Low-Cost, High-Throughput Carbon Fiber with Large Diameter

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Project ID # mat203

Overview

Timeline

- October 1, 2021
- September 30, 2024
- 5%

Budget

- Total project funding $1,500K
  - Although not required, one of the partners had indicated plans to provide ~$150K in in-kind support)
- Funding for FY 2020 NA
- Funding for FY 2021 $500K

Barriers and Technical Targets

- Barriers addressed*
  - Cost of Carbon Fiber feedstock and production
  - Availability of Lower Cost Carbon Fiber at level necessary for large-scale impact in vehicle production

* *Light-Duty Vehicles Technical Requirements and Gaps for Lightweight and Propulsion Materials Workshop Report,* February 2013.

Partners

- Interactions/collaborations
  - 4XT/4M - Advanced Oxidation Co-Developer with ORNL
  - Dralon Gmb, Textile Precusor
- Project lead
  - ORNL

Relevance

Required Impact

- Because industrial carbon fiber costs reside ~50% in precursor and ~50% in conversion, maximizing cost reduction requires addressing both

How to Get There

- To produce carbon fiber at significantly lower costs, the project team will demonstrate the effects of combining
  1) Textile PAN fibers,
  2) Produced via dry spinning,
  3) At substantially larger diameters, and
  4) Oxidized with Atmospheric Plasma Oxidation – *probably the single most critical element to project success*
- This approach will exceed automotive requirements while enhancing processing characteristics and reduce cost >30%
- Work culminates in application demonstration in Year 3 to assess overall technical and economic advantages

FY 21 Milestones

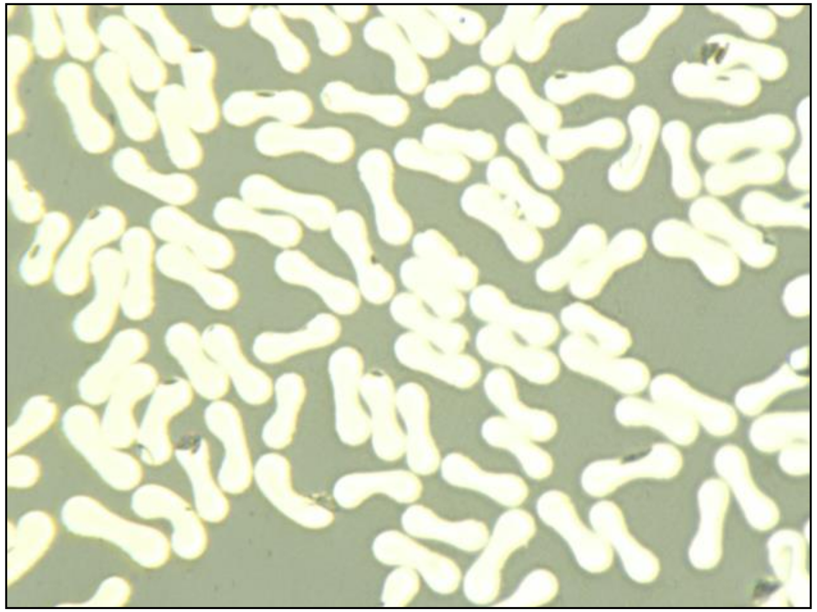
Milestone ID	Milestones description	Due date	Status
M1	**M1:1** Project management plan issued to team members and sponsors detailing task activities and interactions along with key target delivery schedules. Commercial industrial fiber baseline chosen and textile fiber baseline for this project delivered..	12/31/2020	**Projected completion 05/25/2021**
M2	**M1.2** Initial precursor for baseline converted and characterized. Initial precursor for fiber targeted at 25% greater in effective diameter delivered	03/31/2021	**Projected completion 06/15/2021**
M3	**M1.3** Conversion and characterization completed for the larger diameter precursor delivered in Q2.	06/30/2021	**Projected completion 08/15/2021**
M4	**M1.4** Preliminary cost estimation for baseline and larger diameter fiber completed.	09/30/2021	**Projected completion 09/30/2021**
Go/No-Go	**Go/No-Go 1.0** Cost/performance comparison of industrial fiber, project-produced baseline fiber, and first larger diameter fiber compared in order to project potential cost savings in this approach exceed 20% and are likely to meet project objectives.	9/30/2021	**Anticipate being back on schedule**

FY 22 Milestones

Milestone ID	Milestones description	Due date	Status
M5	M2.1 Second generation larger effective diameter precursor produced and delivered to project team for conversion and testing.	12/31/2021	
M6	M2.2 Second generation precursor is converted and tested. Post-treatment (surface treatment and sizing) approaches are down-selected for adaptation at CFTF.	03/31/2022	
M7	M 2.3 Composite panel demo article manufacturing and testing approach are finalized and appropriate precursor is produced.	06/30/2022	
M8	M2.4 Precursor for Year 3 composite evaluation is delivered and CFTF initiates conversion and post treatment activities.	09/30/2022	

Project Approach

- Project Year 1
  - Establish baseline for this approach with carbon fiber converted from the dry spun textile precursor
  - Demonstrate fiber at least 25% larger in "effective" diameter
  - Update ORNL cost models for baseline and 25% larger fiber to compare with commercial industrial fiber



Dralon Dry-Spun Fiber is Typically Dog-Bone Shaped

Go/no-go decision point. Baseline fiber must

- Meet performance of DOE minimum requirements (250 ksi strength, 25 Msi modulus, and 1% strain-to-failure)
- along with projected cost savings of 20% versus modeled commercial fiber.

Project Approach (cont)

- Project Year 2
  - Produce fiber effectively at least 50% larger than project baseline
  - Demonstrate performance minimums of 350 ksi, 33Msi, and 1% strain,
  - Developing and scale fiber post treatment processes to CFTF level production.

We anticipate steam stretching will boost performance

  - Cost target for Year 2 is to demonstrate 25-30% or greater savings are potentially achievable with this approach
  - Select 1) project optimal diameter targets for scaled-up production at CFTF and 2) composite demonstration approach (composite panel evaluation and/or demo part fabrication in order to demonstrate overall advantages).

Project Approach (cont)

- **Project Year 3**
  - **Demonstrate and evaluate attractiveness of the new carbon fiber produced at Pilot Scale.**
  - **Make and test the composite articles required to fully evaluate and define the advantages of this approach.**
  - **Cost models will be completed and utilized to evaluate the cost versus performance benefits in combining effects**
    - **Textile PAN fibers**
    - **Produced via dry spinning, and**
    - **At substantially larger diameters, and**
    - **Oxidized with Atmospheric Plasma Oxidation**

Technical Accomplishments and Progress

Existing Dry Spun Precursor in Filament Sizes of 1.7, 2.2, 3.3 and 5.5 dtex is Being "Repurposed" for this Project

- Expanding on our preliminary work, ORNL has been working extensively with Dralon to finalize specifications and develop delivery schedules for the dry spun precursor and with 4XT to update experimental approaches for converting precursor.
- COVID shutdowns and associated business impacts have delayed planned precursor production at Dralon and significantly slowed progress during our first year
- Recently, partner 4XT has agreed to provide existing stock of similar Dralon precursor they had obtained for other business purposes to initiate previously planned experimental work
- In evaluating recent work utilizing the APO system, we concluded that relatively minor equipment upgrades would enhance the system robustness and consistency

Technical Accomplishments and Progress (cont)

- System enhancements and upgrades in progress include:
  - Flow controllers, flow meters, and gas sensors were calibrated, repaired, or replaced.
  - Rollers that were scratched or damaged were repolished. This will decrease damage to the fibers.
  - Insulation that was damaged or missing was replaced.
  - The compressed air feed gas system was upgraded to include a larger dryer and receiver tank to decrease the variation in water content and line pressure. Moisture has a strong impact on the plasma oxidation process.

- At the time of this submission, detailed plans are being compiled to proceed with the experimental work plans for the first 1-1/2 to 2 years utilizing the existing precursor while continuing to evaluate alternate precursor supply approaches

Collaboration and Coordination with Other Institutions

- ORNL Carbon and Composites Group
  - Technical leadership, project management, composite demonstration
- Dralon GmbH
  - Production of dry spun precursor at various larger diameters
- 4X Technologies, LLC (4XT)
  - Advanced oxidation development and demonstration
  - To be commercialized by 4M Carbon Fiber Corp. (4M)
- ORNL Carbon Fiber Technology Facility
  - Scaled up carbonization and post treatment for demonstration
- ORNL Vehicle Systems Research Group
  - Techno-economic modeling

Barriers and Challenges

- Each of the primary approaches has been addressed on a limited basis separately, but need to be integrated and more fully validated both technical and economically in composites
- Other potential advantages have been identified, but not have yet to be demonstrated in even composite coupons include, but are not limited to:
  - Larger fibers enhance wet-ability in composites production
  - Larger fibers enhance interfacial properties
  - Larger fibers might enhance fiber buckling resistance leading to higher compressive strength in composites
- Advantages in producing these new fibers and making composite coupons need to be demonstrated in potential applications.

Future Research

- Utilizing combination of lower cost dry spun fibers and advanced conversion technologies
- Assessing broader range of diameter versus economics and carbon fiber production/performance tradeoffs than previously explored
- Evaluation of projected resin infusion advantages and possibly improved interfacial properties
- Work culminates in application demonstration in Year 3 to assess overall technical and economic advantages

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

Summary

- Our approach offers high potential for significant cost savings by combining and expanding approaches to lower precursor cost AND advanced conversion technologies leading to higher throughput, lower cost carbon fiber production.
- Team will also seek confirmation of projected advantages of enhanced resin infusion and possibly improved interfacial properties which can further improve cost-effectiveness of this approach
- Progress during the first half of the first year has been slowed due to Covid issues impact on participating organizations
- Work-arounds have been developed and are being implemented while evaluating longer-term options for even broader implementation

Technical Backup Slides

Opportunities for Cost Savings

- Precursor Costs
  - Typically assumed to account for 40-50% of industrial carbon fiber
  - Dry spun PAN is estimated to save 4-15%, regardless of diameter
  - Larger tow sizes potentially save even more over traditional precursor
- Conversion and Post Treatment Costs
  - Oxidation typically assumed to account for 25-35% of fiber costs
  - Carbonization typically assumed to account for 15-25% of fiber costs
  - Other conversion costs estimated at 5-15% of fiber costs
  - At 2X-4X throughput improvement, it is possible costs could be reduced 30-60% for each of the above steps
- Composite Manufacturing
  - Larger diameters could reduce handling costs and potentially improve performance

Background and Preliminary Work

- Commercial carbon fiber today uses conventional approaches for making very round wet spun PAN precursor and converting in traditional ovens and furnaces

- Project team has demonstrated significant advantages for:
  - Textile fiber PAN precursors in non-round shapes
  - Dry spun precursors to reduce solvent issues
  - Advanced Plasma Oxidation

APO Production of large diameter OPF	Density	OPF Diameter	Oxidation Time
	g/cc	micron	min
Typical Conventional	1.36	11	90
Plasma Oxidation	1.36	11	20
	1.36	16	50
	1.36	20	84*

  - Carbon fiber diameters outside of traditional ~5-7 microns

Larger Diameters Also Open Other Doors

- Compressive performance of CF composites is generally thought to be deficient
  - Small diameter – small buckling resistance via Euler buckling theory

$$F_b = \frac{\pi^2 \, E \, I}{(K \, L)^2} \qquad\qquad I = \frac{\pi}{64} \boldsymbol{D^4}$$

  - Small diameter – resin infusion and interfacial property deficiencies
- Although there are design “workarounds”, larger diameters are expected to improve
  - Handling in general
  - Wet-out in intermediate and final production processes, and
  - Significantly enhance structural attractiveness in compressive-critical applications such as wind turbine blades