

RAPID COMMUNICATION

Weavable high-capacity electrodes



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Abstract

Nanomaterials have shown enormous potentials in electronics and energy technology innovations. However, processing of nanoscale materials is challenging and often has extremely limited scalability. We designed and synthesized a Si-carbon nanotube (CNT) yarn which is mechanically as strong and flexible as widely used yarns in the textile industry but simultaneously possesses high electrical conductivity. This composite yarn is readily weavable and the process is highly scalable. As an illustration of potential applications, coin-cell lithium ion batteries were fabricated using the yarns as the anode. Because of the high electrical conductivity, the yarn electrode performs without the need of a copper current collector. A Li-storage capacity of 2200 mA h/g was demonstrated, which is five times higher than graphite electrodes and higher compared with previously demonstrated energy yarns based on pseudo capacitor or ultracapacitor materials.

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Introduction

Nanomaterials such as carbon nanotubes (CNTs) and semiconductor nanocrystals have shown significant promise in

electronics and energy technology innovations due to remarkable new properties arising at the nanoscale [1]. However, nanomaterials processing is challenging and often has extremely limited scalability, which drives the urgent need for scalable nanomanufacturing. Textile technology, on the other hand, has been indispensable since the dawn of civilization and has become an exceedingly sophisticated, large scale, and efficient industry [2]. This technology is typically based on natural or synthetic materials, including cotton, cocoons, wood fiber, polyester and Kevlar, none of

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which can conduct electricity, the key function essential for electronics and energy technology. One key process for textile industry is weaving or twisting from elemental materials to yarns, which serve as the building blocks for the entire textile technologies. Techniques that bring together the merits of these two technologies may pave new routes to energy and electronics technologies such as intelligent clothing, flexible electronics, and wearable batteries [3,4]. Exciting advances at this emerging frontier have been noted [5-7]. Baughman et al., for example, demonstrated a technique to incorporate functional guests into yarns for functional composites, such as TiO_2 -CNT and Si_3N_4 -CNT yarns [5]. Very recently, Lee et al. [6] and Peng et al. [7] reported the fabrication of supercapacitor yarns by electrodeposition, but the specific capacity and voltage are very low partly due to the choice of MnO_x as the active materials.

A particularly intriguing goal is to develop weavable CNT yarn electrodes that incorporate high energy density materials such as silicon for lithium ion batteries (LIBs). LIBs have increasingly become the power sources for consumer electronics and power tools such as cell phones, laptops and electrical vehicles. Portable electronic devices, such as rollup displays and wearable batteries, are technically demanding due to the need for high flexibility and portability, in addition to high energy density [8-10]. On the other side, structural batteries that can be integrated into the body of various system including vehicles, air-planes and tanks are emerging due to their excellent mechanical flexibility. Such integration will largely decrease the dead weight and space toward a high-energy density storage in the system level without the need of a dedicated area for batteries.

Herein, we report for the first time a highly scalable textile-like process to produce mechanically robust, electrically conductive Si-CNT composite yarns for use as high capacity weavable electrodes, as demonstrated in lithium ion batteries as an example. This strategy combines Si, which has the highest theoretical capacity for Li ions [11,12], and CNTs, which provide complementary mechanical durability and electrical conductivity [13], in a scalable process. Although heterostructured Si-CNT electrodes have been previously demonstrated [14-16], they are typically in the form of films with relatively low mechanical durability. Energy yarns with Si anodes have not been demonstrated previously due to the challenge of synthesizing Si-CNT hybrids which are mechanically robust and flexible for spinning and weaving. We overcome the challenge by designing a new process, in which Si was incorporated into spun CNT yarns. The proposed composite yarn electrodes show a discharge capacity as high as 2200 mA h/g based on Si mass, for the first cycle with a cutoff of 0.1-1.0 V, which is 5 times higher than that of graphite anodes [17]. Because the composite electrodes are mechanically strong and electrically conductive, they could be used without additional binders and metal current collectors. These merits benefit the improvement of battery energy density. Moreover, the mechanical durability enables these yarn electrodes to be easily woven and manipulated as an energy textile. This high level of processability may enable new battery fabrication technologies or high performance, portable electronic devices.

Material and methods

Vertically aligned CNT arrays were grown by chemical vapor deposition (CVD) at 815 °C using iron catalyst and acetylene gas precursor as described previously [18]. CNTs with an average diameter of ~50 nm and length of ~750 μm were drawn directly from the CNT arrays and subsequently spun into a yarn with a home-built spindle motor applying a dry spinning process [19,20]. Amorphous Silicon (α -Si) was grown on the CNT yarns and thin films at 460 °C in a low pressure chemical vapor deposition system (Atomate Nanowire Growth System) using 2 Torr of silane as the silicon source and 1 Torr of argon as the protective gas. The thickness of Si-coating was controlled by adjusting the deposition time. The mass of deposited α -Si was determined by measuring the mass change of the CNT yarn after Si-deposition using a microbalance (Citizen Scale Inc., 1 μg resolution).

Raman scattering spectra were collected on a Horiba Yvon LabRam ARAMIS Raman Spectroscopy with a helium neon laser excitation source (632.8 nm). SEM and TEM images were collected with a SU-70 SEM (Hitachi) and a JEOL FEG Transmission Electron Microscope, respectively. Mechanical tensile strength measurements were performed on a Dynamic Mechanical Analyzer (DMA-Q800) at a strain rate of 0.5% per minute with a gauge length of 2 cm. Each yarn was glued with super glue gel (LOCTITE) across a two-centimeter size square hole in the center of a 1.5 cm wide paper frame. After mounted in the tensile test apparatus, opposite sides of the paper frame were cut to free the yarn. The cross-sectional areas of the yarns were measured using SEM to convert the applied force to stress. The Young's Modulus was obtained by calculating the slope of the first linear portion of a stress-strain curve. Electrical conductivities were measured using a four-probe method with platinum metal contacts.

Coin-cell batteries were fabricated to test the battery performance using free-standing CNT or CNT@ α -Si yarns as the working electrode and Li metal foil as the counter electrode. Celgard[®]3501 (Celgard, LLC Corp., USA) was used as separator and 1 M LiPF_6 in a mixture of ethylene carbonate/dimethyl carbonate (EC/DMC, 1:1 by volume) was used as electrolyte. The CNT@ α -Si yarn electrodes were cycled with a charge rate of C/5 and a voltage range of 0.1-1.0 V on a Biologic VMP3 electrochemical workstation. EIS spectra were collected with the same workstation.

Results and discussion

CNTs, each approximately 750 μm in length and 50 nm in diameter, were drawn directly from vertically aligned CNT arrays grown by chemical vapor deposition and then spun into a yarn (Fig. 1). The tube-to-tube van der Waals interactions and well-defined vertical alignment of CNTs in the arrays enables those nanotubes to be drawable and spinnable in a highly scalable process. The spun CNT yarns maintain a porous structure which allows the growth of Si. Amorphous Si (α -Si) was grown on the surface of CNTs by low pressure chemical vapor deposition, at a Si:CNT mass ratio of 0.63 determined from the mass change of the CNT yarn after Si-deposition. The mass loading of the active material

(Si) per area for this yarn was calculated to be ~ 2.5 mg/cm², which can be controlled by adjusting the growth time for Si. Fig. 2b and c are scanning electron microscopy (SEM) images of a spun CNT yarn before and after Si-deposition, respectively, demonstrating a typical yarn architecture. As shown in Fig. 2d, the composite yarn can be repeatedly manipulated and wrapped around a copper rod (radius = 0.42 mm) without breaking. This high level of flexibility and durability enabled the yarns to be woven into a textile fabric (Fig. 2e).

As shown in Fig. 2f and Fig. S2b and c, CNTs within a yarn are arranged almost in parallel, which can provide a well-directed conductive matrix for efficient electron and ion transports. The porosity of the yarns can be controlled by adjusting the twisting force during the spinning process. Fig. 2f and g and Fig. S2e and f shows that the yarns can retain highly porous structures even after Si deposition, providing accessibility for the electrolyte to reach the

interior of the composite yarn, and sufficient space to accommodate the volume expansion of Si upon electrochemical charging. High resolution transmission electron microscopy (TEM) images of individual CNT@ α -Si heterostructures clearly show the CNT-core and α -Si-coating as well as the CNT/Si interface (Fig. 2h and Fig. S3c). The thickness of Si-coating within CNT@ α -Si yarns used in all the characterizations was ca. 25 nm, and could be controlled by adjusting the growth time.

The composition of as-prepared yarns was further confirmed with Raman spectroscopy (Fig. 3). The typical Raman bands of CNTs including disorder mode (D-band), tangential mode (G-band) and the second-harmonic of the D mode (G'-band) were observed at 1335, 1585 and 2665 cm⁻¹, respectively [21]. After Si-deposition, the characteristic peaks of α -Si were present around 150 and 480 cm⁻¹, which are associated with the transverse-acoustic and transverse-optical phonon modes, respectively [22]. There was

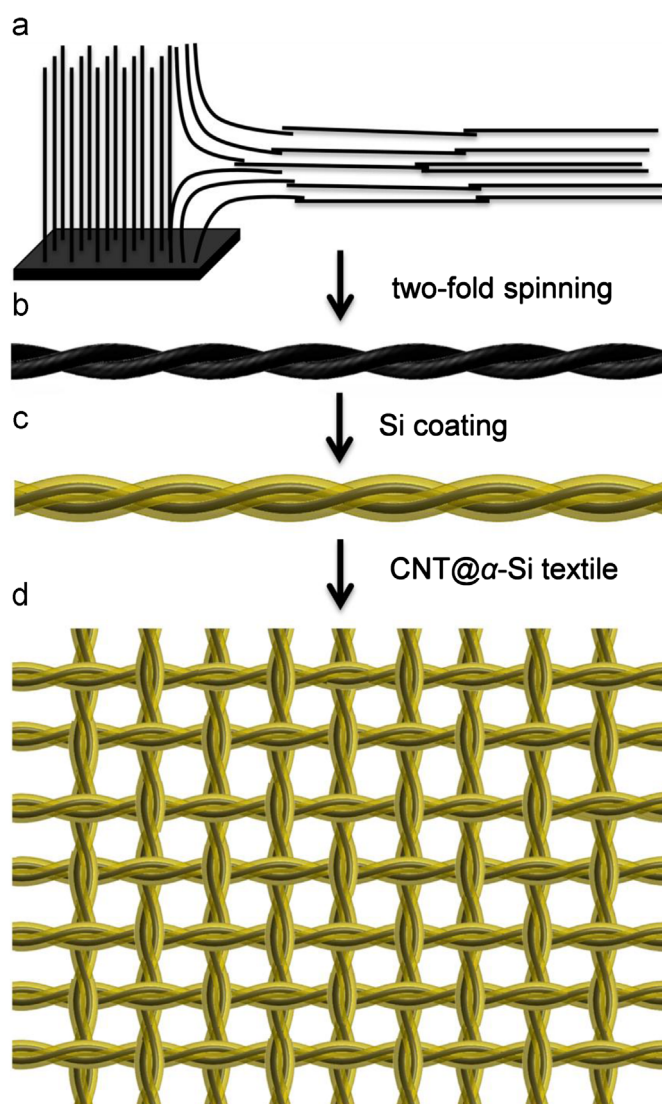


Fig. 1 Schematic illustrations of the fabrication process of weavable CNT@ α -Si composite yarns. (a) Drawing a CNT thin film from a vertically aligned CNT array. (b) CNT yarn spun by a two-fold spinning process. (c) Silicon-coated CNT yarn. (d) Textile woven from the composite yarns.

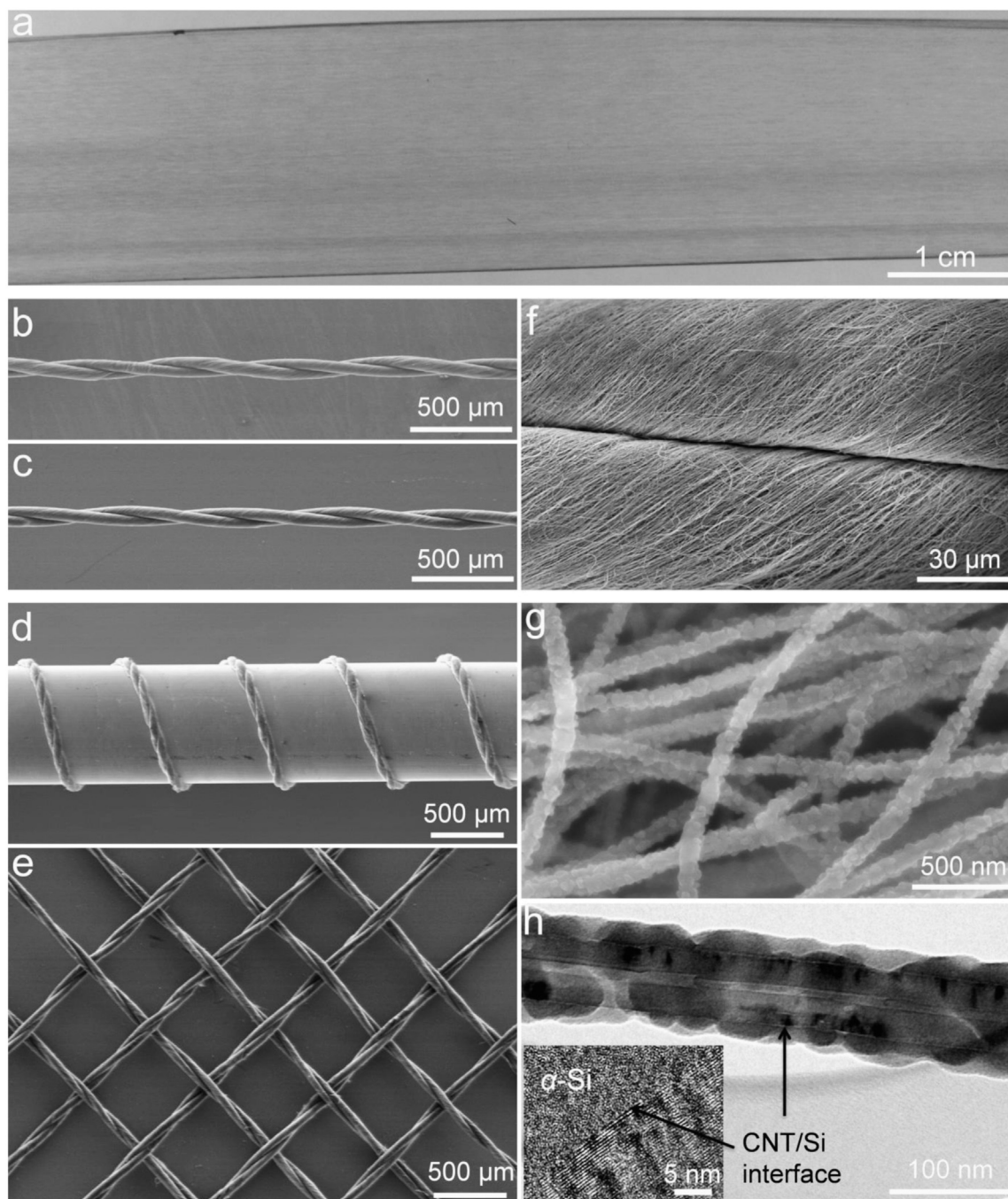


Fig. 2 Electron microscopy characterization of CNT@ α -Si composite yarns. (a) Optical image of a CNT thin film drawn from vertically aligned a CNT arrays. (b) SEM image of CNT yarn. ((c)-(f)) SEM images of CNT@ α -Si composite yarns. (d) The composite yarn is wrapped round a copper rod, showing a high level of flexibility and durability. (e) SEM image of a textile woven from CNT@ α -Si yarns. ((f) and (g)) The porous structure of the CNT@ α -Si composite yarn. (h) TEM images resolving the CNT-core, Si-coating and CNT/Si interface. Inset is a high resolution TEM image that clearly shows the CNT/Si interface.

almost no change of the D-peak before and after Si-deposition. This indicates no increase of defects during Si-deposition process, warranting a high electrical conductivity of the yarns.

We found that the reverse of this spinning and growth process failed to produce robust yarns. The Si-coated CNT thin film could be twisted by a very limited degree, and delamination of α -Si from the surface of CNTs was observed

in such spun yarns (Fig. S4). The obtained CNT@ α -Si yarns exhibit low tensile strengths and tensile fracture occurred even under gentle stretching, which limits their applications especially as weavable electrodes or in other flexible integrated electrical devices. The low tensile strength of such yarns is due to dramatically decreased tube-to-tube van der Waals interactions, as the surfaces of CNTs are increasingly covered by Si.

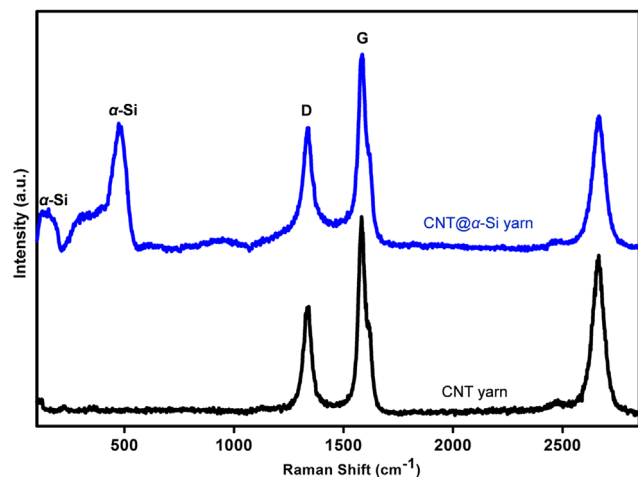


Fig. 3 Raman spectra of CNT@ α -Si composite yarn in comparison with the CNT yarn. The excitation line was 632.8 nm.

Electrical conductivities of CNT and CNT@ α -Si yarns were measured using a four-probe method, giving a value as high as 442 and 419 S/cm, respectively. A slight decline of conductivity after Si-deposition can be attributed to the resistance of Si-coating at the two points of contact with probes. The electrical resistances per unit length of CNT and CNT@ α -Si yarns were measured to be 82 and 86 Ω /cm, respectively. Notably, the electrical conductivity of the Si-coated CNT yarns is 7 orders of magnitude higher than that of Si (1.56×10^{-5} S/cm), ensuring efficient electrical transport within the yarns. This high electrical conductivity makes it possible to abandon conventional metal current collectors in the current battery configuration, a step that may ultimately contribute to improvement of specific capacity and power performance of lithium ion batteries.

For weavable electronics and structural batteries, the mechanical strength is a very important essential. To further investigate the mechanical robustness, we measured the tensile strength of both CNT and CNT@ α -Si yarns. The CNT yarn gives a tensile strength as high as 366 MPa (Fig. 4), which falls within the typical range of 250–460 MPa for two-fold spun CNT yarns [13]. Even with Si-coating, the yarn exhibits a well-preserved mechanical robustness with a very high tensile strength of 342 MPa, much higher than those of conventional textile yarns like wood fiber (11 MPa) [23], polyester (40–90 MPa) [24], Nylon (12.4–94 MPa) [24] and comparable to that of cotton fibers (400 MPa) [24]. This high strength enabled the CNT@ α -Si composite yarn to be easily woven in a highly scalable process, similar as processing conventional fibers in the textile industry. The Young's Modulus of CNT and CNT@ α -Si yarns were calculated to be 33 and 43 GPa, respectively. The higher value of CNT@ α -Si yarns can be attributed to the Si-coating which may serve to restrict slippage among CNTs, like the knots to tie shoelaces.

Electrochemical measurements of CNT and CNT@ α -Si yarn electrodes were performed with Li metal as the counter electrode in a potential range of 0.1–1.0 V vs Li/Li⁺. Fig. 5a shows the voltage profile of CNT and CNT@ α -Si yarn electrodes during the first cycle with a charging and discharging rate of C/5. For pristine CNT yarns, the first-

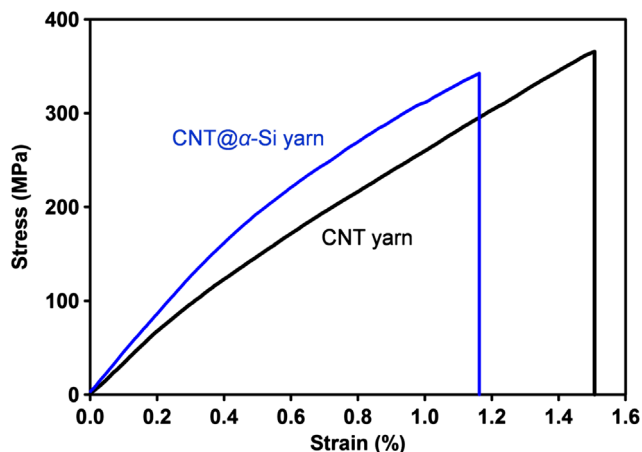


Fig. 4 CNT@ α -Si composite yarns show high mechanical strength comparable with CNT yarns.

cycle charge and discharge capacities are only 267 and 168 mA h/g, respectively, with a relatively low coulombic efficiency of 63%, which is probably due to the large solid electrolyte interphase (SEI) formation and is consistent with literature values for CNT anodes [25]. For CNT@ α -Si yarn, high first-cycle charge and discharge specific capacities of 2440 and 2200 mA h/g (based on the mass of Si) were achieved, respectively, with a coulombic efficiency of 90%. This more than 8-fold improvement in Li⁺ storage capacity can be attributed to the high capacity of Si, the highly conductive matrix and the efficient electrode accessibility for the electrolyte. Notably, the proposed composite yarns show a high capacity comparable with other Si-based electrodes [14,15,26] but afford significant improvement over the mechanical robustness and electrical conductivity that are required for weavable electrodes.

Fig. 5b shows the cycling performance of electrodes with a rate of C/5. Capacity retention is 94% after 20 cycles, and 86% after 30 cycles. The capacity loss can be attributed to inherently weak adhesion between Si and sp² carbon layers [27,28]. The vast mechanical stress arising from the huge volume change between charge and discharge cycles easily leads to fracture and/or interfacial delamination of Si which could be the major cause of battery failure. We note, however, that the interfacial adhesion can be improved by chemically tailored carbon-silicon interface [29].

To understand the cycling performance of our energy yarns as Li-ion battery anodes, we also performed electrochemical impedance spectroscopy (EIS) in the frequency range from 1 MHz to 0.1 Hz with a sinus amplitude of 5 mV. EIS was performed at the same cell before and after cycling (Fig. 5c). The impedance spectrum mainly includes the electrolyte/electrode resistance R_e , charge transfer resistance between electrode/electrolyte interface R_{ct} , and diffusion resistance [30]. R_e is the intercept between the spectra and real resistance at high frequency, which present the major resistance of the cell. R_e was 14 Ω initially and increased to 35 Ω after cycling, which is due to the formation of SEI. The SEI formation and the resulting resistance increase could contribute partially to the capacity decay of our electrodes.

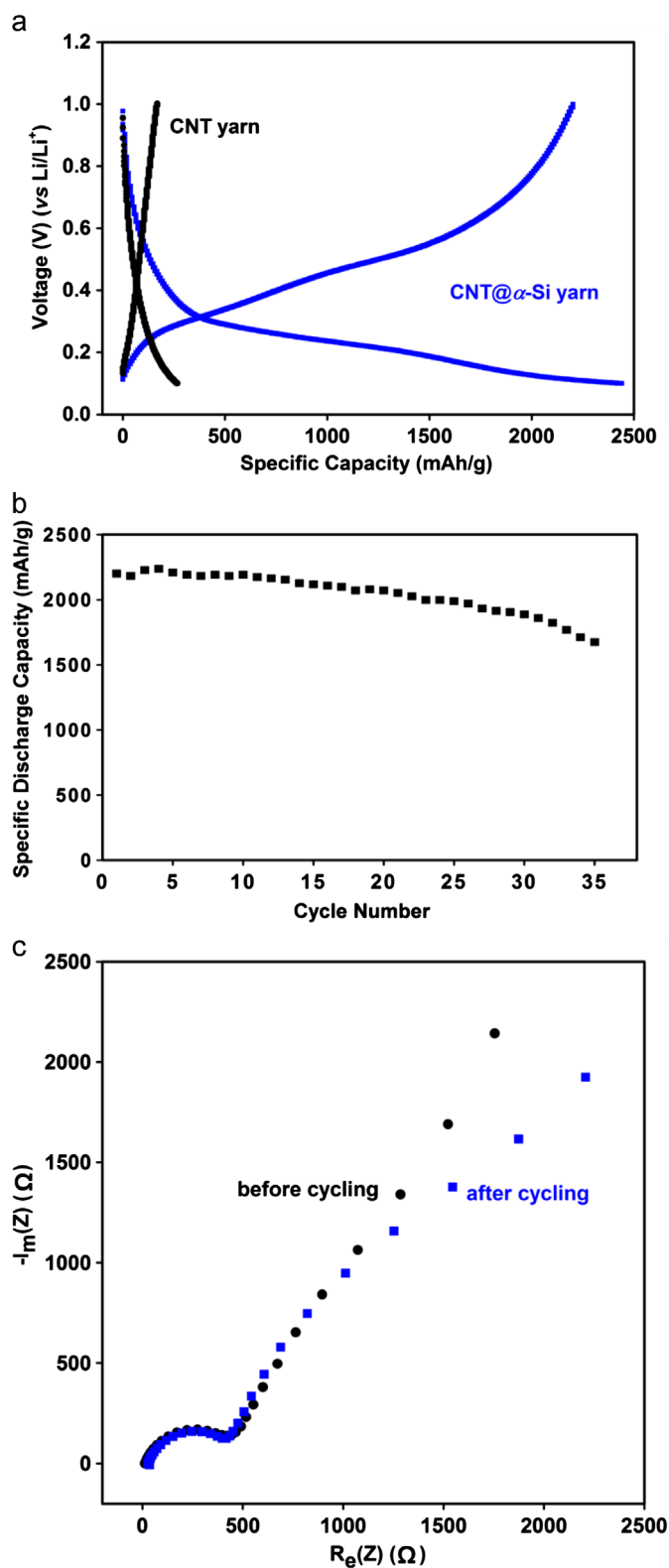


Fig. 5 Electrochemical performances of CNT@ α -Si composite yarns. (a) Voltage profiles of CNT and the composite yarns with Li metal as the counter electrode at 1st cycle with an operation voltage range of 0.1-1.0 V and a charge rate of C/5. (b) Cycling performance of CNT@ α -Si composite yarn (0.1-1.0 V, C/5). (c) EIS data collected from the cell with the composite yarn anode before cycling and after cycling test (0.1-1.0 V, C/5).

Conclusion

We have demonstrated the design and scalable fabrication of a CNT@ α -Si composite yarn for application as lithium battery anodes. This yarn electrode affords five times higher lithium ion storage capacity than that of graphite electrodes, owing to the facial integration of high capacity Si with mechanically robust, highly conductive CNT yarns. This work highlights some of the key advantages of borrowing textile technology for nanomaterials processing, as illustrated through the fabrication of a CNT@ α -Si composite yarn to address the challenge facing the scalable synthesis of high performance silicon-based lithium battery anodes. Li-ion batteries yarns with integrated current collectors, high mechanical strength and high capacitor can be used for wearable electronics and structural batteries for electrical vehicles.

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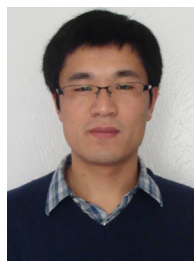
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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at <http://dx.doi.org/10.1016/j.nanoen.2013.03.020>.

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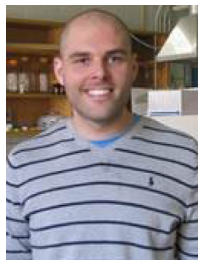


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Edward B. Baker III is currently a Ph.D. student advised by Prof. YuHuang Wang at the University of Maryland, College Park. He received his B.S. in mathematical-physics from Brown University in 2008, and his M.S. in physics from the Weizmann Institute of Science in 2011. His current research interests include the synthesis of carbon nanotubes and their subsequent processing into materials and devices, especially for the purposes of improving energy production and distribution.



Morihiro Okada is a Visiting Professor in the Department of Chemistry and Biochemistry at the University of Maryland. He received B.S. and Dr. Eng. degrees from the University of Tokyo, Japan, in 1985 and 1997, respectively. He has more than two decades of industrial experience in Nippon Steel Corporation. There, he developed a PVD coating system of hard carbon films on steel and studied the scale-growth mechanism in the steel production line. He discovered magnetite whiskers on steel and invented an electron emitter that uses a single magnetite whisker and applied them to spin polarized electron emitters. Since 2008, he has been involved in chloride mediated CVD growth of CNT arrays in Shizuoka University, Japan, and in 2011 he moved to the University of Maryland to collaborate with Prof. YuHuang Wang's group. He has 41 patent disclosures and one US patent.



Jiayu Wan is a Ph.D. candidate at the University of Maryland. His research with Prof. Liangbing Hu is on energy storage and transparent electrode devices. He obtained his BS degree in Huazhong University of Science and Technology in China in 2011.



Adrian Ghemes is currently a postdoctoral researcher at the Research Institute of Electronics, Shizuoka University. He received his B.S. and M.S. from Alexandru Ioan Cuza University, Romania in 2002 and 2004, respectively. In 2008 he received a Ph.D. degree from Shizuoka University, Japan under the supervision of Prof. Hidenori Mimura. He is interested in synthesis and applications of ultralong and spinnable carbon nanotubes.

His research is particularly focused on fabrication and characterization of light, strong and electrically conductive pure CNT yarns.



Yoku Inoue is an Associate Professor of Electrical Engineering at Shizuoka University. He started his research in single electron tunneling effect through Si nanocrystals in 1993 at Kobe University, Japan. He then conducted researches on CVD growth of III-nitride nanomaterials. His research interests are in the area of inorganic nanomaterials. He has been recently working on growth of carbon nanotube (CNT) and its application researches including dry spinning technologies of CNT forest, lightweight and strong CNT yarns, and highly aligned CNT/polymer composite materials.



Liangbing Hu received his B.S. in applied physics from the University of Science and Technology of China (USTC) in 2002. He did his Ph.D. in experimental physics at UCLA, focusing on carbon nanotube based nanoelectronics. In 2006, he joined Unidym Inc (www.unidym.com) as a co-founding scientist. At Unidym, Liangbing's role was the development of roll-to-roll printed carbon nanotube transparent electrodes and device integrations into touch screens, LCDs, flexible OLEDs and solar cells. As a postdoc at Stanford from 2009 to 2011, he worked on various energy devices based on nanomaterials and nanostructures including Li-ion batteries, ultracapacitors and microbial fuel cells. Currently, he is an assistant professor at University of Maryland College Park. His research interests include energy and flexible electronics based on nanomaterials.



YuHuang Wang is an Assistant Professor of Chemistry at the University of Maryland. His research group focuses on materials and physical chemistry of carbon nanotubes, and their biomedical and energy applications. A central theme of his research program is exploiting defect chemistry of sp² carbon lattices for understanding and controlling the coupling of electrons, excitons, phonons, and spin with defects in reduced dimensions. Wang received a B.S. degree in chemistry from Xiamen University, China. He did his Ph.D. studies with Richard E. Smalley at Rice University, where he demonstrated cloning of single-walled carbon nanotubes and contributed to the development of the first macroscopic, neat, single-walled carbon nanotube fiber, an electrically conductive rival of Kevlar. He then joined the laboratory of Chad A. Mirkin at Northwestern University as a postdoctoral fellow, where he invented multiple techniques for high throughput high resolution molecular printing and developed a fundamental understanding of directed assembly and manipulation of nanostructures. He is the author of more than 60 manuscripts and 16 patents and applications in the areas of carbon nanotechnology, nanofabrication and directed assembly.