# C. Low-Cost Carbon Fiber Manufacturing Using Microwave Energy

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## Objective

- Investigate and develop a microwave-assisted technical alternative to carbonize and graphitize polyacrylonitrile (PAN) based precursor.
- Prove that carbon fiber with properties suitable for use by the automotive industry can be produced inexpensively using microwave-assisted plasma (MAP) processing.
- Demonstrate that MAP processing can produce acceptably uniform properties over the length of the fiber tow.
- Show that for specified microwave input parameters, fibers with specific properties may be controllably and predictably manufactured using microwave furnaces.
- Demonstrate the economic feasibility for producing approximately 30-Msi modulus fibers at a significant cost reduction relative to those produced conventionally.

## Approach

- Demonstrate the ability to deliver high fiber mass throughput by increasing line speed and tow count.
- Conduct parametric studies on the continuous carbon-fiber processing pilot unit to continually improve the system design, process parameters, and fiber properties.
- Characterize MAP-processed carbon fibers to confirm that they satisfy program requirements.
- Continually evaluate, develop, and characterize “spin-off” technology, hardware, and ideas that improve upstream or downstream processing, or facilitate more efficient utilization of fiber.

## Accomplishments

- Carbonized 50 K PAN fiber tows successfully at three-tow line speeds up to 13 in./min.
- Determined that improved generator matching and longer plasma length are needed to increase line speed beyond 13 in./min; designed and procured the necessary hardware.
- Investigated the effects of varying fiber tension during carbonization.
- Commenced surface treatment studies in which initial results suggest that MAP-carbonized fibers may accept a higher level of surface treatment than conventionally carbonized fibers.
- Co-authored two papers for 2004 SAMPE Symposium and Exposition, and the principal investigator was the lead author on the 2004 SAMPE Symposium's Outstanding Technical Paper.

## Future Direction

- Increase 3-tow line speed to 40 in./min.
- Continue parametric studies and fiber characterization to better understand process effects and processing window and to quantify fiber properties.
- Achieve integration with other carbon-fiber technology developments, for example, new precursors, rapid oxidation/stabilization processes, advanced surface treatment, advanced downstream formatting, and/or component manufacturing processes.
- Develop and demonstrate related technologies in the area of carbon-fiber manufacturing (e.g., advanced characterization, surface treatment, sensing and control technology, recovery and reuse) as resources and time permit.
- Develop partnership(s) to commercialize the technology.

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## Introduction

The purpose of this project is to investigate and develop a microwave-assisted technical alternative to carbonize and partially graphitize the polyacrylonitrile (PAN) precursor. The project is to prove that carbon fiber with properties suitable for use by the automotive industry can be produced inexpensively using microwave-assisted plasma (MAP) processing. It is to be demonstrated that MAP processing can produce acceptably uniform properties over the length of the fiber tow. The project is also to show that for specified microwave input parameters, fibers with specific properties may be controllably and predictably manufactured using microwave furnaces. Lastly, but most importantly, this project is to demonstrate the economic feasibility for producing approximately 30-Msi modulus fibers at a significant cost reduction below those produced conventionally.

## Project Deliverables

At the end of this project, a continuous, multiple-tow, scalable, high-production line speed MAP carbon fiber prototype unit will have been developed, constructed, and tested. A final report will be issued with the test results of the carbon fibers processed with this unit. Appropriate industry briefings will be conducted to facilitate commercialization of this economically enabling technology.

## Fiber Properties

A key goal of this project is to demonstrate that carbon fibers with satisfactory material properties can be produced by the MAP process. Program goals established for fiber properties are 25-Mpsi tensile modulus, 250-ksi ultimate tensile strength, and 1.0% ultimate strain. Mechanical properties are shown in Figures 1–3. Mechanical property results generally satisfied the specified requirements, except

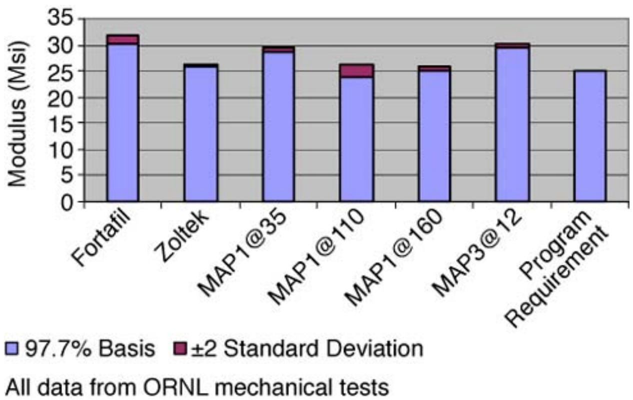


**Figure 1.** Carbon fiber tensile modulus.

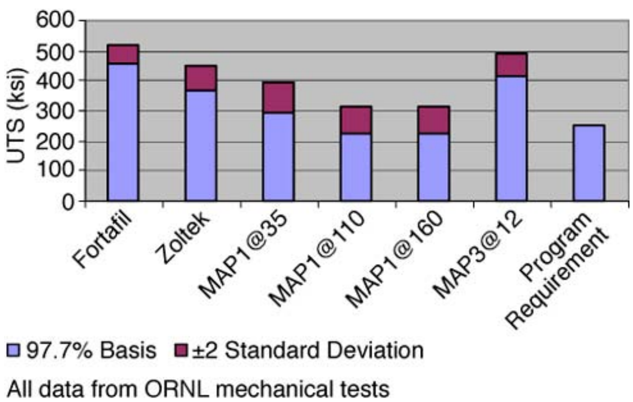


**Figure 2.** Carbon fiber ultimate tensile strength.

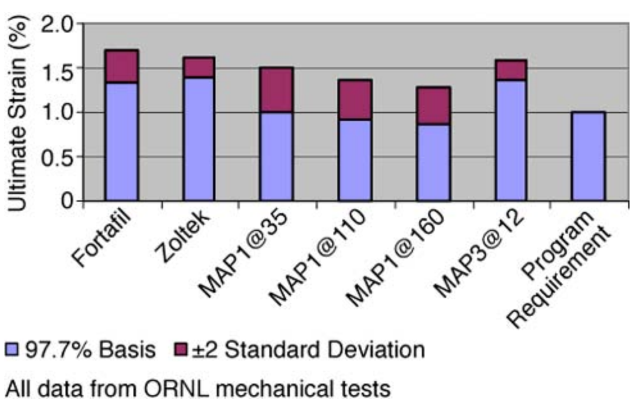


**Figure 3.** Carbon fiber ultimate tensile strain.

that the ultimate tensile strength was slightly deficient at the highest single-tow line speeds. This is attributed to the fiber transport and tensioning system that was not designed for high line speeds. To resolve this problem, a commercial, high-speed, fiber transport and tensioning system was installed during the upgrade to three-tow capacity.

In Figures 1–3, 97.7% basis means that 97.7% of property data should exceed the indicated value. The 97.7% basis value is the mean less two standard deviations. Labels indicate fiber processing, number of tows, and line speed. For example, MAP3@12 indicates three tows MAP processed at 12 in./min.

## Throughput

The economics "figure of merit" is cost per unit throughput. Increasing throughput usually reduces unit cost. Hence the MAP process must be operable at production line speeds and "bandwidth" (number of tows) comparable to or exceeding those of conventional fiber manufacturing lines if it is to realize the desired cost effect. Conventional large-tow carbon fiber manufacturing lines normally run at line speeds in the 90- to 120-in./min range, with about 100 tows per line (a typical line is rated at nominally 1M lb/year when processing 50 K tows).

The researchers have previously demonstrated single-tow operation at line speeds to 200 in./min. The present focus is to achieve high line speed with a three-tow line configuration. Maximum three-tow speed achieved to date was 13 in./min. The limiting factors appear to be generator matching and plasma length. Higher power will be required to run at higher speeds, but raising the power has produced arcing in the waveguide system. Improved generator matching should resolve this problem. Also, the current configuration produces a compact plasma (short length), which results in a severe processing gradient. A simple applicator modification was tried, but it lengthened the plasma by only a small amount.

A major applicator modification is under way, based on scalable mathematical and physical simulations that correlated satisfactorily. The applicator was completely redesigned, and the design was verified by simulation on the subscale physical model shown in Figure 4. Upon completion, the modified applicator should lengthen the plasma processing region by ~6X. New hardware was fabricated and delivered during the last half of FY 2004 and is now ready for assembly.

## Parametric Studies

The fiber properties are affected by many processing parameters such as temperature, pressure, power, plasma density, line speed, and tow tension.

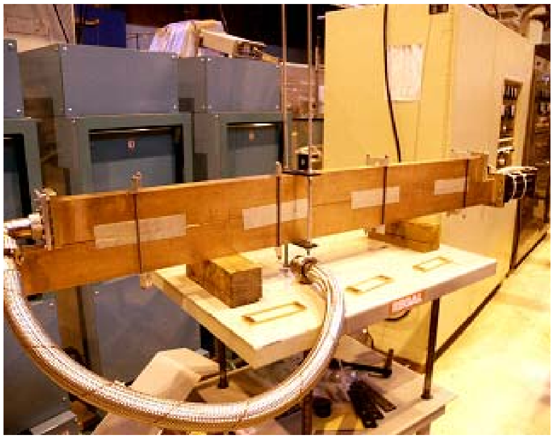

**Figure 4.** Subscale applicator simulation hardware.

During FY 2004, the effects of tow tension were investigated. The results are not published for patent protection and export control purposes.

## Surface Treatment

MAP carbonized fibers were ozone surface treated by the same process as conventionally carbonized fibers. The MAP carbonized fibers consistently exhibited 75%–80% higher concentration of oxygen on the surface than did conventionally carbonized, commercial-grade fibers.

## Economics

During FY 2004, Kline and Company completed a cost study of the Department of Energy's (DOE's) entire carbon fiber development program, estimating profitable selling prices for baseline technology and determining if various technologies under development were implemented. Kline's estimates generally indicated that MAP carbonization and graphitization should reduce the fiber selling price by about 10%. These estimates, however, were based on a line speed comparable to that of conventional conversion lines. Rough calculations suggest that if we can achieve much higher line speeds, the savings could increase dramatically. We have not discovered any inherent physics limits that would prevent us from achieving a large increase in conversion line speed using plasma oxidation and MAP carbonization.

## Education

The materials characterization, notably utilizing scanning electron microscopy (SEM) and spectroscopy methods such as x-ray photoelectron spectroscopy (XPS), has been conducted in partnership with the University of Tennessee's Materials Science Department. A doctoral candidate provided characterization support to the project. This work forms the basis of her doctoral thesis.

## Conclusions

The development of MAP carbonization and graphitization technology has progressed well. Fiber properties have generally satisfied or exceeded targets, and the process knowledge database continues to grow. The three-tow line has been operated at 13 in./min, with modifications underway that should enable operation at 40 in./min or higher line speed.

## Presentations and Publications

1. F. L. Paulauskas and T. L. White, "Temperature-Dependent Dielectric Measurements of Polyacrylonitrile Fibers During Air Oxidation," 49th International SAMPE Symposium 2004, May 16–20, 2004, Long Beach, CA (2004).