Department of Civil Engineering
College of Engineering and Technology
Old Dominion University
Norfolk, Virginia 23529

POLYMER INFILTRATION STUDIES

By
Joseph M. Marchello, Principal Investigator

Progress Report
For the period June 1, 1993 - March 31, 1994

Prepared for
National Aeronautics and Space Administration
Langley Research Center
Hampton, Virginia 23665

Under
Research Grant NAG-1-1067
Robert M. Baucom, Technical Monitor
MD-Polymeric Materials Branch

Submitted by the
Old Dominion University Research Foundation
P.O. Box 6369
Norfolk, Virginia 23508-0369

April 1994

POLYMER INFILTERATION STUDIES

Summary

During the past three months, significant progress has been made on the preparation of carbon fiber composities using advanced polymer resins. The results are set forth in recent reports and publications, and will be presented at forthcoming national and international meetings.

Current and ongoing research activities reported herein include:

- Textile Composites from Powder-Coated Towpreg: Role of Surface Coating in Braiding
- Prepregger hot sled operation in making tape from powder coated tow
- Ribbonizing Powder-Impregnated Towpreg
- Textile Composites from Powder-Coated Towpreg: Role of Bulk Factor in Consolidation
- Powder Curtain Prepreg Process improvements in doctoring of powder
- Hot/Cold shoe for ATP Open-Section Part Warpage Control

Research during the period ahead will be directed toward further development of the new powder curtain pregregging method and on ways to customize dry powder towpreg for textile and robotic applications in aircraft part fabrication.

Studies of multi-tow powder prepregging and ribbon preparation will be conducted in conjunction with continued development of prepregging technology and the various aspects of composite part fabrication using customized towpreg. Also, during the period ahead work will continue on the analysis of the performance of the new solution prepregger.

Polymer Infilteration Studies

Polymer infilteration investigations are directed toward development of methods by which to produce advanced composite material for automated part fabrication utilizing textile and robotic technology in the manufacture of subsonic and supersonic aircraft. This object is to achieved through research investigations at NASA Langley Research Center and by stimulating technology transfer between contract researchers and the aircraft industry.

The powder curtain prepregging system, which was started up successfully last year has been used to produce over three hundred pounds of towpreg. It is currently undergoing modifications. The automated powder return system was contructed and is undergoing tests. Modification are being made to the powder curtain tube, for extended curtain width demonstration. These changes should provide better operating control over fugitive powder and improved towpreg quality control.

Issues in the use of powder coated towpreg for textile applications have been the subject of significant effort. Studies of ways to debulk powder preforms are being conducted, see attachments. Also, work has been initiated on use of gel coating to reduce tow-tow friction during braiding.

Consideration of the ways to customized towpreg for use in automated tow /fiber placement has resulted in several new approaches and will be the subject of a paper to be presented at the SAMPE. Noteworthy among the ideas that have been developed is the potential benefits from use of non-rectangular ribbon, and the thermal wave bonding model of tow placement with on-the-fly-cure, see attachmnent. Several efforts to produce quality towpreg ribbon are underway. In addition to die forming methods, it is planned to investigate making unitape from powdered tow which can then be slit into the desired ribbon geometry.

The following table and attachments provide detailed information about several current and planned research projects.

Forthcoming Presentations and Papers

Attached are abstracts of papers to be presented at 39th International SAMPE Symposium, Anaheim, CA, April 1994

Abstract for the 39th International SAMPE Symposium & Exhibition,
April 11-14, 1994
Abstract Deadline: July 15, 1993

Powder Curtain Process for Fiber Bundle Impregnation

Robert M. Baucom
NASA Langley Research Center
Hampton, Virginia 23681-0001

Maylene K. Hugh and Joseph M. Marchello
Old Dominion University
Norfolk, Virginia 23529

Dry methods of impregnating fiber bundles with polymer powder offer processing advantages over hot-melt, solution and slurry prepregging. They are especially applicable to the prepregging of advanced composites made from polymers with adverse processing characteristics, such as high melt viscosity and limited solubility. Among the dry powder processes, the powder curtain method provides advantages in quality control and powder handling. This paper reports on the progressing development of the powder curtain process.

Uniform metering of powder onto spread tow and subsequent meshing of the powder with the fiber bundle, with minimal fiber damage, are the distinguishing features of the powder curtain process. Recovery of excess powder is achieved using a double belted, powder collection and return system. The powdered tow produced by the process has been used to make textile preforms and ATP ribbon. In addition, towpreg has been processed into unidirectional prepreg tape using the hot sled of the NASA LaRC multi-purpose prepregger.

Submitted for 39th Int'l SAMPE Symposium, April 11-14, 1994
Anaheim, CA

TEXTILE COMPOSITES FROM POWDER-COATED TOWPREG: YARN TREATMENT FOR BRAIDING*

Maylene K. Hugh and Joseph M. Marchello
Old Dominion University
Norfolk, Virginia 23529

J. Timothy Hartness
Cytec Engineered Materials
Anaheim, California 92806

Steve Goodwin
Fiber Innovations, Inc.
Norwood, Massachusetts 02062

Jay G. Shukla
Lockheed Aeronautical Systems Company
Marietta, Georgia 30063-0150

Norman J. Johnston
NASA Langley Research Center
Hampton, Virginia 23681-0001

ABSTRACT

Compression or autoclave molding of textile preforms made from polymer powder-coated yarns offers an alternative to resin transfer molding for the fabrication of net-shape parts. Powder coating the fiber bundle prior to weaving or braiding requires special consideration for both textile processing and for the subsequent debulking of the preform. This study addresses only the former, namely, issues involving the braiding of powder-coated towpreg. Carbon fiber (6k and 12k AS4†, Hercules) coated with epoxy thermoset (AMD0036†, 3M Company) powder was used as the base material. Methods of treating the powder-coated towpreg to improve its "braidability" are reported. These include towpreg twisting, serving with a polyvinyl alcohol (PVA) monofilament, remelting the powder during braiding, and application of surface lubricants, such as zinc stearate, hydroxyl-terminated polyethylene glycol (PEG) and polyacrylic acid. The latter surface treatment provided the most applicable and viable means to facilitate braiding of powder-coated towpreg.

Previous studies have addressed weaving protocol for powder-coated fiber bundles and methods for consolidating the bulky woven preforms. This investigation served to establish a **braiding** protocol for powder-coated yarns by determining ways to maintain yarn integrity and to reduce tow-to-tow friction.

KEYWORDS: Braiding; Manufacturing/Fabrication/Processing; Powder-Coated Towpreg.

---

† Use of trade names or manufacturers does not constitute an official endorsement, either expressed or implied, by the National Aeronautics and Space Administration.

Ribbonizing Powder-Impregnated Towpreg

Donald A Sandusky
The College of William & Mary Williamsburg, VA 23185

Joseph M. Marchello
Old Dominion University, Norfolk, VA 23529

Norman J. Johnston and Robert M. Baucom
NASA Langley Research Center, Hampton VA 23681

ABSTRACT

Dry powder prepregging of thermoplastics is efficient in distributing solid polymer particles throughout continuous filament tows. The resulting towpreg yarn is flexible, bulky and abrasive. Robotic placement material handling systems are generally designed to utilize stiff, preconsolidated ribbons with consistent cross-section. The research included herein summarizes efforts toward developing a bench-scale processing method to convert a single powder coated towpreg yarn into a fully preconsolidated ribbon. A comprehensive study of debulking techniques revealed a variety of issues critical to effective ribbonizing, including towpreg material quality, transverse squeeze flow, appropriate timing for heating and pressure application, and tool contact/release.

Several processing techniques have been designed, built and experimentally evaluated to serve as a basis for understanding the unique characteristics of the towpreg ribbonizing process. Use of reactive plasticizers or solvents was excluded altogether. Due to availability, three powder towpreg yarn materials, Aurum (500)/IM-8 (prepregged by BASF), LaRC-IA/IM-7 and PEEK/AS-4 (prepregged by NASA LaRC), were used in the evaluation of these processes.

By utilizing desirable attributes of several of the experimental processes, a novel processing technique was developed. This powder coated towpreg ribbonizer was comprised of two primary components. The hot bar fixture facilitates transverse melt squeeze flow while the cool nip-roller assembly solidifies the ribbon into a preconsolidated ribbon with consistent cross-section. The process has been shown to provide quality ribbon from various high-temperature performance thermoplastic powder-coated towpreg yarns. The observed experimental rates and temperature ranges indicate that this technique could be readily integrated as a final step in the powder prepreg manufacturing process. The resulting process has been scaled-up to simultaneously convert multiple powder coated yarns into multiple ribbons and has also been used to produce a single 3 inch wide prepreg tape.

KEYWORDS: **Thermoplastic Powder Towpreg, Melt Processing, Prepreg Tape, Polyimides, Ribbonizing.**

Polyimide Composites From Hot-Melt Prepreg Tape Using Powder-Coated Towpreg

S.P. Wilkinson
College of William & Mary
Williamsburg, Va.

N.J. Johnston
NASA Langley Research Center
Hampton, Va.

and

J.M. Marchello
Old Dominion University
Norfolk, Va.

Impregnation of fiber tow bundles with high melt viscosity polymers is a vital step in the preparation of advanced composite materials. The multi-purpose prepregging machine at NASA Langley Research Center provides a means for investigating the prepregging science of solution and powder systems. In this study, carbon fiber powder-coated with several thermoplastic polyimides was used to establish the machine operating protocol for producing consolidated uni-directional prepreg tape. Well consolidated test specimens prepared from the tape exhibited good mechanical properties.

Machine operating data and observations were utilized in the ongoing analysis of the prepregging machine. An extension of the hot melt and solution prepreg flow number (PFN) concept was developed for making tape from powder coated tows.

GRAPHITE/THERMOPLASTIC CONSOLIDATION KINETICS

J. A. Hinkley
NASA Langley Research Center
Hampton, VA

D. C. Working
Analytical Services and Materials Inc.
Hampton, VA

J. M. Marchello
Old Dominion University
Norfolk, VA

ABSTRACT

Continuous carbon fiber/thermoplastic plies were heated briefly under pressure. The degree of bonding achieved was then determined by peeling them apart at room temperature. Unlike previous studies of autohesion, this experiment was designed to allow access to the very short-time regime relevant to *in-situ* consolidation methods. It was shown that the bond strength depends on the time and temperature of heating, and that the net effect of the heating cycle can summarized using time/temperature superposition. It is predicted that measurable bonding could be achieved in 0.01 second at a temperature 50°C above the glass transition temperature.

KEY WORDS: composite consolidation, thermoplastics, tow placement