B. Advanced Stabilization of PAN Fiber Precursor

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Contractor: Oak Ridge National Laboratory
Contract No.: DE-AC05-00OR22725

Objectives

- Develop an improved technique for stabilizing carbon-fiber precursor with increased line speed and reduced carbon-fiber cost.
- Verify that finished fiber properties satisfy automotive and heavy-vehicle manufacturers' requirements.
- Conduct a preliminary evaluation of the cost impact of the new stabilization technique.
- Integrate the stabilization module into an advanced technology pilot-line.

Approach

- Investigate thermochemical, ultraviolet, and electron-beam processing stabilization routes.
- Select one of the aforementioned stabilization routes for detailed equipment and process development.
- Develop fiber-handling protocols for continuous processing.
- Conduct parametric studies to correlate processing parameters and fiber properties.
- Characterize fibers to confirm that they satisfy program requirements.

Accomplishments

- Developed project plan including prospective stabilization routes and appropriate partnerships.
- Initiated subcontract negotiations with Clemson University for ultraviolet processing and Atmospheric Glow Technologies for thermochemical processing routes.

- Met with Ion Beam Applications and commenced planning for electron-beam processing experiments.

Future Direction

- Develop an inexpensive stabilization technique that complements advanced oxidation and carbonization/graphitization processes.
- Conduct parametric studies and fiber characterization to better understand process effects and the processing window and to quantify fiber properties.
- Install advanced stabilization module in advanced-technology pilot-line.

Introduction

The purpose of this project is to investigate and develop a technique to rapidly and inexpensively stabilize a polyacrylonitrile (PAN) precursor. New processing techniques are being developed for the purpose of reducing the cost of carbon-fiber conversion. Previous and ongoing research at ORNL has demonstrated that plasma processing shows great promise for inexpensively and rapidly oxidizing, carbonizing, and graphitizing polymer precursors to convert them to carbon fibers. However, to date, all plasma processing protocols tried by ORNL have damaged virgin precursor, i.e., the precursor needs to be lightly stabilized, or cross-linked, before it is exposed to plasma. A rapid, inexpensive, and robust stabilization technique is needed to complement the aforementioned advanced process modules, and enable the development of an integrated, advanced-technology conversion line that converts polymer precursor fibers into carbon fibers at significantly lower cost than conventional conversion technology.

This project, therefore, intends to develop an advanced stabilization module that integrates with other advanced fiber processing modules to produce inexpensive carbon fiber with properties suitable for use by the automotive industry. Critical technical criteria include (1) $\geq$ 25 Msi tensile modulus and $\geq$ 1.0% ultimate strain in the finished fiber; (2) uniform properties over the length of the fiber tow; (3) repeatable and controllable processing; (4) and significant unit cost reduction compared with conventional processing.

Project Deliverable

At the end of this project, we will have demonstrated satisfactory PAN-precursor-fiber stabilization in a multiple-tow stabilization module operating at line speed exceeding that typical of conventional carbon-fiber conversion lines. The project deliverable is an equipment and processing specification from which advanced stabilization equipment can be purchased and operated in a subscale, advanced-technology, pilot conversion line.

Technical Approach

The researchers are investigating three prospective PAN-precursor-fiber stabilization routes: electron-beam processing, thermochemical processing, and ultraviolet processing. All three routes are based on discoveries previously made in other carbon-fiber projects, and each appears to offer certain advantages. After initial feasibility studies, the researchers will select the most promising route for detailed process development, with the principal criterion for selection being finished fiber cost. The preferred route will then undergo detailed parametric studies to develop the processing recipe, and will be scaled up in both speed and tow count to the point where equipment can be designed and purchased for operation in a subscale, pilot conversion line.

Progress

The project was initiated late in FY05, with sufficient time remaining for planning only. ORNL has engaged partners to provide equipment and assist with or lead the study of each processing route. The researchers have met with Ion Beam Applications, which will provide irradiation services at its Long Island facility, and experiment planning is underway for first electron-beam processing experiments in November 2005. Contract negotiations are ongoing with Atmospheric Glow Technologies, Inc. and Clemson University, the respective providers of thermochemical and ultraviolet processing equipment and services. First experiments for each of these routes are likely to also occur in the November

2005 time frame, assuming there are no unusual delays in contract execution.

Future Direction

FY 2006 will be devoted to parallel studies of the proposed stabilization routes, with down-selection to a single route in early FY 2007.

Education

Educational institutions participating in this project include Clemson University and UT. A Clemson post-doctoral researcher will conduct ultraviolet processing studies under the direction of Professor Amod Ogale. Two University of Tennessee (UT) materials science graduate students will provide characterization support to the project under the guidance of Professor Roberto Benson.

Partners

In addition to the aforementioned partners, ORNL gratefully acknowledges contributions to this project by TohoTenax America and Hexcel. Both have generously provided raw materials and offered technical consultation. Additionally, technical and programmatic consultation has been provided by the Automotive Composites Consortium.

Conclusions

Three prospective, nonthermal processing routes are proposed to lightly stabilize polymer precursors in an advanced carbon-fiber conversion line. Contract negotiations and experiment planning are underway with first experiments scheduled in November 2005.