

Self-Sensing Fiber-Reinforced Composites

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Annual Merit Review

Project ID: mat173

ORNL is managed by UT-Battelle, LLC for the US Department of Energy

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Overview

Timeline

- Start Date: October 2019
- End Date: March 2021
- Percent Complete: 100%

Budget

- Total project funding
 - DOE: \$300k
 - Industrial cost share: \$150k
- Funding for FY21: \$0

Partners

- CRADA with Dronesat, LLC
- Project Lead: ORNL

Barriers and Technical Targets

- Critical Challenge for carbon fiber composites: “Joining, NDE, Life Monitoring and Repair”*
- “The ability to predict performance for material, joints, and parts would allow for optimized design while minimizing cost”*
- Structural health monitoring would help inform models for in-service performance prediction

*From “Materials: Materials Technical Team Roadmap” (2017), by U.S. DRIVE Partnership

Relevance

Impact

- Multifunctional composites with structural health monitoring offer:
 - Increased composite safety
 - Improved estimates of maintenance requirements
- The focus is on cargo transportation and infrastructure monitoring, it also opens other markets:
 - Automotive industry (electric automobile battery enclosures and compressed natural gas storage tanks)
 - Oil and natural gas distribution (composite pipelines and pipeline repairs)
 - Infrastructure repair (patches to concrete bridge pillars)
 - Military (unmanned aerial vehicles with extended flight times)
 - Space vehicles (cryogenic fuel tanks)

Objectives

- Use a roll-to-roll fiber processing method to adhere particles to the surface of different fibers
- Demonstrate a scalable, multifunctional composite with in-situ sensing capabilities as well as improved mechanical performance

Milestones

Milestone/Deliverable Name/Description	End Date	Status
Fabricate multifunctional composites and perform mechanical testing	12/31/2019	Complete
Characterize the active sensing capabilities of the multifunctional composites	6/30/2020	Complete
Synthesize fibers with passive sensing capabilities	9/30/2020	Complete
Fabricate a passive sensing, hybrid, multifunctional composite	12/31/2020	Complete
Characterize the power generation of the hybrid, multifunctional composite in response to strain	6/30/2021	Complete

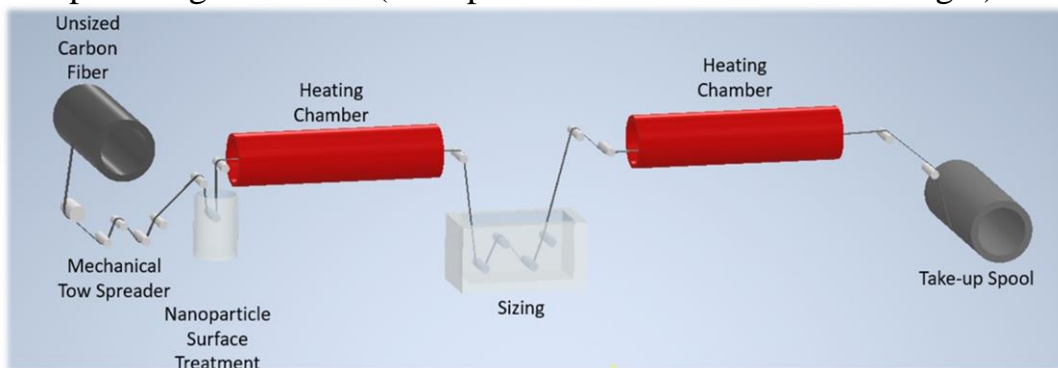
Go/No-Go Decisions

Name	Description	Date	Status
Passive Sensing, Hybrid, Multifunctional Composite	Develop and fabricate a fiber-reinforced composite with at least two different fibers that act as a self-powered sensor	12/31/2020	Complete

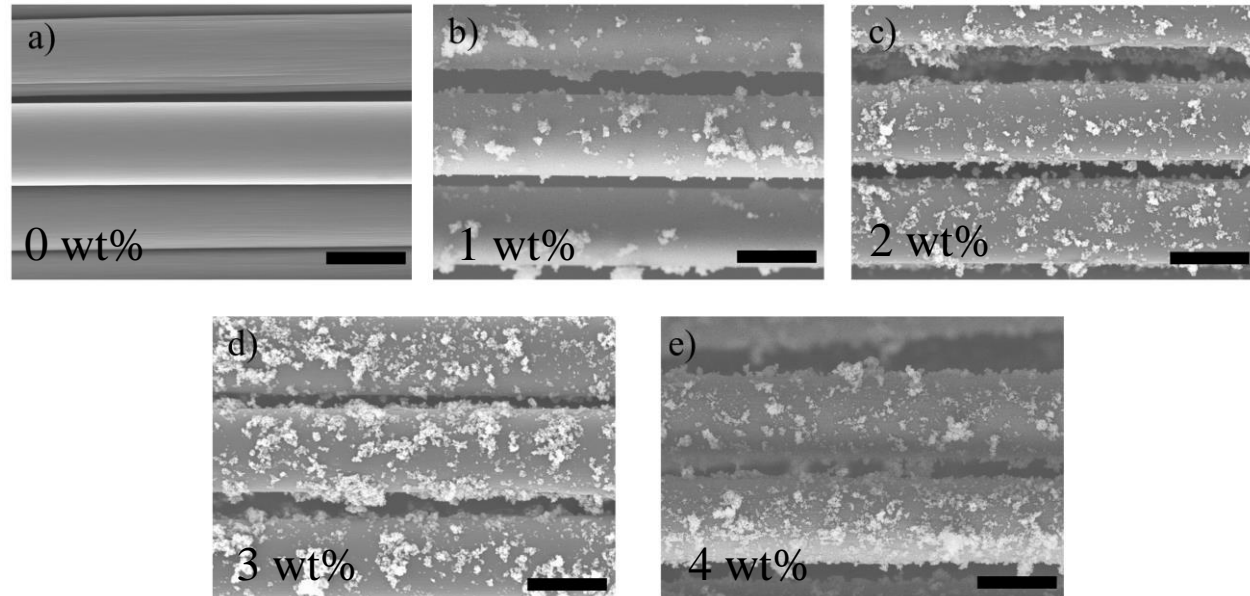
Approach

- Roll-to-roll dip coating deposition process to integrate nanoparticles into the sizing for improved mechanical strength and sensing functionality
 - Concentration of nanoparticles can be easily varied
 - Compatible with many different nanoparticles and fibers
- Use commercially available products combined in a mutually beneficial approach
- Commercially sourced products make scale-up feasible

Dip coating schematic (fiber processed from the left to the right)



TiO₂ nanoparticle (30 nm) coated carbon fiber

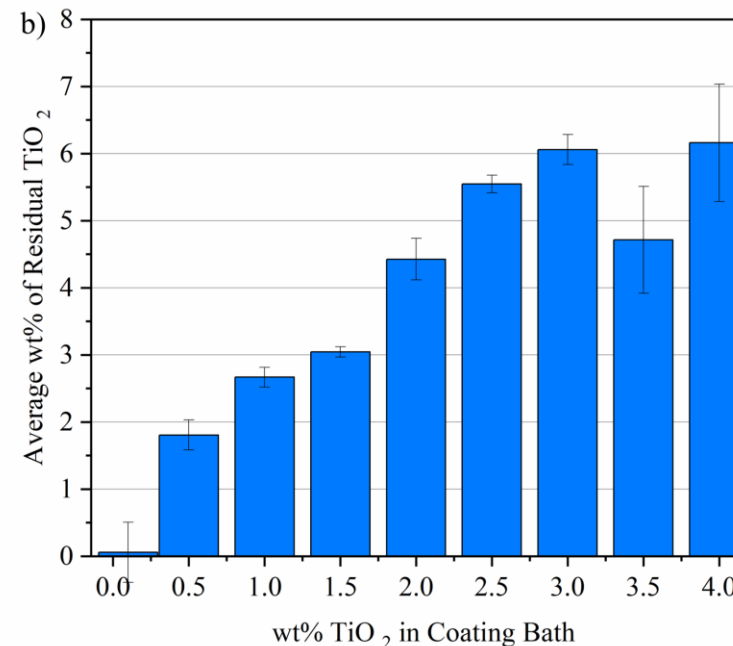
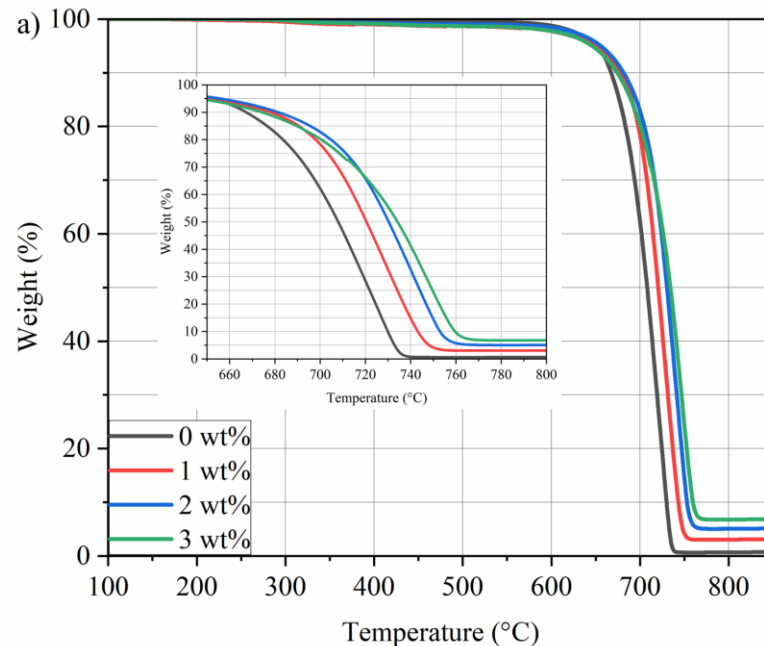


Scale bars are 5 μ m

Accomplishments

- The fiber naming convention is based on the TiO_2 concentration in the first dip coating bath
- Performed thermogravimetric analysis of the fibers to quantify the amount of nanoparticles as a function of the total weight of the fiber (not the total weight of the bulk composite)
- It is seen that we can reliably control the amount of nanoparticles on the surface until the concentration gets too high in the bath (3.5 and 4.0 wt% nanoparticles)
 - The standard deviation increases significantly at the higher concentrations and does not follow the trend of increased nanoparticle concentration deposited on the fiber surface

Thermogravimetric analysis



Control of nanoparticle concentration on the fiber surface was accomplished by modifying the coating bath concentration

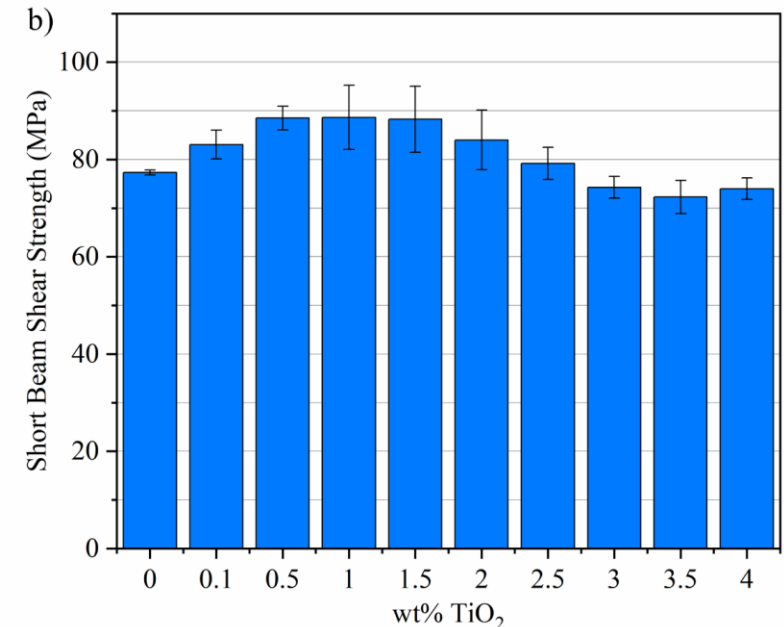
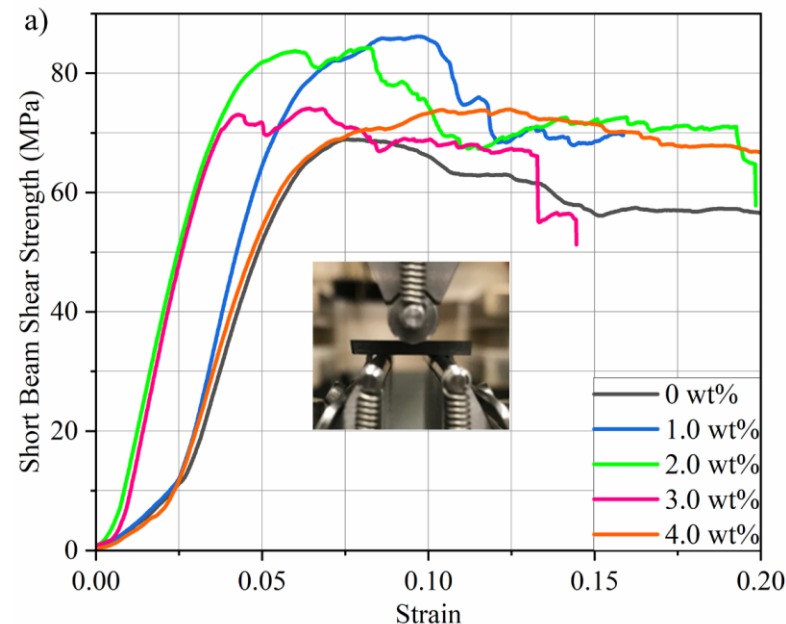
Accomplishments

Interlaminar Shear Strength Testing

- Unidirectional composites were fabricated using a filament winding technique
- Baseline: 77.3 MPa (0 wt% nanoparticles)
- Highest performing composite: 88.7 MPa (1 wt% nanoparticles)
- **Maximum increase of 14.7% increase in interlaminar shear strength, so milestone was achieved**

Milestone: Fabricate multifunctional composites and perform mechanical testing (12/31/2019)

Criteria: Short beam shear test should show an interlaminar shear strength improvement of at least 10%



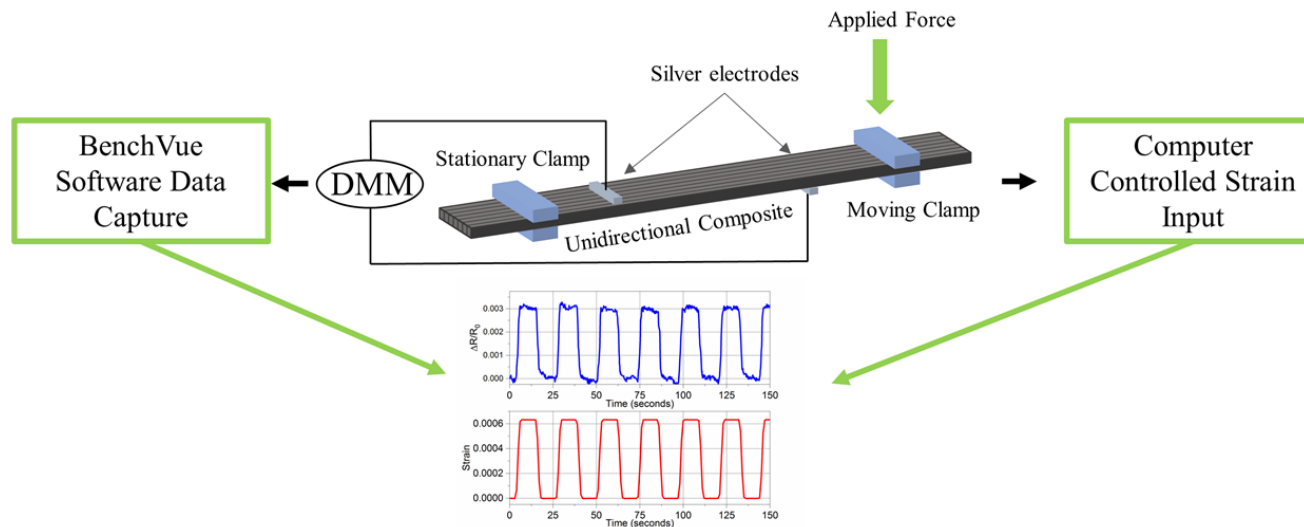
Interlaminar shear strength testing: a) representative stress-strain curves and b) average short-beam shear strength (Error bars signify one standard deviation)

Accomplishments

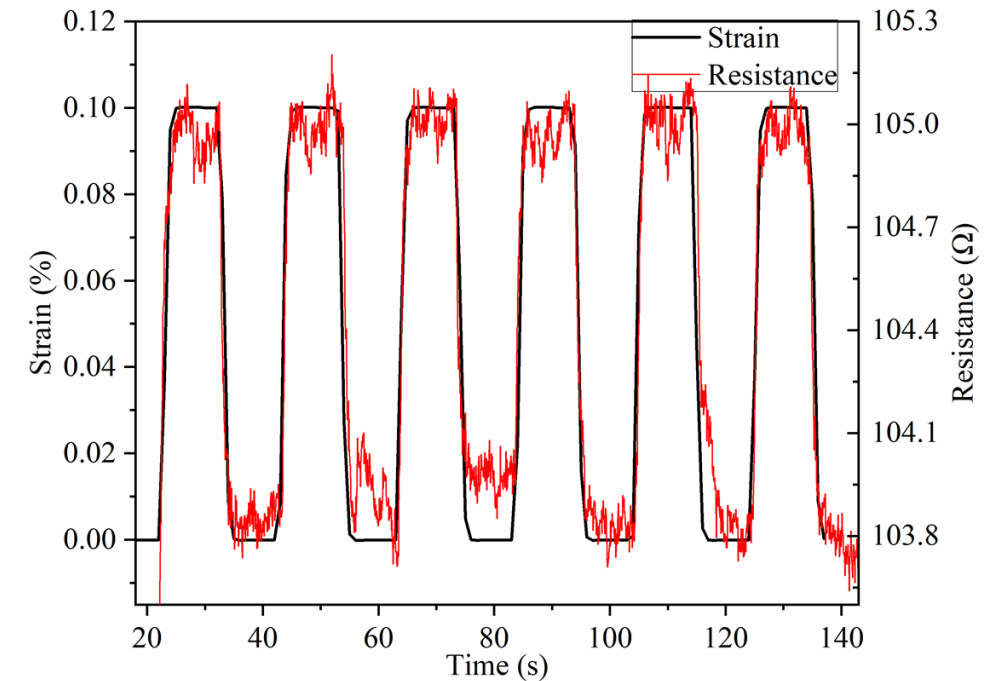
Structural Health Monitoring

- Out-of-plane through thickness electrical resistance was measured of a cantilevered beam during cyclic strain events
- Displacement controlled method using the dynamic mechanical analyzer in a single cantilever configuration
 - Displaced at 3 mm/sec
 - Held at displaced distance for 10 seconds
 - Returned to zero displacement
 - Held for 10 seconds
- Repeated for 10 cycles
- Recorded electrical resistance with a digital multimeter

Schematic of the test configuration



Representative recorded strain and resistance data

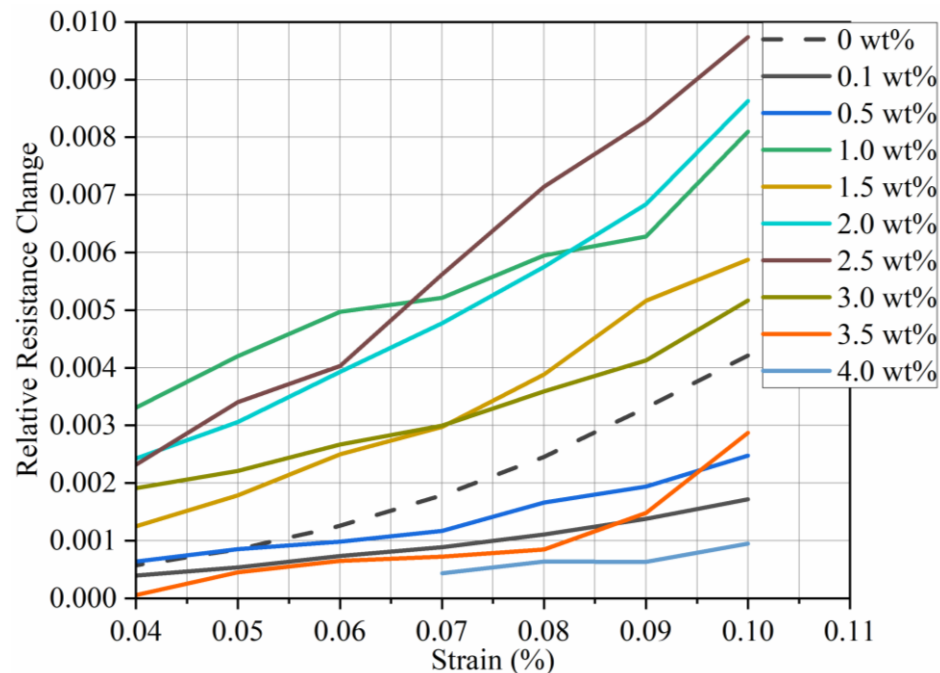


Composites showed a good correlation between input strain and resulting electrical resistance change

Accomplishments

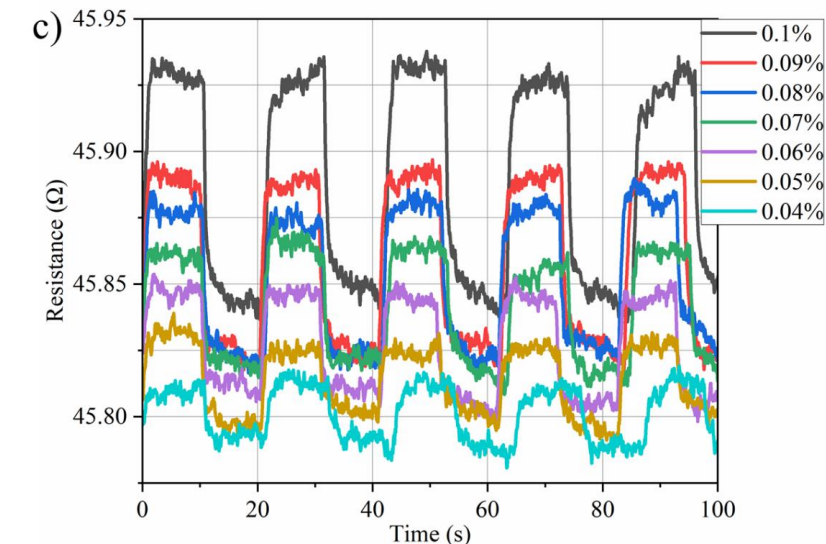
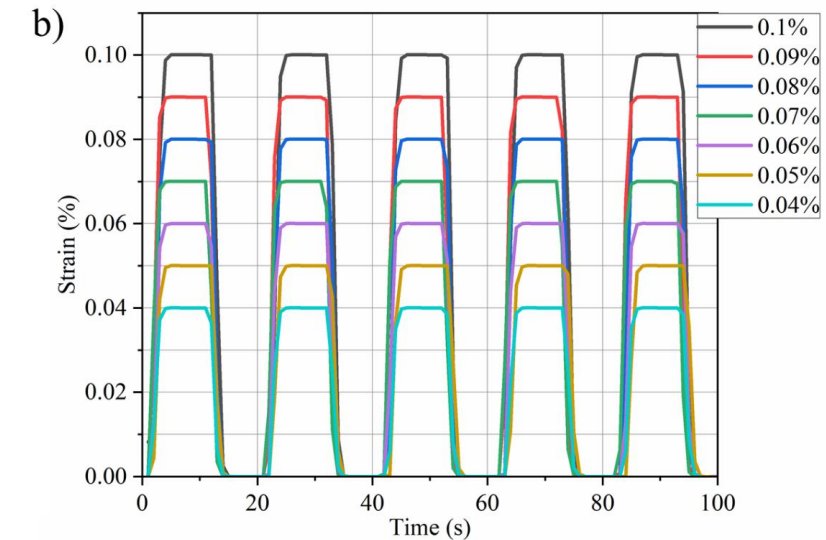
Structural Health Monitoring

- Tested each composite at various repeated strain levels
- Each composite with varying concentrations of TiO_2 were tested
- The electrical resistance measurements were normalized by calculating the relative resistance change
- Relative resistance change for each composite was measured over a range of strains to quantify the sensor sensitivity



Average relative resistive change over a strain range

Composites showed increasing relative resistance change with increasing strain levels



Structural health monitoring tests showing straining of a composite beam with corresponding electrical resistance changes

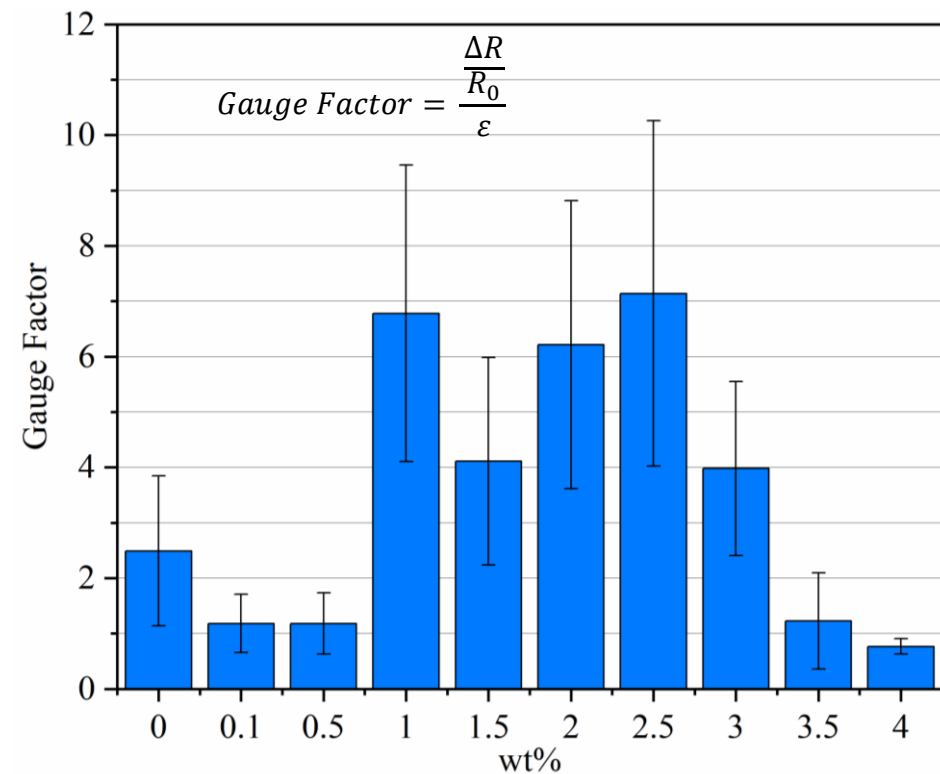
Accomplishments

Structural Health Monitoring

- Averaged the gauge factor at each strain level to calculate an overall average gauge factor for each composite at different nanoparticle concentrations
- Baseline gauge factor: 2.49 (0 wt% nanoparticles)
- Highest gauge factor: 7.14 (2.5 wt% nanoparticles)
- **Maximum gauge factor increase of 187% so milestone was achieved**

Milestone: Characterize the active sensing capabilities of the multifunctional composites (6/30/2020)

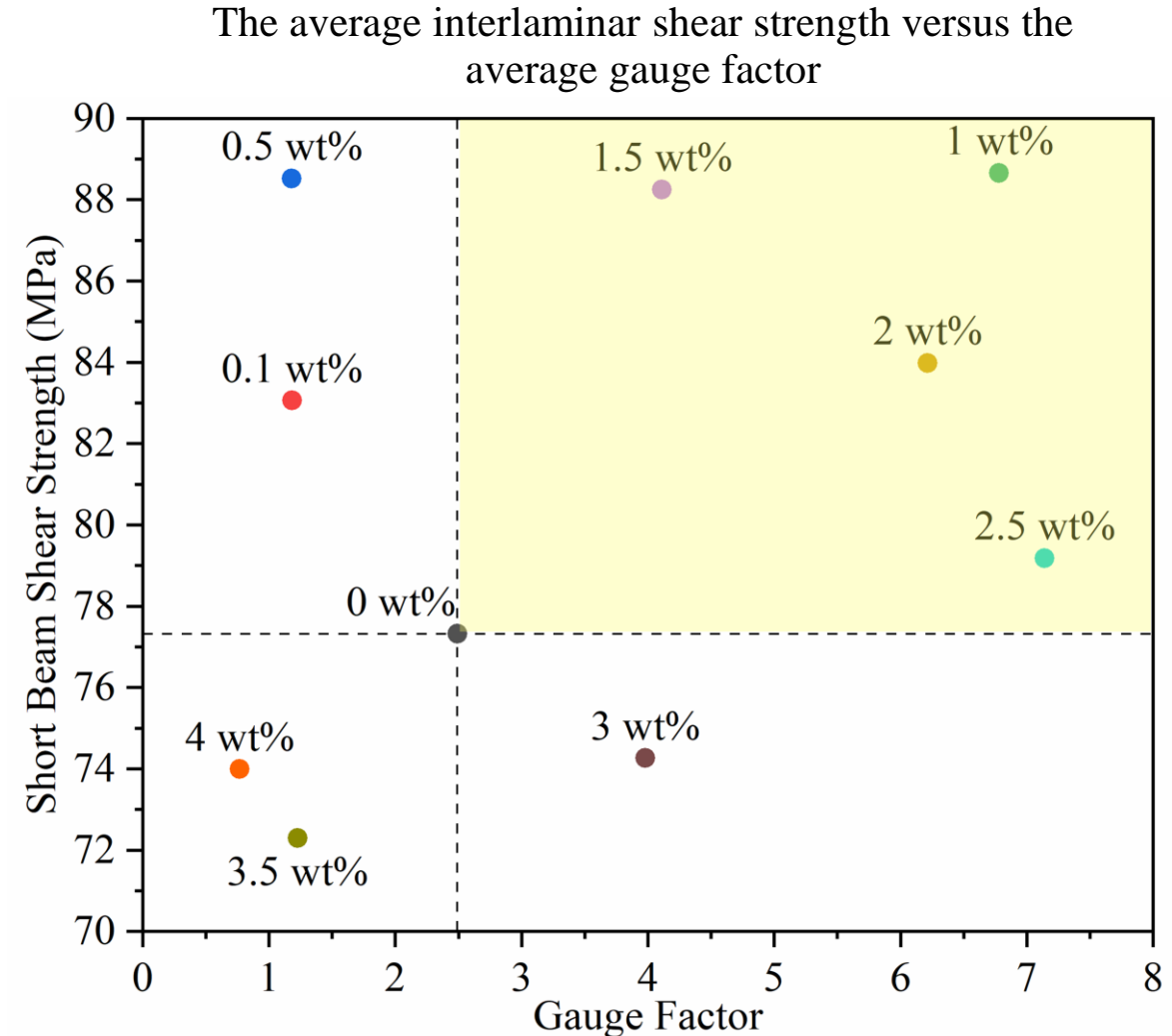
Criteria: Sensor testing in the dynamic mechanical analyzer should reveal at least a 20% improvement in sensitivity



Average gauge factor for each composite over the entire strain range

Accomplishments

- Not all nanoparticle concentrations showed improvements for these measured properties
- Some property trade-offs were observed
- Four composites (1.0, 1.5, 2.0 and 2.5 wt%) exhibited simultaneous improvements in interlaminar shear strength and structural health monitoring sensitivity (gauge factor)
- Best performing composite was 1.0 wt% nanoparticles
 - Interlaminar shear strength: 88.7 MPa (**14.7% increase compared to 0 wt%**)
 - Gauge factor: 6.78 (**172% increase compared to 0 wt%**)

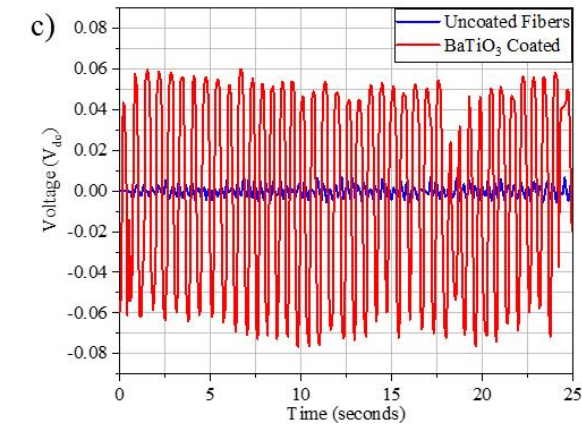
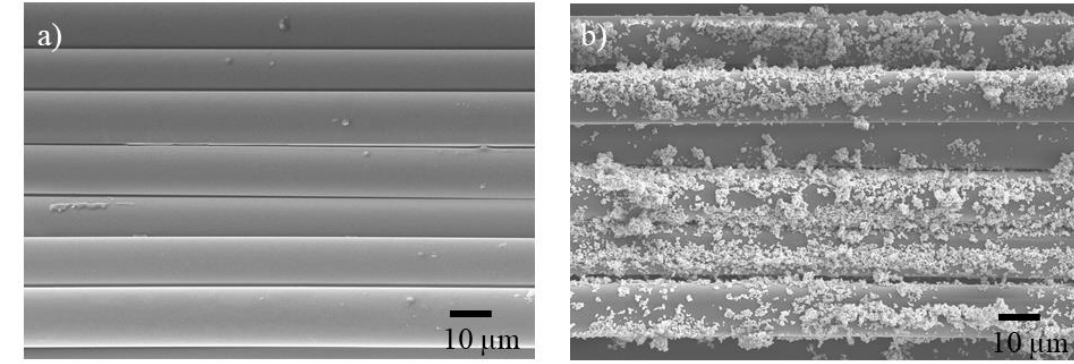
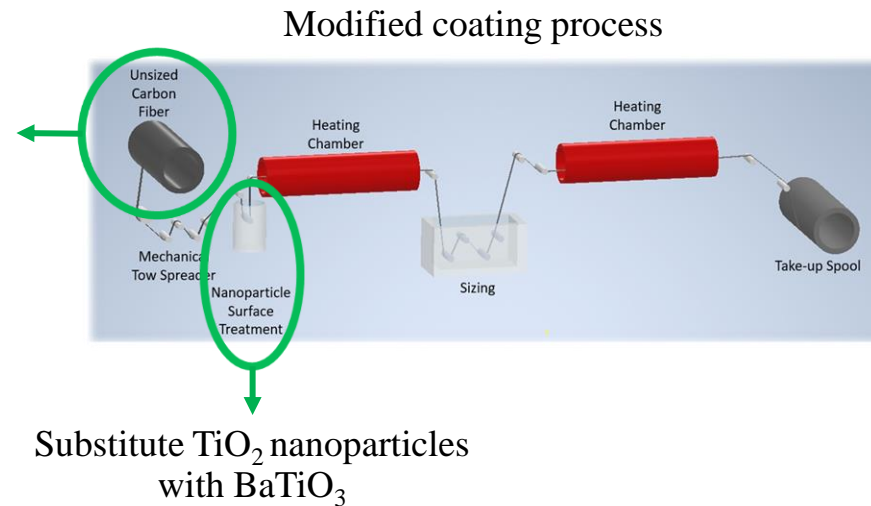


Met the two milestones for the active composite using TiO₂ coated carbon fiber

Accomplishments

Switching from active sensing to passive sensing

- Demonstrated the versatility of the coating process to accommodate different material systems
- Coated tows of fiber with ferroelectric particles
- Fabricated a simple unidirectional composite using one tow of fibers and compared coated vs uncoated fibers
- **Proof-of-concept flexing showed a voltage signal produced by the BaTiO₃ coated fibers**



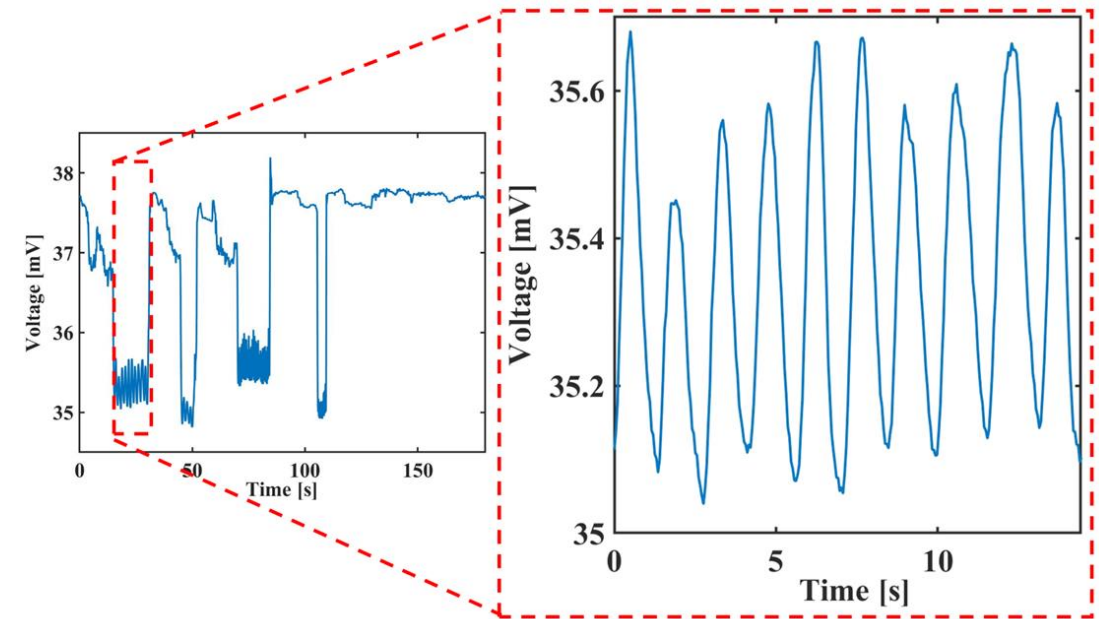
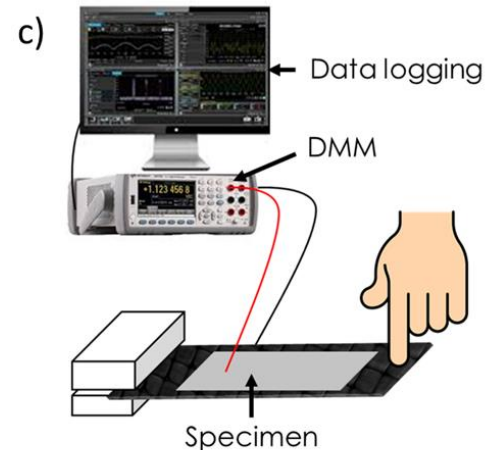
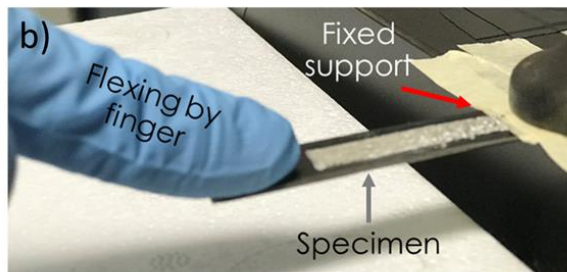
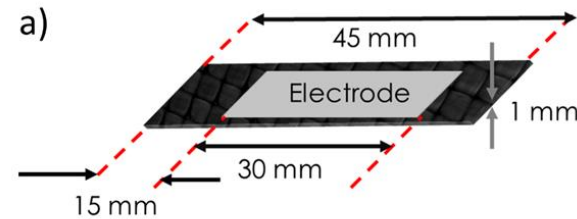
Milestone: Synthesize fibers with passive sensing capabilities (9/30/2020)

Criteria: Sensor testing in the dynamic mechanical analyzer should reveal at least a 20% improvement in sensitivity

Accomplishments

Hybrid Composite

- Larger unidirectional composites were fabricated with one layer of BaTiO_3 coated basalt sandwiched between layers of carbon fiber to make a hybrid composite
- Carbon fiber layers acted as electrodes and were coated with silver epoxy as electrode attachment points
- The hybrid composites produced a voltage in response to flexing and demonstrated the proof-of-concept that the hybrid multifunctional, passive sensing composite is feasible using this deposition approach



Milestone: Fabricate a passive sensing, hybrid, multifunctional composite (12/31/2020)

Criteria: Fabricate a hybrid composite that uses at least two different fibers within the matrix to create a multifunctional composite that can generate its own electrical charge in response to strain input

Response to Previous Year's Reviewers' Comments

- “Since many materials are subject to cyclic loading in service, it would be good to have some data on the impact of the new fiber coating on the fatigue of the composites.”
 - Response: The reviewer is correct in identifying that there is a need for fatigue testing of the composites. While fatigue testing was not included in this project, continuing work in another VTO project has a collaboration established with Columbia University to perform fatigue testing of the particle coated fibers.
- “The funding level is quite modest, but the work performed and progress reported suggest that the resources are sufficient. The reviewer was curious about whether additional funding to support specific real applications should be considered at the conclusion of this foundational work.”
 - Response: Funding has been obtained through another VTO project to further develop the fundamental science behind these multifunctional composites, and additional private industry partners have approached ORNL to investigate real-world deployment of these composites.
- “The approach is very innovative in creating a fiber sensing capability by deposition of nanoparticles on the fiber surface.”
 - Response: We appreciate the reviewer acknowledging the level of innovation of this coating process.

Collaboration/Partner

Dronesat, LLC

- Designed and patented (patent pending) an Unmanned Aerial System (drone), powered from a ground based electrical infrastructure for sensor or cargo configurations
- Identify sensor capability that makes aware, timely information about critical components so actionable decisions can be quickly made about the airworthiness of vehicles
- The ultimate result would reduce maintenance time by early detection of cracks or damage and the deterrence of the costly effects by failures which result in vehicle loss
- Throughout the project, designs for composite integration into damage prone components were designed (images of designs have been omitted due to public release of data)

ORNL interns

- An undergraduate researcher from Virginia Tech (Susan Rankin)
- A post-Bachelor's intern from North Carolina State University as part of the GEM Fellowship Program (Mikayla Moody)

Interns holding TiO₂ nanoparticle coated carbon fiber spools



Susan Rankin (on left) and Mikayla Moody (on right)

Remaining Challenges and Barriers

- Hybrid composite commercialization and integration into real structures
 - These composites are difficult to fabricate due to shorting during the poling process
 - So additional work must be performed to optimize the fabrication process for these composites to see use in real-world applications

Proposed Future Research

Project was closed out on March 25, 2021

No remaining future proposed research for this project

Summary

- **Relevance:** Development of a multifunctional fiber-reinforced composite with structural health monitoring capabilities and improved mechanical performance
- **Approach:** Nanoparticles deposited on the fiber surface via a continuous feed-through process
- **Technical Accomplishments:**
 - Demonstrated a roll-to-roll process to embed nanoparticles in the fiber sizing
 - Showed an interlaminar shear strength improvement of at least 10% (maximum increase was 14%) (Target date: Dec. 2019)
 - Demonstrated active sensing capabilities with at least a 20% improvement in sensitivity (actual improvement was 187%) (Target date: June 2020)
 - Fabricate a hybrid composite that uses at least two different fibers within the matrix to create a multifunctional composite that can generate its own electrical charge in response to strain input. (Target date: Dec. 2020)
- **Future work:** Project has ended

Technical Backup Slides

Technical Backup Slides

Dynamic Mechanical Analysis

