

**Preconference Seminar 1 - December 9<sup>th</sup>, 2009 – 8AM**

**ABSTRACT**

**Carbon Fiber for Tomorrow –  
A Review of Current Technologies and the Path to Wide  
Spread Industrial Growth**

The elevated mechanical properties and low mass of carbon fiber make it an attractive potential material for a number of high volume applications. Industries with potential interest include automotive, wind energy, infrastructure, ground and sea based defense, bio-energy, oil and gas, bio-fuels and sporting goods. The high cost, low relative volumes available and inexperience of many industries with carbon fiber, present significant obstacles to incorporating this material in future structures. This short course will review how pitch, PAN and rayon based carbon fibers are currently used and common manufacturing methods for producing carbon fibers from these precursors. The course will then address individual market sectors that could see wide spread penetration by carbon fiber composites and highlight some of the current obstacles to widespread adaptation for those industries. The course will next give a breakdown of where and what the costs are for producing PAN based carbon fiber and highlight on-going efforts, to reduce these costs. It will also explore other non-cost obstacles to incorporating carbon fiber in high volume industries and what is being done globally to address those. The course will include:

- The History of Carbon Fiber and Who are the Players
- Production Methods for Converting Pitch, PAN and Rayon to Carbon Fiber
- Current Applications for Carbon Fiber
- Future Industries with Potential for Growth of Carbon Fiber Composites
- Entering the Business? PAN Carbon Fiber Production Costs
- Current Technical and Non-Technical Efforts to Reduce Costs
- Other Market Penetration Obstacles

# Carbon Fiber Overview

## Short Course Schedule and Topics

8:00 AM Introductions Scott Stephenson

8:05 AM Course Overview – (Dave Warren)

8:10 AM History of Carbon Fiber – Pitch, PAN & Rayon Mohamed Abdallah

This presentation gives an overview and an introduction to the carbon fiber technologies and the history of carbon fiber industry that includes the following: The history of Rayon-, Pitch-, and Polyacrylonitrile (PAN)-based carbon fiber development; Review of the major historical events that have had large impact on the carbon fiber industry restructuring and market growth; Current industrial technologies for the production of PAN-Based Carbon Fibers; Brief summary of current and potential applications of carbon Fiber composites; Summary of carbon fiber composites manufacturing technologies; Current major carbon producers and expected new producers; Current market status of the estimated production capacity, supply and demand; Expected future growth and the impact of the current global economical slowdown on the carbon fiber composites industry.

8:30 AM Pitch Precursor Spinning & Conversion Fred Baker

This presentation will give an overview of current methods and materials for spinning pitch based precursors and the technologies for converting those precursors into finished carbon fiber. Emphasis will be placed on understanding current technology limitations and advantages.

8:50 AM Pitch & Rayon Fiber Applications, Advantages, & Limitations Nidia Gallego

This presentation will give an overview of current applications for pitch-based carbon fibers and rayon based carbon fibers. The advantages and limitations of pitch-based fibers will be discussed based on the physical and mechanical properties of the fibers. Advantages and limitations of rayon based fibers will also be discussed.

9:05 AM PAN Precursor Spinning Amit Naskar

The presentation will review the existing technologies for obtaining polyacrylonitrile (PAN)-based carbon precursor fibers. The chemistry of precursors and its role on processing will be highlighted. The most common solution-based PAN precursor processing technology, wet spinning and its variants such as dry spinning, melt-assisted spinning, and gel spinning will be discussed. Melt-spinning of PAN copolymers as carbon fiber precursor, common precursor post-spinning operations will be presented. A structure-property relationship of precursors at different spinning conditions will be presented with reference to their effect on carbon fiber properties.

9:20 AM PAN Precursor Conversion Felix Paulauskas

The conversion process is an integral part of the overall carbon fiber manufacturing process. The conversion process, precursor pre-treatments, and carbonized fiber post-treatments are the three most relevant steps in carbon fiber manufacturing. The conversion of precursor fibers into finished carbon fibers requires a sequence of discrete heat treatment steps: stabilization and oxidation, carbonization (low and high temperature), and in some cases graphitization. Each of these thermal processing steps has a distinct role to develop required characteristics of the carbonized fiber. For example, the oxidation stage is the most time consuming and, therefore, rate limiting step in the conversion process. This oxidation step also sets the foundation for mechanical properties of the carbon fiber. If the oxidation step is performed incorrectly it is not possible to retrieve the desired mechanical properties of fiber in subsequent conversion steps. Only the proper thermal control of fibers during conversion is not enough to assure a good quality product. Many additional factors need to be considered to achieve specific quality of the product: precursor condition, pretreatment, fiber handling during each step of the conversion process, proper control of environment, tension control, etc. The precursor molecular structure is very important too. Depending on the precursor characteristics different product can be

achieved. Carbonization and graphitization are the steps that require very high temperature treatment and these steps help to develop required morphology and mechanical properties of produced carbon fiber.

9:40 AM Break

10:05 AM Carbon Fiber Recycling Mohamed Abdallah

This presentation provides an overview the technologies for recycling carbon fiber composite materials. In this presentation we address the following topics: (1)Sources of recyclable carbon fiber materials and structural components technologies for recycling carbon fiber from carbon fiber composite materials and products; (2)Proposed technologies and produces for using recyclable carbon fibers and manufacturing technologies; (3)Impact of government regulations and policies in reinforcing recycling implementations; and (4)Status of the development and implementation of carbon fiber recycling in the USA and Western Europe

10:15 AM PAN Based Fiber Applications, Advantages & Limitations Cliff Eberle

An overview will be given of current applications for PAN based carbon fiber and the advantages that PAN based carbon fiber offers relevant to other competing materials. The advantages and disadvantages of using PAN based carbon fiber will be explored along with the limitations that current fibers have.

10:30 AM Entering the Business? - PAN Carbon Fiber Production Costs Dave Warren

An overview of what it really costs to make PAN based carbon fibers will be given with breakdowns by both cost category and by steps in the production process. Areas of potential cost reduction will be identified. New carbon fiber producers who are employing some of these cost reduction techniques will be identified.

10:50 AM Novel & Unique Carbon Fibers and Fiber Forms Soydan Ozcan

As the price of carbon fiber decreases, there is excitement about the future possibilities for using them in a wide variety of applications such as light-weight fuel efficient vehicles, wind tribune blades, and high pressure storage vessels. The topic areas covered in the talk will include up-to-date information on the broad spectrum of fiber forms and carbon fibers. The talk will include a discussion on comparison of rayon, pitch, and polyacrylonitrile based carbon fibers with ceramic and glass fibers including advantages and disadvantages of the various fiber types specific to their applications. A discussion will also be held on properties by means of processing-microstructure-property relationships.

11:05 AM Current ORNL Development Efforts – 10 min each except last is 5 min

1. Lignin Based Precursors Fred Baker

During the last several years, ORNL has been leading an effort to develop lignin based precursors. Lignin is a co-product from both the paper making process and the ethanol production process. This presentation will give a brief overview of those efforts and the tasks that still remain.

2. Textile Based Precursors Cliff Eberle

Oak Ridge National Laboratory and FISIFE S.A. have partnered to develop a textile based PAN precursor at substantial cost reduction when compared to conventional carbon fiber precursors. This talk will highlight that R&D effort. It will also address other similar efforts that are being undertaken by other current textile manufacturers. Total manufacturing cost reduction will be projected along with benefits and drawbacks to using this new fiber.

3. Advanced Oxidative Stabilization Felix Paulauskas

Oak Ridge National Laboratory is developing an advanced processing technique to rapidly and inexpensively oxidize polyacrylonitrile (PAN) precursor fibers. Conventional oxidation is a slow thermal process that typically consumes more than 80% of the processing time in a conventional

carbon fiber conversion line. A rapid oxidation process could dramatically increase the conversion line throughput and appreciably lower the fiber cost. A related project has already demonstrated the potential for greatly increasing the line speed in the carbonization and graphitization stages, and rapid stabilization techniques are being developed, but the oxidation time must be greatly reduced to effect fast conversion. This presentation will discuss a plasma-based oxidation technology that integrates with other advanced fiber conversion processes to produce inexpensive carbon fiber with properties suitable for use by the automotive industry. Critical technical criteria include (1) 25 Msi tensile modulus and 1.0% ultimate strain in the finished fiber, (2) uniform properties over the length of the fiber tow, (3) repeatable and controllable processing, and (4) significant unit cost reduction compared to conventional processing. Currently advanced oxidation of PAN fibers continues to progress toward the goal of reducing the cost of manufacturing carbon fiber. To date, researchers have reduced oxidation residence time by about two-thirds compared to conventional oxidation. Future goals include refining and scaling the reactor design and processing protocols to achieve a high speed, multiple large tow, semi-continuous (multiple pass) plasma oxidation process.

#### 4. Microwave Assisted Plasma Carbonization Felix Paulauskas

Oak Ridge National Laboratory developed a microwave-assisted technical alternative to carbonize and partially graphitize polyacrylonitrile (PAN)-based carbon fiber precursor. The work proved that carbon fiber properties were suitable for use by the automotive industry and those could be produced at a reduced cost using microwave-assisted plasma (MAP) processing. It was demonstrated that MAP processing produced acceptable uniform properties over the length of the fiber tow. The project showed that for specified microwave input parameters, fibers with specific properties could be controllably and predictably manufactured using microwave furnaces. Lastly, but most importantly, this project demonstrated the economic feasibility for producing 25 MSI tensile modulus fibers at a significant cost reduction below those produced conventionally. The researchers had demonstrated the ability to MAP carbonize and graphitize one large tow at ~5 m/min or three large tow at ~1 m/min, and generally satisfied the program property requirements. Future research will include (1) the continuation of evaluative parametric studies and characterization of the produced fibers, which will provide a better understanding of the process effects and processing window, and (2) the integration of MAP carbonization into the overall conversion line.

#### 5. Surface Compatibility Soydan Ozcan

The study of surface compatibility between the components of composites is truly a interdisciplinary effort together with materials science, physics, chemistry, mechanics, and manufacturing engineering. The talk will first discuss the mechanisms of adhesion between carbon fiber and specific matrix systems, and physico-chemical characterization of the interface properties. A brief review will be provided on current techniques of fiber surface treatment which have been used to improve the bond strength. The on-going research activities at the ORNL based on conventional techniques will be summarized. The talk will include a discussion on the relevance of the research to implement low cost textile-based carbon fibers in high volume composite production. It will also explore the research programs that are underway on advanced technologies to improve the compatibility between the carbon fiber and the matrix at the ORNL.

#### 6. Future Directions Dave Warren

12:00 PM Conclusion (Instructor team stays afterward to answer questions.)

## INSTRUCTOR TEAM

The instructor team is comprised of members of the carbon fiber research program at Oak Ridge National Laboratory, Oak Ridge, Tennessee. They bring their diverse backgrounds together to conduct research on carbon fiber precursor materials and production technologies to develop innovative carbon fibers to be incorporated into high volume applications in transportation, infrastructure, wind energy, oil and gas exploration, gas storage, aerospace and defense applications.

### **C. David Warren**

Dave is currently the Program Manager for Transportation Materials at Oak Ridge National Laboratory and Field Technical Manager for Materials in the Lightweighting Materials Program of DOE Vehicle Technologies. He has been at Oak Ridge National Laboratory working with composite materials for 18 years. Previously he was the program manager for Peacekeeper ICBM propulsion, Small ICBM basing and Rail Garrison support as a Captain in the United States Air Force prior to joining ORNL in 1991. He conducted all undergraduate and graduate work in materials science and engineering at Vanderbilt University. He has 54 technical publications mostly in composite materials development and application and vehicle technologies.



### **Mohamed G Abdallah, PhD, PE**

Dr. Abdallah has more than 27 years experience in all aspect of advanced composite materials; carbon, and precursor fibers manufacturing and processing technologies, and composites materials and products design manufacturing, marketing. He has managed, and directed consortium teams of researchers from industry, academia, and government agencies to producing ultra high modulus, high strength carbon fibers, technologies for low cost carbon fiber production, stretch broken carbon fibers, carbon nanotubes-related carbon fibers, and the applications of carbon fiber for offshore oil exploration and production, automotive transmission devices, and industrial utilization in infrastructure for renewal and new bridge structures. After 24 years working for Hexcel Corporation/Hercules Company in all aspects of carbon fiber production, prepreg manufacturing, composites structures manufacturing, design, and evaluations, he started MGA-Advanced Composites & Engineering Company (MGA-AC&E) to provide engineering services and consulting to the composites and carbon fiber industries. ORNL has awarded a multi-year consulting contract to MGA-AC&E in research area of carbon fiber



precursor materials and conversion processing technologies. Also, MGA-AC&E has been consulting for many organizations in the carbon fiber and composites manufacturing. He has many publications and technical reports on carbon fiber and composite material and product development. Also, he has been invited speaker in international conferences on carbon fiber and composite materials and products development and manufacturing. He has a patent "Apparatus for Remote Recovery of Submerged Pipelines, US Patent Office, 1984", assigned to Exxon Production Research Company

***Positions Held:***

2006 – Consultant, MGA-Advanced Composites & Engineering,  
1995 - 2006 Programs Manager/Senior Staff Scientist, Hexcel Corp, Salt Lake City, Utah  
1982 – 1995 Senior Research Scientist, Hercules, Inc. Salt Lake City Utah  
1980 - 1982 Teaching fellow, ME Department, University of Houston, Texas,  
1976 - 1980 Senior Research Engineer, Exxon Research & Production Company  
1973 – 1976 senior Design Engineer, K Brown & Root, a Halliburton Company

***Formal Education:***

PhD, Mechanical Engineering (Applied Mechanics), University of Houston, Houston, TX.  
MS, Mechanical Engineering, Louisiana State University, Baton Rouge, LA  
BS, Mechanical Engineering, Assuit University, Assuit, Egypt

***Honors:*** Member Tau Beta Pi, Engineering Honor Society

***Professional Societies:*** SAMPE, Professional Engineer in the State of Utah

**Dr. Frederick S. Baker**

Dr. Frederick Baker is a member of the Distinguished R&D Staff at the Oak Ridge National Laboratory, where he is the Principal Investigator for the Low Cost Carbon Fiber (Alternative Precursors) Project. Prior to joining ORNL in 2004, Fred worked for 40 years in the pharmaceutical (antibiotics) and specialty chemicals industries, developing new products and production processes. His work over the past 30 years has been directed to the production and use of activated (nanoporous) carbon products for diverse gas phase



and liquid phase applications. He is widely recognized as an expert in the mechanisms of carbon activation, and how to manipulate process conditions to tailor product properties (porosity, surface chemistry, etc.) to specific applications. He has extensive hands-on experience in transferring R&D technology into the production and commercial environments. Fred has been awarded over 40 United States and foreign patents on the production and applications of activated carbon materials. He is a Fellow and past Chairman of the American Carbon Society. He received his Bachelors degree in Applied Chemistry (with 1<sup>st</sup> Class Honors) in 1969 and his PhD degree in Surface Chemistry in 1974, both from Brunel University in London, England.

Fred's current research interests include: development of processes for low cost production of carbon fibers from sustainable, renewable resource materials (notably lignin) for lightweight automotive composites and also for the manufacture of activated carbon fibers; utilization of activated carbon fibers in electrical swing adsorption (ESA) systems for enhanced gas phase filtration, including more energy-efficient HVAC systems incorporating CO<sub>2</sub> and VOC capture technology to meet regulatory standards in schools and commercial buildings; development of an enhanced and more efficient CO<sub>2</sub> scrubbing and air purification system for submarines, including passive CO<sub>2</sub>/VOC removal in disabled submarines for increased survivability; fuel gas storage in nanostructured carbon materials, including low pressure storage of natural gas as a vehicle fuel and of hydrogen for fuel cell use; and development of cost-effective processes for production of nanoporous carbons for electrical energy storage devices, including electrochemical capacitors ("super" or "ultra" capacitors) and Li-ion batteries.

### **Dr. Nidia C. Gallego**

Dr. Nidia C. Gallego is a member of the Research Staff of the Carbon Materials Technology (CMT) Group within the Materials Science and Technology Division of the Oak Ridge National Laboratory (ORNL), which is engaged in the development and characterization of carbon materials for the U.S. Department of Energy. Dr. Gallego received her M.Sc (1996) and Ph.D. (2000) in Materials Science and Engineering from Clemson University (Clemson, SC). Dr. Gallego is the author of several papers on the subject of high-performance carbon fibers, graphite foams for thermal management and effect of neutron damage on graphite foams. She is the Secretary/Treasurer of the American Carbon Society (since July 2004), and has been an active member of the local chapter of ASM International where she was the chair for 2004-2005.



Dr. Gallego's research interests include: the thermal physical properties of carbon materials; low-cost carbon fibers; high-performance carbon fibers and graphite foams for thermal management applications; fracture behavior and modeling of nuclear grade graphites; the effects of neutron damage on the structure and properties of carbon materials; and, development and characterization of porous carbon materials for gas separation and energy storage applications.

### **Dr. Amit K. Naskar**

Dr. Amit K. Naskar is a staff scientist in the Oak Ridge National Laboratory's (ORNL) Materials Science & Technology Division. His area of research emphasis is on carbon fibers, alternative carbon precursors, polymeric materials (fibers, films, plastisols, and elastomers), and



composites. He is currently working on advanced processing routes for low-cost carbon fiber production, renewable resource polymers, and automotive composites: his research supports the Department of Energy's interests in vehicles of the future. Dr. Naskar earned his Ph.D. degree in Elastomer Technology from Indian Institute of Technology (IIT), Kharagpur, India (2002) and Master of Technology in Fiber Science from IIT, Delhi, India (1998). Prior to joining ORNL he has worked as a post-doctoral researcher with NSF Research Center for Advanced Engineering Fibers and Films, Clemson University, South Carolina on carbonaceous materials, carbon fiber conversion, high performance liquid crystal copolyester fibers, and renewable resource materials. Industrial experiences of Dr. Naskar include his role as a production engineer in a textile melt-spinning and yarn texturing plant, chemist in a fertilizer, vegetable oil industry, pharmaceutical formulation unit, and co-op intern in an elastomer and tire R&D laboratory. He has published extensively in refereed journals, has numerous abstracts in conference proceedings and has disclosed five inventions and filed one patent application.

### **Dr.-Eng. Felix L. Paulauskas**

Dr. Paulauskas is the technical leader in plastics, polymer processing, and carbon fibers processing/conversion technology, with special emphasis in microwave and plasma processing technology, at the ORNL's composite materials group. Dr. Paulauskas is the Principal Investigator (PI) on projects to develop plasma-based techniques for oxidizing, carbonizing, and graphitizing PAN-based carbon fibers. Dr. Paulauskas is also the PI in the low cost textile PAN based carbon fibers, high strength carbon fibers and fiber surface treatments/modifications for matrix adhesion projects. Dr. Paulauskas is named as the lead inventor on five pending or issued patents, three inventions awaiting filing, an R&D 100 Award nomination, and several publications related to processing of carbon fibers. Dr. Paulauskas is credited with 10 patents and about 38 publications in materials processing using microwave energy and plasma energy. Dr. Paulauskas earned his Dr.-Eng. in chemical engineering from the University of Stuttgart, Germany, in 1979. He earned a Diplom-Eng. in polymer processing engineering from the University of Stuttgart, Germany, in 1974. He earned BS and MS degrees in chemical engineering from the University of Zulia, Maracaibo, Venezuela, in 1970 and 1971, respectively. Dr. Paulauskas has been an ORNL staff member since 1990. Before joining ORNL, Dr. Paulauskas worked as a technical consultant in plastics sciences from 1988–1990; for AT&T Bell Laboratories from 1981–1987 in the areas of developing molds, dies, and other tooling for rubber and plastic products; polymer process development and analysis; material failure analysis; and component design; as a consultant in 1988 and 1989 in the injection molding and plastics processing area; in 1971–72 as Assistant Professor of Chemical Engineering at the University of Zulia, Maracaibo, Venezuela; and in 1969–71 as a student assistant in the Venezuelan petroleum industry.



## **Cliff Eberle**

Mr. Eberle is the Technology Development Manager for the ORNL Polymer Matrix Composites group. In this capacity, he identifies strategic research needs, crafts research proposals and programs around those needs, manages projects, and contributes technical expertise. His primary technical and project management emphasis is in the areas of carbon fiber and electron beam processing. His project management expertise includes intellectual property management, partnership development, and commercialization. His technical expertise includes equipment and facility design, scaleability, and economics. He currently develops and manages projects and programs related to carbon fibers, energy, and national security. Mr. Eberle has 25 years of professional experience in various engineering, scientific, management, and program development capacities including approximately 14 years of experience with composite materials. Mr. Eberle earned his M.S. in Mechanical Engineering from Oklahoma State University in 1984 and joined the staff at Oak Ridge National Laboratory in the same year.



## **Dr. Soydan Ozcan**

Dr. Soydan Ozcan is a Research Associate in the Polymer Matrix Composite Group within the Materials Science and Technology Division of the Oak Ridge National Laboratory (ORNL). Dr. Ozcan's current research interests include: carbon fiber processing, carbon fiber composites, interface adhesion, and nano-composites at ORNL in support of the Department of Energy's Lightweight Materials Program. Prior to joining ORNL, he has worked on carbon fiber composite research for aerospace industry with the NSF Center for Advanced Friction Studies at Southern Illinois University. He has over 30 publications in peer-reviewed journals and conference proceedings. Dr. Ozcan received his B.S. degree in Physics from the Ankara University, Ankara, Turkey. He received his M.S. and Ph.D. in Department of Mechanical Engineering at Southern Illinois University, Carbondale.

