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Development of a Lower Cost, High Volume, Commercially Available, Precursor for Lower Cost Carbon Fiber for Automotive and Wind Blade Applications

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Development of a Lower Cost, High Volume, Commercially Available, Precursor for Lower Cost Carbon Fiber for Automotive and Wind Blade Applications

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1. LISTS

1.1 List of Acronyms

DMAC – common precursor production solvent Dimethylacetamide.
DMF – common precursor production solvent Dimethylformamide.
DMSO - common precursor production solvent Dimethyl sulfoxide
PAN - Polyacrylonitrile, chemical name for precursor formulation
DSC – Differential Scanning Calorimetry thermal characterization method
DMA – Dynamic Mechanical Analysis
Ksi – unit of tensile strength in thousands of pounds per square inch
Msi – unit of tensile modulus in millions of pounds per square inch
CF - Carbon Fiber

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1.5 Acknowledgements

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2. EXECUTIVE SUMMARY

Essentially all carbon fibers used in structural applications are presently manufactured from solution spun polyacrylonitrile (PAN) precursor. Solution spinning is accomplished at relatively low temperatures using very large amounts of environmentally unfriendly solvents that impart major impact to line speed and costs involved with handling and reclaiming the solvents and associated "waste streams" required in spinning and extracting those solvents. Dralon and others have demonstrated the dry spinning of PAN in the production of some acrylic fibers for textile applications, but to this point it has not been demonstrated in the production of carbon fibers. In dry spinning, the PAN polymer is also dissolved into similar solvents, although typically less solvent is utilized to maintain a somewhat higher solids content of 20-50%. The use of lower overall solvent quantities and elimination of the need for solvent/water separation simplifies and reduces the cost of solvent recovery operations. Other portions of the precursor production and carbon fiber manufacturing processes are quite similar.

The work described in this report demonstrated that dry spun acrylic fiber can be an attractive candidate for carbon fiber precursor of potential interest to applications looking to capture the specific stiffness (stiffness per unit mass) and specific strength (strength per unit mass) of carbon fiber when produced at a discount relative to commercial carbon fibers in the market today. Dralon has determined that the dry spinning process utilized for producing fibers used as precursors in this project requires 27% less energy versus producing comparable fibers via the industry standard wet spinning process. Identification of carbon fiber manufacturers, as well as applications development and targeted marketing, will be required to achieve significant in-roads into mission areas of interest to DOE, such as automotive applications and wind turbine blades. In support of that activity, initial data produced in this project indicate the innovative approach as described in this report can meet at least the baseline property requirements identified for these applications.

3. INTRODUCTION

This Technical Collaboration Project is focused on the development of a high-volume precursor, which is less expensive due to its production in high volume textile mills. It is intended that this precursor will also be available on the open world market for any Carbon Fiber (CF) manufacturer to purchase and convert into CF. Most carbon fiber manufacturers make their proprietary precursors specifically for their own production needs; these CF precursors are currently not available for other US manufacturers that may wish to enter the market. A high-volume acrylic spinning method, "dry spinning," has been commercialized by Dralon for textile applications and has the potential to provide CF precursors at costs less than typical "wet spinning" technology. The dry spinning technology was explored in this work using conventional conversion approaches, including oxidative stabilization in the air between 200 and 400°C and subsequent carbonization in an inert atmosphere between 1000 and 1700°C as described by Peebles[1]. The resulting lower cost fiber is targeted to be applicable to wind energy and automotive applications as well as other industrial applications that are stiffness (modulus) driven. Additional conversion cost savings could be derived from incorporation of previously developed advanced oxidation technologies utilizing plasma-based techniques to shorten time requirements that are now being commercialized[2]. In future work outside the scope of this project, the advanced oxidation method may be evaluated for applicability to this precursor, resulting in further cost reduction.

This work focused on achieving adequate tensile modulus and reduced cost in the production of carbon

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[1] Peebles, LF, Carbon Fibers Formation, Structure, and Properties, CRC Press, 1995.

[2] "4M Reveals Progress with Plasma Oxidation for CF Production", Composites World, March 2020.

fiber, both of which automotive manufacturers have emphasized as critical needs. The primary avenue for cost savings is the utilization of the “dry spinning” approach in the production of acrylic fiber. In this process, the fibers are vertically spun, just as with air gap spinning. However, rather than the fibers undergoing repeated washing cycles, they are subjected to a heated environment under a moderate vacuum. In dry spinning, the combination of elevated temperature and the slight vacuum allows for evaporation of the solvent as opposed to the repeated washing to remove the solvent in wet spinning or its closely related variant commonly referred to as air gap spinning. (Primary difference in true wet spinning and air gap spinning is that in air gap spinning, the fibers are extruded into a very small open gap before being submerged into the coagulation bath.) It is hypothesized that dry spinning ultimately facilitates the production of more homogenous, less porous fibers than wet spinning. Current dry spun fibers have a more rectangular or dog-boned shape as described later, but with modifications in spinneret design and spinning conditions, it is possible that they could be more round in shape if that proves to be more desirable. Establishing baseline precursor chemistries and spinning conditions for precursor production as close as possible to approaches and equipment already in use for producing textile acrylics was a key portion of early project activities.

Primary carbon fiber demonstration goals were to: (1) characterize the potential of a new source of commercially available precursor; (2) evaluate value for lower cost spinning due to higher throughput potential; and (3) exploit utilization of lower solvent processes to produce adequate fiber while minimizing solvent recycling costs. While not explicit goals of this project, it is believed that experience gained in this work will establish background useful in future work for potentially tailoring fiber cross-sectional geometries and surface roughness via spinneret design and process parameters adjustments. As a result, the potential exists long-term to make not only less expensive precursor fibers but also possibly "better" precursor fibers. Additional cost savings can come from two additional sources: (1) Conventional oxidation may occur more rapidly because the new fiber shape (inherently more rectangular as opposed to circular) will have a shorter maximum oxygen diffusion pathway from the fiber surface to interior resulting in more rapid oxygen diffusion; and (2) the application of the advanced oxidation technology to this fiber in future efforts.

4. BACKGROUND

Essentially all carbon fibers used in structural applications are presently manufactured from solution spun polyacrylonitrile (PAN) precursor. Solution spinning is accomplished at relatively low temperatures using very large amounts of environmentally unfriendly solvents that impart major impact to line speed and costs involved with handling and reclaiming the solvents and associated “waste streams” required in spinning and extracting those solvents. The PAN polymer is dissolved in a solvent such as DMAC, DMF, DMSO, etc., at a solids content of about 15-25% and extruded through spinnerets into an aqueous bath with a large fraction of solvent (50-80%) for coagulation of the filament. After coagulation, the fibers are drawn in a series of washing baths with successively less solvent present in order to orient the fiber and extract the solvent for recovery. The precursor fiber production typically contributes a large fraction (about 50%) to the finished carbon fiber cost. Precursor purity, elongated molecular orientation, and chemical and physical consistency are very important for achieving high fiber strength. Even though PAN polymer is a thermoplastic, probably the number one feature of PAN that makes it attractive for carbon fiber is that it can be cross-linked in the air at relatively low temperatures, which (1) facilitates development and management of fiber orientation, and (2) stabilizes the fiber for subsequent pyrolysis for removal of the non-carbon elements. The principal drawback of this feature is that the low temperature cross-linking makes direct melt spinning of pure or nearly pure PAN impossible.

Dry spinning of PAN as depicted in Figure 1 has been demonstrated by Dralon (believed to be the world's only large-scale producer of dry spun acrylic fiber) and others in the production of some acrylic fibers for textile applications, but to this point has not been demonstrated in the production of carbon fibers. In dry spinning, the PAN polymer is also dissolved into similar solvents, although typically less solvent is utilized in order to maintain a somewhat higher solids content of ~30%. However, in dry spinning, the fibers are extruded through spinnerets into a hot air environment as opposed to a liquid coagulant bath. As the fibers coagulate and solidify, the solvent is evaporated from the fiber more rapidly and cost-effectively in a drying tower as opposed to being extracted in a series of baths. The use of lower overall solvent quantities and elimination of the need for solvent/water separation simplifies and reduces the cost of solvent recovery operations. Other portions of the precursor production and carbon fiber manufacturing processes are quite similar, and specific nuances are beyond the intent of this background discussion. The objective of this work was to demonstrate that adequate carbon fiber can be produced from this lower cost precursor without negating the advantages inherent in the production of the precursor and to characterize potential cost/performance tradeoffs for this approach.

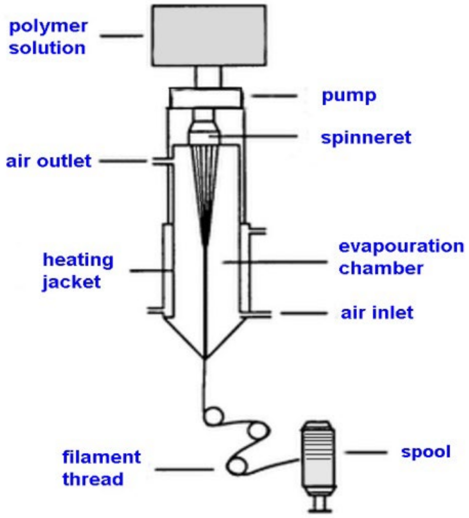

Figure 1 Dry spinning of PAN filaments

During the course of the work, trials were run with both crimped fiber, as is normally sold for textile applications, and fiber from a new setup implemented by Dralon to facilitate removal and spooling of uncrimped fiber. (Fiber crimping is utilized in textile processing to impart desirable textures such as a waviness characteristic to improve handling in some cases and in others to provide a "feel" more similar to that of a natural fiber like cotton than to an engineered fiber like polyester or acrylic.) Another key investigation conducted during this project evaluated the effects of steam stretching prior to conversion versus fibers that went directly into oxidation. Steam stretching provides more orientation to the fiber at lower temperatures than the stretching during oxidation. The steam helps to plasticize the fiber while orienting it prior to the cross-linking effects that occur at the higher temperatures of oxidation. At the conclusion of this project, Dralon provided samples of the preferred precursor approach to ORNL's Carbon Fiber Technology Facility (CFTF) in order to run a larger-scale and continuous run as a demonstration of the advantages of this overall approach.

Key results of this work are summarized in the table below.

RESULTS AND DISCUSSION

A number of batches of dry-spun precursor fibers were produced with varying conditions resulting in differing properties for this project by Dralon. Specifics of how Dralon modified spinning conditions and surface finishes to produce fibers that are able to be handled during conversion and that will not fuse during oxidation or carbonization remain proprietary to Dralon. Based on feedback from ORNL on processability and properties of the resulting carbon fiber, Dralon worked to optimize the spinning conditions. As part of this optimization, Dralon identified and implemented modifications to the industrial pilot line toward improvement in the filament generation process, especially in winding and packaging of the precursor tow. Probably the single most important variable evaluated was the level of crimp. During the course of the project, Dralon supplied samples with "standard" crimp levels, lightly crimped levels, and samples that were uncrimped. These improvements are part of the development path towards adapting the traditional textile acrylic approach to the slightly different approach required to produce an acceptable PAN precursor that can yield a CF with sufficient mechanical properties and at acceptable costs.

The precursor fiber batches supplied by Dralon were evaluated by ORNL to develop the fundamental approach and recipes necessary for the conversion of the precursor into carbon fiber. These pre-conversion evaluations included: (1) Physical Characterization to assess and ensure consistency in fiber diameter, fiber shape, surface texture, and as received fiber density; (2) Mechanical Characterization such as tensile testing and determination of the amount of pre-stretching of the fiber that can be achieved; and (3) Thermal Characterization such as DSC, DMA, etc. to establish key temperature transition regions. The thermal characterization provides information on how much stretch can be applied to the fibers during various stages of oxidation, as well as help pinpoint the temperatures for each stage of oxidation. The data sets a starting point for conversion conditions based upon similarities to precursors with previously determined conversion protocols. A previously developed ORNL proprietary approach was implemented utilizing DSC analysis to mimic the temperature exposure for each stage of the oxidation process and obtain a reasonable starting point for conversion trials.

Based on the results from these early characterization trials, the project team iteratively optimized the conversion process for each batch of the precursor supplied by Dralon in the ORNL Precursor Evaluation System (PES) depicted in Figure 2. Although the focus of this work was to demonstrate the capability to meet previously identified property targets for automotive and wind applications of 400-600ksi tensile strength and stiffness levels of 30-33Msi, the team also evaluated the potential to cost-effectively exceed those levels as well. As is well-known in carbon fiber conversion, key conversion parameters are time, tension, and temperature profiles during each conversion stage. ORNL considers the "standard" conversion approach to consist of potentially some pre-stretching, but the primary approach definitely entails four stages of oxidation and two stages of carbonization. Although parameter details for industrial grades of carbon fiber are export controlled and therefore not included in this report, oxidation is typically conducted in air at varying tensions and successively higher temperatures for each stage between 200-300C while low and high temperature carbonization are also conducted under varying tension but in an inert atmosphere between 500-1500C. These must be done sequentially, completing each processing step before proceeding to the next. Only after completing all these steps can the final properties of the fiber be determined.

Table 1 demonstrates typical data from samples of Dralon X fiber slightly modified by Dralon based on early project results and feedback. In this case, the fiber was uncrimped versus earlier samples which were either crimped utilizing their standard process or only lightly crimped. These samples were not stretched as described below. The samples were processed using a variety of conversion parameters, which, as noted earlier, cannot be provided due to the data being export controlled. The ultimate failure stress ranged from 298ksi to 433ksi, with moduli ranging from 28.3Msi to 30.9Msi and strain-to-failure ranging from 1.00% to 1.43%. As these samples were being processed and tested, ORNL was implementing the custom designed steam stretching system as depicted in Figure 3 below.

Figure 2 ORNL Precursor Evaluation System

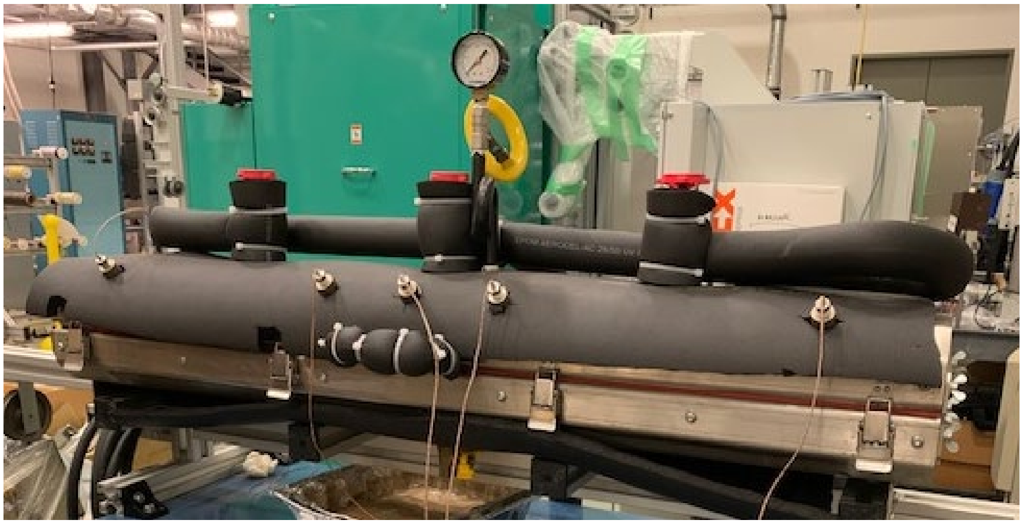

Figure 3 ORNL Steam Stretcher

Table 1 Dralon X Fiber, Uncrimped, Unstretched

Favimat #	Sample	Diameter µm	Ult Stress Ksi	Modulus Msi	Strain %
1316	Dralon Xu run 7	5.70 (0.56)	340.15 (35.86)	29.49 (0.80)	1.13 (0.11)
1317	Dralon Xu run 7	5.89 (0.41)	334.67 (47.13)	28.83 (0.67)	1.14 (0.15)
1318	Dralon Xu run 7	5.73 (0.46)	373.31 (63.27)	28.48 (0.71)	1.28 (0.21)
1327	Dralon Xu run 8	5.94 (0.29)	318.73 (60.21)	28.73 (0.54)	1.09 (0.19)
1328	Dralon Xu run 8	5.79 (0.29)	353.96 (94.53)	29.23 (0.97)	1.19 (0.30)
1334	Dralon Xu run 9	5.98 (0.28)	330.93 (67.27)	30.31 (1.04)	1.08 (0.21)
1335	Dralon Xu run 9	5.89 (0.52)	359.29 (48.18)	29.93 (0.77)	1.18 (0.15)
1336	Dralon Xu run 9	5.58 (0.33)	297.74 (76.29)	29.51 (1.03)	1.00 (0.23)
1421	Dralon Xu run 10	5.92 (0.43)	361.49 (85.97)	28.97 (1.33)	1.22 (0.26)
1422	Dralon Xu run 10	5.77 (0.36)	432.90 (42.77)	29.35 (0.60)	1.43 (0.13)
1534	Dralon Xu run 11	5.47 (0.42)	354.00 (113.90)	30.89 (0.29)	1.06 (0.40)
1535	Dralon Xu run 11	5.89 (0.54)	365.80 (87.59)	29.73 (0.84)	1.21 (0.26)
1561	Dralon Xu run 12	5.85 (0.44)	390.63 (95.91)	29.00 (0.67)	1.32 (0.31)
1567	Dralon Xu run 12	5.82 (0.69)	364.07 (63.22)	28.32 (1.33)	1.25 (0.20)
1568	Dralon Xu run 12	6.21 (0.45)	394.55 (96.09)	27.30 (1.91)	1.39 (0.28)

Figure 4 shows typical data for steam stretching as an independent step upon receipt of the precursor and prior to conversion. Note that one would normally maintain initial stretching well below the point of maximum stress such that there is no danger of breaking filaments in early oxidation where substantial stretching is applied while elevating temperature to achieve cross-linking of the precursor polymer for subsequent conversion steps.

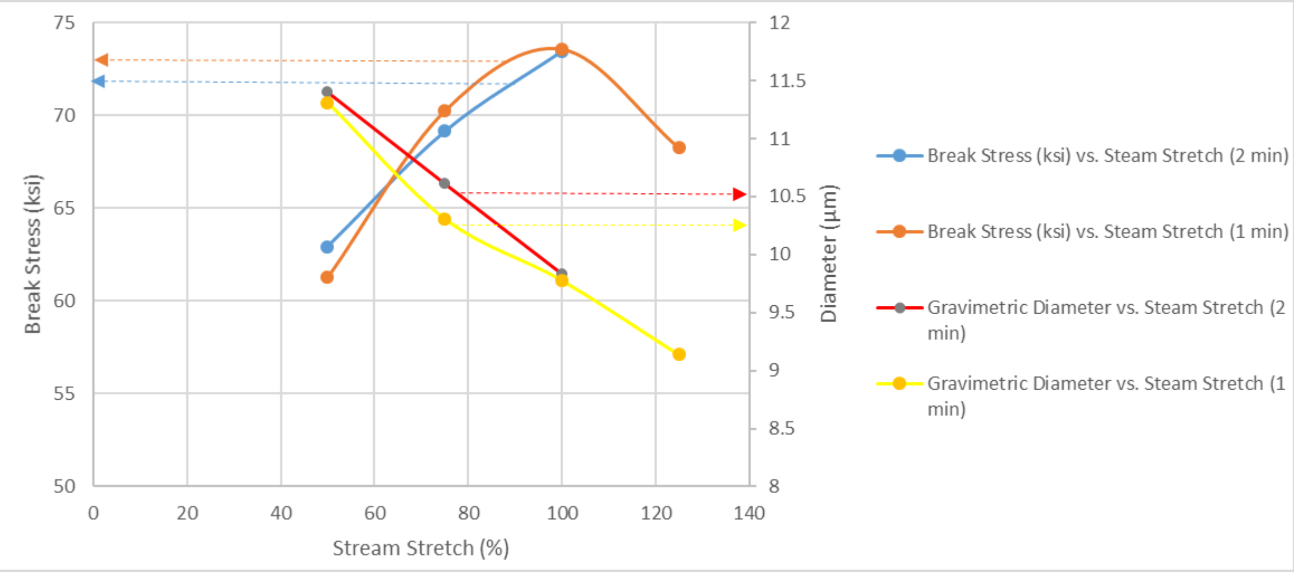

Figure 4 Breaking stress and filament diameter as a function of steam stress levels

Table 2 shows results from converting and testing similar Dralon X uncrimped fiber that has been steam stretched. Again, the samples were processed using a variety of conversion parameters to establish property range capabilities. For this group of samples, the ultimate failure stress ranged from 381ksi to 468ksi, with moduli ranging from 29.8Msi to 31.8Msi and strain-to-failure ranging from 1.23% to 1.46%.

Overall, this data demonstrates that the best results were achieved with uncrimped fiber in conjunction with steam stretching and represents a significant progression of property improvements over the course of the project. Most importantly, the data demonstrates that the Dralon precursor is capable of repeatably achieving project goals of 400-600ksi tensile strength and 30Msi or higher modulus.

Table 2 Dralon X Fiber, Uncrimped, Stretched

Favimat #	Sample	Break Stress Ksi	Diameter µm	Modulus Msi	Strain %
1656	DralonXuSS65R5Ox	34.69 (2.31)	9.61 (0.64)	1.20 (0.04)	20.14 (2.67)
1667	DralonXuSS65R5 RT1	404.68 (37.00)	5.24 (0.21)	31.31 (1.08)	1.26 (0.09)
1669	DralonXuSS65R5 RT1	430.78 (73.11)	5.23 (0.47)	31.80 (0.78)	1.31 (0.20)
1672	DralonXuSS65R5 RT1	381.20 (44.22)	5.41 (0.72)	30.39 (0.65)	1.23 (0.14)
1674	DralonXuSS65R5 RT1	441.19 (77.16)	5.14 (0.46)	29.81 (1.14)	1.42 (0.21)
1675	DralonXuSS65R5 RT1	468.41 (50.35)	5.08 (0.36)	30.91 (0.63)	1.46 (0.15)
1677	DralonXuSS65R5 RT1	425.86 (76.06)	5.27 (0.34)	30.24 (1.38)	1.36 (0.21)
1678	DralonXuSS65R5 RT1	438.42 (61.94)	5.11 (0.32)	31.45 (0.97)	1.35 (0.17)
1686	DralonXuSS65R5 RT1	411.66 (64.81)	5.30 (0.47)	31.77 (0.72)	1.26 (0.19)
1689	DralonXuSS65R5 RT2	425.66 (43.58)	5.38 (0.31)	30.71 (0.67)	1.35 (0.14)
1690	DralonXuSS65R5 RT2	438.52 (69.64)	5.55 (0.33)	31.61 (0.96)	1.35 (0.20)
1691	DralonXuSS65R5 RT2	420.09 (58.14)	5.45 (0.47)	30.52 (1.12)	1.34 (0.18)
1692	DralonXuSS65R5 RT2	456.50 (61.35)	5.23 (0.46)	30.20 (1.19)	1.46 (0.17)

Sample 1656 is precursor that has been oxidized only – balance of the samples have been fully converted.

Based on successful results of the collaborative development work described above, ORNL's Carbon Fiber Technology Facility indicated interest and resources available to run some larger-scale demonstration trials utilizing the precursor down-selected from this earlier work. Dralon provided a larger sample to CFTF and materials were run over a several-day time period utilizing conversion parameters initially based on the earlier work. Like the development trials, the samples were processed using various conversion parameters to establish property range capabilities, which as noted earlier cannot be provided due to the data being export controlled. A data summary is provided below in Table 3. The ultimate failure stress ranged from 377ksi to 437ksi, with moduli ranging from 32.6Msi to 34.4Msi and strain-to-failure ranging from 0.77% to 1.29%. This data is very comparable to data from the developmental trials reported earlier based on single filament testing, but it is important to note that mechanical property results in Table 3 are from impregnated strand testing based on ASTM D4018, which is typical of how carbon fiber manufacturers report data from production runs. This data also completes a key project milestone.

Table 3. Summary of the Best Tensile Property Data from Demonstration Trials at CFTF

Highest Average Tensile Properties:			
	Tensile Strength (Ksi)	Tensile Modulus (Msi)	Elongation (%)
	437.0	32.81	1.23
	429.5	33.43	1.09
	386.5	34.38	0.77
	415.5	33.11	1.16
	377.4	32.84	1.09
	431.3	32.72	1.23
	420.6	33.05	1.18
	416.6	33.01	1.16
	423.0	32.69	1.29
	382.5	32.58	1.10
Average	**412.0**	**33.06**	**1.13**
Stdev.	**21.7**	**0.53**	**0.14**
# of samples	**10**	**10**	**10**
Maximum	**437.0**	**34.38**	**1.29**
Minimum	**377.4**	**32.58**	**0.77**

5. BENEFITS ASSESSMENT

Dralon provided results from internal analysis showing a direct comparison of energy utilized in wet spinning versus dry spinning, as shown in Figures 5 and 6 below. Their analysis points to an energy savings of 27% for the dry spinning process versus producing comparable fibers via the industry standard wet spinning process. If one assumes that the facilities and equipment are fully depreciated, as would be the case for Dralon fiber production, and labor and materials cost are comparable, then overall precursor fiber cost production would be lower for the dry spun fiber. The oxidative stabilization process is dependent on oxygen diffusing from the surface of the fiber to the innermost acrylic molecules in that fiber. It is generally thought that the time required for this process is proportional to the longest pathway distance squared, which in the case of a round fiber would be proportional to the fiber radius squared. For a flat or dog-bone shaped fiber, this maximum distance for a fiber of the same mass would be much less than the radius of an equivalent round fiber. Although beyond the scope and not specifically evaluated as part of this project, the non-round shape of the dry spun Dralon fibers, as shown in Figure 7, would likely also provide energy and cost benefits in carbon fiber conversion.

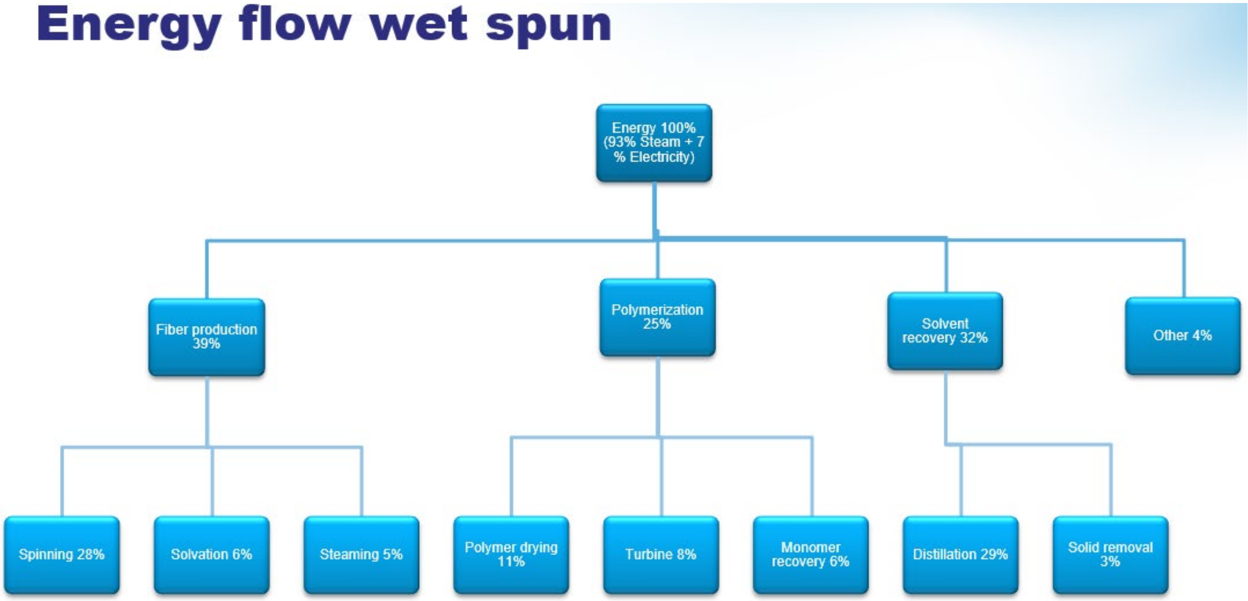

Figure 5 Diagram showing elements of energy contributions and values for wet spinning process

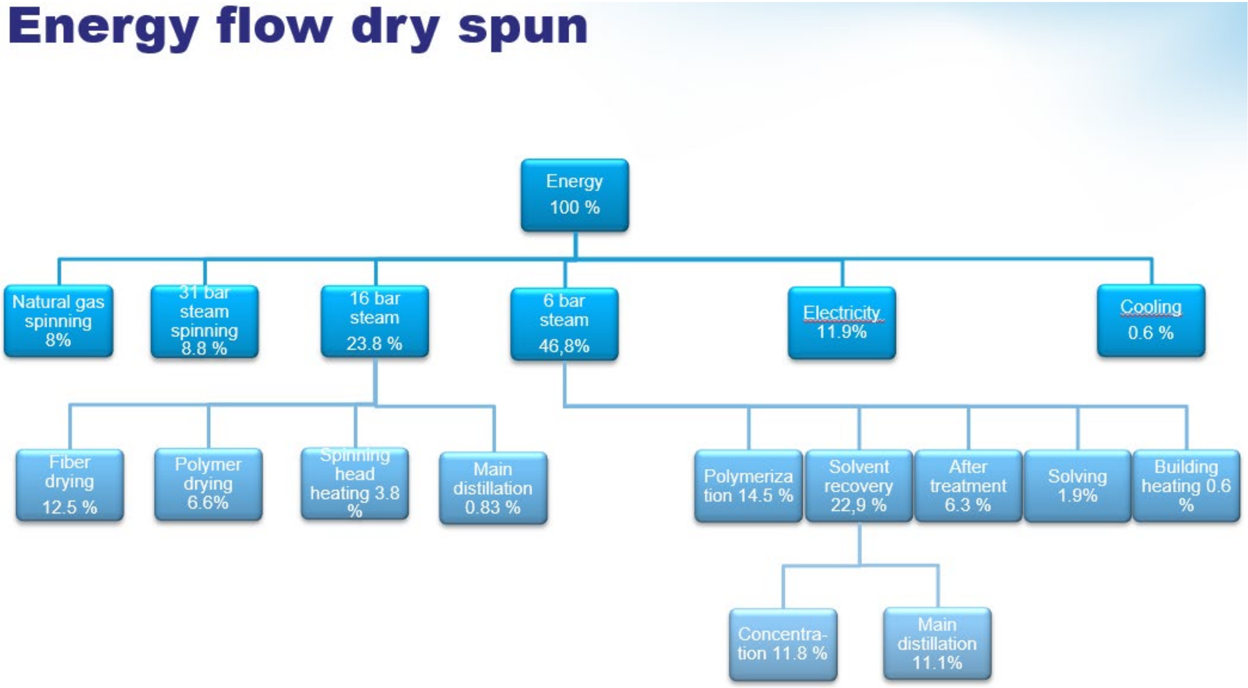

Figure 6 Diagram showing elements of energy contributions and values for dry spinning process

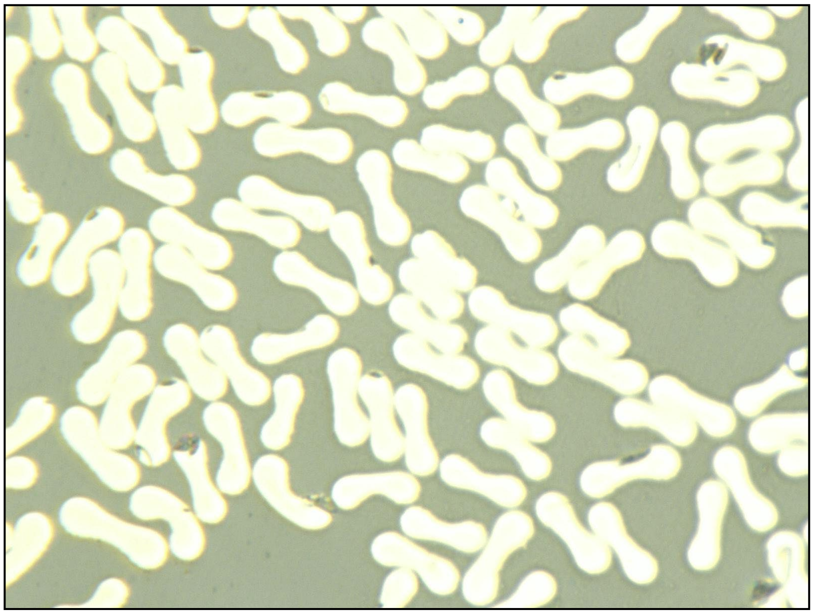

Figure 7 Cross-sectional view of "dog-bone-shaped" Dralon dry spun fibers

6. COMMERCIALIZATION

Now that this acrylic has been demonstrated in lab-scale and CFTF scale experiments to be capable of consistently providing carbon fiber tensile strengths exceeding 400ksi and modulus consistently above 30Msi, project team members are looking for opportunities to facilitate applications development in commercial arenas such as automotive and wind blade markets. While ORNL focuses on DOE program opportunities in those key energy-related areas in collaboration with Dralon, Dralon is developing marketing strategies for how-best to utilize this data in approaching potential carbon fiber manufacturers identified independently as well as through IACMI/ORNL relationships. By introducing a commercially available, lower cost, high performance CF precursor into the worldwide marketplace, multiple CF sources can be made available to manufacturers. The precursor developed in this project is being made available to the open market and thus will encourage new CF manufacturers to enter the market. This will increase both the availability and the price competition for the material.

7. ACCOMPLISHMENTS

This project resulted in the development and demonstration of "recipes" for making small modifications to the production process for dry spun acrylic fiber that can then be converted into carbon fiber with properties meeting levels identified as needed in DOE energy mission areas such as automotive and wind. Specifically, carbon fiber tensile strengths of >400ksi, modulus of >30Msi, and elongation-to-failure >1% were consistently achieved. Best data were achieved with uncrimped fiber that was steam stretched, followed by steam-stretched lightly crimped fiber. Steam stretching of fully crimped fiber caused fiber damage, making the lightly crimped and uncrimped fiber preferred for producing higher properties. Data from this work will be extremely valuable in approaching potential large-scale carbon fiber manufacturers and key decision makers in application development to facilitate strategic discussion on the pathway forward. At the time of this report, the team is only beginning to assess the best means for publicizing results and initiating more formal marketing approaches.

8. CONCLUSIONS

This project has conclusively demonstrated that dry spun acrylic fiber can be an attractive candidate for carbon fiber precursor of potential interest to applications looking to capture the specific stiffness (stiffness per unit mass) and specific strength (strength per unit mass) of carbon fiber when produced at a discount relative to commercial carbon fibers in the market today. Dralon has determined that the dry spinning process utilized for producing fibers used as a precursor in this project requires 27% less energy versus producing comparable fibers via the industry standard wet spinning process. Although identification of carbon fiber manufacturer(s) as well as applications development and targeted marketing will be required in order to achieve significant in-roads into mission areas of interest to DOE such as automotive and wind turbine blades, initial data produced in the project indicate this approach can meet at least entrance property requirements identified for these industries.

9. RECOMMENDATIONS

As per the discussion above, the groundwork has been provided to move into applications development and strategic discussions concerning business relationships for moving forward to commercialization. Additional work to characterize potential improvements in conversion time, energy, and cost believed to be possible due to the elongated shape and smaller oxygen diffusion pathways required due to fiber geometry would be helpful. As with introduction of most revolutionary approaches to producing carbon fiber, additional work is required for product form, filament size, and interfacial compatibility optimization. Based on preliminary work with other non-round shapes, it is recommended that the potential impact of this shape be evaluated for effects on compressive properties since this shape might do well depending on orientation distribution and achievable packing factor when taking into account that surface area for bonding will be larger than for round fibers.