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Environmentally Powered Yarn Arrays that Sense, Actuate, Harvest, and Store Energy (NBIT III)

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Final Report

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14. ABSTRACT In our early NBIT III advance, we used twist insertion and coiling to transform the high strength polymers found in fishing line and sewing thread into large stroke, highly reversible, thermally-powered artificial muscles that (1) generated five times higher gravimetric mechanical power during muscle contraction than the gravimetric power generation capability of a cars combustion engine and (2) functioned as a torsional artificial muscle to rotate a heavy rotor to over 90,000 rpm. By driving this torsional actuation using 19.6C fluctuations in air temperature, we obtained an average output electrical power of 124 W per kg of muscle mass. Our subsequent NBIT III results have expanded our understanding of actuation mechanism, including providing results on the entropy-based mechanocaloric cooling resulting from stretch release and twist de-insertion. A carbon nanotube hybrid muscle that is driven by either fluctuating relative humidity or periodic contact with water was obtained. This bioinspired, muscle provided a giant tensile stroke (up to 78%) and a giant maximum gravimetric work capacity during contraction (2.17 kJ kg ⁻¹), which is over 50 times that of the same weight human muscle and 5.5 times higher than for the same weight spider silk, which is the previous record holder for a moisture-driven muscle. Additional work has focused on engineering comfort adjusting clothing textiles that open and close porosity depending upon ambient temperature (and/or humidity) or the existence of sweat.					
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Project Title: Environmentally Powered Yarn Arrays that Sense, Actuate, Harvest, and Store Energy

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Abstract: In our early NBIT III advance, we used twist insertion and coiling to transform the high strength polymers found in fishing line and sewing thread into large stroke, highly reversible, thermally-powered artificial muscles that (1) generated five times higher gravimetric mechanical power during muscle contraction than the gravimetric power generation capability of a car's combustion engine and (2) functioned as a torsional artificial muscle to rotate a heavy rotor to over 90,000 rpm. By driving this torsional actuation using 19.6°C fluctuations in air temperature, we obtained an average output electrical power of 124 W per kg of muscle mass. Our subsequent NBIT III results have expanded our understanding of actuation mechanism, including providing results on the entropy-based mechanocaloric cooling resulting from stretch release and twist de-insertion. A carbon nanotube hybrid muscle that is driven by either fluctuating relative humidity or periodic contact with water was obtained. This bioinspired, muscle provided a giant tensile stroke (up to 78%) and a giant maximum gravimetric work capacity during contraction (2.17 kJ kg^{-1}), which is over 50 times that of the same weight human muscle and 5.5 times higher than for the same weight spider silk, which is the previous record holder for a moisture-driven muscle. Additional work has focused on engineering comfort adjusting clothing textiles that open and close porosity depending upon ambient temperature (and/or humidity) or the existence of sweat. The challenge here is to control porosity over a large range without significantly changing textile dimensions. We have achieved this by diverse means, including using a fascinating muscle topology that enables an effectively infinite muscle stroke. Also relevant for environmentally powering yarn arrays, we have demonstrated the fabrication and characterization of flexible and stretchable nanotube-yarn-based supercapacitors having record performance for existing and emerging wearable applications. The yarn electrodes of these weavable carbon nanotube yarn supercapacitors were made by a novel biscrolling process that traps above 90 wt% of micron-size MnO_2 particles in the galleries of helically scrolled carbon nanotube sheets, which provide yarn strength and electrical conductivity. Despite the high loading of brittle metal oxide particles, which provides a pseudo capacitance, the realized biscrolled solid-state yarn supercapacitors are flexible and can be made elastically stretchable (up to 30% strain) by over-twisting to produce yarn coiling. The maximum linear and areal capacitances of the highly elastic yarn electrodes were 5.3 times and 2.2 times higher, respectively, than previously described for elastic fibers or yarns. These results have been described in our 27 NBIT III publications (nine of which were published in either *Science* or in *Nature* journals). Unpublished work on our high-performance twistron mechanical energy harvesters will be described.

Brief Overview of Project work:

The goal of our research is to provide “Environmentally Powered Yarn Arrays that Sense, Actuate, Harvest, and Store Energy”. The project results were provided by an intense collaboration between the institute of my Korean brother (Seon Jeong Kim at Hanyang University, HU) and my institute at the University of Texas at Dallas (UTD).

This research has so far produced 26 jointly co-authored NBIT III papers, nine of which were published in either *Science* or in *Nature* journals. An important advance on our below mentioned twistron mechanical energy harvesters was made with Benji Maruyama, Larry Drummy, and Matthew Lucas of AFRL, who will co-author a manuscript that will be submitted to *Science*. Parts of our NBIT polymer and carbon nanotube yarn muscle technology were licensed to Lintec of America, who established a laboratory 5 miles from our NanoTech Institute to commercialize our technologies.

For their NBIT III supported work on polymer artificial muscles, Seon Jeong Kim, Ray Baughman, and collaborators shared the R&D 100 Gold Award for Market Disruptor Product (2015) at a 1000 person gala in Los Vegas, which was one of the two highest awards presented. Seon Jeong’s enabled awards include: 2016 Academic Award of Hanyang University, 2015 Award Certificate of Ministry of Science, ICT and Future Planning of Korea (MSIP), 2015 Award of 100 R&D of Ministry of Science, ICT and Future Planning of Korea (MSIP), and 2013 and 2014 Basic Research Awards of the National Research Foundation of Korea (NRF). In part because of NBIT supported discoveries, Ray was made a foreign member of the European Academy of Sciences (2015) and a Fellow of the National Academy of Inventors (2015). He also received the Tech Titans Technology Inventors Award (2015) at a gala in Dallas and the 2015 Inventor Award for Energy Harvesting Materials and Systems.

Table Summarizing Publication Topics

NBIT III Publications on Environmentally Powered Yarn Arrays that Sense, Actuate, Harvest, and Store Energy			
Sensing	Mechanical Actuation	Electrical Energy Harvesting	Electrical Energy Storage
1 Proc. Natl. Acad. of Sci. 2016		2 Scientific Reports 2016	
3 Smart. Mater. Struct. 2016		7 Advanced Materials 2016	9 RSC Advances 2016
4 Smart. Mater. Struct. 2016		8 RSC Advances 2016	
	5 Scientific Reports 2016	6 Adv. Matls. 2016	13 Adv. Energy Mater. 2016
7 Scientific Reports 2016		14 Energy and Environmental Sci. 2015	
10 Small 2016		15 Journal of Power Sources 2015	
11 Nano-Micro Letters 2016		18 Adv. Eng. Mater 2015	
12 Nanoscale 2016			17 Scientific Reports 2015
	14 Energy & Environmental Science 2015		
	16 Nano Convergence 2015		
	19 Nanoscale 2015	20 Nature Comm. 2015	
	21 Nano Letters 2014		24 Adv. Materials 2014
	22 Science 2014		
	23 Nature Comm. 2014		26 Nature Comm. 2013
27 Nanotechnology 2013			
	25 RSC Advances (provides conducting yarn interconnects)		

Since pdfs of our full publications will be made available for review (including Supplementary files), I will not provide summaries here for our below listed 27 NBIT publications (26 of which are joint with our Korean partner). However, in the table I on the left, I have listed these publications for our project on “Environmentally Powered Yarn Arrays that Sense, Actuate, Harvest, and Store Energy” and used color coding to indicate which papers deal predominately with “Sensing”, “Mechanical Actuation”,

“Electrical Energy Harvesting”, and “Electrical Energy Storage” (papers with titles in black font deal with more than one of these functions).

I here describe major uncompleted work NBIT III that is not described in a publication, which is on our unplanned discovery of a remarkable new way to harvest mechanical energy. Small, large-stroke mechanical energy harvesters having high power densities are required for enabling autonomous sensing, communication, and actuation for diverse needs, from the *Energy-Sufficient Soldier* and the *Internet of Things* to *Wearable Electronics* and the *Wired*

Body. Our NBIT III work has provided high-work-capacity fibers and yarns that can harvest environmental thermal energy and chemical energy (including the energy of sorption/desorption) as torsional or contractile mechanical energy. However, the absence of a small, large-stroke mechanical energy harvester meant the only way we could convert this mechanical energy to electrical energy was by using a conventional electromagnetic generator, which increased total harvester weight ten-fold. Our newly invented twistron mechanical energy harvesters provide a solution to this problem.

We have two main types of twistron mechanical energy harvesters: (1) torsional mechanical energy harvesters based on CNT yarns that are twisted, but not coiled, which we name “twisted twistron harvesters” and (2) tensile mechanical energy harvesters that are fully coiled by twist insertion, which we name “coiled twistron harvesters”. We call these devices twistron harvesters (using “tron” from the Greek suffix, meaning device), since we have discovered that these electrochemical harvesters operate by using mechanically inserted twist to increase yarn density, and thereby decrease yarn capacitance.

Using twist-induced changes in electrochemical capacitance of nanofiber yarn electrodes in response to either tensile stress or twist insertion, we have realized both all-solid-state and liquid-electrolyte-based yarns that serve as twistron mechanical energy harvesters. Though presently far from optimized, and requiring improved basic understanding, we have already demonstrated a gravimetric peak power output up to 250 W/kg. Unlike other capacitance based harvesters, which can require a thousand volt bias voltage, our twistron harvesters (which have provided over 0.26 V output voltages per electrode) *require no externally applied bias voltage*. We have used our NBIT III polymer muscles to harvest environmental energy as the mechanical energy that powers our twistron harvesters. These environmental-to-mechanical energy converters and mechanical-to-electrical energy converters can simply be artificial muscle fibers and twistron harvester yarns that are connected together and tethered at their opposite ends to prevent end rotation. Figure 1 shows the electrochemical cell configuration that we used for initial harvester characterizations, the carbon nanotube (CNT) yarn types that we have evaluated and our initially obtained performance results.

Consistent with our interpretation of a change in yarn twist as being the mechanistic process behind our successful use of coiled twistron yarns for energy harvesting, we have found that the application of a tensile stress to a homochiral yarn produces an opposite change in potential (versus a reference electrode) than does application of the same tensile stress to a heterochiral yarn. This result is extremely important, since it means that there is no need for a mechanical jig to convert an applied tensile stress to an extension stress on one electrode and a compression stress on the counter-electrode, which would be the case if both electrochemical electrodes were either homochiral or heterochiral. We have found experimentally that this use of a mechanical harvesting cell comprising homochiral and heterochiral electrodes works wonderfully to, as predicted, increase the power output from a coiled yarn twistron energy harvester when the applied stretch is in-phase for anode and cathode. Importantly for some categories of applications, we have found that an elastomerically deformable solid-state electrolyte can replace the liquid electrolyte that we initially used for our twistron harvesters, so that a coiled yarn twistron tensile energy harvester can simply comprise electrolyte-coated homochiral and heterochiral yarn electrodes that are interconnected by an elastomeric solid-state gel electrolyte.

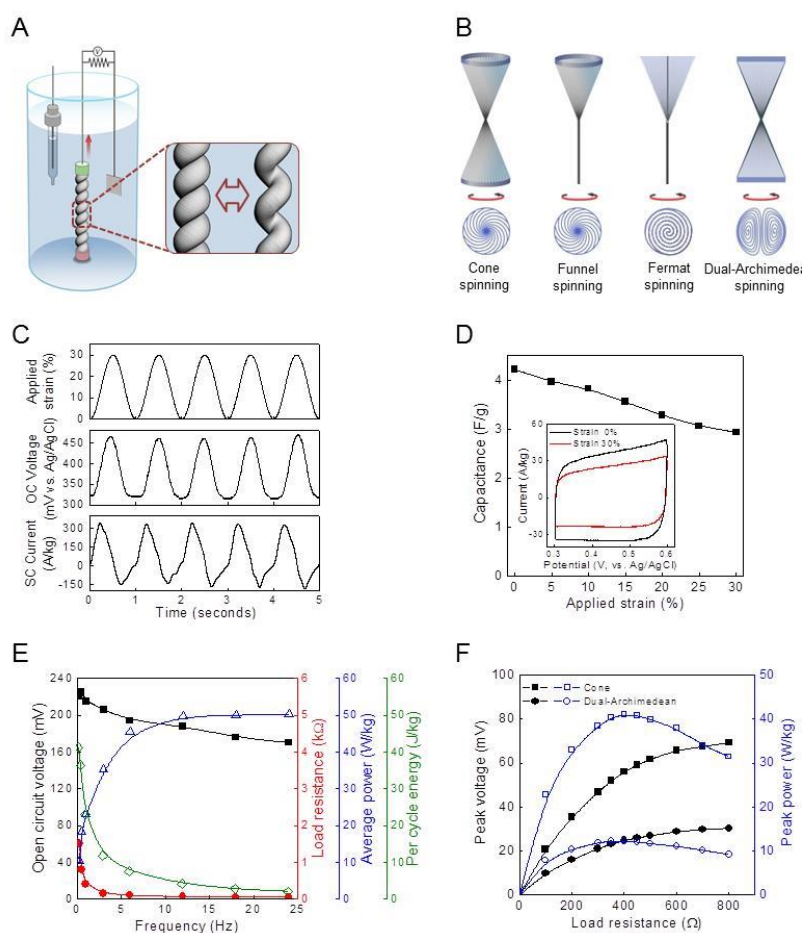


Figure 1 Twistron harvester configuration, structure and performance for tensile energy harvesting in 0.1 M HCl electrolyte. (A) Illustration of a coiled twistron harvester electrode, a Pt mesh/CNT buckypaper counter electrode, and a Ag/AgCl reference electrode in an electrochemical bath. The images on the right illustrate the configurations of the coiled CNT yarn electrode before and after stretch during energy harvesting. (B) Schematic illustration of cone, funnel, Fermat, and dual-Archimedean spinning (top) and the cross sections of the resulting yarns (bottom). (C) The time dependence of a sinusoidal applied tensile strain (up to 30%) (top) and the resulting change in open circuit (OC) voltage (middle) and short circuit (SC) current (bottom) for a 2-cm-long, coiled twistron harvester having a spring index of 0.43, which was made from a 54- μm -diameter twisted carbon nanotube yarn produced by cone spinning. (D) The dependence of capacitance on

applied strain for the tensile harvester of (C). The inset shows the CV curve for 0% strain and 30% strain. (E) The frequency dependence of OC voltage (closed black square), load impedance (closed red circle), average electrical power (open blue triangle), and electrical energy per cycle (open green diamond) for the application of 50% strain to the harvester of (C). (F) Comparison of the dependence of peak voltage (black symbols) and peak power (blue symbols) on load impedance for the cone-spun harvester of (C) and for a dual-Archimedean-spun harvester having identical dimensions. A 1 Hz stretch to 30% strain was used.

This need for high applied voltages for mechanical energy harvesting using a dielectric capacitor poses practical problems. By stretching a twistron carbon nanotube supercapacitor electrode in salt water or other electrolytes, we surprising discovered that we can generate high electrical power output and high electrical energy per mechanical cycle without the need to externally apply a bias voltage. Our explanation for this surprising result is that the chemical potential difference between the electrode surface and the surrounding electrolyte results in either electron or hole injection into the electrode. As a result, simply immersing an electrode into an electrolyte generates an equilibrium charge state on the electrodes, which can be used for harvesting mechanical energy.

In order the measure the equilibrium charge state of a CNT yarn harvester, it is necessary to know its potential of zero charge (pzc). Traditionally, direct measurement of the pzc has been difficult, and the results are often inaccurate. For our NBIT III work, we developed a method to determine the electrochemical pzc, which utilizes the charge-state-dependent response of a CNT electrode to mechanical deformation. This method, hereafter referred to as piezoelectrochemical spectroscopy (PECS), is performed by characterizing an electrode by cyclic voltammetry (CV) while simultaneously applying a sinusoidal mechanical deformation to the electrode. By comparing this CV to a baseline scan without deformation,

the alternating current (ac current) generated by the electrode can be determined as a function of applied voltage. This is shown in Fig. 2, which overlays a CV scan taken at 0% strain with a CV taken during 5 Hz sinusoidal stretching between 0 and 10% strain. Using a lock-in technique, the magnitude and phase of the ac current with respect to the sinusoidal mechanical excitation are calculated as a function of voltage. From this relationship, the pzc is found at the potential of minimum ac current. This is further supported by the phase of the ac current with respect to the sinusoidal mechanical excitation. This phase inverts by 180° at the pzc, which is consistent with the yarn having positive net charge at potentials above the pzc and negative charge below the pzc.

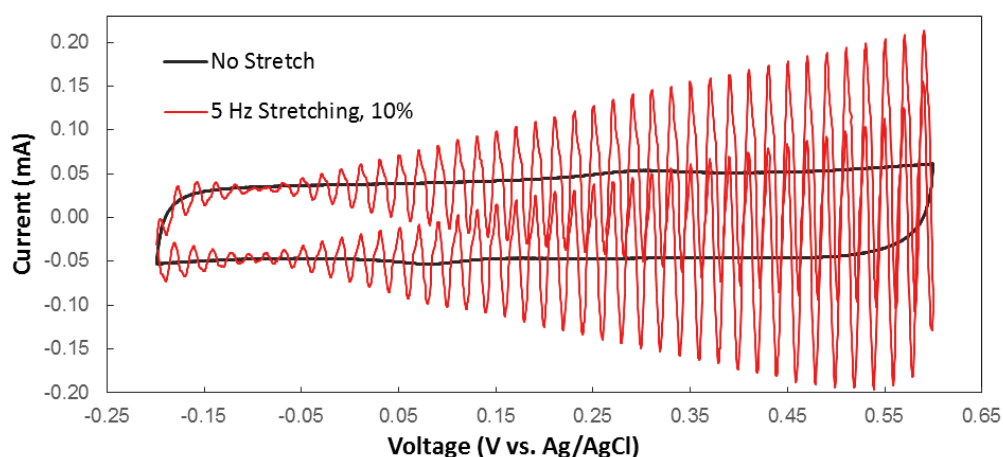


Fig 2. Measuring the potential of zero charge (pzc) by piezoelectrochemical spectroscopy (PECS). Cyclic voltammograms (taken at 100 mV/s in 0.1 M HCl) for a coiled twistron harvester are overlaid for the non-stretched state and during 5 Hz sinusoidal stretching by 10%. The pzc occurs when the stretch-induced oscillations in current are at a minimum (around -0.1 V)

Though we can usefully operate our twistron harvesters in salt water (or sea water for potential energy harvesting of the energy in ocean waves), without applying a voltage bias, higher performance was obtained by using 0.1 M HCl as the electrolyte (since this electrolyte resulted in higher equilibrium charge injection in the electrodes). Using 0.1 M HCl as the electrolyte and a sinusoidal mechanical stretch of 50% at a frequency of 12 Hz, we obtained a peak output power and average output power of 170 W/kg and 50 W/kg, respectively, at a peak-to-peak open circuit voltage of 0.24 V. By operating N identical twistron harvesters mechanically in parallel, and electrically in series, this output voltage increases linearly with N .

We discovered that the performance of our twistron harvesters can be increased by the following process. First, thermally annealing the coiled twistron harvesters for few seconds in vacuum at ~ 2500 °C, like we have described for our patent pending incandescent tension anneal process (ITAP), decreases the pzc by ~ 170 mV. This increases the chemical potential difference between the yarn and an acidic electrolyte, and consequently increasing the bias voltage on the harvester and the amount of hole injection. As shown by the results in Fig. 3D, this increased charge injection increases the open circuit voltage during mechanical energy harvesting, thereby increasing the output electrical power. The origin of this major change in pzc it is likely due to removal of well-known carbonaceous impurities on the nanotube surfaces, which affect the pzc.

Another major performance advance resulted from our discovery that yarn resistance was importantly contributing to twistron resistance. Consequently, twistron performance was improved by providing electrical connections to both ends of the twistron harvester. Even

higher performance (250 W/kg peak power) resulted from coiling a 23 μm platinum wire within the coiled harvester (Fig. 3A). We have not yet characterized the tensile harvester characteristics that we obtain when we take advantage of both the performance increases resulting from ITAP and coiling a Pt wire within the coiled twistrion yarn.

Figure 3, B and C, compare the presently realized performance of our tensile twistrion harvesters with that of alternative technologies, some of which have had decades or centuries to mature. Only sheet-based dielectric rubber harvesters, which require high bias voltages, provide a higher specific energy per cycle and a higher specific power. Moreover, for stretch frequencies of over 1 Hz, no other materials-based harvesting technology provides a higher specific energy per cycle or a higher specific power than are twistrion harvesters, which need no externally applied bias voltage.

A joint invention disclosure with Hanyang University has been filed at the University of Texas at Dallas. It has been agreed informally (a formal agreement is in progress) that the costs of provisional and PCT patent filings, as well as licensing revenues, will be equally shared between the two universities. Each university would be responsible for the costs of patent filing in their own country. A provisional patent filing will be made before the proposed publication of our work on twistrion harvesters in *Science*.

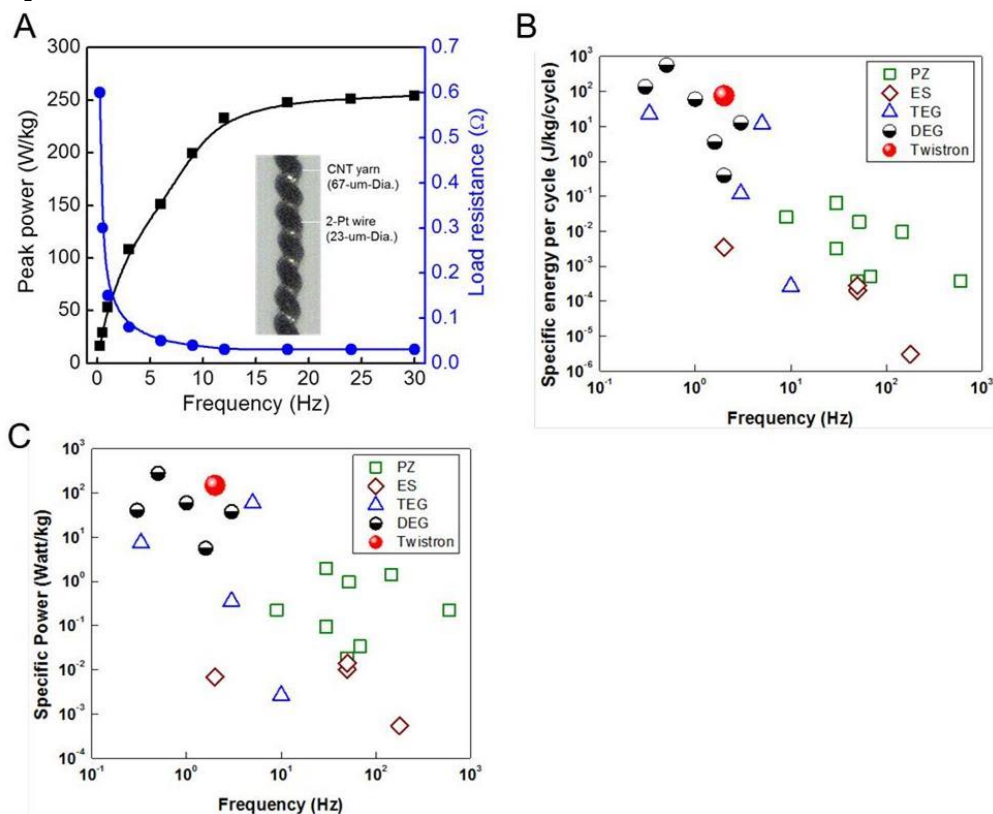


Figure 3. Improved performance twistrion harvester and performance comparisons with that for previously known materials-based harvesters. (A) The frequency dependence of peak power and optimal load resistance resulting from incorporation of a Pt current collector in a coiled twistrion yarn. Comparison for different frequencies of mechanical deformation of the specific energy per cycle (B) and the specific peak power (C) for the tensile torsional harvester with that for piezoelectric (PZ) generators, electrostatic (ES) generators, triboelectric generators (TEG), and dielectric-elastomer generators (DEG). The plots of (C) and (D) show the performance of our harvesters before the advance shown in (A) which increased our maximum power density from 150 W/kg to 250 W/kg.

List of Project Publications:

A. Publications in peer-reviewed journals

1. "New Twist on Artificial Muscles", C. S. Haines, Na Li, G. M. Spinks, A. E. Aliev, J. Dia, R. H. Baughman, *Proceedings National Academy of Sciences* **113**, 11709-11716 (2016), Selected as journal front cover.
2. "Stretchable Triboelectric Fiber for Self-powered Kinematic Sensing Textile", H. J. Sim, C. Choi, S. H. Kim, K. M. Kim, C. J. Lee, Y. T. Kim, X. Lepro, R. H. Baughman, S. J. Kim, *Scientific Reports*, 6:35153 (October 2016)
3. "Ultraviolet-induced irreversible tensile actuation of diacetylene/nylon microfibers", K. Y. Chun, C. Choi, R. H. Baughman, S. J. Kim, *Smart Materials and Structures* | 25 : 075031 | DOI:10.1088/0964-1726/25/7/075031.
4. "Tensile actuators of carbon nanotube coiled yarn based on polydiacetylene-pluronic copolymers as temperature indicators", H. U. Lee, H. Kim, K. Y. Chun, C. H. Kwon, M. D. Lima, R. H. Baughman, S. J. Kim, *Smart Materials and Structures* |25:075021| DOI:10.1088/0964-1726/25/7/075021.
5. "Bio-inspired hybrid carbon nanotube muscles", T. H. Kim, C. H. Kwon, C. Lee, J. An, T. T. Phuong, S. H. Park, M. D. Lima, R. H. Baughman, T. M. Kang, S. J. Kim, *Scientific Reports* | 6 : 26687 | DOI: 10.1038/srep26687.
6. "Woven yarn thermoelectric textiles", J. A. Lee, A. E. Aliev, J. S. Bykova, M. J. de Andrade, D. Kim, H. J. Sim, X. Lepro, A. A. Zakhidov, J.-B. Lee, G. M. Spinks, S. J. Kim, and R. H. Baughman, *Advanced Materials* **28**, 5038-5044 (2016)
7. "Bio-inspired, moisture-powered hybrid carbon nanotube yarn", S. H. Kim, C. H. Kwon, K. Park, T. J. Mun, X. Lepró, R. H. Baughman, G. M. Spinks, and S. J. Kim, *Scientific Reports* 6, 23016; doi: 10.1038/srep23016 (2016).
8. "Mediator-free carbon nanotube yarn biofuel cell", C. H. Kwon, Y. B. Park, J. A. Lee, Y. B. Choi, H. H. Kim, M. D. Lima, R. H. Baughman, S. J. Kim, *Royal Society of Chemistry Advances* **6**, 48346-48350 (2016).
9. "Highly stretchable hybrid nanomembrane supercapacitor", K. J. Kim, J. A. Lee, M. D. Lima, R. H. Baughman, S. J. Kim, *Royal Society of Chemistry Advances* **6**, 24756-24759 (2016).
10. "Carbon nanotube yarn-based glucose sensing artificial muscle", J. Lee, S. Ko, C. H. Kwon, M. D. Lima, R. H. Baughman, S. J. Kim, *Small* **12**, 2085-2091 (2016), selected as a cover for hard cover version.
11. "Temperature-responsive tensile actuator based on multi-walled carbon nanotube yarn", H. Kim, J. A. Lee, H. J. Sim, M. D. Lima, R. H. Baughman, S. J. Kim, *Nano-Micro Letters* **8**, 254-259 (2016).
12. "Biothermal sensing of a torsional artificial muscle", S.-H. Lee, T. H. Kim, M. D. Lima, R. H. Baughman, and S. J. Kim, *Nanoscale* **8**, 3248-3253 (2016).
13. "Elastomeric and dynamic MnO₂/CNT core-shell structure coiled yarn supercapacitor", C. Choi, H. J. Sim; G. M. Spinks, X. Lepró; R. H. Baughman, S. J. Kim, *Advanced Energy Materials* **6**, 1502119 (8 pages), 2016, selected as back cover.
14. "Harvesting temperature fluctuations as electrical energy using torsional and tensile polymer muscles", S. H. Kim, M. D. Lima, M. E. Kozlov, C. S. Haines, G. M. Spinks, S. Aziz, C. Choi, H. J. Sim, X. Wang, H. Lu, D. Qian, J. D. W. Madden, R. H. Baughman and S. J. Kim, *Energy & Environmental Science* **8**, 3336-3344 (2015).
15. "Stability of carbon nanotube yarn biofuel cell in human body fluid", C. H. Kwon, J. A. Lee, Y.-B. Choi, H.-H. Kim, G. M. Spinks, M. D. Lima, R. H. Baughman, S. J. Kim, *Journal of Power Sources* **286**, 103-108 (2015).
16. "High performance electrochemical and electrothermal artificial muscles from twist-spun carbon nanotube yarn", J. A. Lee, R. H. Baughman and S. J. Kim, *Nano Convergence*

2015, 2:8 DOI:10.1186/s40580-014-0036-0.

17. "Stretchable, weavable coiled carbon nanotube/MnO₂/polymer fiber solid-state supercapacitors", C. Choi, S. H. Kim, H. J. Sim, J. A. Lee, A. Y. Choi, Y. T. Kim, X. Lepro, G. M. Spinks, R. H. Baughman, S. J. Kim, *Scientific Reports* | 5 : 9387 | DOI: 10.1038/srep09387 (2015).
18. "Flexible, stretchable and weavable piezoelectric fiber", H. J. Sim, C. Choi, J. Lee, Y. T. Kim, G. M. Spinks, M. D. Lima, R. H. Baughman and S. J. Kim, *Advanced Engineering Materials* **17**, 1270-1275 (2015).
19. "Torsional behaviors of polymer-infiltrated carbon nanotube yarn muscles by atomic force microscope", C. H. Kwon, K. Chun, S. H. Kim, J. Lee, J. Kim, M. D. Lima, R. H. Baughman and S. J. Kim, *Nanoscale* **7**, 2489-2496 (2015).
20. "High power biofuel cell textiles from woven biscrolled carbon nanotube yarns", C. H. Kwon, S.-H. Lee, Y.-B. Choi, J. A. Lee, S. H. Kim, H.-H. Kim, G. M. Spinks, G. G. Wallace, M. D. Lima, M. E. Kozlov, R. H. Baughman, S. J. Kim, *Nature Communications*, 5:3928 DOI: 10.1038/ncomms4928 (2014).
21. "All solid state carbon nanotube torsional and tensile artificial muscles", J. A. Lee, Y. T. Kim, G. M. Spinks, D. Suh, X. Lepró, M. D. Lima, R. H. Baughman and S. J. Kim, *Nano Letters* **14**, 2664-2669 (2014).
22. "Artificial muscles from fishing line and sewing thread", C. S. Haines, M. D. Lima, Na Li, G. M. Spinks, J. Foroughi, J. D. W. Madden, S. H. Kim, S. Fang, M. J. de Andrade, F. Göktepe, Ö. Göktepe, S. M. Mirvakili, S. Naficy, X. Lepró, J. Oh, M. E. Kozlov, S. J. Kim, X. Xu, B. J. Swedlove, G. G. Wallace, R. H. Baughman, *Science* **343**, 868-872 (2014).
23. "Hybrid carbon nanotube yarn artificial muscle inspired by spider silk dragline", K.-Y. Chun, S. H. Kim, J. Park, M. K. Shin, C. H. Kwon, Y. T. Kim, G. M. Spinks, R. H. Baughman, S. J. Kim, *Nature Communications* | 5:3322 | DOI: 10.1038/ncomms4322 | www.nature.com/naturecommunications (2014).
24. "Flexible supercapacitor made of carbon nanotube yarn with internal pores", C. Choi, J. A. Lee, A. Y. Choi, Y. T. Kim, X. Lepró, M. D. Lima, R. H. Baughman, S. J. Kim, *Advanced Materials* **26**, 2059-2065 (2014).
25. "Conductive functional biscrolled polymer and carbon nanotube yarns", S. H. Kim, H. J. Sim, M. K. Shin, A. Y. Choi, Y. T. Kim, M. D. Lima, R. H. Baughman, S. J. Kim, *Royal Society of Chemistry Advances* **3**, 24028-24033 (2013).
26. "Ultrafast charge and discharge biscrolled yarn supercapacitors for textiles and microdevices", J. A. Lee, M. K. Shin, S. H. Kim, H. U. Cho, G. M. Spinks, G. G. Wallace, M. D. Lima, X. Lepro, M. E. Kozlov, R. H. Baughman, S. J. Kim, *Nature Communications* | 4:1970 | DOI: 10.1038/ncomms2970 | (2013).
27. "Free-standing nanocomposites with high conductivity and extensibility", K.-Y. Chun, S. H. Kim, M. K. Shin, Y. T. Kim, G. M. Spinks, A. E. Aliev, R. H. Baughman, and S. J. Kim, *Nanotechnology* **24**, 165401 (9 pp) (2013).

B. Accepted Project Manuscripts

1. Weavable Superelastic Biscrolled Yarn Supercapacitors with Record Power and Energy Densities", C. Choi, K. Min Kim, K. J. Kim, Xavier Lepró, G. M. Spinks, R. H. Baughman, S. J. Kim, *Nature Communications*, provisionally accepted (2016).
2. "Magnetic torsional actuation of carbon nanotube artificial muscle", D. Lee, S. Kim, M. Kozlov, X. Lepró, R. H. Baughman, S. J. Kim, *Smart Materials and Structures*, accepted (2016).

C. Project Manuscripts in Review

"Twistable and Stretchable Sandwich Structured Fiber for Wearable Sensors and Supercapacitors", C. Choi, J. M. Lee; S. H. Kim, J. Di, R. H. Baughman, S. J. Kim, *Nano Letters*.

Patent Activity Resulting from NBIT III Work

1. A joint HU/UTD provisional patent filing on NBIT III twistron harvesters will be filed in 2016 or early 2017, before our public disclosure of this technology (possibly in a manuscript that is being drafted for publication in *Science*). A patent record at UTD has been filed (UTD 1703), and signed by all co-inventors (HU and UTD).
2. A provisional patent filing has been made by UTD on "Twisted, Plied, Uniformly Coiled, and Non-Uniformly Coiled Artificial Muscles for Textile Applications". Conversion to PCT filing will occur before 03/21/2017.

Conference Presentations on NBIT III Work

"Artificial Muscles from Fishing Line and Sewing Thread", 9th Annual International Electromaterials Symposium, University of Wollongong (Feb. 12-14, 2014, Wollongong, Australia).

"Artificial Muscles for Fun and Profit", Nanotechnology Materials and Devices Workshop 2014 (Feb. 24-25, 2014, University of Cincinnati, Cincinnati, Ohio).

"The Evolution of Strong, Fast, Powerful, Durable, and Cheap Polymer Artificial Muscles from Carbon Nanotube Muscles", *Stuttgart NanoDays Workshop* (September 17-19, Stuttgart, Germany, 2014).

"The Evolution of Strong, Fast, Powerful, Durable and Cheap Polymer Muscles from Carbon Nanotube Muscles", *Materials Research Society National Meeting* (April 21-25, 2014, San Francisco).

"The Evolution of Strong, Fast, Powerful, Durable and Cheap Polymer Muscles from Carbon Nanotube Muscles", 11th *International Workshop on Piezoelectric Materials and Applications in Actuators* (Sept. 22-25, 2014, Suzhou, China).

"Biscrolled Multifunctional Nanofiber Yarns for Energy Applications", 10th *Annual International Electromaterials Science Symposium* (University of Wollongong, Wollongong, Australia, Feb. 13, 2015).

"Powerful, Giant-Stroke Artificial Muscles from Twisted and Coiled Carbon Nanotube Yarns", *International Winterschool on Electronic Properties of Novel Materials* (Kirchberg, Austria, March 12, 2015).

Strong, Powerful, Lightweight, Nanotube, and Polymer Muscles for Actuation, and Energy Harvesting for Air and Spacecraft", *Northrop Grumman Nanotechnology Workshop* (Redondo Beach, California, July 6, 2015).

"Powerful Artificial Muscles for Morphing Composites", 20th *International Conference on Composite Materials* (Copenhagen, Denmark, July 22, 2015).

"Powerful, Giant-Stroke Artificial Muscles From Twisted and Coiled Carbon Nanotube Yarns

and Polymer Fibers”, *XXIV International Materials Research Congress 2015* (Cancun, Mexico, August 19, 2015).

“Harvesting Waste Chemical and Thermal Energy Using Carbon Nanotube Yarn and Polymer Fiber Muscles”, *BAMN 2015: Biomimetics, Artificial Muscle, and Nano-Bio* (Vancouver, Canada, August 24-26, 2015).

“Harvesting Waste Chemical and Thermal Energy Using Carbon Nanotube Yarn and Polymer Fiber Muscles”, *10th Energy Harvesting Workshop* (Virginia Tech, Sept. 14, 2015).

“The Evolution of Strong, Fast, Powerful, Durable, and Cheap Polymer Artificial Muscles from Carbon Nanotube Muscles”, *Novel Materials – A Symposium of the National Academies of Sciences, Engineering, and Medicine* (National Academies of Sciences Building, Washington, D.C., Oct. 7, 2015).

“Powerful artificial muscles for morphing composites and other applications”, *Composites at Lake Louise-2015* (Alberta, Lake Louise, Canada, Nov. 8-12, 2015).

“Multifunctional Biscrolled CNT and Polymer Yarns for Energy Storing, and Energy Harvesting Textiles and Artificial Muscles”, *Materials Research Society Fall Meeting* (Boston, Massachusetts, Dec. 2, 2015).

“Strong, Powerful, Torsional, and Tensile, Artificial Muscles from Twisted and Coiled CNT Yarns”, *Materials Research Society Fall Meeting* (Boston, Massachusetts, Dec. 4, 2015).

“Environmentally Powered Yarn Arrays that Sense, Actuate, Harvest, and Store Energy”, *11th Annual International Electromaterials Science Symposium* (Deakin University, Melbourne, Australia, Feb. 10-12, 2016).

“Sheath-Core Conducting Fibers for Weavable Superelastic Wires, Biosensors, Supercapacitors, Strain Sensors, and Artificial Muscles”, Keynote Lecture at IUTAM (International Union of Theoretical and Applied Mechanics) Symposium on Mechanics of Stretchable Electronics), (Hanzhou, China, March 17 and 18, 2016)

“Sheath-Core Conducting Fibers for Weavable Superelastic Wires, Biosensors, Supercapacitors, Strain Sensors, and Artificial Muscles”, 7th Nanotechnology Materials and Devices Workshop (University of Dayton, Dayton, Ohio, May. 23-25, 2016).

“The Evolution of Strong, Fast, Powerful, Durable, and Cheap Polymer Artificial Muscles from Carbon Nanotube Muscles”, *5th International Conference on Smart and Multifunctional Materials, Structures, & Systems*” (*CIMTEC 2015*) (Perugia, Italy, June 5-9, 2016)

“Environmentally Powered Carbon Nanotube Yarns and Polymer Fibers that Sense, Actuate, Harvest, and Store Energy”, *The 24th International Conference on Science and Technology of Synthetic Metals* (Guangzhou, China, June 26-July 1, 2016).

Significant Collaborations Resulting from the AOARD project:

Collaboration is ongoing on our twistron mechanical energy harvesters with Benji Maruyama, Larry Drummy, and Matthew Lucas of AFRL, who will co-author a manuscript that will be submitted to *Science*.

Industrial Relationships

UTD has licensed part of our polymer muscle and carbon nanotube yarn technology to Lintec of America, who established a laboratory five miles from our UTD NanoTech Institute to commercialize our technologies. Important discussions with other major companies (Northrop Grumman Aerospace Systems, Flextronics, Toyobo, Kordsa Global, Samsung, LG Corporation, Under Armour, Kordsa Global, Toyobo, Arc'teryx, and VFC/NorthFace) have occurred and are occurring with our team.

Awards Enabled In-Part by Project Accomplishments

For their NBIT supported work on polymer artificial muscles, Seon Jeong Kim, Ray Baughman, and collaborators shared the R&D 100 Gold Award for Market Disruptor Product (2015) at a 1000 person gala in Los Vegas, which was one of the two highest awards presented. Seon Jeong's enabled awards include: 2016 Academic Award of Hanyang University, 2015 Award Certificate of Ministry of Science, ICT and Future Planning of Korea (MSIP), 2015 Award of 100 R&D of Ministry of Science, ICT and Future Planning of Korea (MSIP), and 2013 and 2013 Basic Research Awards of the National Research Foundation of Korea (NRF). In part because of NBIT supported discoveries, Ray was made a foreign member of the European Academy of Sciences (2015) and a Fellow of the National Academy of Inventors (2015). He also received the Tech Titans Technology Inventors Award (2015) at a gala in Dallas and the 2015 Inventor Award for Energy Harvesting Materials and Systems.

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