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Knitted carbon-nanotube-sheath/spandex-core elastomeric yarns for artificial muscles and strain sensing

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Knitted carbon-nanotube-sheath/spandex-core elastomeric yarns for artificial muscles and strain sensing

Abstract

Highly stretchable, actuatable, electrically conductive knitted textiles based on Spandex (SPX)/CNT (carbon nanotube) composite yarns were prepared by an integrated knitting procedure. SPX filaments were continuously wrapped with CNT aerogel sheets and supplied directly to an interlocking circular knitting machine to form three-dimensional electrically conductive and stretchable textiles. By adjusting the SPX/CNT feed ratio, the fabric electrical conductivities could be tailored in the range of 870 to 7092 S/m. The electrical conductivity depended on tensile strain, with a linear and largely hysteresis-free resistance change occurring on loading and unloading between 0% and 80% strain. Electrothermal heating of the stretched fabric caused large tensile contractions of up to 33% and generated a gravimetric mechanical work capacity during contraction of up to 0.64 kJ/kg and a maximum specific power output of 1.28 kW/kg, which far exceeds that of mammalian skeletal muscle. The knitted textile provides the combination of strain sensing and the ability to control dimensions required for smart clothing that simultaneously monitors the wearer's movements and adjusts the garment fit or exerts forces or pressures on the wearer, according to needs. The developed processing method is scalable for the fabrication of industrial quantities of strain sensing and actuating smart textiles.

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Knitted Carbon-Nanotube-Sheath/Spandex-Core Elastomeric Yarns for Artificial Muscles and Strain Sensing

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Keywords:

Carbon Nanotube, Spandex, Smart Textiles, Actuators, Sensors

Abstract

Highly stretchable, actuatable, electrically conductive knitted textiles based on Spandex (SPX)/CNT (carbon nanotube) composite yarns were prepared by an integrated knitting procedure. SPX filaments were continuously wrapped with CNT aerogel sheets and supplied directly to an interlocking circular knitting machine to form the three-dimensional electrically conductive and stretchable textiles. By adjusting the SPX/CNT feed ratio, the fabric electrical conductivities could be tailored in the range of 870 to 7092 S/m. The electrical conductivity depended on tensile strain, with a linear and largely hysteresis-free resistance change occurring on loading and unloading between 0 and 80% strain. Electrothermal heating of the stretched fabric caused large tensile contractions of up to 33%, and generated a gravimetric mechanical work capacity during contraction of up to 0.64 kJ/kg and a maximum specific power output of 1.28 kW/kg, which far exceeds that of mammalian skeletal muscle. The knitted textile provides the combination of strain sensing and the ability to control dimensions required for smart clothing that simultaneously monitors the wearer's movements and adjusts the garment fit or exerts forces or pressures on the wearer, according to needs. The developed processing method

is scalable for the fabrication of industrial quantities of strain sensing and actuating smart textiles.

Introduction

A wide range of advanced functionalities are being explored for smart textiles, including energy harvesting/storage, force/pressure generation, changes in porosity or color, and sensors for movement, temperature or chemicals. ^[1-8] Since smart textiles are ultimately aimed at the consumer market, material choice and fabrication methods must be commensurate with production on an industrial scale. In this direction, we have developed an up-scalable knitting technique capable of continuously producing electrically conducting 3D fabrics made from hybrid spandex (SPX) – multiwall carbon nanotube (CNT) yarns. These highly stretchable fabrics exhibit excellent performance as sensors and artificial muscles. The CNT content in the yarns is small (below 10% by weight) and commodity SPX fibers are used, so that material costs for the smart textile remain reasonable.

Textile structures are attractive for mechanically actuating devices since they offer a potential practical solution to the contradictory scaling laws for force generation and power output. The mechanical power output from actuator materials per mass or volume tends to increase at smaller dimensions, due to faster mass and heat transport or higher surface area to volume ratios. In contrast, force output varies with the square of diameter even when the stress generated is scale invariant. An often posed solution is to formulate parallel assemblies of many small-diameter actuating fibers or wires, but laborious fabrication has limited this approach to date. Textile processing is an attractive alternative manufacturing process but is only suitable for those actuating materials that can be made into robust, small-diameter fibers in the needed long lengths. One recent example used shape memory alloy wires to form an active knit, where the force output was increased by additive contributions from individual wires. ^[9] Use of the looped fiber architecture of the garter stitch amplified actuation strain and demonstrated the

ability to modulate actuation force and stroke by exploiting the enormous variety of textile architectures that are readily achievable using knitting, weaving and braiding.^[9]

The strategy described here to develop low-cost, mass-producible, actuating textiles demanded a technique to render commodity stretch fabrics electrically conductive without compromising production rates and without major modification to production methods. The actuating textile fabrics were made from commercially available elastomeric SPX copolymer fibers (Spandex) and utilised the thermo-elasticity of the rubber copolymer segments to generate contractile displacements and associated tensile forces. A seamless feed-in process for coating the SPX fibers with CNTs was developed to allow continuous fabrication of the conducting stretch fabrics. The coating method operates at room temperature, requires no solvents and does not compromise textile production speeds. Altering the fabrication parameters provided a convenient way to modify the fabric's conductivity and mechanical properties. Here we report the effect of these properties on the mechanical actuation and strain sensing characteristics of these smart textiles.

A schematic diagram of the developed fabrication process is shown in **Figure 1a**. CNT aerogel sheet was drawn from a CNT forest^[10, 11] and wrapped around the continuous supply of SPX filament(s) fed to an interlocking circular knitting machine (see Movie S1 in the Supporting Information). Knitted structures were prepared using either a single SPX fiber or SPX yarns consisting of 4, 8 or 12 filaments. The tension and rotation of the yarn during the knitting process securely consolidated the CNT coating on the outer surface of the SPX fiber or yarn (Figure 1). Prepared knitted structures (labeled CNT/SPX_{*n*}, where *n*=1, 4, 8, 12 is the number of SPX filaments in the feed yarn), having the structure schematically illustrated in Figure S1, are pictured in Figure 1b-c. The diameter of the circular knitted fabric and the fiber knit angle depended on the number of filaments used in the supply yarn (see Figure S1 and Table S1). The ratio of CNT to SPX in the knitted fabric decreased from 9.1% (w/w) to 2.1% as *n* increased from 1 to 12 (see Figure S2). All knitted textiles were highly stretchable (Figure

S3a) with breaking strains of between 600% to 900% and tensile strengths in the range 75 MPa to 86 MPa. All stress values were calculated based on the non-loaded cross-sectional area of the circular knit (Table S1). The fabrics were also electrically conductive, with approximately the same resistances per fabric length of 3.0 k Ω /m when measured using the two-probe technique and neglecting the contribution from contact resistances. The near equivalent linear resistances indicate that all fabrics had similar conductive path lengths and CNT coating quality and thickness. Apparent bulk conductivities were calculated on the basis of fabric cross-sectional areas and increased from 870 to 7092 S/m with increasing CNT mass loading (Table S1).

The CNT/SPX_n fabrics performed well as thermally driven artificial muscles using direct current (DC) for electro-thermal stimulation (see movie S2). To assess performance as an actuator, the knitted samples were vertically suspended with an attached load, electrically connected at both ends to a power supply and heated electro-thermally using a constant applied voltage to 70°C in approximately 2.5 seconds. The voltage applied was tuned to achieve approximately equivalent heating rates for all samples, as determined by a small thermocouple positioned in close contact with one end of the actuating fabric. Higher voltage inputs were needed for samples with the higher SPX contents because of their larger thermal mass of actuating material. A laser distance transducer measured changes in sample length by continuously monitoring the vertical displacement of the attached load.

The application of the DC voltage to the pre-stretched sample was accompanied by material contraction that was fully reversed by passive cooling when the current supply was terminated. When pre-strained to 100%, the observed contractile strains ($\Delta L/L_0$) were found to be as large as 33% of the non-loaded sample length for knitted CNT/SPX₈ samples heated to 70 °C with 20 V/cm applied voltage at an on-off switching rate of 0.2 Hz (**Figure 2a**). Electro-thermal heating over the same temperature range generated maximum contractile strains of 16%, 25% and 24% for CNT/SPX with $n = 1, 4$ and 12 , respectively. Contractile strains increased

with increasing temperature achieved by applying higher input voltages (Figure 2d). The generated actuation strain was also strongly dependent on the applied stress with the heating-induced contraction strain first increasing and then decreasing with increasing stress for all samples (Figure 2c). Since the work capacity increases linearly with the absolute change in muscle length, unless otherwise noted the contractive strains are normalized with respect to the non-loaded textile length. However, the same general behavior as in Figure 2c was noted when actuation strains were normalized to the loaded sample length (Figure S3). These maximum actuation strokes occurred at pre-strains of 150% for all samples when heated to the same maximum temperature. Strain amplitudes for all samples decreased with increasing voltage switching rates when the same supply voltage was used (Figures S4 and S5). Highly repeatable and stable actuation by contraction and expansion was observed for a CNT/SPX₄ sample for over 10,000 heat and cool cycles (Figure 2b). The maximum work densities were as high as 640 J/kg and the maximum power-weight ratio was 1278 W/kg, which were both achieved with the CNT/SPX₁ textile pre-stretched to 100% and activated at 20 V/cm at 2 Hz. The gravimetric work and power outputs are 16 times and 4 times higher, respectively, than mammalian skeletal muscle^[16], but lower than recently reported for electrothermally actuated coiled polymers fibers that operated at higher actuation temperatures^[12].

The observation of thermally-driven contraction in stretched SPX-based elastomers is not surprising, and has been well known for other elastomers for over a century. The stretched rubber has lower entropy than does the non-stretched rubber, so heating the stretched rubber increases the modulus of the stretched rubber, thereby causing the isotonicity loaded rubber fibers to contract. Stress-strain curves obtained for all samples at room temperature and when furnace heated to 110 °C demonstrate an increase in stiffness as the temperature is increased (Figure S6). The isotonic actuation strain (ϵ_a) for a linear elastic material is directly dependent on the applied stress (σ), free strain (ϵ_o , as occurs in the absence of an applied stress) and the change in modulus (E' and E are final and initial values, respectively) as given by:^[13]

$$\varepsilon_a = \varepsilon_o + \sigma \left(\frac{1}{E'} - \frac{1}{E} \right)$$

The linear thermal expansion coefficient of SPX rubber is of the order of 10^{-4} (m/m K) so that the thermal expansion of the unloaded rubber generates a positive free strain of $\sim 0.5\%$ when heated from room temperature to 70°C . The second term in the above expression is negative for rubbers and increases in magnitude as stress increases. Consequently, the net actuation strain can become negative and larger contractile strains are expected with increasing applied stress, as shown in Figure 2C for small stresses. The non-linear elasticity of rubbers means that more complicated behavior is likely than that described by the above relation and can even account for the declining actuation strain at higher stresses.

The strain sensing capabilities of the CNT/SPX knitted fabrics were assessed by repeated loading and unloading from 0% to 100% strain for a minimum of 1000 cycles. Fabric stretching was accompanied by an increase in electrical resistance R . The fractional change in resistance normalized to the unloaded fabric resistance was calculated as $R^* = (R - R_0)/R_0$. Typical R^* measurements for repeated loading and unloading for CNT/SPX₈ are shown in **Figure 3a** and similar data for other investigated fabrics are shown in Figure S7. The fractional change in resistance was highly repeatable, with the amplitude for each strain cycle changing by less than 2.3% over the 1000 strain/relaxation cycles. A small drift of the resistance at the maximum and minimum strains occurred during the first few dozen cycles after which the resistance response was very stable. The initial drift in resistance can be associated with the adjustments in electrical point contacts between knitted fibers.

A near linear resistance change was observed in all knitted fabrics for strains to $\sim 80\%$ and little hysteresis was observed during loading and unloading (Figure 3b). The absence of substantial hysteresis greatly improves the accuracy of the sensor and the linear response allows a single gauge factor to be obtained as $\alpha = dR^*/d\varepsilon$ for each material^[14]. The gauge factor

increases with increasing number of SPX fibers in the feed supply from 0.12, 0.16, 0.25 to 0.4, for $n = 1, 4, 8, 12$, respectively.

Simple geometrical effects suggest a gauge factor of 2-3 for homogeneous deformation of conductors over the strain range used here which is an order of magnitude larger than the measured gauge factors determined for the CNT/SPX_{*n*} fabrics. Giant gauge factors are achievable when strain disrupts the percolation networks in conductor/non-conductor composites. In contrast, very small changes in resistance have been recently reported for elastomers coated with conductors, whereby surface corrugations or buckled coatings allow large strains without disrupting or damaging the path length or integrity of the conducting layer(s). For example, coating a highly stretched elastomeric fiber with the same CNT aerogel sheet used here followed by stress relaxation produced hierarchically buckled carbon nanotube coated elastomer fibers that show near strain-invariant resistance to extensions of 1000%^[15]. Optical (Figure S2d) and SEM (Figure S8) micrographs of the presently prepared knitted fabrics show CNT coating roughness in the non-stretched fabrics and coating cracking after 100% stretch. Observed resistance changes reflect the combination of resistance increases due to cracking or deformation processes that are partially offset by enhanced inter-fiber contact as the knitted loops are tightened during stretching.

As a demonstration example, the developed textile sensor can be attached to a commercial wireless transmitter for remote acquisition of data from tensile and bending deformations. A knee sleeve prototype based on CNT/SPX₄ wearable strain sensor was developed to assist personal rehabilitation after an injury (see Figure S9 and movie S3).

In conclusion, stretchable and conductive textiles were prepared by combining CNT and SPX fibers in 3D knitted structures. The textiles exhibited excellent strain sensing and actuation performance within the same material. Because of large tensile actuation, low driving voltage, high repeatability, scalability, and stretchability the CNT/SPX knitted textiles could be used for adjustable smart clothing, robotics and medical devices.

Experimental Section

The 40-denier Spandex fibers used, which were 110 μm in diameter, were purchased from Spandex Co. Ltd. (China). The CNT/SPX fibers were knitted using a Harry Lucas circular knitting machine (Type R-1S, Gauge 28, Needles 8). The CNT sheet was obtained from spinnable CNT forest using a drawing procedure that has been previously described.^[10, 11, 16] The morphology of CNT/SPX knitted textile was characterized using a field emission scanning electron microscope (JEOL JSM-7500FA) after sputter coating with a thin gold layer (~ 10 nm, EDWARDS Auto 306). A Leica DM EP optical microscope was used for visual characterization. Thermo-gravimetric analysis (TGA) of the amount of CNT incorporated into the knitted textile was performed using a TA instruments model TGA/SDTA851e in the range from 40 to 1000 $^{\circ}\text{C}$ at the rate of 10 $^{\circ}\text{C}/\text{min}$ under argon flow. TGA shows a weight loss at ~ 300 –450 $^{\circ}\text{C}$ of $\sim 100\%$, 90.9%, 93.2%, 96.9% and $\sim 97.9\%$ for spandex and CNT/SPX $_n$ ($n = 1, 4, 8$ and 12) knitted textiles, respectively. Spandex decomposes at ~ 350 $^{\circ}\text{C}$. Spandex and CNT/SPX showed sharp reduction in mass at 300–450 $^{\circ}\text{C}$. It is, therefore, expected that the weight loss in the CNT/SPX samples at 300–450 $^{\circ}\text{C}$ is principally due to the decomposition of the Spandex polymer. As shown in Table S1, the weight percent of CNT in the textiles that is derived from the weight gain on wrapping Spandex n filament fibers is close to that derived from the TGA measurements on the textiles. Electrical and mechanical properties of the CNT/SPX textiles were evaluated by *in situ* monitoring the sample resistance with a digital multimeter (Agilent 34410A) as cyclic deformations were applied by a tensile testing instrument (Shimadzu EZ-L, 10 N load cell and 10 N clamps). To obtain the strain-to-break of the knitted textiles containing 1, 4, 8 and 12 SPX fibers with and without CNT, 20 mm gauge length textiles were stretched at the rate of 100 mm/min, while stress, strain and electrical resistance were measured. For cyclic testing, 20 mm-gauge-length samples were attached to holder of Shimadzu EZ mechanical tester machine and then subjected to 1000 cycles of stretching/relaxation to 100%

strain at the rate of 100 mm/min. Electrical resistance was measuring continuously during these measurements. Tensile actuation measurements were performed under constant load (isotonic conditions); the sample was clamped vertically to the displacement sensor (Omega LD701) in such way that DC voltage can be applied for Joule heating.

Supporting Information

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

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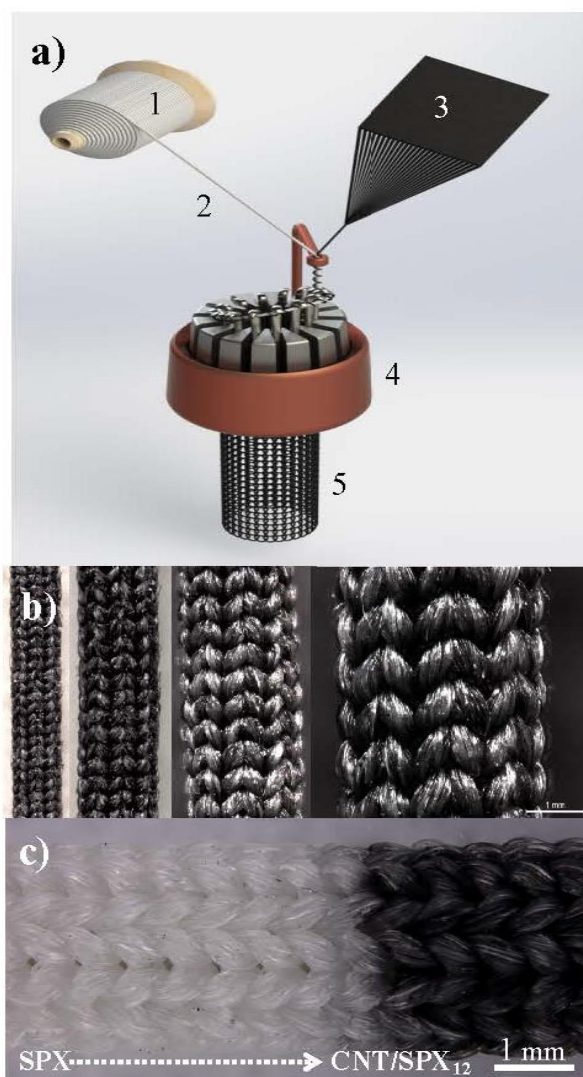


Figure 1 a) Schematic of the process for producing a knitted CNT/SPX textile. The illustrated items are: (1) a “Spool of SPX fibers”, (2) a “ n -fiber SPX yarn”, (3) a “CNT forest”, (4) a “Circular knitting machine”, and (5) a “Knitted CNT/SPX textile”. A CNT ribbon drawn for a CNT forest was wrapped around n SPX fibers and knitted in the knitting machine to produce the circular knitted textile shown in b) and c). Unlike for the case for twist-based CNT yarn spinning, the above schematically-illustrated wedge is a time average of the actual draw process, since a much narrower CNT ribbon migrates over the broader illustrated width in response to stresses applied during wrapping the SPX yarn with CNT. b) Photograph of knitted CNT/SPX structures, from left-to-right containing 1, 4, 8 and 12 SPX fibers, respectively. From left-to-right, the weight percent of CNT in the textile is increasing, as shown in Table S1. c) A knitted CNT/SPX₁₂ textile, showing the transition between CNT non-wrapped SPX₁₂ yarns and CNT wrapped SPX₁₂ yarns.

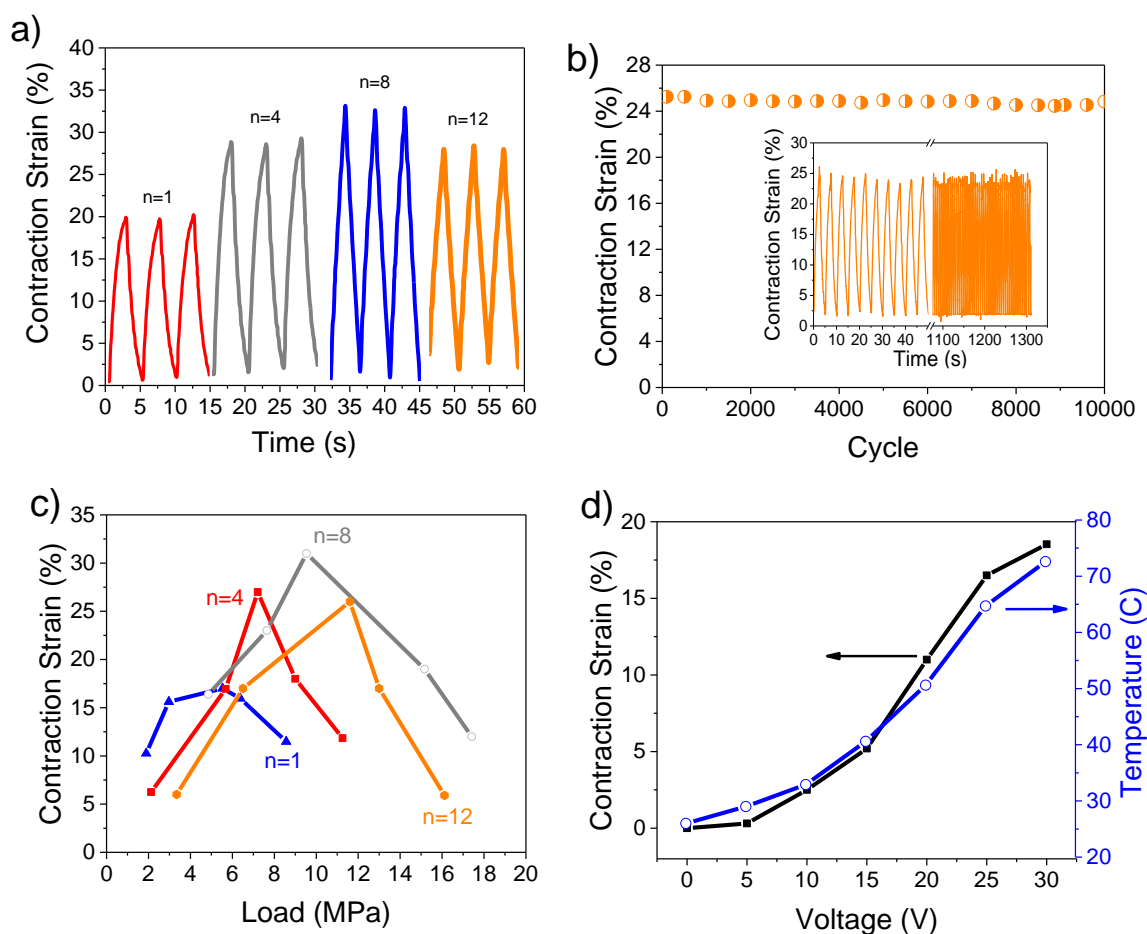


Figure 2 a) Effect of n for CNT/SPX _{n} textiles on isobaric actuator strain during electrothermal heating to 70°C at 0.2 Hz, where all samples were stretched by 100 % strain by an applied load before actuation (which is the non-actuated strain for all of the below isobaric measurements). b) Muscle contraction recorded over 10,000 on-off cycles for knitted CNT/SPX₄ at 0.2 Hz, 30 V/cm. c) Muscle contraction of CNT/SPX _{n} knitted textiles as a function of load at 0.1 Hz and 10 V/cm: (CNT/SPX₁), 20V/cm (CNT/SPX₄), 30 V/cm (CNT/SPX₈) and 60 V/cm (CNT/SPX₁₂). d) Effect of voltage on tensile actuation and sample temperature for CNT/SPX₄ muscles at 0.1 Hz. The contractile strains are normalized with respect to the non-loaded muscle length. Here and elsewhere, applied stresses are engineering stresses, which are normalized by the non-stretched fiber cross-sectional area.

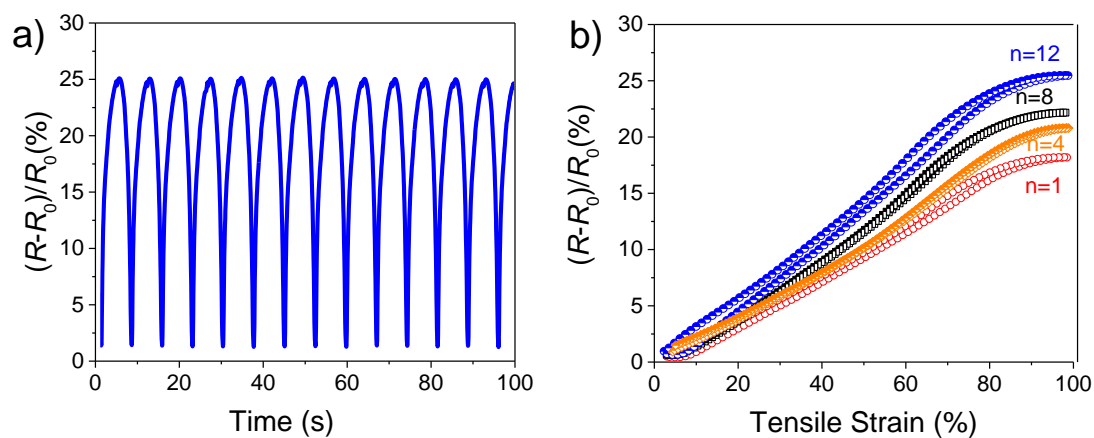


Figure 3 a) Change in resistance, normalized to the non-loaded fabric resistance (R_0), measured during 1000 stretch/relaxation cycles of 0 to 100% strain at 0.2 Hz for a knitted CNT/SPX₈ textile. (b) Change in normalized electrical resistivity versus applied strain during loading and unloading for CNT/SPX_n textiles.

Supporting Information

Knitted Carbon-Nanotube-Sheath/Spandex-Core Elastomeric Yarns for Artificial Muscles and Strain Sensing

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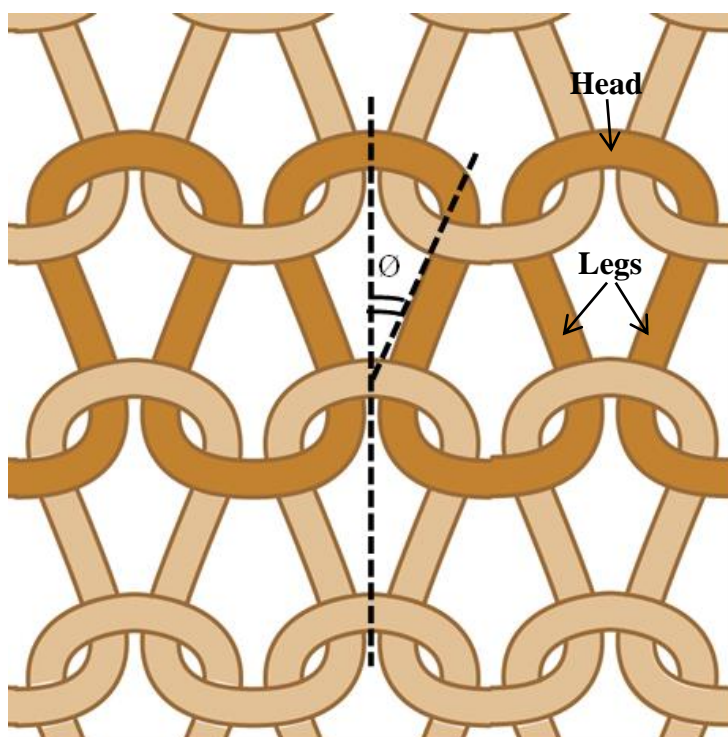


Figure S1: Schematic diagram illustrating the plain stitch knitted structure (weft knitting). The head, legs, and, knit angle (ϕ) are defined.

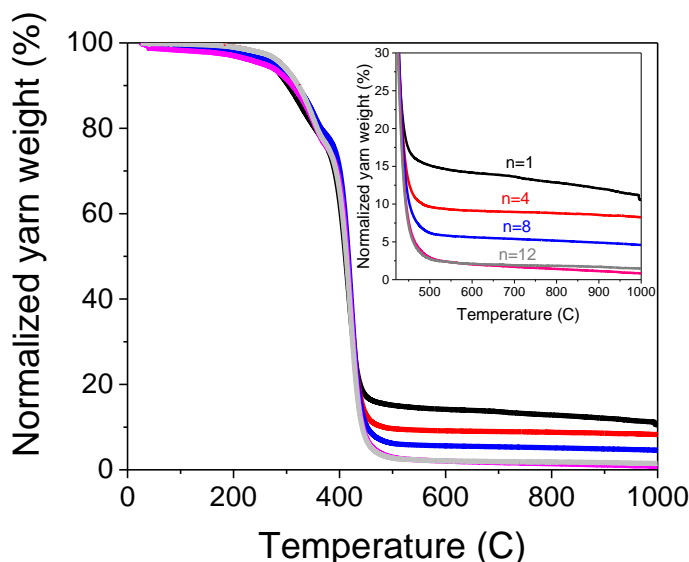


Figure S2 Thermal gravimetric analysis (TGA) results for SPX and CNT/SPX_n ($n = 1, 4, 8$ and 12) knitted textiles (from bottom to top), respectively, in flowing argon at a scan rate of $10^{\circ}\text{C}/\text{s}$. The textile weight, normalized with respect to the initial weight (and expressed as a percent) is plotted versus temperature. The inset is an expanded view of the high temperature region.

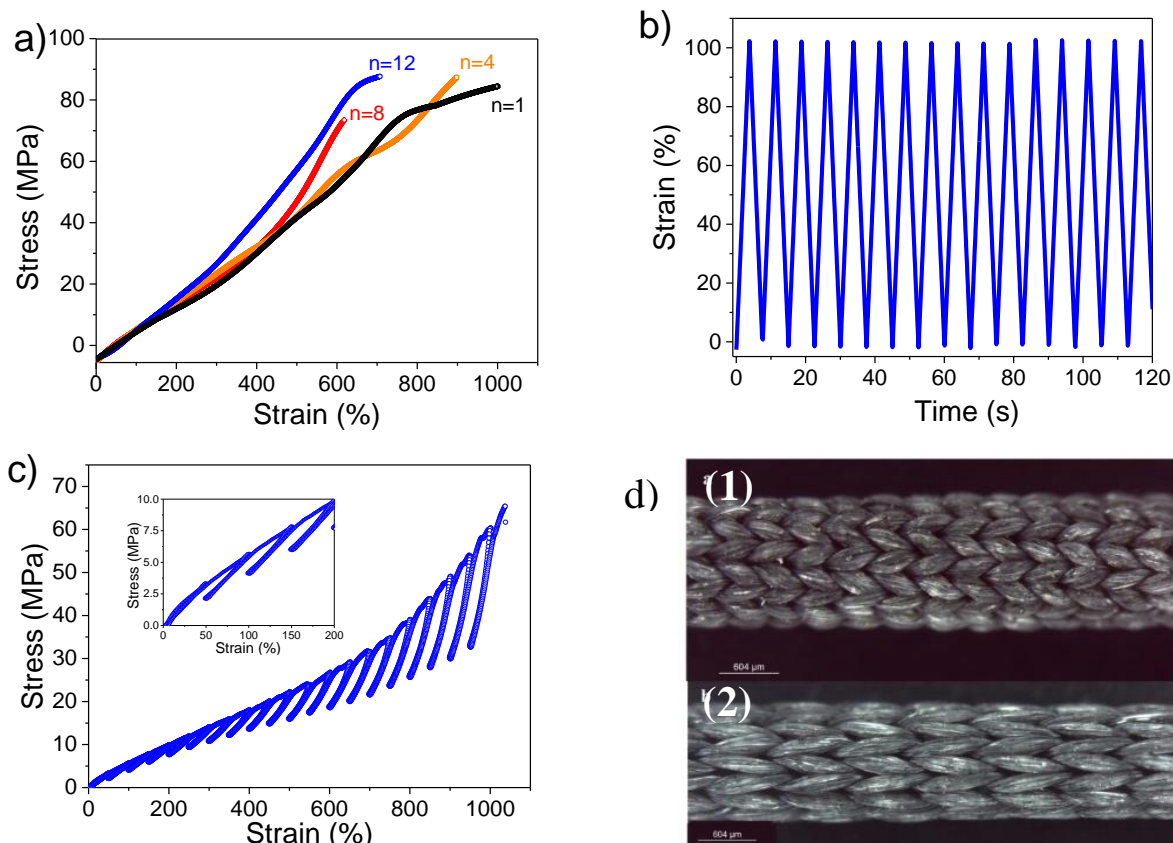


Figure S2 a-d) Mechanical properties of CNT/SPX_n knitted textiles. a) Stress–strain curves for CNT/SPX_n textiles. b) Strain versus time for a CNT/SPX₁ textile that has been stretched for 1000 cycles at 0.2 Hz c) Stress–strain curves (strain rate $100\%/ \text{min}$) observed on unloading and reloading a CNT/SPX₁ textile over a 1000% strain range after differing initial strains. (Inset hysteric stress–strain curves up to 200% strain) d) Photograph of knitted CNT/SPX₈ structures before (d1) and after (d2) 100% strain.

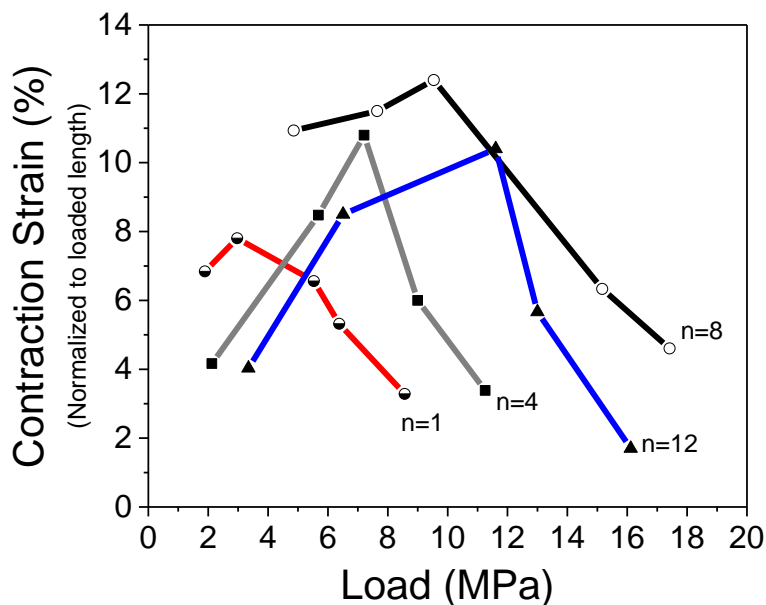


Figure: S3 The tensile actuation data of Figure 2c), when the contractile strain is normalized with respect to the loaded, non-actuated length (rather than the non-loaded, non-actuated length).

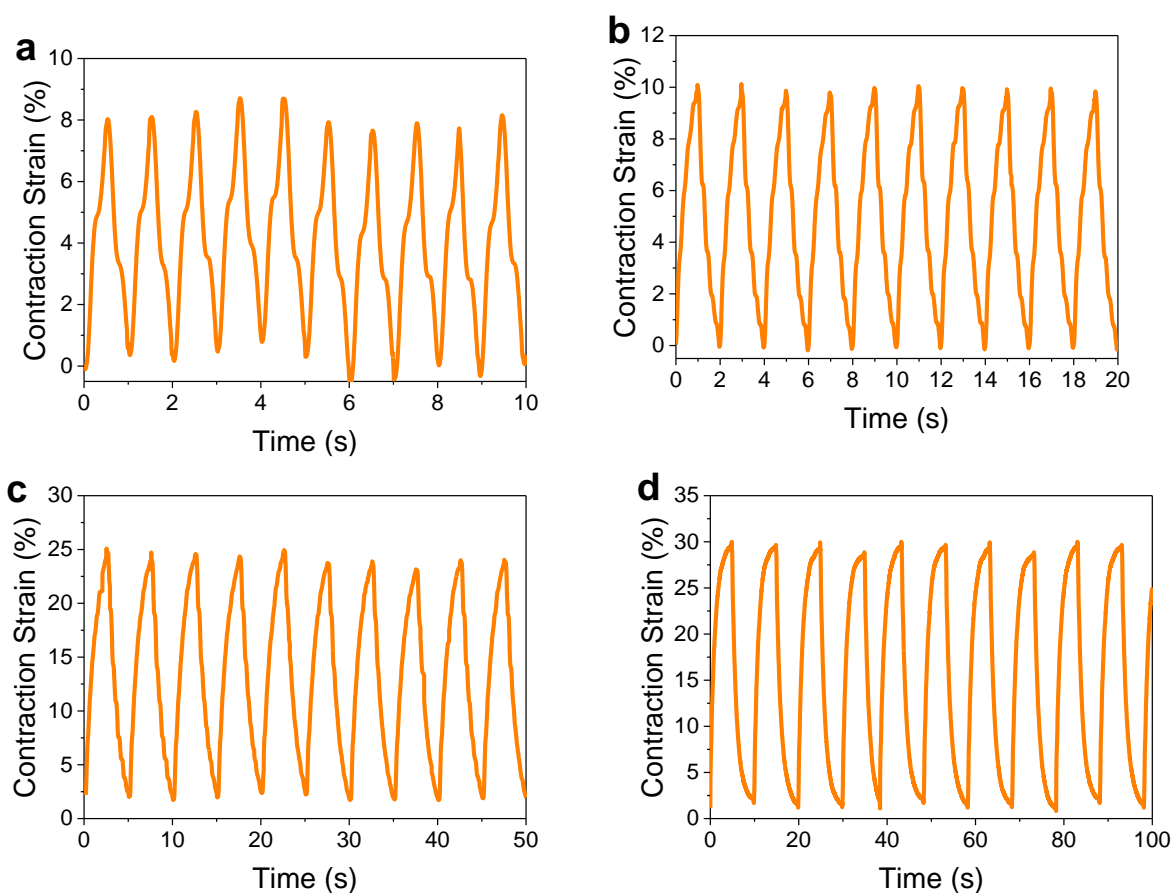


Figure S4 Actuation cycles at different frequencies for a knitted CNT/SPX₄ textile that is under an applied stress of 25 MPa, which provides a tensile strain of 100% for the non-actuated state. The applied square wave voltage is zero to 20 V/cm at frequencies of 1 Hz, 0.5 Hz, 0.2 Hz and 0.1 Hz for the results in a), b), c), and d), respectively.

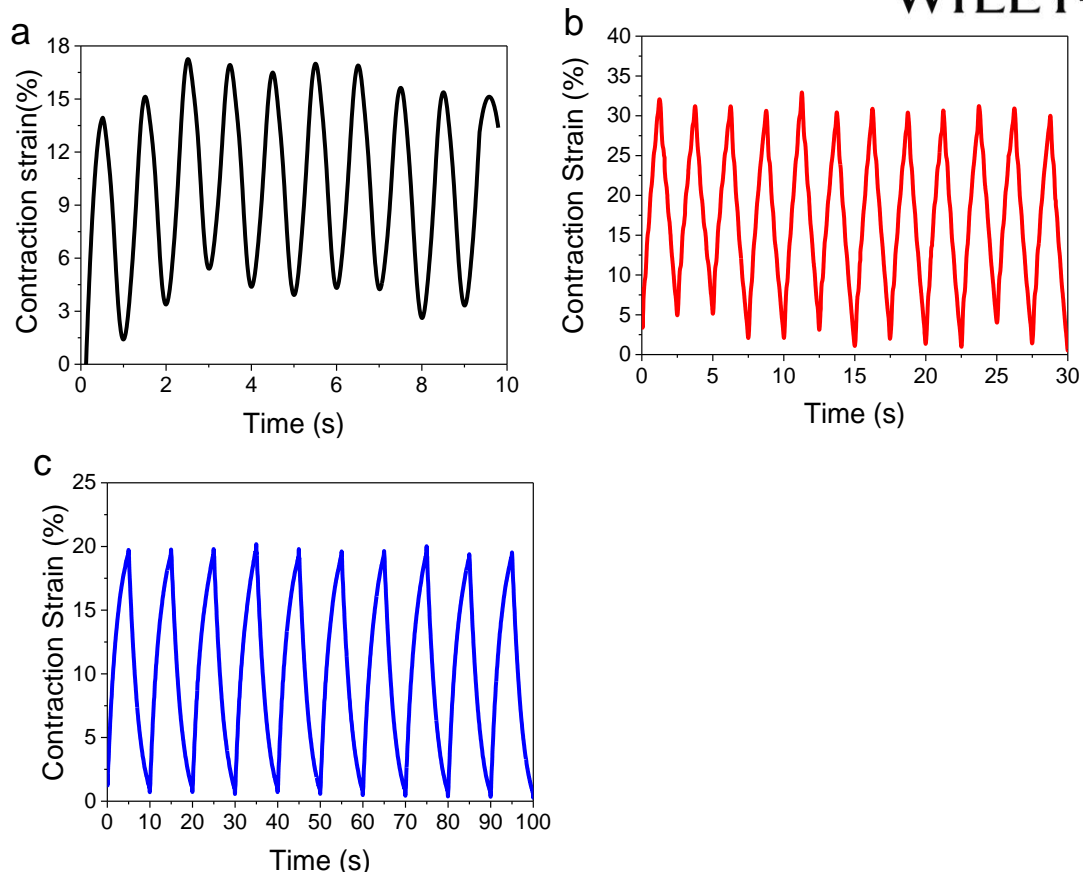


Figure S5 Electrothermal actuation of (a) a CNT/SPX₁ textile at 2 Hz and 10 V/cm; (b) a CNT/SPX₈ textile at 0.4 Hz and 30 V/cm; and (c) a CNT/SPX₁₂ textile at 0.2 Hz and 60 V/cm. The applied stress is 25 MPa, which results in a 100 % engineering strain for the non-actuated state.

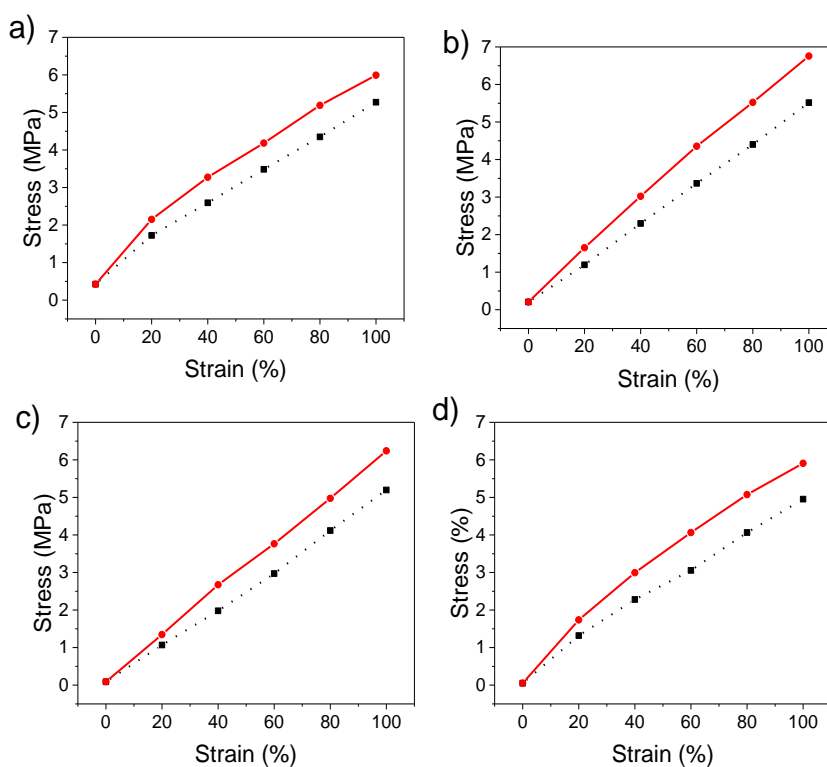


Figure S6 Stress-strain curves of the knitted textiles at room temperature (red data points and solid red lines) and 110 °C (black data points and dashed black lines) for a) CNT/SPX₁, b) CNT/SPX₄, c) CNT/SPX₈, and d) CNT/SPX₁₂ knitted textiles.

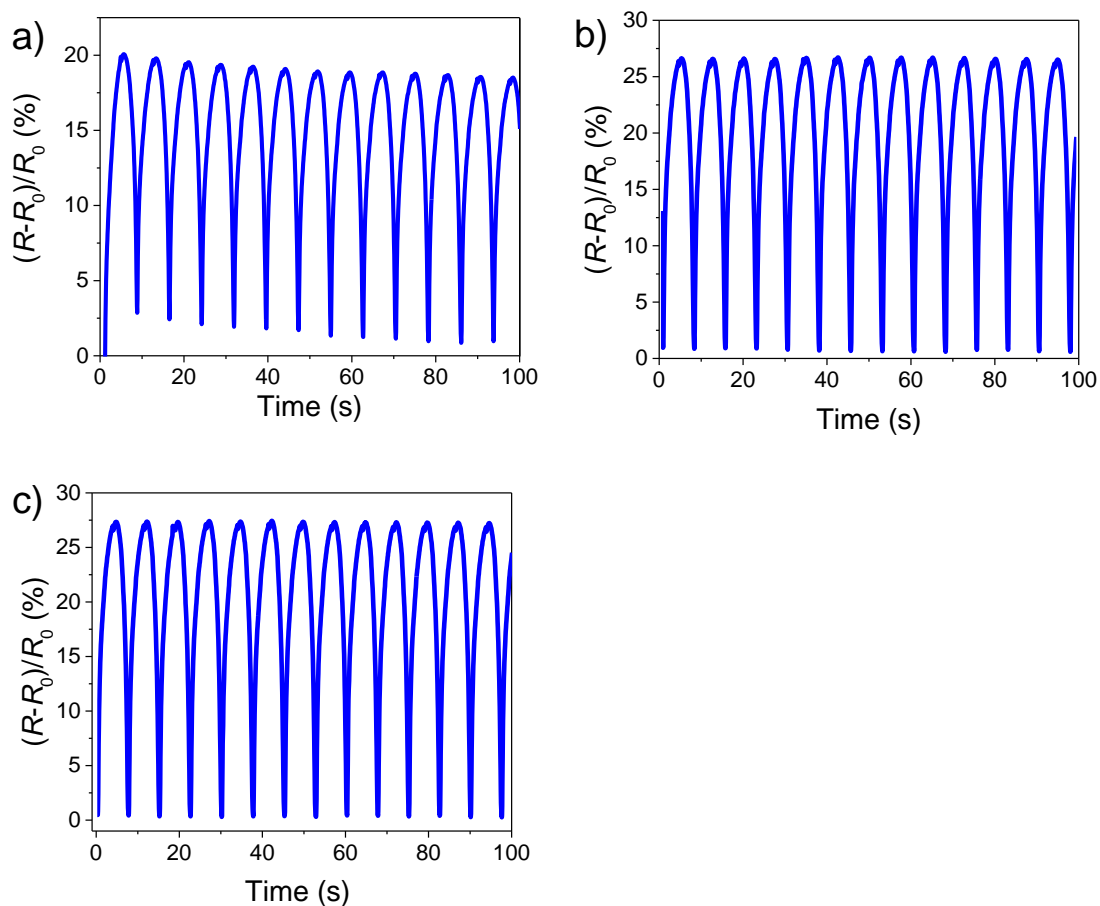


Figure S7 Change in resistance (normalized to unloaded fabric resistance) measured during 1000 stretch/relaxation cycles between 0 and 100% strain at 0.2 Hz for knitted a) CNT/SPX₁ b) CNT/SPX₄ and c) CNT/SPX₁₂ knitted textile.

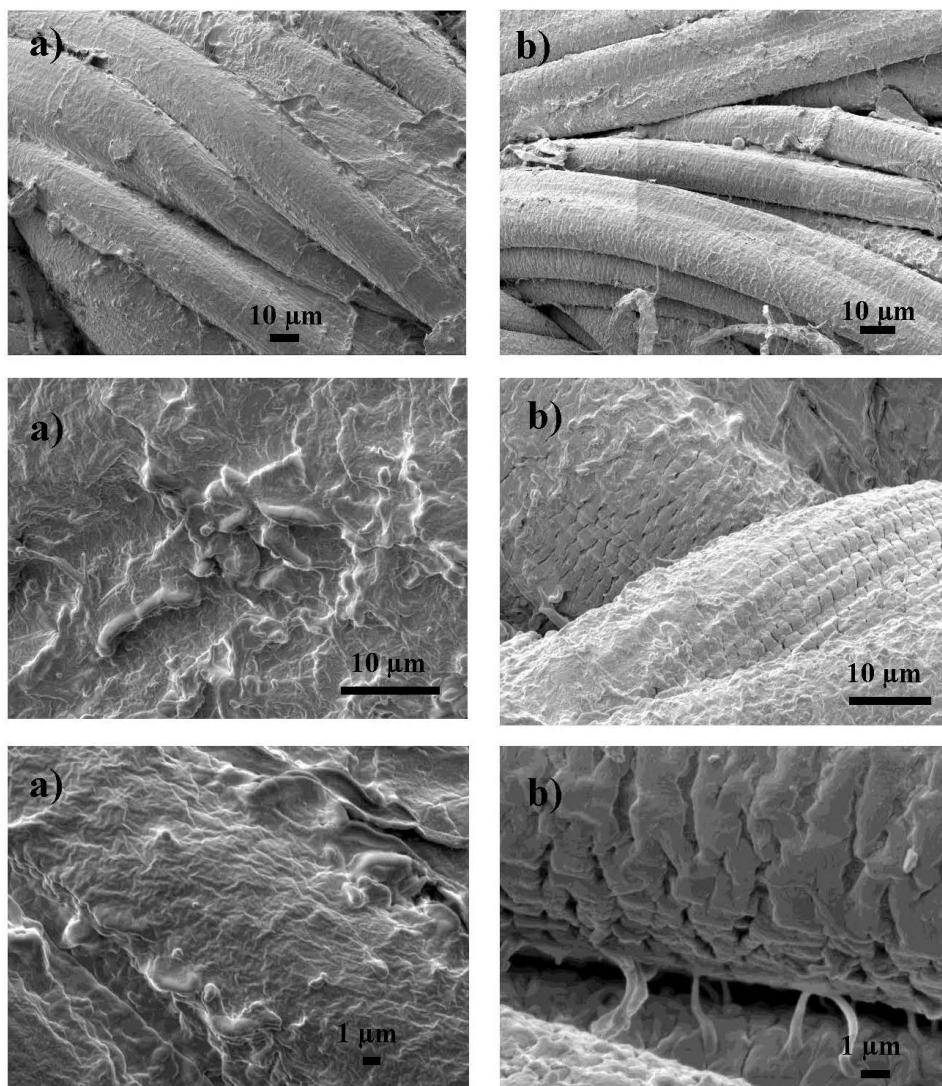


Figure S8 SEM images of CNT/SPX₄ knitted textile a) before stretching and b) after 100% stretching

Application:

The CNT/SPX knitted textile strain gauge can measure applied strain up to 100% in magnitude with high repeatability. The developed textile sensor can be attached to a commercial wireless transmitter for remote acquisition of data on tensile and bending deformations. A knee sleeve prototype based on CNT/SPX₄ wearable strain sensor was developed to help at personal rehabilitation training after an injury (see movie S3).

Using a voltage divider to connect to an expansion board (AnEx board), Shimmer sensor was attached to CNT/SPX₄ knitted textile element (Figure S9). After connecting Shimmer wireless sensor to expansion board it is possible to connect an analog sensor, a digital output sensor, a Serial UART, or a Parallel Bus Interface. AnEx board can be positioned outside the Shimmer box and connected to Shimmer by external connector. The Expansion Board allows knitted textile sensor to use additional function of the Shimmer of adjustment to a specific application and its requirements. The data can be transmitted via Bluetooth to a nearby computer for recording and analysis.

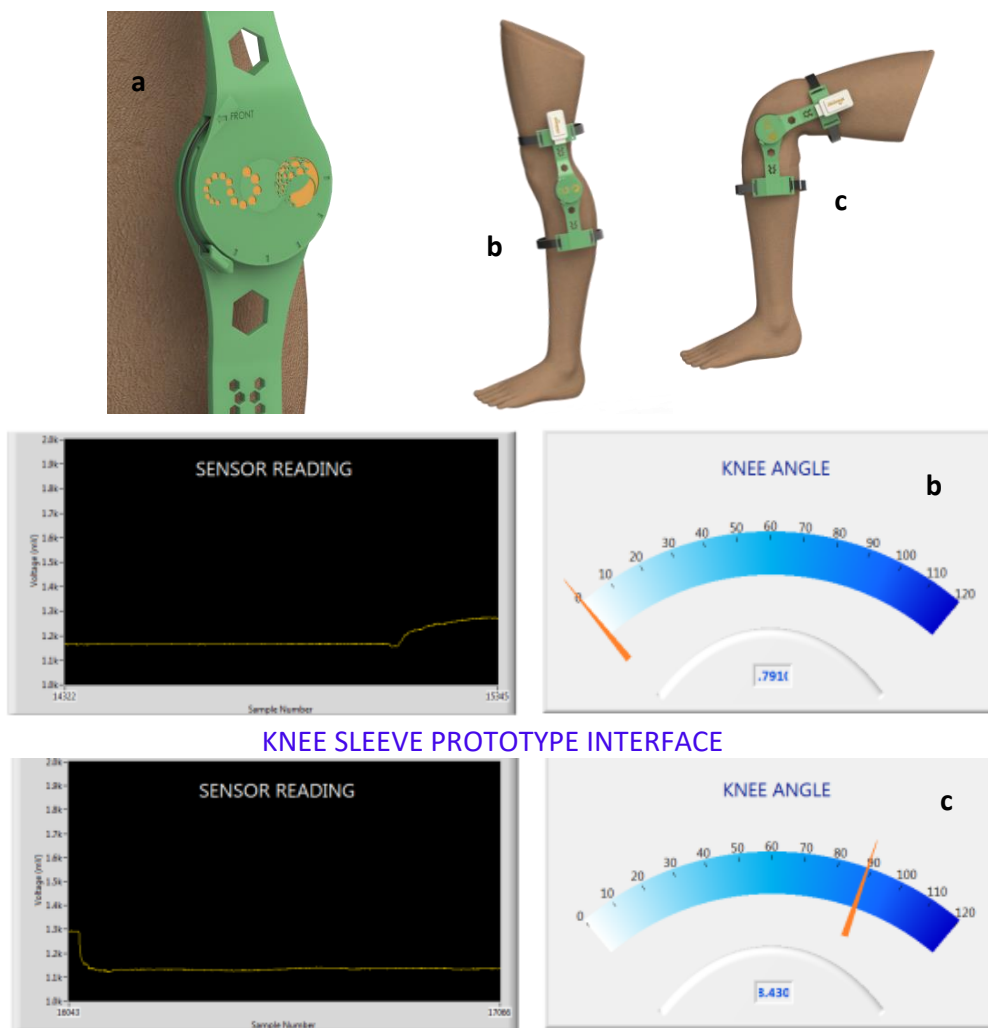


Figure S9 a) Prototype knee sleeve device (wireless sensing) of the CNT/SPX_n knitted strain sensor textile, which connects to the AnEx board of the Shimmer™ wireless sensor platform. b) Schematic illustration of the setup used for wireless strain sensing at initial (zero bending) and c) bent (90° bending).

Table S1 Mechanical and electrical properties of CNT/SPX_n knitted fabrics

<i>n</i>	CNT content (wt %) by TGA	CNT content (wt %) from weight	Conductivity (S/m)	Gauge factor for resistance	Textile diameter (μm)	Cross-sectional area (mm ²)	Stress at break (MPa)	Strain at break (%)
1	0	0	-	-	575	0.045	78	895
1	9.08	8.65	7092	0.12	580	0.047	86	900
4	6.79	6.46	3436	0.16	1000	0.099	84	880
8	3.1	4.27	1515	0.25	1450	0.225	75	600
12	2.11	2.4	870	0.4	1700	0.393	82	700

Movie S1: Fabrication of CNT/SPX_n knitted smart textile. Movie showing how CNT webs from multi-walled carbon nanotube forest and spandex fibers are fed simultaneously into the circular knitting machine.

Movie S2: Electromechanical Actuation of Knitted Carbon Nanotube/Spandex Textile.

Movie showing the electromechanical actuation of a CNT/SPX₄ knitted textile that contracts by 25% while lifting a load of 25 MPa as a result of electrothermal actuation. The applied square wave voltage is zero to 20 V/cm at a frequency of 0.5 Hz.

Movie S3: Strain Sensing from Knitted Carbon Nanotube/ Spandex textile.

Movie showing a prototype knee sleeve using a CNT/SPX₄ knitted textile as strain sensor.