Carbon Fiber Technology Facility

Merlin Theodore, PhD (PI)
Director, Carbon Fiber Technology Facility
theodorem@ornl.gov
(865) 576-6569

Amit Naskar, PhD (Presenter)
Chief Scientist, Carbon Fiber Technology Facility

Alan Liby, PhD
Director, Advanced Manufacturing Program

2017 U.S. DOE Vehicle Technologies Office Annual Merit Review

June 8, 2017 Project ID LM121

This presentation does not contain any proprietary, confidential, or otherwise restricted information

Overview: Carbon Fiber Technology Facility (CFTF)

Timeline

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

Barriers addressed

- Cost of carbon fiber
- Technology scaling to meet high volume manufacturing need
- Market development
- Workforce development

Budget

- $5M in FY 2017
  - $4M AMO
  - $1M VTP
  - Carry-over ($0.3M)

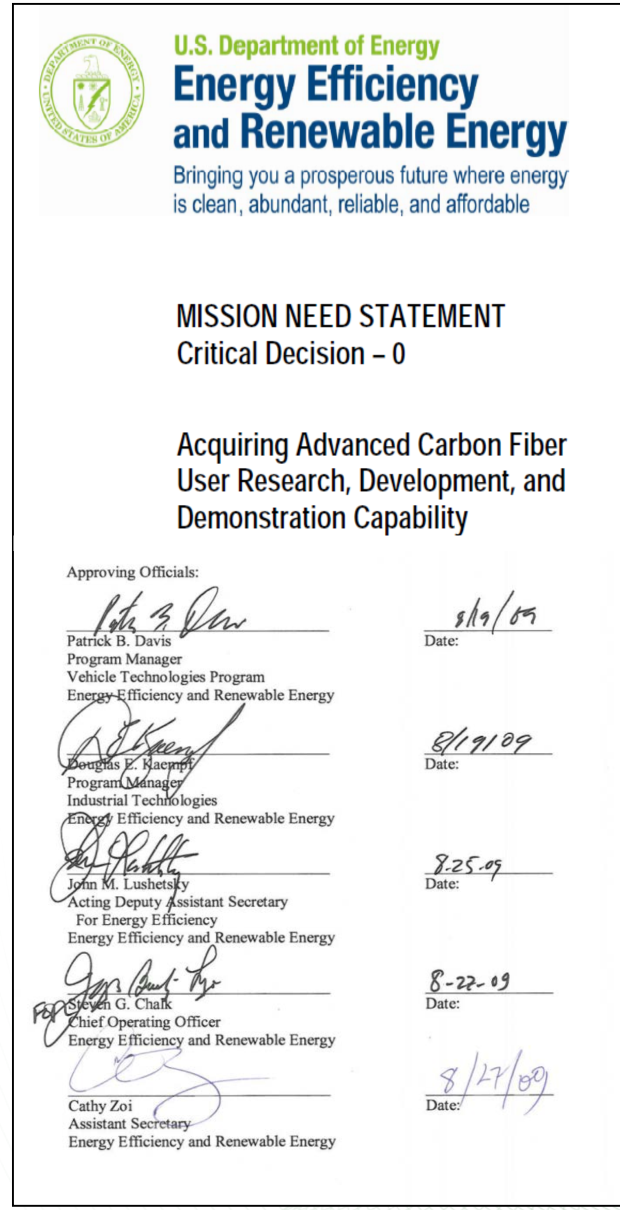
Partners and Collaborators

- Institute for Advanced Composite Manufacturing Innovation - IACMI
- Technical Collaboration Projects & CRADA's
  - More than 6 active projects underway or pending AMO approval
  - two CRADA in negotiation

Relevance: The nation needs a scale-up facility for low-cost carbon fiber technology demonstration

75 Stakeholders from government and industry attended a workshop at ORNL in March 2009

"DOE does not…possess the advanced carbon fiber research, development, and demonstration capability necessary to achieve its mission goals...need to develop a multi-industry, multi-participant technology demonstration and deployment facility that can be easily accessed by researchers and stakeholders for key industries."



U.S. Department of Energy
Energy Efficiency and Renewable Energy
Bringing you a prosperous future where energy is clean, abundant, reliable, and affordable

MISSION NEED STATEMENT
Critical Decision – 0

Acquiring Advanced Carbon Fiber User Research, Development, and Demonstration Capability

Approving Officials:

Patrick B. Davis, Program Manager, Vehicle Technologies Program, Energy Efficiency and Renewable Energy — Date: 8/19/09

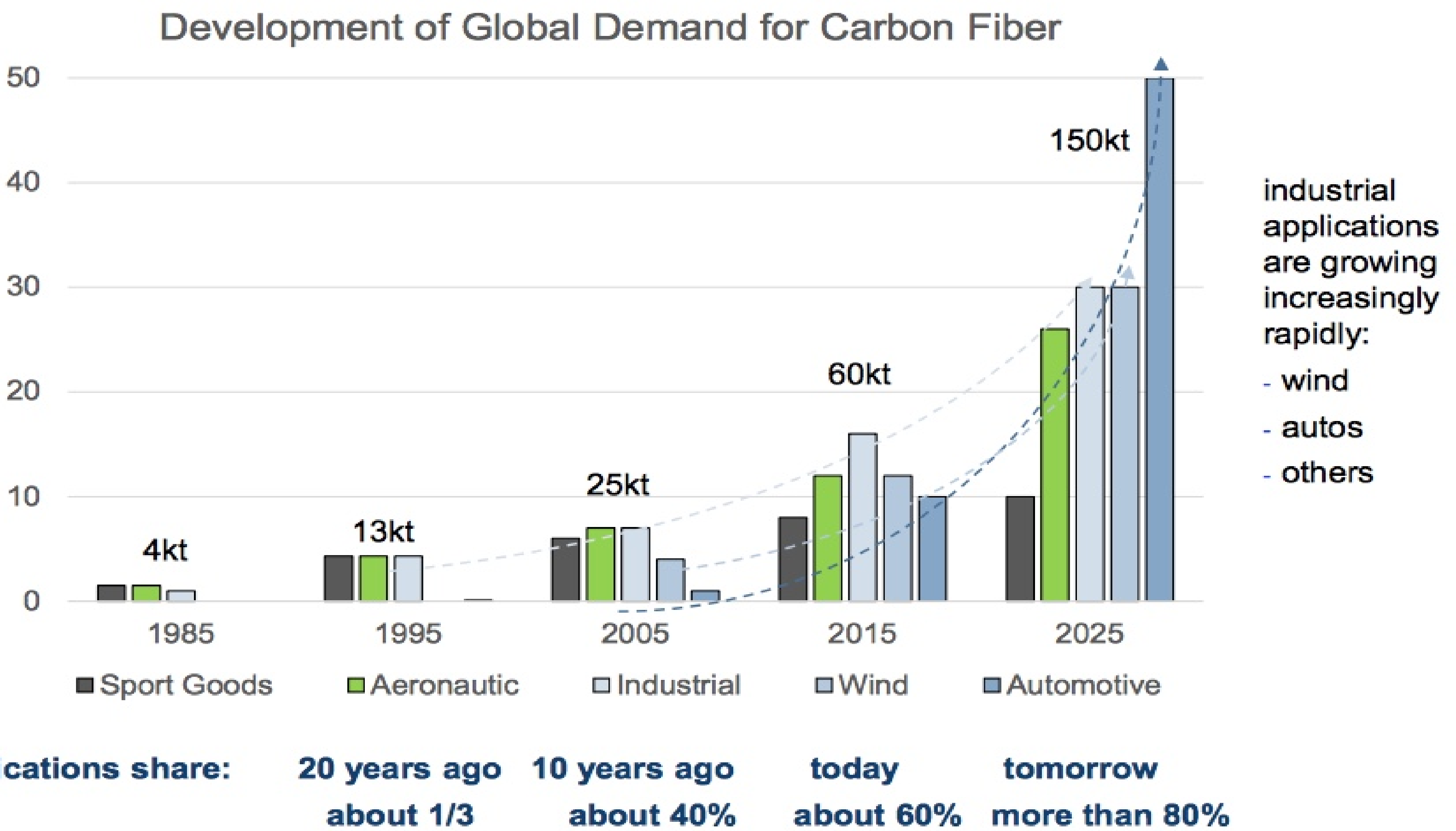
Douglas E. Kaempf, Program Manager, Industrial Technologies, Energy Efficiency and Renewable Energy — Date: 8/19/09

John M. Lushetsky, Acting Deputy Assistant Secretary For Energy Efficiency, Energy Efficiency and Renewable Energy — Date: 8-25-09

Steven G. Chalk, Chief Operating Officer, Energy Efficiency and Renewable Energy — Date: 8-22-09

Cathy Zoi, Assistant Secretary, Energy Efficiency and Renewable Energy — Date: 8/27/09

Relevance: Potential automotive market alone is huge for low-cost carbon fiber



Daniel Pichler "CarbConsult, Germany Status and Outlook for the Maturing Carbon Fiber Industry" Carbon Fibre Futures Conference (2016)

Milestones: FY16 Accomplishments

- Temperature uniformity was measured across the oxidation ovens 1 and 3 with three different temperatures 200°C, 215°C, and 230°C. Due to the location of the controlling thermocouple and the lower fan speed, the temperature uniformity is about 5°C lower than the stated temperature on the sides of the belt and 2°C lower in the middle portion of the belt.
- Study with textile PAN lot-to-lot variability were completed. Lots TE4571150901 and TE4571151006 were produced from 2.0 denier, high-tenacity precursor fiber. Twelve specimens were prepared and tested for each band. Comparison of mechanical property data for both tensile strength and modulus showed only a small variation between the two lots.
- Experiments conducted with other textile PAN precursors. Understanding of the chemistry of the precursors and processibility was developed.
- In situ monitoring and control techniques were identified through early proof of principle.

Background: CFTF Objectives

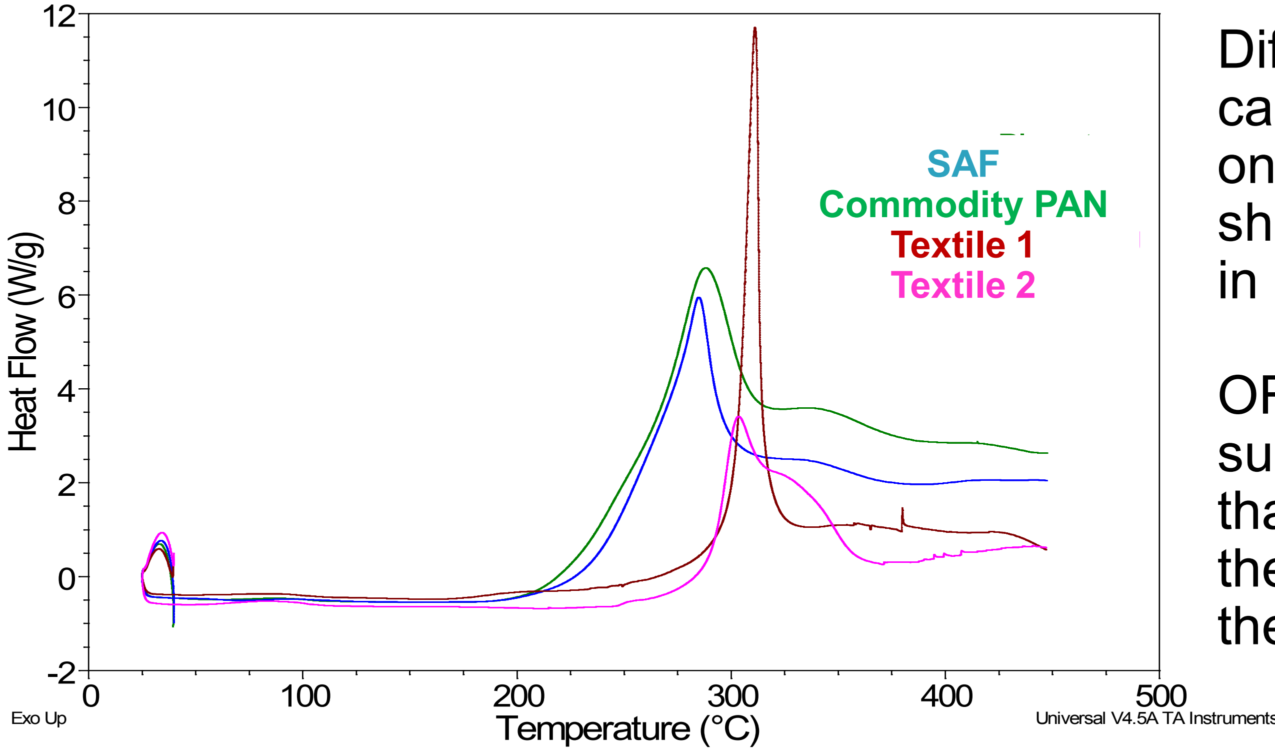
- Demonstrate carbon fiber production using lower-cost precursor materials at semi-production scale.
- Produce and make available low-cost carbon fiber in sufficient quantity to enable evaluation and market development for expanded commercial application of carbon fiber composites.
- Enable development of domestic commercial sources for production of low-cost carbon fiber.

Background: Technical Approach

Integrated approach to low-cost carbon fiber manufacturing R&D

- Identify high potential, low cost alternative precursors
  - PAN-based Textile Precursors
- Develop optimal mechanical properties of resultant carbon fiber from alternative precursors
- Provide sample quantities to industrial partners for testing based on DOE approval
- Address feedback from industrial partners
- Improve carbon fiber manufacturing cost metrics
- Commercialization

Technical Accomplishments and Progress: High throughput conversion of the low-cost precursors offers a key advantage

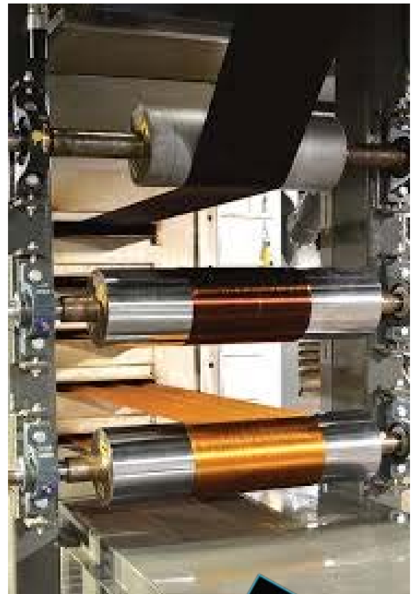


Differential scanning calorimetry (DSC) runs on the precursors show potential difficulty in stabilizing textiles!

ORNL team successfully overcame that deficiency during thermal oxidation of the precursor fibers.

Method of Producing Carbon Fibers from Multipurpose Commercial Fibers, (ORNL ID 3583), Pending U. S. Patent Application

Technical Accomplishments and Progress: Early low-cost carbon fiber production at CFTF using a textile acrylic fiber



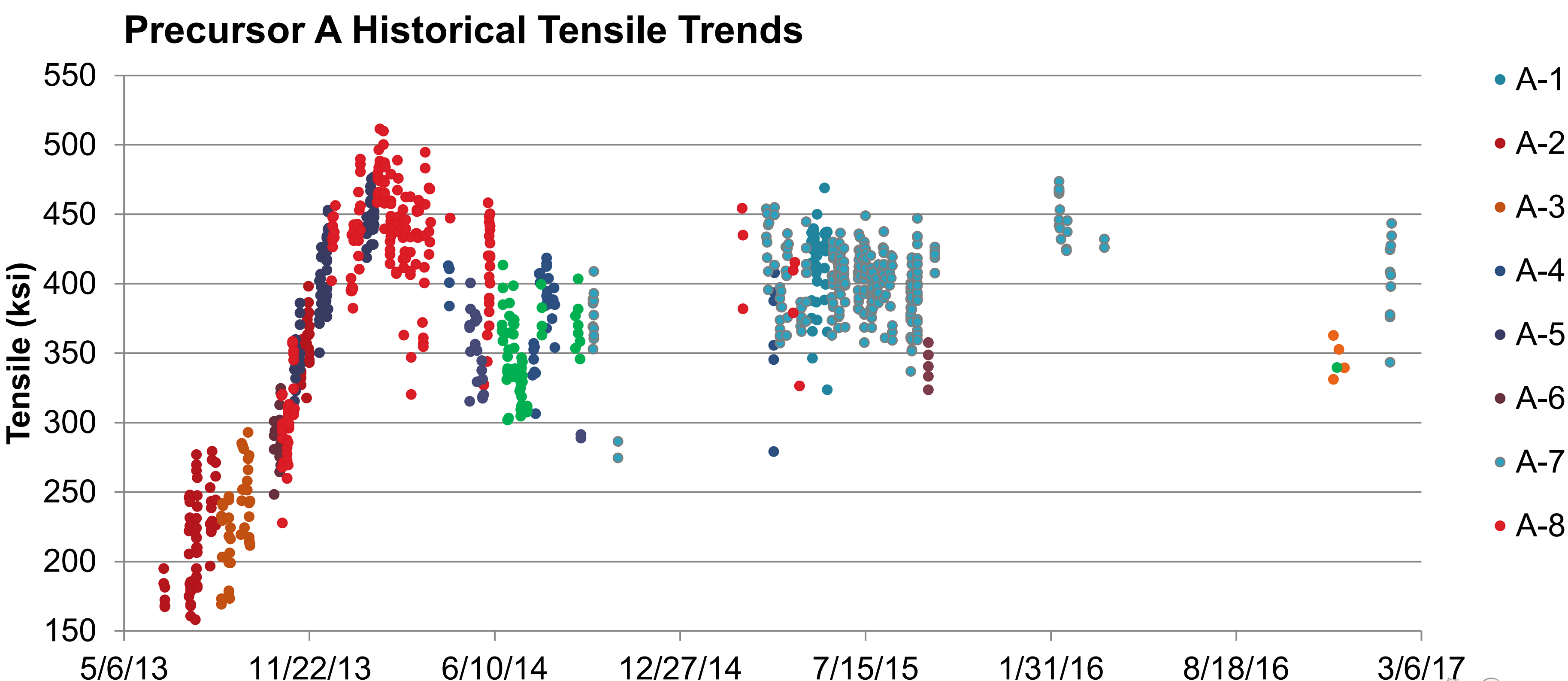
Carbon fiber is produced in a very large tow form (320 – 610 K Tow)

Technical Accomplishments and Progress

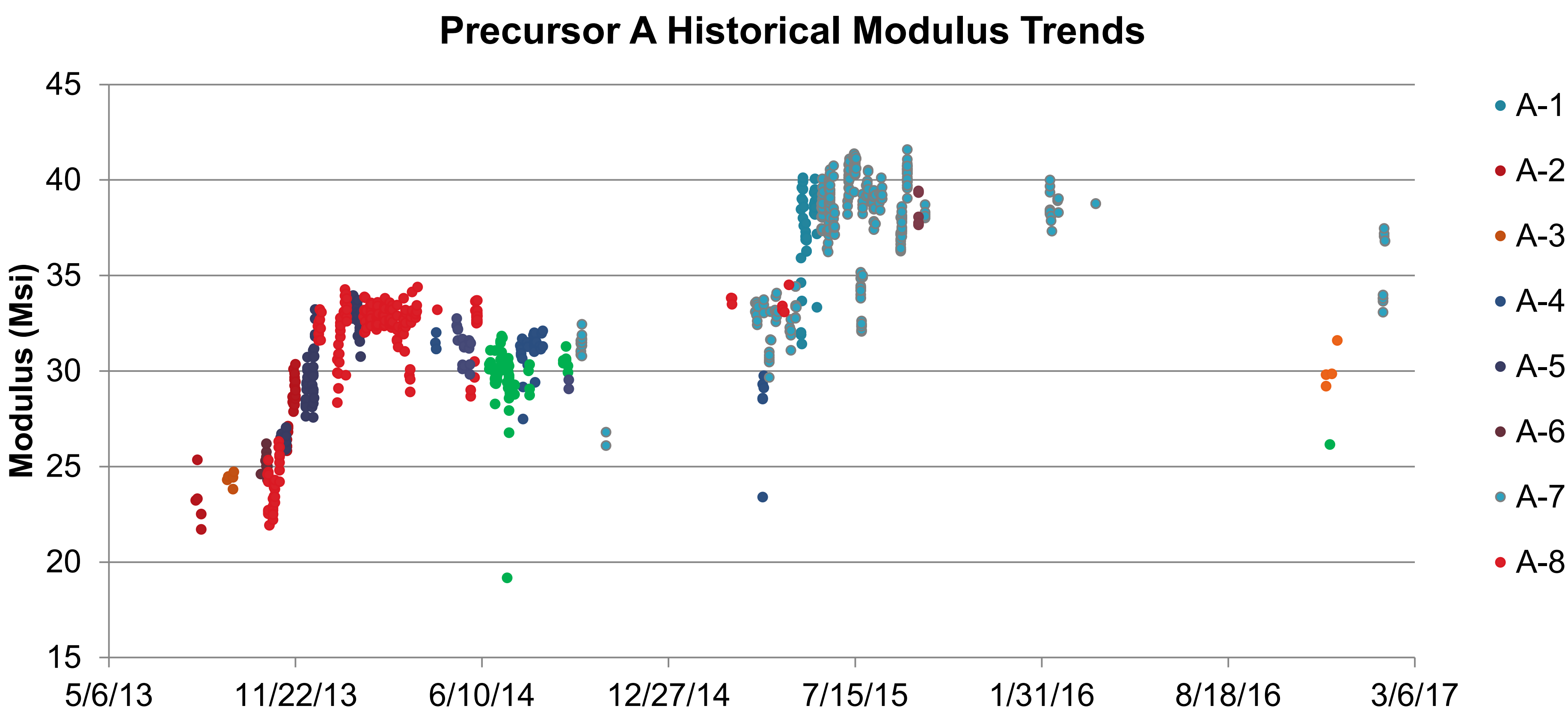
Technical Accomplishments and Progress: Mechanical properties achieved with T-PAN have steadily improved over time

Designed and executed a series of test matrices based on technical knowledge of the process conditions and their relationship to the precursor polymer.

Approaching final stages of development with this precursor. Next phase will focus on repeatability and line speed increases.



Technical Accomplishments and Progress: Mechanical properties - continued

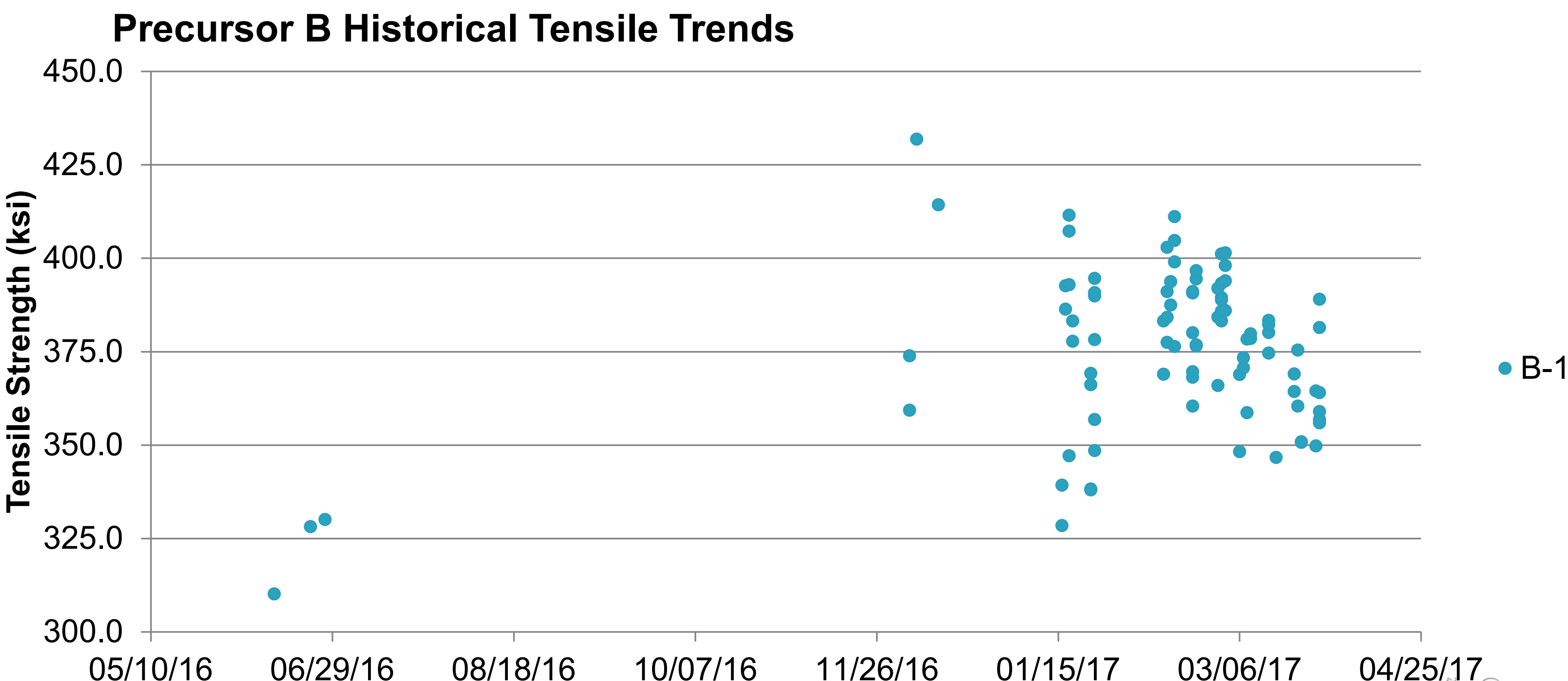


1/15/13

Technical Accomplishments and Progress: Mechanical properties achieved with T-PAN have steadily improved over time

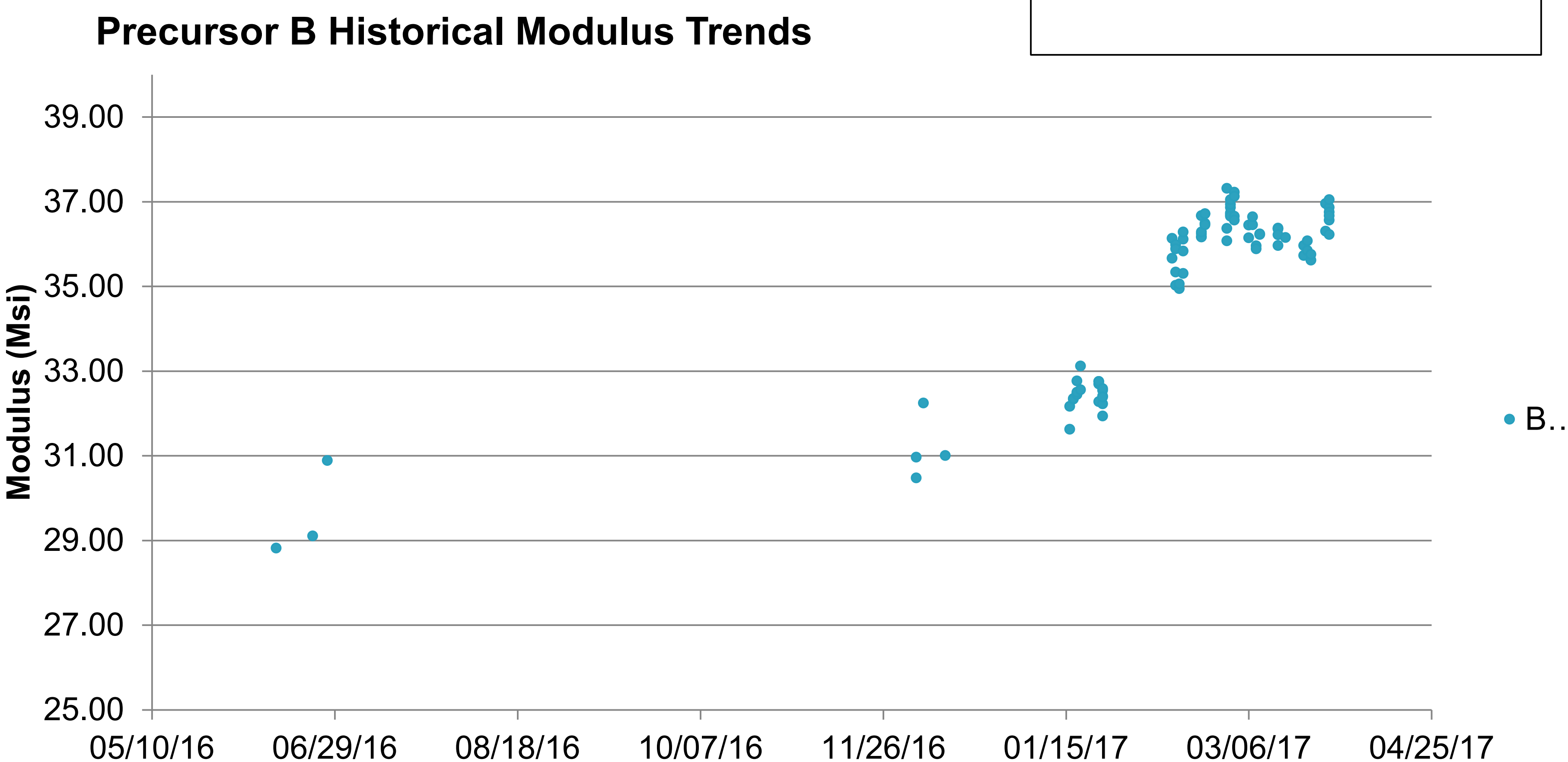
Precursor B with alternative comonomer chemistry, required longer oxidation time based on thermal kinetics results performed by ORNL; therefore, altering composition was recommended to the precursor manufacturer and supplier.

- Oxidation time was decreased significantly
- Technical Strength was increase by 15%



Technical Accomplishments and Progress: Mechanical properties - continued

- Modulus was increase by 20%



 Managed by UT-Battelle
for the U.S. Department of Energy

Technical Accomplishments and Progress: Summary of precursor types and properties achieved

- Demonstrated large volume carbon fiber production using multiple textile polyacrylonitrile (PAN) precursors at semi-production scale
- Demonstrated reproducibility of processing conditions with multiple precursor lots
- Demonstrated commercially viable properties at ~50% reduction in energy consumption and production cost based on volume throughput
  - 510ksi and 33msi – trials to maximize strength (Precursor A)
  - 467ksi and 40msi – trials to maximize modulus (Precursor A)
  - 382ksi and 36msi – trials to maximize strength and modulus (Precursor B)
- Publically announced breakthrough and initiated licensing negotiations for commercialization

Commercialization Accomplishments and Progress : LeMond Composites opens production facility based on ORNL license

A new composites company founded by Tour de France champion Greg LeMond has signed a licensing agreement with the Oak Ridge National Laboratory to commercially produce low cost carbon fiber. The agreement will make the Oak Ridge-based LeMond Composites the first company to offer this new carbon fiber to the transportation, renewable energy, and infrastructure markets and will result in:

- 242 new, highly skilled jobs
- $125M investment locally
- 10 lines producing 140 million lbs by 2020
  - 16-20 million lbs in the first year
- New production method reduces production cost by more than 50% and energy consumption by more than 60%

Response to Reviewers' Comments (Peer Review by AMO Panel on 8/18/16)

Overview-Comments

FY 2016 accomplishments are very significant and major step forward for achieving low cost carbon fiber. FY 2017 plans appear appropriate:

- continue collaborative developments for LCCF; produce materials for prototyping & composites development; process development for alternative textile precursors; continue development of process control & optimization.

We thank the reviewers for their critical comments in specific areas and support to this project. During the last few years, we have strived to make the best use of this national resource by reaching out to industry and establishing a number of collaboration projects. These projects, which are taking the low-cost materials produced at the CFTF and implementing it into composite applications, are intended to create the industry pull needed to drive full scale commercialization of this technology. And this is only the first low-cost precursor material to be scaled – we hope to repeat this process several times over in the future.

Operation -Comments

Validation of one commercial precursor is most significant accomplishment: 2x – 3x increased throughput under continuous running conditions, lot-to-lot variability control, commercial viable properties.

Other precursors evaluated show some potential but need further study.

CFTF is currently evaluating other precursors (see Precursor B results) and associated methods for high throughput manufacturing of LCCF.

Inline process control development shows some potential but needs further development. Concerned about prioritization, timing, & cost of this task compared to needed efforts material development.

The team is currently analyzing the existing processing dataset. However, volume of fiber production with consistent properties and optimization of process parameter remains top priority.

Response to Reviewers' Comments (cont'd)

Major concern over delays created by failures of the HT furnace. This needs to be resolved quickly to allow developments to continue.

> **It got resolved in December 2016 and LCCF production (with HT operation) resumed immediately. HT downtime, however, was utilized to optimize oxidation conditions for precursor B.**

Industry Collaboration and Composite Development-Comments

Industry collaborations are very good for advancing the LCCF technology and acceptance & interest by industry user partners. Composite property data are highly dependent on fiber sizing, and other published data on Zoltek fiber with best sizing for PA6 show much higher properties. While the properties of LCCF fiber show promise, we should be wary of these comparisons.

> **We appreciate the comment. These composite performance analyses are early comparison data produced. We are currently producing LCCF with two different sizing to address compatibility of LCCF with polymer matrices.**

Faurecia collaboration and data look excellent. Faurecia is high probability user partner for LCCF technology. LCCF fiber properties translation are very promising and evaluation of different intermediate product & process concepts is good. More of this type of development is needed.

> **We thank the reviewer for this supportive comment.**

Good direct comparisons of properties for continuous composites using C-Ply product format and VARTM processing. Data shows good translations of LCCF properties. Testing at multiple sites is good. Need to use 2-sided hard tooling to get smooth surfaces on both sides of test panels; this will improve data & variability. This type of development needs to be expanded to look at other intermediate product forms and ways to use the wide tow LCCF product format.

> **At this moment CFTF team is relying on IACMI and industrial partners on composite performance analysis due to lack of CFTF's resources and programmatic fund for such R&D. ORNL composite team is also working on sizing development for LCCF material produced at CFTF using UT-Battelle's internal resources.**

Response to Reviewers' Comments (cont'd)

R&D - Comments

Great work; provides a better understanding of behavior of commercial precursor chemistries in LCCF process, allowing use of exothermic behavior as advantage for increasing throughput.

Continued R&D is extremely important: Precursor stretching effects on oxidation kinetics and end product properties; Studies on spin finishes that can impact process kinetics and end product properties; Additional alternative precursors & chemistries; Melt PAN precursor (BASF technology) has good potential and could be opportunity for U.S. based precursor development.

> **We thank the reviewers for great support to this work. We are currently evaluating current state of the art precursor spinning and stretching technologies. Studies in spin finish will likely be accomplished through CRADA where precursor manufacturer will have ability to tailor fiber spin finish. Melt-processible PAN technology is under evaluation through FCTO. Once the technology becomes mature and scalable CFTF plans to process those materials.**

Should consider ROI analyses for various R&D tasks to help prioritization.

> **CFTF is currently spending the allocated $$ for operation and process development R&D. Additional resources needed to accommodate various R&D tasks. ROI analysis is being done for precursor in-house modification task.**

Should look for additional sources of funding & in-kind support (other gov't agencies, additional industry collaborators, other) due to importance of LCCF technology.

> **The Leadership team along with individual researchers are currently working on this mission. Very likely commercialization effort of the current IP will make a case for R&D investment.**

Commercialization – Comments

Good aggressive schedule, approach, and results. Determination to select limited number of semi-exclusive licenses appears to be best alternative. We are eager to see announcements of licensees in the near future and the plans for these commercial developments. Need to be sure that technology is controlled for U.S. production and U.S. economic advantage. Need to develop strategies & plans for continuation patents.

> **We thank the reviewers for this comment. We are working on continuation patent application.**

Technical collaboration projects

Company	Industry Type	Application	Status	Material Type	Tow
Arkema Inc	End-User	Thermoplastic evaluation sizing for SuperTruck/Volvo/ Caterpillar	Shipped	Carbon Fiber	457K
Asahi Kasei Plastics North America, Inc	End-User	Compounding and testing	Shipped	Carbon Fiber	457K 533K
Auburn University	Academia/R&D	Academic R&D	Shipped	Carbon Fiber	365K 343K 457K 611K
AvCarb Material Solutions	End-User	Evaluate replacement raw material source; preserve US manufacturing jobs.	Shipped	Oxidized PAN	457K 533K 611K
BST Nano Carbon	End-User	Package evaluation sample	Shipped	Carbon Fiber	457K
Chomarat North America	End-User	Fabric evaluation, USCAR project, IACMI 9M wind blade project	Shipped	Carbon Fiber	457K
Chomarat Textiles Industries (France)	End-User	Consortium automotive project	Shipped	Carbon Fiber	457K 533K
Composite Fabrics of America (CFA)	End-User	Large tow weaving evaluation	Shipped	Carbon Fiber	457K
Continental Structural Plastics	End-User	Evaluation of fiber for automotive application	Shipped	Carbon Fiber	365K
Cygnet Group	End-User	Prepreg tape evaluation	Shipped	Carbon Fiber	457K
Cytec	Producer	Sample	Shipped	Carbon Fiber	533K
Faurecia Automotive Composites (France)	End-User	RTM evaluation in automotive and resin trial	Shipped	Carbon Fiber	457K
Vanderbilt University	Academia/R&D	Sample for resonance testing	Shipped	Carbon Fibers	533K 611K
Zoltek	Producer	Wind and auto	Pending approval	Carbon Fiber	363K
Meggitt Aircraft Braking Systems	End-User	Non-braking static application	Pending approval	Oxidized PAN & Carbon Fiber	>300K
Asahi Kasei Plastics North America, Inc	End-User	Large structural applications	Pending approval	Carbon Fiber	457K
Collaborative Composite Solutions	End-User	Automotive application evaluation	Pending approval	Carbon Fiber	Split 457K
The University of Tennessee/IACMI	Academia/R&D	Carbon fiber laminates evaluation	Pending approval	Carbon Fiber	>300K

- **Over 50 collaborated requests/projects**
  - **Academia**
  - **Industry**
  - **Other National Labs**
- **Early feedback from several projects indicate good results**
  - **Final results will be published**

Remaining Challenges and Barriers: Proposed Future Works

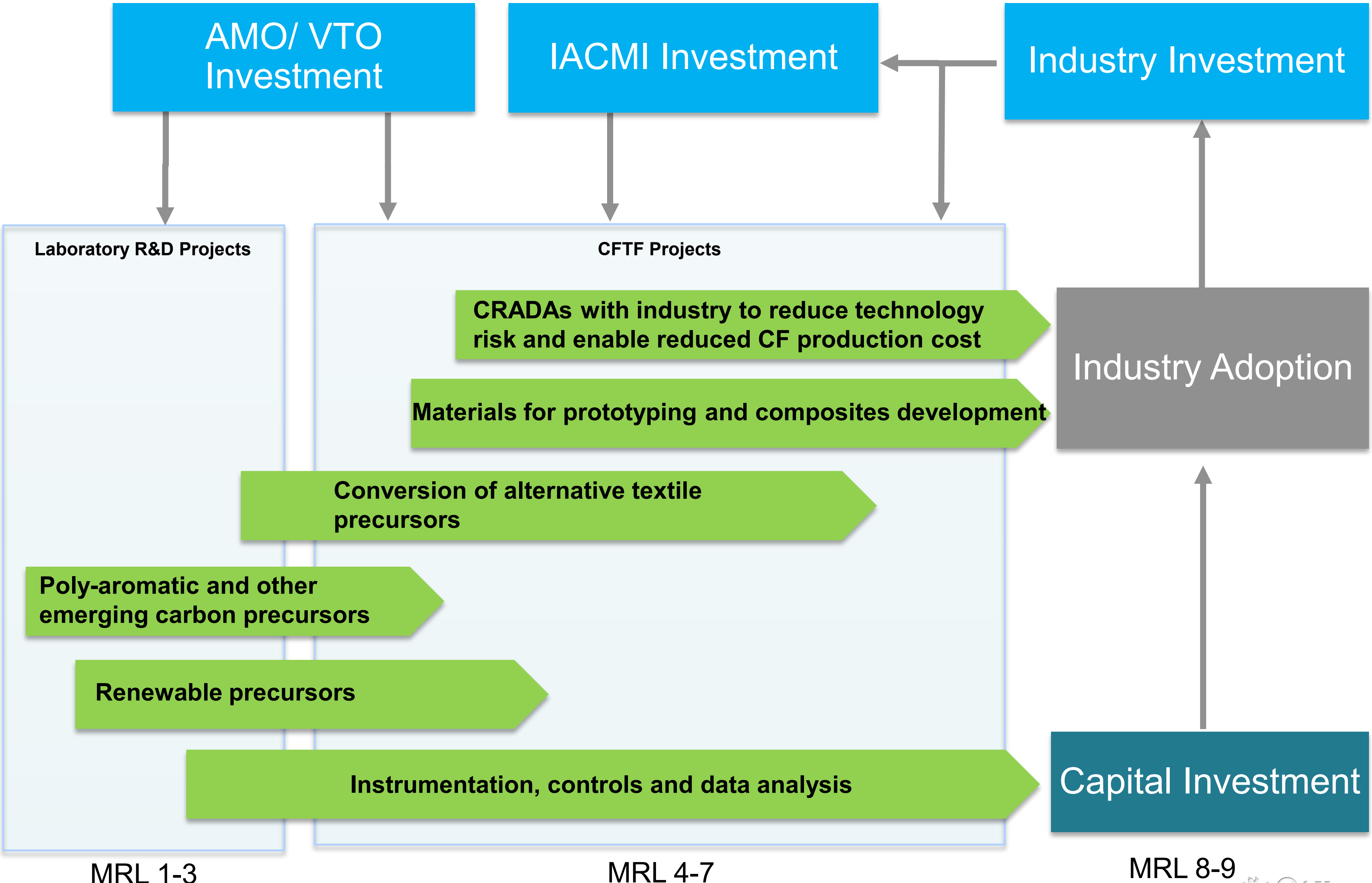
Number	Task Description
1	Establish CRADAs with industry partners to reduce technical risk and further develop technology to produce LCCF from large tow textile precursor.
2	Provide LCCF for prototyping and composites development. ORNL staff will collaborate closely with composites manufacturers using LCCF to understand results and gain feedback to inform further development.
3	Develop process conditions to allow conversion of additional sources of textile acrylic fiber to carbon fiber with properties suitable for use in vehicle applications.
4	Develop process monitoring and data analysis methods to improve process control and accelerate process optimization.

Proposed Future Work

- Textile PAN precursors: Correlating fundamental science to CF process and to the carbon fiber properties. CFTF is continuously focusing on stability and control of the process conditions for the various precursors. In addition to the current inventory, CFTF will develop process conditions for more varieties of textile precursor to target a wider range of applications enhancing engagement with industries and successful commercialization.
- Other Emerging Precursors: Polyaromatic hydrocarbon precursors can be processed at CFTF. Can we adopt high throughput conversion strategy for these groups of precursors?
- Data analysis and process control in carbon fiber manufacturing

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

Proposed Future Work Plan and Investment Strategy



Summary

Relevance: The Carbon Fiber Technology Facility is relevant in proving the technology for scaled-up conversion of low-cost carbon fiber precursor materials and advanced manufacturing technologies. The project is enabling development of domestic production technologies for low-cost carbon fiber.

Approach: Identification and development of high potential, low-cost alternative precursor that can be successfully converted to carbon fibers and commercialized by industrial partners.

Collaborations: Established several technical collaboration projects with industries and academic entities to help create market pull for low-cost, industrial grade carbon fiber.

Technical Accomplishments:

- Demonstrated a full year of operations with zero accidents or environmental non-compliances
- The facility has proven the capability to convert multiple sources of PAN-based textile precursors and developed conversion recipes to optimize properties of carbon fibers
- Acquired 5 interested parties for technology licensing
- Training and development of skilled workforce:
    - Six technology interns are now employed by a licensee
    - Three technology interns are now employed by UT-Battelle

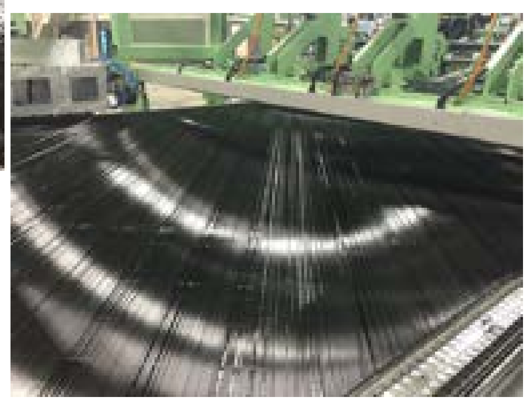
Future Work: Correlating fundamental science to CF process and to the carbon fiber properties. CFTF is continuously focusing on stability and control of the process conditions for the various precursors. In addition to the current inventory, CFTF will develop process conditions for more varieties of textile precursor to target a wider range of applications enhancing engagement with industries and successful commercialization.

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

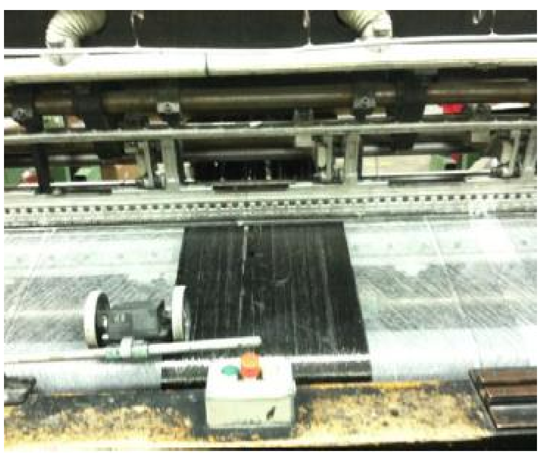
Discussion

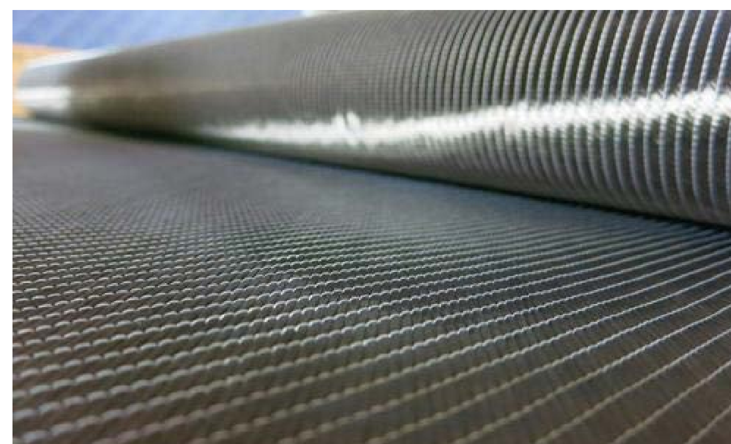

CARBON FIBER
TECHNOLOGY FACILITY
OAK RIDGE NATIONAL LABORATORY
93 PALLADIUM WAY

Fabric/Sheet Fabrication



Chomarat C-PLY non-crimped carbon fiber sheet





Vectorply VectorUltra carbon fiber sheet

Presentations and Major Visits

- Presentations were made throughout the year in order to make industry, academia and other federal agencies aware of CFTF as a national resource
  - Oak Ridge Carbon Fiber Composites Consortium meetings(December 2013 and March 2014)
  - EPRI Advanced Manufacturing Workshop (November 2013)
  - Vehicle Technologies Materials Technology Team (February 2014)
  - DOE Carbon Fiber Technology Facility Peer Review Team (August 2016)
  - U.S. DOE Advanced Manufacturing Office Program Review Meeting (June 2016)
  - IACMI Peer Review (August 2016)
  - Carbon Nexus Carbon Fibre Future Conference (February 2017)
- Numerous industry visits hosted including senior executives from:
  - DOE Office of Fossil Energy
  - Toyota Research Institute
  - Dow Chemical
  - SGL Recycling
  - Advanced Functional Fabrics of America
  - TPI Composites
  - Tesla Factory
  - Lockheed Martin Space Systems
  - Polynt Composites
  - Proper polymers
  - Continental Structural Plastics
  - National Spinning Company
  - Sila Nanotechnologies
  - Harper Internal and Formosa Plastics
  - GKN Aerospace
  - Asahi Kasei Plastics

University of Tennessee Summary of Comparison with Commercial Chopped Carbon Fiber

Resin System	Polypropylene (30% CF)		Polycarbonate (30% CF)		Nylon 66 (30% CF)		Ultem (30% CF)	
Carbon Fiber	**Commercial**	**ORNL**	**Commercial**	**ORNL**	**Commercial**	**ORNL**	**Commercial**	**ORNL**
Impact (ftlb/in)	1.1	0.95	2.74	2.72	2.89	2.41	2.5	1.69
Tensile strength (ksi)	5.94	6.09	19.08	21.99	27.07	25.67	24.88	23.69
Elongation at break (%)	3.56	2.80	2.26	2.36	2.39	2.59	1.46	1.06
Tensile Modulus (msi)	2.37	2.26	2.04	2.59	2.90	2.52	2.01	2.41
Flex Strength (ksi)	10.92	11.62	32.18	31.40	43.52	38.71	35.91	34.34
Flex Modulus (msi)	1.22	1.26	1.74	1.48	1.91	1.63	1.91	2.26

CFTF funding for operations

	FY 2012	FY 2013	FY 2014	FY 2015	FY 2016	FY 2017
Annual operating cost ($MM)	2.1 (1)	5.6	6.63	5.39	5.22	6.5
Carryover		2.3	1.3	0.17	0.28	0.36
VTO commitment	1.0	1.0	1.5	1.5	1.3	1.0
AMO commitment	2.0	2.0	4.0	4.0	4.0	4.0
Other commitment	1.4 (2)	1.6 (3)				1.0 (4)
Surplus/(Shortfall)		1.3	0.17	0.28	0.36	(0.14)

(1) CFTF expense for operations, starting in mid-2012.

(2) $1.4M ARRA contingency released for operations expense December 2011

(3) $1.6M ARRA contingency released for operation expense April 2013.

(4) $1.0M projected IACMI project spending

Carbon Fiber Technology Facility (CFTF) Development

U.S. DEPARTMENT OF ENERGY | Energy Efficiency & Renewable Energy

2011

- $35 million DOE investment under ARRA
- 42,000 sf facility with 390-ft. long processing line. Flexible unit operation configuration
- 25 tons/yr of fiber from multiple precursors in various forms

2012

- Facility occupancy
- Equipment installation complete
- Start-up testing and commissioning

2013

- Facility fully operational **ahead of schedule** and **under budget**

Background

Relevance: Prediction for overall carbon fiber demand and supply through 2020

Carbon fiber demand and supply, metric tonnes (MT)			
	Carbon fiber demand	Carbon fiber supply (nameplate)	Carbon fiber supply (actual)*
2010	48,370	79,650	47,790
2015	82,400	143,595	93,171
2020 (est.)	150,200	180,600	129,965

**Actual output is less than nameplate, due to capacity knockdown.*

Total carbon fiber demand will exceed actual supply. Additional expansion from carbon fiber suppliers is expected.

Source: Composites Forecasts and Consulting LLC
Supply and demand: Advanced fibers (2016), CompositesWorld, article published on 3/17/2016 (http://www.compositesworld.com/articles/supply-and-demand-advanced-fibers-2016) (accessed 4/10/17)

IACMI Summary of Results

- **Useful data for stakeholders for LCCF vs benchmark composites**
- **Beginning of the benchmark comparative testing**
- **Database to include thermoset & thermoplastic composites**
- **Reinforcements – continuous and discontinuous**
- **Processing methods representative of end applications**

Preliminary

Property (Cross-ply)	LCCF (53% W_f) Epoxy	Zoltek (60% W_f) Epoxy
Tensile strength MPa (ksi)	550 (79.77)	700 (101.52)
Tensile modulus GPa (Msi)	60 (8.70)	58 (8.41)
Flexural strength Mpa (ksi)	600 (87.02)	750 (108.77)
Flexural Modulus Gpa (Msi)	50 (7.25)	48 (6.96)
ILSS, Mpa (ksi)	TBD	48 (6.96)

Background: Commercialization objectives

- **Facilitate the establishment of a low cost carbon fiber industrial base in the United States**
  - Select partners who are able and committed to bring the ORNL process to the market
    - Create jobs and economic opportunity in the United States
      - Provide a return on DOE's investment in this technology