ESTIMATION OF THE INTERFACIAL ADHESION STRENGTH IN COMPOSITIONAL LIBRARIES OF EPOXY FILMS*

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Introduction

At the NIST Combinatorial Methods Center (NCMC)[1], we have designed, developed, and demonstrated a combinatorial approach to the edge delamination test[2,3] to characterize the adhesion of thin polymer films.[4,5] This test is based on fracture of a film/substrate interface possessing an initial interfacial crack at a stress-free edge. To fabricate a specimen for the combinatorial edge delamination test, the film/substrate bi-layer is diced into rectangles approximately (1 to 2) cm^2 using a wafer saw or similar technique (see Figure 1). The dicing process initiates small defects (cracks) between the film and substrate. Upon cooling the sample, crack extension (debonding) is driven by stresses generated by the CTE mismatch between the two materials at the interface. When the internal stress equals the interfacial fracture energy, the adhesion at the interface fails and the crack propagates from the edge of the sample towards the center. This metrology provides a rapid screen for interfacial adhesion of a wide variety of materials systems, particularly glassy and thermoset polymeric materials.

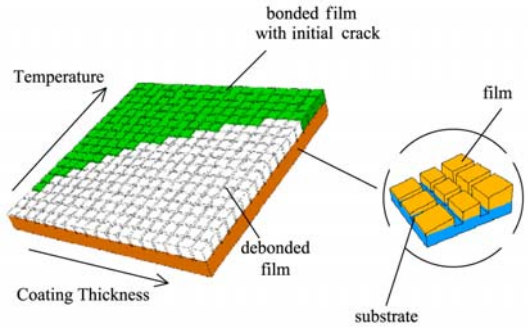

Figure 1. Schematic of the combinatorial edge lift-off test for probing interfacial adhesion strength.

Previous research on the combinatorial edge delamination test focused on applying gradients in thickness of the film, surface energy of the substrate, and temperature.[4,5,6] There would be considerable value in incorporating compositional gradients into this measurement platform. Optimization of material properties of a multi-component material requires time-consuming formulation and exhaustive testing. High-throughput screening offers rapid assessment of new candidate materials and can define appropriate operating windows and tolerances, based on new material suppliers, new formulations, and/or changes in processing conditions. Therefore, we are pursuing methods for depositing compositional gradients of viscous formulations in appropriate library geometries for the combinatorial edge delamination test.

Experimental†

Compositional gradients of an epoxy resin are generated using the meter/mix/dispense system shown in Figure 2. This deposition system consists of two computer-controlled syringe pumps, a static mixer, and an *x-y* translation stage for placement of the epoxy mixture on a substrate. Epoxy resin and catalyst are pre-mixed and loaded into one syringe pump, while the curing agent was loaded into a second syringe pump. A LabView interface is used to send commands to the syringe pumps. By varying the ratio of pumping speeds, the composition of the stream can be tuned either in a discrete fashion or continuously. In all examples discussed here, discrete gradients in composition are generated on copper substrates using various protocols for defining the array size and spacing.

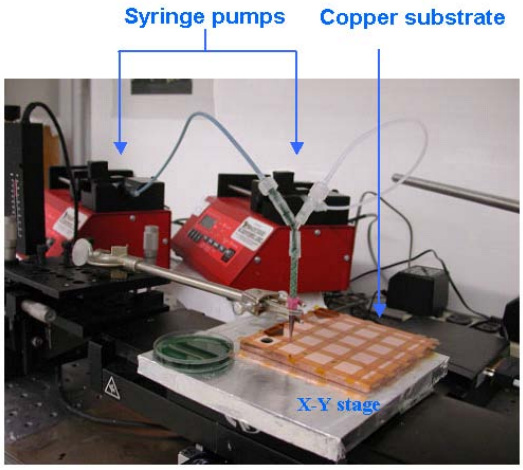

Figure 2. Deposition system for metering, mixing, and deposition of composition gradients of viscous polymer formulations.

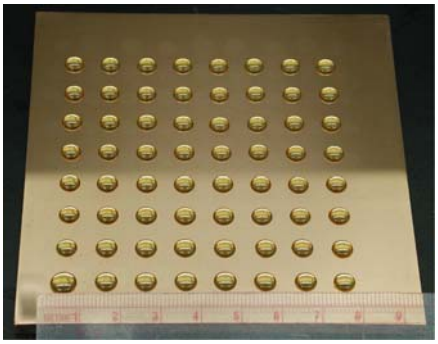

Figure 3. Array of model resin droplets on copper deposited using the deposition system shown in Figure 2.

Figure 3 shows an example of a discrete gradient library for compositional gradients of a model resin system. The accuracy and repeatability of the deposition system was checked via gravimetrics. A lab balance was placed under the substrate being coated, and the accumulated weight of the deposited droplets was recorded. As shown in Figure 4, the deposition system reproducibly meters out the same amount of material in each drop.

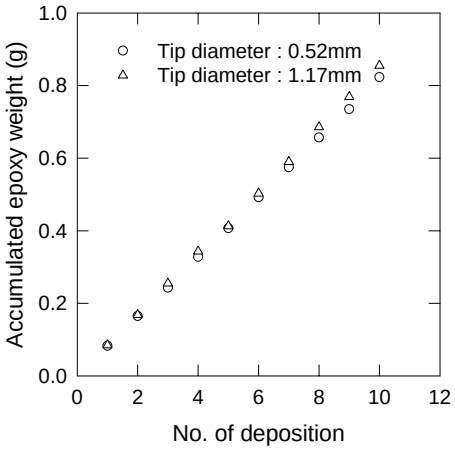

Figure 4. Accumulated weight of epoxy as a function of the number of drops deposited on the substrate.

After deposition, the epoxy is cured at 170 °C for 2 hr. In the droplet geometry, it is critical that the curing oven be level to minimize the flow or smearing of the epoxy droplets during curing. Figure 5 shows the droplet array after curing. The composition of each droplet changes gradually from the bottom right corner to the upper left corner, as labeled in the figure. Currently, we are pursuing high-throughput spectroscopic methods for verifying the composition and homogeneity of each droplet in the array.

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† Equipment and instruments or materials are identified in the paper in order to adequately specify the experimental details. Such identification does not imply recommendation by NIST, nor does it imply the materials are necessarily the best available for the purpose.

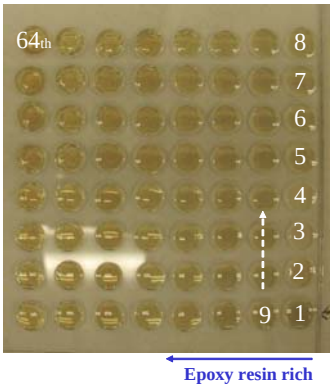

Figure 5. Array of epoxy droplets after curing at 170 °C for 2 hr.

1. http://www.nist.gov/combi
2. Farris, R. J., Bauer, C. L. Bauer, J. of Adhesion **26** (1988) 293.
3. Shaffer, E. O., McGarry, F. J., Hoang, L. *Polym. Eng. Sci.* **36**, 2375 (1996).
4. Chiang, M. Y. M., Wu, W. L., He, J. M. & Amis, E. J. *Thin Solid Films* **437**, 197 (2003).
5. Chiang, M. Y. M., Song, R., Crosby, A. J., Karim, A., Chiang, C. K., & Amis, E. J. *Thin Solid Films* **476**, 379 (2005).
6. Song, R., Chiang, M. Y. M., Crosby, A. J., Karim, A., Amis, E. J. *Polymer* **46**, 1643 (2005).