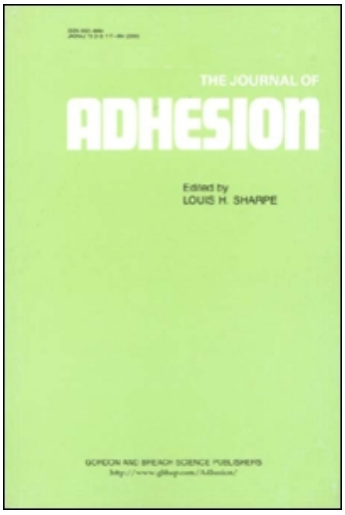

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Critical and Subcritical Adhesion Measurements of a Model Epoxy Coating Exposed to Moisture Using the Shaft-Loaded Blister Test

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The shaft-loaded blister test (SLBT) was used to investigate the adhesion between a model epoxy coating and a silicon oxide surface as a function of relative humidity. Critical and subcritical strain energy release rates were measured using specimens that incorporate reinforcing layers of Kapton® film. A simplified procedure that eliminates the need for video imaging to measure the blister radius and fracture energy was developed. A critical relative humidity level for adhesion loss was observed, in agreement with measurements that have been made previously in a number of polymeric systems. The SLBT was confirmed to be particularly attractive for fracture energy measurements on thin, strongly adhered coatings and films which otherwise tend to be problematic.

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Keywords: Shaft-loaded blister test; Water-assisted subcritical crack growth; Stress corrosion cracking; Epoxies; Moisture; Durability; Microelectronics; Critical relative humidity; Wedge test; Coatings; Thin film

INTRODUCTION

Water is often regarded as the primary agent in the reduction of service life and reliability of adhesive joints and composites [1]. This results from absorption of atmospheric moisture into the polymeric adhesive, which typically accumulates at the interface and displaces the polymer from the adherend surface [2, 3]. Epoxy adhesives are particularly sensitive to moisture, resulting in a dramatic loss of adhesion strength when exposed to moisture above a critical relative humidity level [4–12]. The critical relative humidity level is typically between 50 and 70%.

Subcritical adhesion testing is of practical interest to engineers and scientists because it explores adhesive debonding in a range of crack velocities and applied strain energy release rates, G, which are significantly less than those required for catastrophic failure. Therefore, subcritical adhesion testing simulates the failure occurring in the real-life application or service life of the adhesive. Crack growth can be driven by small applied loads generated by residual stresses, thermomechanical cycling, and mechanical or vibrational loading during service [13]. An additional advantage of subcritical testing over conventional adhesion tests is the reduced ambiguity associated with the dependence on crack velocity on the measured adhesion energy, which is associated with viscoelastic effects at the crack tip and in the bulk adhesive.

The effects of an invasive environment on adhesive bonds can be examined with subcritical adhesion measurements where tests during submersion in heated hostile fluids are possible. The average crack velocity, v (da/dt) and the applied strain energy release rate, G, are measured, where a is the crack length. A schematic of a typical v-G curve is shown in Figure 1. Three regions are usually observed, which are related to the mechanism for crack advancement [13–15]. Region III is associated with critical fracture events, which is independent of the environment for bulk glass fracture. Region II is strongly environment dependent, but only weakly dependent on the crack driving energy. This is characteristic of fracture where diffusion of the penetrant or environmental fluid to the crack tip is the rate limiting step for crack advancement. In Region I low crack velocities and low applied crack driving energies are found. The crack velocity is thought

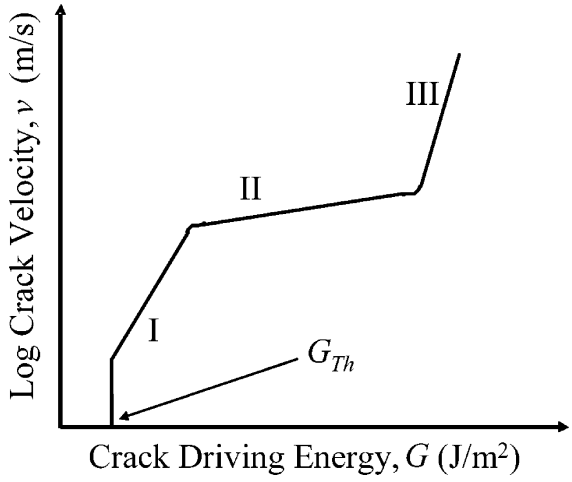

FIGURE 1 Schematic of a typical *v*-*G* curve illustrating the three regions of crack growth and the threshold value of *G*.

to be controlled by a stress-activated chemical reaction of the penetrant with the bonds at the crack tip [16]. At the lowest crack velocities, below Region I, there exists a threshold value of crack driving energy, G_{Th}, below which no crack propagation is observed.

Traditionally, subcritical fracture test specimens have been laminated beams, where the adhesive is bonded between two parallel rectangular rigid adherends. Examples of the laminated-beam-type experiments are the double cantilever beam (DCB) wedge test [5], the asymmetric double cantilever beam test [16], the double cleavage drilled compression specimen [17], and the four-point flexure samples [18]. The advantages of laminated beam-type specimens are 1) the adhesive is loaded elastically away from the crack tip, 2) high strain energy release rates are obtainable, 3) the fracture mechanics models for analysis are well understood, and 4) the specimens can be self-loading. The disadvantage of these types of tests is that they may require a sophisticated experimental set-up to measure the crack length (video camera, acoustic or electrical methods). Additionally, for samples exposed to a fluid environment, the equilibration time may be long because of the two impermeable substrates causing diffusion into the adhesive only from the edge. This leads to a heterogeneous distribution of diffusant in the adhesive joint.

With respect to the laminated-beam-type specimens, there are advantages to utilizing the shaft-loaded blister test. The time for

environmental saturation is relatively short, resulting from the exposed face and short diffusion path [19, 20]. In addition, the specimen geometry is axisymmetric, which reduces any misleading edge effects caused by degradation of the interface away from the crack tip. Some disadvantages of the shaft-loaded blister test are that its fracture mechanics models have not been studied as extensively as the laminated beam specimens and, like most coatings tests, the maximum value of the strain energy release rate before film rupture occurs is limited by the film's mechanical strength and the intrinsic interfacial toughness. Plastic yielding of the coating also complicates the analysis. Furthermore, the strain energy release rate is a function of $1/a^{4/3}$ and therefore will approach a threshold value of G more slowly than beam-type specimens. For the DCB wedge test, the strain energy release rate decreases as a function of $1/a^4$. Therefore, a wide range of G values can be obtained for a relatively small change in crack length and the threshold energy can be rapidly approached.

In this work, the shaft-loaded blister test (SLBT) was used to investigate the effects of relative humidity on an epoxy coating bonded to glass and silicon wafers. A typical experiment utilizes the controlled displacement of a spherically capped shaft, driven by a universal testing machine (UTM) to create a growing blister, as an alternative to applying fluid or gas media to generate the fracture (Figure 2) [21–27]. Although the SLBT has been used extensively to study polymer and thin film adhesion, to date the technique has not been adopted to the study of moisture-assisted subcritical crack growth.

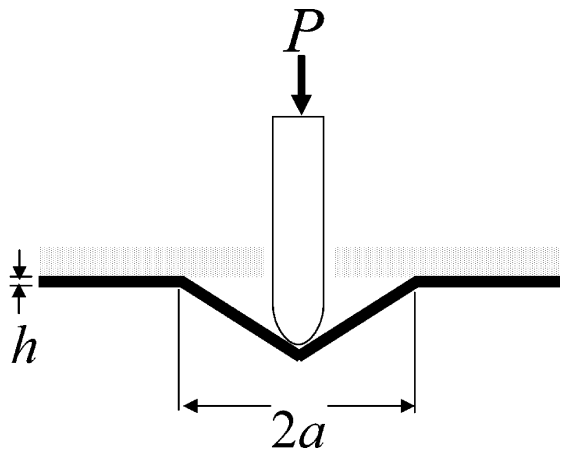

FIGURE 2 Schematic of the shaft-loaded blister test.

Specimens were tested by two methods: 1) the more common critical method where adhesive failure is essentially catastrophic and 2) modification of the SLBT configuration and measuring adhesion over a long timescale in a subcritical crack growth experiment.

EXPERIMENTAL

Materials

The model epoxy adhesive utilized in this work was Epon® 862 bisphenol-f resin (Shell Chemical Corporation, Houston, TX, USA) mixed with 10 parts per hundred resin (phr) of 1,4-butanediol (added to increase the solubility of the curing agent) and cured with 3 phr of 4-methyl-2-phenylimidazole [28]. Details of the physical properties of the model epoxy can be found elsewhere [28]. Quartz and silicon wafers were utilized as substrates. The quartz substrates were obtained from ChemGlass Inc. Vineland, NJ, USA) in a $15.24 \times 15.24 \times 0.9525$ cm ($6'' \times 6'' \times 3/8''$) sheet. The silicon wafer substrates, supplied by Hewlett-Packard Co. (Corvallis, OR, USA) had a 152.4-mm (6″) diameter with a thermally grown silicon oxide 10 nm thick.

Blister Test Specimen Preparation

Critical Adhesion Measurements

Critical adhesion measurements were made on the model epoxy bonded to quartz. Quartz is an appropriate substrate for our investigation because it will support significant loads (due to its thickness) in convenient SLBT geometries. It is also composed mostly of silicon oxide, and has a surface similar to the silicon wafers used in the microelectronics industry. A schematic of the critical shaft-loaded blister test specimens is shown in Figure 3. Quartz sheets were cut into 3.6×3.6 cm (1.5×1.5 in.) squares and a 0.8-cm (0.31″)-diameter hole was drilled in the center. A pre-crack was fabricated by placing a 0.95-cm (3/8″)-diameter piece of Kapton® pressure-sensitive adhesive tape (PSAT) over the hole in the center of the quartz substrate. The Kapton® PSAT consists of a 25-μm (1-mil)-thick Kapton® backing and a 37.5-μm (1.5-mil)-thick acrylic pressure-sensitive adhesive. The tape also provided additional mechanical reinforcement to the thin epoxy film at the highly stressed contact area between the coating and shaft tip. To prepare the specimen, the uncured model epoxy was first coated on the quartz substrate. A 50-micron (2-mil)-thick piece of Kapton® film (no PSA) was then placed on top of the epoxy coating. The Kapton® film acts as a mechanical reinforcing layer for the epoxy

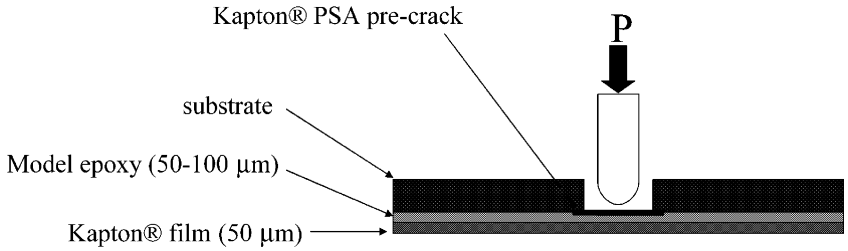

FIGURE 3 Schematic of critical shaft-loaded blister test specimen.

coating. The resulting adhesive coating is therefore a composite of the model epoxy, Kapton® PSAT (located solely in the center) and Kapton® film. A schematic of the test specimen is shown in Figure 3. The sample was then cured in a convection oven for one hour at 130°C and placed in a dessicator to cool slowly (approximately 30 minutes). The typical epoxy film thickness varied between samples from 50 to 100 μm, with ±5 μm uniformity in the films. Samples were conditioned at constant relative humidity (8, 29, 42, 71, or 98%) for 3 days at room temperature. This was sufficient time for the adhesive to be saturated. The relative humidity was regulated using saturated salt solutions [29–31]. After environmental exposure, the samples were immediately tested at ambient temperature and humidity (≈50%). Three samples were tested for each relative humidity using an universal testing machine (UTM) and shaft terminated with a ball bearing approximately 0.63 cm (1/4″) in diameter. The UTM cross head displacement rate was 0.1 mm/sec.

Constant Load Subcritical SLBT Specimens

Subcritical adhesion measurements of the model epoxy were obtained using the silicon wafer substrates. A hole approximately 12.5 mm in diameter was produced in the center of the silicon wafer using a diamond-coated drill bit and a Dremel® tool. Care must be taken when drilling the brittle wafers. The wafer was then rinsed with isopropyl alcohol and dried under a stream of ultra-high purity nitrogen gas. The hole in the wafer was then covered with a 0.95-cm (3/8″)-diameter piece of Kapton® PSAT. The wafer was uniformly coated with epoxy adhesive using a pneumatically driven doctor blade. A 152.4×152.4-mm (6″×6″)-square piece of Kapton® (50 μm thick) was carefully applied on the wafer to avoid disrupting the adhesive and forming air bubbles. The coated wafer (with Kapton®) was cured under identical conditions to the critical specimens described previously.

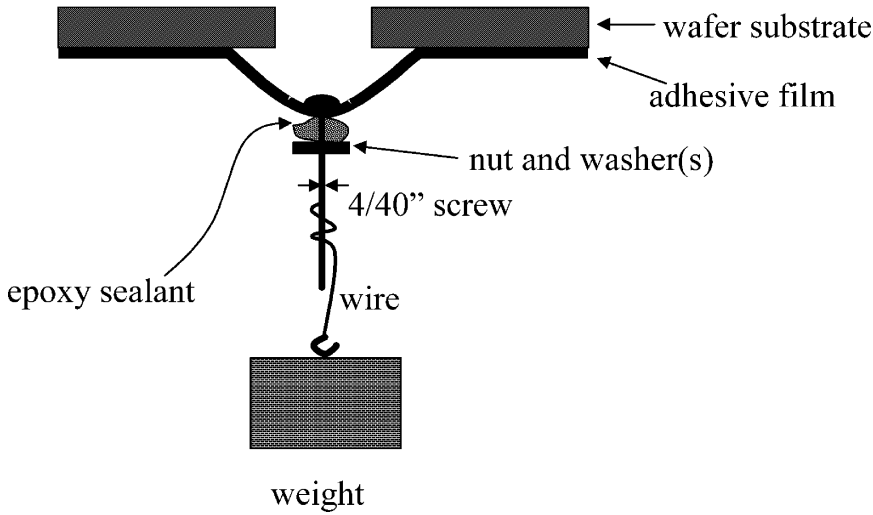

FIGURE 4 Schematic of the subcritical shaft-loaded blister test specimen.

To develop the subcritical blister test specimen, a sample must be self-loading, the integrity of the thin adhesive coating must be maintained, and the sample must be exposed to the fluid. A schematic of a scheme meeting these requirements is shown in Figure 4. To fabricate a simple self-loading, yet constant load, SLBT specimen, a hole was punched in the center of the coating that could accommodate a 2.54-mm (or 4/40″) stainless steel machine screw. The machine screw acts as the fastener from which a "dead load" was suspended *via* a flexible wire. A noncorrosive and high density material such as lead is recommended as the weight because of the corrosive nature of the saturated salt solutions. The punched hole containing the screw was sealed with a room-temperature cure epoxy (Devcon® 2-ton epoxy, Deucon, Danuers, MA, USA). The entire SLBT specimen was placed in a large glass vessel and conditioned at constant relative humidity (42, 71, or 98%) at room temperature. These values span the critical relative humidity value for adhesion loss.

Analysis

The strain energy release rate (crack driving energy), G, can be calculated from the "load-based equation" [Equation (1)] based on the load, P, and blister radius, a [12]:

$$G = \left(\frac{1}{16\pi^4 Eh}\right)^{1/3} \left(\frac{P}{a}\right)^{4/3} \quad (1)$$

where E is the Young's tensile modulus and h is the thickness of the adhesive coating. The modulus of the coating for the bi-layer film (Kapton® and epoxy adhesive) is estimated from a simple rule of mixtures:

$$E_{\text{composite}} = v_{\text{Kapton®}} E_{\text{Kapton®}} + v_{\text{Epoxy}} E_{\text{Epoxy}}$$

where $E_{\text{composite}}$ is the modulus of the composite, $v_{\text{Kapton®}}$ and v_{Epoxy} are the volume fractions of the Kapton® backing and epoxy, respectively, and $E_{\text{Kapton®}}$ and E_{Epoxy} are the moduli of the Kapton® backing and epoxy, approximately 2.5 GPa [28] and 6 GPa [32], respectively.

Equation (1) for the strain energy release rate is independent of the amount of plastic deformation that might occur in the adhesive at the contact zone between the shaft tip and adhesive coating [27]. This is located in the center of the blister and is also where the load is suspended in the subcritical experiments. The expression for the crack driving energy [Equation (1)] is also relatively insensitive to the value of Eh because the film's tensile rigidity (Eh) is raised to the $-1/3$ power. As a consequence, the SLBT is particularly advantageous for environmental durability testing because G is not strongly dependent on the values of the modulus and thickness, which are functions of the moisture content, temperature, and exposure time. The contribution of the Kapton® PSAT is negligible for the same reasons that G is independent of plastic deformation in the center of the blister.

For the critical adhesion measurements, samples were tested using loading and unloading cycles, which were repeated up to seven times, to determine the crack length as a function of load. The load applied by the UTM as a function of the resulting central shaft displacement (w_0) for successive cycles on a single sample is shown in Figure 5. In this test procedure, the film was loaded and the crack was allowed to propagate several mm. The load was removed, as was the sample from the UTM, and two or three measurements were made to calculate the average blister radius. This procedure is different from the typical blister-radius measurement where the crack is allowed to propagate continuously and the diameter is observed simultaneously with a video camera. An example of our typical load as a function of blister radius curve is shown in Figure 6. Note that the fitted line passes through the origin, as Equation (1) suggests. Utilizing the successive loading and unloading cycles, considerable effort involving measuring the blister radius with a video camera has been eliminated. And,

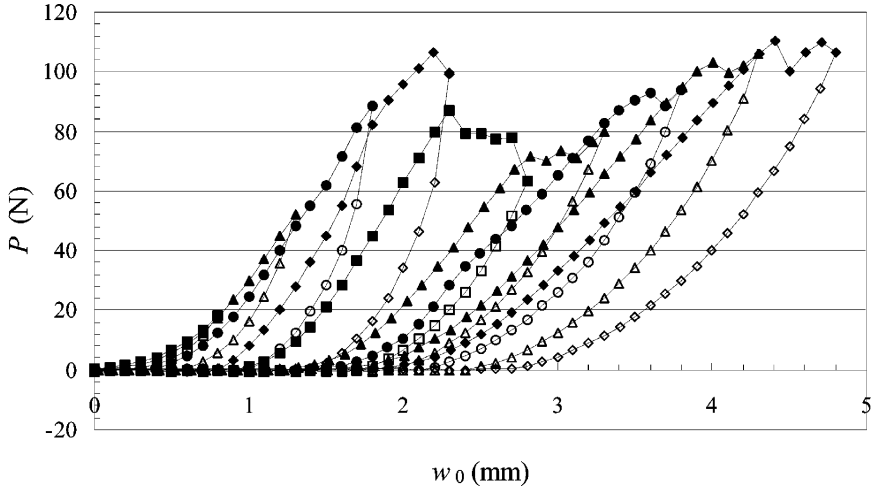

FIGURE 5 Successive load as a function of central shaft displacement curves for 98% relative humidity. The filled-in symbols are the loading curves and the unfilled symbols are the unloading curves.

unlike beam-type specimens that utilize opaque adherends, the crack length can be readily measured in our configuration. Therefore, the SLBT requires only an UTM and eliminates the need for sophisticated crack measuring equipment.

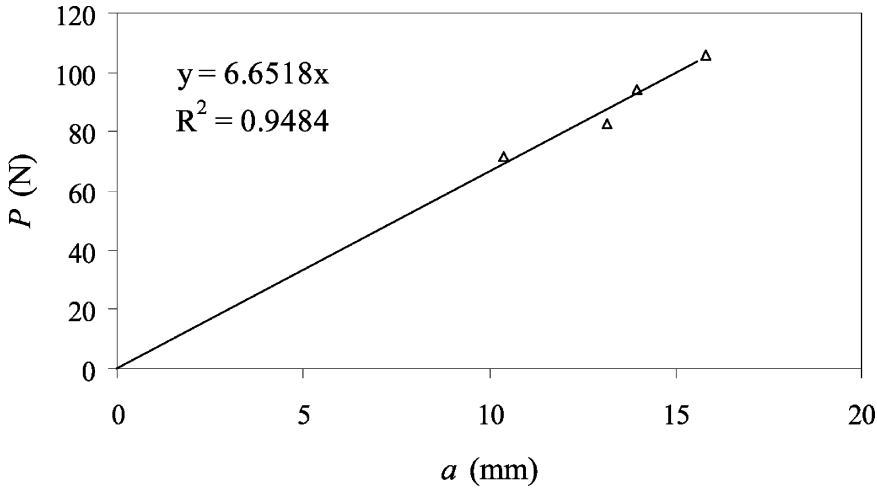

FIGURE 6 Load (P) as function of blister radius (a) at 98% relative humidity.

For the subcritical experiments, the average blister radius was measured periodically. The average crack velocity, $v(da/dt)$, was determined from the relationship between the average blister radius as a function of time. Again, no optical equipment was necessary.

RESULTS AND DISCUSSION

Critical Strain Energy Release Rates

The calculated strain energy release rates are shown in Figure 7 as a function of the relative humidity. At 8, 29, and 42% relative humidity, the film always ruptured before debond growth occurred, indicating very high interfacial toughness. However, the adhesive interface of specimens conditioned at the high relative humidities degrades sufficiently for debonding to occur. The resulting strain energy release rates are 163 ± 27 and 111 ± 27 J/m^2 for 71% and 98% relative humidity, respectively. A dramatic loss of adhesion occurs above 70% relative humidity, suggesting that there is a critical relative humidity environmental conditioning level for adhesion loss. In the case of film rupture, based on the initial hole size, a_0, and the maximum load at break, a lower bound of the strain energy release rate can be estimated. The values of G calculated from the load at rupture are not reported.

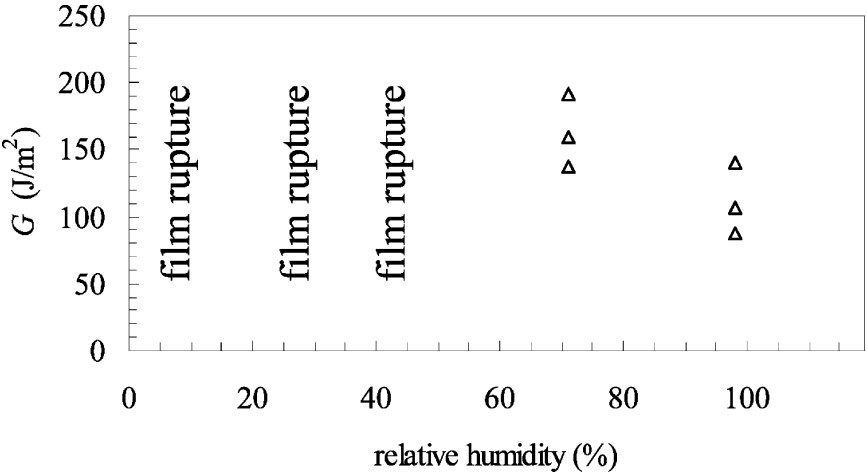

FIGURE 7 Summary of strain energy release rates G (J/m^2) as a function of relative humidity for critical SLBT.

Although exhibiting beneficial attributes such as axisymmetric geometry and the short saturation times, and the relatively simple sample preparation and test procedures, the shaft-loaded blister test has some shortcomings; namely, the adhesive film can rupture before debonding occurs resulting in more qualitative observations. This may be assigned to the large stresses generated within the film near the contact area between the film and shaft tip. Significant, visible, plastic deformation at the contact area may still arise, even if film rupture does not occur and crack propagation takes place. However, if plastic deformation is confined solely to the center of the blister and not at the crack tip, Equation (1) remains valid [27]. One method to reduce plastic deformation and maintain the mechanical integrity of the film is to reduce the applied load and, therefore, the stress on the film. This strategy was employed in the subcritical fracture experiments.

Constant Load Subcritical Fracture

The v-G curves obtained for the model epoxy as a function of relative humidity (42, 71, and 98%) are shown in Figure 8. For each specimen, the initial value of the crack driving energy was approximately 40 J/m^2. As the crack advances, the value of the strain energy release rate gradually decreases as the size of the blister radius increases until the crack appears to arrest at the value of G_{Th}. The averages along with one standard deviation of G_{Th} as function of relative humidity are listed is Table 1. The value of G_{Th} for 42% relative humidity conditioning is significantly greater than either 71% or 98% relative humidity cases. Moreover, G_{Th} is almost identical for the 71% and 98% relative humidity exposures. In some cases, G_{Th} has been found to be dependent on the concentration of water molecules at the crack tip- and, therefore, the relative humidity [16, 18, 33]. Other work has shown that above a critical relative humidity value ranging between 50 and 70%, the adhesive fracture energy is constant and is independent of the vapor pressure [34, 35]. Condensation at the crack tip because of capillary forces may negate any differences in the effect of moisture level conditioning at high humidty. This behavior is predicted from the classic Kelvin equation for the meniscus radius. From that equation, at 70% relative humidity, the meniscus radius is predicted to be approximately 1 nm, about the size of a bond length or molecule.

Residual stress can also affect the adhesion measurement if the G_{Th} is similar in magnitude to G attributable to residual stress. The residual stresses in the coating originate from the contraction of the epoxy

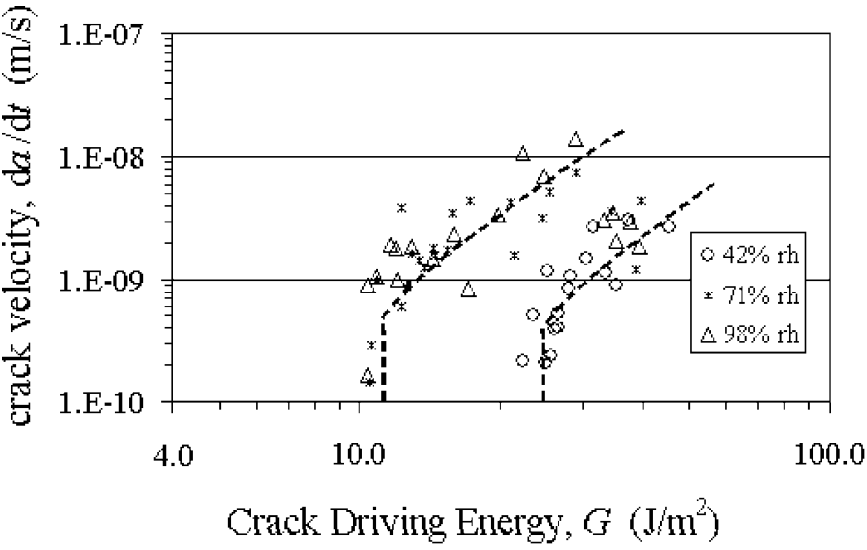

FIGURE 8 SLBT velocity (m/s) as a function of crack driving energy, G (J/m^2), and relative humidity (42, 71, and 98%).

during cure and the difference in the coefficient of thermal expansion (CTE) between the substrate, epoxy, and Kapton® backing. During the critical experiments, residual stress is not expected to be a factor as the stress applied by the UTM is much greater than the residual stress in the film [36]. Depending on whether the residual stress is tensile or compressive, the applied strain energy release rate will be less than or greater than the zero residual stress condition [37, 38].

The tensile residual stress in the model epoxy film bonded to glass was estimated from two different methods. In the first method, residual stress was estimated from the radius of curvature of a bimaterial strip of epoxy to glass and found to be 5.5 MPa [39]. In the second method, the residual stress was estimated from Equation (3) [40]:

TABLE 1 Threshold Crack Driving Energy, G_{Th}, as Function of Relative Humidity

	42% rh	71% rh	98% rh
Average $G_{Th}(J/m^2)$	25.2	10.2	11.5
Standard deviation	4.9	2.1	2.6

$$\sigma_r = -\frac{E}{(1-\nu)}\Delta\alpha\Delta T \tag{3}$$

where $\Delta\alpha$ is the difference in the CTE between the substrate and adhesive and ΔT is the difference between the temperature of the sample and the stress-free temperature. Utilizing values of the stress-free temperature and CTE [39], the residual stress was found to be as high as 20.7 MPa. These values were for the dry coatings that were immediately removed from the oven such that the stresses had little time to relax.

To determine if residual stresses play a role in our subcritical adhesion measurements, the value of G_{Th} was compared with the strain energy release rate of a coating delaminating due to residual stress [see Equation (4)] [40, 41]:

$$G = \frac{h\sigma_r^2(1-\nu)}{2E} \tag{4}$$

where σ_r is the residual stress and ν is Poisson's ratio. Using the values of residual stress determined from the bimaterial specimen and Equation (3), the strain energy release rate in the dry coating was found to be 0.2 and 2.6 $\mathrm{J/m^2}$, respectively.

Below the critical relative humidity, at 42%, the values of G_{Th} obtained by the SLBT are much greater than the G attributable to residual stress. Therefore, at 42% relative humidity, residual stress is not likely a significant factor. However, at high relative humilities, both the G values from residual stress and the SLBT are similar in magnitude, which suggests that residual stress may play a role. However, at high relative humidities, there is evidence that the residual tensile stresses in the dry state relax and decrease to zero or even become compressive [42, 43]. In the case where there is compressive residual stress, the measured threshold value of G_{Th} will be greater than the unstressed condition, leading to an overestimation of G_{Th}, which suggests the interface is more durable than it appears. It should be noted that for this adhesive, after saturation, the bimaterial strips did not exhibit any signs of compressive stresses. The evidence shows that debond growth is not driven by residual tensile stresses, but by the applied load and presence of moisture.

The adhesive fracture energy can also be affected by changes in the mechanical properties of the epoxy and backing because of moisture absorption. Although it is expected that the Kapton® will not be drastically affected by the moisture, some epoxies have shown a decrease in the modulus by as much as 80% in high humidity [19]. The modulus

of the model epoxy used in this work was found to decrease modestly by 18% relative to its dry condition [39]. The modulus change and thickness change because of swelling has little effect on the measured debond driving energy, as $G \sim (1/Eh)^{1/3}$. This insensitivity to changes in modulus makes the SLBT particularly advantageous for environmental durability studies. The strain energy release rate can also be calculated as $G = Pw/(4\pi a^2)$ [44]. This expression is independent of the mechanical properties and it requires, in addition to the load and blister radius, that the displacement, w, be measured. Mechanical properties of the film can also change because of creep of the adhesive and backing. Creep in the subcritical SLBT may be caused by the suspended loads and resulting stress, the plasticizing effects of humidity, and the long duration of the experiment, which in this work was up to 4 months. Creep should not affect the strain energy release rate because Equation (1) depends solely on the mechanical properties of the film at the crack front. Therefore, as the crack continually advances, the film located directly at the crack front is loaded for only a brief period of time.

Experimental uncertainty in the calculation of the values of G, and crack velocity, v, are introduced from the measurement of the blister radius, a, as well as from any slight asymmetry of the blister. The error associated with measuring the blister radius with the micrometer is approximately ± 0.2 mm. Furthermore, the blister radius may not be perfectly symmetric, although this difference is generally small (0.1 to 0.2 mm). In the most extreme cases, the difference between two measurements of the same blister radius is as large as 0.5 mm. Asymmetry can be caused by differences in thickness, residual stress in the epoxy or in the Kapton® backing [45], and any heterogeneities in the intrinsic interfacial adhesive toughness. The experimental uncertainty is essentially unavoidable because of the asymmetry of the blister and is difficult to reduce below 0.1 mm. The error attributable to the blister radius measurement depends on the magnitude of the value of G and crack velocity. For values of G between 20 and 50 J/m^2 the error is ± 1–2 J/m^2, whereas for values of G between 1 and 20 J/m^2 the error is ± 0.1–0.5 J/m^2. Crack velocities on the order of 10^{-8} and 10^{-9} m/s have errors on the order of $\pm 10\%$, which is much less than the inherent scatter. The error is significant for crack velocities of 10^{-10} m/s or less. However, at these crack velocities, the experimental limit of the measurement has been reached and these cracks appear to arrest.

The quartz substrate and the silicon wafer substrates were expected to produce similar adhesion results given that both have surfaces composed of silicon oxide. In the model epoxy–quartz experiments, a

critical level of relative humidity was observed where adhesion loss occurred. That is, at a relative humidity of 42%, the reinforced epoxy film ruptured and the adhesive fracture energy was relatively large. Utilizing the constant load subcritical SLBT, however, the adhesive system could be characterized above and below the critical relative humidity. Therefore, by reducing the applied strain energy release rate and consequently the applied stresses in the adhesive coating, the integrity of the epoxy coating was maintained and the adhesive could be controllably debonded. In addition, from the subcritical SLBT experiments the evidence of a critical level of relative humidity for adhesion loss was much more qualitative. These results show that the constant-load subcritical shaft-loaded blister test is a promising new technique for studying the effects of the environmental degradation on adhesive joints and coatings. Given the great difficulty in testing thin, strongly adhered coatings and films, this technique is particularly attractive. Furthermore, we have demonstrated the subcritical SLBT can be applied to the study of polymer adhesion to brittle silicon wafers which are usually difficult to test given their fragile nature.

The subcritical SLBT specimen has clarified the mechanism for crack growth in this adhesive system within the range of applied crack-driving energies studied. In this work, specimens are different from the classical subcritical crack growth experiments in glasses. This is because the entire adhesive bond is saturated and moisture is already present at the crack tip and vicinity. Therefore, the rate limiting step for crack growth, which produces Region II (the diffusion of moisture to the crack tip), has been "turned off." This strongly suggests that the mechanism for crack growth in these experiments is not dependent on moisture diffusion, but rather also dependent on stress. Similar results that showed diffusion was not the rate limiting step for subcritical crack growth were obtained by Singh and Dillard, who studied subcritical fracture of an epoxy bonded to glass in a fluid environment using the DCB wedge test [46]. They tested two types of samples that controlled the path of diffusion of liquid into the adhesive joint and therefore the role of the fluid molecules on debond growth. One set of specimens were saturated with fluid prior to applying the wedge and therefore the mechanism for Region II crack propagation was turned off. The other set of specimens were initially dry (0% concentration of penetrant) and the wedge was applied at the same time as the specimen was introduced to the fluid environment. They observed identical v-G curves between the two types of samples, which suggests that the diffusion of fluid to the crack tip is not the rate-limiting step for debond growth. This supports the argument that the mechanism for crack growth in our work is a stress-dependent

phenomenon, and not diffusion controlled, indicating that our present results fall in Region I of the v-G diagram.

This work has shown that the SLBT has a number of advantages over other adhesive coating test methods: 1) it is an open face coating that reaches saturation rapidly, 2) it is axisymmetric, 3) it requires simple sample preparation, 4) debond growth is easily measured, 5) samples are self-loading, 6) it is insensitive to plastic deformation and creep away from the crack tip, and 7) measuring adhesion to thin brittle substrates is possible. A few limitations to the SLBT exist that are common to other peel-type test geometries. The most prominent is that when the adhesive interface is strong and the film is thin, the adhesive can experience large stresses, leading to film rupture. Also, a concern is delamination of the backing from the adhesive rather than delamination at the adhesive–substrate interface. Much higher strain energy release rates can be achieved with more conventional beam type specimens, but one may encounter other problems such as sample preparation or crack measurement issues. A possible method to increase the available strain energy release rate in the SLBT is to use a thicker backing that will adhere strongly to the adhesive even in a moist environment and be able to support higher loads without creep. Under these conditions, the adhesive may behave less like a stretching membrane and more like a bending plate and a different expression for the strain energy release rate is applicable [24].

This work convincingly shows that the subcritical SLBT is an excellent method for testing the durability of adhesive coatings. In addition to studying the effect of water vapor, the effects of temperature, various chemical and mechanical stresses (continuous and cyclic), corrosion, and ultraviolet radiation exposure can be investigated with the SLBT. Any changes in the mechanical properties of the adhesive and backing resulting from exposure to aggressive conditions must be considered. However, changes at the crack front and in the adhered coating must be considered. This is due to the same reason creep in the film is not considered. Caution must be taken if the test temperature of the adhesive is close to its glass transition or if its yield stress is sufficiently low that plastic deformation and viscoelasticity at the crack front is possible. In the case where the SLBT specimen is exposed to moisture and chemicals, the adhesive may be significantly plasticized or swollen such that there is no longer linear elastic behavior at the crack front. Therefore, many types of glassy adhesives can be tested within the assumptions of the model. For soft materials, such as pressure sensitive adhesives and rubbers, cavitation and finger-like instabilities caused by confinement of the adhesive between the backing and substrate can complicate the analysis.

CONCLUSION

The SLBT was demonstrated to be a simple and informative method for characterizing the degradation of adhesive bonds accelerated by environmental moisture, without the need for sophisticated crack-length measurements. Notably, the SLBT was modified for subcritical fracture testing by applying a self-loading mechanism whereby a mass is suspended from the center of the inverted blister. Our results illustrate the benefits and shortcomings of the SLBT, suggesting that this test method is advantageous for testing thin adhesive coatings which often rupture under high stresses imposed by more conventional test methods. Furthermore, the low applied stresses enables testing of adhesives bonded to fragile, brittle silicon wafers. This study has also provided an additional example of a critical relative humidity for adhesion loss which has been previously observed in many epoxy systems.

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