Carbon Fiber Technology Facility

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Outline

- Overview
- Relevance
- Milestones
- Approach
- Technical Accomplishments and Progress
- Response to Previous Year Reviewers/ Comment
- Collaboration and Coordination with Other Institutions
- Remaining Challenges and Barriers
- Proposed Future Research
- Summary

Overview

Timeline

- Capital project completed March 2013 (ARRA funded)
- Operations from March 2013 to present

Barriers Addressed

- Cost of carbon fiber manufacturing
- Technology scaling
- Process Validation
- Workforce development

Budget

	FY 16	FY 17	FY 18	FY 19	FY 20	FY 21	FY 22
Total Budget	5.5 M	5.3 M	6.0 M	6.0 M	7.8 M	6.2 M	6.0 M
VTO	1.5 M	1.3 M	1.0 M	1.0 M	1.0 M	1.0 M	1.0 M
AMO	4.0 M	4.0 M	4.0 M	4.0 M	5.0 M	4.0 M	4.0 M
FE			1.0 M	1.0 M	1.0 M	0.2 M	
Other					0.8 M		

Partners

- Institute for Advanced Composite Manufacturing Innovation (IACMI) > 150 Members, 30 states
- University of Tennessee
- Cooperative Research & Development Agreements
  - Project NFE-20-08145 - Advanced Carbon Products
  - University of Virginia

Relevance

CFTF serves as a national resource to assist industry in overcoming the barriers of advanced fiber cost, technology scaling, intermediate formation, and composite product and market development

BRIEFING ROOM

Executive Order on America's Supply Chains

FEBRUARY 24, 2021 • PRESIDENTIAL ACTIONS

By the authority vested in me as President by the Constitution and the laws of the United States of America, it is hereby ordered as follows:

2. Critical minerals are an essential part of defense, high-tech, and other products. From rare earths in our electric motors and generators to the carbon fiber used for airplanes—the United States needs to ensure we are not dependent upon foreign sources or single points of failure in times of national emergency.

- Bridge from Research and Development to deployment and validation of low-cost fiber
- Demonstrate advanced fiber production using lower-cost precursors
- Produce relevant quantities of fiber for evaluation, and composites market development
- Enable development of domestic commercial sources for production of fiber
- Enable an advanced fiber composite industry for high volume energy applications
- Formulate a Workforce Development program for carbon fiber and advance composites workforce

The Carbon Fiber Technology Facility CFTF

Only **Open Access** State-of-the-Art Facility in the U.S

Vehicle Technologies Office

Carbon Fiber is necessary for:

- **highest potential weight reduction, improved energy** efficiency for all vehicle types/classes
  - EV & alt fuel vehicle range extension
- Enhanced CAV sensor & communication device integration, signal transmission & EMI shielding

2017 U.S. DRIVE Roadmap Report, Section 5

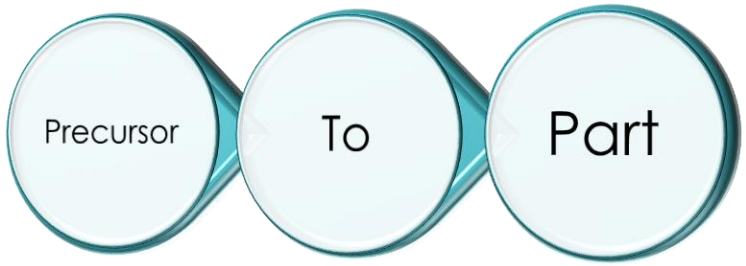
Tasks and Milestones

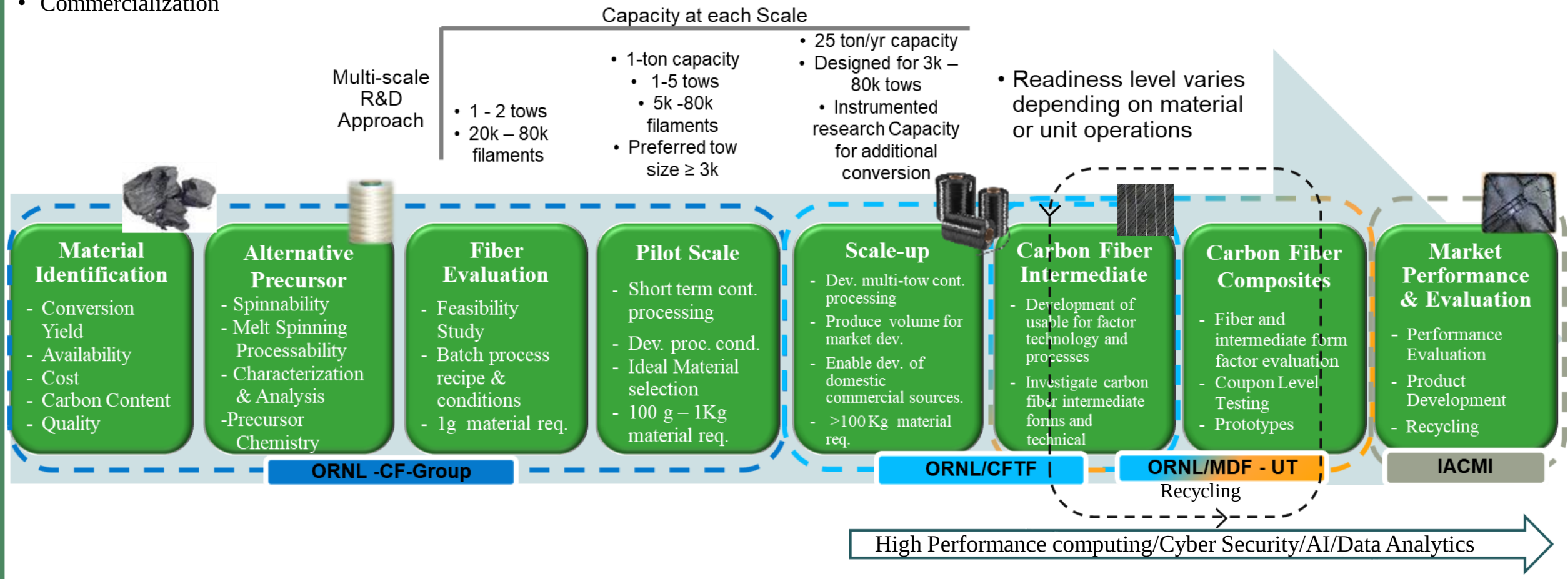
Tasks	
1.	Establish and perform collaborative R&D projects to reduce technical uncertainties associated with scaling technologies
2.	Investigate CF intermediate forms and technical challenges in composite applications
3.	Investigate potential alternative CF precursors.
4.	Establish digital data platform for process, ex-situ, and sensor driven analytics
5.	Investigate and develop in-process measurement, sensing and control methods
6.	Establish a Control Algorithm Development Framework.

	Quarter 1	Quarter 2	Quarter 3	Quarter 3
Milestones	Develop process condition for spinning nylon precursor.	The objective of this task is to demonstrate the feasibility of melt-blowing Participant's mesophase pitch using CFTF facilities.	Evaluate the performance of low-cost advanced fiber in thermoplastic resin system.	Synthesize pitch material for precursor processing .
Criteria	# of spools - 10 Diameter (um) - 24.46 Strain at Break (%) – 85.7 Strength (Gpa) – 0.45 Manu. Strength (Gpa) – 0.08 Modulus (Gpa) – 2.07	Feasibility study of melt-blowing ACP mesophase pitch at scale Characterization and analysis of batch process samples report. Provide melt spinning process condition, characterization analysis report, and video of demonstration.	Characterization & Analysis Report on thermo-mechanical, thermal, mechanical, and physical properties	Criteria for pitch material
Due date	12/31/21	03/31/22	06/30/22	09/30/22
Actual completed	04/20/22	04/04/22 – 04/05/22		
Status	100% Completed baseline for hydrogen project	25 % Completed with Isotropic pitch	On Target	

Technical Integrated Approach

- Identify high potential, low-cost alternative precursors
- Multi-scale approach to develop optimal mechanical properties of resultant carbon fiber from alternative precursors and recycled material
- Provide quantities to industrial partners for testing based on DOE approval
- Address feedback from industrial partners
- Improve carbon fiber manufacturing cost metrics
- Commercialization





Technical Integrated Approach – Integrated Capabilities

Precursor Spinning Processing

Carbon/Advance Fiber Processing

Fiber Chopper

Textile Carbon Fiber Winder

Thermoplastics

D-LFT Plasticator

RocTool induction heating

Tow-preg line

Characterization

DSC/TGA

Twin Screw Compounder

Fourier Transform Infrared

Rheometer

Automation and Molding

Injection over-molding

Automation

Ovens

500-ton fast acting press

Additive Manufacturing

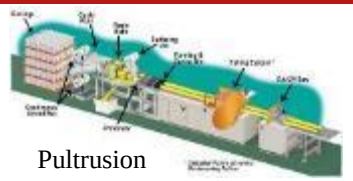
Big Area Additive Manufacturing (Thermoplastics)

Reactive Additive Manufacturing (Thermosets)

Out of Plane Deposition

Thermosets

Sheet Molding Compounding



Pultrusion

Garmut Booth

Filament winder

Recycling & Recovery

OMAX 60120 Waterjet Cutter

ZSE 27 MAX Twin-Screw Extruder

MR40120 Shredder

T5070 Granulator

Technical Integrated Approach - Alternative Precursor Materials for Carbon Fiber Manufacturing - Theoretical Reduction in Cost & Energy

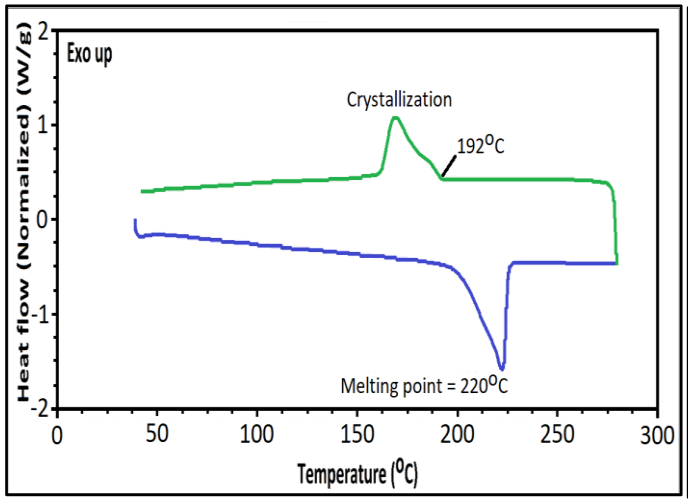
Scale	Precursor/Conversion Process	Cost Δ	Energy Δ
Baseline	**Standard PAN precursor**	0%	0%
Lab	**Melt stable PAN precursor**	- 30%	- 30%
Lab	**Lignin-based precursor**	- 50%	- 40%
Lab	**Silicon Carbide Fibers**	- 85 %	TBD
Lab	**Advanced conversion processing**	- 25%	- 50%
Lab	**Bio-PAN**	TBD	TBD
Scale-up	**Polyolefin/Polyamide precursor**	- 20%	- 50%
Scale-up	**Pitch-based precursor**	- 70%	- 70%
Scale-up	***Textile PAN precursor >30 variations**	*Theoretical **- 25%** Actual **- 54%**	Theoretical **- 30%** Actual **- 41%**
Scale-up	**Recycled CF**	- 60%	- 90%

*Sources: Das, S. and Warren J., "Cost Modeling of Alternative Carbon Fiber Manufacturing Technologies – Baseline Model Demonstration," presented DOE, Washington, DC, 5 April 2012; Unpublished analysis by Kline and Co, 2007; Suzuki and Takahashi, Japan Int'l SAMPE Symposium, 2005;

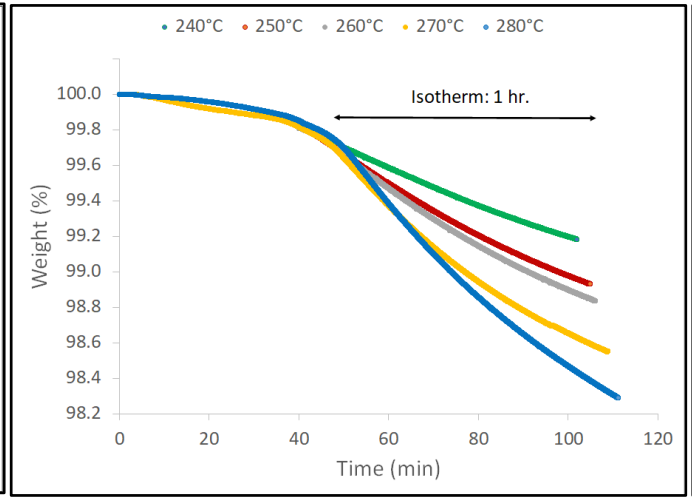
Technical Accomplishments and Results
Science of Scaling – Nylon Thermal Properties

Scale	Precursor/Process	Advantage	Disadvantage	Cost Δ	Energy Δ
Baseline	**Standard PAN precursor**	Strength, elongation, knowledge base, fiber architecture. Conversion Yield is 50%	Feedstock price and volatility, capital cost, energy, yield, processing	0%	0%
Scale-up	**Polyolefin/polyamide precursor (FY 21 Q3)**	Feedstock price and stability, spinning, yield, fiber architecture. Conversion Yield is 65 % – 75 %	Conversion process and equipment, knowledge base, capital cost	- 20%	- 50%

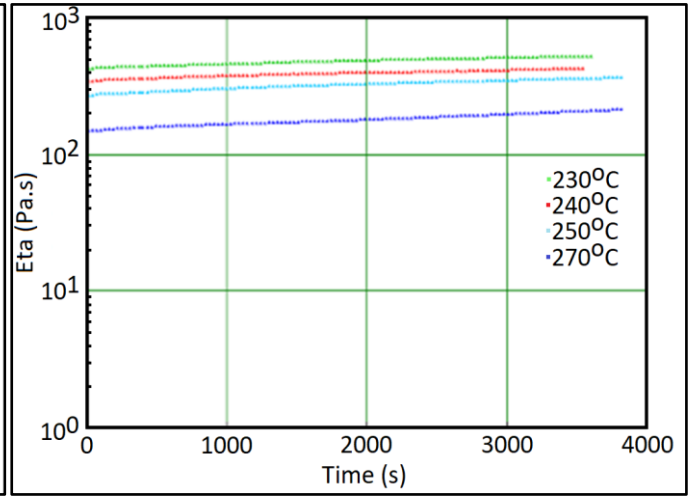
Characterization of thermal properties for melt spinning



Differential Scanning Calorimetry
Melting point



Thermogravimetric analysis in N_2
Thermal stability

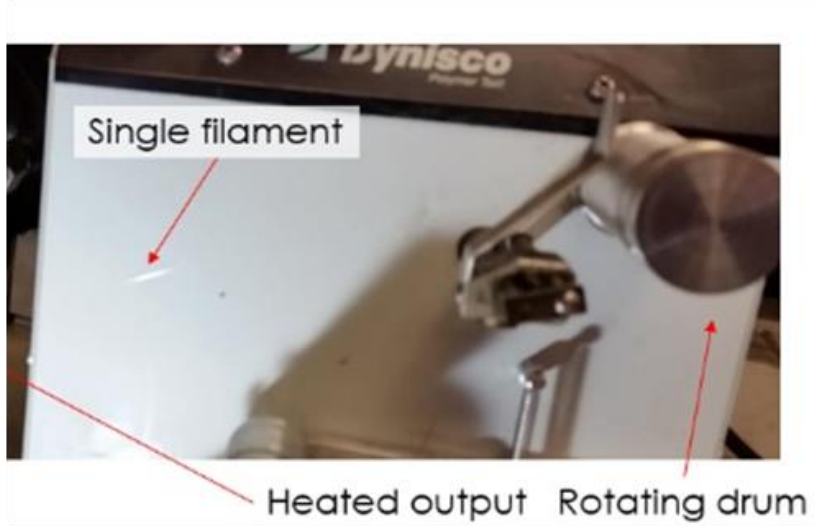


Rheology (rotational shear)
Viscosity

- Determine the starting process condition for bench scale spinning study

Technical Accomplishments and Results
Science of Scaling – Nylon Precursor Spinning Trials

Lab-scale



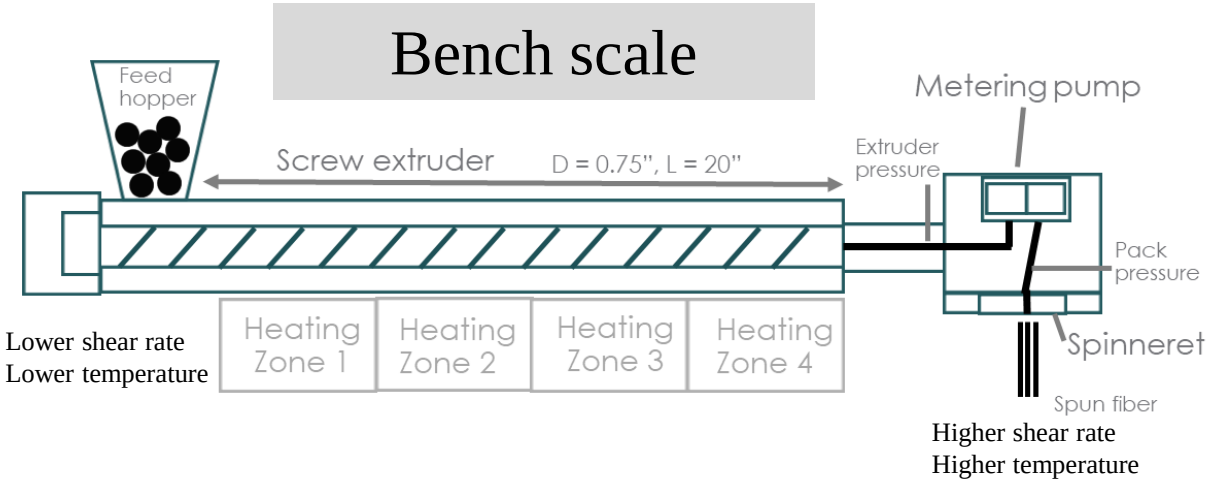
Single Filament spinning

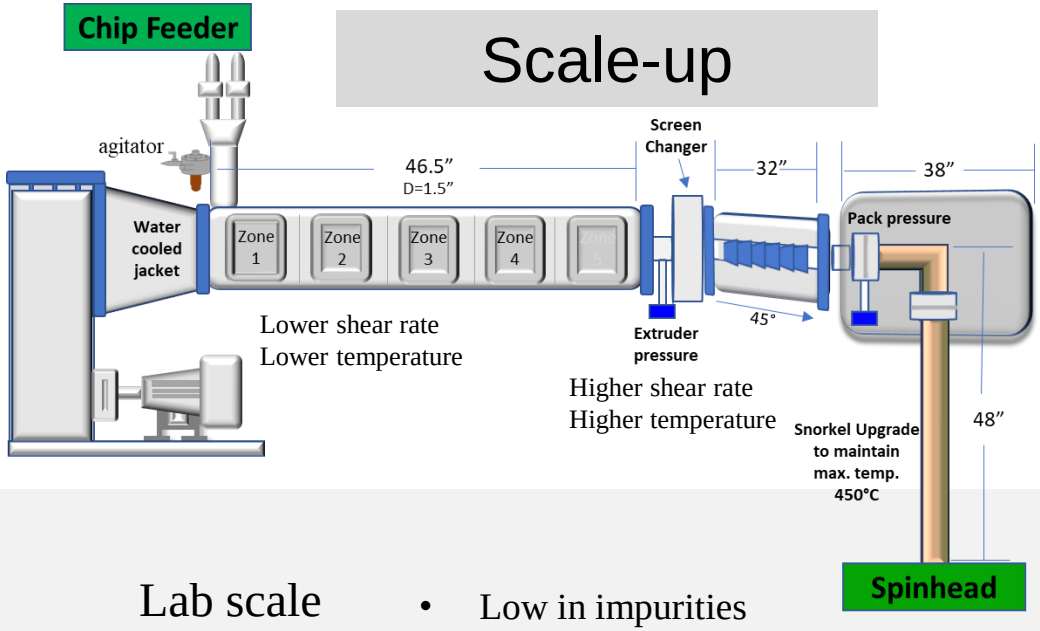
Multi-Filament spinning

Microscopy image of nylon precursor

- Develop spinning process condition/baseline for scale-up spinning study

Technical Accomplishments and Results
Science of Scaling – Bench-scale vs.. Scale-up





- Melt processing not defined by one temperature or shear rate.
  - Specific residence times and shear rates in several zones of increasing temperature.
  - Varying material properties with processing conditions.
- Challenges due to the raw material compositions.
- Uncertainty associated with scaling. Ex. Impact on equipment, volume of material)
- Consistent approach from lab material screening to scale-up:
  - Important parameters: pitch composition and microstructure, rheology (temperature, time, and shear rate dependence), thermal stability, volatilization of low molecular weight components.

Lab scale

- Low in impurities
- Stable phase behavior
- Stable viscoelasticity
- Low volatiles

Bench scale

- Consistent feed
- Controlled melting
- Stable fiber forming dynamics

CFTF scale

- Known scale effects due to temperature, residence time, shear rate, & volume of material.

Technical Accomplishments and Results
Science of Scaling – Scale-up Melt-spinning Nylon Trials

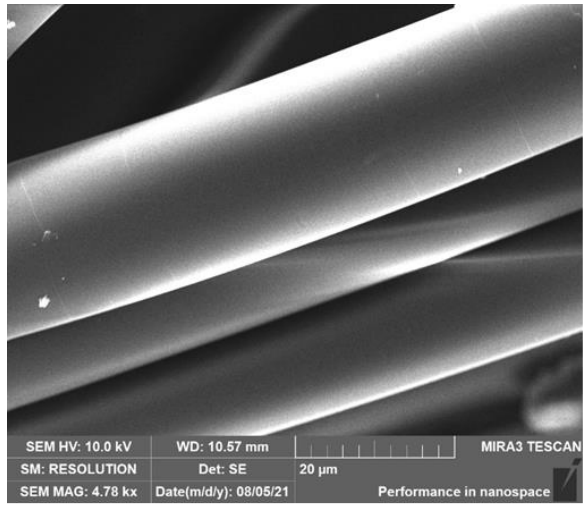
Achievement(s):

- Successfully scaling of Nylon precursor
- Develop the process spinning condition at scale.
- Baseline conditions for candidate Hydrogen Tank Project established for DE-FOA-0002229
- Target properties achieved

Technical Accomplishments and Results
Science of Scaling – Nylon Precursor Properties

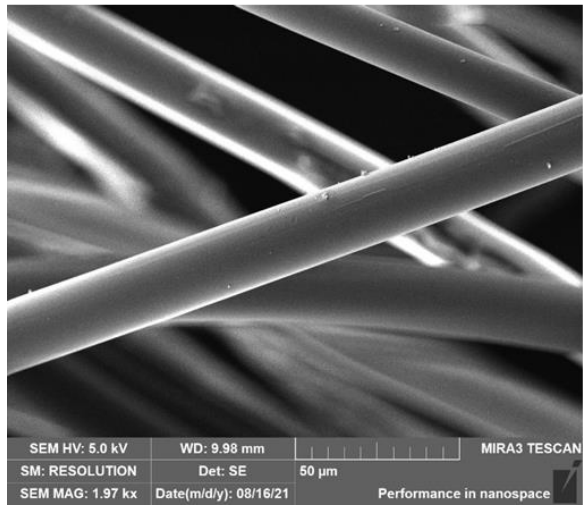
Lab-scale

Avg. Diameter (µm)	21.3 ± 4.2

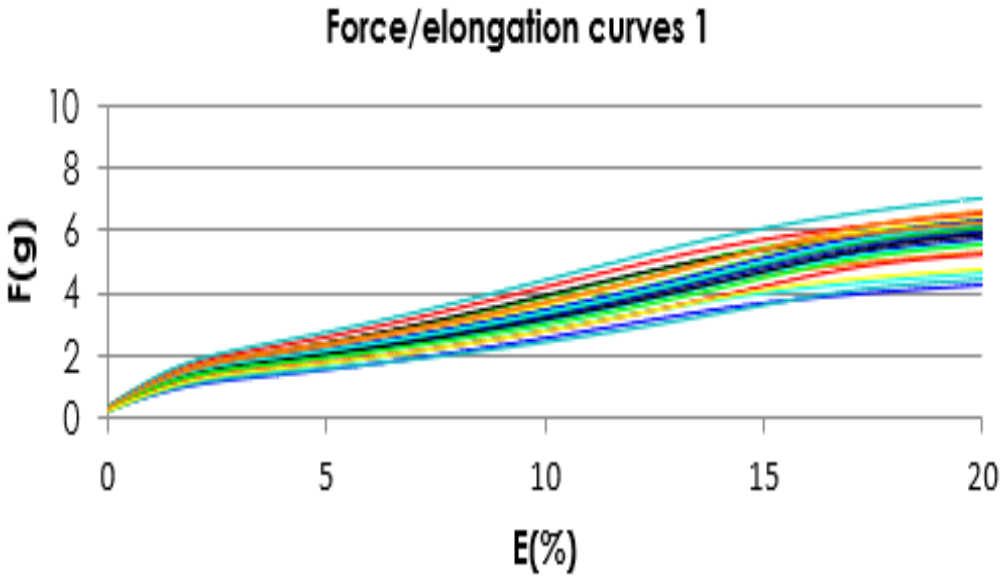


CFTF

Avg. Diameter (µm)	20.3 ± 5.3



CFTF



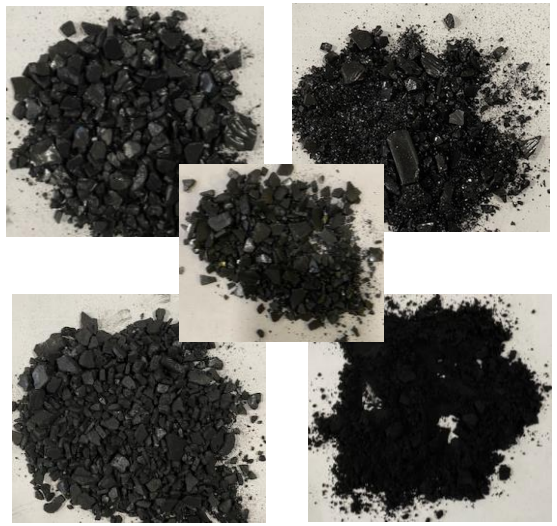
	Target	Trial 1	Trial 2	Trial 3
# of spools	10	13	22	10
Diameter (µm)	24.46	20.7 ± 4.2	20.4 ± 2.30	20.52 ± 1.82
Strain at Break (%)	85.70	103.1 ± 38.0	96.1 ± 24.35	94.4 ± 14.42
Strength (Gpa)	0.45	0.29 ± 0.09	0.36 ± 0.04	0.38 ± 0.03
Manu. Spec	0.08			
Modulus (Gpa)	2.07	3.17 ± 0.21	2.74 ± 0.49	2.13 ± 0.03

Technical Accomplishments and Results
Science of Scaling - Pelletization of Pitch Material

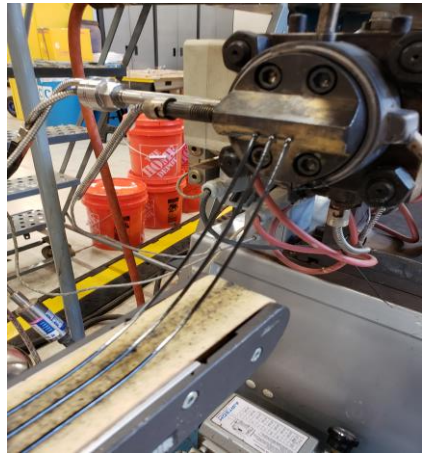
Scale	Precursor/Process	Advantage	Disadvantage	Cost Δ	Energy Δ
Baseline	**Standard PAN precursor**	Strength, elongation, knowledge base, fiber architecture. Conversion Yield is 50%	Feedstock price and volatility, capital cost, energy, yield, processing	0%	0%
Scale-up	***Pitch-based precursor**	Feedstock price and stability, spinning, yield, knowledge base, properties develop w/o stretching, moderate capital. Conversion yield for Mesophase is 80 % – 85 %	Elongation and compression strength, fiber architecture	- 70%	- 70%

Pelletization Trial

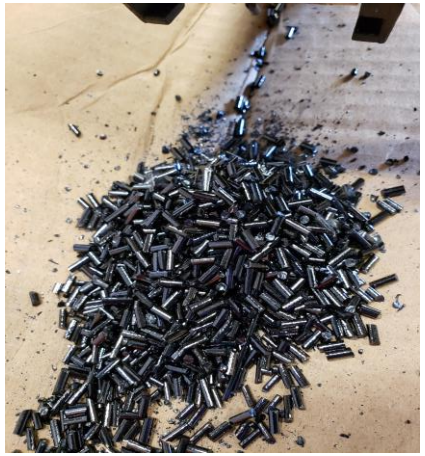
- Challenges
  - Inconsistent Quality of pitch material
- Develop Pelletization process for consistent pitch form factor and easier processing
  - Pitch material w/without additive (4 types)
  - Vary process parameters
- Evaluated impact on spinnability & properties – in progress
  - Thermal profile, Density, Molecular weight, Viscosity, and Melt Flow index
  - Impact on spinnability
  - Impact on spun fibers properties



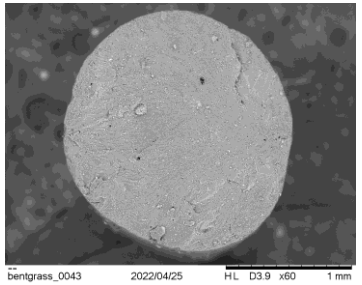
Various pitch material with various particle sizes

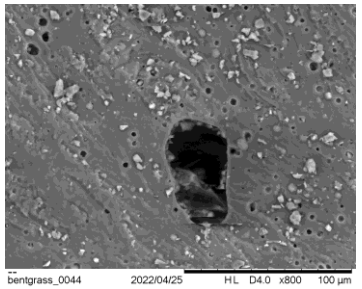


Pelletization process



Pelletized pitch material





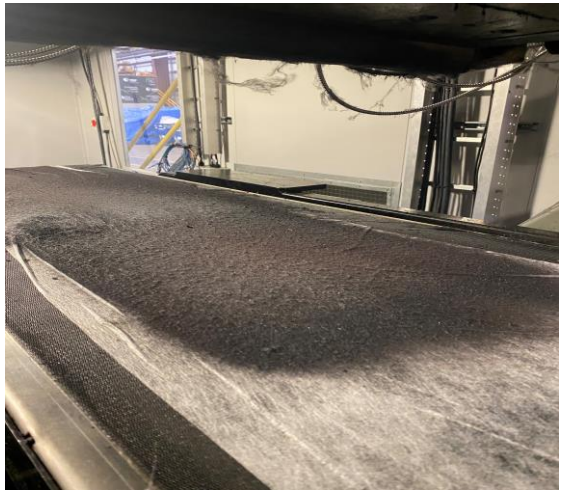
SEM images of pitch pellet

Successfully pelletization process developed

Technical Accomplishments and Results
Science of Scaling - Melt-blown Pitch Precursor at Scale

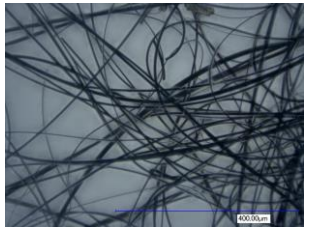
Scale-up Trials:

- Successfully scale-up of pitch precursor.
- Develop melt-blowing process conditions for spinning a new petroleum-based pitch-based material into precursor.
  - Produced: 18 kg (40 LBS)
- Develop baseline for converting the pitch-based precursor into carbon fiber – in progress



Melt-blowing process

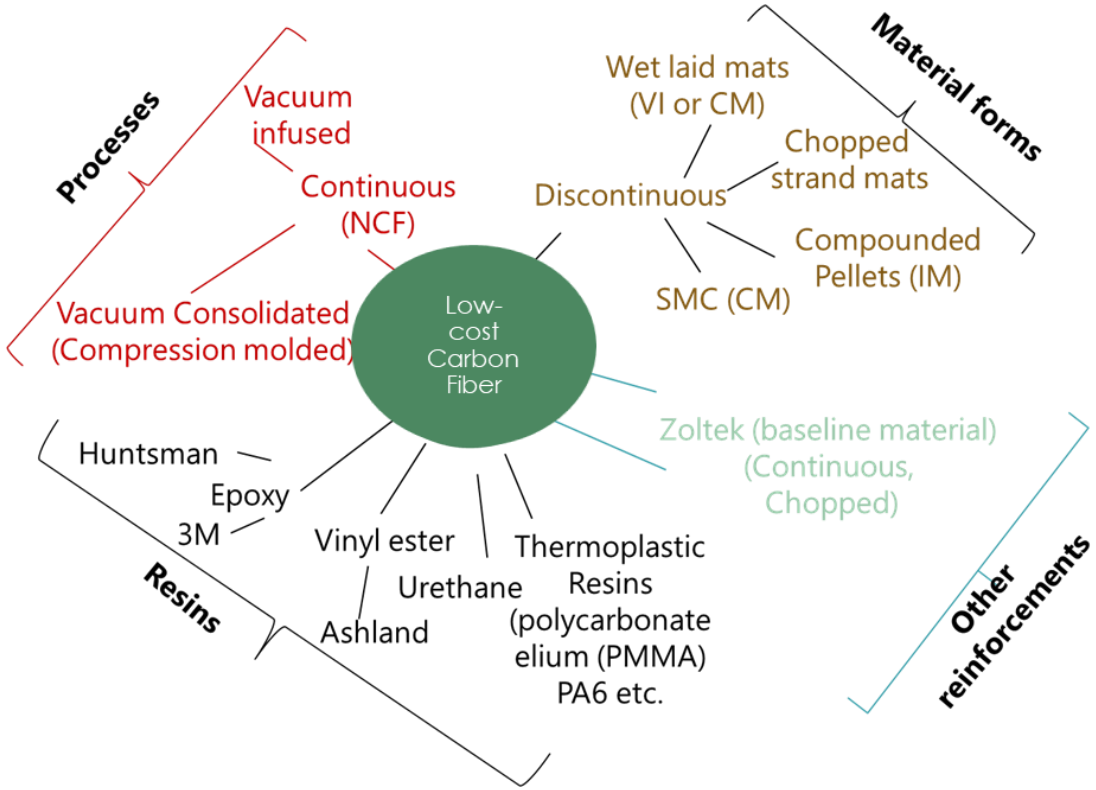
Pitch precursor Fiber spools



Microscopy images of pitch precursor

Technical Accomplishments and Results
Performance Evaluation of Textile Carbon Fibers in Thermoplastic Resin and Composite Methods

Scale	Precursor/ Process	Advantage	Disadvantage	Cost Δ	Energy Δ
Baseline	**Standard PAN precursor**	Strength, elongation, knowledge base, fiber architecture. Conversion Yield is 50%	Feedstock price and volatility, capital cost, energy, yield, processing	0%	0%
Scale-up	***Textile PAN precursor >30 variations**	Properties and knowledge base comparable to standard PAN. Energy consumption and cost reduced. Conversion Yield is 50 %	Capital cost and yield comparable to standard PAN precursor	*Theoretical **- 25%** Actual **- 54%**	Theoretical **- 30%** Actual **- 41%**

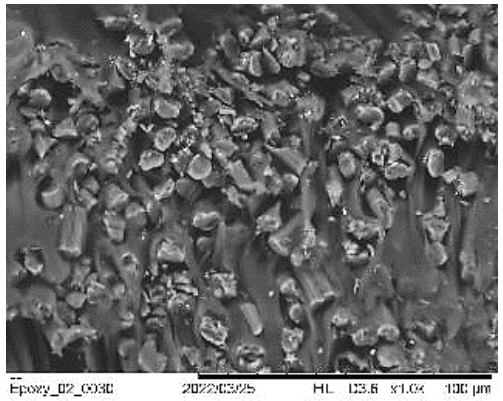


Performance Evaluation – Thermoplastic resin

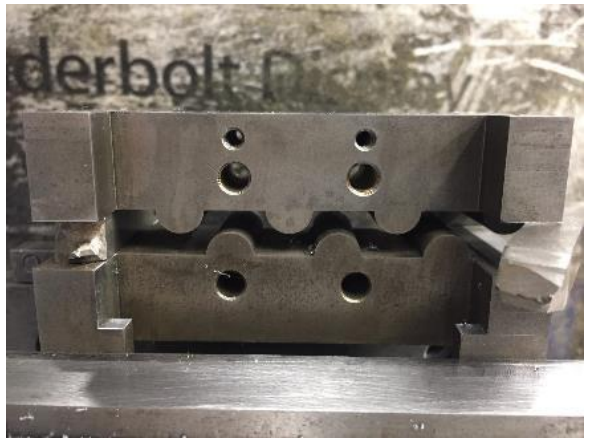
- Thermoplastic tape impregnation
- Plates made from TCF and impact testing
- Extrusion-compression molding of parts
- Extrusion-compression molding of bamboo-PP with TCF
- Injection overmolding in progress
- SMC overmolding with TCF in progress
- Plates produced from thermoplastic-PP tape scrap (Recycling/circular economy, reduced embodied energy)

Technical Accomplishments and Results - Performance Evaluation - TCF Thermoplastic Tape Impregnation

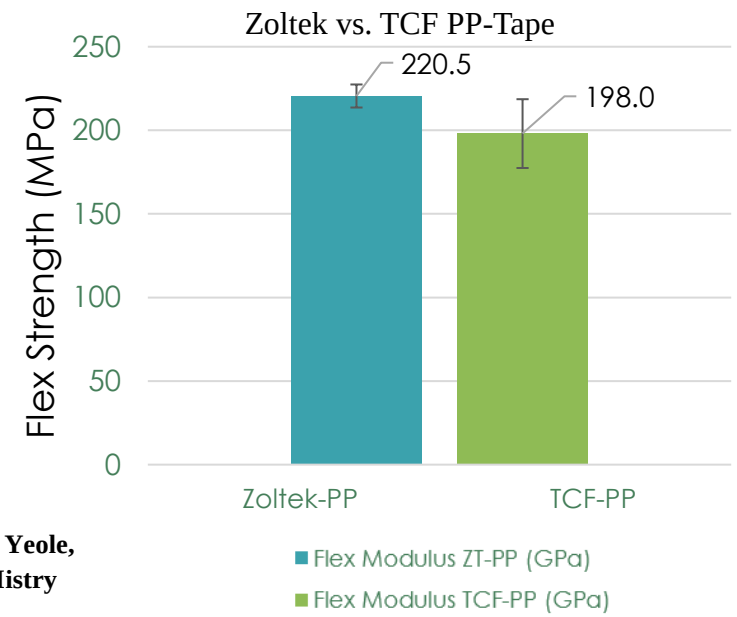
- Various updated have been incorporated since the last briefing on the thermoplastic impregnated tape
- The die designs were further modified
- The electricals were hardened and fortified
- The puller and take up system has been upgraded
- The line has been evaluated for 330k TCF tow
- About 100 m of impregnated tow has been produced
- Excess/scrap has been fully repurposed for testing

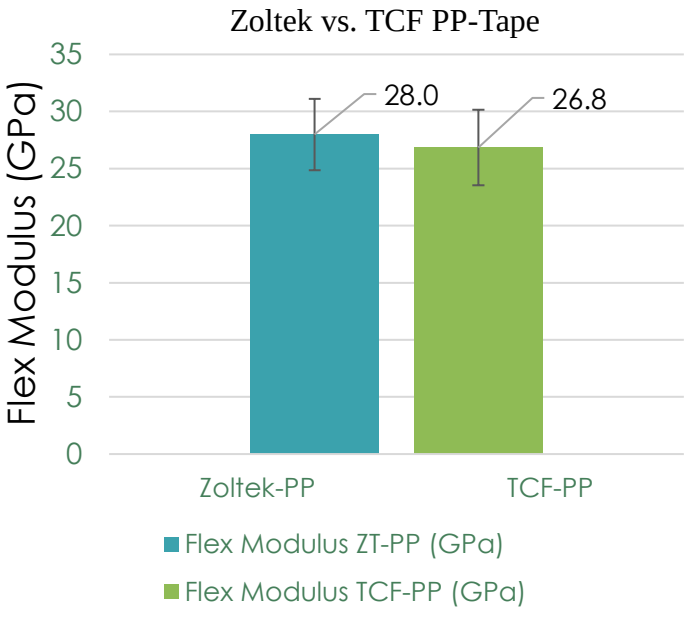


- Excellent fiber wet out
- Comparable properties to commercial fiber



Updated die design for wide tow CF





Slides 17 – 23 are provided by Vaidya Uday, Benjamin Schwartz, Sanjita Wasti, Pritesh Yeole, Saurabh Pethe, Georges Chahine, Nicolas Martin, Joel, Tonial Helmboldt, and Abhi Mistry

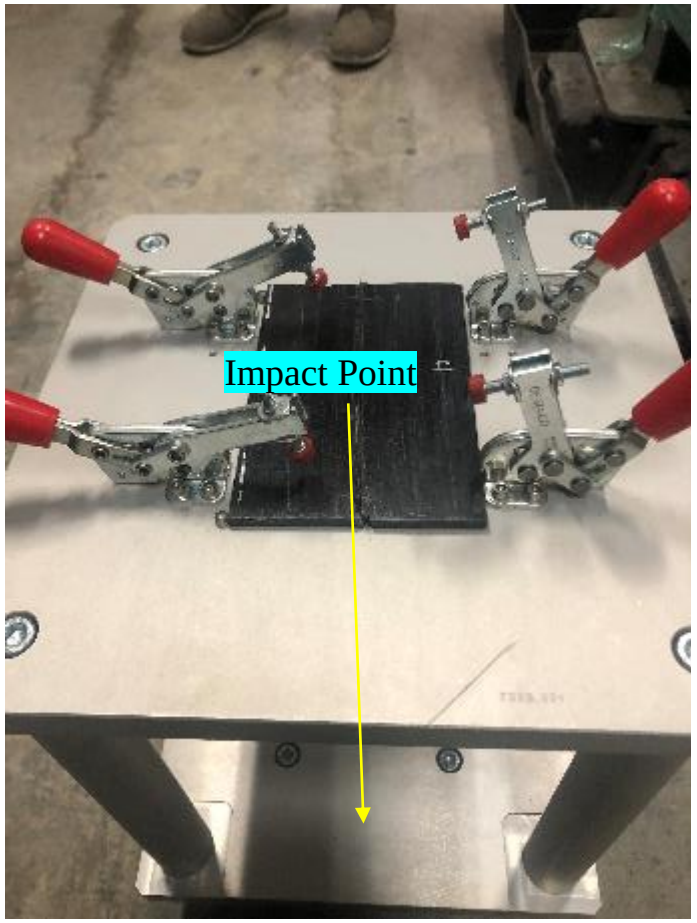
Technical Accomplishments and Results - Performance Evaluation - Impact Response of TCF-PP Thermoplastic Tape-based Plates

All plates were produced by winding tape of flat plate and compression molding

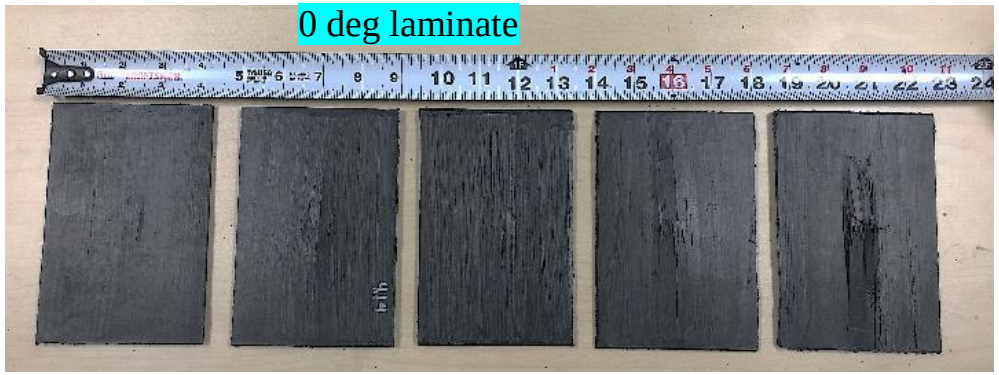
Plate Dimensions (ASTM D7136)

- 4 inches by 6 inches

Specimen mounted on the CEAST 9350 drop tower low velocity impact



- In January 2022 we added a CEAST 9350 drop weight low velocity impact tower | ASTM D7136
- The equipment is being extensively used to evaluate impact damage tolerance of TCF in composite forms.



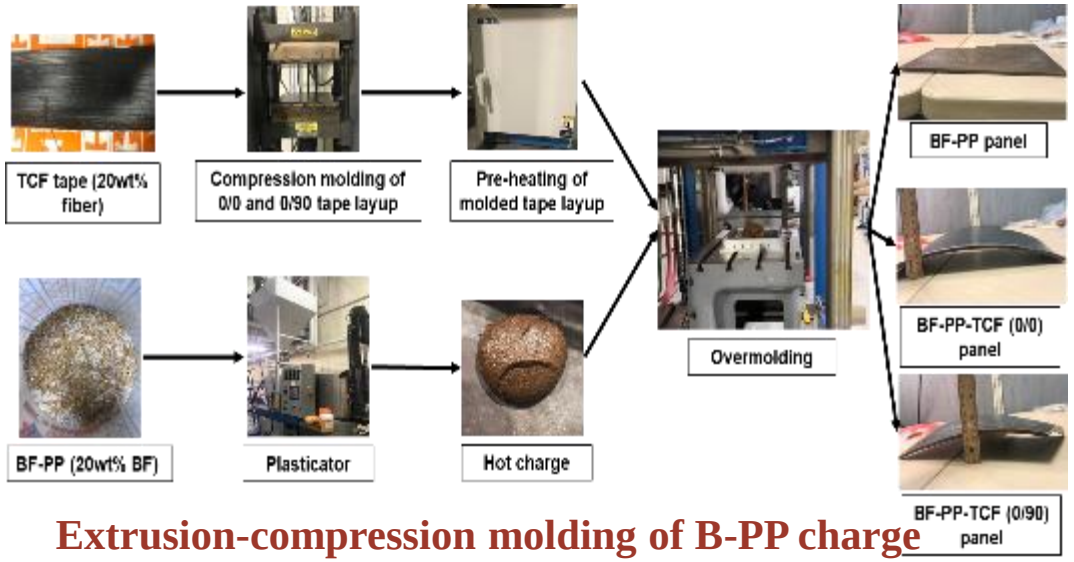
10J 15J 20J 22.5J 25J

20J 25J 30J 40J 50J

- Very repeatable impact response
- The damage tolerance of the cross-ply exceeded the uni.
- The high performance in impact was evident
- In both configurations –uni (0 deg) and 0/90

Technical Accomplishments and Results - Performance Evaluation - Sustainable Natural Materials with TCF

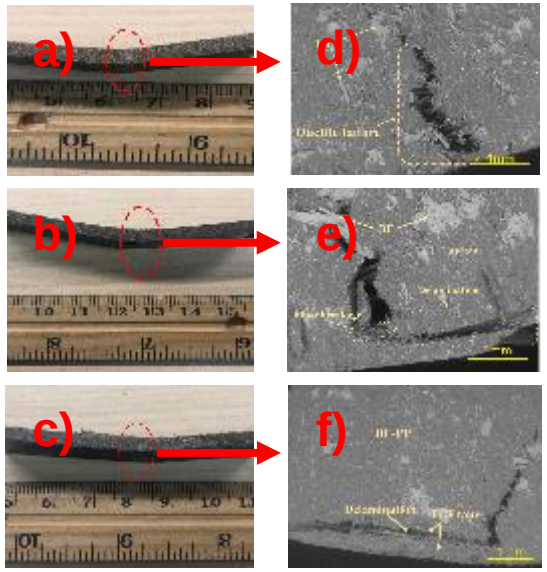
- Lower strength of natural fibers (95- 1200 MPa) compared to synthetic fibers (800 MPa to 1.2 GPa)
- Overmolding is a hybrid composites manufacturing technique where short fiber reinforced thermoplastic composites are extrusion compression molded with continuous fiber-reinforced composite tapes/laminates
- In this work, low-cost textile grade carbon fiber (TCF) thermoplastic tapes were overmolded with bamboo fiber (BF)-polypropylene (PP) for strength & modulus gains



Extrusion-compression molding of B-PP charge overmolding with TCF

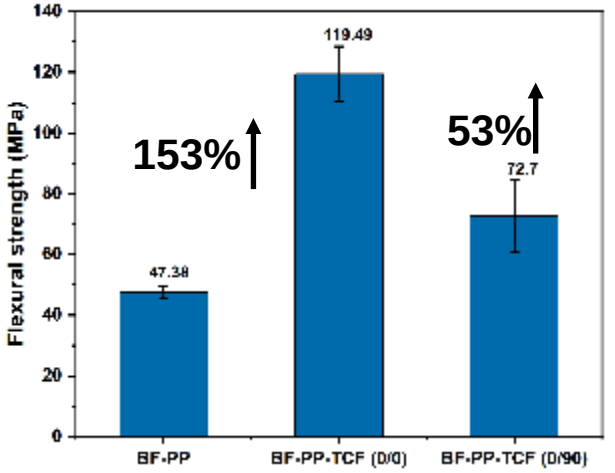
Process flow for Bamboo-PP overmolded with TCF-PP tape

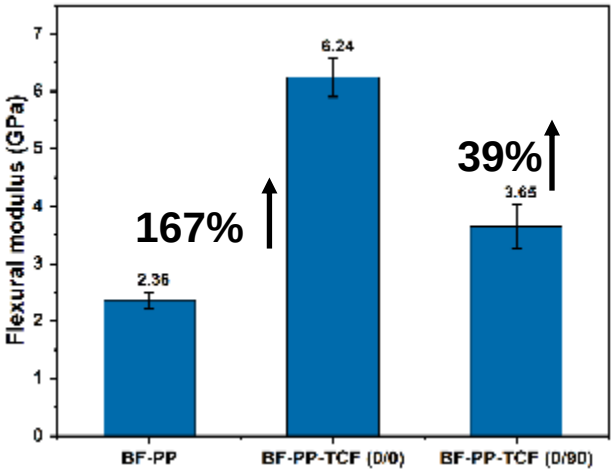
Sample	Density (g/cm^3)
BF-PP	0.9800 ± 0.0006
BF-PP-TCF (0/0)	0.9950 ± 0.0013
BF-PP-TCF (0/90)	0.9853 ± 0.0010



Excellent interface synergy of overmolded TCF with Bamboo-PP

At minimal weight penalty- the flexural strength and flexural modulus of bamboo-PP (BF-PP) overmolded with TCF

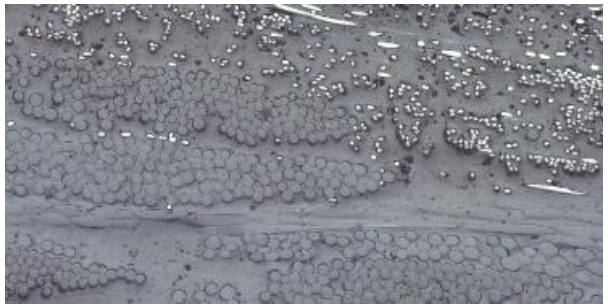




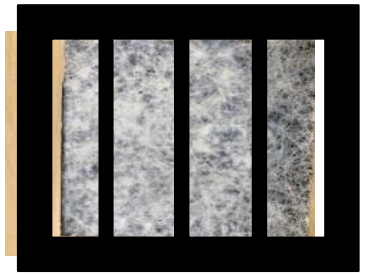
Technical Accomplishments and Results - Performance Evaluation - Multimaterial SMC with TCF Thermoplastic Local Reinforcement

- Evaluate low-cost carbon fiber (TCF) intermediates for interface properties control
- Develop intermediates such as SMC (as example) with thermoset and thermoplastic matrices with TCF
- Demonstrate representative product(s) featuring hybrid SMC-tailored local overmolding – demonstrate load bearing joint features (inserts)

Multimaterial SMC

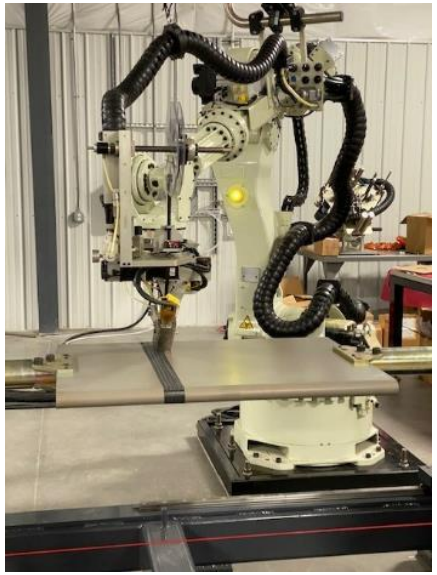


Multimaterial SMC interfaces



Selective overmolding of thermoplastic TCF with SMC for local strength enhancement

Local overmolded tape integration of discontinuous fiber forms & inserts



Automated tape placement (ATP) – Thermoset & Thermoplastic tapes

TCF-PP tape has been produced in wide width and being slit to ¼" and ½" to house in ATP for local placement

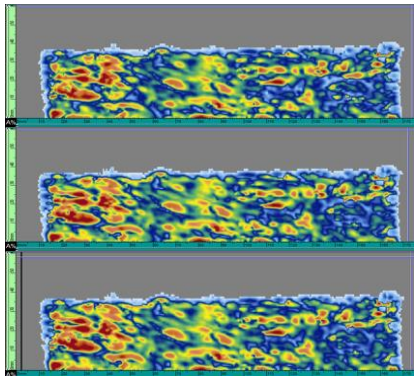
Potential application Automotive fender

Battery tray cover

Technical Accomplishments and Results - Performance Evaluation - Thermoplastic TCF – PP scrap (recycled)

Sample cut in 1” and 1.5” sizes from the first trial tape.

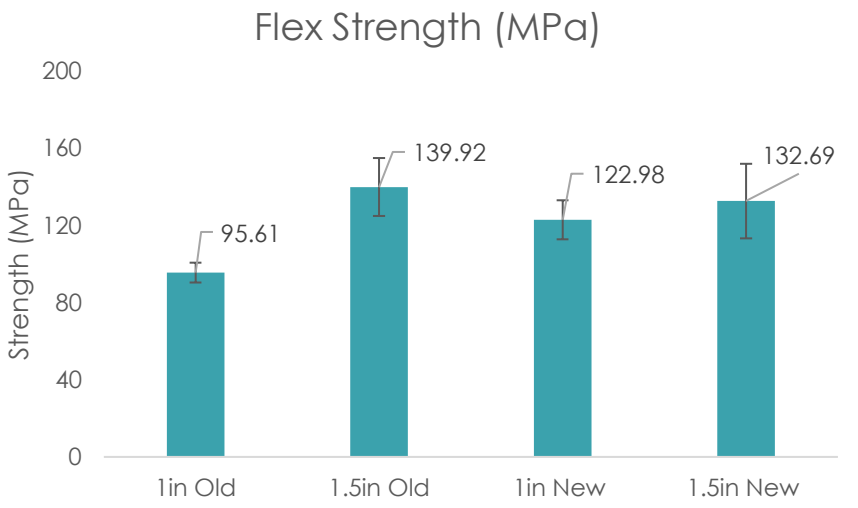
Sample soldered for better alignment of fibers

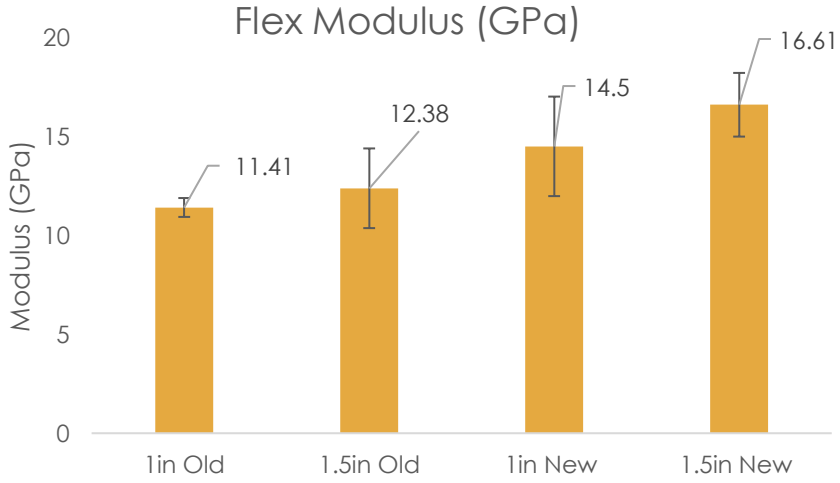


Ultrasonic C-scan of recycled TCF-PP scrap plates. Excellent definition of recycled scrap TCF-PP & orientation in the pane

Recycled scrap from TCF-PP tape production used in reprocessing into extrusion-compression molding plates

Panel dimensions: 6”X6”
Thickness: 4.5 mm





The flexure test the tape cut into 1.5 in. size performs better

R1 and R2 represent different trials with the thermoplastic line.
The R2 had more consistent wet out compared to R1

Response to Reviewers Comment @ FY 22 Peer Review

Positive Responses - Project Relevance and Resources

- 100% of reviewers felt that the project was relevant to current DOE objectives
- 0% of reviewers felt that the project was not relevant
- 100% of reviewers felt that the resources were sufficient
- 0% of reviewers felt that the resources were insufficient
- 0% of reviewers felt that the resources were excessive

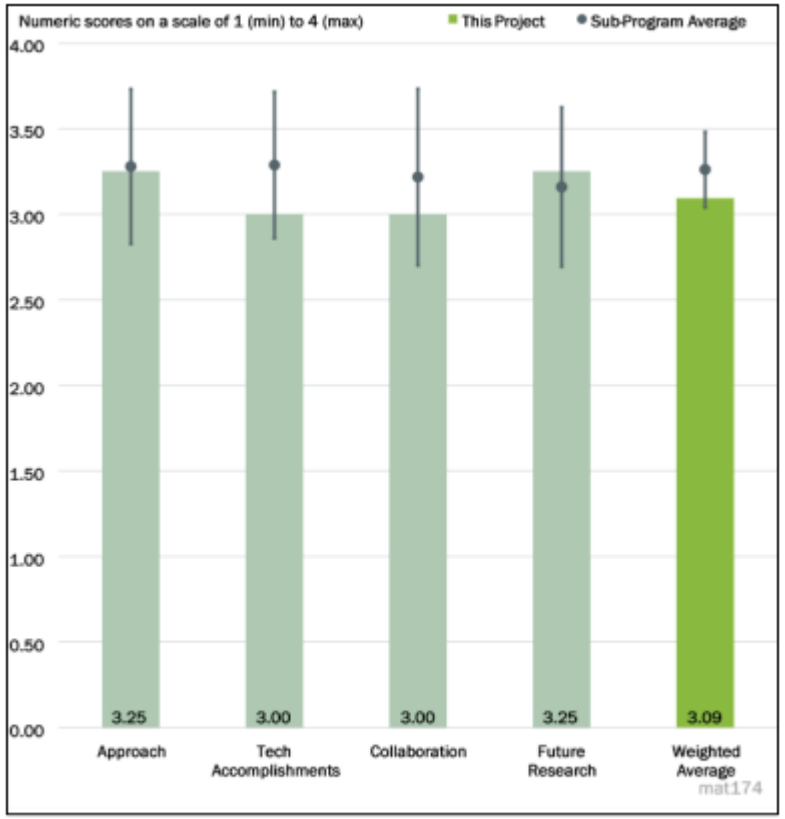


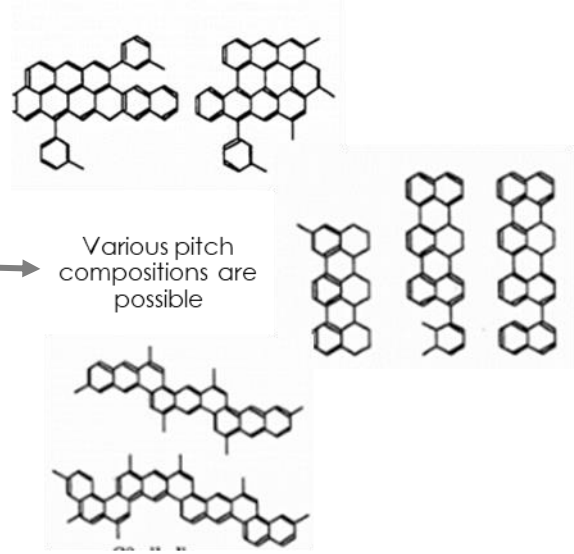
Figure 6-10 - Presentation Number: mat174 Presentation Title: Carbon-Fiber Technology Facility (CFTF) Principal Investigator: Merlin Theodore (Oak Ridge National Laboratory)

Collaboration and Coordination with Other Institutions

Partner	Project #	Project summary
Advanced Carbon Products	NFE-20-08145	ACP has developed several processes for the manufacturing of hydrocarbon precursors for advanced carbon products, including a continuous process to convert low-cost refinery oils into isotropic pitch and a continuous process to convert said isotropic pitch into anisotropic mesophase pitch . The mesophase pitch has the potential to be an excellent precursor to produce high-quality carbon fibers.
Fossil Energy Program – University of Kentucky	FE-AA155	C4WARD, coal-to-carbon fiber initiative
University of Virginia	DE-FOA-0002229	Low-Cost High-Performance Carbon Fiber for Compressed Natural Gas Storage Tanks
Ramaco Resources	NFE-19-07863	The purpose of this work is to demonstrate commercially relevant utilization of coal as a precursor for high value-added products, such as carbon fibers, foams, binder material for composites or other novel carbon products.
Harper International	PR-22-1368	Study on brake processing
Stewart Technologies	NN-22-1364	This purpose is to demonstrate a tow splitting concept for large tow carbon fiber
The Filter Shop	NN-22-1356	Develop a coating process for antiviral N-95 face masks
Premium PPE	NN-22-1353	Scaled production of Antiviral N95 mask
University of Tennessee	NN-19-1221	Spinning/Characterization of SiC Fibers
CCS/IACMI	NFE-22-09103	High Performance computing for carbon fiber manufacturing oxidation process
COI Ceramics	NFE-21-08660	Development of a Low-cost SiC Fiber

Remaining Challenges & Barriers

- **Material**:
  - Pitch
    - Availability of consistently high-quality pitch
      - Stability at temperature, time, and shear conditions, Low in solids (including ash). Low in sulfur and nitrogen,
    - Minimum material required for demonstration/Research at scale is ~100lbs.
  - Nylon
    - Find a spool core that can withstand the tension of the fiber when spun at high speeds to improve strength of fiber. The current cardboard core cannot withstand the tension and fiber must be cut off the core.
    - Very low moisture level of raw material is required to process the material into precursor fiber
- **Process:**
  - Understanding the scale-up processability of pitches based on their origin (coal or petroleum) and structure (isotropic to anisotropic)
    - Effect of isotropic and mesophase content on processing windows
    - Effect of aromatic hydrocarbons, side chains/functional groups on melt processing and oxidation-stabilization. Isotropic pitches can be particularly tricky to stabilize. Need to develop innovative methods of shortening time
  - Melt processing equipment software must be reconfigured to process higher molecular weight pitch material. Upgrades are required.
  - Availability of handling capabilities for newly developed carbon fibers
- **Application:** Understanding the critical end use requirements



Yanagida et al. Carbon 31, no. 4 (1993): 577-582.

Proposed Future Research Projects

Improve Efficiency, Throughput, and Commercialization of Low-cost Carbon Fiber from Alternative Precursor Sources

- Fiber layering through furnaces to reduce nitrogen and energy consumption per pound of fiber
- In-situ measurements for process control (e.g., fiber color, density, modulus, sheen, broken filaments)
- Increase throughput (speed & volume) while maintaining carbon fiber properties
- Petro-based, Coal, and Synthetic Pitch Development for Carbon Fiber and activated carbon from a variety of raw material sources
- Create a criteria for Isotropic Pitch/Mesophase Pitch to understand the chemistry of the various types of pitch.
- Create a criteria for Initiate fundamental studies for both Pan-based, pitch-based, and polyamide-based carbon fibers to develop molecular orientation during the spinning and manufacturing process.

Expand industry partnerships through CRADAs, User agreements, and Tech collaboration projects for scale-up of alternative precursors for low-cost carbon fiber manufacturing

Development of skilled carbon fiber and composites technical professionals

Proposed future work is subject to change based on funding levels

Summary

- **Relevance**: The Carbon Fiber Technology Facility is relevant in proving the scale-up of low-cost advanced fiber precursor , carbon fiber materials, and advanced manufacturing technologies.
- **Approach:** An integrated multi-scale approach expedites the progress of scaling up technology and reduces the risk and technical uncertainties associated with scaling.
- **Collaborations:** Established a couple technical collaboration projects with industry designed to help create market pull for low-cost fiber materials
- **Technical Accomplishments:**
  - Successfully scaled nylon precursor fiber that met target properties requirements
  - Precursor spinning baseline for a phase 2 candidate hydrogen tank project was established
  - Pelletization process was developed for pitch material ensuring easier processability in melt spinning/blowing process
  - Successfully scaled industry partner scaled pitch material
  - Performance evaluation of textile carbon fiber in polypropylene resin was demonstrated
    - Significant advances have been made on thermoplastic TCF tape
    - The TCF thermoplastic tape has been used in overmolding and successfully demonstrated
    - Laminates from the TCF thermoplastic tapes exhibit superior impact properties
    - Overmolding concepts have included SMC and Automated Tape Placement (ATP)
    - Injection and extrusion-compression molding have been focus of interest in this period.
    - Recycled Textile carbon fiber=polypropylene scrap has promise in secondary parts

Summary

- **Technical Accomplishments:**

	Authors	Title	Journal
1	Uday Vaidya, Dayakar Penumadu, Vlastimil Kunc, Raymond Boeman, Brian Knouff, Cliff Eberle and Merlin Theodore	Intermediates, processes and product routes for low-cost textile grade carbon fiber	Submitted - SME Journal of Manufacturing Processes.
2	Brian Knouff, Merlin Theodore, Uday Vaidya.	Carbonization stages in processing of carbon-carbon composites	Draft in progress
3	Uday Vaidya, Saurabh Pethe, Benjamin Schwartz, Merlin Theodore.,	Textile grade carbon fiber tape impregnation	submitted - Journal of Thermoplastic Composites (Oct 2021)
4	Surbhi Kore, Sanjita Wasti, Merlin Theodore, Uday Vaidya.	Natural fiber – Textile grade carbon fiber hybrid composites for automotive applications	submitted - Composites Part B: Engineering (Nov 2021).
5	Uday Vaidya, Merlin Theodore	Technology Roadmap on Low-Cost Carbon Fiber Composites and IACMI elements	submitted -Special Issue, J Comp B (Jan 2022).
6	Uday Vaidya, Merlin Theodore, and Dayakar Penumadu	Intermediates, Processes and Product Routes for Low-cost Textile Grade Carbon Fiber Into Composites	Accepted SAMPE Journal March - April 2022 Issue
7	Uday Vaidya, Mark Robinson, Nitilaksha Hiremath, Pritesh Yeole, Merlin Theodore, Ahmed Hassen, and John Unser.	Multi-process Tooling For Discontinuous Carbon and Hybrid Glass Fiber and Thermoplastics	Accepted 4/26/22

Summary

- **Future Work**:
  - Expand industry partnerships through CRADAs, User Agreements, and Tech collaborations for scale-up of alternative precursors for low-cost advanced fiber manufacturing
  - Identify, research, develop and demonstrate scale-up of new advanced fibers
  - Improve Efficiency, Throughput, and Commercialization of Advanced Fiber Manufacturing Processes
  - Conversion of recently scaled precursor fiber into carbon fiber
  - Performance Evaluation of aforementioned caron fiber in a variety of resin systems and composite methods
  - Life Cycle Analysis of Petroleum Pitch Carbon Fiber Manufacturing
  - “Proposed future work is subject to change based on funding levels”