High-Throughput Measurements of Polymer Adhesion and Mechanical Properties

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INTRODUCTION

Polymer adhesion is important to numerous technologies including electronic packaging, coatings and paints, biomedical implants, and pressure-sensitive adhesives. The challenge is to understand the fundamental driving forces for the development of adhesive strength at polymer - polymer, metal, ceramic, and biomaterial interfaces in multivariable environments. Current methods for characterizing adhesion with combinatorial methodologies are challenged by both experimental geometry and throughput issues. In response to this, our goal is to develop a methodology for quantitatively measuring the adhesive strength of polymer interfaces in a combinatorial manner. Likewise, there is a need for a simple, robust, and flexible measurement technique for the mechanical properties of thin polymer films. This combinatorial technique would benefit the coating, semiconductor, and optical adhesives industry. Conventional techniques to measure the bulk mechanical properties of coatings utilize dynamic mechanical analysis, a universal testing machine, or a depth sensing indentation instrument to measure Young's modulus. These techniques are not easily extendable to thin (thickness < 50 μm) or combinatorial samples. We have developed a combinatorial method for the measurement of the tensile modulus of thin films. This technique quickly provides quantitative information on a variety of sample classes with minimal sample preparation.

LIBRARY DESIGN

The combinatorial adhesion libraries consist of an array of microlenses with a well-defined geometry in contact with a flat substrate[1] (see Figure 1). The multi-lens combinatorial adhesion test (MCAT) method is based on the adhesion theory described by Johnson, Kendall, and Roberts[2] (JKR). The JKR theory relates the contact radius between the lens and substrate to applied load as a function of the system modulus, work of adhesion, and lens radius of curvature. For this technique, each lens represents a single JKR adhesion test and, when tested against a gradient library, constitutes an adhesion test at a unique set of material parameters. The size of the lens array ranges from several hundred to over a thousand lenses. The quantity of data collected during a high-throughput adhesion measurement is very large or difficult to handle and requires an automated approach to data collection and analysis. The key research efforts in our laboratory have focused on the design and preparation of the microlens combinatorial libraries, the instrumentation for controlling contact and separation, and the development of software to automate data collection and analysis.
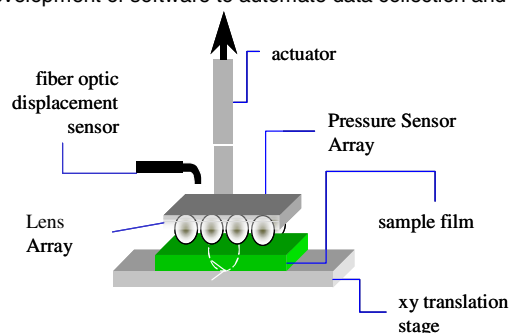

Figure 1. The multilens combinatorial adhesion test (MCAT) method.

We have also developed a high-throughput methodology to measure the modulus of films ranging in thickness from nanometers to several microns and with moduli ranging from MPa to GPa.[3] This technique relies upon a periodic strain-induced buckling instability (buckling wavelength, λ =1 μm to 10 μm for a 100 nm thick film) due to a mismatch of the moduli of a relatively stiff polymer coating on a soft silicone sheet. Knowledge of the thickness of the polymer coating and modulus of the silicone layer underneath allows determination of the specimen modulus. If gradient specimens are employed, multiple independent measurements can be obtained in a high throughput manner.

RESULTS

MCAT provides quantitative information concerning the contact radius, load, and lens displacement required to both unite and separate the lens array from the substrate. The work of adhesion required to produce the lens-substrate interface is determined from loading data (Figure 2), while the unloading data provides information on adhesion hysteresis created by cross-contact bonding and viscous loss. The adhesion data collected during testing is used to create adhesion maps across the combinatorial library. Currently, this technique is being evaluated by measuring adhesion across polymer films possessing both gradients in curing temperature and surface energy. The combinatorial measurements are compared to traditional single lens experiments to determine what effect experimental variables such as lens spacing, size, and array-sample misalignment have on adhesion measurement results.
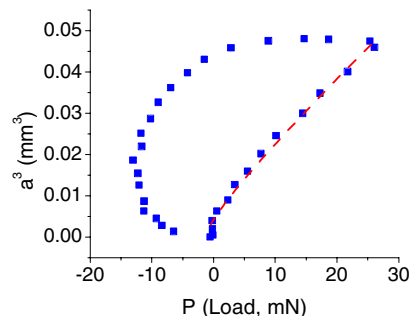

Figure 2. Plot of the cubed contact radius against load. The dashed line represents the fit of the loading data to the JKR equation. The contact area and load measurement uncertainty are ± 20 μm and ± 1 mN, respectively.

Unstrained samples for SIEBIMM were clear, however, upon application of a threshold strain, a buckling abruptly developed that was invariant upon increasing strain. Over a thickness range of 70 nm to 140 nm, the wrinkling wavelength changed proportionately from 3.5 μm to 6.8 μm. The modulus of the polystyrene film was calculated from the dominant wavelength, the measured modulus 3.2 GPa ± 0.1 GPa was independent of both film thickness and strain, and comparable to reported value of 3.3 GPa.

Currently, this technique is being extended to films with more complex structure. Nanoporous films present a heterogeneous structure that could affect the wavelength and amplitude of the buckling instability. The mechanical properties of these film vary significantly as the porosity increases. The ability of SIEBIMM to measure the modulus of heterogeneous films will be presented.

For more details on these and other thin film combinatorial measurements, visit www.nist.gov/combi

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