FINAL REPORT

UNIVERSITY RESEARCH INITIATIVE PROGRAM

OFFICE OF NAVAL RESEARCH
DEPARTMENT OF THE NAVY

Submitted by
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Center for Composite Materials Research
and
Mars Mission Research Center

NORTH CAROLINA A & T STATE UNIVERSITY
GREENSBORO, NC

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**Micro-Macro Studies of FMC Materials**

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The following is a brief summary of (a) undergraduate and graduate students, supporting staff, and faculty that were involved in the research activities undertaken during the course of funding made available by the Department of Navy, Office of Naval Research. Further, a brief technical description of areas that was addressed by the staff in the ONR/URIP is also given here: Program Staff Statistics - a) Total Number of Faculty Involved : 11., b) Total Number of M.S Students Trained : 15., c) Total Number of B.S Students Trained : 11., d) Total Number of Publications by Faculty : 43. Major Technical Areas of Work - a) Micro-mechanics and transverse tensile strength determinations with and without low temp. plasma treatments., b) Evaluation of transverse mechanical properties of unidirectional composites using micromechanics finite element model., c) Studies based on molecular dynamics computer simulations and effect of low temperature plasma treatments of the surface of carbon fiber, and d) Analytical study on the stress analysis of filament wound composite cylinder.
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Forward

This work was performed under a University Research Initiative Program initiated by the Department of the Navy, Office of Naval Research Contract N00014-86-K-0682. Dr. Roshdy S. Barsoum, was the Program Monitor.

The study was conducted primarily in the Department of Mechanical Engineering. The Department of Chemical Engineering was also involved as a co-participating department.

As a part of the ONR/URIP in composite materials, our university was a collaborating and a participating institution in the National Center for Composite Materials Research at the University of Illinois and with its other collaborating institutions during the course of this contract.
Summary

The following is a brief summary of (a) undergraduate and graduate students, supporting staff, and faculty that were involved in the research activities undertaken during the course of funding made available by the Department of Navy, Office of Naval Research. Further, a brief technical description of areas that was addressed by the staff in the ONR/URIP is also given here:

Program Staff Statistics:

* Total Number of Faculty Involved: 11
* Total Number of M.S Students Trained: 15
* Total Number of B.S Students Trained: 11
* Total Number of Publications by Faculty: 43

Technical Areas:

* Micro-mechanics and transverse tensile strength determinations with and without low temp. plasma treatments - Profs. Filatovs & Sadler,

* Evaluation of transverse mechanical properties of unidirectional composites using micromechanics finite element model - Prof. Kelkar,

* Studies based on molecular dynamics computer simulations and effect of low temperature plasma treatments of the surface of carbon fiber - Prof. Kabadi, and

* Analytical study on the stress analysis of filament wound composite cylinder - Profs. Raju and Avva.
Synergistic Effect of ONR/URIP Funding

* Establishment of a Formal Center for Composite Materials Research

- Consisting of 14 faculty members supporting 35 to 40 graduate and undergraduate students. Some of the projects undertaken by the CCMR faculty were/are:

1. **Micro-Macro Studies of Fiber Reinforced Composite Materials** (The basis for this report)

   Principal Investigator: Prof. V. Sarma Avva
   Coinvestigators: Profs. Filatovs, Kabadi (Ch. E), Kelkar, Sadler, and Skeen.
   Sponsor: Office of Naval Research, Dept. of Navy
   Technical Area: Micro and macro behavior of laminated composite materials subjected to a variety of loads.

2. **Testing and Mechanical Properties Characterization of New High Temperature Materials**

   Principal Investigator: Prof. J. Sankar
   Coinvestigator: Prof. Ajit Kelkar
   Sponsor: Naval Air Development Center, Dept. of Navy
   Technical Area: Understanding the behavior of selected glass Ceramics for use in propulsion systems.

3. **Mechanical Properties/Testing of Ceramic Fiber Composites**

   Principal Investigator: Prof. J. Sankar
   Coinvestigator: Prof. Ajit Kelkar
   Sponsor: Martin Marietta/DoE
   Technical Area: Understanding the behavior of SiC/SiC system for Fossil Energy Applications.

4. **Testing and Evaluation of Advanced Ceramics at High Temperatures in Uniaxial Tension**

   Principal Investigator: Prof. J. Sankar
   Coinvestigator: Prof. Ajit Kelkar
   Sponsor: Martin Marietta/DoE
   Technical Area: Understanding the effectiveness of ceramic systems in future heat engines (transportation).
5. Analysis of Composite Laminates subjected to Low Velocity Impact Loading

Principal Investigator: Prof. Ajit Kelkar
Coinvestigators: Profs. J. Sankar and William Craft
Sponsor: Wright Research and development Center, Air Force
Technical Area: Understanding the strength and fracture behavior of selected composites under impact loading.

6. Vacuum Brazing of Aluminum

Principal Investigator: Prof. Devdas Pai
Sponsor: Ford Motor Co
Technical Area: Enhancement and computer control of vacuum brazing process for aluminum automotive parts such as radiators, heaters.

7. Stress Concentration Studies: CMC Materials

Principal Investigator: Prof. V. S. Avva
Sponsor: General Electric Aircraft Engines

8. Testing and Evaluation of Dynamic Tensile Properties of Magnesium-Based Metal Matrix Composites

Principal Investigator: Prof. J. Sankar
Coinvestigator: Profs. Avva and Kelkar
Sponsor: Battelle (PNL)
Technical Area: Determination of dynamic properties of MMC

9. Study of the Behavior of Aircraft Tire Coupons Under Various Loading Conditions

Principal Investigator: Prof. V. S. Avva
Coinvestigators: Profs. Pai, and Sadler
Sponsor: WRDC (Flight Dynamics)
Technical Area: Develop test methods and techniques to evaluate aircraft tire coupons

Current Grants/Contracts*


* Some of the research contracts/grants undertaken by faculty that are not currently associated with the ONR/URI are omitted here.

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**Educational & Student Training Component**

The following courses are available for students associated with the Center:

- ME-226: Manufacturing Processes
- ME-336: Strength of Materials
- ME-346: Strength of Materials Laboratory
- ME-560: Modern Engineering Materials
- ME-569: Engineering Materials Laboratory
- ME-612: Modern Composite Materials
- ME-616: Design by Finite Element Analysis
- ME-650: Mechanical Properties and Structure of Solids
- ME-660: Introduction to Boundary Element Method
In addition, we hold monthly:

- Faculty seminars
- Graduate student seminars

Further, a request to authorize the offering of a Ph. D degree program beginning with the Fall of 1993 by the Department of Mechanical Engineering, home the Center for Composite Materials Research and the Mars Mission Research Center, is now under consideration by The University of North Carolina and the Board of Governors.

- Establishment of Research Laboratories

1. Processing and Fabrication Laboratory
2. Mechanical Testing Laboratory
3. Microscopy and Photography Laboratory
4. Analytical and Computational Laboratory
5. Scanning Electron Microscopy Laboratory

- Major Laboratory Equipment

Processing

1. Autoclave
3. Hydraulic Compression Molding Presses
1. Filament Winding Machine
1. Low Temperature Plasma Equipment
1. Prepreg System
1. Vacuum Oven
2. Electrical Ovens

Evaluation and Characterization

1. Thermal Mechanical Analyzer
1. Thermogravimetric Analyzer
4. Servohydraulic Universal Testing Machines
1. Scanning Electron Microscope
1. Energy Dispersive System With Thin Window Detector
1. Heavy Duty Furnace (-300/+1000 Degrees F)
1. Very High Temp. Furnace (+2800 Degrees F)
1. Nikon Epiphot Microscope (X - 1000)
1. X-Ray (shielded) System
3. Environmental Chambers
1. Polaroid MP-4 Camera and accessories
1. Dynamic Mechanical Analyzer (on order)

**Computational**

1. Tektronix Workstation
6. Sun SPARCstation 1+ GX Workstations

In addition to the above major equipment, the Center has numerous PC's and access to the School of Engineering Convex C 120, CRAY Y-MP at The North Carolina Supercomputing Center, and other computational facilities via network.

* **Established a Mars Mission Research Center**

- This is a multi-year joint research center with North Carolina State University funded by NASA under its University Space Engineering Centers' program.

Associate Director: Prof. V. Sarma Avva


Sponsor: National Aeronautics and Space Administration

Technical Area: Study of the behavior of laminated and textile composites subjected to various loads.
Abstracts research work by URIP faculty


The work was systematic exploration of the microdamage and microfailure in carbon/epoxy composites. The goal was to identify failure originating microstructural features and determine their controlling parameters. The work was comprised of several linked tasks which are briefly discussed as follows:

A method was developed for using small scale specimens, with a test volume of approximately 5 mm³. This method bridges the micro-scale tests such as the single fiber tests, and the macro or bulk tests. The stress state corresponds more to that if the overall composite and the method can reveal micromechanical behavior which may be masked in larger scale tests. The method consists of fabrication procedure and small, automated, testing stage capable of accommodating a variety of specimen such as tension, compression, or shear. The method has potential for obtaining mechanical property profiles of thick section and in-service components.

The above method has been used for extensive in situ (optical and SEM) microtests on transverse specimens. As the transverse strength is of central importance in composites, the program centered on identifying crack-originating microstructural features by direct measurement of through-the-ply properties. Plasma treated and as-received fibers were tested, and based on the fractographic and statistical evidence it was concluded that the failure initiated at Weibullian flaws formed from a complex in which the fiber aggregation and interface strength were the main governing parameters. The distribution patterns of fiber packing in plies and filament winding were studied. Statistical analysis established the most likely distributions for various manipulations of the fibers, and it was shown that in filament winding the usual fiber volume is approximately 2/3, with each fiber having an average of 5.3 nearest neighbors.

Various tests such as compression, shear, and fracture toughness were conducted to study failure events and the influence of variables such as interface condition, notch sensitivity, and fabrication variables.

b. 1. Interfacial Studies Using Molecular Dynamics Computer Simulations, Mr. Daval H. Buch & Prof. Vinayak N. Kabadi

In this work, we have studied the interface between the bulk n-hexane and n-hexane adsorbed on graphite surface. We first carried out simulations for bulk liquid n-hexane and a monolayer of n-hexane on graphite
surface. Next we simulated n-hexane between two flat graphite surfaces. This has allowed us to identify the interface between the bulk and the adsorbed n-hexane phases and characterize the interface in terms of its thickness, thermodynamics and molecular structure. Although a number of simulation studies have appeared in the literature that study monolayers and multilayers of molecules on graphite surface, a study like this one that studies the equilibria between the absorbed and bulk phases and determines the interfacial characteristics has not been attempted before.

This work is a necessary part of stepwise progression that might lead to the application of MD methods to problems in material science, especially the interfacial studies in fiber reinforced polymeric composite materials. The mechanical strength of these materials depends strongly on the interfacial properties. A number additional developments in MD technique would be necessary for this application. There have been investigations in the literature for application of MD technique to systems of real significance, primarily biological systems; but, to our knowledge, this is the first attempt towards an application to problems in material science.


Carbon and Graphite are materials with mechanical properties that are retained at temperatures as high as 25000 K in the absence of oxygen. They are currently used as reinforcing agents in the form of high modulus fibers in the fabrication of high performance resin-matrix composites. These are mainly used in high performance applications such as aerospace and aircraft structures, where the main emphasis is on lightweight along with high strength and modulus. By using composite technology, it is now possible to tailor-make structural materials for specific applications by aligning the fibers in a manner that favors the required performance. The interface between the carbon fiber and the resin matrix is one of the factors that controls the transverse properties in designing a composite.

The aim of the project is to study the effect of low temperature plasma on the surface properties of carbon fibers in order to enhance adhesion with thermosetting resin matrix, and furthermore to study the effect of plasma treatment on other properties of composite laminae such as compressive and tensile strength and fracture toughness. The effects of low temperature plasma treatment on the chemical modification of the surface of carbon fibers were studied using gas plasmas such as air, argon, ammonia, and nitrogen. Plasma surface treatment was observed to enhance the surface concentration of the chemical functional groups which may promote a greater number of chemical bonds between the resin and fiber. It was also observed that the wettability of the fiber
surface was improved by removing the surface contaminants and the weak boundary layer on the fiber surface. Analytical techniques such as Scanning Electron Microscopy (SEM), Auger Electron Spectroscopy (AES), X-Ray Photoelectron Spectroscopy (XPS), and Fourier Transform Infrared Spectrometer (FT-IR) were used to study the surface topography and chemical nature of the plasma treated carbon fiber surface.

In the first phase of the work analysis was made of the enhancement of interfacial adhesion between fibers and the polymeric matrix. Transverse tensile strength (TTS) was used as the measure of the interfacial strength and the fractured surface was analyzed with SEM to observe the adherence of the matrix and fiber. An attempt was made to explain the experimental observations in terms of three major phenomena: i) chemical bonding ii) mechanical bonding, and iii) physical interaction forces. The results show considerable improvement in the transverse tensile properties. Additionally optimum conditions for plasma treatment for best TTS are recommended.


For the analysis of composite structures a variety of mechanical properties are required. These properties can be obtained experimentally by subjecting laminate to axial, shear and transverse loading. Although it is possible to determine the properties of the laminate in axial direction, it is extremely difficult to perform the experimental analysis in transverse direction, as laminate is usually very thin in the transverse direction. An alternate approach is to use the analytical methods which use individual properties of constituent materials to predict the transverse properties of the composite materials. One of these methods is the finite element method. The finite element approach can be effectively used to predict the transverse mechanical properties of composites because the geometry, arrangement, fibers, and matrix can be conveniently described.

In this paper a two-dimensional single fiber and multiple fibers finite element models are presented to predict the transverse mechanical properties of unidirectional composite materials. For both, the single fiber and multiple fibers models, the repeating units were identified. The external surfaces of these repeating units were subjected to uniform displacements. A simple technique based on the superposition principle was developed to simulate multi point constrain boundary conditions. The results obtained by using the present single and multiple fibers finite element models are compared with the results from the classical solutions and other finite element models.

Stress analyses of a composite cylinder consisting of a metal mandrel and a composite winding is considered. Several variables in this composite such as initial fiber tension, dry graphite fiber, etc. are involved in the analyses. A constant tensile force is assumed to exist in the graphite fibers that are being paid off and wound onto a rotating mandrel oriented at a 90° angle to the axis of the cylinder. Two dimensional continuum plane strain analyses are presented for the composite cylinder. The first analysis assumes that the cylinder consists of two different isotropic materials. The second analysis assumes that the cylinder consists of an isotropic and a polar orthotropic materials. Finite element analysis were performed for these two cylinders and the solutions are compared to the continuum solutions.

Experimental investigations showed that thick composite cylinders fail at stress levels at almost one half of failure stresses obtained by thin cylinders tests. This is a significant loss of strength in these thick cylinders. The loss of strength is hypothesized as due to fiber buckling, the snaking motion of fibers along the length of the cylinder, etc. To understand these phenomenon, a good model of the composite cylinder and loading is needed. In this study the stress distributions in a simple 2D-plane strain of a thick composite cylinder subjected to winding tensile load are considered. Several finite element models were developed and analyzed using Computational Mechanics Testbed, COMET finite element software system. These solutions are compared with the analytical solutions obtained and with other solutions in literature. A 2D plane strain model mesh generation computer program for use with the Testbed was developed. This mesh generator yields several finite element models that have gradual transition and good aspect ratio elements with only very few input parameters. These FE models are generated as a part of the Testbed input stream.

Axisymmetric analyses of cylinders with an isotropic mandrel and a polar orthotropic composite are first considered. Exact solutions are obtained for these composite cylinders with external pressure. The exact solutions obtained here degenerated to the Lame's solution when the mandrel and composite are both made up of isotropic materials. The FE solutions are obtained using the Testbed and these results are compared with the exact solutions. Excellent agreement was observed between the two sets of solutions. Also, the present exact solution is compared to Beddoe's axisymmetric solution. Excellent agreement was observed between the two sets of results for the composite with thickness varying from 0.5 to 6.0 inches. The axisymmetric analyses and comparisons, confirmed the validity of the mesh generation, Testbed implementation and Testbed analysis.
The FE Testbed analysis is used to obtain the stress distributions in composite cylinders due to asymmetric loading. Once again plane-strain deformations were assumed. The cylinder winding loading is the loading on the cylinder. Two different boundary conditions were hypothesized. In the first case, three nodes on the inner surface of the mandrel were arbitrarily chosen and zero displacements were prescribed at these nodes to prevent rigid body movement. This boundary condition yielded stresses that showed peak values at these boundary condition nodes. These stresses and the boundary conditions are unrealistic and hence were not pursued further. In the second case, the winding load is hypothesized to be equilibrated by shear stresses on the inner surface of the mandrel. This assumption is probably realistic because the winding load is equilibrated by the rotation of mandrel. A simple two-term stress distribution is hypothesized. The constants in this two-term approximation are calculated using the force and moment equilibrium conditions. Finite element analyses were performed for various thick composite cylinders for this asymmetrical loading. These solutions showed that as the thickness of the composite increases, the stresses at the mandrel-composite interface decrease. Through the thickness hoop stresses also decrease as the composite thickness increases. These results suggest that linear analysis such as the one discussed here may not be sufficient to predict the lower strengths for thick cylinders.

**Brief Summary of Accomplishments and Program Impact**

**Accomplishments:**

(i) Presentations and publications by our faculty is an on-going activity. Currently, the emphasis is in getting the technical accomplishments published in referred journals and conference proceedings.

(ii) On an annual basis, about 30 to 50 undergraduate and graduate students are being trained in the sponsored research projects by the faculty in the Center for Composite Materials Research and the Mars Mission Research Center.

(iii) The formal establishment of a Center for Composite Materials Research in September, 1988 may be viewed as an enhancement of our research and training activities in composite materials. The number of faculty working with advanced materials is growing faster.

As a result of significant funding received from ONR through its URI program in 1986, we were able to enhance our research facilities, in particular, thereby enabling our faculty and students to contribute to the national research endeavors in advanced composite materials.
The annual funding level generated by the CCMR faculty is now in excess of $2,000,000. Besides supporting several undergraduate and graduate students, tenured and adjunct faculty, this funding also supports three full-time technicians and two secretaries on a "permanent" basis.

Further, as a result of our activities and strength in composite materials, we were able to compete successfully (with the Department of Aerospace and Mechanical Engineering, and the School of Textiles at North Carolina State University as acolloaborating partners) for the award of a multi-year major research center, now known as the Mars Mission Research Center, by the National Aeronautics & Space Administration under its University Space Engineering Research Centers Program in June, 1988. After an initial five-year funding, the accomplishments in the MMRC program were independently reviewed critically by a peer and a technical committee in April/May, 1992. Based on these independent and favorable reviews, NASA has extended the period and the funding for an additional three-year period through October, 1995.

**ONR/URIP Impact:**

**New Areas:**

(i) Research with textile composites.

(ii) Newer processing and fabrication techniques (Resin transfer molding).

(iii) Search for cost affective composite fabrication technologies.

(iv) Create interest among students to develop newer composite technologies to advanced professionally with further education.

(v) Further explore the limits on the fabrication of thick-walled cylindrical pressure vessels using the textile composite technologies.
Selected Publications/Presentations
by our ONR/URIP faculty

"Effect of Fatigue Load on Graphite Fiber-Reinforced Glass Matrix Composite Material", (V. S. Avva, D. Kelkar, J. Sankar and P. Chander), International Conference on Analytical and Testing Methodologies for Design with Advanced Materials (ATMAM 87); August 26-28, 1987; Montreal, Quebec, Canada.

"Non-Linear Flexural Response of Graphite Fiber Reinforced Glass Matrix Composite Beams" (A. D. Kelkar, J. Sankar, V. S. Avva and A. Sinha), International Conference on Analytical and Testing Methodologies for Design with Advanced Materials (ATMAM 87); August 26-28, 1987; Montreal, Quebec.

"Teaching and Evaluation of Si_3 N_4 in Uniaxial Tension at Room Temperature", (R. Vaidyanathan, J. Sankar, and V.S. Avva );5th Automotive Technology Development Contractors' Coordination Meeting; Department of Energy; October 26-29,1987; Dearborn, Michigan.


Engineering Fibers and Textile Structures for Composites, October 15-17, 1991, North Carolina State University, Raleigh, NC.


R. L. Sadler, “Lower Cost Composites with Resin Transfer Molding (RTM), presented at N.C. A & T State University, Mechanical Engineering Seminar Series, October 9, 1992


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*Personnel could have worked full to part-time during the semester*