

Effects of mechanical stress and moisture on packaging interfaces

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When microelectronic packages fail in accelerated stress testing, it is often because of mechanical stress and/or moisture acting upon interfaces between polymeric adhesives or encapsulants and other package components such as solder interconnects, chip passivation, heat sinks, and chip carrier surfaces. Once the polymer loses adhesion, even if only in a small area, delamination at the interface can occur over time, leading to package failure. This paper describes an adhesion test methodology, using model materials and interfaces rather than actual packages, which has increased our understanding of the effects of mechanical stress and moisture and how they interact to induce adhesion failures. The effects of increasing severity of moisture exposures at elevated temperature and humidity conditions were measured using adhesion testing of epoxy/steel interfaces with and without adhesion promoters. An important aspect of this investigation pertained to the effect of the combination of mechanical stressing and exposure to moderate moisture conditions followed by solder reflow temperatures, again comparing the results for interfaces with and without adhesion promoters. Epoxy interfaces were weakened by the combination of mechanical stress and moisture exposure, thus allowing pockets of water to collect and cause delamination during subsequent solder reflows. Some insights are offered on how best to prevent this package failure mode, referred to as "popcorning," caused by vaporization of moisture at the interface.

Introduction

For microelectronic packaging applications, durable adhesion is one of the most important considerations in developing a reliable structure. For packaging structures that include interfaces with organic polymers, the greatest challenge is usually to obtain adhesion that will survive humidity stressing, since all organic polymers are to some degree permeable to water vapor, and water molecules can easily adsorb at a polymer interface and degrade the adhesion. If, after absorbing moisture, the organic package is subsequently subjected to high temperatures such as those experienced in solder reflow operations in card assembly, vaporization of water can cause a serious loss of adhesion ("popcorning"), resulting eventually in delamination and package failure. The popcorn phenomenon was first identified with molded plastic wire-bond packages [1-4]; but it has been recognized more recently to occur more generally at plastic interfaces [5],

e.g., between underfill and chip passivation in flip-chip packages [6].

To design and build robust packages, it is important to understand the response of the materials and interfaces to the conditions to which they will be subjected. Adhesion testing contributes to that understanding, especially if it can be shown that such tests are predictive, i.e., they provide results which correlate with the performance of polymer interfaces in actual products. The use of silane adhesion promoters or couplers [7] is one aspect of engineering the interfaces in microelectronic packages that is important to investigate using adhesion testing. Both the composition [8] and the network structure [9] of the adhesion promoters are important to optimize for the particular materials and structures being used, and this is more efficiently done by using adhesion tests rather than by building actual packages. Epoxy adhesives and their interfaces with other materials have been studied quite

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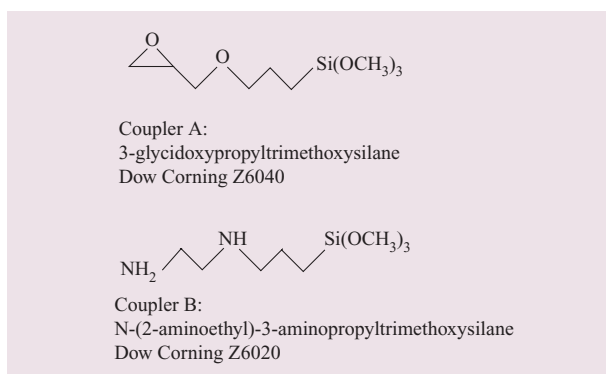


Figure 1

Couplers A and B: chemical structures and names.

extensively [9, 10] because of their wide utility. For microelectronic packaging, epoxy materials are used extensively as die attach adhesives [11], molded encapsulants [12], flip-chip underfills [13], and structural reinforcement [14]. The topic of this paper is epoxy adhesion at a stainless steel test surface, including a detailed analysis of the combined role of moisture and mechanical stress in inducing adhesion loss during subsequent exposure to high temperature, as experienced by packages during solder reflow. Adhesion testing in this study is used not as a quantitative measure of interfacial fracture toughness, but as a comparative tool to optimize the ability of adhesion promoters to strengthen package interfaces and to understand how to prevent popcorning at such interfaces. A review of the important fields of adhesion and adhesion testing is outside the scope of this paper, but the interested reader is referred to selected references in the field—for example, [9, 15–19].

Experimental methods

Materials

Test coupons ($12.5 \times 16.5 \times 0.18$ mm) of stainless steel (SS) 304 were used in this study. The SS coupons were particularly amenable to adhesion testing because the high modulus and high yield stress of SS304 allowed quantitative measurements to be made using a cantilever beam technique (see below). The epoxy is a standard industrial structural adhesive based on epoxy novolac chemistry. The glass transition temperature (T_g) of the fully cured material ($130^\circ\text{C}/4$ hr) was about 90°C . Two commercially available silane adhesion promoters, Coupler A and Coupler B (**Figure 1**), were evaluated. The different chemistries of these two couplers were important to this study because of their different reactivity with the epoxy novolac adhesive. The epoxy groups of Coupler A

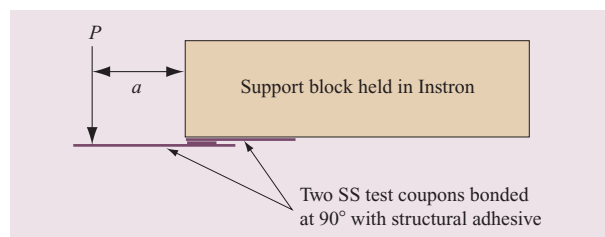


Figure 2

Schematic for cantilever beam test.

have reactivity similar to that of the adhesive itself, and thus react only during the adhesive cure cycle [20]. Coupler B, on the other hand, contains highly reactive amine groups that react on contact with the epoxy novolac [21]. Both couplers have silyl ether end groups which, upon hydrolysis prior to application, bond with inorganic surfaces [7].

Application of silane adhesion promoters

The adhesion promoters were dissolved in aqueous alcohol mixtures and diluted to 0.1%–1.0% by volume. The promoters were applied to the SS coupons by a simple dip process followed by a short bake at 90°C – 120°C . Variables that were explored to optimize the coverage of Couplers A and B included coupler concentration, water content of the solvent, pre-mix time, number of dips, post-application rinses, and post-application bake temperature. Each set of application conditions was characterized for coupler coverage by X-ray photoelectron spectroscopy (XPS) and used for adhesion testing.

Adhesion testing

The two adhesion tests used in this study were a cantilever beam test [22, 23] (**Figure 2**) and a wedge test [24–28]. The objective of each test was to measure epoxy adhesion to steel after exposure to stress conditions which were designed to simulate and accelerate the effects of stresses on adhesion at packaging interfaces.

Cantilever beam test

The cantilever beam test samples were prepared by dispensing epoxy novolac structural adhesive onto one of two SS test coupons, using two double strips of polyimide adhesive tape to define the boundaries of a stripe. After the adhesive was dispensed by using a glass microscope slide to draw down an even coating between the tape strips, the double strip was peeled off to remove excess structural adhesive. The second test coupon was placed across the first one to form a “T.” The assembly was then cured in a convection oven at 130°C for four hours. Most

samples were subjected to 24 hours of pressure cooker testing (PCT) at 121°C/2 atm of steam and then dried for at least 12 hours at room temperature. The samples, usually two or three duplicate assemblies, were then each held in a mechanical testing instrument (e.g., Instron 1125, Figure 2) and the force P required to detach the bottom coupon was recorded. Each failed adhesive joint was then examined in order to determine the predominant failure mode (adhesive or cohesive). The method relies on determining the load required to propagate a crack and calculating a critical strain energy release rate G_C (in.-lb/in.² or J/m²) to cause failure of the adhesive bond.

For a system with a crack of area A loaded by a force P , the general expression for strain energy release rate G is

$$G = -[\delta U / \delta A + (\delta U / \delta P)(dP / da)], \quad (1)$$

where U is the strain energy stored in the loaded system [29]. For linear elasticity and a sample of constant width B and crack length a , Equation (1) becomes

$$G = (P^2 / 2B)(dC / da), \quad (2)$$

where C is the compliance of the beam and P is the force measured by the mechanical tester. Values of C can be calculated or measured; the equation in **Figure 3** is for the measured values. As P increases, G increases until a critical value G_C is reached, at which point the crack propagates. Using a 0.005-m span, which is the effective crack length a from load point to crack tip (Figure 2), gives

$$G_C = 0.0105 * P_f^2 / B, \quad (3)$$

where G_C is in J/m² and the right side of Equation (3) is expressed in N and m. Although it is recognized that crack tip radius affects measured G_C , no effort was made to sharpen the crack beyond the initial sample preparation; and the measured G_C values are likely higher than those that would have been obtained with crack sharpening. With this methodology, however, standard deviations less than 10% were achieved, and the values were useful for comparison of different surface treatments.

Wedge test

Wedge testing has been used extensively in the past to measure the energy of fracture at an adhesive joint, usually with some form of crack initiation [24–28]. Our use of the wedge configuration is different from prior uses in that the wedge is used to apply a static mechanical stress, estimated to be equivalent to that experienced by the adhesive joint in an actual package, while the sample is simultaneously exposed to elevated humidity and temperature. The wedge was not used to fracture the

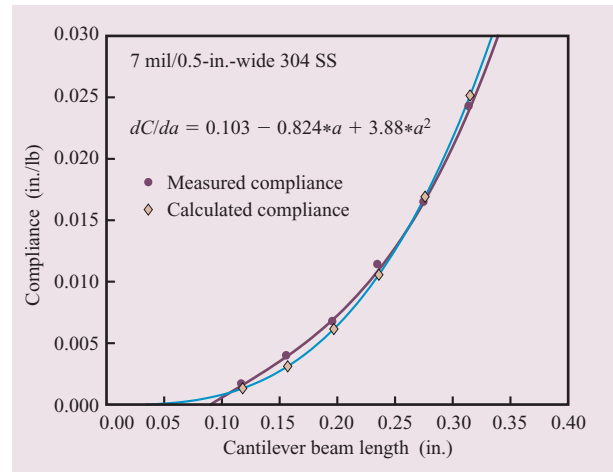


Figure 3

Measured and calculated compliance of stainless steel coupons used in cantilever beam test.

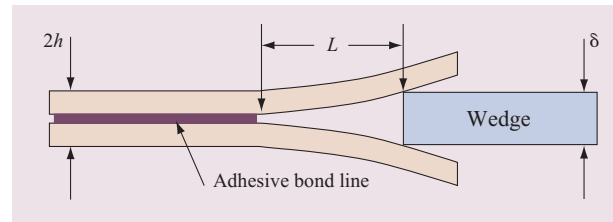


Figure 4

Schematic for wedge test.

adhesive joint. As explained below, the output data from this test was based on examination of the interface of adhesive with the SS coupons. For preparation of the test samples, two SS coupons were bonded face to face using Kapton adhesive tape to delineate the epoxy adhesive in a manner similar to that used for the cantilever beam test, except that the coupons were superimposed and the bonded area was offset to one end of the pair to allow space for a wedge, as shown in **Figure 4**. Following the usual cure cycle for the structural adhesive, the wedge was applied to simulate the mechanical stresses that an adhesive interface might experience in an actual package. The strain energy release rate G can be determined by the following equation and varied by adjusting L , the distance between the edge of the adhesive bond line and the front edge of the wedge (Figure 4):

$$G = 3Eh^3\delta^2 / 16L^4, \quad (4)$$

where E is the modulus of SS304, h is the steel thickness,

Table 1 Cantilever beam test results incorporating PCT stressing.

<i>Test sample</i>	G_C (J/m ²) $\pm$ standard deviation	<i>Failure mode*</i>	<i>At.% Si</i> (XPS)
No coupler	0	A	Not applicable
Coupler A	258 $\pm$ 12	C	2.2
Coupler A	165 $\pm$ 15	A	1.2
Coupler A	255 $\pm$ 50	C	8.9
Coupler A	280 $\pm$ 27	C	4.1
Coupler A	232 $\pm$ 19	C	3.4
Coupler A	206 $\pm$ 26	A & MAC	2.4
Coupler A	249 $\pm$ 6	C	6.1
Coupler A	253 $\pm$ 25	C	6.8
Coupler A (after 24 hr, 85°C/100% RH, no PCT)	122 $\pm$ 15	MAC	3 [†]
Coupler B (after 24 hr, 85°C/100% RH, no PCT)	191 $\pm$ 13	C	5 [†]

*Failure modes: A – adhesive, C – cohesive, MAC – mixed adhesive and cohesive.

[†]Representative values measured on similarly prepared test coupons.

and δ is the thickness of the wedge. From finite element modeling of various package structures, it was estimated that, at temperatures well below the T_g of the adhesive, G values as high as 50 J/m² may be caused by differences in coefficients of thermal expansion of an adhesive or encapsulant and other components of the package such as silicon chips, ceramic chip carrier substrates, or laminate chip carrier substrates. At solder reflow temperatures of about 220°C, the G value at the adhesive interface would be an order of magnitude less, or about 3 J/m², primarily because of the reduced modulus of the adhesive above its T_g . To simulate and accelerate the effect of conditions experienced by actual packages, the following sequence of stress conditions was applied to the wedge test samples: a) humidity exposure (24 hr/70°C/60% RH) under $G = 50$ J/m² wedge; b) moisture bakeout with wedge removed from sample (24 hr/130°C); c) humidity preconditioning (6 hr/85°C/85% RH); and d) three solder reflow heat cycles to 220°C with $G = 3$ J/m². Note that this test sequence also simulates the Joint Electronic Device Engineering Council (JEDEC) standard preconditioning test for plastic packages [30]. Following these stresses, wedge test samples were examined for delaminations using acoustic microscopy and by peeling the two coupons apart. The results are reported in terms of the percentage of the area of the adhesive stripe that showed failure at or near the interface between adhesive and SS.

Flexural strength testing of epoxy adhesive

Bar samples of epoxy novolac adhesive were cured in polytetrafluoroethylene cavity molds in order to evaluate

cohesive strength as a function of humidity conditioning. The bars were weighed to ± 0.1 mg before and after exposure to various temperature/humidity conditions to determine total moisture weight gain. The bars were then tested to failure in a three-point flexural test using a mechanical tester. The force-deflection curve was recorded, and the maximum flexural strength at failure was calculated.

Surface analysis for adhesion promoter concentration

It was important to differentiate the coverage of Couplers A and B on the SS test coupons to help characterize the resulting epoxy adhesion. Since the coverage of adhesion promoters on various surfaces can be as thin as a few monolayers, the surface sensitivity of a Perkin-Elmer Phi 5500 X-ray photoelectron spectroscopy (XPS) system with MgK α excitation was needed for these measurements. Photoemitted electron take-off angles were 15° and 45° from the sample surface in order to collect data to depths of about 1.5 nm and 10 nm, respectively. A survey scan was recorded for each SS test coupon, and the atomic percent of Si_{2p} at a binding energy of 102.5 eV was recorded as a measure of coupler coverage on the test coupon. In all cases, strong signals for the SS itself were observed, indicating that the thickness of the coupler was less than the sampling depth of the XPS. For comparison, cleaned but uncoated SS coupons were found to have at most 0.3% Si. XPS was also used to characterize the failure interfaces after adhesion testing. For the purposes of this study, an

adhesive failure was defined as one in which the elements of SS could be detected by XPS at the failure surface. Since the XPS sampling depth under the conditions of our measurements is only 1.5–10 nm, any failure surface in which any residue of adhesive could be seen by close inspection could be safely classified as cohesive. Spot checks by XPS of failures after humidity exposures in which the shiny surface of the SS could consistently be seen by eye showed strong XPS signals for Fe, Cr, etc. from SS, and these have thus been classified as adhesive failures by our operational definition.

Results and discussion

The objective of any reliability test of microelectronic packages is to accelerate the effects of field conditions to predict how various material and process options will actually perform in the field. To this end, packages are commonly subjected to thermal cycling in order to induce mechanical stresses from mismatched thermal expansion coefficients in component materials and exposed to humidity in order to induce failures associated with effects of moisture. Adhesion testing of test structures which model the packaging interfaces without requiring actual packages to be built and sacrificed to reliability testing can be a valuable adjunct to package reliability testing if the structures can be designed to incorporate stresses similar to those that exist under field conditions. Such tests are valuable both for initial stages of package design and for process monitoring during manufacturing.

Cantilever beam test results

PCT was incorporated into the cantilever beam test because, without aggressive moisture exposures, the epoxy adhesive showed uniformly good adhesion results even without any adhesion promoter, e.g., $G_C = 489 \pm 89 \text{ J/m}^2$ with 100% cohesive failure. To provide some degree of differentiation, the PCT was added. The results shown in **Table 1** indicate that PCT does provide the desired differentiation in adhesion results but with widely variable results depending on specific coupler application conditions when Coupler A is used. Characterization of adhesion promoter coverage by XPS analysis gave a rough correlation to adhesion testing results (see **Figure 5**). These results suggested that, to ensure the robustness of adhesion under PCT, XPS values of more than 4 at.% Si were required; and it appeared that the cantilever beam adhesion test with PCT was a useful screening test for process improvements.

A more detailed study of the effects of PCT compared with other humidity exposure conditions revealed a surprising result, however. Our intent in choosing PCT was to differentiate among process alternatives by forcing failures at the interface between the adhesive and the SS test coupons. Strikingly, the results in **Figure 6** (all

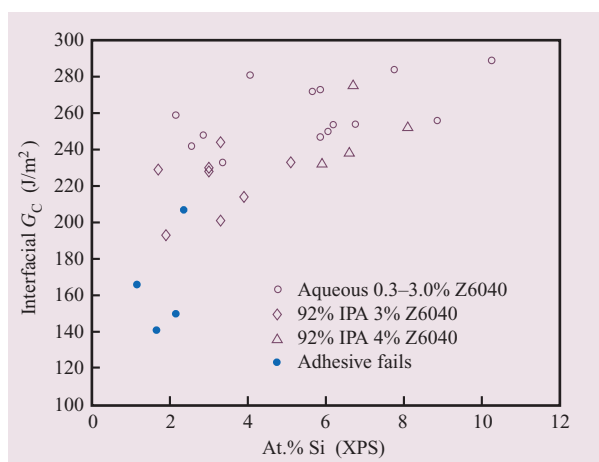


Figure 5

Correlation of coupler concentration on SS test coupons and cantilever beam adhesion test results.

measured on test coupons with Coupler A applied at about 3 at.% Si) show that moderate humidity exposures cause the cantilever beam test to show a higher degree of adhesive failure than the more aggressive conditions of either HAST (highly accelerated stress testing, 130°C, 85% RH) or PCT. Using more moderate humidity conditions allowed differentiation between Couplers A and B. As shown in the last entry of **Table 1**, use of Coupler B resulted in consistent cohesive failures even under moderate humidity stress conditions.

To explain the counterintuitive observation that exposure to moderate humidity conditions caused more adhesive failures using Coupler A than did more aggressive conditions, bar samples of the epoxy were prepared and tested on the mechanical tester to measure the flexural strength of the adhesive after various humidity exposures. The results are plotted in **Figure 7** vs. the measured weight gain of water absorption. The cantilever beam adhesion G_C results vs. moisture uptake are also plotted in **Figure 7** using the right vertical axis.

On the basis of these results, a plausible explanation for the adhesive failures after exposure to moderate humidity conditions can be offered. As moisture is absorbed by the epoxy/steel test structure, it initially finds its way to the epoxy/steel interface, where it weakens the adhesive strength, resulting in an increase in adhesive failure. In **Figure 6(b)**, the location of the adhesive failure around the periphery of the epoxy is indicative of the path of the moisture as it displaces the epoxy at the interface by inserting water molecules at sites where the epoxy is hydrogen-bonded to the surface. This observation is consistent with prior work by Zanni-Deffarges and Shanahan [31], who showed evidence of faster diffusion of

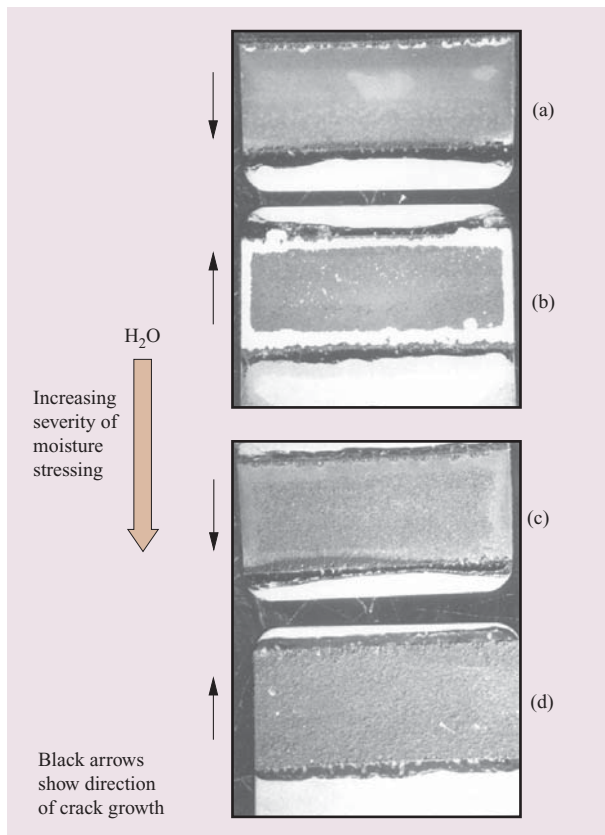


Figure 6

Cantilever beam adhesion test results after various moisture exposure conditions: (a) No moisture exposure; (b) 250 hr 85°C/85% RH; (c) 96 hr 130°C/85% RH (Highly Accelerated Stress Testing, or HAST); (d) 96 hr 121°C/2 atm water vapor (PCT).

water at an epoxy adhesive interface than through the bulk. As more moisture is driven into the bulk by the more aggressive conditions of HAST and PCT, however, the reduced flexural strength of the bulk adhesive allows the failure to occur again within the epoxy (cohesive failure), resulting in an apparently superior adhesion result. The important lesson from these results is that adhesion testing will have predictive value only if the humidity exposure conditions are chosen with a complete understanding of the response of the component materials. Unrealistic test conditions may cause responses with no correlation to the responses of microelectronic packages to field or package assembly conditions.

Wedge test results

The cantilever beam test was useful in its ability to quantitatively measure the robustness of adhesion after exposure to humidity and thereby to differentiate

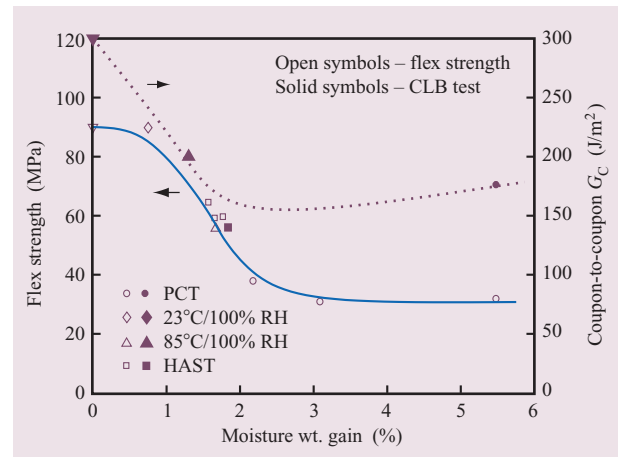


Figure 7

Measured modulus of epoxy adhesive and cantilever beam adhesion results vs. moisture absorption.

between samples having different levels of Coupler A or between those with Coupler A and Coupler B. Real packages, however, are simultaneously exposed to moisture and mechanical stress at polymer interfaces. Real packages are also exposed to high temperatures (220°C or higher) during card assembly solder reflows. Although one could consider using an environmental test chamber for the cantilever beam test to allow temperature and humidity to be varied while using the tester to mechanically stress the sample, it was more convenient to devise an alternate test method using a wedge to apply the mechanical stress. The stepwise stress conditions of the wedge test, as described in the section on experimental methods, were chosen to probe the effects of a three-way combination of mechanical stress, humidity, and solder reflow on the reliability of package interfaces.

It is well known [18, 32] that mechanical stress can increase the sensitivity of an adhesive interface to attack by incoming moisture. The stress applies tensile or shear forces at the interface. These forces may be insufficient to break the adhesive bonds until water arrives to replace these bonds with hydrogen bonds: Hydrolysis in combination with mechanical stress is more effective than either alone. Sufficient displacement of the adhesive by water molecules may then occur to allow the formation of a defect at the interface. Condensation of a pocket of liquid water at such defects and its subsequent rapid vaporization during the temperature excursion associated with solder reflow (popcorning [1–6]) provides a strong driving force for delamination. This sequence of events is represented schematically in **Figure 8**. This scheme is an

oversimplification in one important respect.¹ It neglects the role of absorbed water in the bulk of the epoxy and the kinetic effects of diffusion of this absorbed water on the interfacial defects during solder reflow,² contributing to the popcorn failure at the interface. However, as is elucidated below from the wedge test results, the interfacial defects play the lead role in triggering the popcorn effects, and eliminating them is the best hope for controlling this form of package failure because absorption of water in the bulk of the adhesive is usually unavoidable in real packages.

Some important earlier work, moreover, supports the scheme in Figure 8 with respect to the ability of moisture to collect at adhesive interfaces. The phenomenon of capillary condensation [33–35] has been cited as playing an important role in polyimide adhesion [36]. Wu et al. [37] and Kent et al. [38] have used neutron scattering by D₂O to show high concentrations of water at polyimide and polyurethane interfaces, respectively. In both cases the concentration of interfacial water without adhesion promoters was significantly higher than with them. Nguyen et al. [39] have used infrared spectroscopy to measure the accumulation of water at an epoxy/SiO₂ interface and correlate it to weakened adhesion. Loss of adhesion and accumulation of water at the interface were both accelerated in the absence of adhesion promoter. As mentioned above, prior work [18, 32] has shown that mechanical stress at an adhesive interface can accelerate the accumulation of water at that interface. What we are investigating here is the role of the interfacial water in adhesion failures during subsequent high-temperature excursions.

For the subsequent discussion of wedge test results, therefore, the scheme in Figure 8 is a useful working hypothesis. The results are summarized as follows (with each result substantiated and interpreted in subsequent paragraphs and figures):

1. Epoxy delamination detectable by acoustic microscopy appears only after solder reflow.
2. Longer wedge stressing below the T_g of the adhesive induces larger delaminations.
3. If the wedge stressing is omitted but the moisture exposures and solder reflows are duplicated, little or no delamination occurs.
4. Both types of wedge stress (i.e., during humidity exposure below the T_g of the adhesive and during solder reflow) cause delaminations, but stress below the T_g of the adhesive is sufficient to cause quite large delaminations.

¹D. W. Henderson, personal communication.

²D. Swenson and D. W. Henderson, unpublished results.

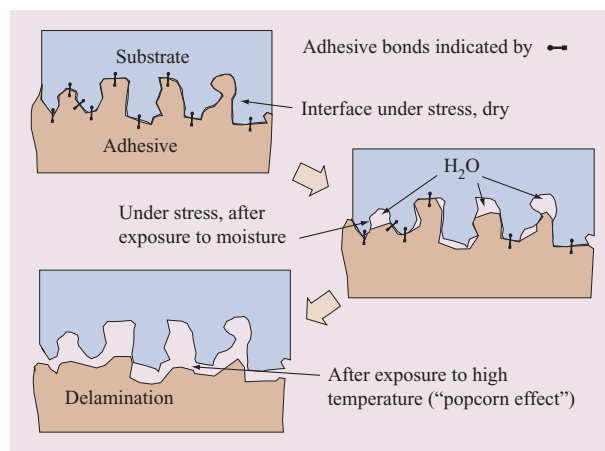


Figure 8

Schematic representation of steps in popcorn delamination.

5. Omitting couplers altogether results in very large areas of delamination.
6. At similar concentrations (about 3–4 at.% Si), Couplers A and B show similar large areas of delamination in wedge testing but show excellent results in cantilever beam testing after PCT.
7. An optimum level of Coupler B exists where delaminations are consistently absent.
8. The wedge test can be used to probe other packaging interfaces.

Delaminations occur after solder reflow. This result is required if the mechanism illustrated in Figure 8 is correct. Damage to the interface is not detectable prior to solder reflow because the moisture and mechanical stress are able to cause only small areas of the interface to separate, i.e., form “proto-delaminations.” The high-temperature popcorn effect causes these areas to grow significantly because of the pressure exerted by water vapor at that temperature. **Figure 9** shows wedge samples before and after solder reflow. Delaminations are absent before solder reflow, but sizable delaminations are detected after solder reflow.

Longer wedge stressing induces larger delaminations. This result is also required by the proposed mechanism. If moisture and mechanical stress are jointly responsible for the formation of proto-delaminations, which expand by the popcorn effect, a longer time for them to act should produce a stronger delamination response. **Figure 10**, in which the sub- T_g wedge stressing was twice as long as that of Figure 9 (24 instead of 12 hours), shows the increased area of delamination caused by the longer stress time. An alternative explanation, that the delaminations are simply the result of moisture absorption and popcorning during

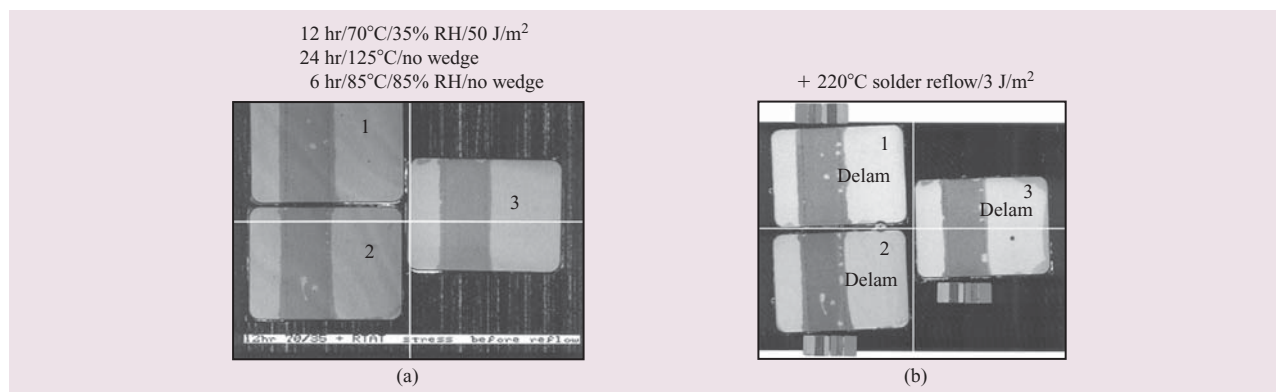


Figure 9

Acoustic microscopy of wedge test samples (a) before and (b) after solder reflow, showing some loss of adhesion at the interface after solder reflow. Similar samples, numbered 1–3, of cured epoxy adhesive on SS treated with Coupler A (3–4 at.% Si), were exposed to the given sequence of conditions. After the sonoscan images were made, the samples (see small sample images) were peeled open by hand to confirm the delamination. The delaminated area, matching the sonoscan image, could be clearly seen with the naked eye in the peeled samples.

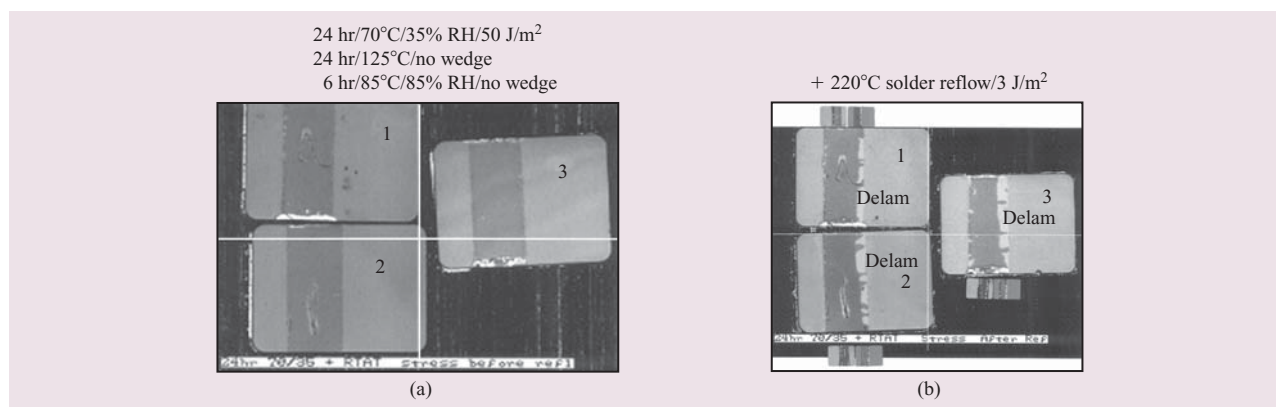


Figure 10

Acoustic microscopy of wedge test samples (a) before and (b) after solder reflow, showing more extensive damage caused by longer sub- T_g wedge stressing. The three samples were identical in preparation and treatment to those of Figure 9 except for an increase in the sub- T_g wedge stressing time from 12 to 24 hr.

solder reflow, would not explain this result. The inclusion of the 125°C bake-out followed by 6 hr/85°C/85% RH (see the section on experimental methods) was designed to equalize the moisture content of all of the samples prior to solder reflow. For this reason, it can be argued that the key factor determining the level of delamination that occurs during solder reflow is not how much moisture has penetrated into the sample but the extent of damage at the interface brought about by the combination of mechanical stress and moisture.

Little or no delamination occurs without wedge stressing. This result strengthens the above argument. Acoustic microscopic images were recorded on three sets of

samples prepared and exposed to moisture and solder reflow in exactly the same manner as samples with wedge stressing. The delamination results are summarized in **Table 2**. Note that a moisture exposure time of 48 hours showed no delaminations, in contrast to the sample in Figure 9 that showed delaminations after a moisture exposure time of only 12 hours with wedge stressing.

All plastic packages absorb some moisture because of the nature of polymeric materials; they contain too much free volume to block the diffusion of water, and they contain polar bonding sites to bond with water molecules. The challenge then is not how to keep moisture out, but how to prevent it from causing damage. The significant

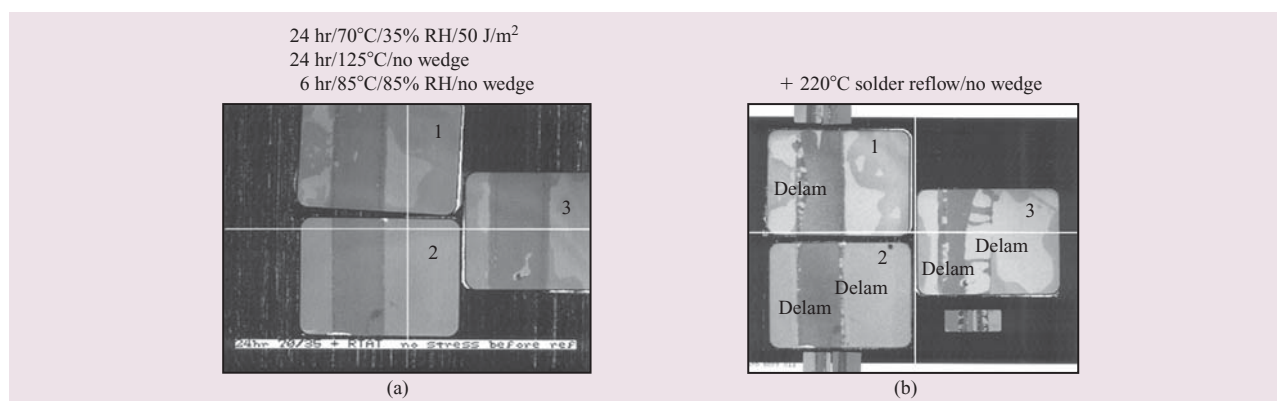


Figure 11

Acoustic microscopy of wedge test samples (a) before and (b) after solder reflow, showing that loss of adhesion does not require wedge stressing during solder reflow. The three samples were identical in preparation and treatment to those of Figure 10 except for omission of the wedge during solder reflow.

lesson here is that mechanical stress can work in concert with moisture to cause interfacial damage other than that caused by moisture alone. This finding implies that plastic packages can be made more robust without excluding moisture if the interface can be strengthened or if the mechanical stress can be reduced.

Stress below the T_g of the adhesive is sufficient to cause quite large delaminations. The mechanism illustrated in Figure 8 hypothesizes that stress together with ambient conditions of temperature and humidity causes interfacial damage, or proto-delaminations, that allow further damage (moisture popcorning) during solder reflow. To verify this mechanism, it is necessary to separate the effects of stress at ambient conditions from the effects of stress during solder reflow. It has been assumed for this study that, as long as the adhesive material is kept in its glassy state, or sub- T_g , a temperature higher than ambient could be used to accelerate the combined effects of stress and moisture so that what might occur over days or weeks at ambient conditions could be observed after a shorter period of time. It is evident, however, that the response of the adhesive well above its T_g , as during the rapid rise to solder reflow temperatures, is very different from its response below T_g . Of course, real packages experience both temperature regimes, but to verify the mechanism, it is important to determine whether wedge stressing above room temperature but below T_g is sufficient to induce delaminations without wedge stressing during solder reflow. Previous modeling of the effects of the pressure of interfacial moisture [5, 40] has estimated the vapor pressure of the interfacial water to be comparable to that of the yield strength of epoxy materials above their glass transition temperatures (3–5 MPa); thus, it is plausible to expect that defects

Table 2 Wedge test samples (SS coupons with epoxy and Coupler A) exposed to test conditions without wedge stress. Test conditions: N hr/70°C/35% RH; 24 hr/130°C bake-out; 6 hr/85°C/85% RH; 3X 220°C simulated solder reflow.

Hours at 70°C/35% RH	Delaminations observed by acoustic microscopy after solder reflow
0	None
24	Small
48	None

introduced by moisture and stress below T_g could cause delaminations to grow at solder reflow temperatures without the assistance of the mechanical stress applied by the wedge.

Figure 11 shows delaminations observed with sub- T_g wedge stressing with no wedge stressing during solder reflows. Together with the near absence of delaminations with no wedge stressing and the progressive increase in delaminations with increasing time of sub- T_g wedge stressing, the significant delaminations apparent in Figure 11 provide strong evidence that the water vapor pressure is sufficient to drive the interfacial failure, as depicted in Figure 8. As mentioned, however, the interfacial water is not the only source of water vapor to drive the delamination, since the absorbed water in the polymer close to the interface can also contribute.^{1,2} The interfacial water should properly be considered as a nucleation site for the formation of water vapor whose

¹D. W. Henderson, personal communication.

²D. Swenson and D. W. Henderson, unpublished results.

Table 3 Wedge testing results as a function of Coupler B concentration.

Calculated Coupler B amount (μg) (volumetric application by syringe)	Wedge test samples delaminated
None	4 of 4
0.15	4 of 4
1.5	1 of 4
2.06	0 of 4
15.5	0 of 4
20.6	0 of 4
206	4 of 4

expansion at high temperatures drives the failure at the interface.

Omitting coupler results in large areas of delamination. The function of an adhesion promoter (coupler) is to provide covalent bonding between an adhesive and the surface to be bonded, i.e., across the interface. Under the proposed mechanism for the combined effect of mechanical stress and moisture, it is clear that the coupler is the first line of defense against displacement of adhesive by moisture. Thus, it is reasonable to expect that omitting the coupler would result in very poor results in the wedge test, if the mechanism is correct and if the test is providing meaningful results. Large delaminations were in fact observed without a coupler.

Similar concentrations of Couplers A and B show similar levels of delamination in wedge testing. Levels of Couplers A and B of about 3–4 at.% Si by XPS could be reproducibly applied to the SS coupons by dipping in dilute isopropyl alcohol solutions of A and B. At this level of either Coupler A or B, the test coupons routinely passed the cantilever beam adhesion screening test (with PCT) with virtually no delaminations. Both, however, showed large delaminations in the wedge test. Remarkably, the very aggressive moisture conditions of PCT are not sufficient to cause delaminations, in contrast to the results from quite moderate moisture conditions in combination with mechanical stress and a subsequent high-temperature excursion (the simulated JEDEC preconditioning [30] of the wedge test). It is significant that the dip technique commonly used to apply adhesion promoters gives apparently acceptable results from the cantilever beam test for both Couplers A and B, but the wedge test shows potential problems with both. In experiments outside the scope of this paper,³ application methods were found to reproducibly apply Coupler B at higher concentrations than Coupler A. It is sufficient to note that Coupler B, with its basic amine groups, can

be rapidly hydrolyzed, in aqueous or partly aqueous solution, to its active trisilanol form [41], which facilitates bonding of a thicker layer of the coupler to the SS surface. Coupler A, lacking amine groups, is activated more slowly; attempts to accelerate the hydrolysis by adjusting the pH of the solution can easily hydrolyze the epoxy groups of Coupler A as well as the silyl ether groups, with probable detrimental effects on its ability to function as a coupler.

An optimum level of Coupler B can be found using the wedge test. According to Plueddemann [42], silane couplers are effective not only because they provide covalent bonds across the interface but also because these bonds are dynamic; i.e., they can relieve stress by breaking at one site and reforming a short distance away. If this picture is correct, there may be an optimum density of bonds across the interface. If there are too few bonds, breaking a few will be sufficient to separate the adhesive from the surface. If there are too many, the added rigidity imparted by the additional bonds may concentrate the mechanical stress locally, causing adhesion loss. In addition, if the coupler thickness is excessive, the cohesive strength of the coupler layer may not be high enough to prevent failure within the coupler. For these reasons, use of the wedge test to compare the robustness of adhesion at a wide range of coupler concentrations was an attractive possibility.

Table 3 summarizes the results for test coupons to which Coupler B was applied in a broad range of concentrations. The best way to control the relative amount of Coupler B applied to the coupons was to use a different method of applying the coupler to the coupons. The usual method of dipping the coupons in dilute 2-propanol solutions would not allow the amount of coupler on the surface to be precisely controlled. Instead, a coupler solution of known concentration was applied volumetrically, using a syringe, to the surface of the coupon. After evaporation of the solvent at room temperature, the coupler was baked on as usual, and the wedge test was then performed. Then, by varying the concentration or volume of solution, a wide range of coupler amounts could be tested. Because of this change in the method of coupler application, the results for these coupons were not directly comparable to those for coupons treated by the normal dip method. By various estimations, however, the coupler concentration for the best dip process for Coupler B (6–8 at.% Si) was roughly equivalent to 2–10 μg in Table 3.

The wedge test can be used to probe other packaging interfaces. This paper has been focused on the model structure of one particular epoxy adhesive bonded to stainless steel. However, the test can easily be extended to real package interfaces such as those between underfill and solder mask, underfill and chip passivation materials,

³K. W. Lee, M. Gaynes, and S. L. Buchwalter, unpublished results.

die attach and chip, and heat sink adhesive and chip. It is necessary to apply a thin film of one of the materials on the test coupons and then perform the test as described herein. The choice of which material to apply to the test coupons as the thin film is usually obvious, because it is important to replicate the order of deposition in the actual package. As an example, to model an adhesive bonded to the back side of a silicon chip, silicon dioxide can be sputter-coated on the SS coupons and the wedge test performed with the epoxy adhesive. The results of such an experiment with Coupler B applied to the sputtered silicon dioxide prior to bonding the adhesive gave excellent results in the wedge test.⁴ Similarly, coating SS coupons with chip passivation films and substrate solder masks allowed the wedge test to be used to compare the adhesion of various underfills.⁴ Because there is an additional interface introduced between the test coupon and the film material, careful failure analysis must be performed to ensure that the interface of interest is the one failing.

Conclusions

Polymeric materials and their interfaces with other materials in microelectronic plastic packages are almost always subjected to moisture and mechanical stress as well as exposure to high temperatures during solder reflow cycles. This paper has described two test methodologies aimed at learning how to optimize the adhesion at a polymeric interface while minimizing the need to build and test actual packages. The cantilever beam test with PCT was found to provide a useful, coarse level of screening between surface treatments, but it failed to predict the performance of an epoxy/stainless steel interface under realistic package conditions for three reasons. First, exposure to moisture at the increased temperature and pressure of PCT actually weakened the bulk properties of the adhesive sufficiently to cause cohesive failures to appear when the interface might otherwise have failed. This unexpected result was a reminder that characterization of the response of the bulk materials to stresses is a requirement for developing predictive adhesion tests. Second, the samples used in the cantilever beam test were not exposed to moisture and mechanical stress simultaneously, in contrast to real package interfaces. Third, no solder reflow simulations were performed on the samples.

The design of the wedge test addressed these deficiencies and, more significantly, it elucidated several important insights about adhesive interfaces in plastic packages. First, the combination of moisture and mechanical stress does act synergistically to weaken the adhesive interface, particularly with respect to subsequent

solder reflows. The likely explanation for this observation is that the mechanical stress at the interface accelerates the displacement of the adhesive by moisture. Second, the root cause of moisture-driven delaminations at adhesive interfaces during high-temperature exposures such as solder reflow, known as popcorning, is not moisture absorption into and diffusion through the plastic materials. Plastic packages are inherently non-hermetic, but the wedge test results with Coupler B show that the interfaces can be made robust to moisture by modifying the surfaces to be bonded. Third, moisture popcorning applies forces on the interface greater than any applied by mechanical stress. This conclusion can be inferred from the fact that, after moisture stressing but before solder reflow, acoustic microscopy or peeling apart the test samples after moisture stressing did not show delaminations at all comparable to those observed for similar samples subjected to solder reflow. Referring to the schematic representation of the popcorn mechanism in Figure 8, the argument is that moisture and mechanical stress nucleate the damage to the interface, but it takes the forces applied by vaporization of water during solder reflow to drive the delamination.

Finally, it can be concluded that the wedge test is useful for evaluating the robustness of adhesion against moisture popcorning, thus providing a predictive test for developing robust interfaces in new packaging structures. Although this paper has been limited to the model structure of one particular epoxy adhesive bonded to stainless steel, the test can easily be extended to real package interfaces such as those between underfill and solder mask, underfill and chip passivation materials, die attach and chip, and heat-sink adhesive and chip.

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