Soft Smart Tools Using Additive Manufacturing

Project ID: mat206

PI/Co-Is: Jay Gaillard (SRNL), Srikanth Pilla (CU), Richard Czerw (MS)

FY 2021 DOE Vehicle Technologies Office Annual Merit Review

June 21 – 25, 2021

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Program Overview

Goal: Develop soft, smart composite tooling by additive manufacturing

Timeline:

Project Start Date: 10/01/2020

Budget I Period End Date: 10/31/2021

Barriers and Challenges:

- Achieving thermal conductivity targets
- Mechanical properties, uniformity, durability.

Project Year 1 Budget

DOE Share	$325,000
Subrecipient Clemson U	$ 75,000
Subrecipient Mainland	$100,000
Total Budget	$500,000

Project Team and Roles:

Savannah River National Laboratory (Lead)
- EM Susceptor design
- Continuous fiber and sensor 3D printing

Clemson (CU-ICAR)
- Mechanical testing
- Advise on industry standards.

Mainland Solutions, LLC
- Produce and compound nanomaterials and fabricate the 3D printing filaments
- Produce materials needed for the embedded sensors

Relevance

Goal: Develop soft, smart composite tooling by additive manufacturing

Impact

Targeting vehicle tooling will lead the way for producing a multitude of different car parts based on our proposed Smart Soft Tooling. Advantages over standard soft tooling include:

- Reduced tool cost, by lowering scrap rate
- Reduce cure cycles by 20%-40%, by *in situ* cure monitoring,
- Lower thermal gradients, leading to more consistent part production.
- Increased durability, leading to longer operational lifetime over traditional soft tools

Smart tooling enhances this by providing immediate feedback about a part through its manufacture and use. In addition to the VTO office, the proposed technology supports advancements for the Advanced Manufacturing Office (AMO) office in process heating, smart manufacturing, additive manufacturing, and composite materials.

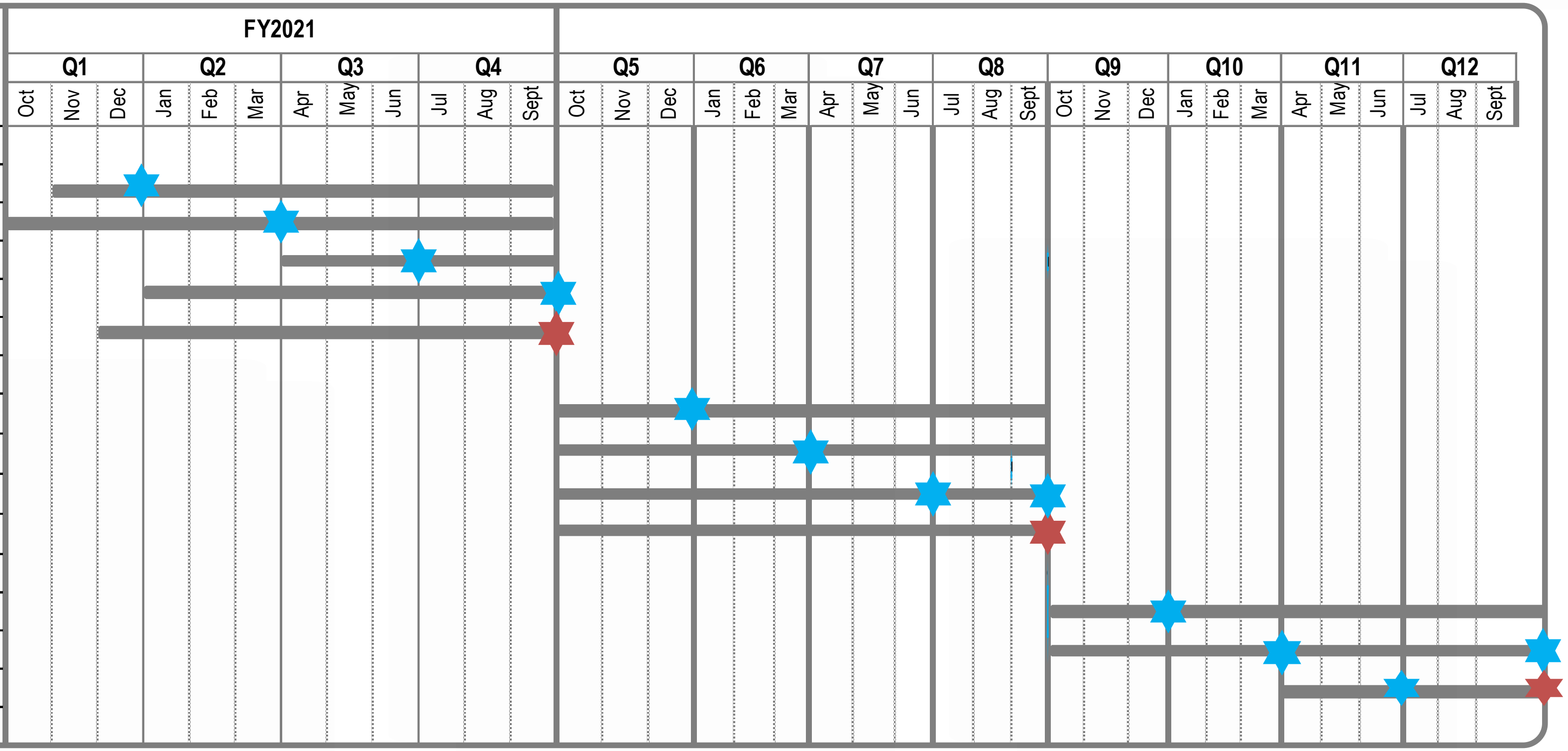
Objectives

- Increase the strength of a 3D printed part through susceptor-enabled EM annealing
- Increase the thermal conductivity through embedding carbon nanomaterials and carbon fiber in the filament
- Develop sensor designs for incorporation into 3D printed parts
    - Strain sensor
    - Thermocouple
    - Capacitive curing sensor

Program Milestones and Approach

Any proposed future work is subject to change based on funding levels.

Task	Activity	Team Member: SRNL	Team Member: CU	Team Member: MS	Budget
1.0	**Year 1**				$500k
1.1	Compounding and filament production			●	$90k
1.2	3D-print continuous carbon fiber ASTM test coupons	●			$150k
1.3	3D print temperature, pressure, and strain sensors	●			$150k
1.4	Evaluate sensors	●		●	$35k
1.5	ASTM testing of coupons		●		$75k
2.0	**Year 2**				$500k
2.1	Optimization and downselection			●	$100k
2.2	Produce integrated test panel	●			$275k
2.3	Evaluate test panels		●		$75k
2.4	Technoeconomic analysis	●			$50k
3.0	**Year 3**				$500k
3.1	Intermediate scale-up of thermoplastic filaments			●	$100k
3.2	Print prototype tool	●			$305k
3.3	Perform testing on tool		●		$95k
	Total Budget				$1.5M



Year 1 Milestones

M1.1: Demonstrate proof of concept of significant mechanical/thermal property enhancements using EM coupled heating.

M1.2: Provide final SMART sensor design for integration into 3D printing.

M1.3: Provide sufficient evidence of >10% increase in tensile strength and > 10% increase in thermal conductivity for small test coupons.

M1.4: Demonstrate SMART sensor integration of at least two sensors into a test coupon.

Year 2 Milestones

M2.1: Provide sufficient evidence of >20% increase in tensile strength and > 20% increase in thermal conductivity for small test coupons.

M2.2: Demonstrate analytically the potential to reduce energy intensity by at least 40% by combined process heating and SMART sensing.

M2.3: Provide expert opinions from car manufacturing industry partners.

M2.4: Provide final optimization of SMART SOFT tool for prototyping.

Year 3 Milestones

M3.1 3D print first small-scale tool for part production at ICAR.

M3.2 Provide evidence of formed composite coupon meeting typical performance parameters.

M3.3 Provide complete test plan for fully integrated prototype tool.

M3.4 Demonstrate at least 25% reduction in energy consumption using EM-energy post heated tools with prototype fully integrated SMART sensors.

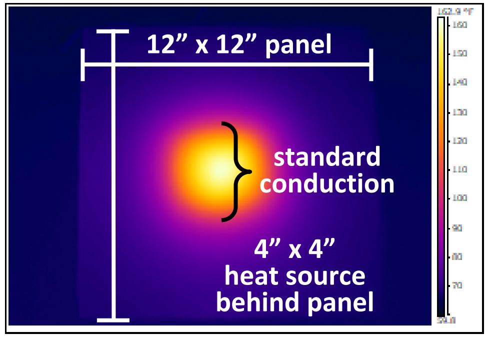
Technical Approach

To increase process throughput, reduce scrap rates and lower soft tooling costs, the program will prototype soft smart tooling using additive manufacturing techniques:

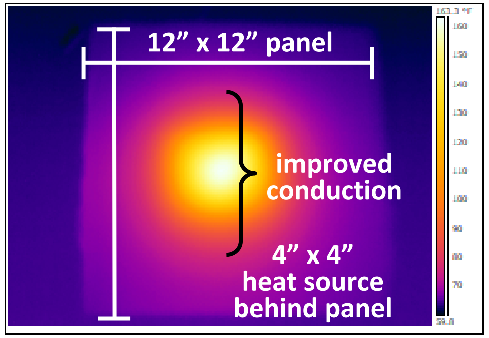
1. Nanomaterial filled thermoplastics will be used to improve thermal conductivity with added tool strength in 3D continuous fiber printing. This will allow for thinner tooling with less thermal gradient and lower thermal lag.

2. Post curing/annealing will be used by coupling microwave or induction RF energy directly to susceptor-enhanced nanomaterials that serve to reinforce and improve polymer crystallinity for greater mechanical and thermal properties. EM heating is more efficient than conventional heating.

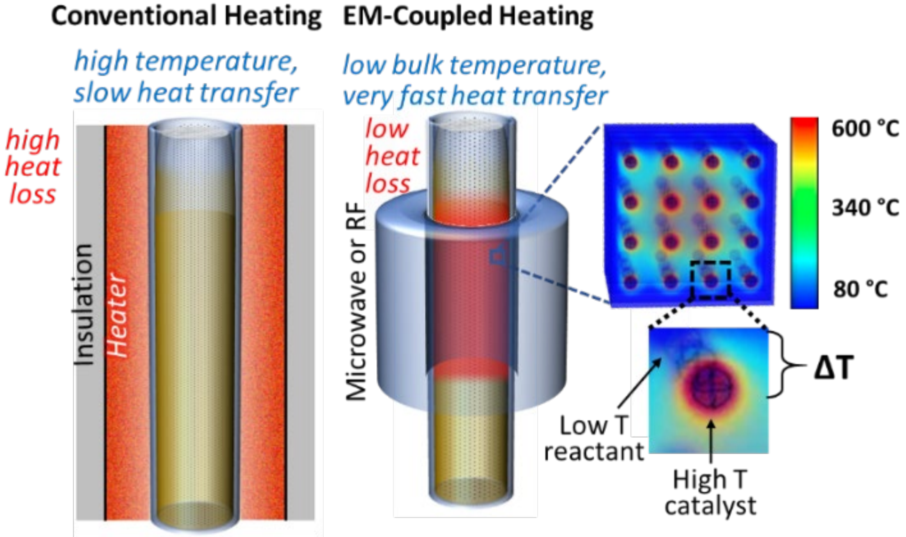
3.Temperature, heat flux, strain, and pressure sensors will be printed into the tool to monitor cure kinetics, voids (especially for resin transfer molding (RTM)), and spring-in.



Standard panel with no CNTs.



Panel with CNTs in the resin.

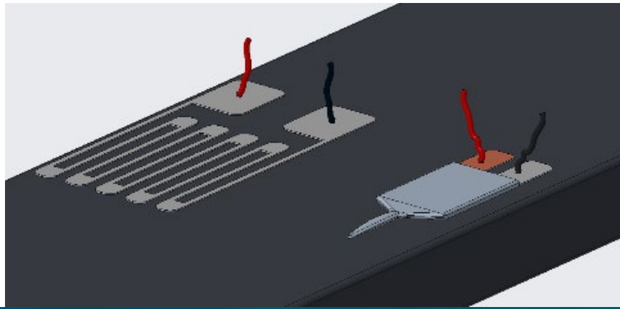


Part Progression

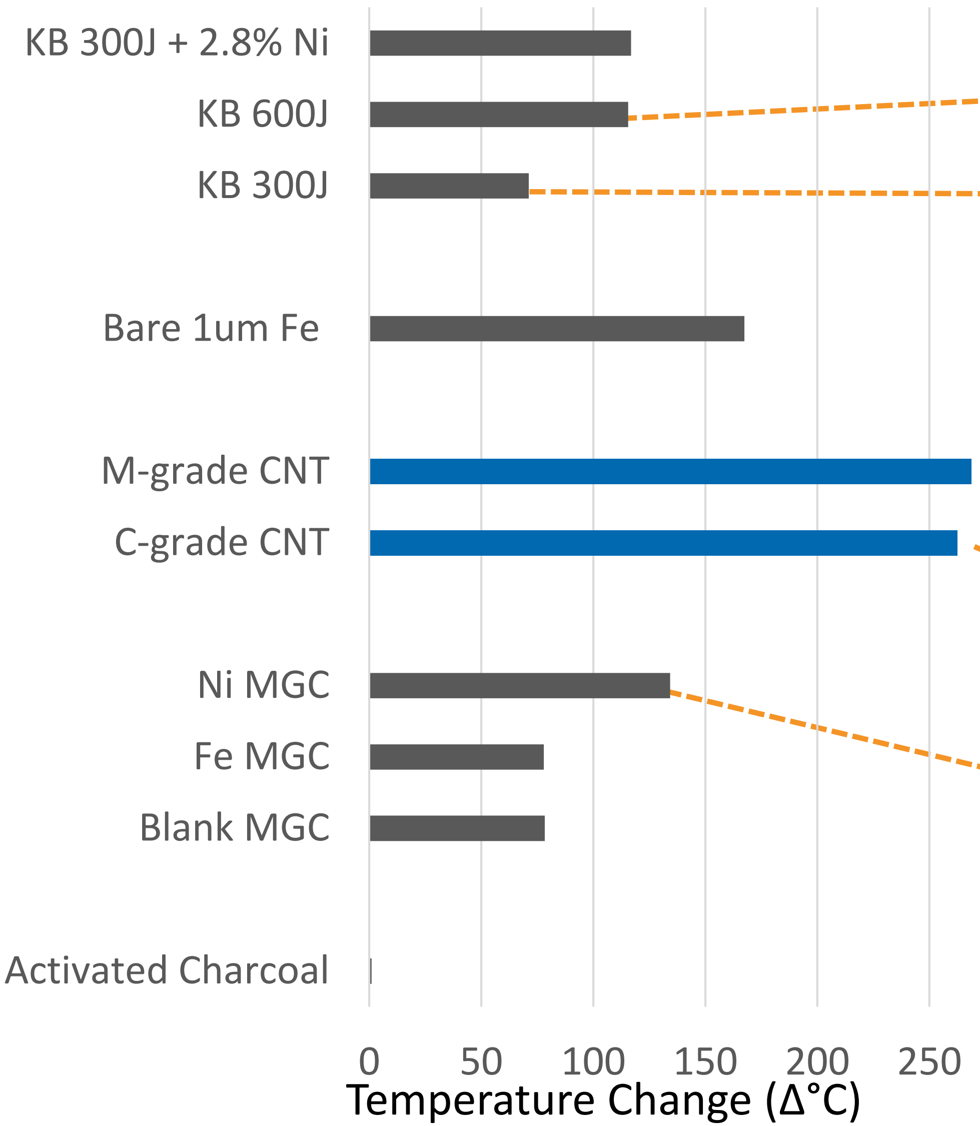
3D print nanomaterial-Continuous Carbon Fiber test parts

3D print sensors

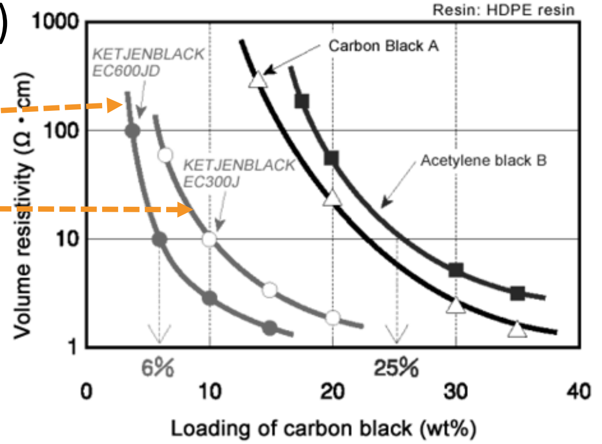
Fully-integrated sensors within 3D-printed tooling



EM Susceptor microwave heating



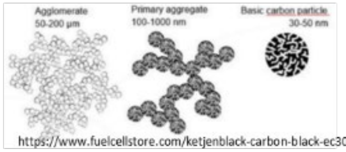
1)



https://www.lion-specialty-chem.co.jp/en/product/carbon01.htm

Why Ketjen Black?

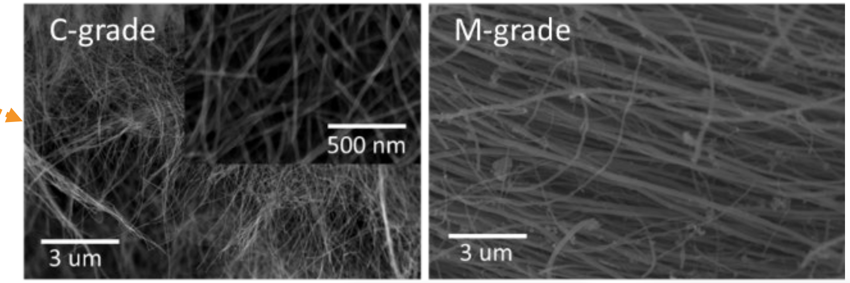
- Ketjen Black cost is commercially relevant. Can be purchased by the ton ($2.5/kg)
- High electrical conductivity – high Joule heating
  - 0.01 - 0.1 [Ω·cm]



https://www.fuelcellstore.com/ketjenblack-carbon-black-ec300j

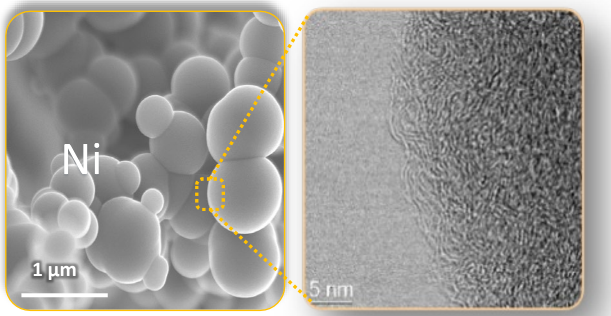
2)

~30nm diameter CNTs ~150nm diameter CNTs



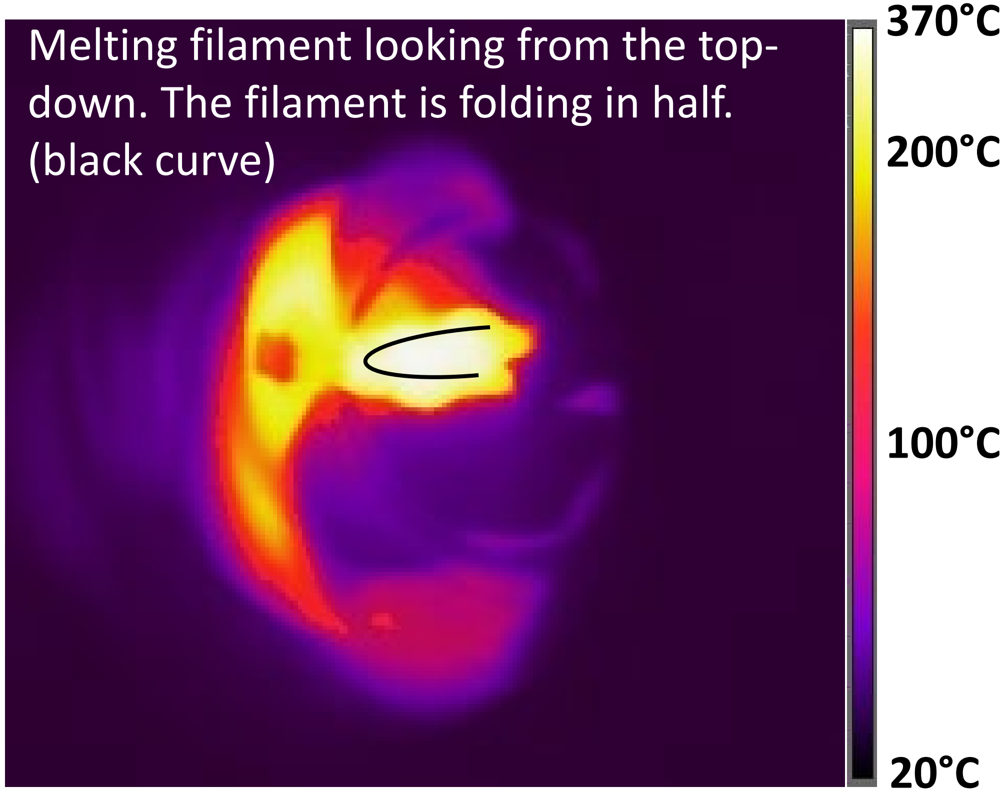
Carbon nanotubes produced using chemical vapor deposition

3)

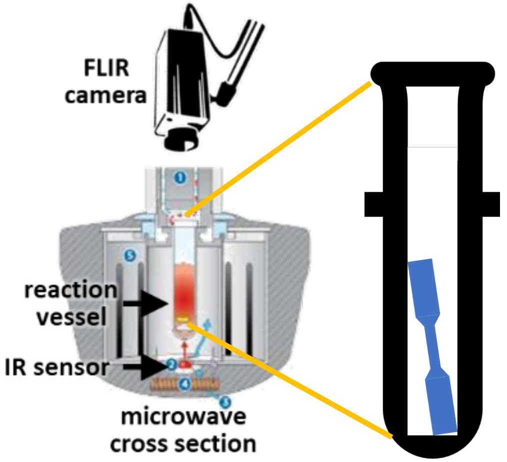


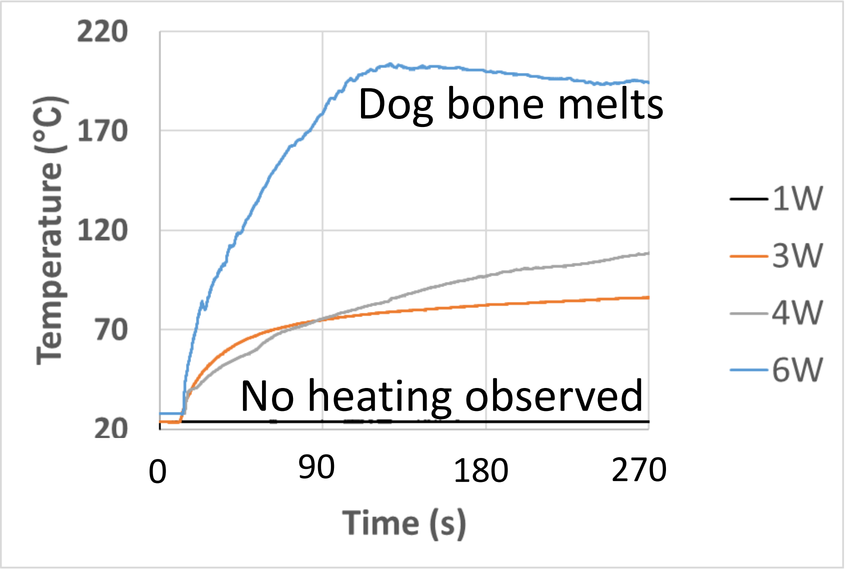
Carbon Fiber Nylon: Microwave Heat Treatment

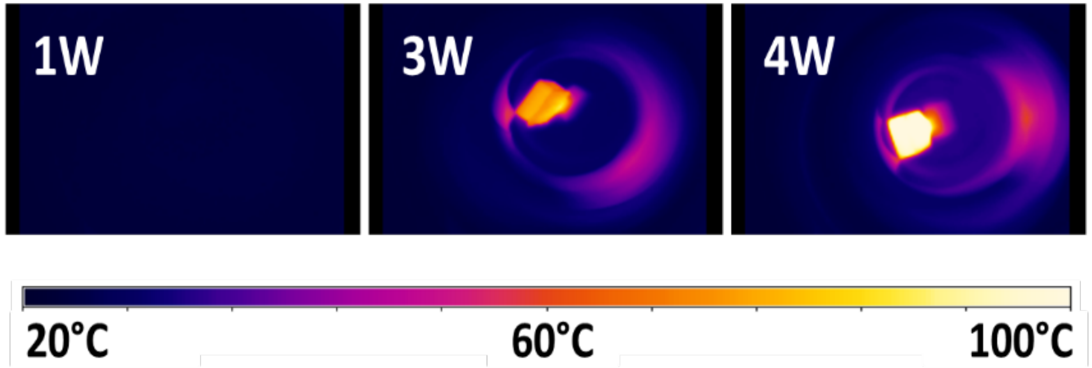
- Dog bones conforming to ASTM mechanical testing requirements
- Power-correlated heating experiments performed on the 3D printed dog bones
- Temperature-temporal plots recorded to show the heating ability of the material



10 W Irradiation. Sample heated to ~400°C in <60s.
Material: DuPont Zytel 20% Chopped Carbon Fiber Reinforced Nylon Filament



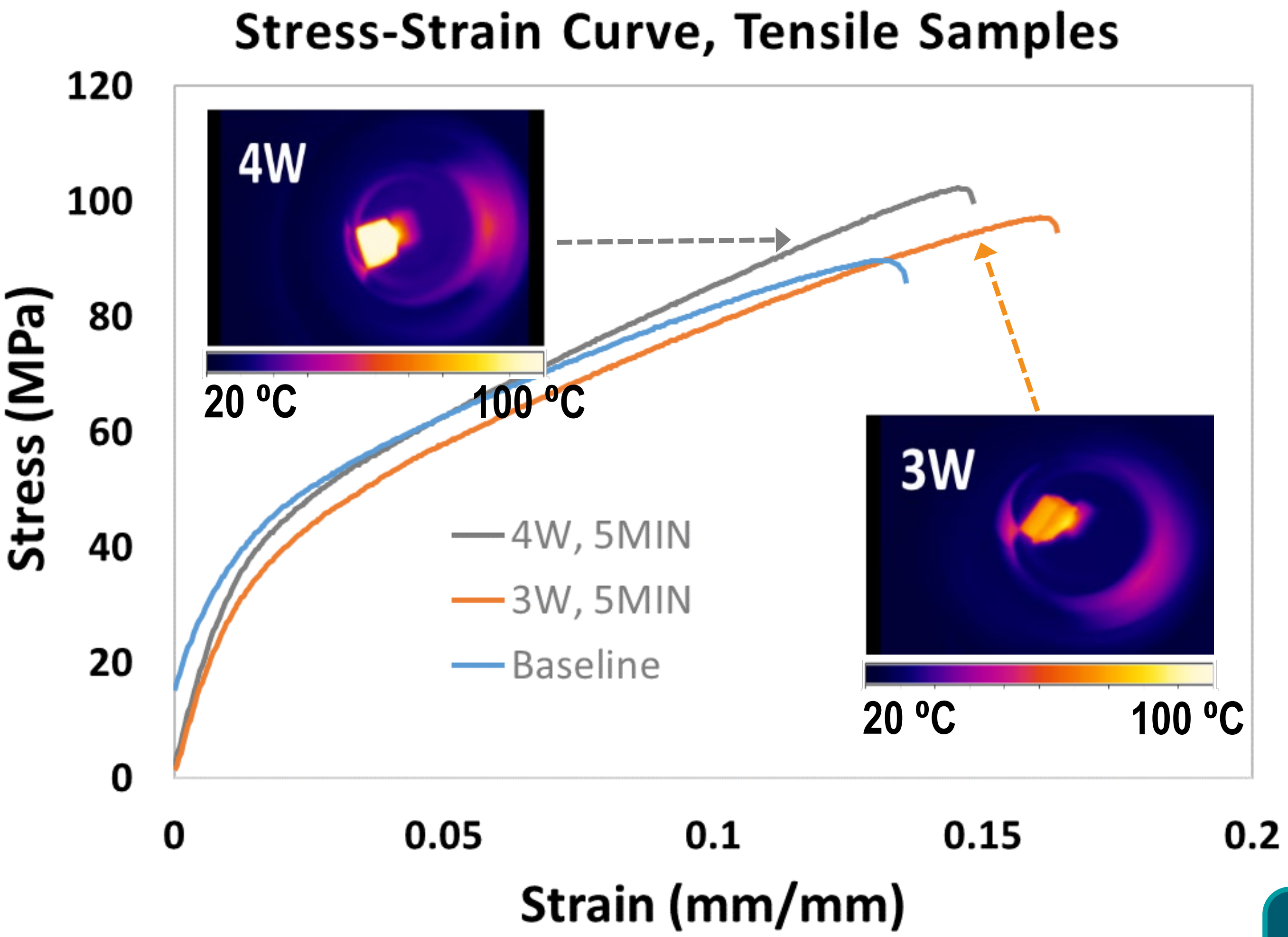


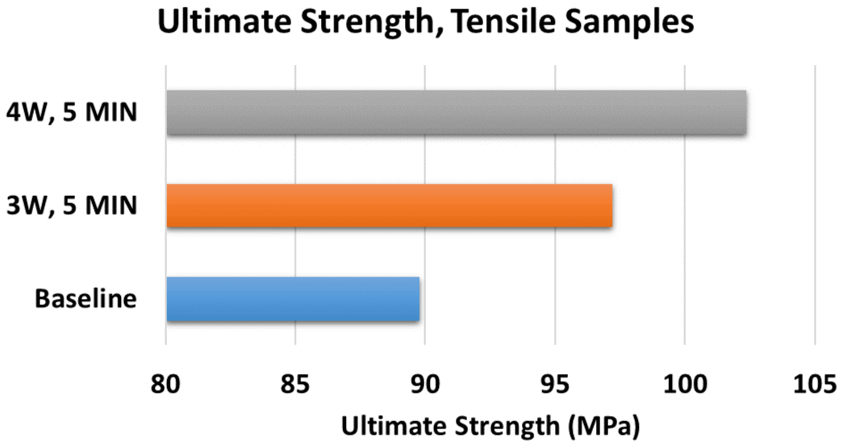


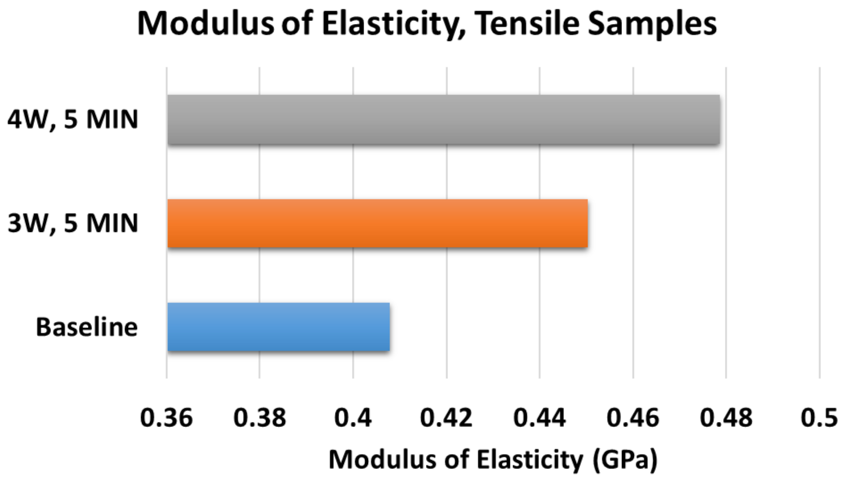
Thermal images of chopped carbon fiber 3D-printed dog bones under microwave irradiation.

Carbon fiber within 3D printed dog bones successfully anneals, and the annealing temperature can be controlled by the irradiation power.

3D Printed Dog Bones: Tensile Testing



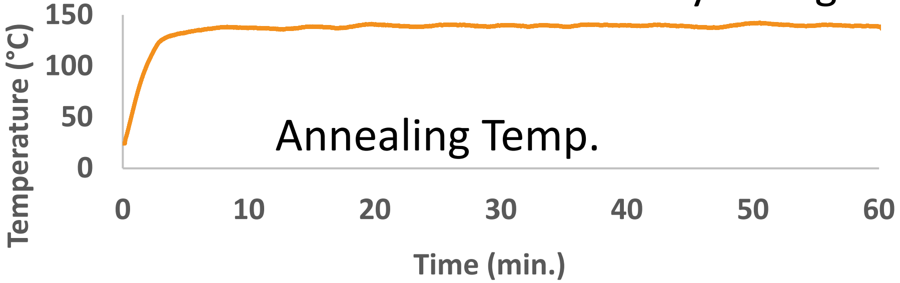


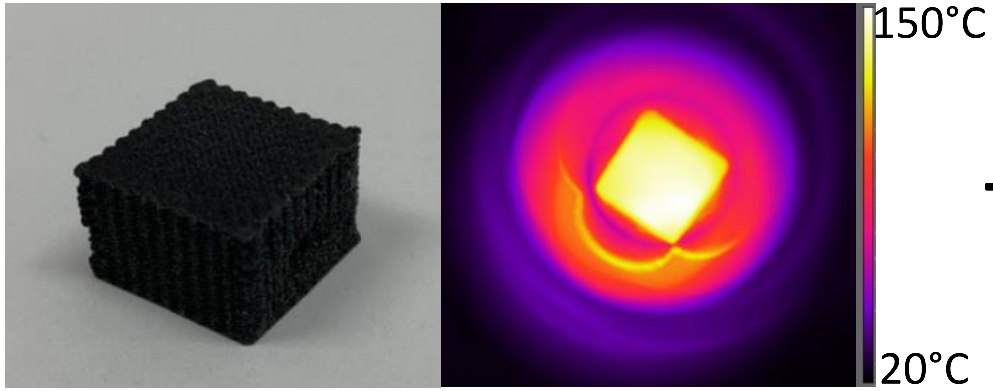


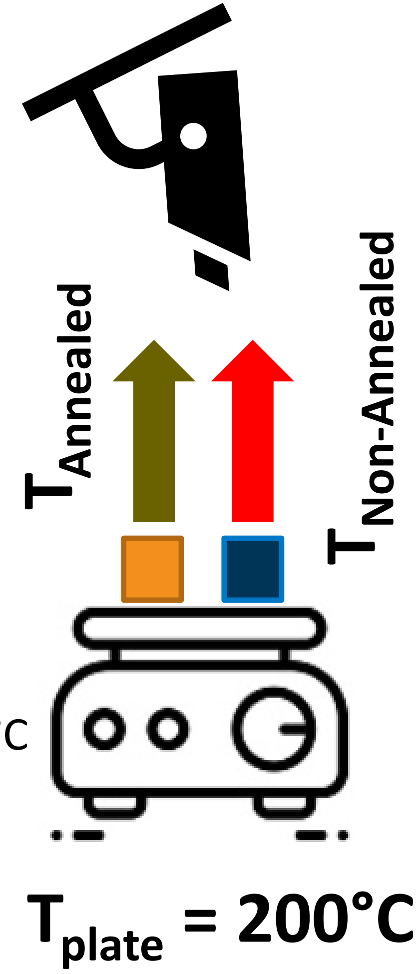
Microwave annealing shows a marked increase in the sample tensile strength with increasing power

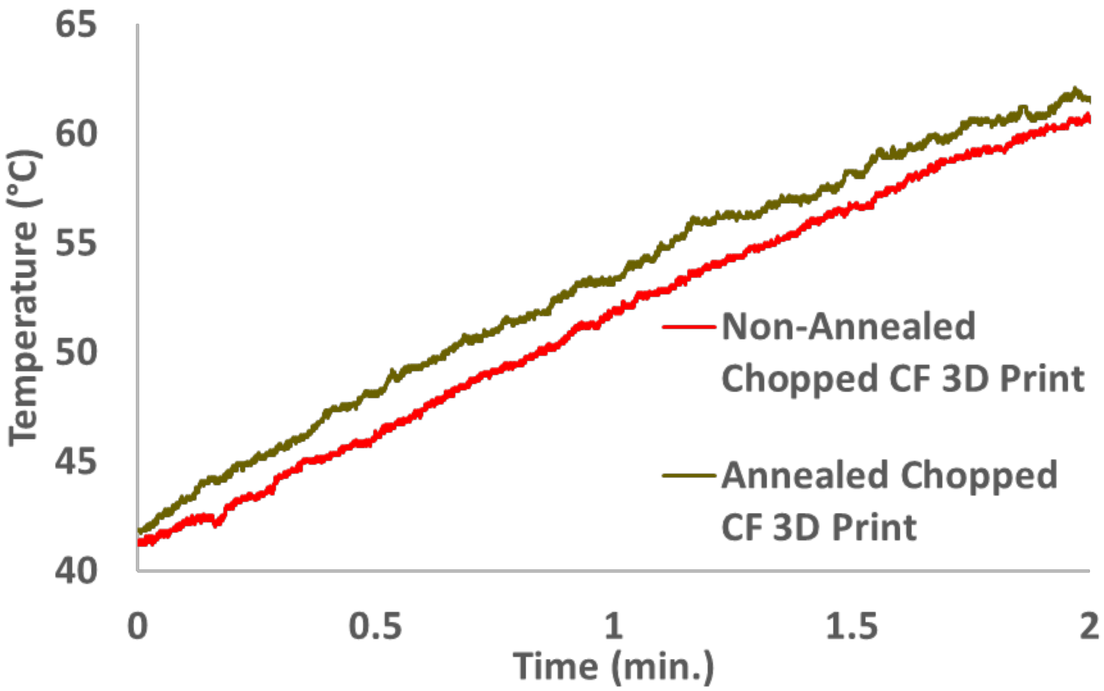
Thermal Conductivity of Chopped and Continuous Carbon Fiber Composites

- Two 3D-printed cubes made from chopped-carbon fiber filled nylon (20% wt.)
- One cube was as-printed without annealing. The second was a microwave-annealed cube (1 hour, 5-7 W controlled)
- Hotplate set to 200°C. Measured the time-dependent temperature change to demonstrate thermal conductivity change.





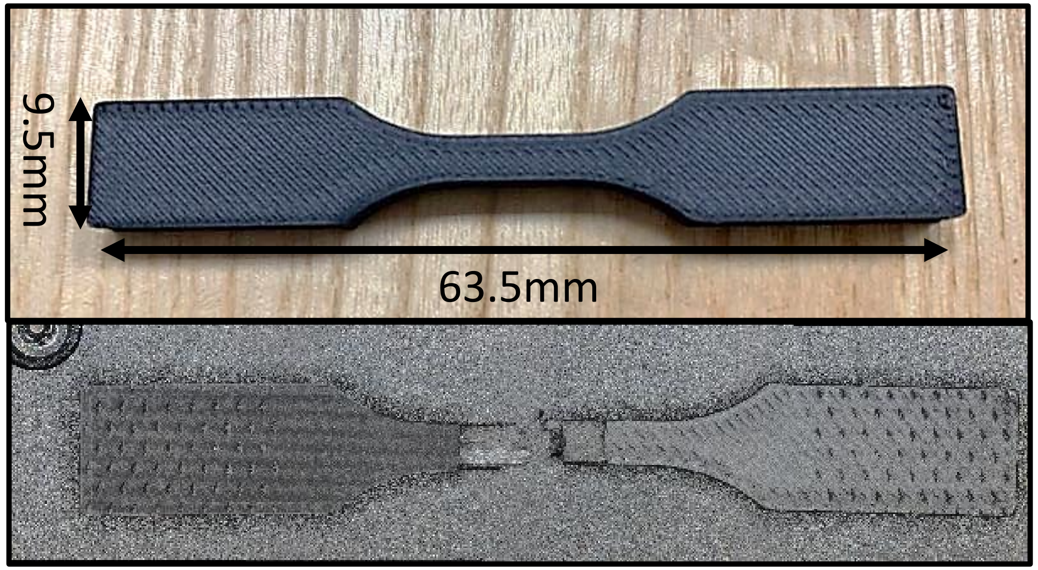


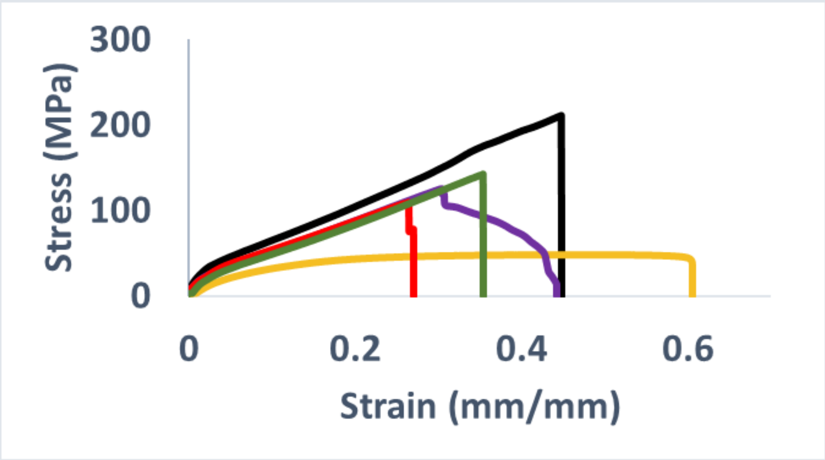


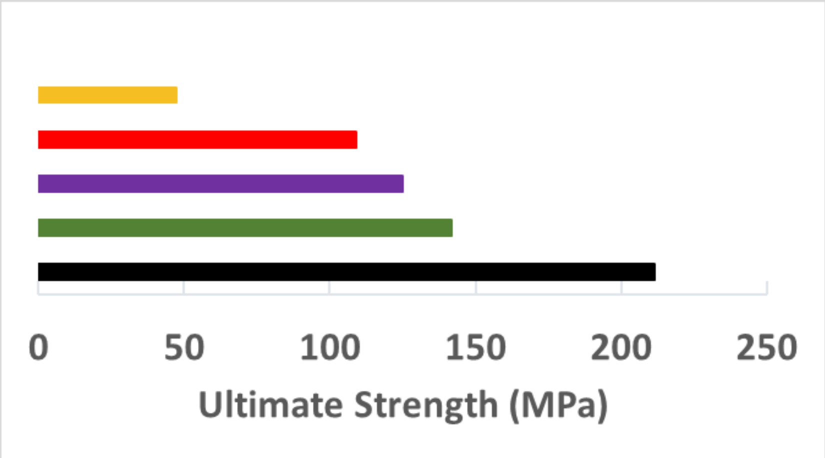
1 hr. of microwave annealing at relatively low power (<10W) demonstrates a noticeable increase in the thermal conductivity.

Printing w/ Continuous Carbon Fiber

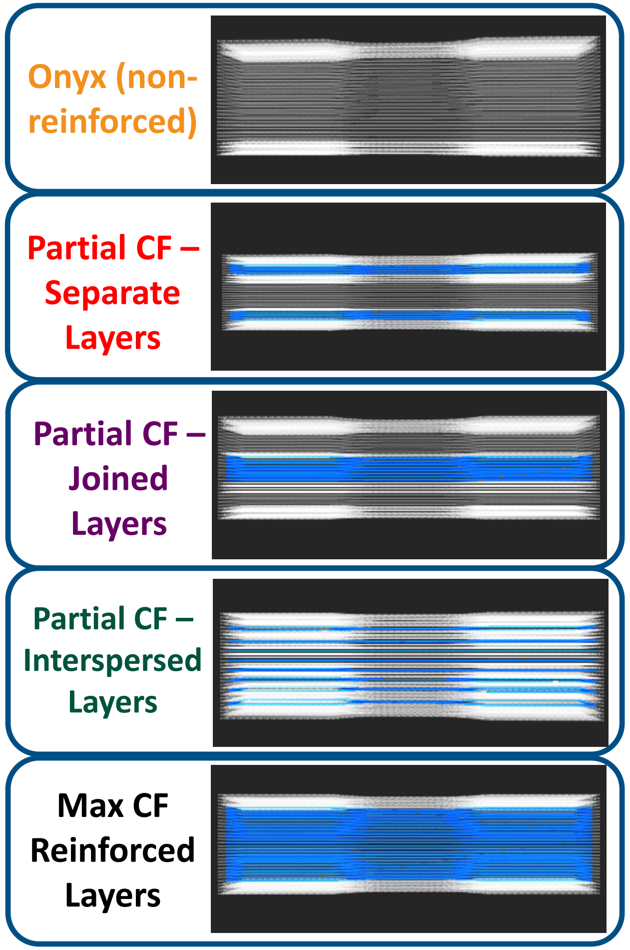
- Dog bone specimens printed from Onyx with varying amounts of carbon fiber reinforcement on Markforged X7 Printer
  - Onyx – Markforged Chopped CF Nylon material
    - Maximum CF layers
    - Partial CF layers
    - Interspersed CF layers
- Tensile tests performed using ASTM D638 standard





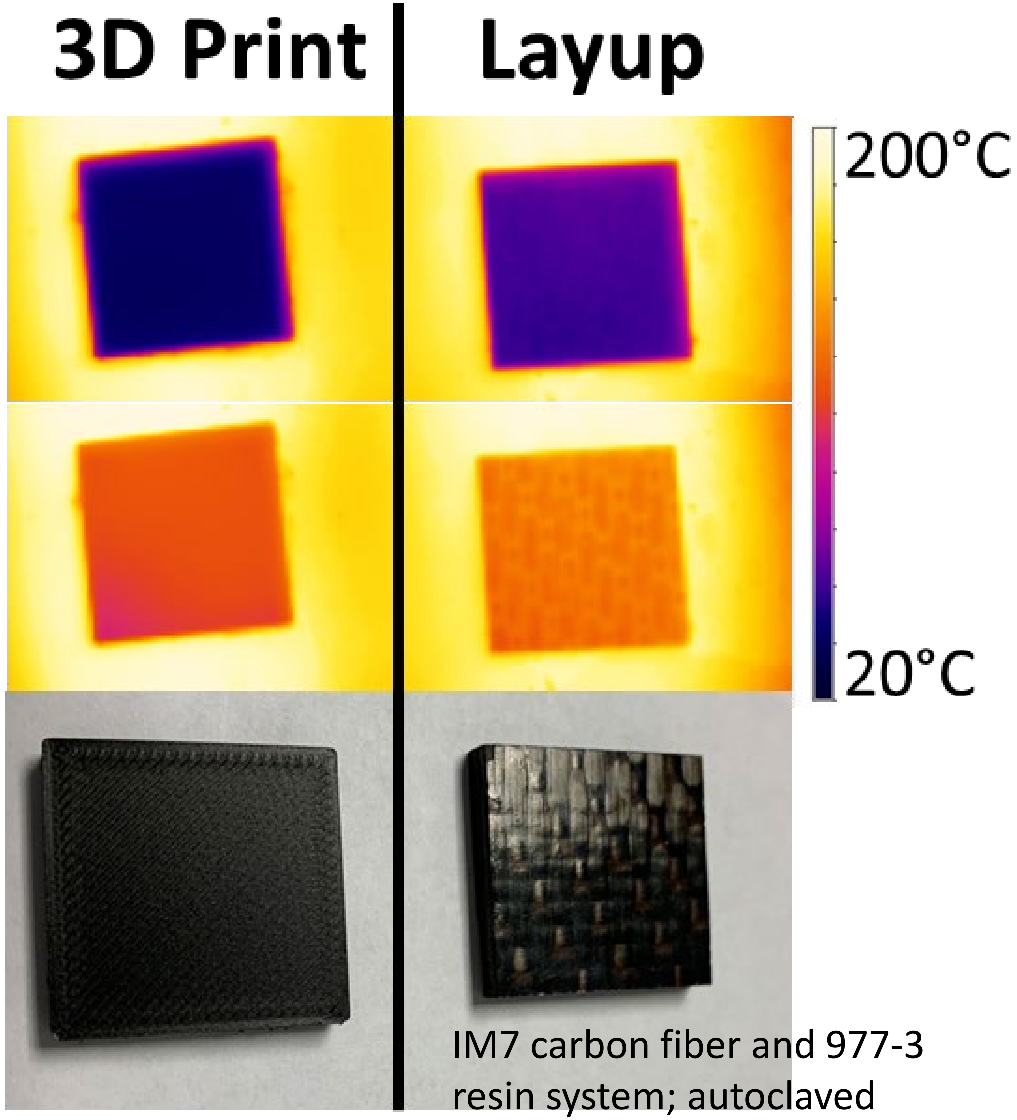


Dog Bone Side Profiles



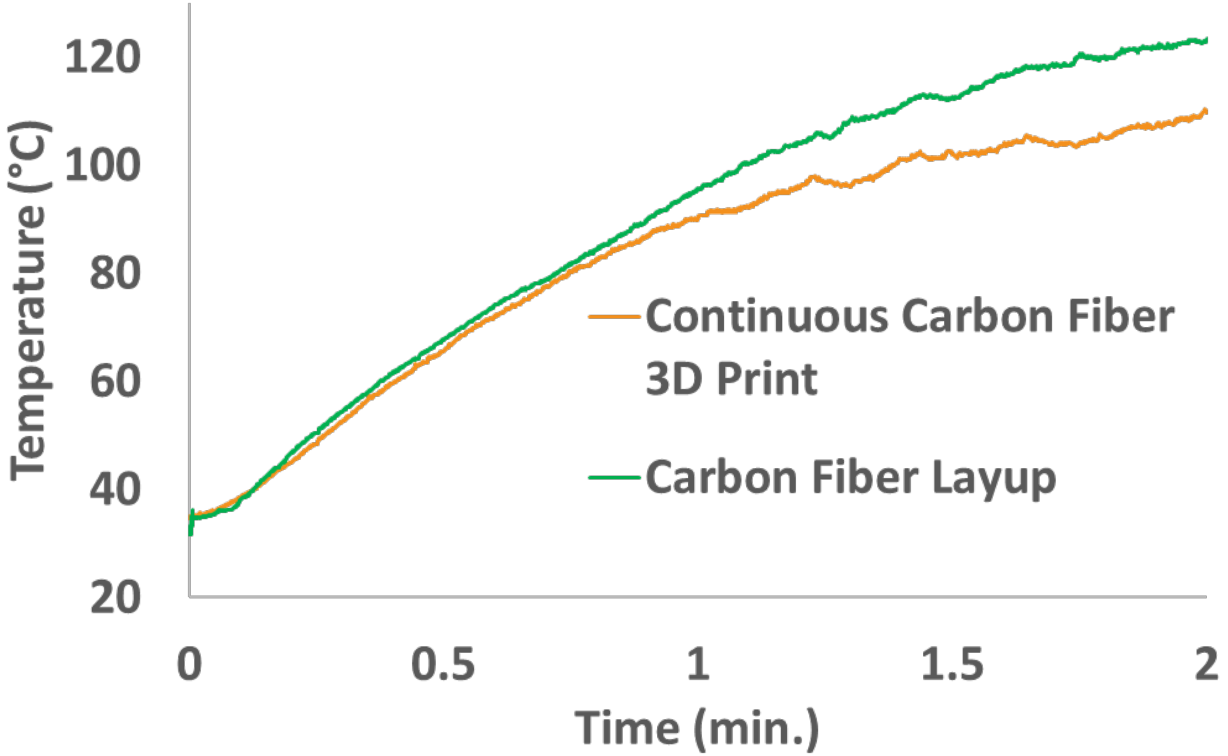
Adding 50% interspersed carbon fiber layers in the material provides about 70% the total strength of a fully reinforced part.

Thermal Conductivity of Chopped and Continuous Carbon Fiber Composites



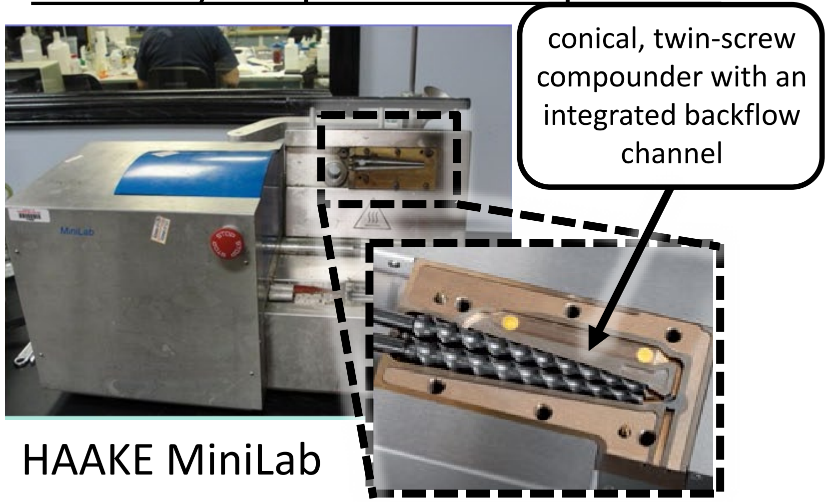
IM7 carbon fiber and 977-3 resin system; autoclaved

A 3D-printed part with chopped-carbon fiber resin and **continuous carbon fiber interlayers** has nearly the thermal conductivity as a "gold-standard aerospace-grade thermally conductive CF layup", even without post-print treatment.



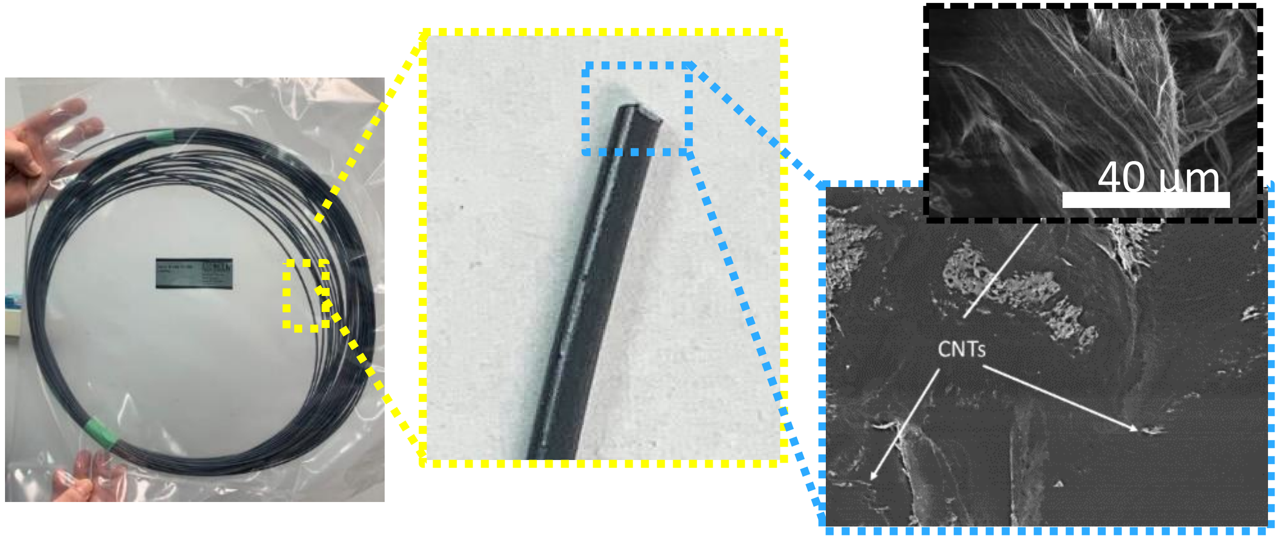
M-Grade Carbon Nanotubes in Nylon-12 Filament

Extruder/compounder components

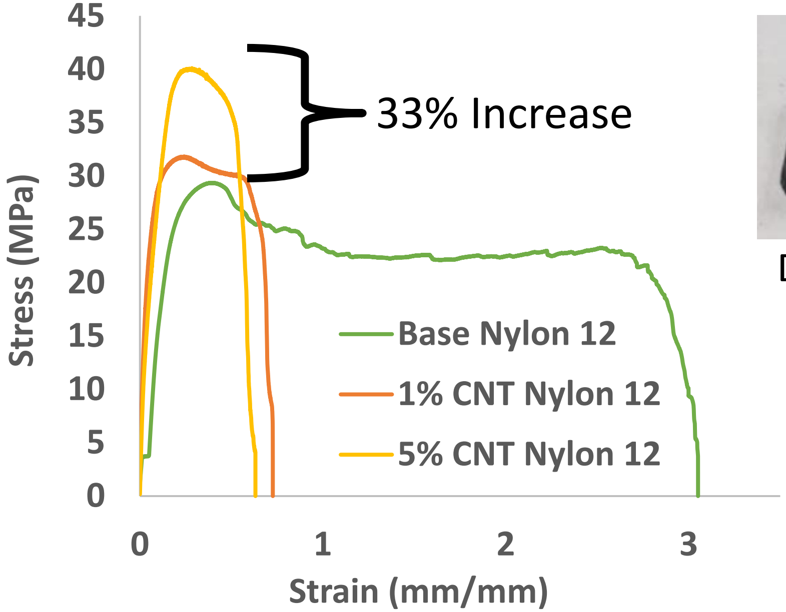


HAAKE MiniLab

1. Neat Nylon 12 was extruded through a 1.75 mm die at the outlet of the Haake to produce baseline filament material.
2. M-grade carbon nanotubes were dry mixed in a multi-axis mixer between 1% and 5%wt to polymer.
3. CNTs and nylon were melt mixed and extruded 3x to ensure proper dispersal of the CNTs.



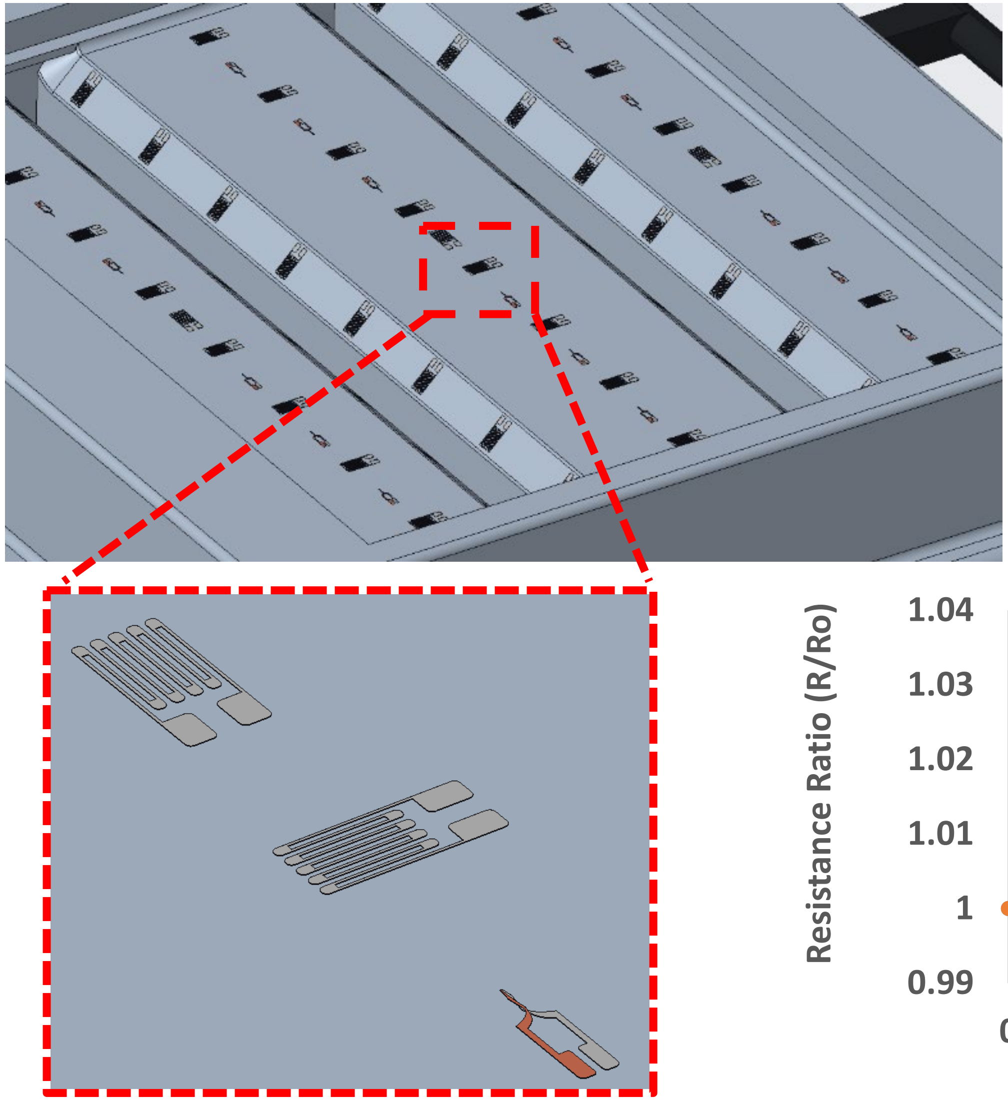
Images show a CNT-reinforced filament at 1% wt. filling.

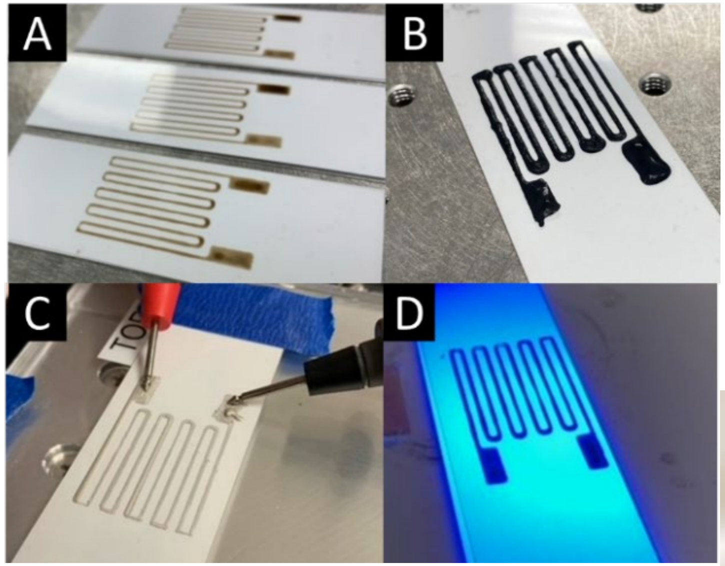


Dog bone printed from 5% wt. CNT-filled Nylon 12

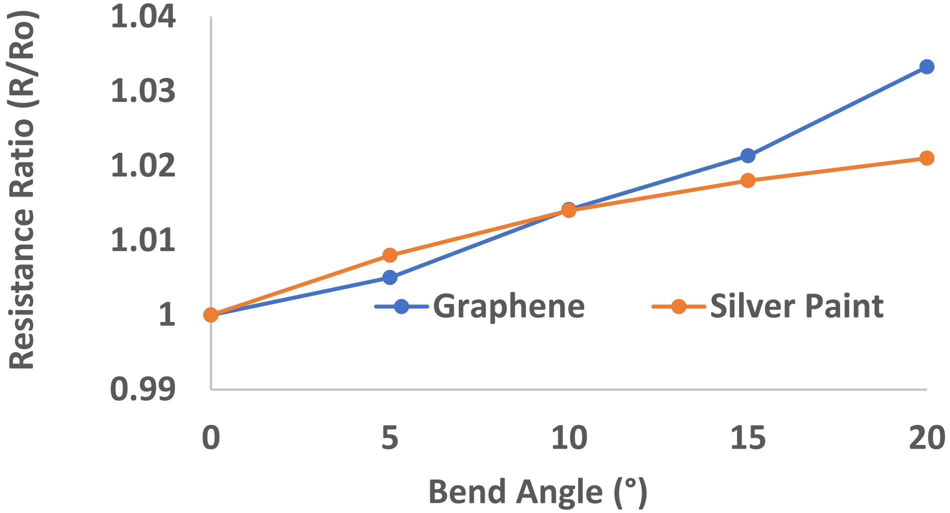
Adding carbon nanotubes to the polymer composite adds both strength and a susceptor within the filament.

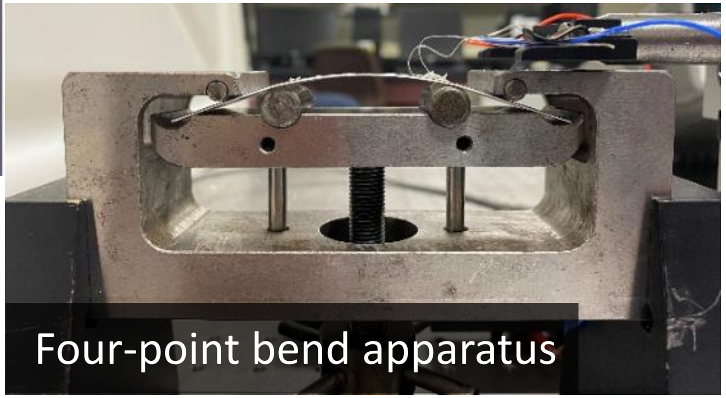
Printed sensors to be incorporated into Smart Tooling





Formulations Printed:
- (A) Graphene Oxide
- (B) Graphene-PVP Ink
- (C, D) Silver Paste
- Copper Paste
- Constantan Paste
- GO-CNT Ink




Four-point bend apparatus

Strain sensors have been successfully printed out of multiple materials and tested for their resistance change.

Responses to Previous Years' Comments

This is the first year this project has been reviewed.

Institution Coordination

Savannah River National Laboratory®

OPERATED BY SAVANNAH RIVER NUCLEAR SOLUTIONS

Prime, National Laboratory
Located in Aiken, SC
Within VTO

- EM Susceptor design
- Continuous fiber and sensor 3D printing

Mainland Solutions LLC

Subcontractor, Industry
Located in Yadkinville, NC
Within VTO

- Produce and compound nanomaterials and fabricate the 3D printing filaments
- Produce materials needed for the embedded sensors

CUICAR

CLEMSON UNIVERSITY INTERNATIONAL CENTER FOR AUTOMOTIVE RESEARCH

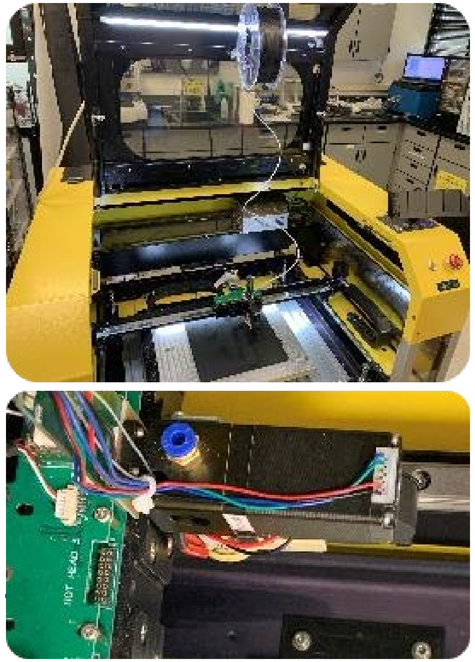
Subcontractor, University
Located in Clemson, SC
Within VTO

- Mechanical testing
- Advise on industry standards.

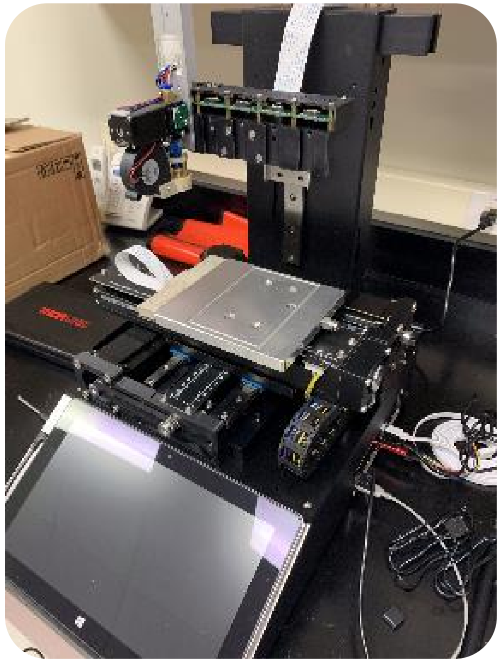
Remaining Challenges

- Compounded filaments containing CNTs must be co-printed with continuous carbon fiber tow to realize strength and thermal conductivity gains from both systems.
- Custom compounded carbon fiber tow must be formulated.
- Sensor ruggedness must be demonstrated on 3D printed test coupons.
- Sensor formulation to be developed with significant resolution in the deflection regime expected of soft tooling tolerances.
- Sensor wiring must be determined to hook to external measuring devices.
- Annealing conditions of printed parts must be identified so as to enhance the thermal conductivity of composite tools beyond the state of the art of thermally conductive carbon fiber layup tools.

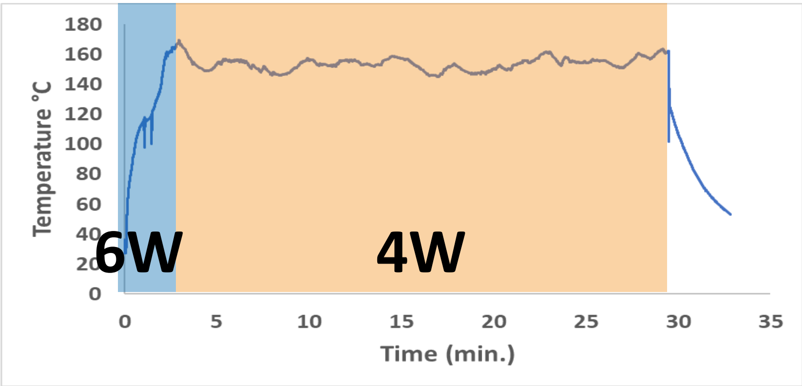
Any proposed future work is subject to change based on funding levels.



Hyrel Hydra



Hyrel Engine HR

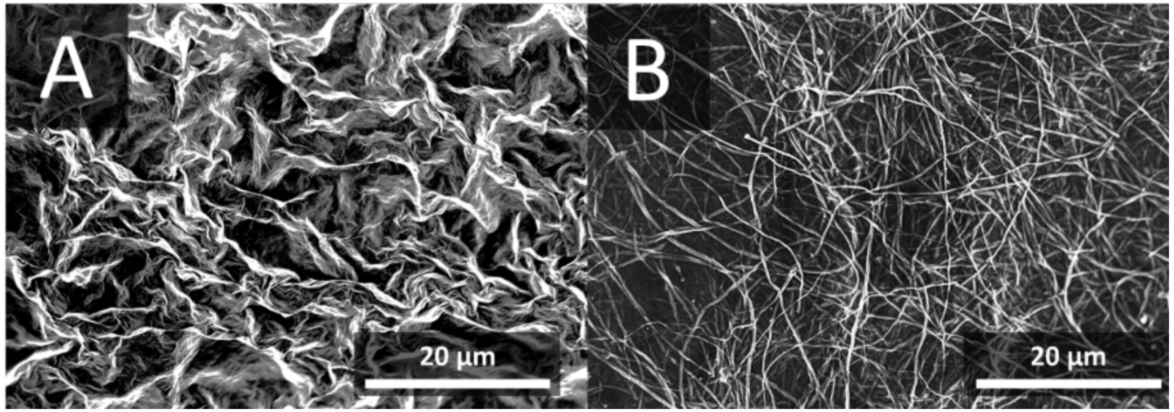


Annealing is controllable via power modulation

Proposed Future Research

FY21 Future Research

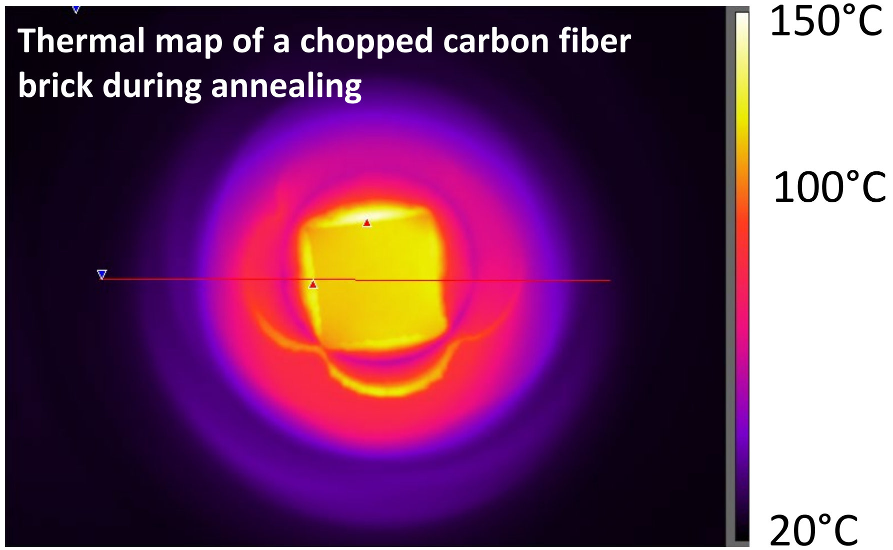
- Optimize microwave parameters for part annealing. (M1.3, Yr 1)
- Investigate the parameters necessary to anneal a part using RF magnetic induction (M1.3)
- Optimize formulation of 3D-printed nylon-susceptor part (M1.3)
- Develop the code for printing sensors and test coupon on a single printer utilizing multiple heads (M1.4)



SEM images of dried inks made from (A) Graphene oxide and (B) Graphene oxide coated onto carbon nanotubes.

FY22 Proposed Research

- Incorporate continuous carbon fiber tow within a SMART coupon, printed on a single 3D printer (M2.1)
- Formulate high-temperature thermoplastic test parts using PEEK/PEKK base polymer (M2.1)
- Determine process energy used in making a part including post-annealing via conventional heating and EM susception. (M2.2)



Any proposed future work is subject to change based on funding levels.

Summary

- Susceptor-filled 3D-printed nylon parts can be EM annealed under **low power** and for much shorter periods of time compared to traditional heating and show marked improvements in tensile strength and thermal conductivity.
- Incorporating continuous carbon fiber tow within a 3D printed part strongly improves thermal conductivity.
- Incorporating carbon nanotubes into the filament improves microwave susception.
- Resistive strain gauges have been printed out of several different materials aiming to optimize for resolution and ruggedness.
- Thermocouple sensors have been printed into arrays on test coupons.

Milestone Name/Description	Deadline	Status
M1.1: Demonstrate proof of concept of significant mechanical/thermal property enhancements using EM coupled heating.	12/31/20	Complete
M1.2: Provide final SMART sensor design for integration into 3D printing.	3/31/21	Complete
M1.3: Provide sufficient evidence of >10% increase in tensile strength and > 10% increase in thermal conductivity for small test coupons.	6/30/21	On Track
M1.4: Demonstrate SMART sensor integration of at least two sensors into a test coupon.	9/30/21	On Track
Phase 1 (Yr 1) Go/No-Go milestone: Demonstrate > 20% higher modulus/strength and thermal conductivity with at least one integrated SMART sensor.	9/30/21	On Track