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**Morphological and Interfacial Effects in Aramid-Graphene Composites for
Structural Energy and Power**

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1. Accomplishments

1.1 Research Objectives

Here in this project, the team's long-term goal is to intimately understand, predict, and verify the major factors toward enhancing energy and mechanical properties for structural capacitors and batteries. In this two-year project, the team investigated the following objectives that contributed to this goal. The objectives included:

- Objective 1. Micromechanically investigate morphological control for higher performance in capacitors
- Objective 2. Experimentally quantify electrochemomechanical coupling for capacitors
- Objective 3. Determine the significance of the interphase between the aramid nanofibers and reduced graphene oxide sheets
- Objective 4. Demonstrate aramid nanofiber structural separators of controlled porosity for capacitors and batteries
- Objective 5. Tailor optimal combinations of mechanical and electrochemical performance by using Kevlar as the structural support.

1.2 Details of Accomplishments

In the following section, we provide key highlights on selected discoveries from the project in the areas of (1) structural batteries, (2) structural supercapacitors, (3) electrochemo-mechanical coupling, and (4) multifunctional efficiency modeling.

Structural Batteries

Structural batteries combine the energy storage properties of batteries with that of structural composites. Competing groups use carbon fibers as the structural matrix, but carbon fibers can have low energy densities and poor loading of the electrochemically active filler. Here, we used branched aramid nanofibers (BANFs) and reduced graphene oxide (rGO) nanosheets as the structural matrix for various electrochemically active fillers. ANFs/rGO matrices had never before been used as structural elements in battery electrodes. Key findings centered on the processing of these electrodes as well as the relationships among active material loading, electrochemical performance, and mechanical properties are discussed.

Conventional electrodes for batteries are optimized for high energy storage and suffer from poor mechanical properties. More specifically, commercial lithium-ion battery anodes and cathodes demonstrate tensile strength values < 4 MPa and Young's modulus of < 1 GPa. Here, we show that using branched aramid nanofibers (BANFs), or nanoscale Kevlar fibers, as a binder leads to mechanically stronger lithium-ion battery electrodes. BANFs are combined with lithium iron phosphate (LFP, cathode) or silicon (Si, anode) particles, and reduced graphene oxide (rGO), **Figure 1**. Hydrogen bonding interactions between rGO and BANFs are harnessed to accommodate load transfer within the nanocomposite electrodes. Overall, we obtained up to two orders of magnitude improvements in Young's modulus and tensile strength compared to commercial battery electrodes while maintaining good energy storage capabilities, **Figure 2**. This work demonstrates an efficient route for designing structural lithium-ion battery cathodes and anodes with enhanced mechanical properties using BANFs as a binder.

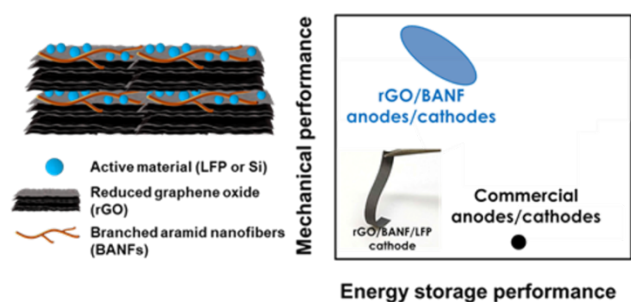


Figure 1. A schematic of the structural battery electrode containing silicon (Si) or lithium iron phosphate (LFP) active materials. These electrodes show significant improvements in stiffness compared to commercial anodes and cathodes.

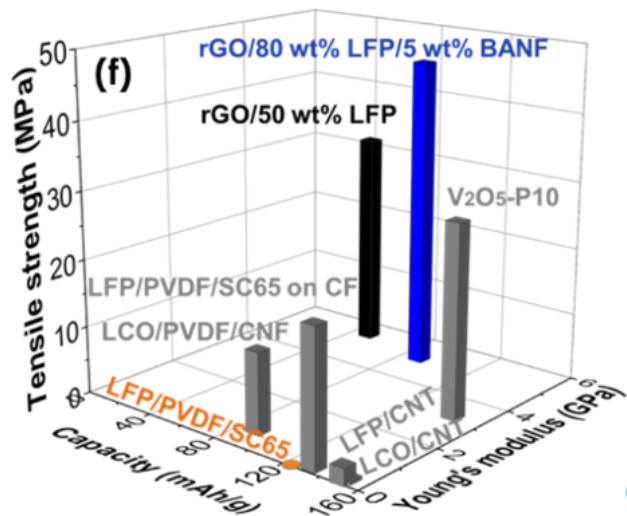


Figure 2. An Ashby plot showing the superior mechanical properties of the rGO/LFP/BANF structural cathode (blue) as compared to the conventional LFP/PVDF/SC65 cathode (orange).

Every battery requires that the anode and cathode do not touch to prevent short-circuiting and self-discharge. We developed structural separators from ANFs to replace the current polypropylene Celgard separators that are easily deformed. Thermogravimetric analysis shows a high 5 wt % decomposition temperature of 447 °C, which is over ~175 °C higher than commercial Celgard separators. The ANF separator also possesses a high Young's modulus of 8.8 GPa, which is ~1000% higher than commercial separators, **Figure 3**. Even when dry or when soaked in battery electrolyte, the ANF separators self-extinguish upon exposure to flame, whereas commercial separators melt or drip. We show that these features, although adventitious, present a trade-off with electrochemical performance in which a lithium nickel manganese cobalt (NMC) oxide-based battery possessed a capacity of 123.4 mA h g⁻¹.

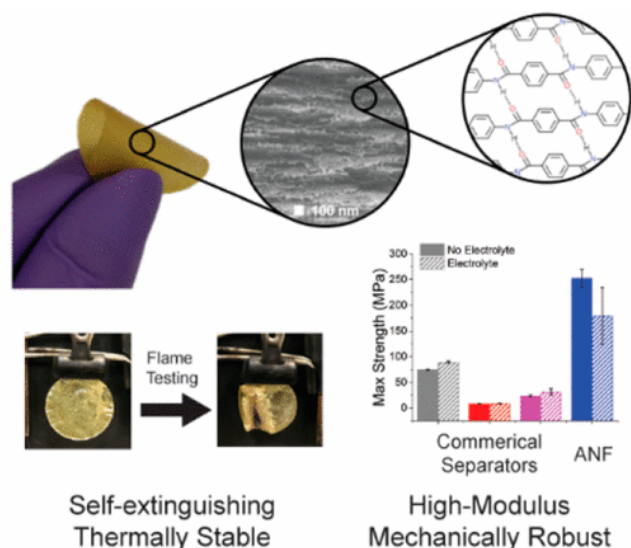


Figure 3. Aramid nanofiber battery separators possess mechanical properties that far exceed that of commercial separators, and they self-extinguish upon exposure to flame.

Key papers:

Flouda, P., Oka, S., Loufakis, D., **Lagoudas**, D.C., & **Lutkenhaus**, J.L., Structural Lithium-Ion Battery Cathodes and Anodes Based on Branched Aramid Nanofibers. *ACS Applied Materials & Interfaces* 13 (29), 34807-34817 (2021). doi:10.1021/acsami.1c06413

Patel, A., Wilcox, K., Li, Z., George, I., Juneja, R., Lollar, C., Lazar, S., Grunlan, J., Tenhaeff, W.E., & **Lutkenhaus**, J.L., High Modulus, Thermally Stable, and Self-Extinguishing Aramid Nanofiber Separators. *ACS Applied Materials & Interfaces* 12 (23), 25756-25766 (2020). doi: 10.1021/acsami.0c03671

Structural Supercapacitors

Structural supercapacitors combine the energy storage properties of capacitors with that of structural composites. These differ from batteries in that supercapacitors store charge in an electrical double layer or at a surface where batteries store charge through an electrochemical reaction throughout the volume of an active material. Competing groups have used carbon fibers as the structural matrix and the surface for the electrical double layer, but carbon fibers can have low energy densities because of the extremely low surface area. Here, we used branched aramid nanofibers (BANFs) and reduced graphene oxide (rGO) nanosheets as both structural matrix and as the surface for charge storage by the electrical double layer in a high surface area platform. In this reporting period, we focus upon our new findings regarding interfacial engineering of the rGO/ANF interface and effect of electrode morphology.

We used tannic acid to create strongly bonded interfaces between rGO nanosheets and BANFs to create structural supercapacitors. Without interfacial modification, rGO nanosheets can slide past one another, resulting in a relatively lower modulus. Tannic acid bears numerous phenol groups that can hydrogen bond with both materials, leading to Young's moduli as high as 25 GPa (yellow rGO-TA/BANF/Fe³⁺ in **Figure 4c**). For comparison, rGO electrodes have a Young's modulus of about 5 GPa. This concept was also extended to polyaniline/rGO/BANF supercapacitors to leverage the pseudocapacitive nature of polyaniline, which led to a Young's modulus of 4 GPa and a specific capacitance of 245.4 ± 2.6 F/g. In comparison, the polyaniline-based structural supercapacitor displayed higher capacitance but lower stiffness than the tannic acid-modified rGO/BANF ones.

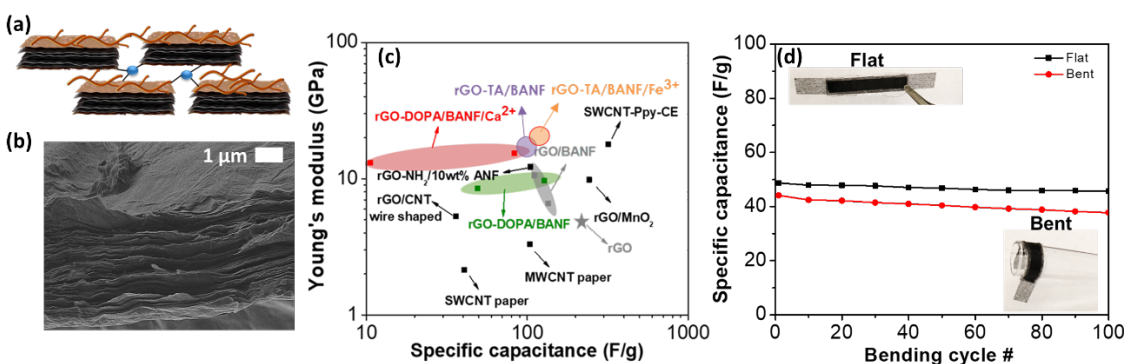


Figure 4. (a) Schematic representation and (b) cross-sectional scanning electron microscopy image of the structure of the tannic acid and Fe³⁺ modified electrodes. (c) Ashby plot of Young's modulus vs. specific capacitance. (d) Specific capacitance vs. scan rate for the prototype device in flat and bent states.

All the preceding electrodes possessed a dense brick-and-mortar morphology, which may hinder ion diffusion at high rates of discharge. To overcome this, we explored porous rGO/ANF aerogels prepared by hydrothermal reaction, **Figure 5**. Indeed, the aerogels were able to sustain higher specific capacitances at higher rates of discharge as compared to their dense, brick-and-mortar counterparts (i.e., 140 F/g vs 100 F/g, respectively).

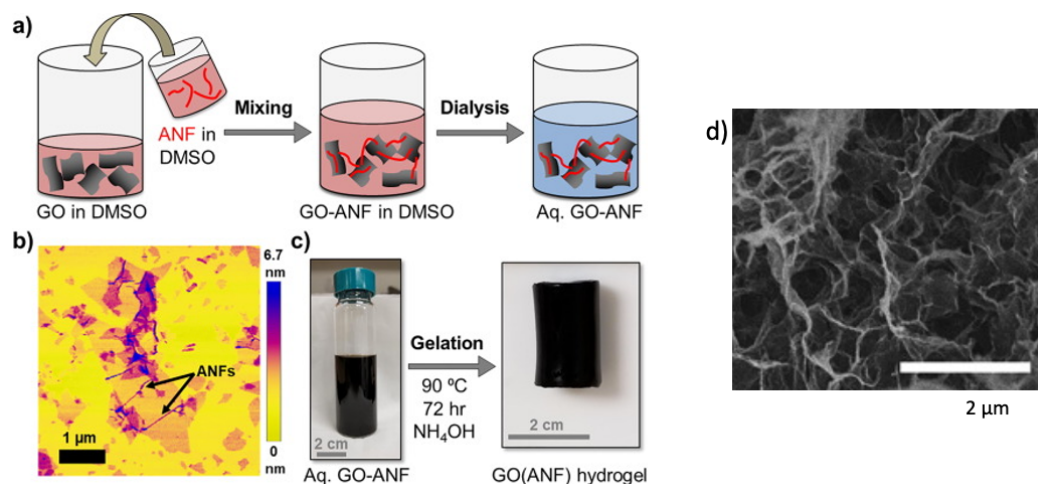


Figure 5. A schematic of the rGO/ANF aerogel preparation, along with AFM and SEM images.

Key papers:

Flouda, P., Yun, J., Loufakis, D., Shah, S.A., **Green, M.J., Lagoudas, D.C., & Lutkenhaus, J.L.**, Structural reduced graphene oxide supercapacitors mechanically enhanced with tannic acid. *Sustainable Energy & Fuels* 4 (5), 2301-2308 (2020). doi:10.1039/C9SE01299K

Flouda, P., Quinn, A.H., Patel, A.G., Loufakis, D., **Lagoudas, D.C., & Lutkenhaus, J.L.**, Branched aramid nanofiber-polyaniline electrodes for structural energy storage. *Nanoscale* 12 (32), 16840-16850 (2020). doi: 10.1039/D0NR04573J

Shah, S.A., Kulhanek, D., Sun, W., Zhao, X., Yu, S., Parviz, D., **Lutkenhaus, J.L., & Green, M.J.**, Aramid nanofiber-reinforced three-dimensional graphene hydrogels for supercapacitor electrodes. *Journal of Colloid and Interface Science* 560, 581-588 (2020). <https://doi.org/10.1016/j.jcis.2019.10.066>

Coupling of Mechanical and Electrochemical Properties

In both supercapacitors and batteries, charging and discharging can cause the evolution of mechanical stresses within the electrode. It is extremely challenging to observe such changes *in situ* due to the complexity of the working device. Here, we have built and created an instrument characterizes electrochemo-mechanical couplings *in-situ* on a single electrode using an electrochemical cell that is interfaced with a micro-tensile stage, **Figure 6**.

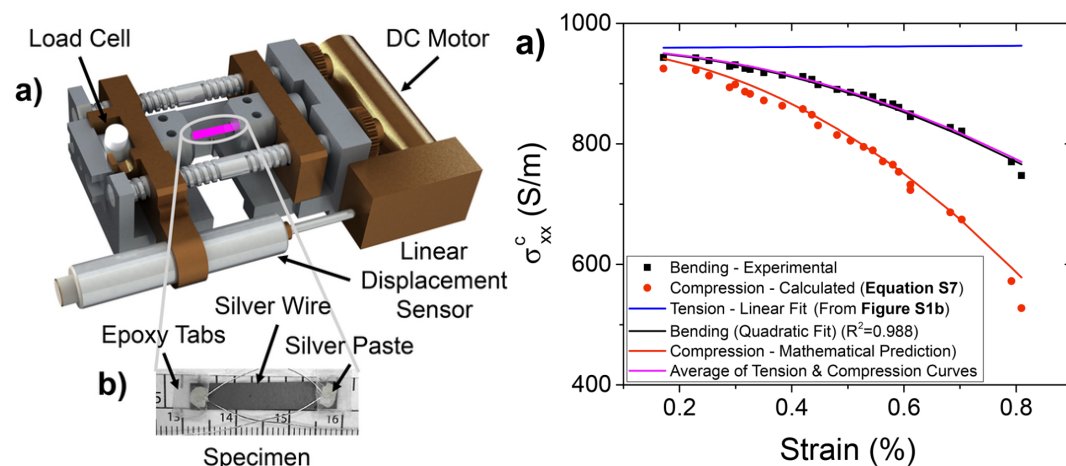


Figure 6. (Left) A schematic of the custom-built electrochemo-mechanical tester built by our team. (Right) Measured and calculated conductivities as a function of strain.

We first used this newly developed instrument to measure the conductivity of simple rGO electrodes under varying strain, allowing us to extract both the contributions of the bending and compression components to the conductivity using appropriate physical models. Our future work is now focused upon quantification of the developed stresses during electrochemical cycling.

Key paper and patent:

Loufakis, D., Boyd, J. G., Lutkenhaus, J. L. & Lagoudas, D. C. Experimental determination of the compressive piezoresistive response of a free-standing film with application to reduced graphene oxide. *Journal of Applied Physics* **131**, 035105, (2022). doi:10.1063/5.0063318

Loufakis, D., Lutkenhaus, J., Lagoudas, D.C., & Boyd, J.G., Submitted to U.S. Patent Office.

Energy-based Multifunctional Efficiency Metric for Multifunctional Devices and Materials

A new energy-based multifunctional efficiency (MFE) metric was developed using micromechanics solutions for structural supercapacitors consisting of composite electrodes that can store electrical energy and sustain mechanical loads. MFE metrics quantify the volume and/or mass savings when structural and functional materials are replaced by multifunctional materials and evaluate the trade-off between different functionalities. Commonly used multifunctionality metrics for structural supercapacitors are based on the rule of mixtures for both mechanical and electrical performance. These metrics provide an adequate approximation for some electrode geometries and loading conditions, such as longitudinal direction for aligned fibers in multifunctional composite electrodes and in-plane directions for laminate composite electrodes. However, if supercapacitors with complex microstructure or multiple electrode materials encompass more complex geometries or orientations of the structural and functional phases, a more comprehensive method is required to accurately capture the MFE. The MFE proposed accounts for complex geometries and different mechanical loading conditions by using micromechanics methods. The shapes considered here include layered composite supercapacitors, fibrous films and any shape that can be derived from an ellipsoid. When calculated utilizing the proposed metric, the MFE varies by orders of magnitude due to the difference in shapes and applied mechanical fields to the supercapacitors, while existing metrics provide a constant upper bound. The influence of Young's modulus difference between multifunctional electrodes and solid electrolytes is also discussed.

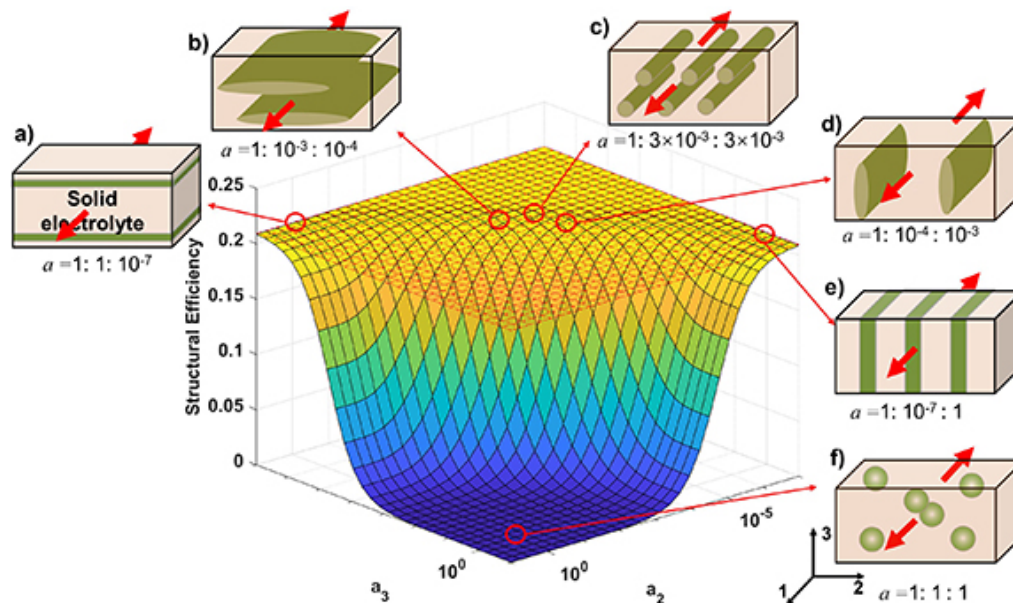


Figure 7. Structural efficiency of multifunctional electrode as a function of aspect ratio under uniaxial loading along axis 1. The multicolored gradient surface plot represents the results based on the new MFE metric. The red meshed plane is the result based on rule of mixtures. Variables a_2 and a_3 are in logscale.

Key paper:

Zhou, T., Dickinson, E., **Boyd, J.G.**, **Lutkenhaus, J.L.**, & **Lagoudas, D.C.**, Multifunctional efficiency metric for structural supercapacitors. *Multifunctional Materials* 3 (4), 044002 (2020). doi: 10.1088/2399-7532/abcd87

1.3 Dissemination of Results

For the duration of the project, 16 manuscripts were published in peer-reviewed journal and 22 conference presentations were given.

Publications:

2022

1. Loufakis, D., **Boyd, J. G., Lutkenhaus, J. L. & Lagoudas, D. C.** Experimental determination of the compressive piezoresistive response of a free-standing film with application to reduced graphene oxide. *Journal of Applied Physics* **131**, 035105, (2022). doi:10.1063/5.0063318

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2. Aderyani, S., Flouda, P., Shah, S.A., **Green, M.J., Lutkenhaus, J.L., & Ardebili, H.**, Simulation of cyclic voltammetry in structural supercapacitors with pseudocapacitance behavior. *Electrochimica Acta* **390**, 138822 (2021). <https://doi.org/10.1016/j.electacta.2021.138822>
3. Flouda, P., Oka, S., Loufakis, D., **Lagoudas, D.C., & Lutkenhaus, J.L.**, Structural Lithium-Ion Battery Cathodes and Anodes Based on Branched Aramid Nanofibers. *ACS Applied Materials & Interfaces* **13** (29), 34807-34817 (2021). doi:10.1021/acsmi.1c06413

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4. Flouda, P., Yun, J., Loufakis, D., Shah, S.A., **Green, M.J., Lagoudas, D.C., & Lutkenhaus, J.L.**, Structural reduced graphene oxide supercapacitors mechanically enhanced with tannic acid. *Sustainable Energy & Fuels* **4** (5), 2301-2308 (2020). doi:10.1039/C9SE01299K
5. Flouda, P., Quinn, A.H., Patel, A.G., Loufakis, D., **Lagoudas, D.C., & Lutkenhaus, J.L.**, Branched aramid nanofiber-polyaniline electrodes for structural energy storage. *Nanoscale* **12** (32), 16840-16850 (2020). doi: 10.1039/D0NR04573J
6. Shah, S.A., Kulhanek, D., Sun, W., Zhao, X., Yu, S., Parviz, D., **Lutkenhaus, J.L., & Green, M.J.**, Aramid nanofiber-reinforced three-dimensional graphene hydrogels for supercapacitor electrodes. *Journal of Colloid and Interface Science* **560**, 581-588 (2020). <https://doi.org/10.1016/j.jcis.2019.10.066>
7. Aderyani, S., Shah, S.A., Masoudi, A., **Green, M.J., Lutkenhaus, J.L., & Ardebili, H.**, Comparison of Nanoarchitecture to Porous Media Diffusion Models in Reduced Graphene Oxide/Aramid Nanofiber Electrodes for Supercapacitors. *Acs Nano* **14** (5), 5314-5323 (2020). doi: 10.1021/acsnano.9b07116
8. Patel, A., Wilcox, K., Li, Z., George, I., Juneja, R., Lollar, C., Lazar, S., Grunlan, J., Tenhaeff, W.E., & **Lutkenhaus, J.L.**, High Modulus, Thermally Stable, and Self-Extinguishing Aramid Nanofiber Separators. *ACS Applied Materials & Interfaces* **12** (23), 25756-25766 (2020). doi: 10.1021/acsmi.0c03671

9. **Lutkenhaus, J.L.** & Flouda, P., Structural batteries take a load off. *Science Robotics* 5 (45), eabd7026 (2020). doi:10.1126/scirobotics.abd7026
10. Patel, A., Loufakis, D., Flouda, P., George, I., Shelton, C., Harris, J., Oka, S., & **Lutkenhaus, J.L.**, Carbon Nanotube/Reduced Graphene Oxide/Aramid Nanofiber Structural Supercapacitors. *ACS Applied Energy Materials* 3 (12), 11763-11771 (2020). doi: 10.1021/acsaem.0c01926
11. Zhou, T., Dickinson, E., **Boyd, J.G.**, **Lutkenhaus, J.L.**, & **Lagoudas, D.C.**, Multifunctional efficiency metric for structural supercapacitors. *Multifunctional Materials* 3 (4), 044002 (2020). doi: 10.1088/2399-7532/abcd87

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12. Zhou, T., **Boyd, J.G.**, Loufakis, D., **Lutkenhaus, J.L.**, & **Lagoudas, D.C.**, Fabrication, characterization and micromechanics modeling of the electrical conductivity of reduced graphene oxide/aramid nanofiber nanocomposites. *Smart Materials and Structures* 28 (9), 094001 (2019). doi: 10.1088/1361-665x/ab2c39
13. Aderyani, S., Flouda, P., **Lutkenhaus, J.L.**, & Ardebili, H., The effect of nanoscale architecture on ionic diffusion in rGo/aramid nanofiber structural electrodes. *Journal of Applied Physics* 125 (18), 185106 (2019). doi: 10.1063/1.5087280
14. Sun, W., Shah, S.A., Lowery, J.L., Oh, J.H., **Lutkenhaus, J.L.**, & **Green, M.J.**, Lightweight Kevlar-Reinforced Graphene Oxide Architectures with High Strength for Energy Storage. *Advanced Materials Interfaces* 6 (21), 1900786 (2019). doi: 10.1002/admi.201900786
15. Patel, A. & **Lutkenhaus, J.L.**, Structural Energy and Power for Lightweight Vehicles and Powered Body Armor. *Homeland Defense & Security Information Analysis Center* 6 (2), 4-9 (2019).

https://www.hdiac.org/wp-content/uploads/2019/08/HDIAC_Journal_Volume_6_Issue_2-fixed-2.pdf
16. Flouda, P., Shah, S.A., **Lagoudas, D.C.**, **Green, M.J.**, & **Lutkenhaus, J.L.**, Highly Multifunctional Dopamine-Functionalized Reduced Graphene Oxide Supercapacitors. *Matter* 1 (6), 1532-1546 (2019). <https://doi.org/10.1016/j.matt.2019.09.017>

Conference Abstracts Submitted or Presented, 2019-2021

1. Structural lithium-ion battery cathodes based on organic redox-active polymers and Kevlar® aramid nanofibers, *Chemical Engineering Graduate Student Association Annual Symposium*, College Station, TX, USA, March 11, 2021
2. Structural lithium-ion battery cathodes based on organic redox-active polymers and Kevlar® aramid nanofibers, *Texas A&M Conference on Energy*, College Station, TX, USA, Dec 2-3, 2021

3. Energy-based Multifunctional Efficiency Metric for Multifunctional Composite Anodes in Structural Batteries, *American Society for Composites (ASC) Thirty-Sixth Technical Conference on Composite Materials*, Virtual, Sept 19-22, 2021
4. Interphase Influence on the Elastic Moduli of rGO and ANF Supercapacitor Electrodes, *Materials Research Society (MRS) Spring Meeting & exhibits*, Virtual, Apr 17-23, 2021
5. Experimental Electro-chemo-mechanical Coupling of Reduced Graphene Oxide Electrodes, *Texas A&M Conference on Energy*, College Station, TX, USA, Dec 2-3, 2021
6. Structural Lithium-Ion Battery Electrodes Based-on Branched Aramid Nanofibers, *2021 MRS Spring Meeting*, Virtual, April 17-23, 2021
7. Structural Lithium-Ion Battery Electrodes Reinforced with Kevlar Aramid Nanofibers, *ACS Spring 2021*, Virtual, April 5-16, 2021
8. Structural Nanocomposite Battery Electrodes Reinforced with Kevlar Aramid Nanofibers', *Texas A&M Polymer Technology Industrial Consortium Meeting*, Virtual, April 9th, 2021
9. Thermally Stable Aramid Nanofiber Separators, *Annual American Institute of Chemical Engineers Meeting*, Orlando, FL, USA, Nov 10-15, 2019
10. Thermally Stable Aramid Nanofiber Separators, *8th Annual Texas Soft Matter Meeting*, Lubbock, TX, USA, August 16, 2019
11. Comparison of Nanoarchitecture to Porous Media Diffusion Models in Reduced Graphene Oxide/Aramid Nanofiber Electrodes for Supercapacitors. *2019 IMECE, International Mechanical Engineering Congress & Exposition*, Salt Lake City, UT, Nov. 2019
12. Nanoarchitectural Modeling vs. Porous Media Approach, *UH-MRS*, Oct. 2019
13. The effect of nanoscale architecture on ionic diffusion in rGO/aramid nanofiber structural electrodes, *56th Semiannual TCSUH Student Symposium*, Houston, TX, USA, April 2019
14. Highly Multifunctional Dopamine Functionalized Reduced Graphene Oxide Structural Supercapacitors", *C3E 2019*, College Station, TX, USA, November 13-14, 2019
15. Nature-inspired Structural Supercapacitor Electrodes Based on Dopamine Functionalized Graphene, *Polymers for Advanced Technologies (PAT 2019)*, College Station, TX, USA August 8-10, 2019
16. *In-situ* Mechano-Electrochemical Coupling Measurements of Structural Supercapacitor Composite Electrodes, *Texas A&M Conference on Energy*, College Station, TX, Sept 2019
17. Electrical Resistance Response of Reduced Graphene Oxide - Aramid Nanofiber Films to Bending-Induced Strain, *235th ECS Meeting*, Dallas, TX, May 2019
18. Effects of Waviness and Discontinuous Interphase on the Multifunctional Aramid Nanofiber-Functionalized Reduced Graphene Oxide Electrodes, *European Congress and Exhibition on advanced materials and processes (EUROMAT)*, Stockholm, Sweden, Sept 1-5, 2019

19. Micromechanics Modeling on the Electrical Conductivity of Aramid Nanofiber-Functionalized Graphene Electrodes, *Conference on Smart Materials, Adaptive Structures and Intelligent Systems (SMASIS)*, Louisville, KY, USA, Sept 9-11, 2019
20. Electrical Resistance Response of Reduced Graphene Oxide - Aramid Nanofiber Films to Bending-Induced Strain *235th ECS Meeting*, Dallas, TX, USA, May 26-30, 2019
21. Thermally Stable Aramid Nanofiber Separators, *235th ECS National Meeting*, Dallas, TX, USA, May 26-30, 2019
22. Bioinspired Structural Supercapacitor Electrodes, *235th ECS National Meeting*, Dallas, TX, USA, May 26-30, 2019

2. Impacts

2.1 Development of the Principal Discipline of the Project

This project resulted in new components for structural batteries and capacitors that exhibited stiffnesses far higher than conventional components found in energy storage devices. To accomplish this, the team examined experimentally and theoretically a family of electrodes based upon high-strength nanomaterials: reduced graphene oxide nanosheets and aramid nanofibers. This resulted in first-ever demonstrations of battery electrodes containing these materials, as well as first models that captured the effect of material shape for multifunctional efficiency. Last, a new instrument was developed and constructed that could measure mechanical changes in the electrode during charging and discharging. Taken together, the new knowledge generated supports the long-term goal of creating batteries and capacitors with exceptional mechanical properties.

2.2 Other Disciplines Impacted

Besides the area of structural energy and power, this work impacts the disciplines of composites and multifunctional materials. This work has established the structure, morphology, and properties related to reduced graphene oxide/aramid nanofiber composites which was not known prior. Further, the theoretical concepts of electrochemomechanical coupling and multifunctional efficiency developed here may be extended to the broader discipline of multifunctional materials.

2.3 Impact in This Reporting Period on Human Resources

In this reporting period 2 undergraduate students and 5 graduate students were trained. Two of the graduate students have entered the US workforce, and another has continued on to a post-doc at Georgia Tech.

2.4 Impact on Teaching and Educational Experiences

None to report.

2.5 Impact on Physical, Institutional, and Information Resources that Form Infrastructure

None to report.

2.6 Impact on Society Beyond Technology

This work has highlighted the idea of unconventional batteries that could broadly impact electric vehicles, aerospace, and extreme environment applications. The composites disclosed here could be translated to other applications to afford high-stiffness materials with Kevlar-like properties.

3. Changes

3.1 Changes in Approach

None to report.

3.2 Problems or Delays

The team experienced delays related to COVID-19 (lab closures and reduced capacities), as well as Texas's February 2020 winter storm in which the power grid failed. This caused several pipes to break in the building housing the Green and Lutkenhaus labs, leading to severe damage and

loss of some equipment and extended time away from the office due to subsequent repairs and renovations.

3.3 Expenditure Impacts

Because of the delays outlined in Section 3.2, there were delays in expenditure. An extension was requested and granted.

3.4 Significant Changes in the Use or Care of Human Subjects, Vertebrate Animals, or Biohazards

None to report.

3.5 Changes to the Primary Place of Performance

None to report.