Eighteenth Asilomar Conference on Polymeric Materials

Eric Baer

Case Western Reserve University
Cleveland, OH 44106-7202

U.S. Army Research Office
P.O. Box 12211
Research Triangle Park, NC 27709-2211

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Eighteenth
Asilomar Conference
on Polymeric Materials

February 12-15, 1995

Pacific Grove, CA
COOPERATIVE FIBER MICROBUCKLING

C. Mueller, A. Gorius*, S. Nazarenko, A. Hiltner and E. Baer
Center for Applied Polymer Research
Department of Macromolecular Science
Case Western Reserve University
Cleveland, Ohio USA 44106

*Rhone-Poulenc Fibers et Polymeres
25 Quai Paul Doumer
92408 Courbevoie, France

ABSTRACT

Cooperative fiber microbuckling is a compressive failure mechanism in unidirectional fiber-reinforced composites. This phenomenon was studied in a simplified three-dimensional system composed of polyamide fibers in a silicone matrix which permitted in situ observation of fiber microbuckling during compressive deformation. First, the microbuckling behavior of a single fiber was analyzed by determining the critical wavelength of microbuckling which was shown to depend on the diameter and modulus of the fiber, and the modulus of the matrix. An excellent quantitative analysis of fiber microbuckling in terms of existing stability theories was achieved.

Subsequently, to study cooperative fiber microbuckling, this system was used to model composites with two fibers. Cooperative shear microbuckling occurred when the fibers were close to one another. When the fibers were farther apart, a new noncooperative microbuckling mode was observed rather than the commonly expected cooperative transverse mode. These observations led to a new experimentally derived stress overlap criterion for cooperative shear microbuckling stability. When the total in-plane shear stress at the midpoint between the fiber is greater than 30% of the shear stress at the fiber surface cooperative microbuckling will occur. Using this criterion and Rosen's models of cooperative microbuckling, it was predicted that the cooperative transverse mode, if it appears at all, exists only for very special cases.

References:
The Processing, Structure, and Properties of PBO Fibers: A Lyotropic Liquid Crystalline System

by

R. A. Bubeck

The Dow Chemical Company
Midland, Michigan 48674

There has been considerable interest recently in fibers of very high modulus and tensile strength composed of semi-rigid rod molecules such as polybenzothiazole (PBZT) and polybenzoxazole (PBO). These fibers possess very high modulus, strength, and thermal resistance. The general scheme for the drawing of PBO fibers starts with a dry-jet wet-spinning process from a 13 - 15% solution of the polymer in polyphosphoric acid. Upon coagulation of the drawn lyotropic solution, a filament/fiber with a microfibrilar microstructure results. The fundamentals of PBO and PBZT fiber drawing were studied through a series of novel synchrotron-based in-situ X-ray scattering experiments of monofilament drawing from lyotropic solutions of poly(cis-benzoxazole) (PBO) and poly(trans-benzothiazole) (PBZT). The purpose of the study was to determine orientation and microstructure development in the draw zone as a function of shear rate in the capillary die, spin draw ratio (SDR), and temperature. The filament orientation parameter (f) was found to depend strongly on spin draw ratio, but not shear rate. The transition of the extrudate from opaque to transparent is complete at about a SDR = 3 and f of 0.9. The orientation was found to increase down the extrudate with more completion of the draw down as one proceeds further from the die face. Coherence lengths on the order of 19nm (axial), and 4.5nm (lateral) have been observed. These "microdomain" sizes are consistent with the "crystallite" sizes typically observed in coagulated fiber, and with the columnar nematic character of the drawn lyotropic extrudate. The occurrence of the microdomains in the draw zone as a precursor to the microfibrilar structure is believed to be the origin of low filament compressive strength.
HIGH PERFORMANCE FIBERS FOR COMPOSITES

R.J. Diefendorf
Materials & Engineering Program
Clemson University
Clemson, SC 29634-0907

The NMAB commissioned a report on "High Performance Synthetic Fibers for Composites" which issued in 1992. Much has occurred on both the national and international political scenes since. There has been a decrease in world-wide activity in the development of new fiber types or even new grades, partially because of the economic downturn in Japan and Europe, but also because companies realize that markets will take years to develop. However, the decrease in the number of companies already has probably stabilized the industry, and some capacity is being added for industrial applications. One point, emphasized in the report, was the financial difficulty of producing small quantities of a critically needed fiber, but at a profit. The formation of a virtual company among fiber producers and end-users (jet-engine companies), under government urging, may be a solution to this problem. The urgency of some of the recommendations of the report has decreased, since progress elsewhere has slowed. Yet, the report properly predicted several policies would hurt the competitiveness of the U.S. fiber companies. Other recommendations, as yet unimplemented, are still required to maintain a strong textile and high performance fiber industry.
POLYMER ADHESION TO FIBER SURFACES
IN COMPOSITE MATERIALS

Lawrence T. Drzal

Michigan State University
Department of Chemical Engineering
East Lansing, Michigan 48824-1326
517-353-5466

It has been shown that adhesion between the polymer matrix and the fiber surface is a necessary condition for acceptable composite mechanical performance. Fiber surface treatments that improve fiber adhesion to polymeric matrices have been the subject of many research efforts over the last decade. As a result, researchers have proposed that various mechanisms have been the sole factor responsible for improvements in adhesion measured after surface treatment including the formation of interfacial chemical bonds between fiber and matrix; increases in fiber surface area; non-chemical polar/dispersion type interactions; removal of the native fiber surface; and the role of the polymer in the interphase.

This study has sought to quantify these interactions and establish the interrelationships between these potential variables. Surface and interfacial chemical characterization was coupled with fiber-matrix interfacial shear strength measurements in a carbon fiber-polymer composite system fabricated from fibers with variable fiber surface treatments combined with both thermoset and thermoplastic matrices. In thermoset systems, chemical bonding could be detected. Chemical bonding of the matrix constituents at levels of less than 5% is responsible for changes in adhesion as large as 40%. Quantitative surface topographical analysis using scanning tunneling microscopy has shown that after conventional surface treatments on carbon fibers, there is a systematic increase in the fiber surface topography which increases the mechanical interlocking mechanism and thereby increases adhesion by 25%. Molecular segregation of lower molecular weight polymers has been shown to take place in an amorphous thermoplastic system. Molecular rearrangement at the fiber surface favors low molecular weight segregation and low adhesion results. The interphase polymer mechanical properties (shear strength in thermosets and yield strength in thermoplastics) limits the overall level of adhesion regardless of the surface treatments methods used.
Abstract

NOVEL ADSORPTION SYSTEMS BASED ON HIGH SURFACE AREA FIBERS

by

James Economy

During the past several years we have made significant progress in the design of new improved high surface area fibers for removing trace contaminants from air or water streams. Use of fibers is considered essential in order to obtain high contact efficiency with trace contaminants and also to permit rapid regeneration of the fibers and recovery of the contaminant.

One of the key features of these activated fibers is the major improvement in selective adsorption of contaminants through control of the pore size and shape, surface area and pore surface chemistry. With this new knowledge we have been able to tailor separation systems for removing trace contaminants down to a few ppm without the excessive cost penalties usually ascribed to the removal of contaminants down to this level. In fact in one case we were able to remove contaminants down to below 1 ppb starting with a solution containing 15 ppm of a H₂O soluble impurity.
Grafting of Microparticles Onto Fiber Surfaces

Ian R. Hardin
The University of Georgia, Athens, Georgia

The overall objective of this work is to create new technology that will permit addition of several types of protective functions to fabrics, such as barriers to chemical agents, absorption of gaseous vapors, and heat and flame protection. Some types of radiation protection may also be possible. The protective functions are added by incorporating very small particles to the surface of cotton and various manufactured fibers. These particles are attached to surfaces through the novel formation of polymer grafts onto the surfaces of fibers in such a way that the polymers retain their reactivity. Further reaction of the polymers with various types of very small particles can occur by covalent bonding rather than using adhesive bonding. This enables maximum use of the protective qualities of the particles since much less of the surface area of the particles will be inactivated. If large amounts of polymer chains are attached to each fiber and these chains react to particles, then the total surface area of the particles will be very large. This in turn will allow an extensive area for absorption or for coverage of the surface of the fiber. The polymers that are grafted to the fiber surfaces are vinyl polymers and will be attached by two different methods. By the judicious selection of monomer structures, functions such as soil release, fire resistance, and antibacterial activity can be added to the fiber even before the particles are added to the fiber surface. Among the particles that will be attached to the still reactive graft polymers are activated carbon, titanium dioxide, silicon dioxide, zeolites, and particles containing phosphorus and/or bromine. The development of polymerization methods for grafting will involve methods of activation of the fiber surfaces, control of the molecular weights of the graft polymers, and development of methods to attach particles to the end of the polymer chains. The specific fibers that will be included in the study are cotton, wool, and nylon. These fibers all contain potential reactive groups for the graft polymers and have properties that are inherently desirable for clothing. The crosslinking of the polymer chains to the fibers will be achieved by free radical techniques. The preferred method is to graft copolymers of mono and polyfunctional monomers to the particles first. These grafted copolymers contain unsaturations that are used to bind them with the fibers in a subsequent step. The incorporation of small particles to the fibers is accomplished by grafting these fibers with polymer chains that are terminated by the particles. Based on preliminary work, systems of grafting the polymers, combined with techniques that allow the polymers to remain reactive, should allow reaction with a variety of particles that can be incorporated for protective functions. The feasibility of incorporating various types of particles is being examined. These include zeolite crystals, several inorganic oxides such as titanium dioxide and silicon oxides, activated carbon, and particles that include phosphorus and bromine in their structures. It is expected that these particles will be effective as a protective barriers in various ways.
Delamination Failure Mechanisms of PC/SAN Microlayers

A. Hiltner, T. Ebeling, and E. Baer
Department of Macromolecular Science
Case Western Reserve University
Cleveland, OH 44106

and

J. Im
Dow Chemical Company
Midland, MI 48674

The mode of delamination failure, and the corresponding delamination toughness, of coextruded microlayer sheets consisting of alternating layers of polycarbonate (PC) and styrene - acrylonitrile copolymer (SAN) were studied with the T-peel test. Four delamination modes were observed; the mode depended on both the SAN layer thickness and the PC layer thickness. The SAN layer thickness determined whether the crack propagated along a PC-SAN interface (interfacial delamination) or through crazes within a SAN layer (crazing delamination). Only interfacial delamination was observed if the SAN layers were thinner than 1.5μm. For SAN layers thicker than 1.5μm, the amount of crazing delamination increased as the thickness of the SAN layers increased. The corresponding toughness, measured as the critical load, was lowest for interfacial delamination failure and increased with the amount of crazing delamination. With both interfacial and crazing delamination, the crack could either propagate along a single layer or could jump from one layer to the next. This depended on the PC layer thickness. If the PC layers were thin enough, the crack jumped from one layer to another with tearing of the PC layer. This significantly increased the delamination toughness.
"LIQUID CRYSTALLINE POLYMER FIBERS - THE NEXT GENERATION"

DR. MICHAEL JAFFE & DR. GERALD FARROW
HOECHST CELANESE CORPORATION
SUMMIT, N.J. & CHARLOTTE, N.C.

Thermotropic liquid crystalline polymers offer a route to fiber, film and molded products of high thermal, chemical and mechanical performance. Based on a disrupted p-phenylene polyester structure, chemistries leading to a broad range of processing and performance characteristics have been developed, with melting points ranging from about 225°C to over 350°C and fiber moduli between 70 and 150 GPa have been developed with a concomitant increase in fiber strength. Other LCP properties of note include cryogenic toughness, low creep, high vibration damping, low moisture regain and excellent barrier properties to gases and other low molecular weight species.

Investigation of the structural origins of LCP properties, from the molecular to the macroscopic, leads to the definition of hierarchical assembly of fibrillar units, similar to that found in natural systems such as collagen. Analysis of the various size scales of structure comprising the hierarchy indicates that, in highly perfect uniaxial structures such as fibers, mechanical properties are dominated by molecular parameters, while thermal and chemical stability are more directly linked to the size and perfection of crystalline units. Very recent work in our laboratory indicates that raveled or intertwined chains may also be a key to the understanding of LCP performance. These considerations appear to be generally true for all highly oriented polymer systems, including the lyotropic aramids and even high modulus polyethylene. Inherent in these highly oriented chain structures is a high degree of property anisotropy, leading to low toughness and poor compressive behavior transverse to the molecular chain axis. The strategy for successful market exploitation of the fiber properties of these LCP's is to therefore utilize in end uses where the positive attributes are manifest, including cost vs. performance parameters.

Over the past decade, markets for LCPs have grown substantially, with products ranging from electrical connectors widely used in the computer industry to cut-resistant gloves used by meat packers. The rate of future growth will primarily be determined by the economics of monomer production. If either existing or new, suitable, monomers can be produced at significantly lower cost, then additional market opportunities will present themselves for exploitation. Innovative technical processing could also spur development in certain fields.
PROCESSING, STRUCTURE AND PROPERTIES OF
EXTENDED CHAIN POLYETHYLENE FIBERS

Sheldon Kavesh
AlliedSignal, Inc.

ABSTRACT

Extended chain polyethylene fibers have the highest specific strength of any man-made fiber and a unique and exceptional combination of strength, modulus and toughness. U.S.P. 4,413,110 described for the first time novel polyethylene fibers of specific modulus three-fourths that of diamond.

SPECTRA® extended chain PE fibers are beneficially applied in artificial tendons, ligaments and joint prosthesis. Protective gloves with remarkable cut resistance are saving the hands of surgeons and meat packing workers. Sports equipment such as kayaks, canoes, bicycles, boats, sails, skis and others are employing SPECTRA fibers and composites to provide toughness and strength at low weight. SPECTRA fibers and SPECTRA SHIELD® are protecting the lives of our police and our military in bullet resistant vests, helmets and armor.

SPECTRA fibers do have limitations. Among them are low melting point and tendency to creep. The relationships among processing, structure and properties is examined. The crystallinity and perfection of the crystalline phase are remarkably independent of processing conditions whereas the orientation of a less ordered phase controls tensile properties.

The potential strength of SPECTRA is several fold higher than presently available commercially. The utility of extended chain polyethylene is just beginning to be realized.
Abstract: New Structural Polymers Modeled after Natural Materials

John P. O'Brien
DuPont Central Research and Development
Wilmington, Delaware

Nature provides examples of structural materials in the form of composites and fibers that exhibit combinations of physical properties and functionality not yet achieved in man-made materials. The basic building blocks of natural polymers are excellent paradigms for the design and synthesis of new polymers for advanced materials applications. Protein polymers are of particular significance because protein biosynthesis is now comparatively well understood and can be genetically manipulated with a high level of precision. Over the past decade, several ab initio designed, structural protein polymers have been synthesized, demonstrating both the feasibility of the approach and the potential breadth of accessible, new polymeric materials. These new synthetic methods thus offer hope for a family of next generation structural polymers with precisely specified chemical and physical properties for both specialty and commodity applications.
INTERFACES IN MULTIPHASE BLENDS

D. R. Paul
Department of Chemical Engineering and
Center for Polymer Research
The University of Texas at Austin
Austin, TX 78712

Abstract

Blends of immiscible polymers offer attractive opportunities for achieving unique property combinations and for improving mechanical toughness. A key to the development of successful multiphase blends is to understand the nature of the interface between the phases. In many cases, it is necessary to alter the interface by some form of compatibilization in order to achieve a desirable blend morphology and to strengthen the interface. This presentation briefly examines three commercially important systems that serve as useful case studies.

The first is polycarbonate/ABS blends. The relevant polymer-polymer interaction energies have been determined and used to provide a theoretical characterization of the interfaces in this system. The calculated interfacial tensions and thicknesses correlate well with interfacial strength and blend morphology and toughness. It is interesting to note that no compatibilizer is employed in these commercial blend systems.

The second example involves toughening of polyamides using maleated elastomers. The graft copolymers formed during processing exert an enormous influence on morphology and allow particles of an optimum size to be generated. The effect of polyamide functionality and processing conditions on morphology generation will be described. The extent of chemical reaction at the interface appears to be strongly influenced by the nature of the polyamide-elastomer physical interaction or interfacial thickness.

The final example considers nylon 6/ABS blends that have been reactively compatibilized using imidized acrylic polymers. These materials lead to supertough compositions when the morphology is properly controlled. Studies of deformation mechanisms suggest that rubber particle cavitation is an important trigger for subsequent massive shear yielding of the polyamide matrix; cavitation does not appear to be possible in uncompatibilized blends owing to premature interfacial failure.
Abstract for 18th Asilomar Conference 1995

Novel Methods of Fiber Production and Use

by Roger S. Porter
University of Massachusetts

Unusual methods of fiber production will be discussed, such as preparation of polyolefin and teflon fibers directly from reactor powder and ultra fine fibers of Kevlar made by the electrospinning method of Reneker. New fiber composites concepts to be discussed will include bonding methods and preparation of novel materials such as molecular velcroix and compositions with a negative Poisson's Ratio. Two and three dimensional composite reinforcement will be discussed.

Continuous fiber constructions are also an avenue to smart composites. Concepts on utilizing fibers to monitor temperature and stress excursions, including composite damage, will be presented. Fiber properties that may be monitored to evaluate composite performance include Raman spectra or electrical and thermal conduction. Examples will be offered. The use of optical fibers to measure composite fatigue will also be discussed.
CRYSSTALLIZATION OF THERMOPLASTIC COMPOSITES:
POLY(ETHYLENE TEREPTHALATE)/POLYCARBONATE BLENDS

Dr. Ludwig Rebenfeld

TRI/Princeton
P. O. Box 625
Princeton, New Jersey 08542

ABSTRACT

Blends of poly(ethylene terephthalate) (PET) and polycarbonate (PC) over a full range of compositions were studied in isothermal crystallization from the melt using differential scanning calorimetry (DSC). The crystallization rate of PET as well as various thermal transitions were found to be dependent on the PC content. In cooling scans from the melt, the dynamic crystallization temperature of PET was observed to be affected by the PC. In blends with high PC content, a novel dual-peak crystallization was observed.

PET/PC blends reinforced with aramide and glass fibers were studied in order to establish the influence of reinforcing fibers on PET crystallization. Aramide fibers enhance the crystallization rate while glass fibers depress the crystallization rate in 100% PET. However, in PET/PC blends, both fiber types depress the crystallization rate of PET. These effects of fibers on crystallization rate are interpreted in terms of fiber nucleation and impingement phenomena. The degree of crystallinity, melting temperature, and glass transition temperature were also characterized in relation to PC content in the blend for both neat and fiber reinforced systems.
Ballistic Evaluation of Microlayered Plastics: Polycarbonate/Polyester and Polycarbonate/Poly(Styrene Acrylonitrile)

Heidi L. Schreuder-Gibson and William G. Kohlman
U.S. Army Natick Research, Development and Engineering Center
Fiber and Polymer Science Division, S&TD
Natick, MA 01760-5020
(508) 651-5487

Extruded microlayer sheets of polycarbonate and two other plastics (poly(cylohexane-1,4-dimethyl terephthalate) and poly(styrene-acrylonitrile), produced by DOW Chemical Central Research, were tested for ballistic impact performance. Although much impact testing has been reported for these blends, showing improved impact properties as microlayer thickness decreased, these are the first ballistic tests that have been conducted. Normalized ballistic test results showed that some microlayered samples performed as well as and slightly better than injection molded polycarbonate samples. The failure mechanism was affected by the composition and the number of layers. Increasing composition of the polycarbonate fraction and decreasing the thickness of the microlayer had the effect of decreasing the percent of brittle failures.
New Stilbene-Based Copolymers for Fibers

Heidi L. Schreuder-Gibson, Walter Yeomans, Peggy Cebe* and Y.Y. Cheng*
U.S. Army Natick Research, Development and Engineering Center
Fiber and Polymer Science Division, S&TD
Natick, MA 01760-5020
(508) 651-5487

A series of polycarbonates with 5 different stilbene mesogens has been
prepared and characterized, expanding upon past work by Jackson¹, Blumstein² and
Percec³ with polyesters and polyethers of stilbene. It has been found that the stilbene
mesogen, mono- or di- substituted with methyl or ethyl groups, significantly affects
polymer morphology. Methyl substituted stilbenes are semicrystalline and appear to
be liquid crystalline, whereas ethyl substituted stilbenes are amorphous. Disubstitution
lowers the transition temperatures from those values observed for monosubstituted
stilbenes.

These characteristics will be discussed along with plans to copolymerize
stilbene mesogens with other meso-phase forming monomers.


* MIT, Department of Materials Science and Engineering
NANOSCOPIC SUPERMOLECULES - CONTROLLED THREE
DIMENSIONAL DENDRITIC POLYMERS

Donald A. Tomalia

Michigan Molecular Institute
1910 W. St. Andrews Road
Midland, Michigan 48640

Generation: 0  1  2  3  4  5
Surface Groups: 3  6  12  24  48  96

ABSTRACT: Synthetic strategies have been developed for the preparation of precise
macromolecular building blocks referred to as Starburst\* dendrimers. The
predictable precision of mass and valency (i.e., number of reactive surface groups)
displayed by these dendrimers, as a function of generation, validates their proposed
role as fundamental nanoscopic building blocks. Ideal, defect free structures of
Starburst\* polyamidoamine (PAMAM) dendrimers (e.g., NH₃ core; generation = 2.0,
MWt. 2,414) have been synthesized in kilogram quantities with overall yields of 60-
70%. Electrospray ionization mass spectroscopy techniques have confirmed a typical
polydispersity for a polyamidoamine (PAMAM) dendrimer (Generation = 4.0; MWt.
= 10,632) to be; M_W/M_N = 1.0005. The precise masses and surface valencies
associated with these dendrimer structures allows one to view these entities as
"nanoscopic, three dimensional analogues" to atoms. As such, basic rules of
chemical combination between dendrimers to give definite, stoichiometric
compositions can be defined much as first noted by Dalton for atoms. The use of
these nanoscopic building blocks to construct supramolecular and supermolecular
structures such as membranes, nanoscopic rods, clusters, compounds, necklaces and
microcrystallitites will be reviewed.

\*Trademark of Dendritech Inc.

D.A.T.
1/4/95
"Oscillating Catalysts for Block Polymer Synthesis"

Robert Waymouth, Geoffrey Coates, Elisabeth Hauptman, Anne-Lise Mogstad, Michael Bruce, Mark Krejchi

Department of Chemistry, Stanford University

We report a new strategy for the dynamic control of stereochemistry in an olefin polymerization reaction. We report the synthesis of thermoplastic elastomeric polypropylene based on the unbridged metallocene (2-PhInd)$_2$ZrCl$_2$ [2-PhInd = 2-phenylindenyl]. This catalyst was designed to isomerize between chiral and achiral coordination geometries during the polymerization reaction to produce atactic - isotactic stereoblock polymers. The metallocene precursor (2-PhInd)$_2$ZrCl$_2$ in the presence of methylaluminoxane polymerizes propylene to yield rubbery polypropylene. The physical properties of this polymer are most readily explained in terms of a stereoblock structure consisting of isotactic sequences that can crystallize and provide physical crosslinks for the amorphous atactic stereosequences. The microstructure of the polypropylene, described by the isotactic pentad content [mmmm], increases with increasing propylene pressure and decreasing polymerization temperature to produce polypropylenes with isotactic pentads ranging from 6.3% to 28.1%. Thus, this catalyst is capable of producing polymers which range in properties from gum elastomers to thermoplastic elastomers.
PROCEEDINGS

EIGHTEENTH ASILOMAR CONFERENCE ON POLYMERIC MATERIALS

February 12-15, 1995

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SUBMITTED BY

Eric Baer
Department of Macromolecular Science
Case Western Reserve University
Cleveland, Ohio 44106

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CASE WESTERN RESERVE UNIVERSITY
CLEVELAND, OH

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PROFESSOR RUSSELL DIEFENDORF
CLEMSON UNIVERSITY
CLEMSON, SC

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PROFESSOR LAWRENCE DRZAL
MICHIGAN STATE UNIVERSITY
EAST LANSING, MI

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MICHIGAN MOLECULAR INSTITUTE
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PROFESSOR ROBERT WAYMOUTH
STANFORD UNIVERSITY
STANFORD, CA
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Prof. Eric Baer
Case Western Reserve University
Cleveland, OH 44106

Prof. Larry Drzal
Michigan State University
East Lansing, MI 48824

Dr. Robert Bubeck
The Dow Chemical Company
Midland, MI 48674

Prof. James Economy
University of Illinois
Champaign, IL 61801

Dr. David Buscheck
3M Company
St. Paul, MN 55144

Dr. Gerald Farrow
Hoechst Celanese Corporation
Charlotte, NC 28232

Dr. Alfredo Causa
Goodyear Tire & Rubber Company
Akron, OH 44309

Dr. Stephen Foster
Dow Chemical Company
Granville, OH 43023

Dr. Larry Charbonneau
Hoechst Celanese Corporation
Summit, NJ 07901

Dr. Gunilla Gillberg
Kimberly Clark Corporation
Roswell, GA 30076

Dr. George Collins
Hoechst Celanese Research Division
Summit, NJ 07901

Dr. Andre Goriou
Rhone-Poulenc
Courbevoie, FRANCE

Dr. Andrew Crowson
U.S. Army Research Office
Research Triangle Park, NC 27709

Dr. Ian Hardin
University of Georgia
Athens, GA 30602

Dr. Ronald DeMartino
Hoechst Celanese Corporation
Summit, NJ 07901

Prof. Anne Hiltner
Case Western Reserve University
Cleveland, OH 44106

Prof. Russell Diefendorf
Clemson University
Clemson, SC 29631

Dr. Michael Jaffe
Hoechst Celanese Corporation
Summit, NJ 07901

Dr. Marvin Doerr
Hoechst Celanese Corporation
Charlotte, NC 38232

Dr. Sheldon Kavesh
Allied Signal
Morristown, NJ 07960
Dr. Patrick Kelly  
John Wiley & Sons, Inc.  
New York, NY 10158

Dr. Doug Kiserow  
Army Research Office  
Research Triangle Park, NC 27709

Dr. James Klug  
3M Company, Austin Center  
Austin, Texas  78726

Dr. Kenneth Ko  
Armstrong World Industries  
Lancaster, PA 17604

Dr. Jacqueline Kroschwitz  
John Wiley & Sons, Inc.  
New York, NY 10158

Dr. George Mayer  
The Pentagon  
Washington, D.C. 20301

Dr. Edward McEntire  
PPG Industries  
Allison Park, PA 15101

Dr. Charles Myers  
Amoco Chemical Company  
Naperville, IL 60563

Dr. Stam Mylonakis  
54 Wilshire Drive  
Belle Mead, NJ 08502

Dr. John O'Brien  
E.I. DuPont Experimental Station  
Wilmington, DE 19880

Dr. Rudolph Pariser  
851 Old Public Road  
Hockessin, DE 19707

Dr. Richard Parker  
BFGoodrich Central R&D  
Brecksville, OH 44141

Prof. Donald Paul  
University of Texas  
Austin, TX 78712

Dr. Brian Pengilly  
Goodyear Tire & Rubber Company  
Akron, OH 44306

Prof. Roger Porter  
University of Massachusetts  
Amherst, MA 01003

Dr. Ludwig Rebenfeld  
TRI/Princeton  
Princeton, NJ 08542

Dr. Daniel Roberts  
Raychem Corporation  
Menlo Park, CA 94025

Dr. David Schiraldi  
Hoechst Celanese Corporation  
Charlotte, NC 28210

Dr. Heidi Schreuder-Gibson  
U.S. Army Natick RDE Center  
Natick, MA 01760

Dr. Mark Shmorhun  
BFGoodrich Company  
Brecksville, OH 44141
Dr. Robert Singler  
U.S. Army Research Laboratory  
Watertown, MA 02172

Dr. Mark Sinsky  
Goodyear Tire & Rubber Company  
Akron, OH 44305

Dr. David Squire  
1032 Diamond Crest Court  
Santa Barbara, CA 93110

Prof. Vivian Stannett  
North Carolina State University  
Raleigh, NC 27695

Dr. James Summers  
The Geon Company  
Avon Lake, OH 44012

Dr. Donald Tomalia  
Michigan Molecular Institute  
Midland, MI 48640

Prof. Robert Waymouth  
Stanford University  
Stanford, CA 94305

Dr. Debra Wilfong  
3M Company  
St. Paul, MN 55144

Dr. Marty Woods  
The Geon Company  
Avon Lake, OH 44012

Dr. Statis Zachariades  
POLTECO Incorporated  
Burlingame, CA 94011