a wide range of current density. Figure 4c and Supporting Information Figure S8 show the cyclic performance of supercapacitor textiles based on bare CNT and CNT/ PANI composite textiles both with high capacitance retention. As expected, no decay occurs in capacitance for the bare CNT textile after 2000 charge–discharge cycles. Although conducting





**Figure 4.** Electrochemical characterizations of the supercapacitor textile with PANI weight percentage of 50%. a) CV curves at increasing scan rates. b) Galvanostatic charge/discharge profiles at increasing current densities. c,d) Dependence of specific capacitance and Coulombic efficiency on cycle number at 1 A g<sup>-1</sup>, respectively.

polymers generally suffer from low cyclic stability as electrode materials, the CNT/PANI composite textile produces a high cyclic stability, e.g., over 90% in capacitance after 2000 charge– discharge cycles, possibly due to the remarkable mechanical and electronic properties from aligned CNTs. In addition, the Coulombic efficiency is also maintained by almost 100% during 2000 charge–discharge cycles (Figure 4d).

Due to a thin and grid structure, the supercapacitor textile is transparent (**Figure 5**a) and shows an optical transmittance of  $\approx$ 90% from the UV–vis spectrum (Supporting Information Figure S9). Here the CNT/PANI composite fiber exhibited a density range of 0.52–0.72 g cm<sup>-3</sup>, so the supercapacitor textile is lightweight and can be lifted up even by a tender flower (Figure 5b). The supercapacitor textile is flexible and can be bent with different angles without obvious fatigues in structure (Figure 5c). The CV curves are also maintained during the bending (Figure 5d), demonstrating stable electrochemical properties. The specific capacitance retains by 96.4% after bending for 200 cycles with a bending angle of 150° (Figure 5e).

The supercapacitor textiles can be further assembled in series or in parallel to tune the output voltage or capacity. Supporting Information Figure S10 compares the galvanostatic charge/discharge curves of single and three assembled supercapacitors in series at the same charge–discharge current. The potential window is extended from 0.8 V for a single supercapacitor to 2.4 V for the three supercapacitors connected in series with the same charge/discharge time. The three supercapacitors connected in series can efficiently power a red light emission diode (Supporting Information Video S1). When three supercapacitors are connected in parallel, the potential window remains the same while the charge/discharge time is tripled compared with the single supercapacitor.

The wearable supercapacitor textile was found to be suitable for the further integration with the other energy device such as solar cell. Photoelectric conversion (PC) and electrochemical storage are generally two separated parts and have been mainly connected by external circuits during use. This conventional connection is unfavorable for the portable and wearable electronic facilities.<sup>[27]</sup> Learning from the multilayered textile structure in the clothes (**Figure 6**a), the supercapacitor textile may be integrated with the PC function by designing a layered textile structure.

The PC part was first fabricated from a Ti wire textile grown with perpendicularly aligned TiO<sub>2</sub> nanotubes by the electrochemical anodization. Figure 2g shows a SEM image of an anodized Ti wire textile. The TiO<sub>2</sub> nanotubes shared a length of appropriately 29 µm (Figure 2h) and diameter of ≈100 nm (Figure 2i). A CNT fiber textile was then covered onto the Ti wire textile with the electrolyte injected between them to produce the PC part. For liquid and gel electrolytes, power conversion efficiencies of 3.2% (Supporting Information Figure S11) and 2.2% (Supporting Information Figure S12,S13) had been achieved, respectively. Here the gel electrolyte was prepared mixing poly(vinylidene fluoride-co-hexafluoropropene) bv (10 wt%) and 3-methoxypropionitrile solution containing 0.1 M LiI, 0.05 M I<sub>2</sub>, 0.5 M 4-tert-butylpyridine and 0.5 M 1-propyl-2,3-dimethylimidazolium iodide. The gel electrolyte was mainly studied for the integrated energy textile due to the convenience in fabrication.

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**Figure 5.** a) Photograph of a transparent supercapacitor textile. b) Photograph of a supercapacitor textile being lifted up by a tender flower. c) Photograph of a supercapacitor textile being curved without damage. d) CV curves of a supercapacitor textile being bent into different angles at a scan rate of 20 my s<sup>-1</sup>. e) Dependence of specific capacitance on bend cycle number of a supercapacitor textile with PANI weight percentage of 50% (bending angle of 150°).  $C_0$  and C correspond to the specific capacitances before and after bending, respectively.

The integrated energy textile was fabricated by stacking the PC part with the supercapacitor textile (Figure 6b). Figure 7a,b have showed the photocharge and discharge curves of the integrated all-solid-state energy textiles based on bare CNT fiberbased and CNT/PANI composite fiber-based supercapacitor textiles, respectively. The discharge process was realized by a galvanostatic method with a constant current density of 1 A  $g^{-1}$ . The voltage was maintained at  $\approx 0.70$  V, slight lower than the open-circuit voltage of the part, possibly due to the loss of electric power during connection. For the bare CNT fiber-based integrated textile, the voltage was increased to 0.70 V in  $\approx$ 0.5 s; while for the CNT/PANI composite fiber-based integrated textile, the voltage was increased to 0.70 V in ≈25 s due to a higher specific capacitance in the CNT/PANI fiber-based supercapacitor. Accordingly, the glavanostatic discharge time for the CNT/ PANI composite fiber was  ${\approx}100$  s,  ${\approx}33$  times longer than the bare CNT fiber. The energy storage efficiencies of the integrated energy textile were calculated as 68.4% and 60.3% for the bare CNT and CNT/PANI composite, respectively. Entire photoelectric conversion and storage efficiency had been calculated by

multiplying the storage efficiency and photoelectric conversion efficiency and used to characterize the overall performance of the energy textile. For the bare CNT fiber-based integrated allsolid-state energy textile above, it was calculated to be 1.5%; when the liquid electrolyte was used for the PC part (Supporting Information Figure S14,S15), a higher entire photoelectric conversion and storage efficiency of 2.1% was achieved.

In summary, a thin, lightweight, transparent and flexible supercapacitor textile has been developed on the basis of the unique grid structure of the CNT fiber-based textile electrode. Due to the aligned structure of CNTs in the grid that enables a high mechanical strength and electrical conductivity and uniform deposition of PANI that provides a high electrochemical activity, the supercapacitor textile displays stable specific capacitances up to 272.7 F g<sup>-1</sup>. The supercapacitor textile has been further integrated to create a new energy textile that can convert solar energy to electric energy besides storing it, and a high entire photoelectric conversion and storage efficiency of 2.1% is achieved. This work also presents a general and effective strategy for the development of high-performance and





**Figure 6.** a) Photograph of multilayered clothes. b) Schematic illustration to the integrated energy textile. The enlarged view shows the working mechanism.

multifunctional energy devices by designing composite textile electrodes.

## **Experimental Section**

The synthesis of spinnable CNT array is described in the Supporting Information. Aligned CNT fibers were prepared from the CNT array.<sup>[21,28]</sup> The CNT fibers were finally woven into textiles by a textile technology. CNT/PANI composite fiber-based textile was synthesized by electrochemically depositing aniline onto the CNT fiber-based textile in a three-electrode system, where the platinum foil and Ag/AgCl were used as counter and reference electrodes, respectively. Prior to the electrochemical deposition, the CNT fiber textile was immersed into the electrolyte for 5 min. Electrochemical deposition was performed at a potential of 0.75 V in a solution containing 0.1 M aniline and 1.0 M H<sub>2</sub>SO<sub>4</sub>. After deposition, the resulting CNT/PANI composite fiber-based textile was washed with deionized water for three times and then dried at 70 °C for 1 h. The weight percentage of PANI was calculated from the total Faradic charge consumed in the electrochemically



**Figure 7.** Typical photocharging and galvanostatic discharging curves of the integrated all-solid-state energy textiles based on a) the bare CNT fiber and b) aligned CNT/PANI composite fiber textile electrodes.

depositing reaction with an average of 2.5 electrons per monomer. The supercapacitor was fabricated by stacking two textile electrodes separated by a layer of gel electrolyte. The gel electrolyte was prepared by dissolving 1 g of PVA in 9 mL of deionized water at 95 °C for 1 h, followed by addition of 1 g of H<sub>3</sub>PO4 solution (85 wt%) when the solution was cooled down to room temperature.

To fabricate the integrated energy textile, a Ti wire textile (diameter: 130  $\mu$ m, size: 80 meshes) was anodized by a an ethylene glycol solution contained NH<sub>4</sub>F (0.3 wt%) and deionized water (8 wt%) at a voltage of 60 V for 6 h.<sup>[29]</sup> The anodized method was performed in a two-electrode electrochemical container with the Ti wire textile and Pt sheet as anode and cathode, respectively. The anodized Ti wire textile was washed with deionized water thoroughly, and then annealed at 500 °C for 1 h. The annealed Ti wire textile was immersed into an aqueous solution of TiCl<sub>4</sub> at 70 °C for 0.5 h, followed by annealing at 450 °C for 0.5 h, and then immersed into a solvent mixture of tertbutanol and dry acetonitrile (1:1, v/v) for 16 h. The PC part was first fabricated by stacking the modified Ti wire textile with a CNT fiber textile, followed by sealing and injecting the electrolyte. Finally, the PC part and supercapacitor textile were assembled into the integrated energy textile.

The structures were characterized by SEM (Hitachi FE-SEM S-4800 operated at 1 kV) and Raman spectroscopy (Renishaw in Via Reflex with an excitation wavelength of 633 nm and laser power of 20 mW). Cyclic voltammograms were obtained from an electrochemical analyzer system (CHI 660D). Galvanostatic charge–discharge characterizations were carried out by an Arbin multi-channel electrochemical testing system (Arbin, MSTAT-5 V/10 mA/16 Ch). The average specific capacitance of the electrode derived from the galvanostatic discharge curve was



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calculated according to the equation of  $C = 2i_0 / [m(\Delta V / \Delta t)]$ , where  $i_0$ , m, and  $\Delta V / \Delta t$  correspond to the discharge current, the mass of active material in the electrode and average slope of the discharge curve, respectively.

## Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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