



**Report from the 2nd Workshop on:  
Characterization and Modeling of the  
Interface/Interphase of Polymeric  
Materials and Systems  
December 7 and 8, 1998**

**Tinh Nguyen, BFRL  
Charles Han, MSEL  
Richard R. Cavanagh, CSTL  
Rose Ryntz, The Ford Motor Company**

U.S. DEPARTMENT OF COMMERCE  
Technology Administration  
National Institute of Standards  
and Technology  
100 Bureau Drive  
Gaithersburg, MD 20899

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**NIST**



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U.S. DEPARTMENT OF COMMERCE  
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## ACKNOWLEDGEMENTS

**The authors wish to express their gratitude to Dr. Robert Hebner, former NIST Deputy Director, for giving the welcoming remarks at the workshop and to all the participants who offered their insights to help advance the measurements and modeling of the interface/interphase of polymeric materials and systems.**

**Last, but not least, the authors wish to thank Lori Phillips Buckland and Patrice Boulanger of NIST Conference Facilities for their help with the arrangements for the workshop.**

## Executive Summary

The interface/interphase region plays a fundamental role in a wide range of polymeric materials and systems such as polymer blends and alloys, nanocomposites, particle-filled systems, paints on plastics and metals, electronic packagings, and fiber-reinforced polymer composites. However, an understanding of the physical, chemical, mechanical and morphological properties of the interface/interphase and how it affects the performance and durability of polymeric materials and systems is still in its infancy. The National Institute of Standards and Technology, in collaboration with the Ford Motor Company, held a 2<sup>nd</sup> workshop on the **Characterization and Modeling of the Interface/Interphase of Polymeric Materials and Systems** at NIST on December 7 and 8, 1998. The objectives of the workshop were:

- 1) To present lectures on the state-of-the-art in characterization and modeling of the interface/interphase of polymeric materials and systems.
- 2) To identify industry needs related to the characterization and modeling of the interface/interphase region of polymeric materials and systems.
- 3) To draft a research agenda to address measurement and other needs of the industry as they relate to the interface/interphase region.
- 4) To discuss the feasibility of forming a consortium on characterization and modeling of the interface/interphase region of polymeric materials and systems.

Forty eight participants representing a cross section of interested parties attended the one-and-a-half-day workshop. To provide a background for working group discussions, 19 invited speakers presented lectures on the state-of-the-art in characterization and modeling of the interface/interphase of polymeric materials and systems. After the presentations, the participants were divided into two working groups, characterization and modeling, to discuss the industrial needs, and prepare recommendations for presentation at the summary session. Examples of industrial needs to which the groups drew attention in their reports were:

### Characterization:

- Reference materials.
- Low-cost, fast, real-time, on-line, large-area, high-resolution, chemical-specific characterization techniques.
- Characterization of dispersion of fillers.
- Methods for better dispersion of fillers.
- Measuring adhesion and adhesion loss.

- Better understanding of the mechanisms of molecular interactions and degradation in the polymer/substrate interface/interphase region (polymer/polymer, polymer/filler, polymer/fiber, polymer/inorganic).
- Well-controlled weathering tests.
- Characterization of phase separations.
- Characterization of surface appearance

### **Modeling:**

- Modeling of polyolefin blends
- Modeling of injection molding process.
- Modeling of surface properties.
- Modeling of multiphase transition.
- Modeling of effects of shear rate/temperature on interphase formation.
- Mechanical properties of the interphase (polymer/polymer, polymer/inorganic)

### **Role of NIST and Industry**

**Industry:** Serves as validator and provides direction vector for areas of involvement.

**NIST:** Helps identify needs and provides measurements and models.

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## 1. Introduction

Polymeric materials and systems, such as polymer blends and alloys, nanocomposites, particle-filled plastics, pigments and fillers in paints, adhesives and sealants, paints on plastics and metals, electronic packagings, and fiber-reinforced polymeric composites, are comprised of a polymer matrix, a substrate (including pigments, fillers, and fibers), and an interface/interphase between the matrix and substrate. The interface is defined as the two-dimensional boundary between the matrix and substrate, while interphase is the three-dimensional region including the interface plus a zone of finite thickness on both sides of the interface. In addition to the matrix and substrate layers, the interphase has the following characteristics: chemical, physical, and morphological properties that are different from those of the bulk materials. Further, the interphase often includes impurities, unreacted molecules, or additives. It is also affected by processing conditions, which may cause chemical reactions, species diffusion, volumetric changes and generation of stresses. The resulting interphase can be a very complex structure, which is not easily analyzed or modeled.

The interface/interphase controls many of the properties of a polymeric material and system. It may also be a weak link, in that failures of a system often initiate from the interface/interphase region. Considerable research on the characterization has been conducted to improve the strength and durability of the interface/interphase region through surface treatment or modification of the substrates. However, little progress has been made on the understanding of the morphological, physical, chemical, and mechanical properties of this region, how these properties change with external stresses and processing conditions, and how this region affects the performance and service life of a polymeric system. Since the performance, service life, and reliability of polymeric systems depend on the integrity and durability of the interface/interphase region, NIST should consider an effort to carry out research on the characterization and modeling of the interface/interphase of polymeric materials and systems. This research effort would aid the industry in reducing costs and increasing our national competitiveness.

Recognizing the importance of this subject, NIST held the 1<sup>st</sup> Workshop on the Characterization and Modeling of Polymer/Polymer Interface/Interphase region in 1997. The 1<sup>st</sup> workshop, which attracted nearly 60 participants, addressed only polymer/polymer systems. However, because the interface/interphase problems cover a wide variety of polymeric materials and systems, the industry expressed interest in expanding the scope of the workshop to include the interface/interphase of other polymeric systems such as fiber-reinforced polymer composites, particle-filled plastics, nanocomposites, and thin films. For that reason, NIST, in collaboration with Visteon, a division of the Ford Motor Company, held the 2<sup>nd</sup> Workshop on the same theme, i.e., measurement and modeling of the interface/interphase, with the focus on polymers used in association with other materials. In addition to presentations on the state-of-the-art in measurement and modeling and identification of the industry needs, the 2<sup>nd</sup> Workshop also included discussions on a research agenda and the feasibility of forming a consortium on the characterization and modeling of the interface/interphase in polymeric materials and systems.

This report summarizes the activities of the 2<sup>nd</sup> Workshop, which was held at NIST on December 7 and 8, 1998. It is hoped that the information included in this report will provide a framework for 1) planning research to address the needs of the industry related to the measurement and modeling of the interface/interphase in polymeric materials and systems, and 2) discussing the feasibility of forming a consortium on the subject.

## 2. Workshop Organization

The workshop was a collaboration between Visteon and three NIST Laboratories, namely, Building and Fire Research Laboratory, Chemical Science and Technology Laboratory, and Materials Science and Engineering Laboratory. NIST and Ford representatives defined the workshop objectives, drew up the workshop program, and drew up the invitation list of speakers and participants. The workshop program and list of invited speakers, which were publicized on the WEB and in NIST conference announcements, are given in Section 4 of this report.

Forty eight participants attended the workshop. After the opening remarks by the NIST Deputy Director, Dr. Robert Hebner, presentations were made by 19 invited speakers on the state-of-the-art in characterization and modeling of the interface/interphase of polymeric materials and systems. Abstracts of the invited presentations are given in Section 5. Following the presentations, the industry needs were discussed. After a brief introduction by the moderators, the participants were divided into two working groups, Characterization and Modeling. The working groups were instructed to address the workshop objectives and be prepared to present recommendations summarizing the industry needs. In case they wished to use them in promoting the group discussion, each working group chair person was provided with a list of topics (Appendix III) that had been suggested in advance by the organizers based on inputs from the 1<sup>st</sup> Workshop. Following the industry needs session, each group's recommendations were presented to the assembled participants in the plenary session. Finally, there was a general discussion on the feasibility of the formation of a consortium on the subject, where each participant expressed her or his view on the advantages and disadvantages of a consortium. However, no immediate action was taken on this issue, and it was recommended that a core group of companies should explore the issue.

In this report, the remaining sections present the workshop objectives, the workshop program, abstracts of the invited presentations, recommendations of the working groups, and concluding remarks. The report includes an appendix providing the advance notice, a typical invitation letter to a speaker, suggested areas for working group discussion, compilation of the recommendations by working group participants, and a list of participants.

## 3. Workshop Objectives

- To present lectures on the state-of-the-art in the characterization and modeling of the interface/interphase of polymeric materials and systems and its changes with service and processing conditions
- To identify industry needs related to the characterization and modeling of the interface/interphase.
- To draft a research agenda to address the measurements and other needs of the industry as they relate to the interface/interphase region.

- To discuss the feasibility of forming a government/industry consortium on characterization and modeling of the interface/interphase of polymeric materials and systems.

#### **4. Workshop Program**

### **2<sup>nd</sup> Workshop on Characterization and Modeling of the Interface/Interphase of Polymeric Materials and Systems**

**National Institute of Standards and Technology  
Gaithersburg, Maryland**

**December 7 and 8, 1998**

**Monday, December 7, 1998**

8:00 a.m. Registration

8:30 a.m. Welcoming Remarks, Robert Hebner, NIST Deputy Director

#### **Characterization and Modeling I: Moderator, Charles Han, NIST**

8:45 a.m. The Inter-Dependence Between Fiber-Matrix Adhesion, Interphase Formation and Processing in Polymer Composite Materials. Lawrence T. Drzal, Michigan State University.

9:15 a.m. The Accurate Prediction of Surface Properties of Polymers, Gregory Dee, DuPont Company.

9:45 a.m. Investigation of Elastomer Interfaces, Jay Dias, Exxon Company.

10:15 a.m. Break

10:45 a.m. Novel Probes of Interfaces in Biotechnology, Anne Plant, NIST; Characterization of Materials Surfaces and Interfaces with NSOM, Richard Cavanagh, NIST.

11:15 a.m. Tire: The Most Durable Man-Made Composite, Marty Cohen, The Goodyear Tire and Rubber Company.

11:45 a.m. Tailoring the Polymer/Solid Interface Using Block Copolymer Adhesion Promoters, Russ Composto, University of Pennsylvania.

12:15 p.m. Influence of Compositional Variations in Compounded Thermoplastic Olefins (TPOs) on the Physical and Mechanical Attributes of Injection Molded Plaque

Interphase Management, Rose Ryntz, Visteon Automotive Systems, The Ford Motor Company.

12:45 p.m. Lunch

**Characterization and Modeling II, Moderator: Richard Cavanagh, NIST**

1:45 p.m. Characterization of Thin Polymer Blends and Bilayers with NEXAFS Microscopy, Harald Ade, North Carolina State University.

2:15 p.m. Multilayer Polymer Optical Interference Filters, Andrew Ouderkirk, 3M Company.

2:45 p.m. Polymers on Heterogeneous Surfaces, Alamgir Karim, NIST.

3:15 p.m. Break

3:30 p.m. On the Use of Micro-FTIR and TOF-SIMS Spectroscopy to Follow the Photodegradation of Interfaces in Multi-Layer Automotive Paint Systems, T.J. Gerlock, The Ford Motor Company.

4:00 p.m. Chemical Imaging of Polymer Surface Composition and Structure, P. Treado, ChemIcon, Inc.

4:30 p.m. Trends in Computational Materials Science for Polymer Interface/Interphase, Sharon Glotzer, NIST.

5:00 p.m. Adhesion of Automotive Coatings to Thermoplastic Olefins, Ndiba Diah, Equistar Chemical Company.

**Tuesday, December 8, 1998**

**Characterization and Modeling III, Moderator: Rose Ryntz, Ford Motor Company**

8:30 a.m. Characterization of the Gloss of Polymer Films with Novel Scattering Techniques, Sanat Kumar, Penn State University.

9:00 a.m. Nanophases in Silicone-Based Polymers, Wei Chen, Dow Corning.

9:30 a.m. Quantifying the Water Layer at the Polymer/Substrate Interface with FTIR-Multiple Internal Reflection Spectroscopy, Tinh Nguyen, NIST; Characterization of Painted Plastics Interphase by AFM, Mark VanLandingham, NIST.

10:00 a.m. Break

**Discussion of Industrial Needs, Moderators: Rose Ryntz, Ford Motor Company, Bruno Fanconi, NIST; and Geoff Frohndorff, NIST**

- 10:30 a.m. Group Discussion
- 12:45 p.m. Lunch
- 1:45 p.m. Discussion of Industrial Needs and Feasibility of Establishing a Consortium on the Subject.
- 3:45 p.m. Adjourn

**5. Abstracts of Presentation**

**The Inter-Dependence Between Fiber-Matrix Adhesion, Interphase Formation and Processing in Polymer Composite Materials**

*Lawrence T. Drzal  
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Michigan State University  
East Lansing, MI*

Fiber reinforced polymer composites are examples of a structural material system that depends on fiber-matrix adhesion and the interfacial/interphaseal region for initial and long-term mechanical properties. The ability to measure fiber-matrix adhesion and to characterize the interphase has been the key to making significant advances in developing the structure-processing-property relationships in fiber reinforced composite materials. Results of research in carbon fiber/epoxy; glass fiber/vinyl ester; glass fiber/epoxy; and carbon fiber/polycarbonate interphases will be used to illustrate the material property based mathematical relationships which have been developed. Relationships between the surface chemical, energetic, and topographical characteristics of the reinforcement surface; the composition and processing dependency of fiber sizings and finishes; and the physical properties of the interfacial/interphaseal region in these systems will be presented. Areas where further research and advances in interphase characterization will be identified.

## **Investigation of Elastomer Interfaces**

*Jay Dias*  
*Exxon Company*  
*Baytown, TX*

The use of polyisobutylene based elastomers in many applications is limited by its poor interaction with fillers and its poor adhesion to other polymeric and inorganic materials. The surface and interfacial chemistry of these polymer formulations has not been investigated in detail. In addition, elastomers are used in complex vulcanized blends. The influence of each of the commonly used formulation ingredients on the resulting surface and interfacial chemistry (also the resulting adhesion) is unknown. The surface chemistry of blends containing polyisobutylene are examined in this work using valence band X-ray photoelectron spectroscopy. As expected the lower surface energy component dominates the free surface of the blend; we show that the surface of the blends are dominated by the saturated rubber component (polyisobutylene).

Our preliminary investigation of rubber - rubber interfaces is also presented. This work utilizes neutron reflectivity to probe the interface between a pair of elastomers. Thin layers of deuterated polybutadiene were laminated with a brominated copolymer of poly(isobutylene-co-4-methylstyrene). The interfacial width was found to be a function of the composition of the copolymer. The kinetics of the Interdiffusion was also a function of the film thickness. Film thickness below 20 nanometers did not undergo interdiffusion. Filler addition to thick films resulted in a dramatic reduction in the interdiffusion.

## **Novel Probes of Interfaces in Biotechnology**

*Anne Plant*  
*Biotechnology Division*  
*National Institute of Standards and Technology*  
*Gaithersburg, MD*

Surfaces are important in biotechnology applications such as implants and diagnostic devices. A biomimetic approach to surface fabrication provides the possibility of producing relatively rugged materials that resemble biological interfaces such as cell membrane surfaces. We employ self-assembling alkanethiols as components in lipid bilayer membrane mimics by addition of a second monolayer of natural lipid or cell membrane components to an alkanethiol monolayer. These biomimetic bilayers are tethered to gold films supported on silicon or glass. The planar configuration, the stabilization provided by tethering, and the association with metal make it possible to study these bilayers with a large number of surface analytical techniques. We have employed atomic force microscopy, x-ray photoelectron spectroscopy, reflection-absorption infrared spectroscopy, surface enhanced Raman spectroscopy, nonlinear optical spectroscopy, ellipsometry, surface plasmon resonance, electrochemistry, electron microscopy, and neutron

reflectivity in studies of these membranes. Applying a combination of analytical techniques allows careful assessment of the structure and function of complex cell membrane mimics.

### **Characterization of Materials Surfaces and Interfaces with NSOM**

*Richard Cavanagh  
Surface and Microanalysis Science Division  
National Institute of Standards and Technology  
Gaithersburg, MD 20899*

The ability of scanned probe techniques to characterize surfaces and interfaces shows great promise for providing insights into the structure/performance issues in polymer systems. These techniques provide exquisite topographic information, but they generally lack the capability to characterize the chemical inhomogeneity of a system. Yet knowledge of the chemical variation associated with interfaces and interphases can be central in determining the properties of a material. Consequently, there is significant interest in microscopic methods that can distinguish between different compositional domains and interfacial regions of complex systems. Near-field scanning optical microscopy (NSOM) offers a method to obtain optical contrast on a spatial scale that is an order-of-magnitude greater than that set by the diffraction limit of light. Two variants of NSOM will be presented that incorporate vibrational spectroscopy as a mode of compositional contrast – Raman NSOM and Infrared NSOM. In the Raman NSOM work, a demonstration of the near field signal is reported, and a method for calibration of the tip size is addressed. In the infrared NSOM work, preliminary performance criteria are presented and initial images of TiO<sub>2</sub> nanoparticles in an acrylic melamine matrix are discussed.

### **Tire - The Most Durable Man-Made Composite**

*Martin Cohen  
The Goodyear Tire & Rubber Company  
Akron, OH*

The foundation of tire technology is surface science spanning from molecular to macro scale. The structures and chemistries of a variety of interfaces which comprise the composite nature of a tire were reviewed, including: polymer-polymer, polymer-filler, fiber-rubber, wire-rubber, and component-component. State of the technology and future challenges in materials and analysis were described.

## **Tailoring the Polymer/Solid Interface Using Block Copolymer Adhesion Promoters,**

*Russ Composto  
University of Pennsylvania  
Philadelphia, PA*

Tailoring of the interface between a polymer and a solid substrate using block copolymer adhesion promoters has been investigated. Silicon oxide-covered Si as the substrate, P(MMA-*b*-d*B*) as the adsorption species, and PS and poly(styrene-*ran*-bromostyrene) as the polymer matrices were used for the investigation. The adsorption was studied by forward recoil spectroscopy (FRES) and neutron reflectivity, and the bonding strength between the polymer and the adhesion promoter-treated substrate was measured by a peel test. The effects of adsorption kinetics of block copolymer adhesion promoters, matrix chain length, matrix block copolymer interaction parameter, and block copolymer additives on thin film dewetting are presented. Adsorption of block copolymer on the substrate surface has been shown to greatly improve the adhesion between the polymer matrix and the substrate. Mechanism for the enhanced adhesion is discussed.

## **The Influence of Compositional Variations in Compounded Thermoplastic Olefins (TPO) on the Physical and Mechanical Attributes of Injection Molded Plaque Interphase Management**

*Rose A. Ryntz  
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Dearborn, MI 48121*

Compounded TPO continues to make inroads into automotive applications due to the excellent price/performance balance reached in this thermoplastic substrate. Thermoplastic olefin (TPO), a blend of elastomer and poly(olefin), achieves its balance of properties through the choice of compounded ingredients. The injection molding conditions through which the desired plastic part is achieved are known to also influence the attained properties of the blend. In this paper, the influence of poly(olefin), namely poly(propylene) homopolymer, and elastomer utilized in the compounded blend, in conjunction with molding properties used to produce plaques, are studied as they relate to the physical and mechanical properties of the interface/interphase achieved. Paint adhesion and friction induced paint damage resistance of coated plaques are shown to be directly related to poly(propylene) molecular weight and elastomer crystallinity. Molding conditions, mainly influenced through the shear induced injection molding process, are also correlated.



## Characterization of Thin Film Polymer Blends and Bilayers with NEXAFS Microscopy

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In many cases, polymer systems of interest are not a homogeneous or single-component system, but a blend, composite, or copolymer. Sophisticated analytical methods are required for their characterization. Amongst them is Near Edge X-ray Absorption Fine Structure (NEXAFS) microscopy. Excitations of core electrons into unoccupied molecular orbitals provide sensitivity to a wide variety of chemical moieties, including sensitivity to isomeric substitution in some cases [1,2]. This sensitivity complements and resembles that achieved in infrared (IR) spectroscopy. Although NEXAFS spectra are in most cases not as specific and “rich” as IR spectra, the spatial resolution achieved in NEXAFS microscopy is about 50 nm, and thus superior to that achieved in IR microscopy [3]. In addition, similar to IR dichroism the orientation of specific molecular orbitals can be assessed with NEXAFS [4]. The advantages of x-ray microscopy have opened up new avenues for the characterization of polymers, particularly in those cases where quantitative information about composition at high spatial resolution is required and where preferential staining in the electron microscope is limited.

Several systems have been successfully characterized with NEXAFS microscopy to date. We will discuss representative applications with an emphasis on characterizing the dynamics of and morphology formation during phase separation and dewetting in thin polymer films [5-9].

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## **Phase Separation of Polymer Blend Films: Homogeneous versus Patterned Substrates**

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Phase separation in ultrathin polymer blend films of polystyrene (dPS) / polyvinylmethylether (PVME) leads to a variety of film morphologies depending on polymer composition. For a homogeneous substrate, bicontinuous spinodal decomposition patterns form for near-critical blend films, while “mounds” and “holes” form for PVME-rich and dPS-rich off-critical mixtures, respectively. Reverse temperature jumps are performed to confirm the phase separation origin of these film structures. The influence of film thickness on the kinetics of film phase separation will be discussed. Both lower and upper critical solution temperature blends of PS/PVME and PS/PB(Polybutadiene) respectively are investigated, leading to generalized observations of phase separation in ultrathin films. Finally, the role of substrate inhomogeneity in influencing ultrathin film phase separation is considered using self-assembled monolayer (SAM) patterns in which the SAM end-group functionalization is used to direct the phase separation.

## **On the Use of Micro-FTIR and TOF-SIMS Spectroscopy to Follow the Photodegradation of Interfaces in Multi-Layer Automotive Paint Systems**

*T. J. Gerlock*

*The Ford Motor Company  
Dearborn, MI*

The Ford Research Weathering Tests are the result of 15 years of sustained research at the Ford Research Laboratory. Traditional paint weathering tests rely on measurements of physical performance; gloss loss, color changes, and time to observe cracking and peeling. While such information is valuable, it is not adequate to quickly appraise the weathering performance of modern systems, which comprise of many complex layers. The Ford Research Weathering Tests are designed to cope with these complex systems by shifting emphasis from measurements of physical property changes to measurements of chemical composition changes. These measurements are then supplemented by a direct measurement of the mechanical repercussions of chemical composition change. When the results of chemical composition and mechanical change measurements are combined, they provide a sound basis to greatly reduce the risk of introducing inferior paint systems. Tests include measurements of a) clearcoat photooxidation resistance, b) clearcoat ultraviolet light absorber (UVA) effectiveness and longevity, c) clearcoat hindered amine light stabilizer (HALS) effectiveness and longevity, and

d) the ability of a clearcoat to cope mechanically with the photooxidative degradation that invariably occurs during outdoor exposure.

This presentation provides results to illustrate the applications of micro-FTIR and TOP SIMS to measure the photodegradation in the interface region in a multilayer automotive paint systems. Unexposed multilayer paint samples and samples that had been exposed in an artificial weather device and in Florida were analyzed, and  $^{18}\text{O}$ -labeled was used to follow the photooxidation products by SIMS. The results showed that micro-FTIR and SIMS can be used to characterize and assess the relative photodegradation of individual coating layers in fully-formulated, multi-layer coating systems.

### **Chemical Imaging of Polymer Surface Composition and Structure**

*Patrick J. Treado, Ph.D.*

*ChemIcon Inc.*

*Pittsburgh, PA 15208*

Chemical imaging combines the molecular analysis power of Raman, IR or fluorescence spectroscopy with high-resolution optical imaging. Chemical imaging is used to characterize the molecular architecture of organic coatings applied to blended polymer substrates. Chemical imaging instruments have been applied as laboratory analysis tools to support polymer and coatings materials R&D. When appropriately configured, chemical imaging systems have demonstrated capability as quality monitoring platforms for use in coatings manufacturing environments. A typical application is the monitoring of the migration of volatile molecular species from surface applied coatings to the underlying bulk substrate. Species migration can be visualized rapidly, non-invasively and quantitatively. Chemical images are based on the molecular 'fingerprints' of components of interest and are typically generated without the use of dyes or intensive sample preparation. Chemical imaging is applicable to a variety of coating systems. For example, chlorinated polyolefin adhesion promoter applied to thermoplastic olefin (TPO) polymers has been studied in detail. In this presentation, chemical imaging technologies and analysis strategies, as well as general implications for characterization of interfacial molecular architecture will be discussed.

### **Adhesion of Automotive Coatings to Thermoplastic Olefins**

*Ndiba Dioh*

*Equistar Chemical Company*

*Cincinnati, OH*

Results are presented from a newly developed fracture mechanics based test method, which can be used to quantify the adhesion of coatings to Thermoplastic Olefins (TPOs). Adhesion energy values are reported for injection molded plaques and automotive fascia painted with an automotive coating system consisting of an adhesion promoter layer, a basecoat and a clearcoat. The results indicate that superior adhesion is obtained for in-reactor TPO resins with optimized

rubber morphology and dispersion. The superior adhesion of these resins is demonstrated by the cohesive failure observed between the substrate and the coating in both injection molded plaques and bumper fascia. Transmission electron micrographs (TEM) are presented which relate the enhanced paint performance to the rubber surface morphology. This morphology is characterized by a bi-modal distribution of rubber particles within the polypropylene matrix. It is demonstrated that the modifier and matrix rheological properties of these in-reactor TPO resins are responsible for the rubber surface morphology, which results in better adhesion.

### **Nanophases in Silicone Based Polymers**

*Wei Chen*

*Dow Corning Corporation  
Midland, MI 48686*

Applications of silicone polymers are often involved combinations of other materials, either as matrix matrixes or additives. The dispersion phases in such hybrid materials are of great interests. In most applications, we would like to find ways to design and control the morphologies of mixed materials. We used the concepts of rheology match and reactive mixing to control the silicone dispersion phases in a series of silicone/polyethylene blends. In a certain temperature range where the viscosities of silicone component matches with the low density polyethylene, the dispersed silicone phase reaches an average size of 0.56  $\mu\text{m}$ . After we pursued the reactive mixing route on the same blend, the silicone dispersion phase size is further down to 100-200 nm. The inter-diffusion (interface) areas are significant in the reactive blend. Using layered silicates as additives, the silicates can be well aligned in a polypropylene film and dispersed as single layer with large aspect ratios. The silicate layers typically 100 nm in length and a few nanometer in height. The TEM micrographs demonstrate the layered structure in the film. Silica fillers are often mixed into polydimethylsiloxane as the reinforcement agents. Colloidal silica fillers are used to reinforce silicone rubber. With the proper surface treatments, colloidal silica can mostly stay as primary particles in silicone rubber. We determined the morphologies of these treated colloidal silica particles using TEM and AFM.

### **Trends in Computational Materials Science for Polymer Interface/Interphase**

*Sharon Glotzer*

*Polymers Division and Center for Theoretical and Computational Materials Science  
National Institute of Standards and Technology  
Gaithersburg, MD.*

Computational Materials Science is playing an increasingly important role today in materials development and processing, and in the design of new materials and materials applications. Researchers use numerous computational techniques, from quantum molecular dynamics (MD) and quantum Monte Carlo (MC) methods which allow "first principle" calculations of electronic

structure, to classical MD and MC in which the effect of electrons is implicitly included via classical, semi-empirical pair potentials, to mesoscopic and macroscopic modeling of continuum equations and constitutive laws. The hierarchical coupling of length scales and methods is called multiscale modeling of materials, and is one of the most important trends in Computational Materials Science today.

In the field of polymers, simulations provide insight and predictions that guide experiments to control patterns at mesoscopic length scales. For example, spinodal patterns that emerge in immiscible blends have been found to be sensitive to perturbations that break the isotropy of the coarsening process, providing a way to produce diverse morphologies through the adjustment of molecular architecture and the type of perturbation. In this talk, we discuss three examples of simulations that investigate such phenomena:

(i) Phase separation of ultrathin polymer-blend films on patterned substrates, (ii) target patterns in filled polymer blends, (iii) pattern formation in liquid crystal display materials.

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## **Characterization of Interfaces and Interphases using Atomic Force Microscopy**

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Recent advances have been made on two fronts regarding the capability of the atomic force microscope (AFM) to characterize the mechanical response of polymers. Phase imaging with the AFM has emerged as a powerful technique, providing contrast enhancement of topographic features and, in some cases, revealing heterogeneity in the polymer microstructure that is not apparent from the topographic image. The enhanced contrast provided by phase images often allows for identification of different material constituents. However, while the phase changes of the oscillating probe are associated with energy dissipation between the probe tip and the sample surface, the relationship between this energy dissipation and the sample properties is not well understood. As the popularity of phase imaging has grown, the capability of the AFM to measure nanoscale indentation response of polymers has also been explored. Both techniques are ideal for the evaluation of multi-component polymer systems. For these types of materials, micro- and nano-scale properties related to the interfaces and interphases between constituents

are difficult to evaluate with current experimental techniques, and often these properties control important aspects of the material's performance.

In this presentation, the use of the AFM to characterize multi-component polymer systems is discussed. Previous work related to the development of the AFM as a nanoindentation device is briefly reviewed. This technique is then used to measure property differences between polymer samples and property variations across an interphase region in a polymer composite. The combination of phase imaging and indentation is also used to identify constituents in a polymer blend sample.

### **Quantifying the Water Layer at the Polymer/Substrate Interface with FTIR-Multiple Internal Reflection Spectroscopy**

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Quantitative molecular information on the water layer at the organic coating/substrate interface is crucial for understanding the adhesion loss of organic coating systems exposed to moist environments. A technique, based on a two-layer model derived rigorously from internal reflection theory, has been developed for quantifying in situ the water layer at the organic coating/substrate interface. In this technique, water at the coating/substrate interface is detected by the evanescent wave, which is generated by the total internal reflections within the substrate. The technique gives new insight into the processes by which water degrades the coating/substrate bonds. Experimentally, a transparent or an opaque organic coating of sufficient thickness is applied to a prism with or without a thin metallic film, which is used as the substrate. A water chamber is attached to the organic-coated specimen. After adding water to the chamber, Fourier transform infrared spectra in the multiple internal reflection mode are taken at specified time intervals without disturbing the specimens or the instrument. Water uptake in the coatings, and optical properties of the substrate, the water, and the coatings are used for quantitative analysis of water at the coating/substrate interface. Examples of clear, pigmented water-reducible, and powder coatings on several types of substrates are given to demonstrate the technique. The effects of adhesion promoter and surface salt contamination on the interfacial water layer and adhesion loss of coating systems and polymer/fiber composites will be presented. Results of water accumulation at the coating/iron interface with and without applied electrical potentials are given. Implications of the interfacial water layer on the adhesion loss of coating/substrate systems are discussed.

**\*The Accurate Prediction of Surface Properties of Polymers**

*Gregory Dee  
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**\*Multilayer Polymer Optical Interference Filters**

*Andrew Ouderkirk  
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**\*Characterization of the Gloss of Polymer Films with Novel Scattering Techniques**

*Sanat Kumar  
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University Park, PA*

\* Abstract is not available.

## **6. Working Group Recommendations**

Each of the working groups spent two hours in discussion and preparing recommendations for presentation of the industry needs at the final plenary session. A compilation of the working group recommendations is given in Appendix IV. They could be viewed as possible research areas for a consortium.

Examples of needs to which the working groups drew attention in their summary reports at the session were:

### **Characterization:**

- Reference materials.
- Low-cost, fast, real-time, on-line, large-area, high-resolution, chemical-specific characterization techniques.
- Characterization of dispersion of fillers.
- Methods for better dispersion of fillers.
- Measurements of adhesion and adhesion loss.

- Better understanding of molecular interactions and degradation mechanisms of the polymer/substrate interface/interphase (polymer/polymer, polymer/filler, polymer/fiber, polymer/inorganic)
- Well-controlled weathering tests.
- Characterization of phase separations.
- Characterization of surface appearance.

### **Modeling:**

- Modeling of polyolefin blends.
- Modeling of injection molding processes.
- Modeling of surface properties.
- Modeling of multiphase transitions.
- Modeling of shear rate/temperature gradients.
- Mechanical properties of the interphase (polymer/polymer, polymer/inorganic).

### **Roles of NIST and Industry**

**Industry** serves as validator and provides direction for areas of involvement; while **NIST** helps to identify needs and provides measurements and models.

## **7. Concluding Remarks**

Although the interface/interphase region plays a fundamental role in a wide range of polymeric materials and systems, an understanding of the physical, chemical, mechanical and morphological properties of this region and how it affects the performance and durability of polymeric materials and systems is still in its infancy. The two workshops held at NIST on the characterization and modeling of the interface/interphase of polymeric materials have identified industry needs related to this area. It is hoped that the recommendations made by a broad cross-section of knowledgeable participants will stimulate actions, which will help to provide industry with understanding of the interface/interphase region in polymeric materials and systems.



## APPENDIX I

### Advance Notice

#### **2<sup>nd</sup> Workshop on Characterization and Modeling of the Interface/Interphase of Polymeric Materials and Systems**

National Institute of Standards and Technology  
Gaithersburg, MD

Lecture Room B, Administration Building  
December 7 and 8, 1998

The interface/interphase region plays a fundamental role in a wide range of polymeric materials and systems such as polymer blends, nanocomposites, particle-filled systems, electronic packagings, fiber-reinforced polymer composites, and paints on plastics and metals. Yet, understanding of the physical, chemical, mechanical and morphological properties of the interface/interphase and how they affect the performance and durability of polymeric materials and systems is still in its infancy. The National Institute of Standards and Technology, in collaboration with the Ford Motor Company, will hold a 2<sup>nd</sup> workshop on the **Characterization and Modeling of the Interface/Interphase of Polymeric Materials and Systems** to be held at NIST on December 7 and 8, 1998. This workshop will 1) present lectures on state-of-the-art of the characterization and modeling of the interface/interphase of polymeric materials and systems, 2) identify industry needs related to the characterization and modeling of the interface/interphase region, 3) draft a research agenda to address the measurements and other needs of the industry as they relate to the interface/interphase region, and 4) discuss the feasibility of forming a consortium on the subject.

The workshop is limited to 80 attendees, and the registration will be accepted on a "first come, first serve" basis. For further information, please visit web site: <http://ciks.cbt.nist.gov>, or contact:

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## APPENDIX II

### Typical Letter of Invitation to a Speaker

September 24, 1998

Name:  
Title:  
Company  
Address

Dear ...:

The polymer/substrate interfacial region plays a fundamental role in a wide range of polymeric materials and systems. Yet understanding of the physical, chemical, mechanical, and morphological properties of this region and how they affect the performance and durability of polymeric composites is still in its infancy. The National Institute of Standards and Technology, in collaboration with the Ford Motor Company, is organizing a 2<sup>nd</sup> workshop on the "*Characterization and Modeling of the Interface/Interphase of Polymeric Materials and Systems*" to be held at NIST on December 7 and 8, 1998. The objectives of this workshop are to 1) present the state-of-the-art of the characterization and modeling of the polymer/substrate interface/interphase region and its changes in service, 2) identify industry needs relating to characterization and modeling of the interface/interphase region, 3) draft a research agenda to address the needs of industry, and 4) discuss the feasibility of forming a consortium on the subject.

In view of your research activities in this area, we would like to invite you to give a 30-minute presentation in this workshop. Your participation would greatly enhance the success of this workshop. A workshop announcement and preliminary topics to be covered are enclosed for your information. We very much hope that you will be able to accept our invitation. We look forward to hearing from you.

Sincerely,

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## APPENDIX III

### Suggested Research Areas for Discussion

Following is the list of suggested research areas for discussion at the:

#### 2<sup>nd</sup> Workshop on Characterization and Modeling of the Interface/Interphase of Polymeric Materials and Systems

- I. Effects of processing conditions and variables on morphology and properties (mechanical, physical, chemical, thermal, and optical) of polymeric materials and systems interphase.
  1. Development of theoretical tools to quantify (model) the effects.
  2. Development of measurement techniques to characterize the morphology and properties.
  3. Processing/crystallinity relationship, crystallinity/property relationship.
  4. Role of polymer functionality/tailoring on performance.
  5. Relationship between polymer architecture and properties (cost/performance).
  
- II. Effects of molecular structure on properties
  1. Functionality
  2. Architecture
  3. Microstructure
  4. Crystallinity
  
- III. Reactive Processing
  1. Characterization of the interphase
  2. Optimal functionality and performance
  3. Processing optimization and performance
  
- IV. Durability and service life prediction of polymeric materials and systems interphase
  1. Modeling
  2. Measurement
  3. Accelerated testings
  4. Mechanisms of degradation
  5. Identification of controlling factors
  
- V. Molecular interaction at the interface/interphase
  1. Measurement techniques
  2. Its role in adhesion

3. Effects on morphology and properties
  4. Modeling
  5. Its role in durability
- VI. Multi-layer adhesion
1. Measurement
  2. Modeling
  3. Processing
  4. Effects of environmental conditions
  5. Geometry of coated surface (flat/curved surface)
- VII. Rheological effects on interphase formation
1. Characterization of flow profile-boundary effects
- VIII. Kinetics and morphology spinodal/Oswald ripening/coalescence as a function of processing conditions
1. Post manufacturing effects
  2. Solvent, temperature, and induced stresses
- IX. The Role of clay and fillers on morphology and properties of filled polymers
1. Effect of polymer/particle compatibilization on morphology and properties.
- X. Development of techniques for the characterization of polymeric materials and system interphase, with particular emphasis on in situ, online, and nondestructive.
- XI. Processing in thin films/multi-component thin films
1. Surface effect
  2. Flow effect
  3. Field effect

**Applications of Polymeric Materials and Systems Requiring Understanding of Interface/Interphase**

- 1) Automotive
- 2) Solid resins/plastics
- 3) Coatings, ink and varnish
- 4) Polymer/fiber composites
- 5) Building and construction materials
- 6) Transportation
- 7) Membrane

- 8) Aerospace
- 9) Military
- 10) Electronic packagings
- 11) Food packagings
- 12) Textiles
- 13) Paper
- 14) Wood and pulping products
- 15) Pharmaceutical
- 16) Recreational industries

## APPENDIX IV

### Compilation of Working Group Recommendations

#### Characterization Group

- 1) Low cost, fast, real time, on-line, friendly, for untrained personnel tools.
- 2) Sample preparations.
- 3) Availability of different techniques.
- 4) Weathering tests.
- 5) Method for dispersion of pigments.
- 6) Characterization of dispersion.
- 7) Standards for characterization.
- 8) Reference materials.
- 9) Techniques to characterize phase separations, chemical compositions, polarity.
- 10) Tests for adhesion loss.
- 11) Chemical mapping techniques.
- 12) 3-d imaging of structures.
- 13) Chemical modifications of surfaces.
- 14) Controlling surface preparation processes.
- 15) Characterization of surface appearance.
- 16) Scaling characterization techniques.
- 17) Characterization of chemical interactions across interfaces.
- 18) Samples for inter-comparison between labs and researchers.
- 19) Data base.
- 20) Knowledge-based expert systems.

#### Modeling Group

- 1) Thermoplastic focus.
- 2) Processing as important parameter (predict process effects for new products development).
- 3) Models for predicting morphology (multi-scale, multi-time...).
- 4) Theory as the experimental tool (cross check model vs results).
- 5) Utilization modeling as another characterization technique.
- 6) Can models be sold to industry (cut down the development time, appreciate limitations, prove realistic capability, change mindset, etc).
- 7) 1<sup>st</sup> principle simulation of material behavior vs mathematical description of data set.
- 8) Prediction vs empirical.
- 9) Fast, quantitative, well-defined, flexible, meet industry needs (materials properties, rheology of blends, correlate with industry results)
- 10) Define requirements models need (parameters to be included, parameters need to be quantified ...).
- 11) Modelers need to interact with designers/engineers.
- 12) Vision 2020: fundamental physics implemented from common platform.

## **Summary of Industry Needs**

- Reference materials.
- Low cost, fast, real time, on-line, large area, high resolution, chemical specific, and 3-d imaging techniques.
- Knowledge-based systems.
- Dispersion of pigments and fillers.
- Methods for better dispersion of pigments and fillers.
- Durability - characterization of adhesion loss and understanding mechanisms
  - Polymer-flat substrate, polymer-filler.
  - Easy to understand weathering tests.
- Characterization of interface interactions, phase separations, and surface appearance
- Modeling of polyolefin blends, injection molding process, surface properties, multiphase transition, shear rate/temperature gradient, and mechanical properties.
- Modeling of the interphase (polymer-polymer, polymer-inorganic).

## **Roles of Industry and NIST**

Industry serves as validator and provides direction vector for areas of involvement.

NIST identifies needs and provides measurements and models.

## APPENDIX V

### List of Participants

#### 2nd Workshop on Characterization and Modeling of the Interface/Interphase of Polymeric Materials and Systems

December 7-8, 1998

National Institute of Standards and Technology, Gaithersburg, MD

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