POLYMERS DIVISION

Measurement Facilities: NIST Combinatorial Methods Center

A key part of the NIST Combinatorial Methods Center (NCMC) is a set of facilities dedicated to the design and fabrication of polymer materials combinatorial libraries, and for developing and demonstrating high-throughput measurements of polymer materials structure and performance. In addition to being the central test bed for the Division's Combinatorial Methods Group, these facilities help to advance NIST programs in biomaterials, nanomaterials, electronic materials, and polymer processing. Moreover, these labs serve as an essential demonstration and technology transfer facility through which industry part-ners and collaborators from academia and govern-ment can evaluate first-hand, NCMC methodologies and devices for use in their research.

Polymer Film Library Fabrication and Characterization

The central NCMC facilities house a suite of devices for creating gradient combinatorial libraries that systematically explore factors that govern the structure and behavior of polymer coatings and thin films. The NIST Flow Coater generates film libraries that gradually increase in their thickness. This instrument is capable of creating well-behaved libraries that span 10 nm to a μ m or more in film thickness. These libraries are characterized via automated interferometry and ellipsometry instrumentation in the same facility. Another device, pictured below, can generate a variety of libraries by producing a gradient in light exposure. One implementation of this instrument can create substrates that systematically vary in their surface energy. Characterized via an automated contact angle goniometer, such libraries are a key



Device for creating gradients in light exposure

tool for examining the role of substrate hydrophobicity on film performance measures such as stability. This same instrument can create gradients in photopolymerization, UV-aging and other quantities related to light-exposure. Gradient hot stages in the facility enable high-throughput analysis of temperature processing, and provide a route to gradient polymer phase diagrams. The micro- and nano-structure of film libraries created in these facilities can be rapidly characterized through two automated optical microscopes, and a large-platform atomic force microscope.





Newest NCMC library fabrication tools

Recently, devices for creating libraries of grafted copolymers have been added to the NCMC facility. As shown in the left-hand image above, this instrumentation delivers graded combinations of monomers to an initiator-functionalized substrate via a microfluidic channel. The resulting library of surface grafted polymers can exhibit systematic variations in polymer molecular weight, composition or architecture. In addition, this year we commissioned fabrication of a new instrument capable of depositing libraries of viscous polymer mixtures. Pictured above on the right, this direct-write instrument incorporates a miniature mixing rod in the deposition head, allowing delivery of discrete and continuous film libraries of up to three input fluids. In coming months, the device will be optimized for creating arrays of multi-component polymer specimens for advanced coatings applications.



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High-Throughput Mechanical Testing

Housing instruments developed in the Division's Mechanics of Complex Interfaces Project, a second NCMC facility focuses on the mechanical performance of polymer materials. A keystone set of devices supports the NISTdeveloped buckling metrology, which harnesses wrinkling instabilities to quantify the mechanical properties of thin film libraries. This equipment includes precision strain stages, environmental control chambers, and a static light scattering instrument to provide rapid, essential, measurements of the modulus of thin films.



Lens contact adhesion instrument

Two pieces of equipment in this facility can accomplish highthroughput measures of surface and inter-facial adhesion in polymeric systems. The NCMC lens contact adhesion instrument, pictured above, uses the Johnson-Kendall-Roberts (JKR) approach to measure the weak adhesion inherent to biosystems and pressure sensitive adhesives. This year, the in-strument was modified to accomplish measurements in the aqueous environments relevant to cell adhesion and marine coatings, and a positionable probe enables systematic measurements across gradient libraries. For strongly adhering systems, such as structural adhesives, electronics packaging, and coat-ings materials, the NCMC has developed combinato-rial edge lift-off testing instrumentation capable of parallel delamination measurements across library arrays. This device drives a pre-formed array of flaws at the adhesive-substrate interface, and monitors this crack growth in parallel across a library. This instrument is being optimized to examine the adhesion between dissimilar materials inherent to application systems such as flip-chip epoxies and aerospace coatings.

Microfluidic Device Prototyping and Integration

The NCMC Polymer Formulations Project produces microfluidic technologies appropriate for the high-throughput analysis of the polymer solutions and mixtures central to a range of technologies. Central to this effort are facilities for prototyping microfluidic devices that hold up to the rigors of organic solvents, polymerization reactions, and higher temperatures. Key infrastructure includes the light sources and other equipment necessary for a NIST-developed process for fabricating microfluidic devices from thiolene, a UV patternable optical adhesive that exhibits enhanced solvent resistance. In addition, machining tools enable the production of metal microfluidic de-vices that can withstand both solvents and high-temperatures.



Metal microfluidic device fitted with DLS probe

For in-situ measurements on microfluidic devices, this facility has a suite of fiber-based optical probes for assessing fluid structure and chemistry. Raman and near-IR spectroscopy probes enable chemical analy-sis of a wide range of fluid specimens, and the speed to gauge composition in real time. In addition, NIST-developed microfluidic Dynamic Light Scattering (DLS) probes allow measurements of nanoscopic polymer structures such as micelles and latexes, and fluid dispersed particles.

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