

# HIGH THROUGHPUT ADHESION MEASUREMENTS USING A MODIFIED EDGE LIFT-OFF TEST

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## Introduction

The strength of an adhesively bonded joint depends on a host of factors such as surface treatment, roughness, materials properties, cure conditions and environmental variables. Therefore testing of adhesion within this large parameter space can be very time-consuming and expensive. The ability to screen candidate materials quickly remains a measurement challenge for coating manufacturers to meet rigorous adhesion requirements. It would be attractive to perform simultaneous measurements of interfacial adhesion on samples or libraries having discrete or continuous changes in two or more of these controlling factors<sup>1</sup>.

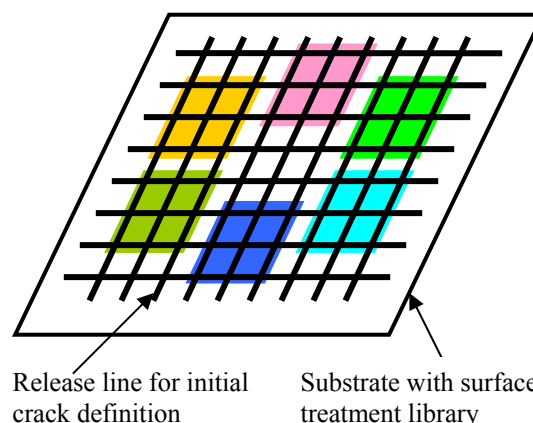
The modified edge lift-off test (mELT) is commonly used in the electronic packaging industry to evaluate the adhesion of interfaces with multilayer structures<sup>2-8</sup>. This test requires a rectangular coupon of a defined substrate material to be coated with a thin film of interest. A thick polymer backing layer is coated onto the test film as a stress-generating layer. The sample is subjected to thermal quenching which leads to delamination events due to the thermal expansion coefficient mismatch between the polymer and substrate. Previous work at the National Institute of Standards and Technology<sup>9-11</sup> demonstrated a combinatorial approach to the edge lift-off test, allowing the simultaneous interfacial adhesion measurements to be performed on a single substrate possessing a combinatorial library on its surface. In the present work, we will discuss our recent developments in library design and fabrication for adhesion testing via mELT. In particular, we demonstrate that this test can be used as a high-throughput tool to examine the effect of composition and processing conditions of adhesion promoters on adhesion strength for polymer/inorganic interfaces.

## Experimental<sup>12</sup>

**Materials:** The model epoxy system used in this work consisted of EPON 828 epoxy resin (Hexion) and An-camine AEP (Air Products) curing agent. The resin and curing agent (100:23 ratio by mass) were mixed thoroughly. Then, the mixture was degassed by centrifugation and spin-coated onto different substrates. The substrates used in this study include borosilicate glass, aluminum, and copper. All substrates were rinsed in an acetone bath and dried with nitrogen. The copper substrate was immersed in a 1 % by mass sulfuric acid solution for 30 s and

then rinsed with deionized water. After the cleaning process, the substrate was placed into an ultraviolet-ozone cleaner for 20 min.

**Library Fabrication:** A discrete compositional gradient of adhesion promoter was generated on substrates by the following protocol. Solutions of glycidoxypropyltrimethoxysilane (GPS) and aluminum sec-butoxide ( $\text{Al}(\text{O}^i\text{Bu})_3$ ) in ethanol/water (1:1) (1 % by mass of GPS and  $\text{Al}(\text{O}^i\text{Bu})_3$  combined) with different GPS/ $\text{Al}(\text{O}^i\text{Bu})_3$  ratios were prepared. These solutions were then deposited onto a single 100 mm  $\times$  100 mm substrate at different locations using a NIST-built flow-coater<sup>13</sup>. The solvent evaporated rapidly leading to uniform films of adhesion promoter with approximately 10 nm in thickness. The library of film thickness was fabricated in a similar fashion by depositing solutions of different concentrations.



**Figure 1.** mELT sample preparation using a release layer approach to generate defined pre-cracks prior to testing.

After applying the adhesion promoter, a cross-hatched pattern of a 10 nm to 20 nm thick release layer is deposited through a mask as shown in Fig. 1. The epoxy was then spin-coated onto the substrate and cured at 120 °C for 2 h. The resultant epoxy films had film thickness of 150  $\mu\text{m}$  to 500  $\mu\text{m}$ . Finally, the epoxy coated sample was diced into 10 mm squares along the middle of the release layer strips using a precision wafer dicing saw. The release layer served as pre-crack of the individual square samples. An alternative way of introducing pre-cracks was also tested by contact printing, where a cross-hatched PDMS stamp inked with octadecyltrichlorosilane was pressed into contact with the substrate.

**Testing:** The specimens with libraries of surface treatment were placed into a cryogenic temperature chamber to drive the debonding process by decreasing the temperature. The temperature of the cryogenic chamber was controlled by the flow rate of liquid nitrogen through the chamber. Debonding events and corresponding temperature were recorded by a digital camera.

## Results and Discussion

Our results showed that the pre-crack definition is critical in controlling the locus of failure so that reliable measurement can be made. Data scattering reduced drastically when a well-defined pre-crack is introduced. Our preliminary results indicate that the adhesion strength of the epoxy/metal interfaces depends on the GPS/Al(O<sup>s</sup>Bu)<sub>3</sub> ratio of the adhesion promoter. Presumably the crosslinking density or the modulus of the adhesion promoting layer would change with the GPS/Al(O<sup>s</sup>Bu)<sub>3</sub> ratio. Previous results have shown that Al(O<sup>s</sup>Bu)<sub>3</sub> has a catalytic effect on the crosslinking of the GPS silane<sup>14</sup>. XPS analysis of the locus of failure will reveal some details of the adhesion mechanisms at the polymer/metal interfaces. We are also pursuing to look at the effect of drying conditions of the adhesion promoter. For example, a library of drying time can be easily constructed by depositing the solution at different times. This will be the focus of future research.

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