NIST/Industry Polymer Surface/Interface Consortium Meeting

Oct 24-25, 2017

NIST, Gaithersburg, MD

Conference Room: B205-9/226

Oct 24, 2017, Tuesday

Industrial Practice and Scientific Exchange (Invited presentations)

8:30- 8:45a	Social networking
8:45- 9:00a	**Welcome** Deputy Division Chief – Dr. Ken Snyder ***Materials and Structural Systems Division***, **EL/NIST**
9:00- 9:40a	**Mapping Viscoelastic and Plastic Properties of Polymers and Polymer-Nanotube Composites** Dr. Robert Cook, ***Materials Measurement Science Division***, **MML/NIST**
9:40 – 10:20a	**Stone Chip Performance of Automotive Paint Systems** Dr. Mark Nichols, ***Ford Motor Company***
10:20 – 10:40a	Break
10:40 – 11:20a	**Combination of indentation and scratch testing to characterize polymer films** Dr. Pierre Morel, ***Anton Paar***
11:20– 11:50a	**Visualization of damage in a single fiber epoxy composite using a fluorescent molecular probe** Dr. Jeremiah Woodcock, ***Materials Science Engineering Division MML***
12:00- 1:00p	Lunch at NIST Cafeteria
1:10 – 2:20p	**Tour: National Fire Research Laboratory, Bldg 205/EL** Dr. Matt Hoehler, ***Fire Research Division, EL/NIST***
2:30- 3:10p	**Non-linear optical Lab Tour, F112/Bldg 217/ MML** Dr. Ryan Beams***, Materials Measurement Science Division/MML***
3:10 – 3:30p	Break
3:30 – 4:00p	***In Situ* Nanoscale Topographic Measurements at the Solid-Liquid Interface using Digital Holographic Microscopy** Dr. Alex Brand, ***EL/NIST***
4:00- 4:30p	**Fluorescence imaging analysis and microscale characterization of depth-dependent degradation in photovoltaic laminates** Dr. Yadong Lyu, ***EL/NIST***

Mapping Viscoelastic and Plastic Properties of Polymers and Polymer-Nanotube Composites

Dr. Robert F. Cook
Materials Measurement Science Division, National Institute of Standards and Technology, Gaithersburg, MD 20899, USA

Abstract: An instrumented indentation method is developed for generating maps of time-dependent viscoelastic and time-independent plastic properties of polymeric materials. The method is based on a pyramidal indentation model consisting of two quadratic viscoelastic Kelvin-like elements and a quadratic plastic element in series. Closed-form solutions for indentation displacement under constant load and constant loading-rate are developed and used to determine and validate material properties. Model parameters are determined by point measurements on common monolithic polymers. Mapping is demonstrated on an epoxy-ceramic interface and on two composite materials consisting of epoxy matrices containing multi-wall carbon nanotubes. A fast viscoelastic deformation process in the epoxy was unaffected by the inclusion of the nanotubes, whereas a slow viscoelastic process was significantly impeded, as was the plastic deformation. Mapping revealed considerable spatial heterogeneity in the slow viscoelastic and plastic responses in the composites, particularly in the material with a greater fraction of nanotubes.

Bio: Dr. Robert F. Cook is a NIST Fellow at the National Institute of Standards and Technology (NIST), Gaithersburg, Maryland, USA. He received a B.Sc.(Hons I) in physics from Monash University, Melbourne, Australia in 1981 and a Ph.D. in physics from the University of New South Wales, Sydney, Australia in 1986, spending time as a Guest Researcher at NIST (then known as the National Bureau of Standards) in 1982 and 1984. The author of over 170 peer-reviewed publications and 14 patents, his research interests center on mechanics and mechanical properties of materials, especially brittle fracture. Recent research has focused on small-scale stress and strain mapping.

Stone Chip Performance of Automotive Paint Systems

Tony Misovski and Mark Nichols; Ford Motor Company
Dearborn, MI USA

Abstract: A single projectile impactor was designed to quantify the stone chip resistance of automotive paint systems. The damage mechanisms during stone impact are not well understood, as they are a complex function of projectile geometry and speed, paint system constitutive properties, impact angle, and temperature. High speed photography and non-contacting profilometry were used to quantify the volume of material removed during chipping and the details of the removal process. Impactor speed, paint system chemistry/process, basecoat color, and impactor angle were all shown to significantly affect the amount of material removed during impact. The potential sources of energy dissipation as well as potential mechanisms to improve stone chip performance will be discussed.

Bio: Dr. Mark Nichols received his B.S. from the University of Michigan in 1987, his M.S. from the University of Illinois in 1989, and his Ph.D. from the University of Michigan in 1992. All his degrees are in Materials Science and Engineering. Dr. Nichols joined Ford Research Laboratory in 1992 to work on the durability of elastomers. His current research is focused on automotive coatings. Topics of particular interest include: coating weatherability, corrosion, the fracture behavior of coatings and films, color science, and functional surfaces. He is currently a Technical Leader and heads the Paint and Corrosion Research group in Ford's Materials and Manufacturing Department. In addition, Dr. Nichols is the Editor-in-Chief of the Journal of Coatings Technology and Research. Dr. Nichols is the co-author of the books; *Mechanical Properties of Coatings, 2nd ed.*, and *Organic Coatings: Science and Technology, 4th ed.*; has published over 65 peer-reviewed research papers; and holds 9 U.S. patents. Dr. Nichols was a 2008 recipient of the Henry Ford Technology Award for his role in the implementation of a 3-wet paint process at Ford.

Combination of indentation and scratch testing to characterize polymer films

Dr. Pierre Morel, Anton Paar USA

Abstract: A combined approach using Instrumented indentation and Scratch testing was proposed to characterize polymer films and specifically automotive paints. The instrumented indentation yields the viscoelastic properties of the materials while the scratch testing assesses their resistance to surface damage. This combined technique allows a more quantitative and repeatable assessment of the paints.

Bio: Dr. Pierre Morel is the technical sales director for the indentation / scratch / Tribology and AFM instrument of Anton Paar USA. Using 15 years of experience in material testing, he directs a team of engineers supporting and advising customers testing materials at the nano and micro scale. He received a master degree in mechanical engineering from ENISE, and a second master's degree in materials science from the University of Tennesse Knoxville under the guidance of Pfr. George Pharr.

Visualization of damage in a single fiber epoxy composite using a fluorescent molecular probe

Dr. Jeremiah Woodcock, ***Materials Science Engineering Division MML***

Abstract: Exploitation of the photo-physical properties of fluorescent molecules, and advanced optical microscopy techniques has enabled important advancements in the medical and biological communities. This is due to the sensitivity of the spectral properties of fluorescent molecules to the chemical environment, and to the recent advances in instrumentation. Here we have developed a powerful new measurement platform for the study of localized phenomena in materials. This measurement approach has been applied to the single-fiber fragmentation test (SFTT), a conventional composite materials test, which is used to determine the interfacial shear strength (IFSS) between the fiber and matrix. In this work, the observation of damage, recorded by a mechanically-responsive dye (mechanophore, MP), in the SFFT of an epoxy composite will be presented. The SFFT sample was deformed under uniaxial, stepwise quasi-static strain

conditions that favored fiber fragmentation. Imaging, using two-photon fluorescence lifetime microscopy, revealed regions where the MP experienced significant mechanical activation. The images from regions of higher activation and shorter fluorescence lifetime are interpreted as sites of high energy transfer from the fiber to the matrix during fiber fragmentation. These results dramatically change the interpretation of the SFFT/IFSS measurements. We propose that the IFSS measured using the SFFT is not of a pristine composite, but the IFSS of a damaged, or fatigued, composite. Therefore, this method should be useful for developing damage tolerant composites.

Bio: Dr. Jeremiah Woodcock is a research chemist in the Functional Polymers group headed by Chris Soles, and he works under Jeffrey Gilman in the composites project. He received his B.S. from Middle Tennessee State University where he began his studies of fluorescent molecules under the guidance of Andrienne Friedli. He obtained his Ph. D. from the University of Tennessee Knoxville under Bin Zhao with a focus in stimuli response and polymer design/synthesis. His current work involves the development of stimuli responsive fluorescent probes for the characterization of composite materials. Employment of this technique enables regionally specific visualization of damage.

In Situ Nanoscale Topographic Measurements at the Solid-Liquid Interface using Digital Holographic Microscopy

Dr. Alexander Brand, Materials and Structural Systems Division, EL

Abstract: A novel technique known as digital holographic microscopy (DHM) can be utilized for fast, *in situ* micro- and nanoscale characterization of surfaces. Three-dimensional surface topography data can be collected at tens of frames per second with submicron lateral resolution and potentially subnanometer vertical resolution. This presentation will cover a brief overview of DHM and how it is being applied to innovate the study of rate kinetics for mineralogical systems by monitoring nanoscale surface topography evolution during dissolution. The findings strongly suggest that the dissolution rate is spatially and temporally heterogeneous, owing to intrinsic variability in crystal surfaces. The presentation will conclude with an overview of the versatility of reflection DHM and how it can be applied to other engineering and scientific research problems, including geochemistry, particle and cell tracking, steel corrosion, and static and dynamic surface metrology.

Bio: Alexander S. Brand, Ph.D., is in the Materials and Structural Systems Division of the Engineering Laboratory at NIST, where he started in January 2016 as a National Research Council Postdoctoral Research Associate. He received his Ph.D. in civil engineering from the University of Illinois at Urbana-Champaign in December 2015. His research interests focus on the materials science, microstructure development, and reaction kinetics of cementitious materials.

Fluorescence imaging analysis and microscale characterization of depth-dependent degradation in photovoltaic laminates

Dr. Yadong Lyu

Abstract: Depth-dependent degradation of packaging materials for photovoltaic (PV) applications has serious implications for system performance, however, it is still poorly understood. In this work fluorescence imaging and other microscale characterization techniques are utilized to visualize the degradation depth-profile of aged glass/encapsulant/backsheet laminates, a model system for PV. Samples were weathered at 85°C and 0% relative humidity (RH) under different ultraviolet (UV) light intensities and wavelengths for 3840 h. In cross-sectional fluorescence imaging a non-uniform distribution of degradation species is observed across the thickness for the ethylene vinyl acetate (EVA) encapsulant for all exposure conditions. Changes in the depth-profile of optical (yellowness index), chemical (oxidation, UV absorber (UVA) concentration), mechanical (Derjaguin-Muller-Toporov (DMT) modulus), and thermal (melting enthalpy) properties of the EVA encapsulant are correlated to the fluorescence profiles. An increase in the yellowness index and oxidation, and a decrease in the modulus and UVA concentration of the exposure side of the EVA encapsulant after aging are identified. In the backsheet a degradation gradient in the EVA middle layer is found for samples aged under different light intensities. In addition, the two adhesive layers of the backsheet exhibit a strong fluorescence emission, indicating the degradation of these adhesives and potentially an increased propensity for delamination within backsheet layers. These results demonstrate that fluorescence imaging, as a non-invasive, spatially resolved and sensitive method, enhances the ability to understand the reliability of each layer in multicomponent and multilayer PV systems.

Bio: Dr. Yadong Lyu graduated from Sichuan University with a bachelor’s degree of polymer science and engineering in 2010. Then he began his doctoral studies at the same institute under the guidance of Prof. Guangxian Li working on the aging mechanism investigation and lifetime prediction of polyolefin materials under multiple weathering factors. During the last year of his PhD study, he worked at NIST as a visiting student under the supervision of Dr. Xiaohong Gu. His work involved the multiscale characterization and durability evaluation of polymeric materials for photovoltaic applications. After being awarded his PhD degree in 2016, he continued his work at NIST in the same group as a guest researcher.