Carbon Fiber Technology Facility

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2021 U.S. DOE Vehicle Technologies Office Annual Merit Review
June 22, 2021
Project ID mat174

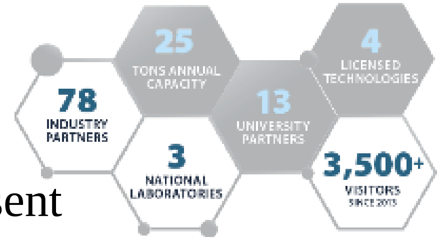
Outline

- 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
Total Budget	5.5 M	5.3 M	6.0 M	6.0 M	7.8 M	6.2 M
VTO	1.5 M	1.3 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
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
- Technical Collaboration Projects & Cooperative Research & Development Agreement (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

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

- 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

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.

Vehicle Technologies Office

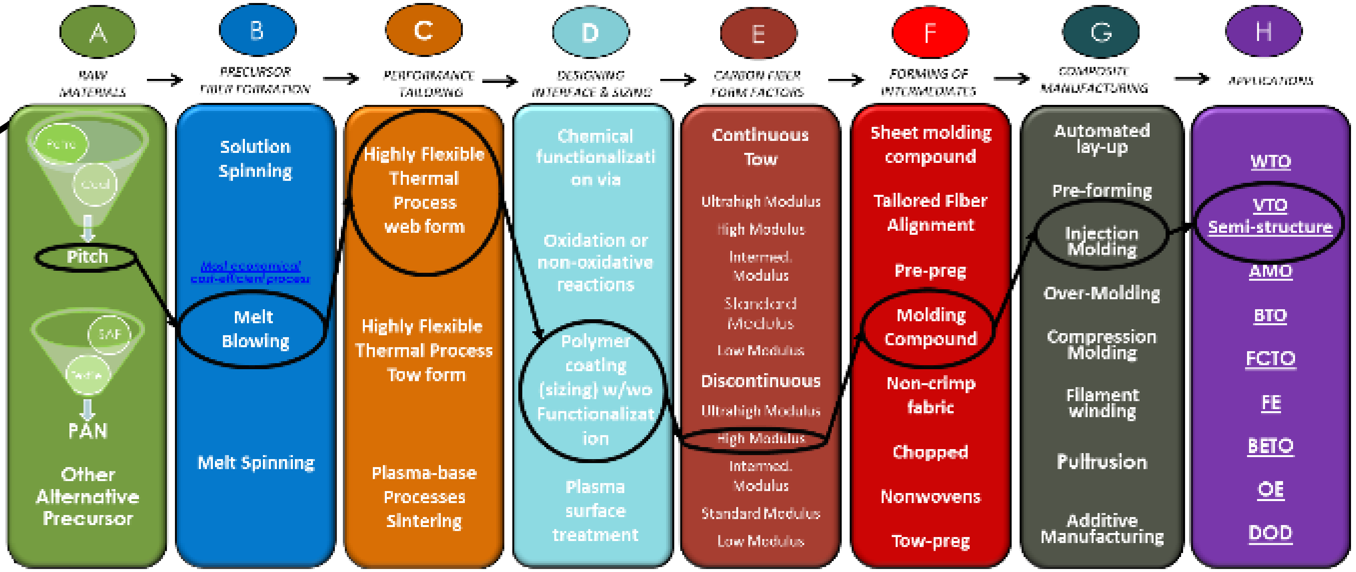
Carbon Fiber is necessary for:

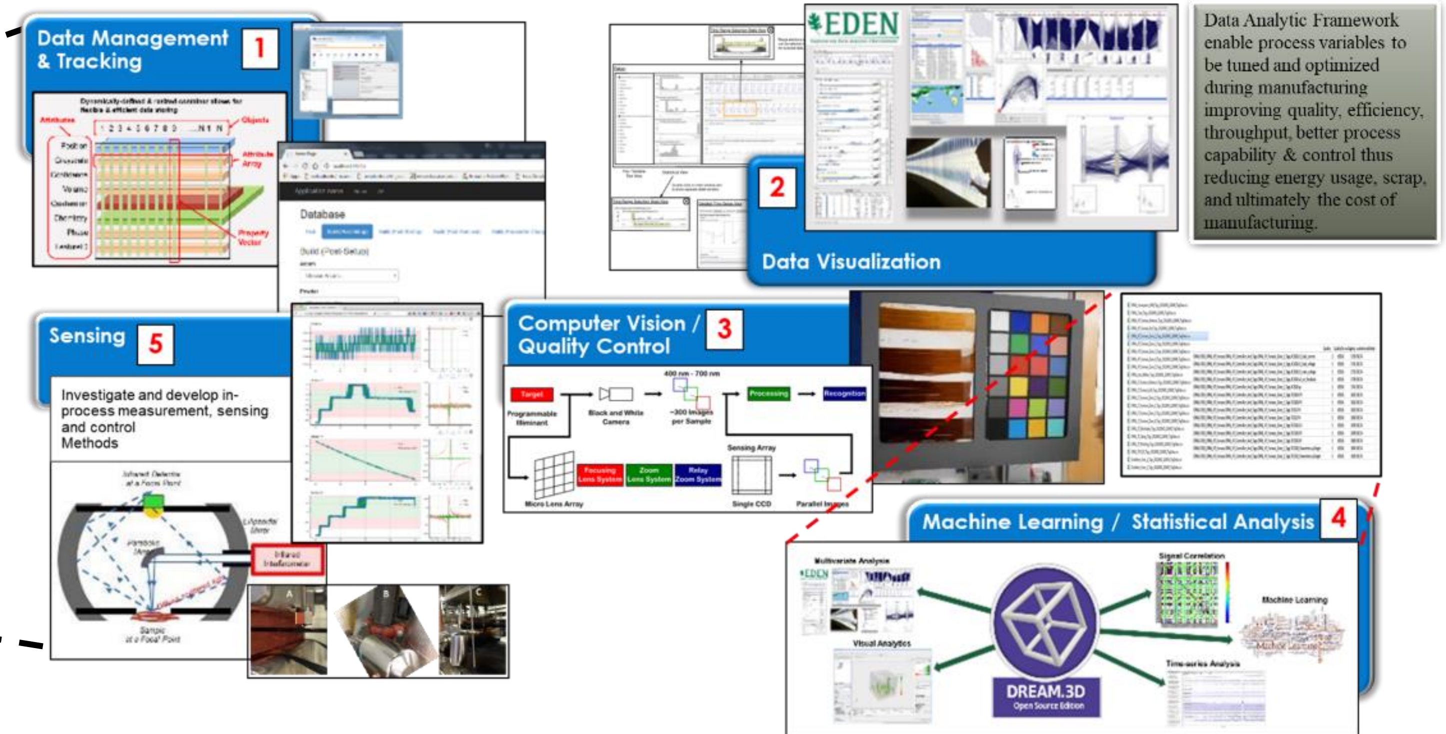
- **highest potential weight reduction, improved energy** efficiency for all vehicle types/classes
  - EV & alt fuel vehicle range extension
- Enhanced CAV sensor & communication device integration, signal transmission & EMI shielding

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.





Tasks and Milestones

Tasks
3. Investigate potential alternative CF precursors. This Task is 100% funded by VTO

Task/Objective	1st Quarter	2nd Quarter	3rd Quarter	4th Quarter
Investigate potential alternative CF precursors	Feasibility study of melt-blowing ACP mesophase pitch at scale Characterization and analysis of batch process samples report	Develop and demonstrate spinnability of nylon precursor at scale. A pilot production demonstration of 12,000 filament tows will be at ORNL CFTF.	Develop and demonstrate spinnability of mesophase pitch material from Kopper's Industry partners at scale. Produce CF from 5-8 kg batch of petroleum pitch mesophase exhibiting >25 Msi modulus and >250 ksi on multi-filament melt spinning equipment (Q3)	Develop and demonstrate spinnability of bio-pan precursor. Identify formulations of pre-ceramic polymers that provide >40 % silicon carbide yield at 1000 °C. Spin and oxidize bio-PAN tow for carbonization at CFTF exhibiting >25 Msi modulus, >250 ksi strength and > 1 % elongation. Provide technical report on formulations of pre-ceramic polymers.

Technical Integrated Approach

Precursor To Part

- 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
- Improve carbon fiber manufacturing cost metrics
- Commercialization

Capacity at each Scale

Multi-scale R&D Approach

- 1 - 2 tows
- 20k – 80k filaments

- 1-ton capacity
- 1-5 tows
- 5k -80k filaments
- Preferred tow size ≥ 3k

- 25 ton/yr capacity
- Designed for 3k – 80k tows
- Instrumented research Capacity for additional conversion

- Readiness level varies depending on material or unit operations

Material Identification
- Conversion Yield
- Availability
- Cost
- Carbon Content
- Quality

Alternative Precursor
- Spinnability
- Melt Spinning Processability
- Characterization & Analysis
- Precursor Chemistry

Fiber Evaluation
- Feasibility Study
- Batch process recipe & conditions
- 1g material req.

Pilot Scale
- Short term cont. processing
- Dev. proc. cond.
- Ideal Material selection
- 100 g – 1Kg material req.

ORNL -CF-Group

Scale-up
- Dev. multi-tow cont. processing
- Produce volume for market dev.
- Enable dev. of domestic commercial sources.
- >100 Kg material req.

Carbon Fiber Intermediate
- Development of usable for factor technology and processes
- Investigate carbon fiber intermediate forms and technical

ORNL/CFTF

Carbon Fiber Composites
- Fiber and intermediate form factor evaluation
- Coupon Level Testing
- Prototypes

ORNL/MDF - UT

Market Performance & Evaluation
- Performance Evaluation
- Product Development
- Recycling

IACMI

Technical Integrated Approach

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%
Bench	**Melt stable PAN precursor**	Throughput and energy in spinning, strength, elongation, fiber architecture. Conversion Yield 50%	Same as standard PAN, but higher energy productivity and lesser knowledge base	- 30%	- 30%
Bench	**Lignin-based precursor**	Feedstock price and stability, renewable domestic feedstock. Conversion Yield is 30 % – 45 %	Mechanical properties, yield, processing, knowledge base	- 50%	- 40%
Bench	**Silicon Carbide Fibers**	high-temperature oxidation resistance, high hardness, high strength, high thermal stability, corrosion resistance, and low density. good performance under extreme conditions.	Feedstock price and volatility, capital cost, energy, yield, processing	- 85 %	TBD
Bench	**Advanced conversion processing**	Speed, energy, capital cost	Knowledge base, risk	- 25%	- 50%
Scale-up	**Polyolefin 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%
Scale-up	**Bio-PAN (FY21 Q4)**	Renewable; pricing decoupled from oil	Knowledge base, scale	TBD	TBD
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%
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%**
Scale-up	**Recycled CF**	Cost, energy, capital cost, yield, fiber architecture (future)	Feedstock availability, fiber architecture (current), knowledge base, risk	- 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;

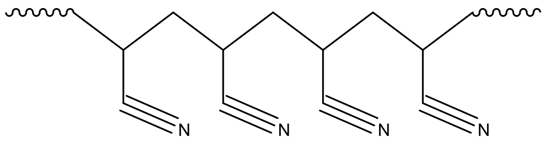
Technical Integrated Approach

Road to Lower Cost Carbon Fiber: Pitch vs PAN

Polyacrylonitrile (PAN)

$4-6 /kg (textile grade)

Precursor composition



More controlled molecular weight distribution and structure.
- Less impurities.

Carbon fiber processing

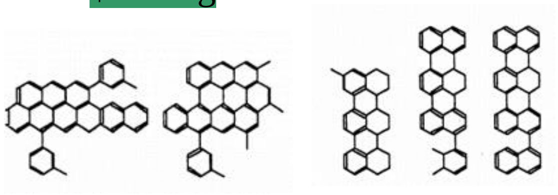
Solution spinning
↓
Oxidation-stabilization
↓
Carbonization

Stretching to align PAN molecules and the growing carbon structure

Pitch (coal or petro-based)

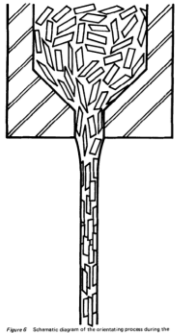
$1-5 /kg

Precursor composition



Highly aromatic with functional groups.
Large distribution of molecular weights and structures.
- Heterogenous
- Impurities

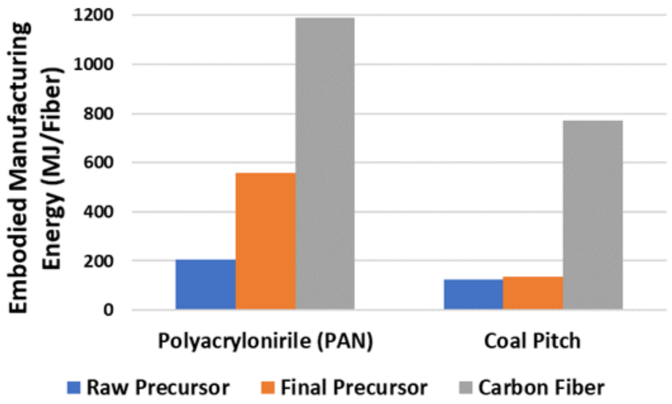
Carbon fiber processing



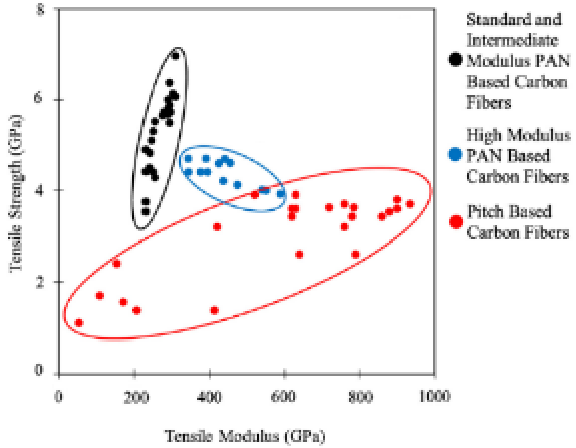
Melt spinning/Melt blowing
↓
Oxidation-stabilization
↓
Carbonization/Graphitization

Shearing and extension orient the molecules (mesophase pitch)

Singer, Fuel 60, no. 9 (1981): 839-847



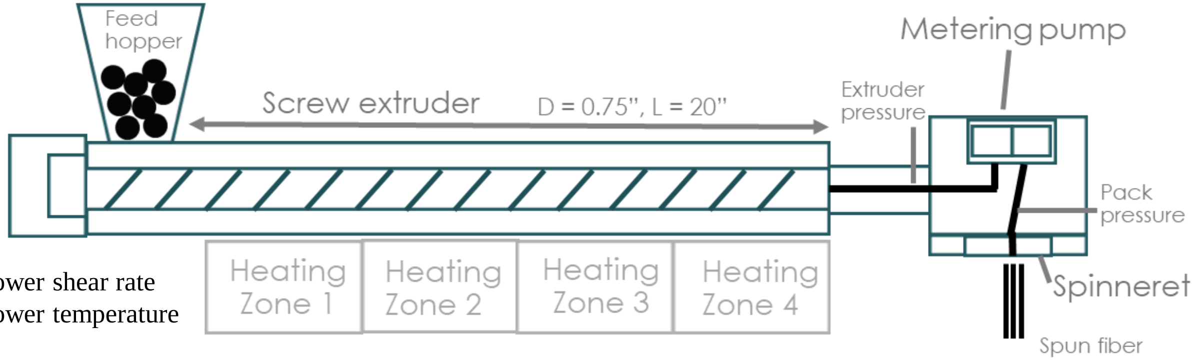
Source: [1]SimaPro 7.3..3 (LCA Software)[2]Liu, X. and Yuan, Z. (2016). Journal of Cleaner Production," Vol. 112, 1292-1301



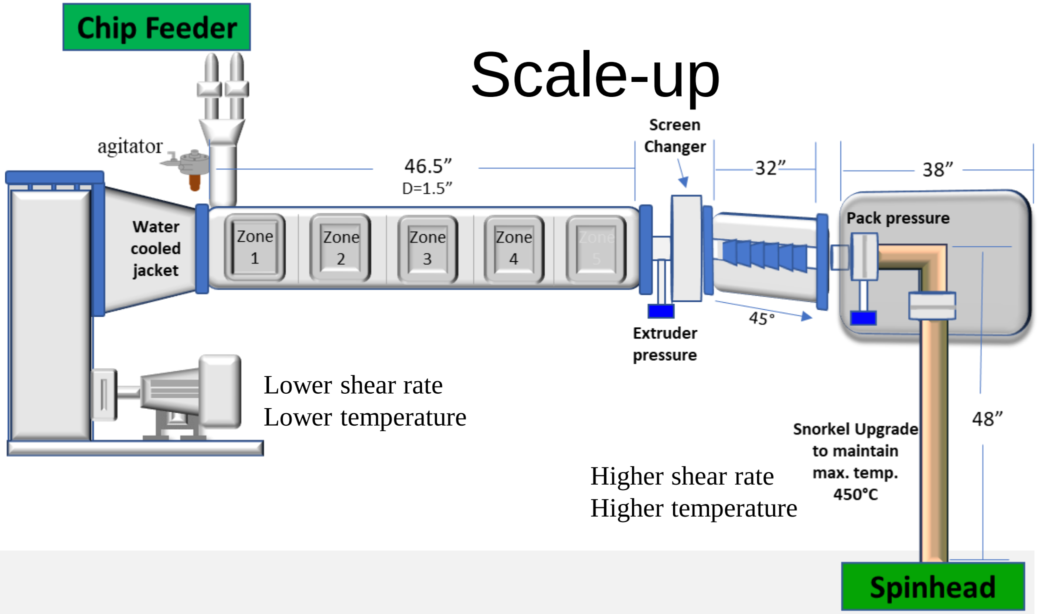
Processing, Structure, and properties of carbon fibers
B. A. Newcomb. Composites: Part A 91 (2016) 262–282

Technical Integrated Approach
Scale-Up

Bench scale



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 pitch unique and heterogeneous compositions.
- 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

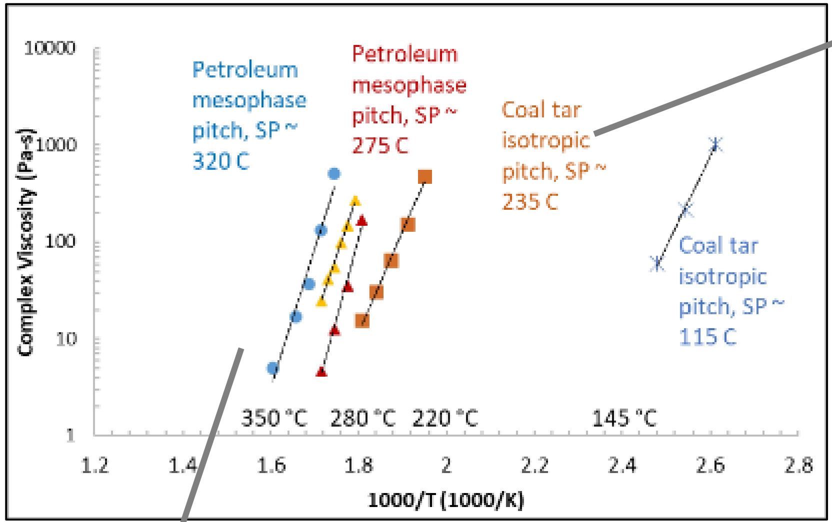
CFTF scale
- Known scale effects due to temperature, residence time, shear rate.

Oak Ridge National Laboratory

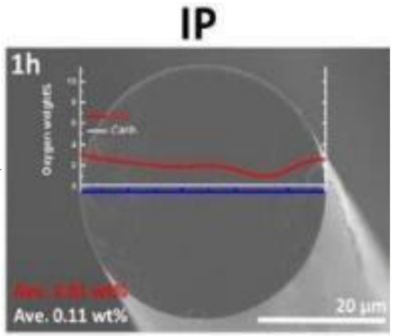
Technical Accomplishments and Results

Each Pitch is Different

Pitch rheology is highly temperature and composition/microstructure dependent



0% mesophase

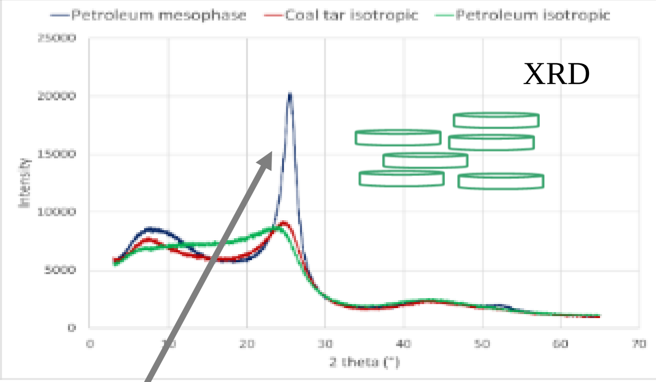


Isotropic pitch fiber
Low modulus carbon fiber

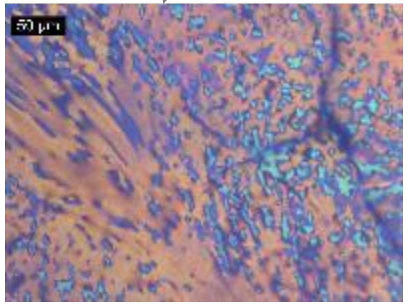
No liquid crystal content and no long-range orientation in pitch or fiber

Lim et al.,*Scientific Reports* 7.1 (2017): 1-12.

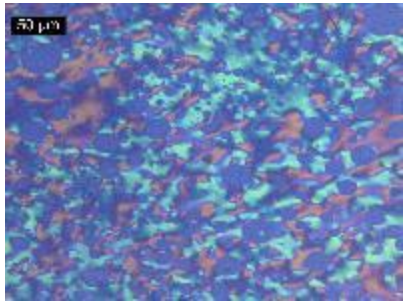
50% mesophase



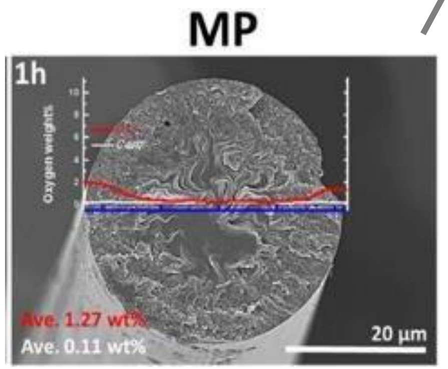
MALDI-TOF-MS	Mn (g/mol)
Petroleum mesophase	542
Coal tar isotropic	485
Petroleum isotropic	512



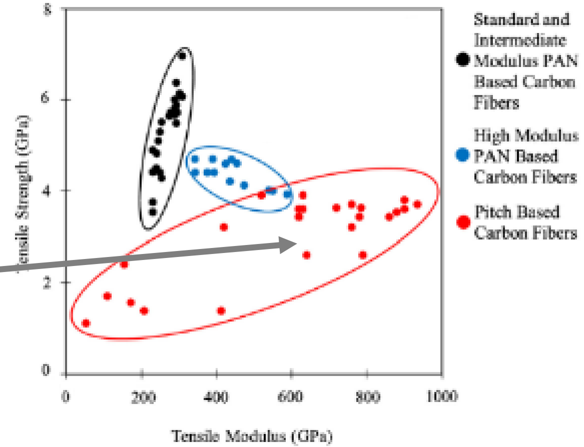
100% mesophase



70% mesophase



Liquid crystal mesogen stacking in mesophase pitch seen with XRD

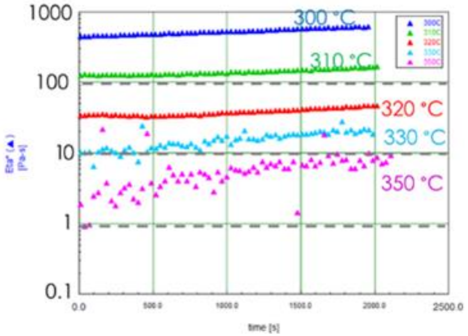


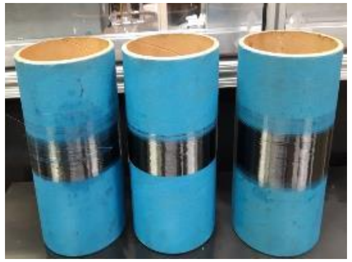
Technical Accomplishments and Results

Carbon Fibers From Petroleum and Coal Tar Results

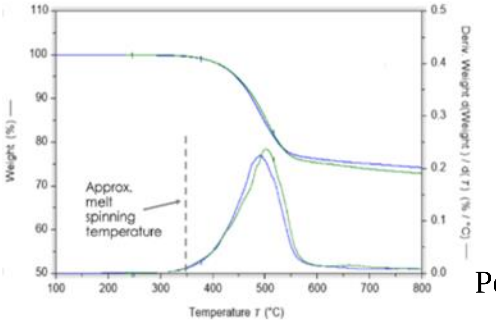
Melt spinning of petroleum 100% mesophase pitch:

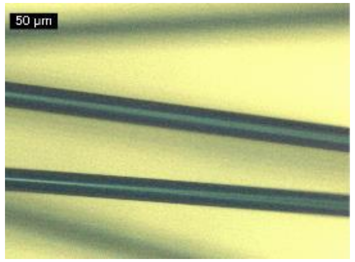
- Spinning temperature: 340-350 °C
- Very low impurities, continuous fiber possible
- Scale-up is planned





Petro-based pitch precursor fiber



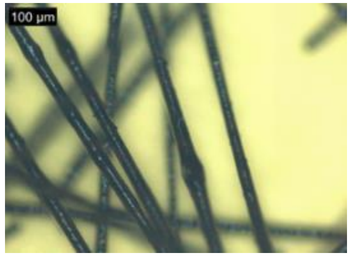


Petro-based pitch precursor filaments

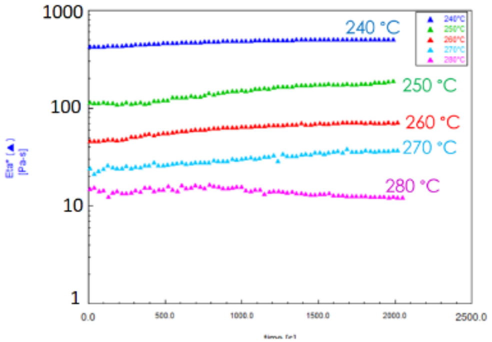
Melt spinning of commercial coal tar based 100% isotropic pitch:

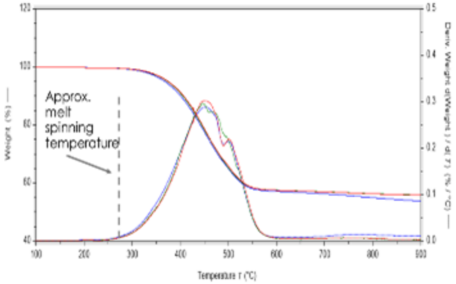
- Spinning temperature: 260-270 °C
- Up to 15% impurities, challenges with filtration
- Scale-up work is in progress

Coal-based pitch precursor fiber



Coal-based pitch precursor filaments





Technical Accomplishments and Results

Permits for Processing Alternative Precursor materials

CITY OF
OAK RIDGE

POST OFFICE BOX 1 • OAK RIDGE, TENNESSEE 37831-0001

Dear Industrial User,

This letter is to notify you of a change to the Industrial User Discharge Permit. The following amendment has been added to the permit:

"1.3.4 However, a waiver from monitoring for a pollutant neither present nor expected to be present in the discharge may be granted in accordance with Section 18-312(4)(b) of the Sewer Use Ordinance irrespective of monitoring requirements appearing in Appendix A."

Please note that this change does not in any way reflect a change in the discharge and/or monitoring requirements in the discharge permit. Should you desire to submit a waiver for any of the pollutants in the Industrial User Discharge Permit, you may request a copy of the waiver form. Once processed, your discharge permit will be modified to reflect any necessary changes. If you have any questions or comments, do not hesitate to contact me.

Regards,

Zachariah Seiden
Environmental Compliance Coordinator
City of Oak Ridge

P: 865-425-1610
E: zseiden@oakridgetn.gov

Water Permit Modification

STATE OF TENNESSEE
AIR POLLUTION CONTROL BOARD
DEPARTMENT OF ENVIRONMENT AND CONSERVATION
NASHVILLE, TENNESSEE 37243

Operating Permit (Conditional Major) Issued Pursuant to Tennessee Air Quality Act

Date Issued: February 24, 2020 Date Expires: October 1, 2029	Permit Number 474951 Facility ID: 73-1108
Issued To: U.S. Dept. of Energy, Office of Science Carbon Fiber Technology Facility UT-Battelle, LLC – Operator	Installation Address 93 Palladium Way Oak Ridge
Installation Description Carbon Fiber Process Line, Thermal Oxidizer as Control	Emission Source Reference No. 73 1108 01 **Conditional Major**

The holder of this permit shall comply with the conditions contained in this permit as well as all applicable provisions of the Tennessee Air Pollution Control Regulations (TAPCR).

CONDITIONS:

G1. The application that were utilized in the preparation of this permit are dated March 6, 2014, May 12, 2016, and September 12, 2018, and are signed by John E. Powell, Director Environment, Safety, and Health for the permitted facility. If this person terminates their employment or is assigned different duties and is no longer the responsible person to represent and bind the facility in environmental permitting affairs, the owner or operator of this air contaminant source shall notify the Technical Secretary of the change. Said notification shall be in writing and submitted within thirty (30) days of the change. The notification shall include the name and title of the new person assigned by the source owner or operator to represent and bind the facility in environmental permitting affairs. All representations, agreement to terms and conditions and covenants made by the former responsible person that were used in the establishment of limiting permit conditions on this permit will continue to be binding on the facility until such time that a revision to this permit is obtained that would change said representations, agreements and covenants.

TAPCR 1200-03-09-.03(8)

(conditions continued on next page)

Michelle W. Owenby
TECHNICAL SECRETARY

No Authority is Granted by this Permit to Operate, Construct, or Maintain any Installation in Violation of any Law, Statute, Code, Ordinance, Rule, or Regulation of the State of Tennessee or any of its Political Subdivisions.

Air Permit Modification

Equipment & Upgrades required
Resource
Water Permit Modification
Air Permit Modification
+ 2 Spin packs
Controls system repair
AMP Cherokee TOX data logger compliance device
LT piping Upgrade
Software upgrade
Graphite belt replacement
Upgrade temperature capacity of melt blowing kit

Other Funding sources was leveraged for new upgrades

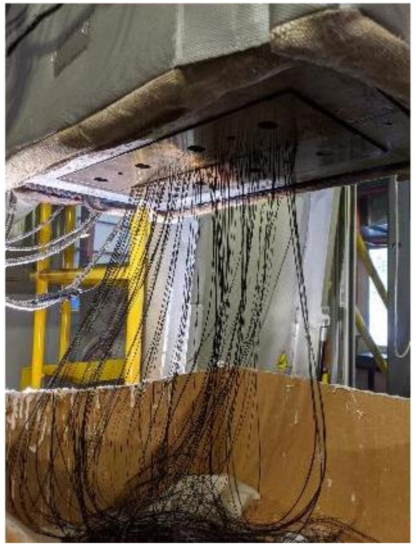
Not considered a technical accomplishments. However, No work is initiated without these permits in place. Some materials have never been explored at this scale. This process may take up to a year, depending on the revision required.

OAK RIDGE
National Laboratory

Technical Accomplishments and Results
Melt-blown Pitch Precursor at Scale-up

Preparation for Scale-up:

- Baseline Conditions:
  - Develop baseline for spinning mesophase petroleum-based pitch-based material into precursor – in progress
  - Develop baseline for converting the pitch-based precursor into carbon fiber – in progress
- ACP pitch-based material:
  - CRADA approval – not yet approved
  - Develop spinning and conversion process conditions for ACP proprietary pitch-based material from precursor to carbon fiber
  - Characterize and analyze properties of pitch-based carbon fibers

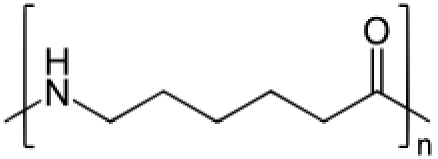


Oak Ridge National Laboratory

Technical Accomplishments and Results
Nylon as a low-cost-fiber precursor

- Global production ~ 4 million tons/year.

Precursor composition



Controlled molecular weight distribution and low impurities.

Nylon
$2 /kg

Carpets
Tire cords
Apparel
Seat belts
Ropes and industrial cords

Carbon fiber processing

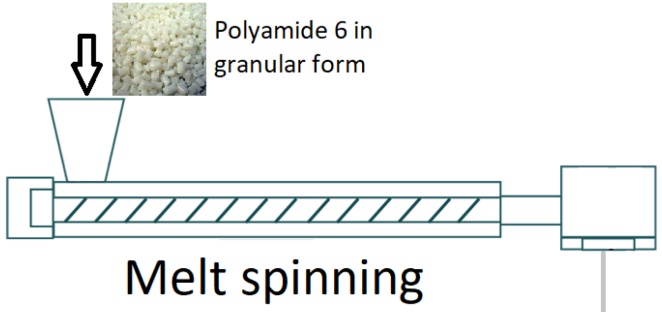
Melt spinning/Melt blowing

Stabilization

Carbonization

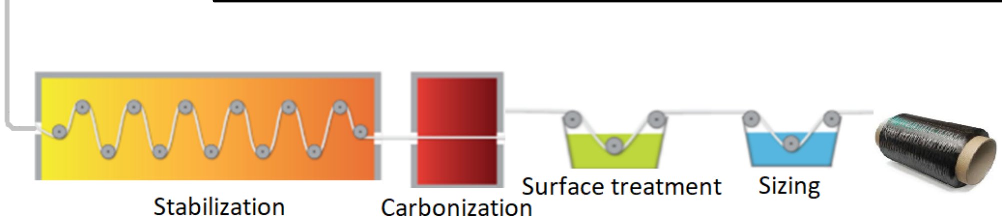
L. Karacan and G. Baysal
Fibers and Polymers 13 (2012) 864-873
Use of a cross-linking agent

On-going project at ORNL.



Goal: Integration of melt spinning extrusion in the carbon fiber processing line. First step : Demonstrate melt spinning of commercial polyamide 6 fibers at CFTF.

→Demonstration of melt spinning at bench scale.

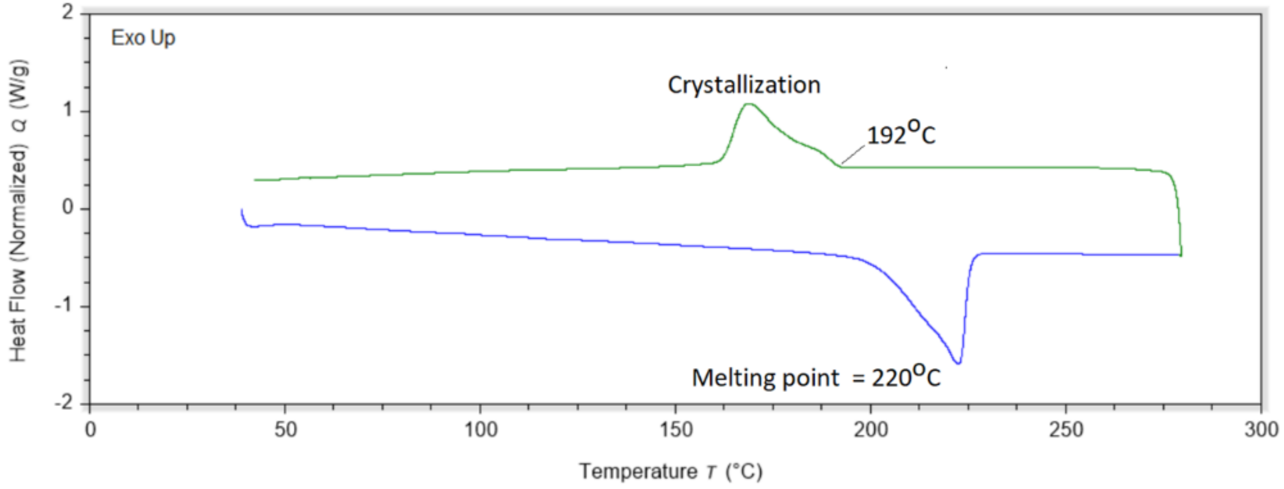


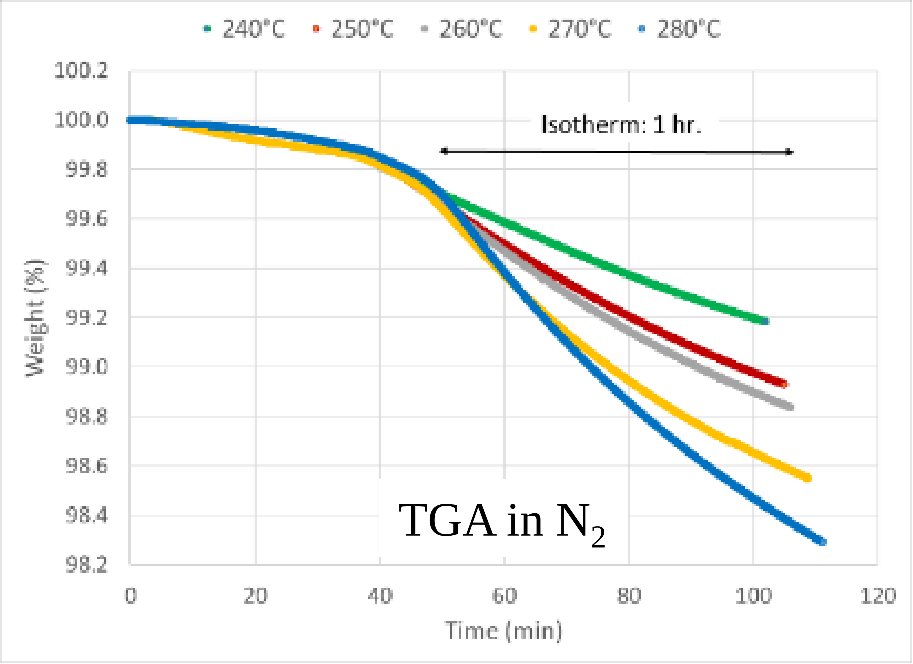
OAK RIDGE National Laboratory

Technical Accomplishments and Results

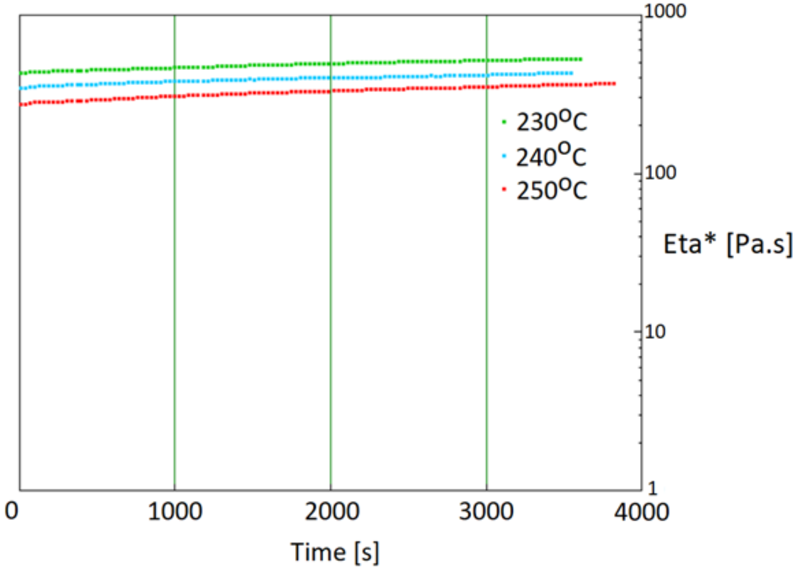
Nylon as a low-cost-fiber precursor

- Reception of material 04/21
- Commercial sample from BASF (Ultramid® B27 E 01)
- Characterization for melt spinning at bench scale
- Demonstration of Melt spinning of Polyamide 6 fiber as scale (CFTF)





TDS: Moisture content at 50% Relative Humidity: 2.6%



TDS:
Melting point: 220°C
Temperature range for melt processing: 240-285°C

Response to Reviewers Comment @ FY 20 Peer Review

Question 1: Approach to performing the work—the degree to which technical barriers are addressed, the project is well-designed and well planned.

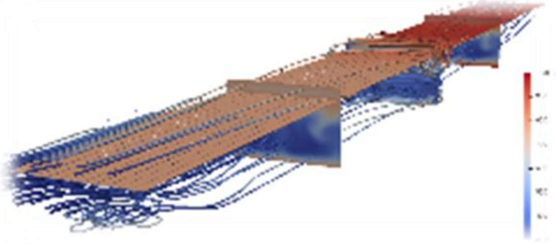
Reviewer 2: The broader mission of the CFTF is not clearly delineated from the specific VTO project work. IT would be helpful, and improve the quality of the research, if a more explicit plan is included to interrogate other properties of the resulting blown fiber mats (including nominal fiber diameter, variance in fiber diameter, specific gravity, modulus, etc.). Finally, it is not clear if conversion to structural materials (via impregnation and cure) is planned, and more importantly, followed by testing and evaluation of those resulting composites. It is also not clear whether a path toward aligned fiber reinforcements using these candidate (or down-selected) precursors is being planned. It also seems important to seek out useful molecular models that can inform or predict process parameters and resulting properties to support a more rapid screening and drive future of mesophase pitch selections and designs.

Ans: **A** strategic technological path have been developed for the various types of carbon fiber produced by the different precursor.

Office	Application	Typical Design Drivers	Preferred Fiber Spec	Developmental CF	CF Product Form	Intermediate Form	Compositing Method
Vehicles	Crash mgmt structure	Energy absorption (ult. strain)	TS > 5 GPa TM 200 - 240 GPa	SM melt-spun PAN or gel-spun textile PAN	Continuous tow	Pre-preg, tape, fabric, braid, etc.	HP-RTM, compression molding variants
	Primary structure	Stiffness	TS > 2.7 GPa TM > 270 GPa	IM Textile PAN	Continuous tow	Pre-preg, tape, fabric, braid, etc.	HP-RTM, compression molding variants
		Strength	TS > 4 GPa TM > 200 GPa	SM melt-spun PAN or gel-spun textile PAN	Continuous tow	Pre-preg, tape, fabric, braid, etc.	HP-RTM, compression molding variants
		Strength and stiffness	TS > 4 GPa TM > 270 GPa	IM melt-spun PAN or gel-spun textile PAN	Continuous tow	Pre-preg, tape, fabric, braid, etc.	HP-RTM, compression molding variants
	Semi-structure	Stiffness	TS > 2 GPa TM > 400 GPa	HM pitch CF	Nonwoven	Molding compound	Injection or compression molding
		Stiffness & intermediate strength	TS > 2.7 GPa TM > 270 GPa	IM Textile PAN	Continuous tow	Engineered molding compound, non-crimp fabric	Compression molding, injection over-molding
	Cosmetic components	Appearance & stiffness	TM > 400 GPa	HM pitch CF	Nonwoven	Molding compound	Injection or compression molding

Molecular models and data analytics for carbon fiber manufacturing have been developed for characterizations of the precursor at various stages along the carbon fiber process. It is not shown here because we leveraged other sponsors for that effort.

A three-dimensional, multi-physics computational model of the processing conditions in the LT carbonization furnace.



Srikanth Allu, Srdjan Simunovic, Tae-Seok Lee, Peter Witting, "Development of mathematical model and simulation tool for the high-capacity production of carbon fiber" ORNL/TM-2018/1062.

OAK RIDGE National Laboratory

Response to Reviewers Comment @ FY 20 Peer Review

Question 1: Approach to performing the work—the degree to which technical barriers are addressed, the project is well-designed and well planned.

Reviewer 3: Manufacturing of carbon composites using pitch showcases the capability of the CFTF. However, it does not clearly demonstrate the facility's capability in terms of the barriers the project addressed, i.e., the cost of manufacturing, process validation and technology scalability, etc.

Ans: This work is currently still in progress. LCA analysis of different pitch material is a project currently in process…slide 8

Question 2. Technical Accomplishments and Progress toward overall project goals—the degree to which progress has been made and plan is on schedule.

Reviewer 3: The accomplishments related to melt-blowing three of the five candidate precursors demonstrate important progress but leave many of the challenges and barriers in place and do not answer important questions related to the cost of the resulting fiber. Similarly, the referenced disadvantage visa-vis PAN precursors, strain to failure, and tensile strength has not been addressed as part of this year's technical accomplishments. The reviewer could not assess whether the results are promising or not.

Ans: At the time of the review last year the final properties were not yet completed.

Rain Isotropic Fibers	Mean	STDev*
Tensile Strength [MPa]	184	80
Tensile Modulus [GPa]	15.3	4.15
Strain to Failure [%]	2.36	1.39

Pitch	Mat Density (g/cm^3)	Thermal Conductivity (W/mK)
Pilot Production Rain (250MP)	0.0375	0.035

Question 3: Collaboration and Coordination Across Project Team.

Reviewer 3: There is little offered that demonstrates how the collaboration (whether through the IACMI partnership or other noted collaborators) impacted the technical approach or accomplishments. It would be helpful to understand what market drivers, technical performance parameters, or specific cost targets come from the end-users or collaborators.

Ans: Strategic Drivers - Projected demand for Carbon fiber and composites, Increase in demand for fuel-efficient vehicles, Focus on energy production from renewable energy, Cost of carbon fiber and composites manufacturing, Insufficient U.S. owned production capacity, Potential opportunities in new applications, Growth rate in emerging markets, Focus on Mass Market Opportunities, Economic Opportunities.

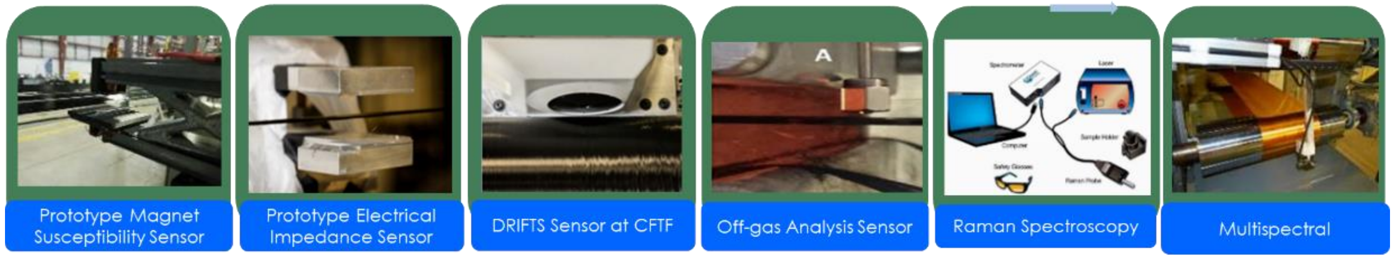
- Cost Target was mandated by VTO as cost of manufacturing - $5/lb, tensile strength >250 Ksi, tensile modulus >25 Msi
- 12 Active IACMI projects for Composites Materials and Processing Technology Area that utilizes resources from CFTF

Response to Reviewers Comment @ FY 20 Peer Review

Question 4: Proposed Future Research—the degree to which the project has effectively planned its future work in a logical manner by incorporating appropriate decision points, considering barriers to the realization of the proposed technology and, when sensible, mitigating risk by providing alternate development pathways.

Reviewer 2: The proposed future research is clearly necessary, and those specific steps make sense to the reviewer. What does not make sense is the order in which the proposed work is presented. It seems that establishing criteria should come before efforts to increase throughput, take “in situ measurements,” etc. It would also be more compelling if the research team proposed specific interrogation of the produced materials in the application that is most impactful for these materials, that is, as a reinforcing element in a structural composite. No material testing is proposed toward that end.

Ans: This is being done, it is just not mentioned in the task associated with VTO. ORNL have developed a suite of in-situ measurement concepts under the sensing module for the data analytic framework for carbon fiber manufacturing.



Question 5: Resources—How sufficient are the resources for the project to achieve the stated milestones in a timely fashion?

Reviewer 3: The reviewer remarked that resources are insufficient based on the lack of progress in identifying specific work to address challenges and barriers as presented by the research team. The proposed work does not appear to address the specific concerns (“challenges and barriers”) related to “availability” and “quality” of mesophase pitch feedstocks. If the goal is to produce high performance (meaning high specific stiffness and strength) materials, make sure the work being planned provides an accurate view of the status and performance of the production materials.

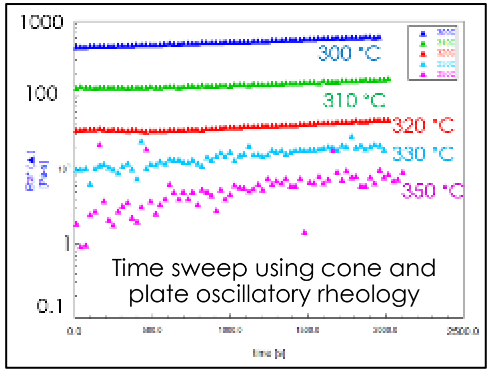
Ans: DOE has started the C4Ward project to address the issues of availability of mesophase pitch. ORNL is working with several companies developing mesophase pitch supplies, however they are in other programs at the lab, and as they develop enough product for scaling, CFTF will be positioned to start scale up trials and evaluations of those pitches. Also, ORNL is in the middle of starting a CRADA with Exxon to develop mesophase pitch supplies for large scale applications of fiber, not just structural.

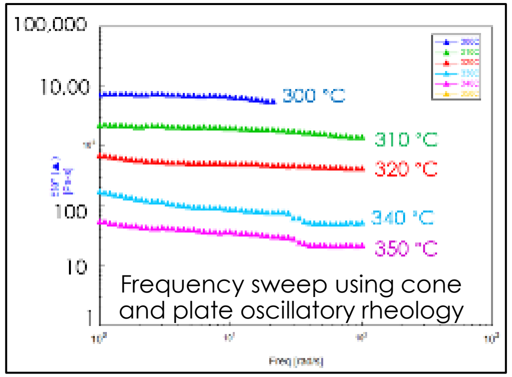
Technical Collaboration Projects

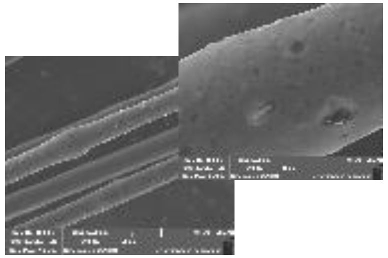
Partner	Project #	Project summary
Not yet started		
Advanced Carbon Products	NFE-20-08145	ACP has developed several processes for the manufacturing of hydrocarbon precursors for advanced carbon products, including a continuous process to convert low-cost refinery oils into isotropic pitch and a continuous 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	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.
Life cycle Analysis/Assessment (LCA)	PI -Sujit Das	LCA of petro-based carbon fiber manufacturing

Remaining Challenges & Barriers

- **Material**: Availability of consistently high-quality pitch
  - Stability at temperature, time, and shear conditions
  - Low in solids (including ash)
  - Low in sulfur and nitrogen
  - Controlled pellet or particle sizing
  - Mesophase content vs softening point

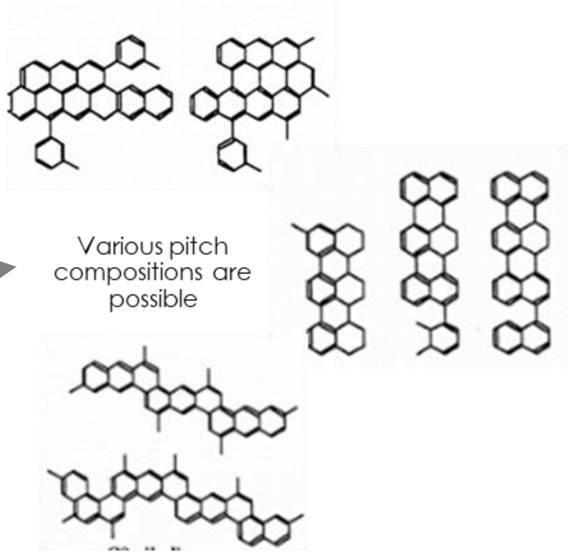






Fiber from high solids pitch

- **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 designed for polymers may not be ideal for pitch



- **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 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

Development of skilled carbon fiber and composites technical professionals

Proposed future work is subject to change based on funding levels

Integrated Team

Advanced Fiber Manufacturing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Sefa Yilmaz (MSD)

Fiber Characterization


Tonia Robinson (MSD)


Parans Paranthaman(CSD)

C-Fiber Science (CSD)


Nidia Gallego


Ryan Paul


Frederic Vautard


Amit Naskar

Digital Thread and On-Line Diagnostics


Yarom Polsky (EEID)


Jim Parks (MSD)


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Alex Melin (EEID)


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Compliance Specialist

Katherine Gaul
Technical Project Specialist

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Kenneth Hardie
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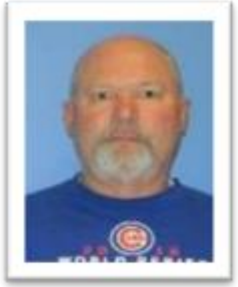

Kim Sitzlar
Operation Supervisor

Jason Newport
Shift Lead

Terry Wyrick
Line Technician

Justin Walls
Line Technician

Timothy Dexter
Line Technician

Samantha Aycock
Line Technician

Donnie Buck
Line Technician

Summary

- **Relevance**: The Carbon Fiber Technology Facility is relevant in proving the scale-up of low-cost advanced fiber precursor 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 alternative pitch-based carbon fiber
- **Technical Accomplishments:**
  - Demonstrated a full year of operation with zero accidents or environmental non-compliances
  - Identified pitch-based material suitable for scaling
    - Melt-blown and carbon fiber processing of mesophase pitch still a work in progress
  - Initiate bench scale development of nylon fibers
  - Obtain air/discharge permit for processing pitch fibers at CFTF – no work can be initiated without it
  - Upgrades to equipment to help overcome some challenges associated with processing materials is completed
- **Future Work**:
  - Expand industry partnerships through CRADAs for scale-up of alternative precursors for low-cost carbon fiber manufacturing
  - Identify, research, develop and demonstrate scale-up of new advanced fibers
  - Improve Efficiency, Throughput, and Commercialization of Pitch based Carbon Fiber
  - Life Cycle Analysis of Petroleum Pitch Carbon Fiber Manufacturing
  - "Proposed future work is subject to change based on funding levels"

Technical Back-Up Slides

Pitch Material

Left - Purge material, Right – As-is Pitch material

Pelletized Pitch material- preferred quality