D. 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

- Completed relocation of the carbon-fiber conversion laboratory to ORNL main campus.
- Achieved what we believe to be record spatial resolution in measuring spatial distribution of surface functional groups.
- Correlated surface topography and spatial distribution of surface chemistry.

Future Direction

- Re-assemble and commission the MAP carbonization module at ORNL main campus location.
- Increase multiple-tow line speed.
- Continue parametric studies and fiber characterization to better understand process effects and processing window and to quantify fiber properties.
- Integrate into advanced technology, subscale carbon-fiber pilot conversion line (future project).
- Develop partnership(s) to commercialize the technology.

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.

Facility Relocation

In FY 2005 the carbon fiber conversion laboratory and associated equipment were moved from the Y-12 site to the ORNL main campus. Most of the equipment has now been re-assembled at the new facility. Completion of reassembly, installation, and checkout is scheduled in early FY 2006.

Characterization

Carbon-fiber surface chemistry greatly affects bonding to the resin, and hence the composite material properties. MAP carbonization has been shown to produce different, and apparently superior, surface chemistry than conventional carbonization. While spatially-averaged surface chemistry is useful information, it is extremely desirable to understand the spatial variation of surface chemistry and how it correlates with surface topography.

PAN-based carbon fibers are typically covered by ridges that run approximately parallel to the fiber axis and are somewhat regularly spaced. Typical ridge height and width both tend to be on the order of 100 nm, so any technique for correlating surface topography and surface chemistry must include the ability to locate surface functional groups with about 10 nm spatial resolution. Furthermore, depth-of-field needs to be in the nanometer range so as to focus on the top atomic layers.

Auger Electron Spectroscopy (AES) satisfies spatial and depth-of-field resolution requirements. By using chemical derivitization techniques, AES can identify elemental composition and indirectly determine the chemical states present on the sample surface. ORNL's High Temperature Materials Laboratory employed this technique to produce the images shown in Figure 1. AES and secondary images were both obtained in the same instrument (PHI 680, Physical Electronics, Cahnhassen, MN). In the AES images, high carboxyl concentration is indicated by dark (blue for readers who are viewing color images) areas. The images clearly show the high carboxyl concentrations imparted by plasma treatment, as well as the correlation between microstructural features and the distribution of acid groups. The researchers are unaware of previous measurements of surface chemical spatial distribution at this resolution. It is observed that most carboxyl groups were distributed along the bottoms or sides of valley regions. Functional groups are believed to attach to the edges of graphene planes, so it appears that, compared to ridges, there are more graphite edge sites located along the valley. This is surprising, as the conventional view has been that functional groups attach to the ridge tops. The orientation of graphene planes with regard to the fiber surface in both ridges and valleys, and the graphitic organization in both areas, i.e., any distortion present, need to be further investigated.

Patents and Publications

An invention disclosure was submitted to ORNL's patent office on the design details of the MAP system. The patent will be filed in early FY 2006.

Education

The materials characterization, notably utilizing Auger microanalysis and 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

Relocation of the carbon-fiber conversion lab commenced during the first half of FY 2005.

The researchers developed a method to accurately determine the spatial distribution of surface functional groups and their correlation with topographical surface features. A noteworthy achievement was that, to our knowledge, the spatial resolution of these measurements was the best ever achieved in measuring functional group locations.

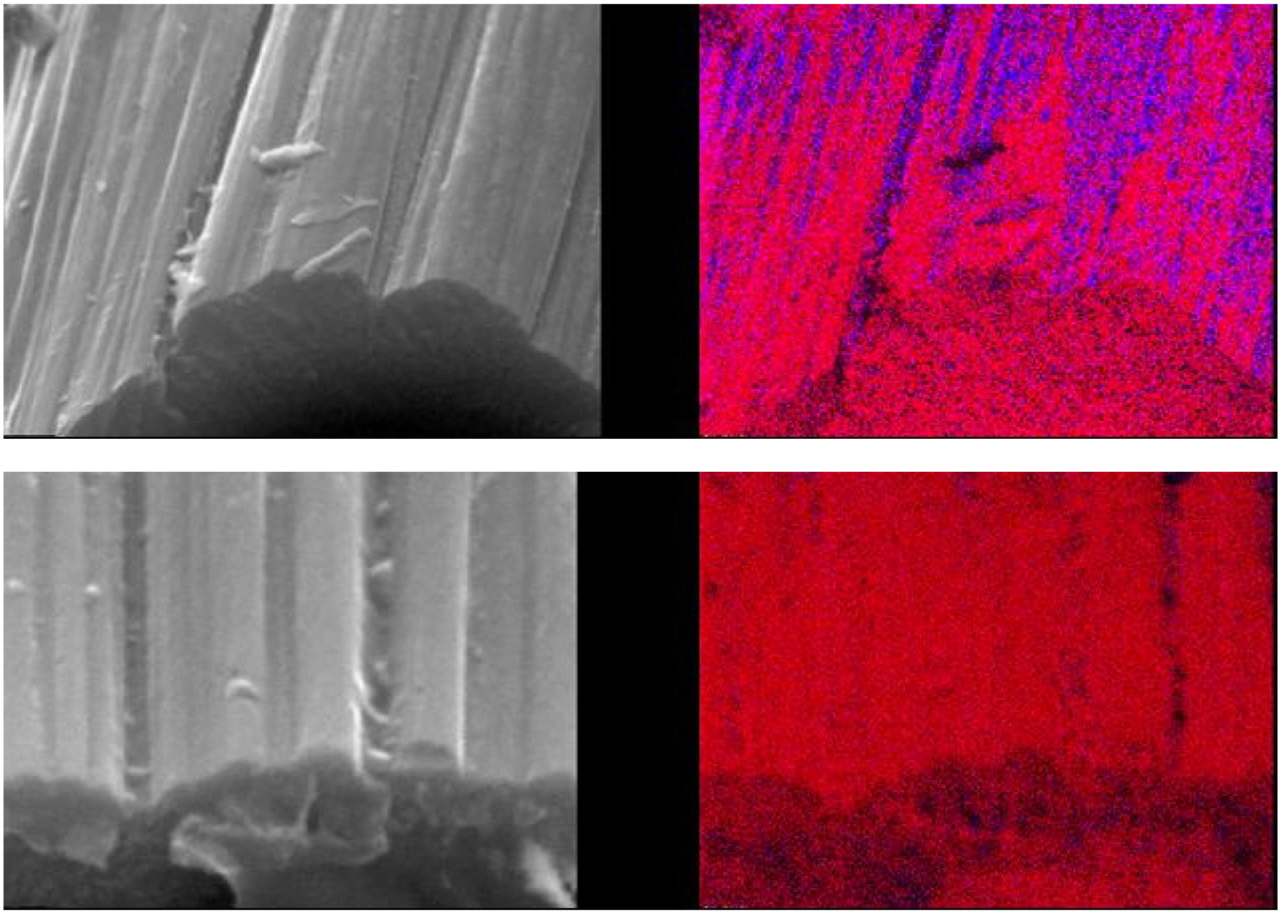

Figure 1. Images of carbon fiber. Top: untreated, unsized commercial fiber. Bottom: Plasma-treated fiber. Left: scanning electron micrograph. Right: Auger electron spectrograph, in which dark (blue, for readers viewing color images) areas indicate high carboxyl concentrations.