U.S. Department of Energy Workshop Compressed Gas Storage for Medium and Heavy Duty Workshop

Carbon fiber R&D progress and technology status towards commercialization and deployment for various applications

Merlin Theodore
Director Of Carbon Fiber Technology Facility (CFTF)

Oak Ridge National Laboratory

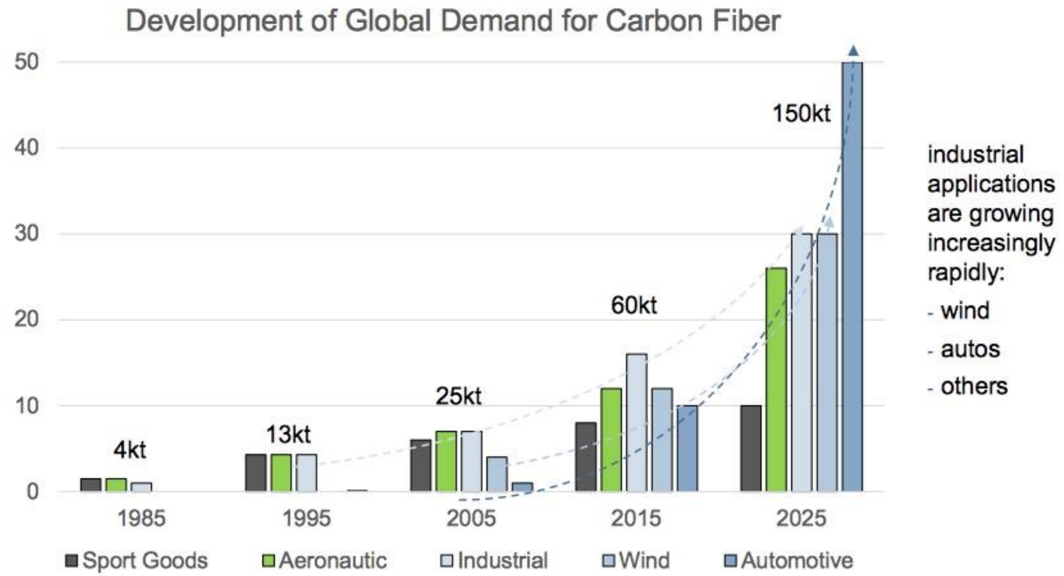
ORNL is managed by UT-Battelle, LLC for the US Department of Energy

Carbon Fiber Manufacturing

Carbon Fiber Production Challenges

CF Manufacturing: Issues

- Costly
- Precursor quality/reliability
- Processability
- Energy intensive process
- Purity level
- Sources for new precursors



Industrial applications share:	20 years ago	10 years ago	today	tomorrow
	about 1/3	about 40%	about 60%	more than 80%

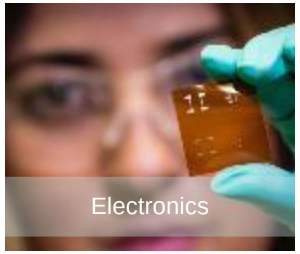
www.carbonfibrefutures.com.au/wp-content/uploads/20017/03/20170301-CF-Futures-Deakin-2017-Pichler-v4.pdf

ORNL is addressing these challenges by conducting multi-scale research

Necessary for 50+% Mass Reduction

Only material with enough strength and weight

Needed for longer blade designs

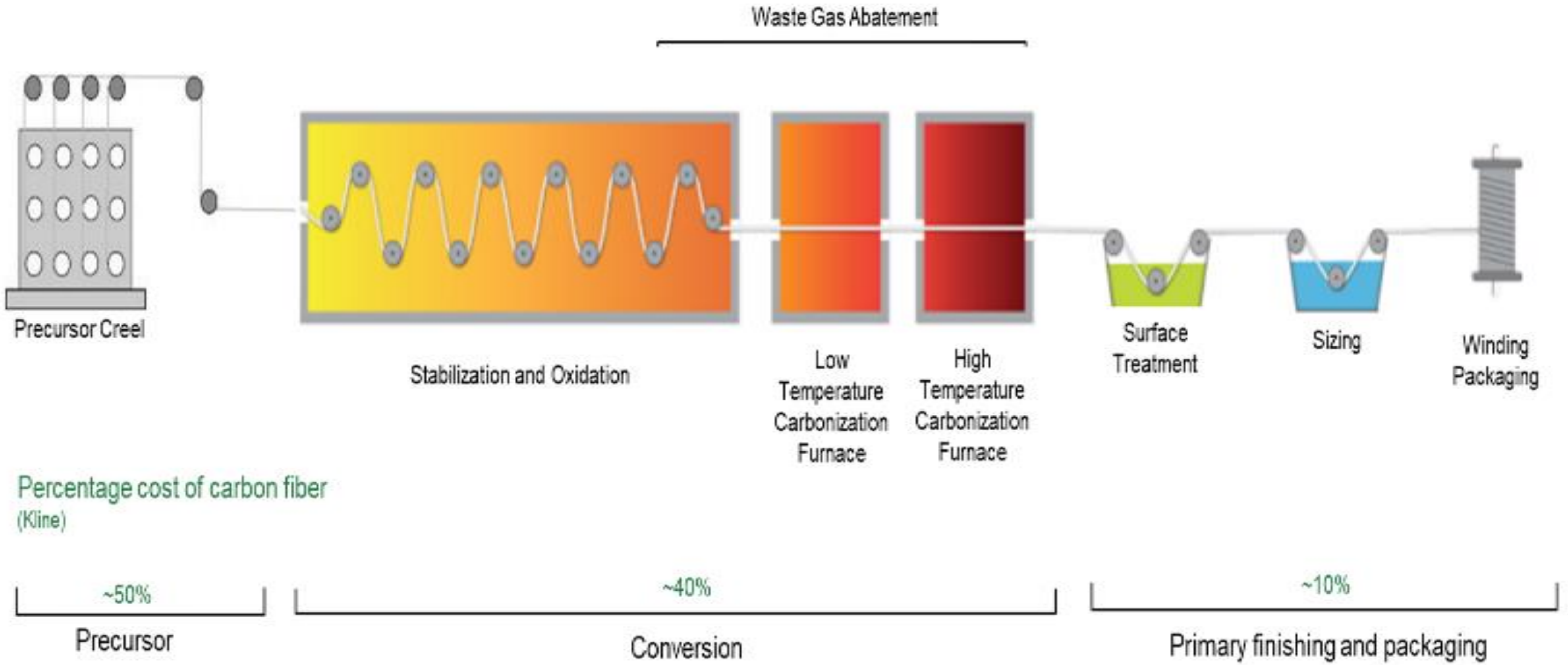


Lightweight, EMI shielding

Offshore structural components

Rapid repair and installation, time and cost savings

Carbon Fiber Manufacturing Process



With conventional processing using a carbon fiber-grade (CF) PAN, precursor is over 50% of the carbon fiber cost

Research & Development at ORNL

Precursor/ Process	Advantage	Disadvantage	Cost Δ	Energy Δ
Standard PAN precursor	Strength, elongation, knowledge base, fiber architecture	Feedstock price and volatility, capital cost, energy, yield, processing	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	~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	~ 30%	~ 30%
Bio-PAN	Renewable; pricing decoupled from oil	Knowledge base, scale	TBD	TBD
Polyolefin precursor	Feedstock price and stability, spinning, yield, fiber architecture	Conversion process and equipment, knowledge base, capital cost	~ 20%	~ 50%
Pitch-based precursor	Feedstock price and stability, spinning, yield, knowledge base, properties develop w/o stretching, moderate capital	Elongation and compression strength, fiber architecture	~ 70%	~ 70%
Lignin-based precursor	Feedstock price and stability, renewable domestic feedstock	Mechanical properties, yield, processing, knowledge base	~ 50%	~ 40%
Recycled CF	Cost, energy, capital cost, yield, fiber architecture (future)	Feedstock availability, fiber architecture (current), knowledge base, risk	~ 60%	~ 90%
Advanced conversion processing	Speed, energy, capital cost	Knowledge base, risk	~ 25%	~ 50%

Sujit Das
Life Cycle Analysis
dass@ornl.gov

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;

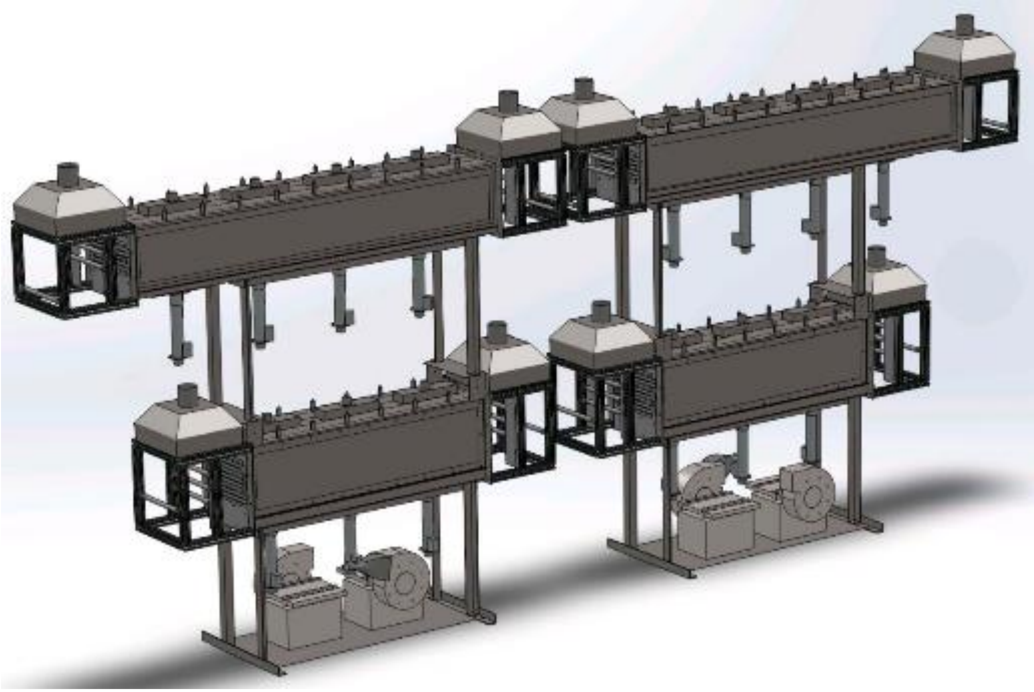
Advanced Carbon Fiber Manufacturing – Example Advanced Oxidation Technology

- Recently demonstrated Close Proximity Indirect Exposure (CPIE) plasma approach is being refined and optimized with textile PAN - confirmed that the localized damage that was seen in the old approach is no longer a problem.

- **Successfully oxidized textile grade PAN in under 35 minutes(~2 – 4 x faster). Exceeded strength and strain program requirements for carbonized fiber.**

- **Improves Production Throughput**

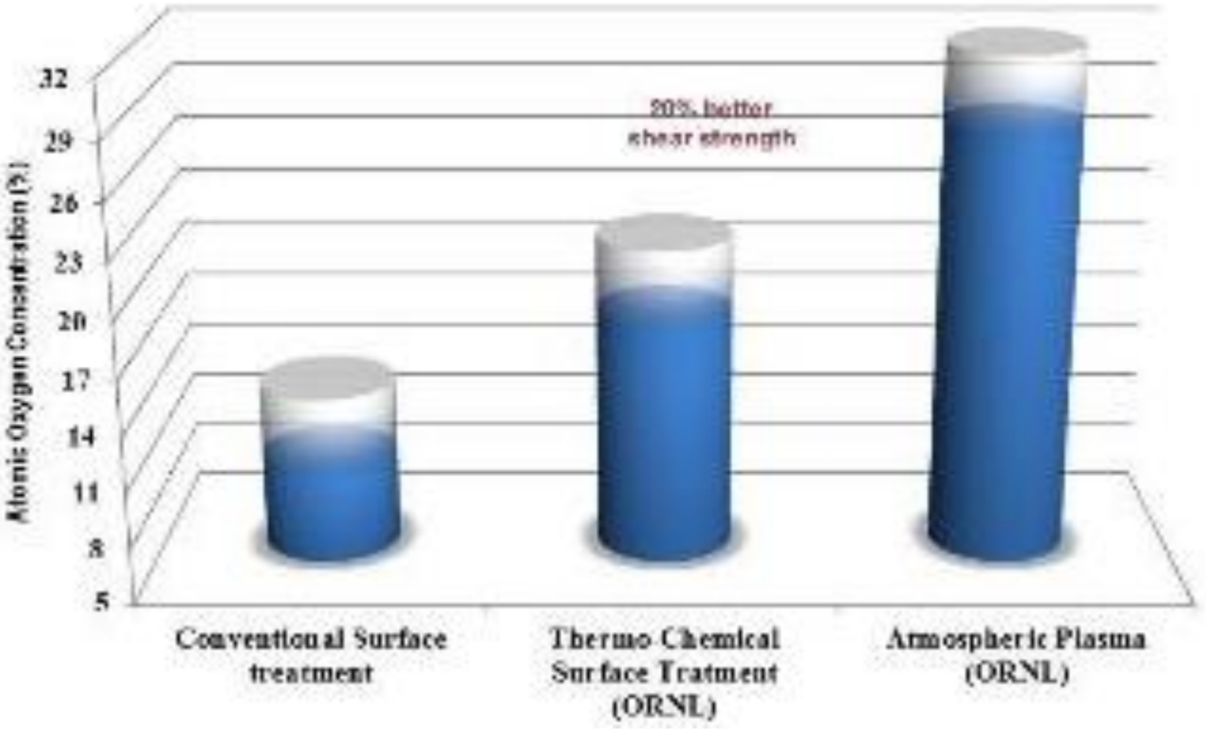
- **Completed design and began construction of the scaled CPIE reactor.**



Paulauskas et al. US Patent 7,649,078 (2010)

- RMX Technologies (Knoxville, TN) licensed the technology from ORNL.

Advanced Carbon Fiber Manufacturing –Examples of Advanced Surface Treatment



1 tpy dry surface treatment module

- Current CF post-treatment not tailored for commodity resins
- SBS strength increased by 40+% with proper fiber surface engineering

Strategic Path From Precursor-to-Part from - Lab Scale-to-Application

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 Composite
- Fiber and intermediate form factor evaluation
- Coupon Level Testing
- Prototypes

ORNL/MDF - UT

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

IACMI

CARBON FIBER RESEARCH GROUP
- Carbon fiber science
- Custom melt spinning
- Activated carbon R&D
- Carbon precursors
- Carbon Fibers

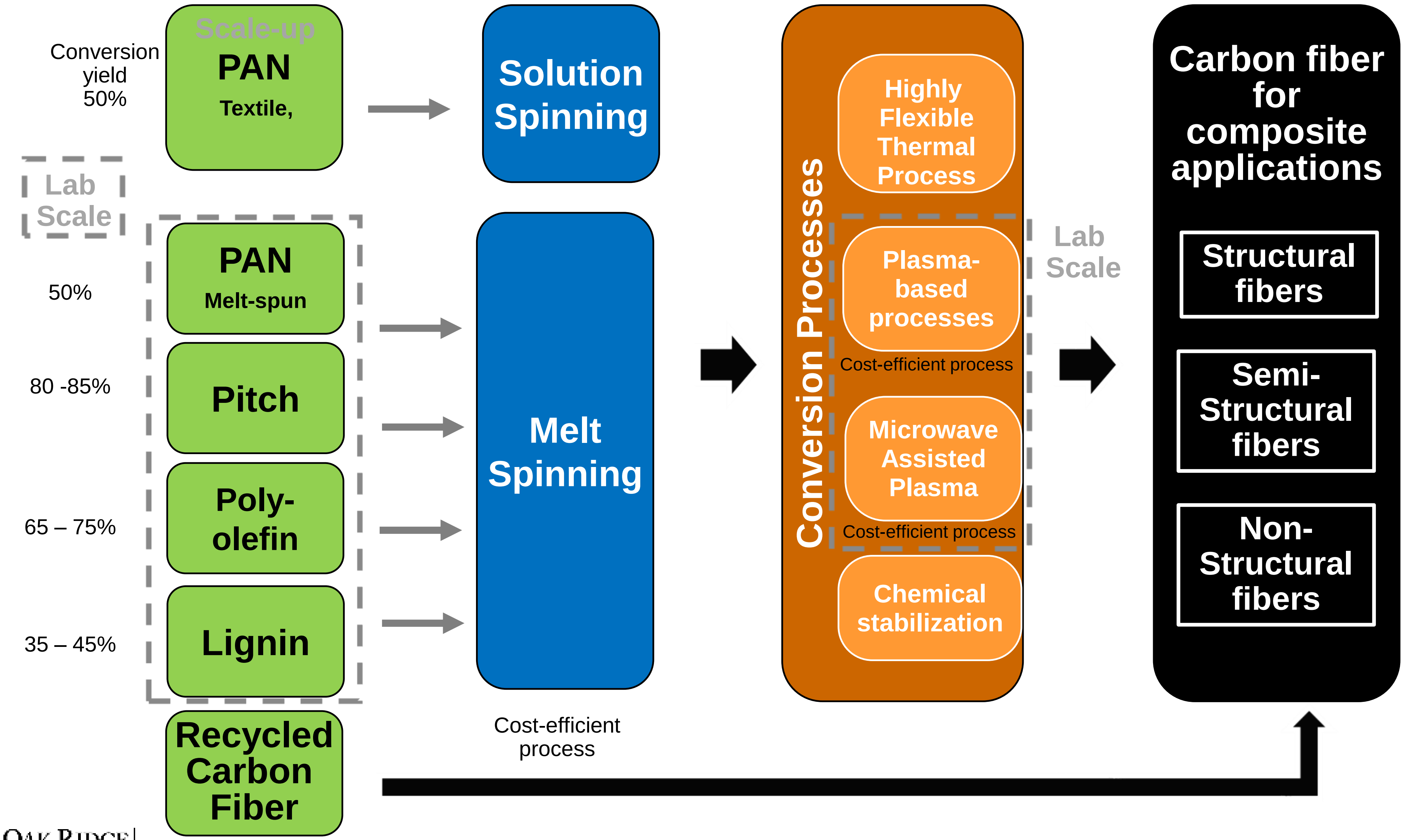
CARBON FIBER TECHNOLOGY FACILITY
- Low cost carbon fiber
- 25 ton per year carbon fiber production
- 65 tons/yr melt spinning precursor production
- Custom sizing and characterization
- Intermediates fabrication
- Instrumented & flexible CF line

MANUFACTURING DEMONSTRATION FACILITY
- Intermediate to large scale composite fabrication
- Hybrid materials
- Additive - Conventional Composites
- BASIC SCIENCE –> LAB SCALE –> INTERMEDIATE SCALE – PROTOTYPING – MANUFACTURED PRODUCTS – SERVICE and END OF LIFE
- ~ 70 Systems

UNIVERSITY OF TENNESSEE COMPOSITES
- Fibers & Composites Manufacturing
- Mechanical Characterization
- Joint Institute for Advanced Materials
- Center for Renewable Carbon (CRC)

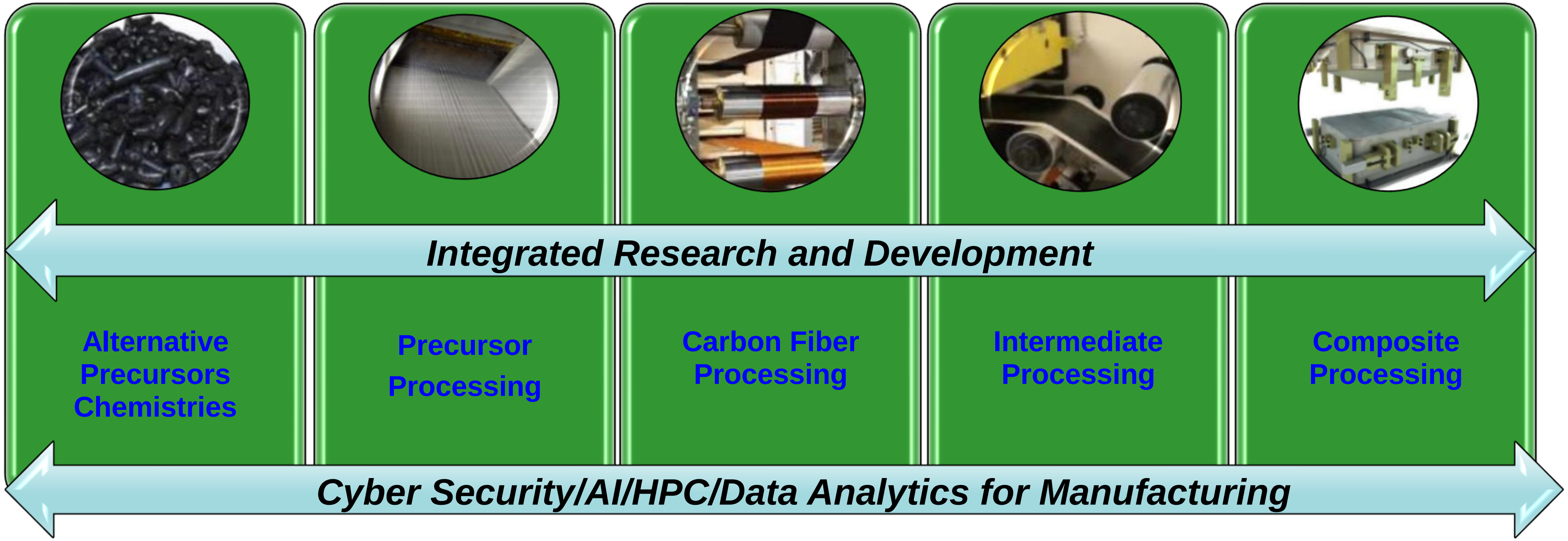
Institute Advanced Composites Manufacturing Innovation
- 160 members representing 31 states, 130 companies, 17 Academia
- Compositing Prototyping Center,
- Technology areas: Vehicles Technology Area, Material & Processing Technology Area, Design Modeling & Simulation Technology Area. Wind Energy Technology Area, Recycling Technology Area, and Compress Gas Storage Technology Area

Research & Development at ORNL



Fiber & Fiber Composites Vision

Integrated Carbon Fiber and Carbon Fiber Composites Strategy that delivers significant ENERGY and PROSPERITY impacts to the United States.



Technical Approach

Barriers to overcome	Technical Approach	CF & Composite Technology
1- Cost of Material	•Alternative Precursor •Low-cost, •High cconversion yield	1- Cost-effective, High yield Precursor & CF Material
2 - Cost & Scalability of Manufacturing Process	Innovative high throughput heat treatment process	2 - Cost Effective Manufacturing Processes/Processing
3 - High Energy Intensive Process	Innovative high throughput heat treatment process	3 – Energy-efficient CF & composite Processes/Processing
4 – Challenges in compositing applications	Innovative high-rate low cost intermediate and compositing processes	4 – Low-cost composites

Scaled-up Research & Development at CFTF

25 TONS ANNUAL CAPACITY

4 LICENSED TECHNOLOGIES

13 UNIVERSITY PARTNERS

3 NATIONAL LABORATORIES

78 INDUSTRY PARTNERS

3,500+ VISITORS SINCE 2013

Advancing Carbon Fiber Manufacturing Technology

The Carbon Fiber Technology Facility (CFTF), established in 2013, is the Department of Energy's only designated user facility for carbon fiber innovation. The CFTF, a 42,000 sq. ft. facility, provides a platform for identifying high-potential, low-cost raw materials, including textile, lignin, polymer, and hydrocarbon-based precursors. Using the CFTF, ORNL is developing optimal mechanical properties for carbon fiber material, focusing on structure property and process optimization.

The facility, with its 390 ft. long processing line, is capable of custom unit operation configuration and has a capacity of up to 25 tons per year, allowing industry to validate conversion of its carbon fiber precursors at semi-production scale. The CFTF supports the technology development and commercial deployment of carbon fiber in the United States for use in clean energy applications. Additionally, research focuses on further understanding the kinetics of carbon fiber manufacturing, energy consumption, and environmental impact.

Thermal (Conventional) Conversion Line
Rated for 25 tons/year of polyacrylonitrile (PAN)–based fiber with ability to convert both melt-spun and solution-spun precursors.

Melt-Spun Precursor Fiber Production Line
Rated for 65 tons/year of polyethylene fiber and designed to also spin lignin and pitch-based precursors.

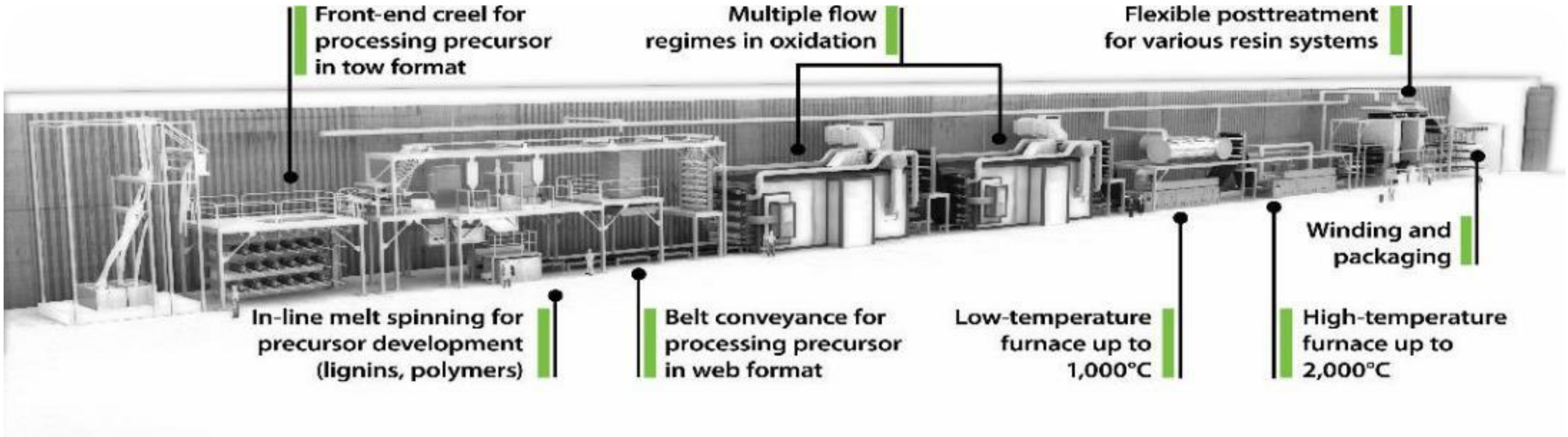
Pultruder
Speed range of 1 to 120 in./min. and enables manufacturing of custom composite materials with constant cross sections.

Steam Stretching
Enhances processability of textile-grade fiber with 32% reduction in oxidation time.

Textile Winder/Packaging
Provides packaging for large-tow textile carbon fibers for robust delivery.

Chopper
Designed to distribute chopped strands of fiberglass or carbon fibers; continuous carbon fiber chopping as small as 6 mm.

Carbon Fiber Technology Facility (CFTF)



The Carbon Fiber Technology Facility (CFTF) serves as a national resource to assist industry in overcoming the barriers of carbon fiber cost, technology scaling, and product and market development.

- Demonstrate carbon fiber production using lower-cost precursors and lower energy at semi-production scale.
- Produce low-cost carbon fiber available for evaluation and market development .
- Enable development of domestic commercial sources for production of low-cost carbon fiber.

- Highly flexible, highly instrumented low-cost carbon fiber technology demonstration facility
- Rated capacity 25 tons/year based on 24k PAN tows • Designed for PAN, polyolefins, lignin, and pitch precursors; upgradable for rayon and high-modulus carbon fibers
- Designed for 3k to 80k tows and web up to 300 mm wide x 12.7 mm loft
- Oxidation temperature up to 400°C with airflow configurable to be parallel, cross, or downflow
- Carbonization: Low-temperature carbonization up to 1000°C, High-temperature carbonization to 2000°C
- Post-treatment system designed for compatibilizing fibers with performance or commodity resins

Challenges associated with Carbon Fiber Scaling

- Availability and amount of precursor material required at scale
- Health and safety concerns
- Environmental concerns
- Capability and flexibility of Equipment (specifications)
- Process conditions control at scale (ex. Temperature & humidity)
- Precursor quality/reliability
- Fundamental Understanding of Carbon Fiber in the CF Manufacturing Process
- Manufacturing Costs
- Packaging
- Understanding the requirements of the downstream processing and applications

CFTF Objectives

Core Research and Development

Leveraging ORNL's Science Capabilities to Solve Challenges in carbon fiber and composites manufacturing.

Industry Collaborations

Cooperative research to develop and demonstrate low cost CF manufacturing to reduce the cost promoting and expanding the use of CF and its composites in clean energy applications

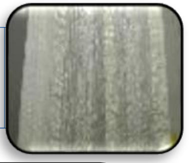
Education and Training

Internships, academic collaborations, workshops, training programs, and course curriculum for universities and community colleges.

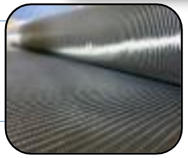
The Carbon Fiber Technology Facility

1. Establish and perform collaborative R&D projects to reduce technical uncertainties in CF manufacturing process

2. Investigate potential alternative carbon fiber precursors



3. Investigate carbon fiber intermediate forms and technical challenges in composite applications



4. Establish artificial intelligence-based framework and correlate process data to product characteristics

5. Investigate and develop in process measurement, sensing and control methods

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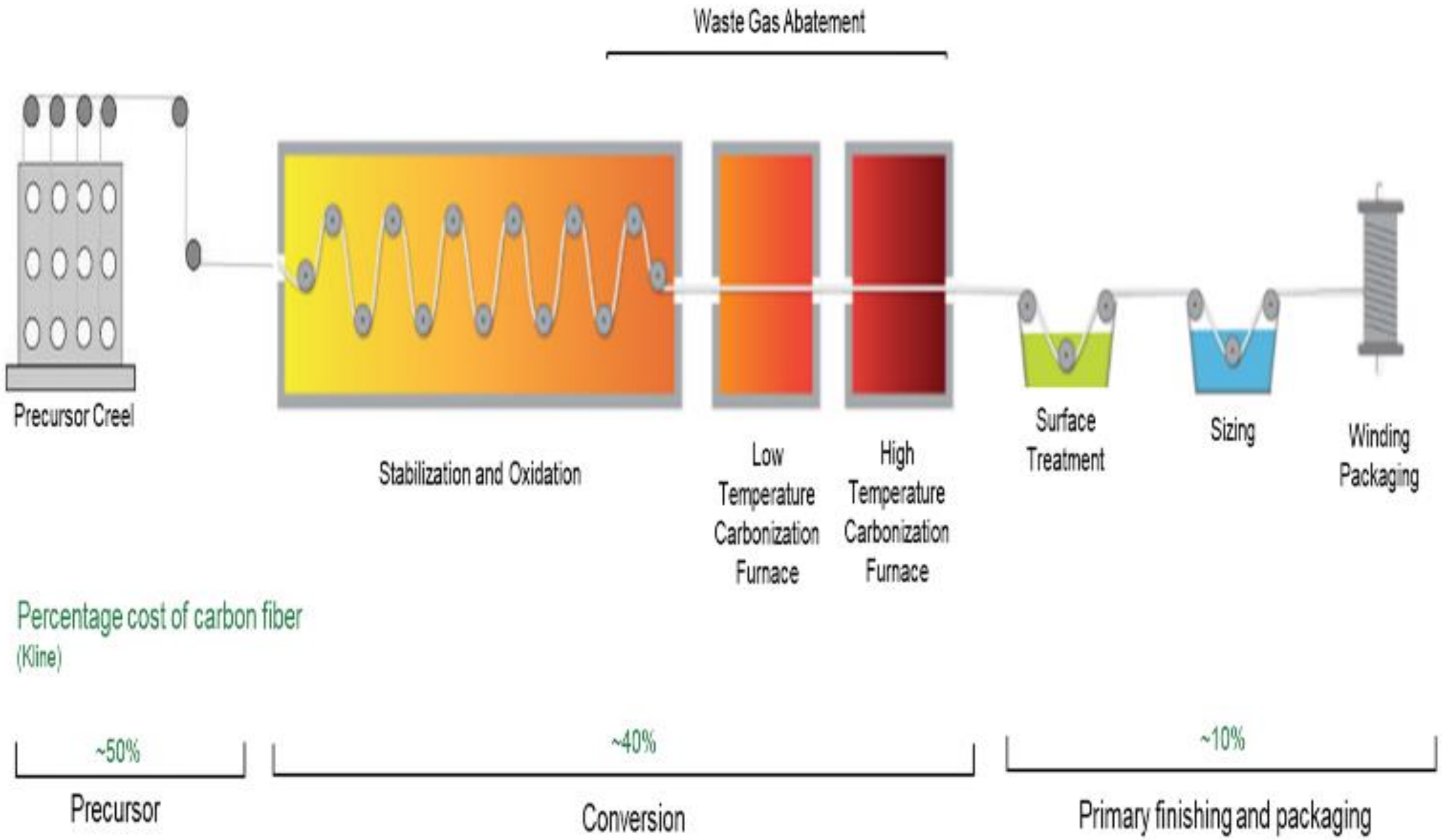
Textile Carbon Fiber Research & Development at ORNL



With conventional processing using a carbon fiber-grade (CF) PAN, precursor is over 50% of the carbon fiber cost

- 2x throughput
- IP developed
- ~60% Energy reduction in heat treatment
- ~ 50% manufact. cost reduction
- Textile Pan-base precursor made for textile industry processed into carbon fiber
- Equivalent properties, more specifically modulus, to conventional standard carbon fiber

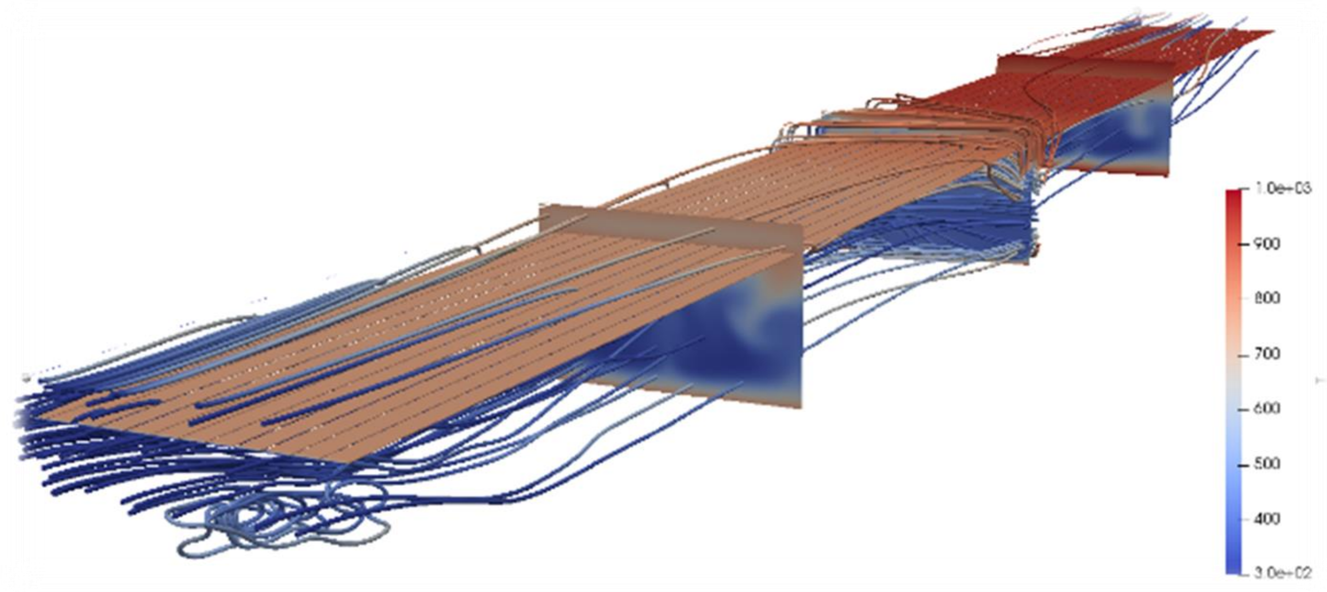
Oak Ridge National Laboratory

Cost Drivers – Raw Material, Capital, and Energy

Carbon Fiber Manufacturing – Computational Models for Carbon Fiber Carbonization

Comprehensive three-dimensional, multi-physics computational model for high capacity production of Carbon Fiber

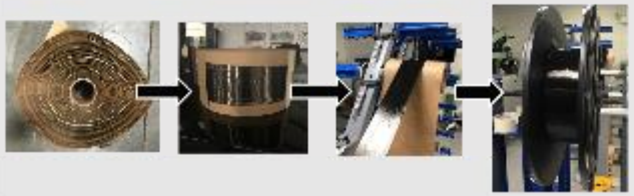
- Developed a blueprint for new, faster, more energy efficient processes which can enable the rapid introduction of carbon fiber reinforced polymer composites in the various applications.
- Ex…gas flow color coded with off-gas(CH_4) fraction distribution is shown. Also we can see heating in fiber tow bands due to radiation and non-uniform temperature distribution due to exhaust location and stagnation/re-circular region on the wall opposite to the exhaust where the off-gas fraction is maximum.



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.

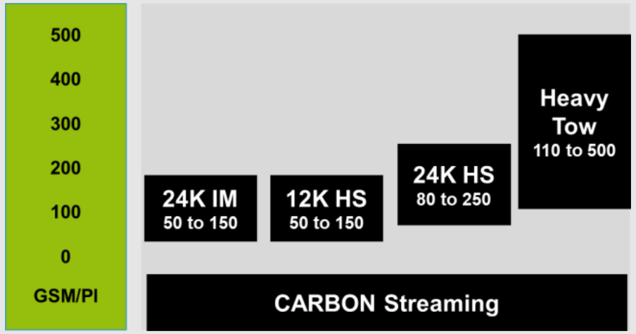
Leveraging ORNL Core Strengths and other programs (HPC4MFG)

Example of Joint Industry Projects



Develop commercial relevant packaging for TCF

Team: ORNL – IACMI



Non-crimp Fabric using TCF

Team: ORNL- IACMI

Pelletization & Compounded Textile Carbon Fiber

- Techmer compounded PA66 with 10%, 25%, and 40 % LCCF

Team: ORNL-IACMI

Sheet Molding Compound Reinforced by Recycled or Textile Carbon Fibers

Team ORNL-IACMI

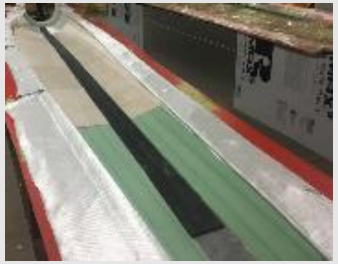
TCF in Prepreg production

Team: ORNL-IACMI

Develop Chopping Equipment for chopping TCF at various length with various Sizing

Team: ORNL- IACMI

Example of Joint Industry Projects



Pultrusion of carbon fiber wind turbine spar caps has demonstrated cost reductions and improved performance versus infusion.

Use of textile carbon fibers (TCF) will lower that further. Conventional pultrusion is not designed for large tow form typical of the TCF.

Team: ORNL-IACMI

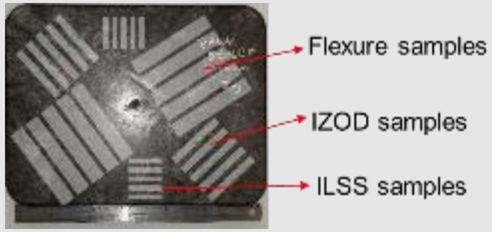
Fenders
75 secs cycle time

https://www.youtube.com/watch?v=NRk_v3fPyCI&t=163s

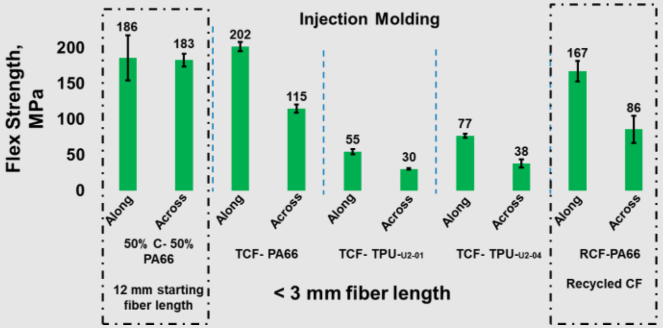
		40% CF in PA66		10% CF in PA66	
Property	Units	Commercial CF	CFTF Fiber	Commercial CF	CFTF Fiber
Tensile Strength @ Yield	Psi	37,000	28,100	21,700	19,800
Tensile Elongation @ Break	%	1.8	1.0	2.4	2.2
Flexural Strength	Psi	37,300	37,000	30,000	28,500
Flexural Modulus	Psi	3,900,000	3,840,000	1,000,000	1,260,000
Notched izod	ft-lb/in	1.39	1.01	0.50	0.60
HDTUL @ 264 psi	°F	489	484	479	468
Surface Resistivity	ohms/sq	2.20E+03	1.10E+04	3.20E+05	1.99E+12

Injection Molded Saturn Fenders using
Team: ORNL-IACMI-MSU

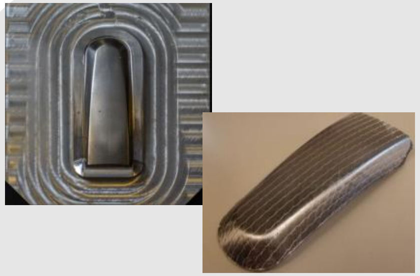
Injection molding → Battery Tray



Textile Thermoplastics Battery Tray Flex Molds
Team: ORNL-IACMI



Textile carbon fiber Performance Database (Coupon Testing) use in design, modeling and application development
Team: ORNL -IACMI



Bike Form produced using Non-crimp textile carbon fiber fabric and Elium Resin via compression molding

Team ORNL-IACMI

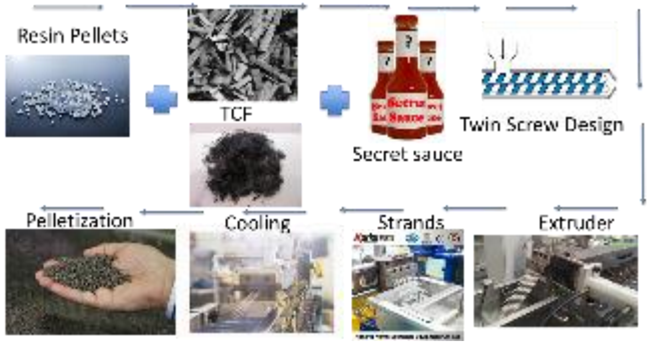
Car hood Mold Printed using the BAAM System

Textile carbon Fiber in Additive Manufacturing

Team ORN-IACMI

OAK RIDGE National Laboratory

Example of Joint Industry Projects



Developed low-cost carbon fiber

Compound 10% LCCF pellets with PA66

Injection molded parts

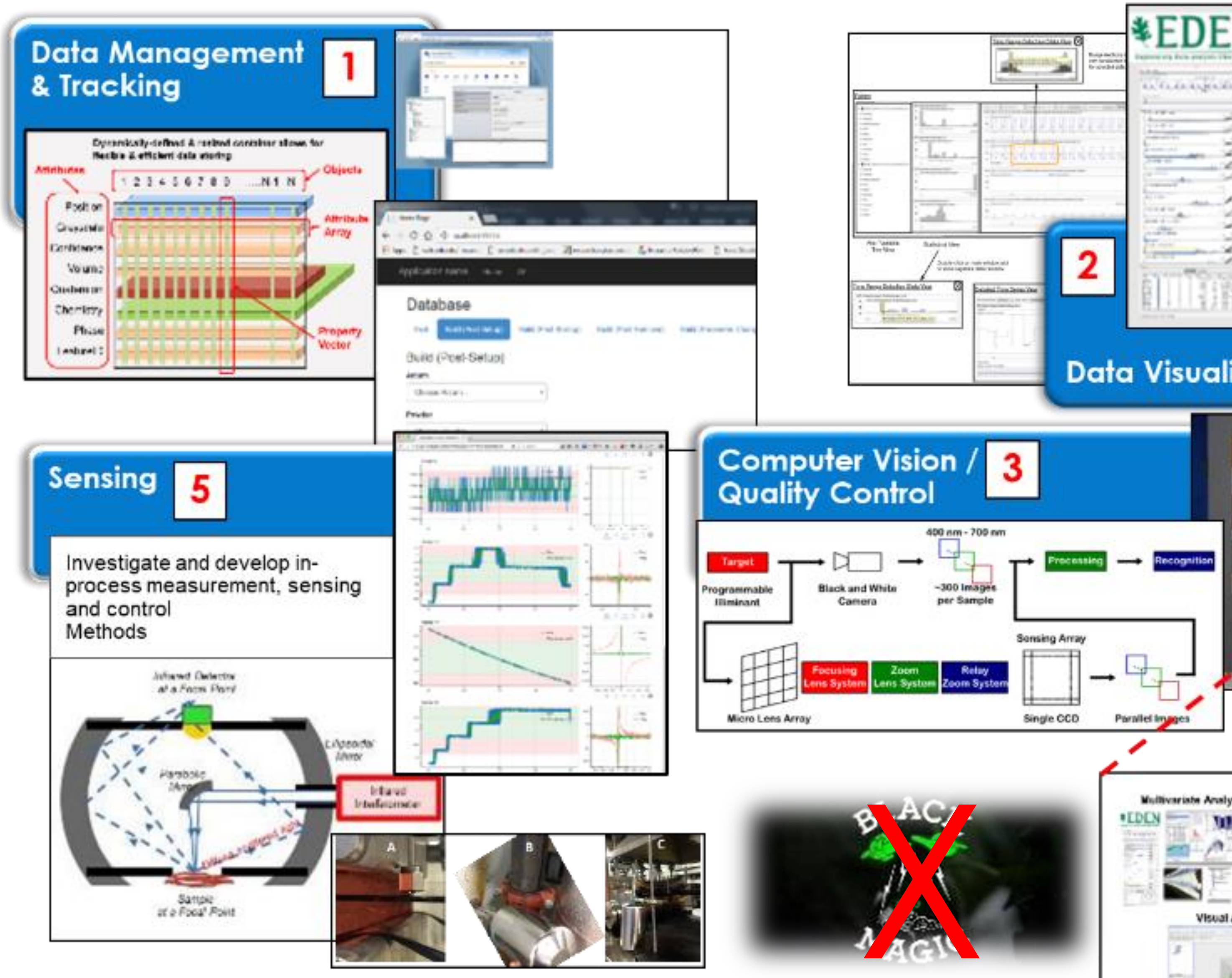
OAK RIDGE National Laboratory

TECHMER PM POLYMER MODIFIERS

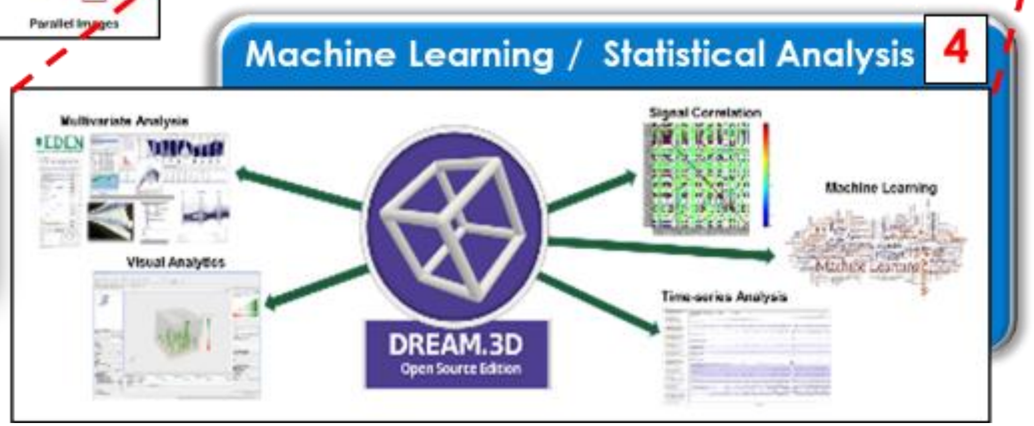
iacmi

			10% CF in PA66	
Property	Units	Method	Commercial CF	CFTF Fiber
Tensile Strength @ Yield	Psi	ASTM D638	21,700	19,800
Tensile Elongation @ Break	%	ASTM D638	2.4	2.2
Flexural Strength	Psi	ASTM D790	30,000	28,500
Flexural Modulus	Psi	ASTM D790	1,000,000	1,260,000
Notched izod	ft-lb/in	ASTM D256	0.50	0.60
HDTUL @ 264 psi	of	ASTM D648	479	468
Surface Resistivity	ohms/sq.	ASTM D257	3.20E+05	1.99E+12

Data Analytics Framework for CF Manufacturing



Data Analytic Framework enable process variables to be tuned and optimized during manufacturing **improving quality throughput**, better processability, capability & control thus **efficiency reducing energy usage**, **scrap**, and ultimately the **cost** of manufacturing.



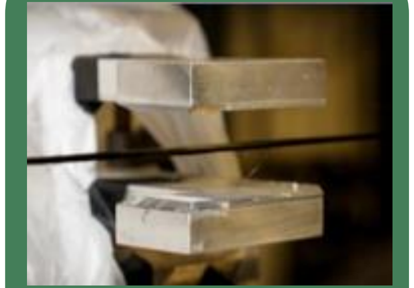
Insitu-Measurement Concepts Developed

Sensing Module (5) in Data Analytic Framework
In-situ measurements provides valuable information and characteristic of the precursor material and how it changes at different stages within the process. Combining this information with information in the other Modules of the data analytic framework will help design a closed loop system in CF manufacturing.

Reactive CF Process → Predictive/Proactive Process

Prototype Magnet Susceptibility Sensor

Relationship between degree of oxidation and crosslinking is expected to be observable through magnetic susceptibility measurement. Value of magnetic susceptibility of fiber tow can be measured and compared with a strength-related set point.

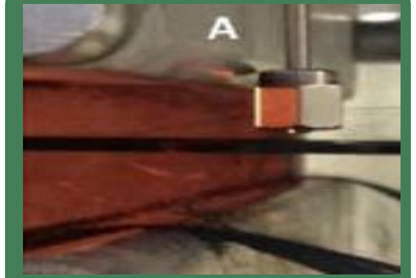


Prototype Electrical Impedance Sensor

**The use of software defined radios as a low-cost method to wirelessly probe the carbon fiber tow to determine material properties of the tow. Data can be correlated with process conditions.*

DRIFTS Sensor at CFTF

The application Fourier Transform Infrared (FTIR) spectroscopy techniques will enable measurement of the specific fiber chemistry associated with crosslinking and cyclization reactions characterizing the chemical bonds present in the fiber material



Off-gas Analysis Sensor

Off-gas measurements Extractive samples is analyzed via standard methods including MS, FTIR and GCMS to identify the control species, which in process-control applications can be monitored via the real-time analytical tech. or other in-situ sensors.

Raman Spectroscopy

Online, non-invasive indication that fiber coming from high temperature furnace adequately carbonized.

Multispectral

In-line and off-line multispectral measurements of the fibers, to capture the colorimetric information used by operators to estimate the quality of the fiber along the production line

**"Carbon Fiber Tow Inspection Technique Using Software Defined Radios" James R. Humphries*, Roger A. Kisner, Stephen M. Killough*, Merlin Theodore, and Yarom Polsky, Sensor Application Vol. 2, No. 4, Dec. 2018*

OAK RIDGE National Laboratory

Carbon Manufacturing R & D Collaboration

Ever Growing Partnerships: Integrating the AM Supply Chain

Materials Suppliers

- KALTEX
- TAEKWANG
- Birlacril
- Bluestar Fibres
- SGL GROUP THE CARBON COMPANY
- Montefibre
- MICHELMAN

Equipment Suppliers

- EISENMANN
- McCOY
- LITZLER DRIVEN BY RIGHT
- CDI
- Harper
- MAE

End Users

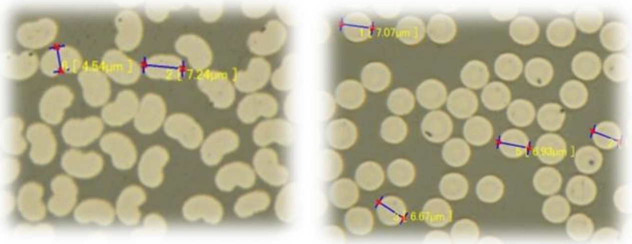
- iacmi
- CHOMARAT
- United Technologies
- NATIONAL SPINNING CO., INC.
- Ashland always solving
- HONDA
- AvCarb
- Lawrence Technological University
- Martin Pultrusion Group
- ARKEMA
- faurecia
- MEGGITT smart engineering for extreme environments
- ASCEND MANUFACTURING
- AUBURN UNIVERSITY
- LeMond COMPOSITES
- OWENS CORNING
- AsahiKASEI ASAHI KASEI PLASTICS NORTH AMERICA
- AIR FORCE RESEARCH LABORATORY
- CRTC COMPOSITE RECYCLING TECHNOLOGY CENTER
- Diversified Structural Composites
- GREEN DYNAMICS
- Tennessee Tech University
- Composites UK Hub
- TECHNICAL FIBRE PRODUCTS LTD
- MAGNA
- MICHIGAN STATE UNIVERSITY
- IMERYS
- ZOLTEK Toray Group
- POLIMOTOR
- NC STATE UNIVERSITY
- PLASTECH CORPORATION
- CHOMARAT
- PLASTIC OMNIUM
- RMX TECHNOLOGIES
- TECHMER PM POLYMER MODIFIERS
- Continental Structural Plastics
- VECTORPLY
- WEBER STATE UNIVERSITY
- TOUCHSTONE RESEARCH LABORATORY, LTD.
- MITSUBISHI HEAVY INDUSTRIES
- SUMITOMO BAKELITE CO., LTD.
- EPSILON COMPOSITE The alternative
- Milliken

Scaled-up Research & Development at Manufacturing Demonstration Facility (MDF)

Vlastimil Kunc
Project Lead
Manufacturing Science Group

Manufacturing Challenges and Opportunities

- Format of low cost carbon fiber
    - Processing of large tow fibers
    - Tow splitting
    - Accepting “fuzzy fiber”
- Liner performance
    - Permeability
    - Thermal Properties
- Boss and Liner integration
- Structural Health Monitoring
- Recycling



From 6 μm fiber

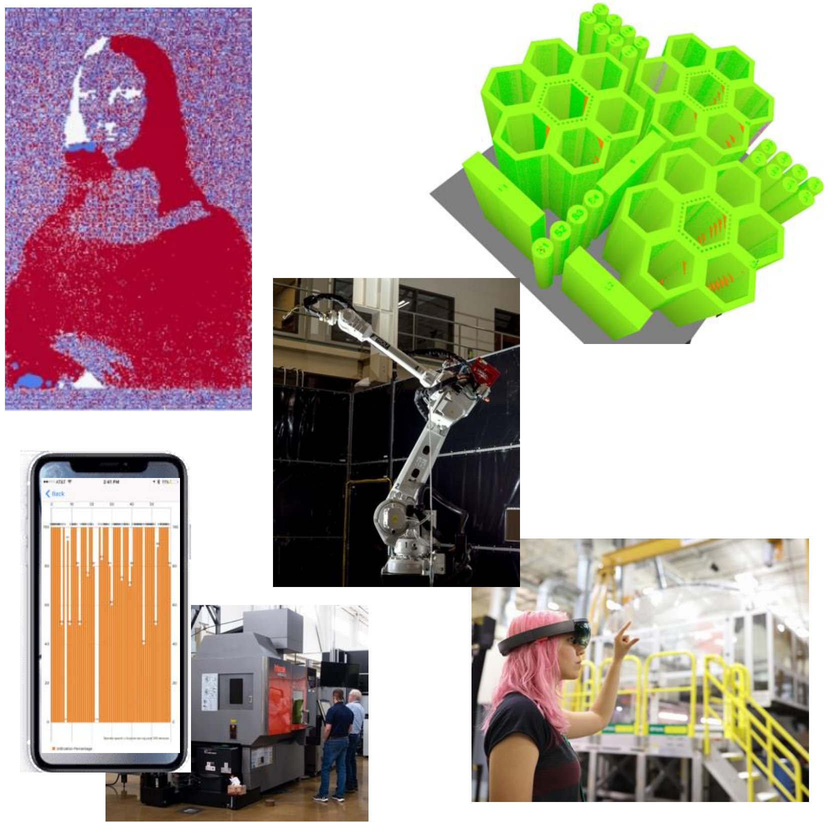
To 2.4 m wide vehicle

Manufacturing Demonstration Facility

Core Research & Development Goals:

1) **Improve Performance of Advanced Manufactured Components**
2) **Establish Framework for Born Qualification of Advanced Manufactured Components**
3) **Develop Next Generation Manufacturing**
4) **Enable Technologies for Digital Factories**
5) **Develop Future Manufacturing Leaders**

Core R&D	Industry Collaborations	Education and Training	Demonstrations
• R&D in materials, systems, and computational applications to develop broad of additive manufacturing	• Cooperative research to develop and demonstrate advanced manufacturing to industry in energy related fields	• Internships, academic collaborations, workshops, training programs, and course curriculum for universities and community colleges.	• Executing on "moon shot" demonstrations to integrate partners, understand technical challenges, and accelerate technologies.



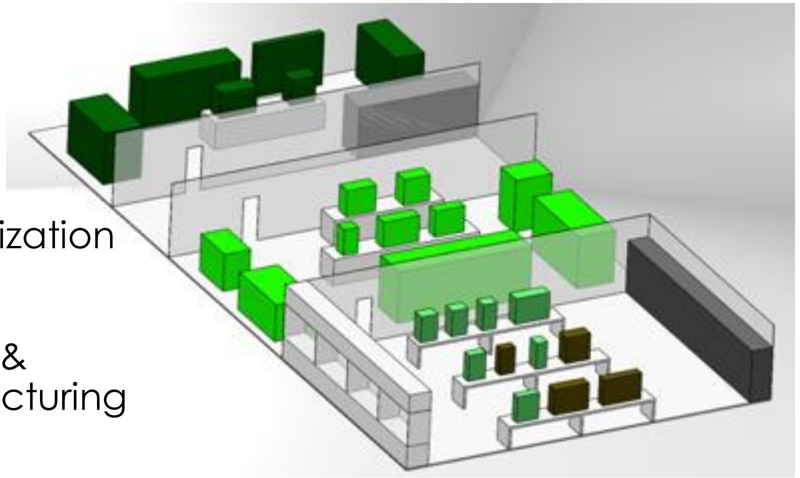
Oak Ridge National Laboratory

Rapid Innovation and Commercialization Lab Floor Plan

No currently available open research facility with the combined capabilities

Rapid Innovation Capability

Low Bay
50’ x 100’
- Characterization
- Bench Top Feasibility, Recycling & Remanufacturing



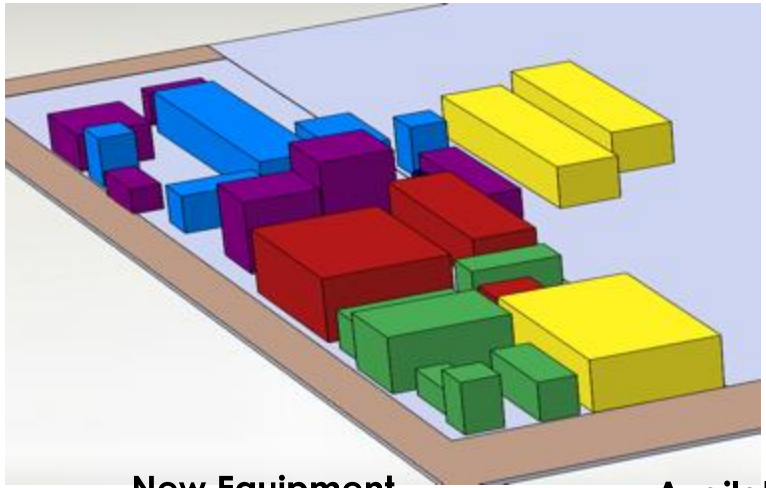
New Equipment

Lab-Scale Recycling & Recovery

Manufacturing

Characterization

Scale up Capability



High Bay
50’ x 200’
- Commercial Scale Manufacturing Systems
- Recycling & Recovery Equipment

New Equipment

Recycling & Recovery

Available Equipment

Thermosets

Thermoplastics

Additive Manufacturing

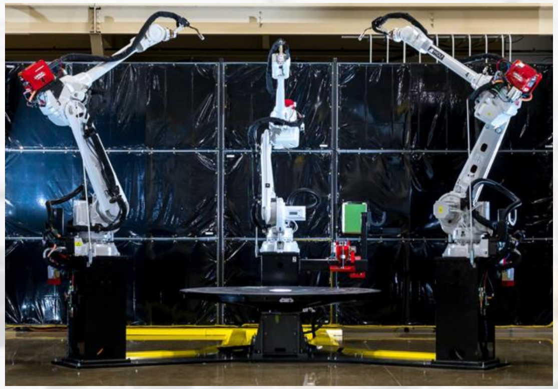
Automation and Molding

<u>Circular economy supporting capabilities:</u>
- **Green** boxes are new equipment
- Other colors are MDF existing capabilities

Advanced Manufacturing Capabilities

~***70*** *advanced systems, of which 60%* ***(>$20M)*** *is industry-provided*

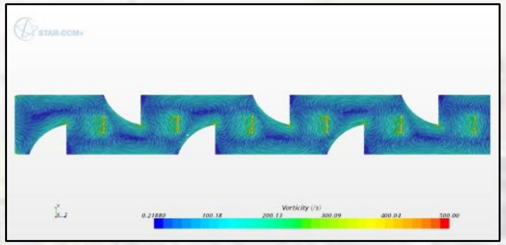
Large-scale metal AM



Polymers AM

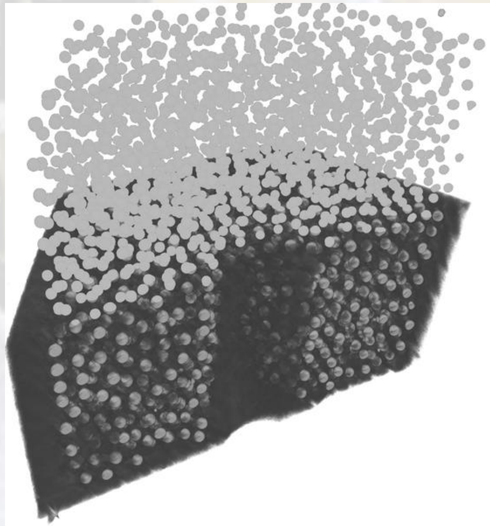
Composites Processing

Simulation and Data Analytics

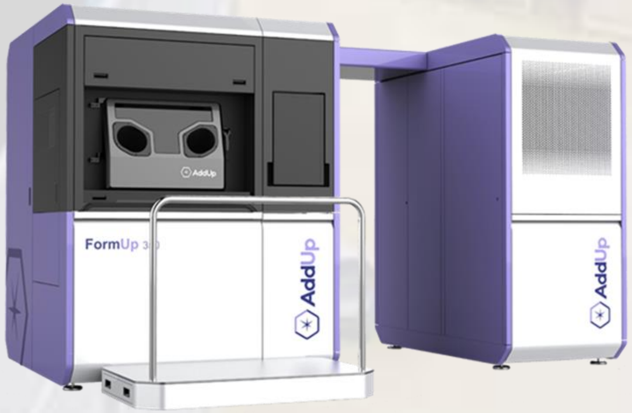


Machining

Metrology & characterization



Metal powder systems



MDF by the Numbers

170 partnerships

100 publications/year

131 awards since MDF's inception

80-**100** internships per year

More than **50** university collaborations

Over **60** advanced manufacturing systems

More than **110,000** sq. ft. of facility space

82 staff members

Over **$30M** of equipment (**$20M** industry-provided)

More than **4,100** visiting companies since 2012

Over **28,150** visitors since inception

Impact of over **$1B** for U.S. manufacturing

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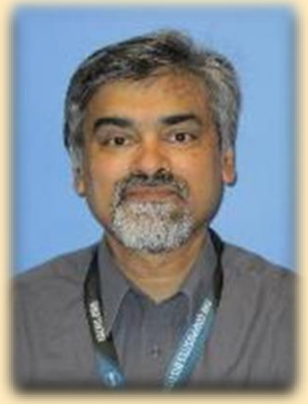
Composites



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Thank You

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