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TECHNICAL HIGHLIGHTS

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**MONSANTO/WASHINGTON UNIVERSITY
ONR/ARPA ASSOCIATION**

March 1975

FINAL TECHNICAL REPORT

ONR CONTRACT NO. N00014-67-C-0218

ARPA ORDER NO. 976

**PROGRAM MANAGER
ROLF BUCHDAHL**

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MONSANTO RESEARCH CORPORATION
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Final Technical Report
Covering Research on High Performance
Composites being conducted for the
ADVANCED RESEARCH PROJECTS AGENCY
Under ONR Contract No. N00014-67-C-0218
Formerly No. N00014-66-C-0045

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TECHNICAL HIGHLIGHTS

Introduction

The Monsanto Company/Washington University Association was formed under Office of Naval Research/Advanced Research Projects Agency sponsorship in 1965 with concurrent, interdependent objectives.

These were:

- (a) to establish a broader technical base for the area of composite materials through fundamental research and an active communications effort,
- (b) to develop an interdisciplinary teaching program to train scientists and engineers in efficient utilization and design with composites and
- (c) to demonstrate a means by which an effective working relationship can be formed between a university and private industry.

Since the goals were all somewhat exploratory in nature, maximum flexibility was permitted in choosing and modifying the approaches to be followed, in staffing, and in reporting. Furthermore, initial funding and the first contract renewal were for two-year periods to

facilitate planning and to allow time for the impact of the program to be measured. In general, all the goals were satisfactorily met. In the following paragraphs the major accomplishments made under the contract are briefly summarized. More detailed lists of reports and publications, patents, and degree recipients are appended in later sections.

Major Technology Advances

As measured by their acceptance and use in the technological community, the Association contributed major technical advances in three general areas: Chemistry and Physics, Mechanical Analysis and Characterization, and Fabrication and Processing.

The chemistry of silane coupling agents was unraveled enabling improvement of a wide range of coupling agents. Industrial producers of coupling agents are now making use of the work to develop new types of coupling agents. A new flexible innerlayer and a process for applying it to continuous, large diameter glass filament were developed. It is highly likely that this technology will be applied directly to the manufacture of glass reinforced pipelines. The innerlayer greatly improves fatigue resistance and elevates the strength properties of the large diameter glass system to those of small diameter glass, making a superior, economical pipeline system available.

In the area of mechanical analysis and characterization, many of the structure-property relations currently used in designing composite structures were developed under the ONR/ARPA contract. The stiffness design equations currently employed in the Air Force Design Manual were developed at Washington University. A tensor failure criteria

for continuous fiber laminates, as well as fracture toughness measuring procedures, were developed under the contract and are currently in use by the technological community.

With regard to fabrication and processing, basic rheological parameters important in the transfer and injection molding of filled melts were identified and characterized. These concepts are currently in use within the Monsanto Company as well as in other corporations. New ways to produce short-fiber molding compounds for both thermosetting and thermoplastic matrices were developed and patented.

In order to disseminate the technology and data generated under the contract, 170 research reports were written over the contract period. Over 85 publications in reputable journals resulted. Seven patents have so far been issued on technology developed under the contract. Detailed lists of patents, publications, and reports are appended in later sections of this report.

Examples of Technical Exchange and Interaction with the Materials Community

One of the measures of success in accomplishing the above objectives is the degree of technical exchange and interaction with the materials community. Under the ONR/ARPA contract, this interaction took the form of people-to-people contact first via collaboration with industrial and government organizations.

In 1971, the Monsanto/Washington University Association participated in a structural design project involving the McDonnell-Douglas F-15 aircraft. Part of the aircraft's wing, the spar root fitting for the

intermediate torque box, was selected as the component for design and five design concepts were developed utilizing high performance fibers with both plastic and metal matrices.

The Association also participated in an evaluation of a fractured experimental speed brake with members of Goodyear Aerospace Corporation, at their request. The component was fabricated for the F-5 plane by Goodyear under Air Force Contract. Results from the study defined 1) the contribution of the continuous fibers to the structure and the extent to which maintaining the position of these fibers during molding is a problem, 2) the extent to which fiber position and orientation changes when molding chopped prepreg using notched metal dies, and 3) the source and cause of the fracture.

Consultations were held with the Polymers Division of the National Bureau of Standards concerning possible directions that composites research might take at NBS.

Samples of very highly aligned dispersed graphite fibers in an epoxy matrix were obtained for evaluation from two sources in the United Kingdom, the Explosives Research and Development Establishment and Fothergill and Harvey. These highly aligned systems were characterized both mechanically and morphologically.

Concepts and technology developed under the Contract are now being used by the Rubber Chemicals Division of Monsanto Industrial Chemicals Company in Akron to develop new reinforced rubber systems containing unregenerated cellulose short fibers as reinforcement. Some of these systems are nearly ready for marketing.

Several small testing contracts have been completed by Washington University using specialized equipment developed under the contract especially for anisotropic materials. Among these participating industrial concerns are Arundale Manufacturing, Inc., Rawlings Sporting Goods Company, Western Textile Products, and Tiffany Industries, Inc.

Consultations have recently been held with Owens-Corning Corporation, Air Products Company, and Lord Corporation concerning ways in which these companies might productively interact with the Washington University research program.

A second form of interaction with the materials community involved efforts by the Association to stimulate education of the materials community in the area of composite materials and to serve as a vehicle for gathering and disseminating the work of others in the field.

Early in the Contract (1967), the Monsanto/Washington University Association sponsored a Composite Materials Workshop on the Physical Aspects of Composite Materials. The proceedings of this workshop were published by Technomic Publishing Company as Volume I of their Progress in Materials Science Series.

The authoritative Journal of Composite Materials was initiated by the Association in 1967. It is published quarterly by Technomic Publishing Company and continues to be the leading journal in the area of the physics and mechanics of composite materials.

The Monsanto/Washington University Association organized six symposia on advanced composites during the duration of the Contract. These meetings were held annually, and attracted national and

international attention. The proceedings of the last of these, "Composite Materials in Engineering Design", were published by the American Society for Metals in 1973 (one volume 727 pages).

For the last several years, Washington University has organized and conducted a short course entitled, "Advances in Reinforced Plastics Technology". This 3-day intensive course is sponsored by the Plastics Institute of America and draws heavily on members of the Association for lecturers.

University/Industry Coupling An Evaluation

A number of requirements and possible barriers to an effectively coupled effort have become apparent in the course of the Program. The requirements include in order of importance:

1. Geographic proximity so that day-to-day interaction is possible (and convenient).
2. Mutual dedication to the goals (with clear cut benefits to each of the partners) and mutual dependence on one another to provide key expertise and/or facilities (thus forcing interaction where otherwise there might be hesistance).
3. Flexibility in administrative procedures, use of equipment and facilities, experimental approaches and reporting on the part of both organizations.
4. A single program manager, but interaction between counter-parts at all administratives and technical levels.
5. Willingness of the industrial partner to become involved in work sufficiently fundamental to meet academic degree standards,

and willingness of the academic partner to maintain a practical focus to the work and facilitate use of, but protect (patents, etc.), results of possible commercial value.

6. Freedom to publish and broad interactions within the technical community. These require that the problems be recognized as important and long range enough for thesis research, yet not so intensely competitive as to hamper meaningful technical exchange with others in the field outside the parent organizations.

As implied by the various requirements listed, among the more important barriers to an effective coupling program are:

1. Wide geographic separation.
2. Insistence on such rigid procedures and protocol that communication is reduced.
3. Demand for poorly defined organization or existence of complete autonomy or independence by either partner.
4. A highly competitive atmosphere preventing publication or technical exchange outside the program.
5. Unwillingness to accept some practical focus and/or insistence on complete use orientation.
6. Inability of either partner to make allowances for and adjust to the pace of the other (that of the university will necessarily be slower).

Tendencies for some of these occasionally cropped up in the Monsanto/Washington University program from time-to-time; fortunately, these were largely overcome.

The success of the coupling demonstration is exemplified by the fact that the two organizations have continued to work together in bidding for additional contracts, by appointment of some of the faculty members as Monsanto consultants and some of the Monsanto staff as affiliate faculty, and by voluntary cost-sharing by Monsanto of a portion of the coupled program having possible commercial implications.

Educational Program

The Materials Science and Engineering Program of Washington University School of Engineering and Applied Science is a flexible, interdisciplinary activity designed to encourage graduate study and research in the area of homogeneous and multi-phase structural materials with heavy emphasis on reinforced plastics. It was developed largely over the last eight years under the auspices of the ONR/ARPA contract. The Program stresses the integration of basic and applied research on reinforced plastic systems and provides for the education of materials scientists and engineers in this field.

The Program is now well established and enjoys international recognition as one of the few in the United States dealing with the preparation and long-term performance evaluation of polymeric composites. Since 1967, 22 master's degrees and 28 doctoral degrees have been awarded; 8 graduate students and one post-doctoral fellow are currently in residence. Faculty in the Program hold their appointments from the Departments of Chemical Engineering and Mechanical Engineering. Affiliate faculty participate from Monsanto Company and the Air Force Materials Laboratory. A list of current faculty in the Program is

appended. Most graduates of the Program are now employed by industry and are highly sought after by materials-oriented companies. A list of graduates and their current employers is included in a later section.

Preparation of a Monograph on Short Fiber Composites

From a technical point of view, the unique feature of this contract was its emphasis on short fiber composites as structural materials. We undertook the writing of a monograph to define the role of short fiber systems in structural materials, to outline the state-of-the-art in short fiber technology development, and to summarize the Monsanto/Washington University Association's contributions to this fast-growing area of Materials Science and Engineering.

Unfortunately, writing of the monograph did not start until near the end of the Contract. As Association members went on to different things after the Contract concluded, it became more difficult to meet target milestones for completion of the book. Completion is now set for Fall, 1975. Editing, rewriting and drawing are now proceeding. A chapter-by-chapter status report appears below.

Title: Engineering Composites Reinforced With Short Fibers

Publisher: Marcel Dekker, New York

Preface: 1st draft completed

Chapter 1 - Introduction - 1st draft completed

Chapter 2 - Constituent Materials - 1st draft completed

Chapter 3 - Prediction of Composite Performance - 1st draft completed

Chapter 4 - Practical Performance Levels - 1st draft not complete, resource references and data complete

Chapter 5 - Laboratory Processing of Composites - 1st draft completed

Chapter 6 - Commercial Processing of Short Fiber Composites - 1st draft completed

Chapter 7 - Composite Characterization Techniques - 1st draft completed

Chapter 8 - Composite Testing - 1st draft not completed (all subsections completed but not drawn together)

Chapter 9 - Design with Composite Materials - 1st draft completed, 2nd draft completed

Appendices

Data Analysis and Reduction - 1st draft completed

Bibliography - not completed

Glossary and Index - not completed

PATENTS

To date the following patents based on Contract-sponsored research have been issued.

U.S. 3,518,221 - Shaped Composites Comprised of Filler Particles Enveloped
in a Thermosetting Resin - A. S. Kenyon and R. J. Slocombe.

U.S. 3,562,198 - Epoxide Resin Solution - R. J. Slocombe.

U.S. 3,580,882 - Mica Reinforced Composites - L. E. Nielsen and J. E. Fields.

U.S. 3,598,693 - Molding Composition and Method - H. M. Anderson and J. E. Calfee.

U.S. 3,626,041 - Apparatus and Process for Making Continuous Filament -
J. E. Fields and E. H. Mottus.

U.S. 3,708,456 - Polycondensate Reinforcement Process Using an Interfacial-
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U.S. 3,790,438 - Ribbon Reinforced Composites - L. E. Nielsen and T. B. Lewis.

PUBLICATIONS FROM MONSANTO/WASHINGTON UNIVERSITY ASSOCIATION

Internal HPC No.	Authors	Title - AD No.	Publication Information
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65-2	D. F. Siegmund	Crack Theory and Stress Analysis of Composite Systems Monsanto Lit. Survey 1674 AD 488381	
65-3	* W. J. Calvin	Rheology of Filled Systems Monsanto Lit. Survey 1675 AD 488382	
65-4	* W. J. Calvin	Surface Energy Monsanto Lit. Survey 1676 AD 488383	
65-5	R. A. Landy	Tape and Ribbons in Composites Monsanto Lit. Survey 1677 AD 488384	
65-6	M. K. Collins	The Use and Function of Coupling Agents in Glass-Reinforced Plastics Monsanto Lit. Survey 1678 AD 488385	

* Indicates author(s) from Washington University

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Monsanto Lit. Survey 1679
AD 488386 |
| 65-8 | W. C. Peterson | Fibers for High Performance Composites
Monsanto Lit. Survey 1680
AD 488387 |
| 65-9 | M. J. Farrell | Surface Migration or Diffusion of Gases or Liquid Along Solid Interfaces
Monsanto Lit. Survey 1681
AD 488388 |
| 66-1 | C. M. Bower | Computerized Research Index
AD 481654 |
| 66-2 | D. C. Morris &
C. H. Adams | Isotropic and Composite Materials for High Performance (Structural) Applications
AD 481654 |
| 66-3 | * H. J. Duffey | Evaluation of Mathematical Analyses of Discontinuous Fiber Reinforced Composites
AD 481654 |
| 66-4 | P. E. Chen | Stress Fields Around Edge Cracks
AD 481654 |
| 66-5 | A. S. Kenyon | Thick and Multiphase Zone Between Matrix and Reinforcing Agent
AD 481654 |
| 66-6 | J. F. Schaefer &
D. J. Moritz | Diffusion of Water at a Glass-Resin Interface
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66-7	R. E. Lavengood	Characterization of Whisker Fibers AD 481654
66-8	R. B. Weil	Solid State Physics of High Performance Fibers (internal progress report)
66-9	R. M. Anderson	Relationships Between Matrix Properties and Dynamics Fatigue Resistance in Filament Wound Composites AD 481654
66-10	M. K. Collins	The Rheology of Fibrous Materials in Low Viscosity Media: A Literature Survey-- Monsanto Lit. Survey 1682 AD 488389
66-11	C. M. Bower	Glossary to the Science of Composites AD 634606
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<u>Organic Coatings and Plastics</u>
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M. Riley

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M. Riley

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Ultimate Tensile Properties of Flow Molded Short Fiber Composites

L. A. Goettler

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Rate Effects in the Flow Assignment of Short Fiber Molding Compounds in Convergent Channels

L. A. Goettler

72-150

Characterization of Fiber Orientation Patterns Produced in the Flow Molding of Reinforced Thermosets

L. A. Goettler

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D. Ruhmann and E. M. Wu

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J. Shea | Tensile (Compressive) Properties
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| 73-165 | M. Takano | Viscosity Effect on Slow Orientation
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| 74-166 | S. V. Kao, L. Nielsen
C. T. Hill | Rheology of Concentrated
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I. Effect of the Liquid-
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C. T. Hill | Rheology of Concentrated
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II. Highly Concentrated
Suspensions and Pastes |
| 74-168 | S. V. Kao, L. Nielsen
C. T. Hill | Rheology of Concentrated
Suspension of Spheres.
III. Suspensions Agglomerated
by an Immiscible Second Liquid |
| 74-169 | Richard O. Maschmeyer &
Christopher T. Hill | Rheology of Concentrated Suspensions of
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Study |
| 74-170 | Richard O. Maschmeyer &
Christopher T. Hill | Rheology of Concentrated Suspensions of
Fibers in Tube Flow: III. Suspensions
With the Same Fiber Length Distribution. |

Faculty in Materials Science and Engineering Program

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|-------------------|--|
| R. Buchdahl | Ph.D., Johns Hopkins University, 1940, Affiliate Professor Chemical Engineering, Director of Fundamental and Exploratory Research, Central Research Department, Monsanto Company |
| R. M. Christensen | Ph.D., Yale University, 1961, Professor Mechanical and Aerospace Engineering |
| L. B. Gulbransen | Ph.D., University of Utah, 1949, Professor of Metallurgy |
| C. T. Hill | Ph.D., University of Wisconsin, 1962, Associate Professor of Chemical Engineering |
| J. C. Halpin | Ph.D., University of Akron, 1969, Affiliate Professor of Chemical Engineering, Senior Scientist, Air Force Materials Laboratory, Wright-Patterson AFB |
| J. L. Kardos | Ph.D., Case Institute of Technology, 1965, Chairman of the Materials Science and Engineering Program, Director of the Materials Research Laboratory, Professor of Chemical Engineering |
| J. M. McKelvey | Ph.D., Washington University, 1950, Dean of the School of Engineering and Applied Science and Professor of Chemical Engineering |
| L. E. Nielsen | Ph.D., Cornell University, 1945, Affiliate Professor of Chemical Engineering, Distinguished Science Fellow, Central Research Department, Monsanto Company |
| C. Thies | Ph.D., Michigan State University, 1962, Associate Professor of Chemical Engineering |

S. W. Tsai

D.Eng., Yale University, 1961,
Affiliate Professor of Mechanical
Engineering; Chief, Mechanics and
Surface Interactions Branch, Air
Force Materials Laboratory

E. M. Wu

Ph.D., University of Illinois,
1965, Associate Professor of
Mechanical and Aerospace Engineering

DEGREES RECEIVED UNDER ONR/ARPA PROJECT
COMPOSITE-RELATED ACTIVITIES

<u>Name</u>	<u>Degree Earned & Date Received</u>	<u>Advisor</u>	<u>Employer After Graduation</u>
David C. Morris	M.S. - Jan. 1966	Gulbransen	Monsanto Company
Jacques Joseph	M.S. - Jan. 1967	Kardos/Nielsen	Industry, Paris, France
Richard Lavengood	M.S. - Jan. 1968	Gulbransen	Monsanto Company
William McDonnell	M.S. - June 1968	Kardos	Oil Shale Company, Australia
Aron Preis	M.S. - June 1968	Isnai	Technion, Israel
Robert Johnson	M.S. - Jan. 1970	Duffey	Aeronautical Company
Stephen Lowy	M.S. - Jan. 1970	Kardos	Monsanto Company
Gopel Gaonkar	D.Sc. - Jan. 1967	Hohenemser	Washington University
Dieter Droste	D.Sc. - Jan. 1969	DiBenedetto	DuPont, Delaware
Samiron Chatterjee	D.Sc. - Jan. 1968	Gulbransen	
Jimmy Ho	D.Sc. - Jan. 1969	DiBenedetto	TRW, Inc., California
Jing Ming Lin	D.Sc. - June 1969	DiBenedetto	Parks Aeronautical College
Ashok Dhingra	D.Sc. - Jan. 1970	Gulbransen	DuPont
Arlo Moehlenpah	D.Sc. - Jan. 1970	DiBenedetto	Hydro-Air Engrg., St. Louis
Kenneth Trachte	D.Sc. - Jan. 1970	DiBenedetto	Esso Research & Engrg., Texas
Allan Wamback	D.Sc. - Jan. 1970	DiBenedetto	General Electric, Mass.
Francis S. Cheng	D.Sc. - June 1970	Kardos/Tolbert	Esso Research & Engrg., Texas

<u>Name</u>	<u>Degree Earned & Date Received</u>	<u>Advisor</u>	<u>Employer After Graduation</u>
Bing Lin Lee	M.S. - June 1971	Nielsen	
Luigi Nicolais	M.S. - June 1971	DiBenedetto	Research Laboratory for Polymer Technology and Rheology of the National Research Council, Naples, Italy
Charles L. Johnson	D.Sc. - June 1971	Bagley	Denver Broncos
Roger L. Kaas	D.Sc. - June 1971	Kardos	General Motors, Dearborn, Michigan
Kenneth Jerina	M.S. - June 1971	Wu	Air Force Materials Laboratory, Dayton, Ohio
Theodore Neisen	D.Sc. - June 1971	Bagley	Exxon, New Jersey
M. Scigliano	D.Sc. - June 1971	Bagley	Monsanto Company
Adrian Cooper	D.Sc. - June 1972	Wu	Babcock & Wilcox
James V. Gauchel	D.Sc. - June 1972	DiBenedetto	U.S. Navy Res. Lab.
B. S. Mehta	D.Sc. - June 1972	DiBenedetto/Kardos	Mobil Oil Company
Daniel Peretz	D.Sc. - June 1972	DiBenedetto	Technician, Haifa
Ken Yang	D.Sc. - June 1973	Kardos	IBM
Roger Fountain	D.Sc. - June 1973	Kardos	American Standard
S. V. Kao	D.Sc. - Dec. 1973	Nielsen	McGill U. (post doc)
F. Nazem	D.Sc. - June 1973	Hill	U. of Wisconsin (post doc)
P. K. Agarwal	M.S. - June 1971	Bagley	U. of Pittsburgh
H. M. Li	M.S. - June 1971	Kardos	U. of Pittsburgh (doct)

<u>Name</u>	<u>Degree Earned & Date Received</u>	<u>Advisor</u>	<u>Employer After Graduation</u>
T. C. Nguyen	M.S. - June 1971	Kardos	Iowa State U. (other field)
D. J. Stubblefield	M.S. - June 1972	DiBenedetto	Inter. Am. Devel. Bank (Mex.)
R. Elvin	M.S. - June 1972	Kardos	Tamplastic, Inc., Turkey
W. Fujimoto	M.S. - June 1972	Noton	McDonnell-Douglas
D. A. Hurwitz	M.S. - June 1972	Roberts	Small Coating Industry (Maine)
W. Kiang	M.S. - June 1972	Roberts	Washington University (doct.)
K. D. Roberts	M.S. - June 1973	Hill	U. S. Army
J. Raison	D.Sc. - Aug. 1974	Kardos	Ford Motor Company
J. Sandoval	M.S. - Aug. 1974	Kardos	U. of Mexico
D. R. Ruhmann	D.Sc. - Aug. 1974	Wu	McDonnell-Douglas Corporation
S. V. Murty	D.Sc. - Aug. 1974	Kardos	Sherwood Medical
R. Maschmeyer	D.Sc. - June 1974	Hill	Corning Glass Company
B. Whipple	D.Sc. - June 1974	Hill	Whirlpool Corporation
T. A. Duffy	M.S. - June 1974	Kardos/Michno	M & T Chemical
K. Jerina	D.Sc. - June 1974	Kardos	MTS Systems, Inc.
L. Teasley	M.S. - June 1974	Hill	Monsanto Company