Title: Carbon Fiber Technology Facility (CFTF)

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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 Slides

Overview

Timeline

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



Budget

	FY16	FY 17	FY18	FY 19	FY20
Total Budget	5.5 M	5.3 M	6.0 M	6.0 M	7.0 M
VTO	1.5 M	1.3 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
Other			1.0 M	1.0 M	1.0 M

Barriers Addressed

- Cost of carbon fiber manufacturing
- Technology scaling
- Process Validation
- Workforce development
- USDRIVE Materials Technical Team Roadmap, October 2017, sections 4 and 5

Partners

- Institute for Advanced Composite Manufacturing Innovation (IACMI) 150 Members, 30 states
- Technical Collaboration Projects & CRADA's
 - Field Work Proposal FEAA155 Fossil Energy Program FWP University of Kentucky
 - Project NFE-19-07863 Ramaco
 - Project NFE-20-08145 Advanced Carbon Products

Relevance

<u>The Carbon Fiber Technology Facility CFTF</u> Only **Open Access** State-of-the-Art Facility in the U.S



The Carbon Fiber Technology Facility is relevant in proving the scale-up of low-cost carbon fiber precursor materials and advanced manufacturing technologies

- Bridge from Research and Development to deployment and validation of low-cost fiber
- Demonstrate carbon 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 a carbon fiber composite industry for high volume energy applications
- Formulate a Workforce Development program for carbon fiber and advance composites workforce

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



Milestone

5

	Milestones			
Task/Objective	1 st Quarter	2 nd Quarter	3 rd Quarter	4 th Quarter
Investigate potential alternative CF precursors	Demonstrate spinnability of pitch material less than 20 microns in melt blown or fully drawn precursor yarn form.	Demonstrate spinnability of a minimum of 2 variations of pitch material into precursor.	Evaluate and report the mechanical properties, micro-structure, diameter, density, and stabilizing temperature of the pitch precursor(s).	Demonstrate the ability to stabilize the pitch precursor in line through CFTF oxidation process and Determine the thermal profile of the stabilize precursor using DSC.

*Pitch-based precursor offers the highest potential cost and energy reduction potential

Precursor/ Process	Advantage	Disadvantage	Conversion Yield	Cost A	Energy Δ
Standard PAN precursor	Strength, elongation, knowledge base, fiber architecture	Feedstock price and volatility, capital cost, energy, yield, processing	50%	0%	0%
Textile PAN precursor	Properties and knowledge base comparable to standard PAN. Energy consumption and cost reduced	Capital cost and yield comparable to standard PAN precursor	50%	-25%	-30%
Melt stable PAN precursor	Throughput and energy in spinning, strength, elongation, fiber architecture	Same as standard PAN, but higher energy productivity and lesser knowledge base	50%	- 30%	- 30%
Bio-PAN	Renewable; pricing decoupled from oil	Knowledge base, scale	TBD	TBD	TBD
Polyolefin precursor	Feedstock price and stability, spinning, yield, fiber architecture	Conversion process and equipment, knowledge base, capital cost	65% – 75%	- 20%	- 50%
*Mesophase Pitch-based precursor	Feedstock price and stability, spinning, yield, knowledge base, properties develop w/o stretching, moderate capital	Elongation and compression strength, fiber architecture	80% - 85 %	- 70%	- 70%
Lignin-based precursor	Feedstock price and stability, renewable domestic feedstock	Mechanical properties, yield, processing, knowledge base	35% - 45%	- 50%	- 40%
Recycled CF	Cost, energy, capital cost, yield, fiber architecture (future)	Feedstock availability, fiber architecture (current), knowledge base, risk	N/A	-60%	-90%
Advanced conversion processing	Speed, energy, capital cost	Knowledge base, risk	N/A	- 25%	- 50%

²Sources: 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'I SAMPE Symposium, 2005;

Technical Approach

Why Pitch?

- Substitution of polyacrylonitrile by pitch is more economical route to carbon fiber via melt spinning and conversion processing.
- Pitch carbon fiber (PCF) potentially offer the lowest cost/stiffness among carbon fiber, hence a compelling value in cost-sensitive, stiffness-driven, high-volume applications.
- Low thermal expansion, high thermal conductivity, electrical conductivity and low friction.



• A significant reduction potential in coal pitch carbon fiber embodied manufacturing from a Lower raw material embodied energy and Higher process yield

- Manufacturing Energy Carbon Fiber -1188 MJ/kg (PAN) vs. 770MJ/kg (Coal)
- Lower Raw Material Embodied Energy Acrylonitrile (90 MJ/kg)1 vs. Tar (46 MJ/kg)1,2
- Higher Process Yield Raw Precursor

Source: ¹SimaPro 7.3..3 (LCA Software)²Liu, X. and Yuan, Z. (2016). "Life Cycle Environmental Performance of By-Product Coke Production in China," Journal of Cleaner Production," Vol. 112, 1292-1301



Technical Approach

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

Multiscale

R&D Approach

Material

Identification

Conversion

Availability

- Carbon Content

Yield

Quality

- Cost

• Improve carbon fiber manufacturing cost metrics

Alternative

Precursor

Spinnability

Evaluation

Melt Spinning

Processability

& Analysis

- Characterization



- 3/5 Pitches were successfully melt blown into Carbon Fibers
 - AR Mesophase synthetic from naphthalene
 - L3 Mesophase petroleum derived
 - Rain Carbon 270 Isotropic petroleum derived
 - Conoco pitch petroleum derived
 - Koppers pitch coal derived isotropic pitch
- Rheology was performed to determine optimum blowing conditions.
- Samples were blown into a mat
- Oxidation was optimized
- Samples carbonized and then mat was measured for thermal insulation

AR Mesophase Rheology L3 Mesophase Rheology Eta* Mesophase-test-1-355C-N2 . hase-test-1-3450-N . 10 Eta' Mesophase-test-1-335C-N2 . L3-MP-test-1-320C-N2 . Mannennas-test-2:335/ 104 . L3-MP-test-1-330C-N2 L3-MP-test-1-340C-N2 103 Eta* (>) 10 Eta" (A) 10 10 ETA* Isotropic-270-test-1-320C-N2.RSD0 10⁰ . Isotropic-270-test-1-310C-N2.RSD0 . 500.0 1000.0 1500.0 2000.0 0.0 2500.0 Isotropic-270-test-1-300C-N2.RSD0 10' . 500.0 1000.0 1500.0 2000.0 0.0 2500.0 time [s] time [s] 10 Rain Carbon 270 Eta* (▲) [Pa-s] 10 Rheology 10 101 500.0 1000.0 1500.0 2000.0 0.0 2500.0

- During blowing, multiple spinning conditions were tested, based on initial conditions suggested from rheology.
- This was conducted for each sample of pitch
- For example, with the AR mesophase pitch, 3 fiber mats were prepared and then measured for thermal insulation.

Results indicate similar thermal conductivities

	B1	B2	B3	\triangleright
	[°C]	[°C]	[°C]	
Air Temp	473	476	466	
Needle Temp.	356	357	346	
Front Temp.	355	345	335	
Mid. Temp.	325	325	325	
Back Temp	320	319	319	
Feed Temp	100	100	100	
Packed Density [g/cc]	0.048	0.063	0.053	
Thermal Conductivity [W/mK]	0.0385	0.0339	0.0385	

Scanning Electron Microscopy Analysis







• Thermal Analysis with cooperation with Buildings Technology group at ORNL

Pitch	Mat Density	Thermal Conductivity	
	[g/cm ³]	[W/mK]	
AR Mesophase	~0.05 ¹	0.034	
L3 Cytec Mesophase	~0.05 ¹	0.039	
Rain Carbon Isotropic Pitch	~0.051	0.033	

- Results show the thermal conductivity is all remarkably close
- Results are also very similar to that of commercial building insulation
- Carbon fibers also do not burn

- Air Permit approved by Tennessee Department of Environment and Conservation Division of Pollution Control on 2/24/20.
- Hazardous waste evaluation completed
- Export control evaluation completed
- Secured a supply of pitch material
- CFTF upgrades/modifications
 - Melt-blow kit temperature capability upgrade in procurement system
 - TOX compliance kit- in procurement system
 - Controls upgrades first milestone payment made
 - HT Belt plates material upgrade first milestone payments made
 - Conveyor Belt upgrade for faster speeds completed
 - Implementation delayed due to COVID-19 will be scheduled immediately upon returning to normal work conditions

Front-end creel for processing precursor in tow format	Multiple flow regimes in oxidation	Flexible posttre for various resin :	systems	
n-line melt spinning for precursor development (lignins, polymers)	Belt conveyance for processing precursor in web format	-temperature furnace up to 1,000°C	Winding and packaging High-temperature furnace up to 2,000°C	CFTF Melt-spinning and Carbon Fiber Line

STATE OF TENNESSEE AIR POLLUTION CONTROL BOARD DEPARTMENT OF ENVIRONMENT AND CONSERVATION NASHVILLE, TENNESSEE 37243	Ť
Operating Permit (Conditional Major) Issued Pervaset to	Tenaessee Air Quality Act
Date landed. Fromary 24, 2020	Pennal Manifert
Date Explana: October 1, 2029	474951
	Facility ID: 73-1108
Isrued To:	Installation Address
U.S. Dept. of Eastryy, Office of Science	95 Palladium Way
Carbon Fiber Technology Facility UT-Banelle, LLC – Operator	Oak Rulge
installation Description	Emission Source Reference No.
Carbon Fiber Precess Line, Thermal Oxidizer as Centrol	73-1108-01
	Conditional Major

Response to Reviewers' Comments

There are no recent reviews or reviewers' question to address

Technical Collaboration Projects

Partner	Project #	Project summary			
Not yet started					
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	The Consortium on Coal-based Carbon Materials Manufacturing (COAL-MAT) is a collaborative Field Work Proposal (FWP) involving coordinated research and technology development between national laboratories, universities, and industrial stakeholders.			
DOE FOA 2197 DOE FOA 2203 DOE FOA 2229		Partnered with 7 industry partners Partnered with 1 industry partners Partnered with 4 industry partners			
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.			
Alberta Innovates	Draft completed	The latter are amongst the world's largest hydrocarbon resources, with proven reserves of approximately 170 billion barrels and current production of 3 million barrels per day. The purpose of the work is to accelerating the development of large-scale production pathways for carbon fiber from bitumen-derived asphaltenes and promoting their use in manufactured products.			

Remaining Challenges and Barriers

- Availability and amount of mesophase pitch for isotropic precursor required at SCALE
- Availability of isotropic with a high enough softening point to allow for stabilization at Scale. Currently there is only one source.
- Quality of Pitch material
 - Insoluble in coal derived materials can have ash and sulfur and thus create difficulty in processability via melt spinning/blowing
 - Variations in pitch material
 - Amount of sulfur in the petroleum pitch

Proposed Future Research

Improve Efficiency, Throughput, and Commercialization of Pitch based Carbon Fiber:

- 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
- 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- and pitch-based carbon fibers to develop molecular orientation during precursor processing, oxidation, and carbonization process.

Expand industry partnerships through CRADAs for scale-up of alternative precursors for low-cost carbon fiber manufacturing

Life Cycle Analysis of Pitch Carbon Fiber Manufacturing

• Techno Economic analysis

Development of skilled carbon fiber and composites technical professionals

Any 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 carbon fiber precursor materials and advanced manufacturing technologies.
- Approach: A 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 pitch-based carbon fiber
- Technical Accomplishments:
 - Demonstrated a full year of operations with zero accidents or environmental non-compliances
 - Successfully melt-blown pitch material from various sources
 - Obtain air permit for processing pitch fibers at CFTF
 - Hazardous and export control evaluation completed at Scale
 - Procured required upgrades associated with scaling pitch base carbon fibers for implementation at CFTF.
- Future Work:
 - Expand industry partnerships through CRADAs for scale-up of alternative precursors for low-cost carbon fiber manufacturing
 - Improve Efficiency, Throughput, and Commercialization of Pitch based Carbon Fiber
 - Life Cycle Analysis of Pitch Carbon Fiber Manufacturing

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

ORNL Team

CF & Materials and Processing Team



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Composites

Discussion



Technical Back up Slides

Carbon Fiber Processing



Technical Results and Accomplishments

PARAMETER	BASELINE	HEAVY TEXTILE TOW (Current: 1.6X Capacity)	HEAVY TEXTILE TOW (2X Capacity)
Precursor Cost	\$3.63/kg	\$2.24/kg	\$2.24/kg
Tow Size	50K	457K	457K
Tow linear density (g/m)	3.4	15	15
Tow Spacing	24 mm	50 mm	30 mm
Strands/Line	120	58	96
Line Speed	211 kg/hr (9 m/min)	338 kg/hr (7 m/min)	420 kg/hr (5.4 m/min)
Annual Prodn. Volume	1,500 tonnes/yr	2,400 tonnes/yr	3,000 tonnes/yr
Capital Investment	\$58M	\$58M	\$58M
Final Fiber Cost	\$18.11/kg	\$11.19/kg	\$9.92/kg





Fiber Embodied Energy

