

Niobium Nanowire Yarns and their Application as Artificial Muscles

Seyed M. Mirvakili, Alexey Pazukha, William Sikkema, Chad W. Sinclair,
Geoffrey M. Spinks, Ray H. Baughman, and John D. W. Madden*

Metal nanowires are twisted to form yarns that are strong (0.4 to 1.1 GPa), pliable, and more conductive ($3 \times 10^6 \text{ S m}^{-1}$) than carbon nanotube yarns. Niobium nanowire fibers are extracted by etching a copper-niobium nanocomposite material fabricated using the severe plastic deformation process. When impregnated with paraffin wax, the niobium (Nb) nanowire yarns produce fast rotational actuation as the wax is heated. The heated wax expands, untwisting the yarn, which then re-twists upon cooling. Normalized to yarn length, 12 deg mm^{-1} of torsional rotation was achieved along with twist rates in excess of 1800 rpm. Tensile modulus of $19 \pm 5 \text{ GPa}$ was measured for the Nb yarns, which is very similar to those of carbon multiwalled nanotubes.

1. Introduction

When made sufficiently fine, metallic nanowires (whiskers) are found to be defect free and to have mechanical properties that approach those predicted by the intrinsic bond strength.^[1–6] The resulting tensile strengths (e.g., $\approx 9 \text{ GPa}$ for Cr whiskers)^[4] and elongations at break ($\approx 5\%$ for Fe whiskers)^[1] approach those of the much vaunted carbon nanotube, with carbon multiwalled

nanotubes for example demonstrating tensile strengths in the range of 11 to 63 GPa and elongations of 3 to 13%.^[7] Such metallic whiskers have been used successfully as reinforcements in nanocomposites. The whiskers can be produced in-situ by phase transformation^[8] or through deformation processing^[9–11] in which nanowhisker reinforced metallic wires are created through a repeated process of 'bundle-drawing'. Copper-niobium wires with tensile strengths of $\approx 2 \text{ GPa}$ produced by this method are used as ultra-high strength electrical conductors for high field magnets.^[12] In this work, copper

has been etched from the Cu-Nb wires to extract highly aligned Nb nanofilaments, as described in the supplementary materials. These Nb filaments, having a diameter of approximately 100 nm,^[13] are twisted into yarns for mechanical and electrical characterization, and for use as actuators (Figure 1a,b).

Piezoelectrics, magnetostrictives, shape memory alloys, and electroactive polymers are materials that generate linear and volumetric displacements when stimulated,^[14,15] enabling them to be used as actuators. None of these materials have demonstrated significant torsional actuation except when patterned in such a way as to convert linear motion into twist and untwist.^[16]

Carbon nanotube containing sheets and yarns have been shown to undergo dimensional changes when electrochemically charged in an electrolyte^[17,18]—like when an electrochemical double-layer capacitor is charged. A recent discovery has shown that the highly twisted carbon nanotube yarns, which have a built-in asymmetry (handedness), can be made to torsionally actuate when electrochemically swollen with ions^[19] or when otherwise made to change in volume.^[20] Wax exhibits a large volumetric expansion when heated (30% between 20 °C and 210 °C),^[20] a property that has been previously used to achieve actuation, and is very effective in microactuators since the thermal time constants are small.^[21] The impregnation of nanotube yarns with wax leads to exceptional actuation properties.

The carbon nanotube yarns, and the niobium nanowire yarns presented here, provide a conductive matrix in excellent thermal contact with the inserted wax. The degree of yarn twist determines the extent wax volume change is converted to rotational actuation and linear motion. The stiff nanofibers, surrounded by expanding wax, are effectively inextensible. Three motions can result from an increase in yarn volume caused by expanding wax—a change in diameter, a change in length, and

Prof. J. D. W. Madden
Department of Electrical and Computer Engineering
Advanced Materials and Process Engineering Laboratory
University of British Columbia
Vancouver, BC, V6T 1Z4, Canada
E-mail: jmadden@ece.ubc.ca



S. M. Mirvakili, A. Pazukha, W. Sikkema
Department of Electrical and Computer Engineering
Advanced Materials and Process Engineering Laboratory
University of British Columbia
Vancouver, BC, V6T 1Z4, Canada

Prof. C. W. Sinclair
Department of Materials Engineering
University of British Columbia
Vancouver, BC, V6T 1Z4, Canada

Prof. G. M. Spinks
Intelligent Polymer Research Institute
Australian Research Council Centre of Excellence
for Electromaterials Science
University of Wollongong
Wollongong, NSW 2522, Australia

Prof. R. H. Baughman
The Alan G. MacDiarmid NanoTech Institute
University of Texas at Dallas
Richardson, TX 75083, USA

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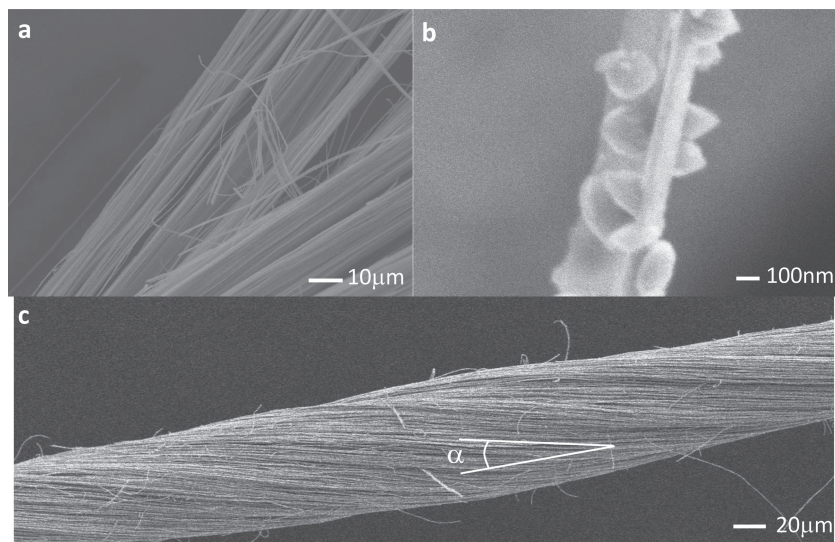


Figure 1. SEM image of niobium fibers. a) Strands of niobium remaining after copper etching, b) close-up of fibers showing un-etched pyramids of copper on the nanowires, c) niobium twist spun with twist angle α of 13° .

untwisting. The carbon nanotube yarns (like the niobium yarns in this work) have a twist angle (α in Figure 1c), which is the angle the outer fibers make with the yarn axis, given by $\alpha = \tan^{-1} 2\pi rT$, where r is the distance from yarn center and T is the inserted twist in turns per yarn length.^[19] Volume expansion at constant length is associated with yarn untwist which in turn enables an increase in yarn diameter, as described in the supplementary materials and depicted in Supporting Information Figure S5. If the yarn is not allowed to change twist (for example if both ends are clamped), then yarn volume increase is predicted to be associated with a contraction in length and an increase in diameter for twist angles that are sufficiently small.

Recent work with wax-infiltrated carbon nanotube yarns shows changes in twist of up to 100° per millimeter of length, maximum rotational speed of 11 500 rpm, and maximum torque to mass ratio of $8.42 \text{ N} \cdot \text{m} \cdot \text{kg}^{-1}$ can be obtained from wax-impregnated yarns upon thermal expansion and melting of the wax.^[20] The gravimetric torque achieved is greater than those observed in electric motors. In the present work we report, for the first time, that torsional and linear actuation is also obtained using wax-impregnated Nb yarns, with the high conductivity of these yarns enabling higher heating rates for the same applied voltage.

2. Results and Discussion

The conductivity of the niobium yarns is found to be $3 \times 10^6 \text{ S m}^{-1}$ at room temperature, which is half that of bulk niobium and is large compared to the $3 \times 10^4 \text{ S m}^{-1}$ of carbon multiwalled nanotube yarns.^[22,23] The difference in conductivity suggests that for yarns of identical geometries about 10 times lower voltage can be used to generate the same heating power in niobium. The yarns burn in air at current densities above $3 \times 10^6 \text{ A m}^{-2}$ —which is similar to the current rating of insulated copper wires.

Stress-strain curves were obtained at loads of up to $120 \pm 14 \text{ MPa}$, with 7 ramp cycles each 4 s long, performed sequentially (Supporting Information Figure S3a). The neat niobium yarn tensile modulus of $19 \pm 5 \text{ GPa}$ is found from the slopes up to 15 MPa stress (Figure 2a and Supporting Information Figure S3b). This value is similar to the 15–20 GPa moduli seen in carbon multiwalled nanotube yarns.^[22] The tensile modulus for fully wax-infiltrated yarns containing molten wax and solidified wax is found to be $7 \pm 2 \text{ GPa}$ and $10 \pm 1 \text{ GPa}$, respectively. The yarn showed a creep of $\approx 0.07\%$ over the period of the test, with larger creep being observed in the first run of this experiment (Supporting Information Figure S3b). The tensile strength (Figure 2b) reached $1.1 \pm 0.1 \text{ GPa}$ in a $14 \mu\text{m}$ diameter sample, higher than the 460 MPa obtained in the carbon nanotube yarns employed in actuation,^[22] but lower than the 2 GPa achieved for centimeter gauge length yarns spun from carbon nanotubes synthesized in the gas phase.^[24] Thicker samples ($20\text{--}50 \mu\text{m}$) showed tensile strengths as low as 400 MPa, and that of the wax-containing yarn ($80 \mu\text{m}$ diameter) is only $\approx 110 \text{ MPa}$. The tensile strength appears to drop with increasing diameter, perhaps due to damage induced in the thicker yarns from the twisting. The niobium has a lower elongation at break than the carbon multiwalled nanotubes,^[1,7] which may limit the achievable twist.

The torsional stiffness (the spring constant in twist) was found by hanging a rod from the yarn in a torsional pendulum configuration, and recording the natural frequency of the twist via a video camera.^[25] The torsional stiffness is found to be $7.1 \times 10^{-10} \text{ N} \cdot \text{m} \cdot \text{rad}^{-1}$ without wax, based on the torsional oscillation frequency of the yarn with a suspended paddle.^[26] The shear modulus is estimated to be $17 \pm 6 \text{ GPa}$, which is similar to that estimated from torsional stiffness data in carbon nanotube yarns (17.7 to 21.6 GPa).^[19] Torsional stiffness in the waxed yarns is expected to be lower, probably by a factor of 1.5 to 2.5 seen in the tensile modulus.

Active rotational actuation was obtained by infiltrating wax into the yarn,^[26] which was then alternately electrically melted and re-solidified to induce untwist and then re-twist. As suggested in work on carbon nanotube yarns,^[19] half of the yarn's length is infiltrated with wax, with the other half of the yarn providing a restoring force to assist recovery of the twist (Figure 3a). Melting is produced by applying 0.32 V cm^{-1} (890 W cm^{-3}) to 12 V cm^{-1} (30 kW cm^{-3}) (Supporting Information Figure S6a) square pulses through the yarn, thereby providing a range of input power to volume of 300 W cm^{-3} to 30 kW cm^{-3} .

Figure 3c presents an example of the temperature profile obtained for a case where a pulse of 1.36 V cm^{-1} is applied (300 W cm^{-3}) for $\approx 8 \text{ s}$. The image indicates that there are lower temperature sections of the yarn (middle and left portions). These correspond to wax-filled regions. Presumably the higher heat capacity and large latent heat of melting of these wax-infiltrated regions leads them to be cooler. The waxed regions

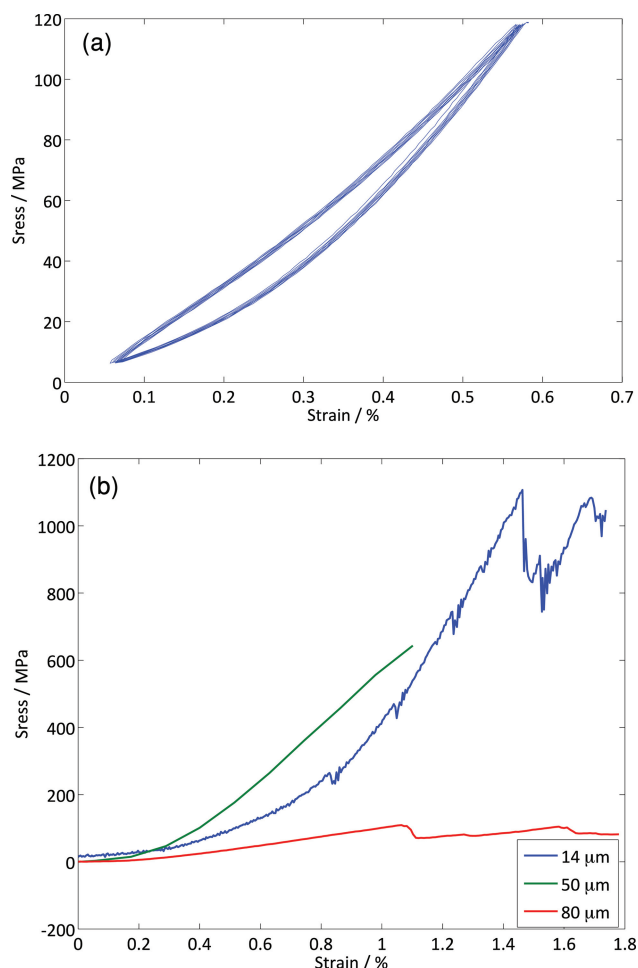


Figure 2. a) Stress-strain curves obtained by repeated loading and unloading of a $110 \pm 15 \mu\text{m}$ diameter niobium yarn having an inserted twist of 516 turns per meter and a length of 31 mm. Maximum load of 118.7 MPa is achieved in the stress-strain curve. b) Results of tensile tests of $14 \pm 1 \mu\text{m}$, $50 \pm 1 \mu\text{m}$, and $80 \pm 2 \mu\text{m}$ diameter yarns with identical inserted twists of 800 turns per meter. The $80 \mu\text{m}$ yarn is wax impregnated. The strain rates for these tests are $0.17\% \text{ s}^{-1}$, $4\% \text{ s}^{-1}$, and $0.33\% \text{ s}^{-1}$, respectively.

also have longer thermal time constants for heating and cooling, as observed in video sequences provided online (Supporting Information Movie S1).

Rotation as a function of time in response to a 100 ms long pulse is shown in Figure 3b, as determined by frame-by-frame video analysis at 200 frames per second. There is an initial rapid unwinding (blue line) in response to a voltage spike (dashed line), followed by a damped oscillation and a gradual re-twisting. The cooling time constant of several hundred milliseconds determines the rate of recovery of the twist. In this experiment (Supporting Information Movie S2) the rotation rate reaches 650 rpm, which is similar to the 592 rpm achieved in electrochemically driven carbon nanotube yarns, but slower than the 11 500 rpm average torsional rotation rate demonstrated in wax infiltrated carbon nanotube yarns. The resonant response, as indicated by the damped oscillation following

the 100 ms excitation (Figure 3b and Supporting Information Movie S2), demonstrates that paddle inertia is playing an important role in limiting the rate.

The initial acceleration of the paddle (Figure 3b) is found to be 2500 rad s^{-2} ($150\,000 \text{ deg s}^{-2}$). Given the initial angular acceleration, and the paddle moment of inertia ($I = m \frac{L^2 + W^2}{12}$, with $m = 22.49 \text{ mg}$ and $I = 3.34 \times 10^{-10} \text{ kg m}^2$), the torque produced is 0.8 mN m . The mass of the active portion of the yarn and the wax is $960 \mu\text{g}$, and the torque to mass ratio is 0.9 N m kg^{-1} . The torque to mass is about 1/10th that of wax-infiltrated carbon nanotube yarns,^[20] and the torque to volume is about 1/3rd as large. The heating power per volume is higher in the niobium case, suggesting that the rate of heating is not the cause of the lower torque per volume. Given the resonant response, the paddle inertia is almost certainly limiting rate. The lower twist angle (21°) compared to those of the nanotube yarns (up to 43°) is an important factor reducing the torque and speed generated, as discussed below. The torque to mass ratio is nevertheless substantial—at about 1/4 that produced by commercial direct drive motors.^[15] The power to mass is 35 W kg^{-1} ($\frac{1}{2} \frac{\omega^2}{\Delta T}$), compared to 61 W kg^{-1} obtained in electrochemically driven carbon nanotube yarn actuators,^[19] with the power to volume in the niobium yarn being higher than in these electrochemically driven carbon nanotube yarns.

Fast torsional actuation was achieved by using a loop of yarn to observe twist instead of a paddle (henceforth a “paddle-free yarn”) in a half-waxed yarn. In this low inertia paddle-free yarn, that is once again half waxed, the peak rotation rate reaches at least 1800 rpm. This rate is determined by the observed 360-degree rotation in less than one video frame (1/30th of a second) upon application of a 4.8 V pulse (3 kW cm^{-3}). Untwist is slower, taking about 1 s, due to the thermal time constant of the yarn. This thermal time constant should be dramatically reduced in thinner yarns, as found for carbon multi-walled nanotubes, where $11.5 \mu\text{m}$ diameter yarns show a large recoverable response at 20 Hz, while $150 \mu\text{m}$ yarns have a time constant of more than a second. The niobium yarns are twisted and manipulated by hand, making it a challenge to achieve the very thin yarns machine spun from carbon nanotubes.

In this work, we have introduced a new configuration for structuring the twisted yarns to improve the mechanical stability and actuation performance. In the structure of this fast acting sample, two yarns are twisted together with a thin polymer fiber (Supporting Information Figure S6b,c), which results in some coiling and an increase in heat capacity. The yarn employed is 3 cm long and $60 \mu\text{m}$ in diameter, with an inserted twist of $2300 \text{ turns m}^{-1}$. Increasing pulse voltage amplitude leads to faster rotation (up to 7200 rpm but also to burning. Observations in other yarns indicate that burning of the yarn and the resulting failure begins near the center of the yarn where the wax-free and waxed portions of the yarn meet (as seen in the Supporting Information Movie S3). The non-waxed portion of the yarn reaches higher temperatures than the waxed portion, presumably due to its lower heat capacity, leading to the ignition of the neighboring waxed regions. Adding the polymer fiber strand increases the heat capacity, which results in lower temperatures on the neat side of the yarn, and may enable

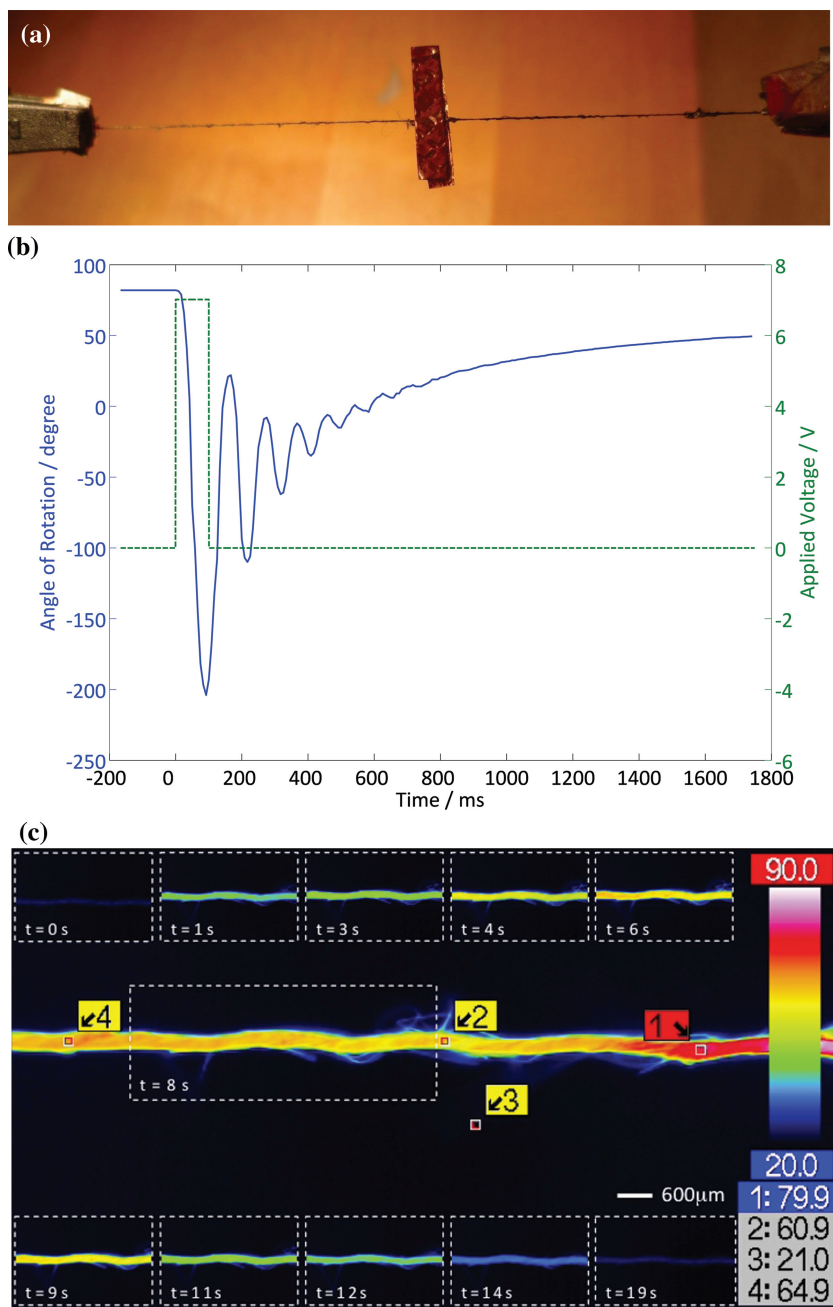


Figure 3. a) The torsional actuation configuration, with the right half of the yarn infiltrated with wax and melting induced by current pulses. The central paddle is used to determine rotation. It has a mass 23 times larger than the yarn's and a moment of inertia of $3.3 \times 10^{-10} \text{ kg}\cdot\text{m}^2$. The yarn is held at constant length. The yarn diameter without wax is $51 \mu\text{m}$, the inserted twist is $2,381 \text{ turns m}^{-1}$, the distance between clips is 6.3 cm , and the paddle is 13 mm long by 3 mm wide. b) Angle of rotation versus time upon pulse voltage actuation of the Nb yarn and paddle shown in Figure 3a. The resistance is 35Ω and melting is produced by a 100 ms square pulse with amplitude of 7.02 V , represented by the dashed line. The power per volume produced by this pulse is 11 kW cm^{-3} . c) Thermal profile of a half-wax-infiltrated yarn three seconds after the application of a square wave voltage pulse of 1.36 V cm^{-1} . The red region is without wax. Arrows and the legend correspond to the approximate temperatures reached. The yarn diameter without wax is $310 \mu\text{m}$, the total yarn length between clamps is 22 mm , and the input power per volume was 300 W cm^{-3} . The emissivity of the Nb is <1 so the temperature scale may underestimate the true temperature at the surface of the yarn.

higher wax temperatures before failure in the yarn is observed.

Inertia no longer appears to be limiting the rate of twist in the paddle-free yarns. This is evident in the lack of oscillation seen in the responses of these yarns. The speed of response and torque are likely instead to be set by the heating rate. The use of shorter pulses whose thermal energy is carefully controlled may lead to even fast rotation while avoiding burning. Increasing length and twist angle, as well as reducing thickness of the yarn should also lead to still faster rotation, as discussed in the supplementary materials.^[26] The twist achieved in the niobium is not as large as that in carbon nanotube yarns, perhaps because carbon nanotubes exhibit higher elongation at break and greater tensile strength than the niobium nanofilaments. The result of lower twist is less rotation per actuator length, and for the same heating rate, lower rotation speed. In addition, the large Nb diameter, compared to CNT diameter, can play a role in reducing the allowable twist on the Nb yarn.

The higher electrical conductivity of the Nb yarns enables lower voltage operation than in carbon MWNT yarns for the same heating rate. Lower voltage operation is beneficial in portable battery operated applications (e.g., toys, valves and implanted medical devices). The yarn used to generate the data in Figure 3c is activated using 1.36 V cm^{-1} , whereas a $150 \mu\text{m}$ carbon nanotube yarn^[20] activated over a similar time frame employs 15 V cm^{-1} . A centimeter length of the niobium yarn thus requires only a single alkaline cell to activate, whereas the same length of nanotube yarn needs ten.

In addition to the torsional actuation, an isobaric tensile (linear) actuation of 0.24% was observed, as shown in Supporting Information Figure S4b. This strain is larger than that achieved for a non-coiled carbon multiwalled nanotube yarn, but much smaller than for the coiled nanotube yarn.^[20]

The presence of torsional actuation in both the carbon nanotube and the niobium nanowire yarns confirms that the actuation is not specific to carbon. Indeed, torsional actuation might be expected in any helically wound structure in which a volume change is induced as long as the yarn guest can remain confined in the yarn. The use of nanofiber yarns provides surface energies that result in wax confinement when the interfacial energy between nanofiber and air is much less energetically favorable than between guest and nanofiber.

Table 1. Comparison between properties of Niobium nanowire twisted yarns and twist-spun carbon multiwalled nanotube yarns.

	Niobium Nanowire Twisted Yarn	Twist-spun Carbon Multiwalled Nanotube Yarns ^{a)}
Conductivity	$3 \times 10^6 \text{ S m}^{-1}$	$3 \times 10^4 \text{ S m}^{-1}$
Ultimate Tensile Strength	1.1 GPa	460 MPa
Young's Modulus	$19 \pm 5 \text{ GPa}$	15 to 20 GPa
Shear Modulus	$17 \pm 6 \text{ GPa}$	18 to 22 GPa
Tensile Actuation	0.24% (@20 MPa)	0.12% (non-coiled @ 4.8 MPa)
Torsional Actuation Speed	7200 rpm	11500 rpm
Torque to Mass	$0.9 \text{ N}\cdot\text{m}\cdot\text{kg}^{-1}$	$8.4 \text{ N}\cdot\text{m}\cdot\text{kg}^{-1}$

^{a)}Values are obtained from refs. [19,20,22,23].

3. Conclusions

Niobium offers an alternative material for creating high strength yarn. **Table 1** compares the properties of the Nb yarns to those of twist spun carbon multiwalled nanotube yarns. Although carbon nanotubes offer much larger linear actuation strains (in a coiled configuration), and higher strength with wax added, nevertheless metal-based nanostructured fibers provide some key advantages that make them worthy of attention. Tensile strength and modulus of the neat niobium yarns are in the same range or higher than those observed in carbon multiwalled nanotube yarns. A key advantage of the metal nanowire yarns is their high electrical conductivity (100 times that of carbon multiwalled nanotube yarns). The high conductivity of the niobium yarns makes them promising for use in small, tightly wound magnetic coils and for flexible metal contacts, where conventional wires are not sufficiently compliant. In thermally induced torsional actuation, the higher conductivity allows heating to be achieved with lower voltages than are needed in carbon nanotube yarns, making them of potential interest in portable devices including implantable drug delivery systems, guided catheters, active toys and miniature valves.

4. Experimental Section

Niobium Yarn Preparation: Nanometer-scale niobium fibers are extracted from copper-niobium composite wires that are drawn using a severe plastic deformation (SPD) process.^[13] These composite wires were provided by National High Field Magnet Laboratory (Tallahassee, FL). The SPD process used to create these wires involved accumulated drawing and bundling to produce a copper-niobium matrix.^[13] The repetition of three hot-extrusion, cold drawing, and bundling cycles produce a sample with roughly 620 000 niobium nanowires in a cross-section. The volume fractions of copper and niobium are 80% and 20%. Free-standing niobium nanowires were prepared by etching 4.0 mm diameter, 46 mm long sections of niobium-copper wires in nitric acid solution (50% HNO₃ and 50% H₂O) at room temperature for 3 days and then rinsing. The copper is almost completely removed (Supporting Information Figure S1), as determined using a Philips XL30 Scanning electron microscope equipped with Bruker Quanta 200 energy-dispersion X-ray microanalysis system. Small nodules remain on the niobium fibers in some locations that are likely copper (Figure 1b; main text).

The resulting niobium wires are twisted to form a yarn (Figure 1c). The niobium copper wires are available in much longer lengths, since

they are used as windings in ultra-high field magnets, so longer samples should be readily produced by using larger etching baths and longer starting lengths of wire. Twist is inserted into the yarns by hand, and is between 1200 and 5300 turns per meter. The twist angle is in the range of 10° to 40°. The technique used for twisting the yarns produces low twist angle yarns. In some samples twist is performed with a thin strand of hot-melt adhesive (Stanley Works GS15DT DualMelt, referred to as a "polymer fiber") included in the twist (Supporting Information Figure S6). This addition made the yarn/strand combination less brittle.

Wax Impregnation: A low melting temperature wax (Glimma brand, paraffin/vegetable wax, IKEA) is added by hand, with a soldering iron used to melt in the wax.

Supporting Information

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

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