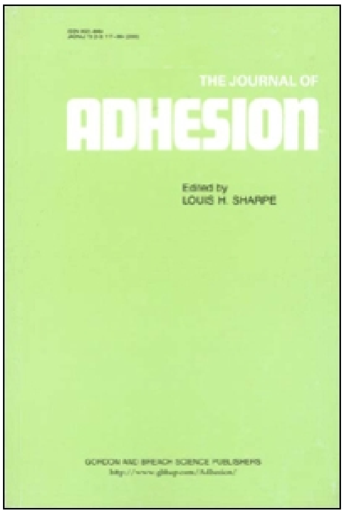

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# Relative Adhesion Measurement for Thin Film Microelectronics Structures

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# Relative Adhesion Measurement for Thin Film Microelectronics Structures*

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There is a difference between technologically-important adhesion or practical adhesion, and fundamental or basic adhesion. What is important in the understanding of fundamental adhesion may be of insignificant interest to technology. A manufacturer is interested in how to improve the reliability of the structure being built should an interface problem exist, rather than knowing the precise value of the fundamental adhesion. It is not possible to measure fundamental adhesion for technologically-important structures due to the inability to account for all energy dissipating processes during the test. Adhesion measurements are plagued with the mode of interface loading issue: the resemblance of test interface loading to that of the actual manufactured part. What technology needs is a simple adhesion test method that is practical for product development, giving reliable information about the interface integrity. The present paper compares the value of two adhesion tests for microelectronics applications and emphasizes the importance of locus of failure analyses. A realignment of structure reliability modeling is suggested by the usage of effective fundamental adhesion instead of the standard undeterminable fundamental adhesion.

*Keywords:* Adhesion measurement; modified edge lift-off test; MELT; peel test; locus of failure; fundamental adhesion; practical adhesion

## 1. INTRODUCTION

What is the purpose of adhesion testing? Is it to find the weak interfaces? Usually not, as the weak interfaces tend to become

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apparent during the development cycle of a new product. Therefore, the more likely purpose of adhesion testing is to find ways to improve the known weak interface. The weak interface can be made stronger either by improving the cohesive strength of the materials forming the interface or by improving the interfacial bonding. The trick is to know which one of these two should be done.

In spite of the great progress in the field of adhesion during the past several years, interfaces continue to present challenges to microelectronics structures containing them. These challenges used to be primarily due to one variable: surface cleanliness. The case is somewhat different today as our attention is directed to low dielectric constant insulators that are porous, taking advantage of air's fundamentally low dielectric constant. Porosity can cause cohesive weakness in a material so much so that some standard cross-sectional analytical procedures are not possible. For example, porous glasses can be so weak that the attempts to make transmission electron microscopy (TEM) samples can result in pulverization of the insulator. Changes in the type and shape of porosity as well as pore surface chemistry can be of significant importance to the material's cohesive strength. Today, more than ever before, the cohesive strength of the insulator is perhaps more important than the adhesion itself. The microelectronics industry needs a well-characterized methodology to determine the reliability of multilayer structures it attempts to build with significantly weaker materials than was done during the days of standard silicon dioxide ($SiO_2$) or polyimide (PI) insulators.

The problem can be approached from two different directions: (1) Chemistry, and (2) Mechanics. These approaches will meet, as both are instrumental in the understanding of adhesion, which is an important component of the process development for next-generation device or packaging structures. Adhesion is influenced by the substrate surface characteristics. Problem solution from this vantagepoint focuses on surface chemistry related issues: is the surface clean of contaminants, and is its chemistry right for bonding with the material being deposited on it? Mechanics, on the other hand, focuses on the issues of the interface loading and the fundamental adhesion calculation. It is important that the loading during the test, as well as yielding conditions, relate well to the device or packaging structure interface loading characteristics and material yielding. If not, it is difficult to extend the data from the measurement to application. Assuming that loading and yielding conditions are appropriate, the fundamental adhesion calculated can then be used as a variable in structure modeling to determine its long-term reliability. Having mechanics and chemistry of adhesion work together can potentially result in relatively complete understanding of the interface at hand. However, most often the loading and/or yielding conditions are not known in the test or in the application. Neither is there a method to assure that true fundamental adhesion is ever measured in practical adhesion tests. Gent and Hamed's statement from 1978 still holds [1]: "Herein lies one of the basic problems in adhesion – the inability to obtain from a simple mechanical test a measure solely of the interaction across the interface." That is what fundamental adhesion is: the *chemical* interaction across the interface.

This report will address the above issues in some detail, comparing two of the more popular adhesion measurement techniques. The paper is organized as follows:

(1) Adhesion measurement: purpose and definitions
(2) Peeltest
(3) Modified edge lift-off test (MELT)
(4) Locus of failure characterization
(5) Summary

The peel test and the MELT were chosen because they are more practical for microelectronic applications, more easily implemented and they allow locus of failure characterization with surface-sensitive analytical tools. MELT is the newer one of the two techniques and, therefore, the comparison of it with the well-known and characterized peel test may serve the adhesion community well at this time. Covering the plethora of adhesion tests reported in the literature would be prohibitive and take too long to discuss in any detail within one publication – even if it were limited to tests applicable to thin film adhesion characterization.

The main purpose of this paper is not only to compare the two tests but also to address the issue and necessity of the fundamental adhesion. Can the fundamental adhesion be measured and is it important for the purpose of microelectronics structure fabrication and adhesion problem solving in the same?

## 2. ADHESION MEASUREMENT: PURPOSE AND DEFINITIONS

There are three primary purposes for the adhesion testing: (1) to rank materials, (2) to improve adhesion at poor interfaces, and (3) to determine the fundamental adhesion. At this juncture it is appropriate to define the fundamental adhesion, which is the energy required to break the bonds at the weakest plane in the adhering system under the adhesion measurement conditions used [2]. The fundamental adhesion value should in theory correlate with the bond energies across the interface per unit area. Therefore, if one were to know the type of interface bond and the bond density, the fundamental *interfacial* adhesion may be calculated. In the same manner, the fundamental adhesion may be calculated if bond type and density are known at the locus of failure. It should be noted that the weakest plane, as well as the energy required to break the bonds, may change as the measurement conditions change (*i.e.*, interface loading) [3, 4]. Practical adhesion [2] is defined as the force or the energy required to disrupt the adhering system irrespective of the locus of failure. This includes the component representative of fundamental adhesion and the energy dissipated in a number of other processes during the testing.

To rank materials or to improve adhesion at any given interface, any adhesion test will work which has a mode of interface loading and yielding conditions resembling that in the application and which can show the relative differences between interfaces of interest.

The values measured represent practical adhesion. To determine the fundamental adhesion will require a test that will allow separation of the other energy-dissipating processes from the fundamental adhesion. In many, if not in all, practical measurement techniques for adhesion, it is difficult to account for all energy-dissipating processes such as energy dissipated as heat [5], in sonic emission, in fractoemission [6], or locally in the deformation ahead of the crack front [7]. Corrections accounting for macroscopic deformations are usually possible. *To determine the fundamental adhesion requires that all energy-dissipating processes be accounted for.*

There are two important issues, alluded to above, which need to be considered explicitly: (1) interface mode of loading (the ratio between crack opening mode (mode I) and shear or sliding mode (mode II) [8];

mode III, tearing, is ordinarily omitted), and (2) yielding conditions (stretching of materials beyond the yield point resulting in plastic deformation). Interface mode of loading should resemble that in the application. This is important as both the locus of failure and the strain energy release rate can change as the mode of loading changes. It is not possible to conclude that if the locus of failure is at one interface that the other would be stronger [9]. This is only true at the mode of loading used. Therefore, it is imperative that the mode of loading is known both in the test and in the application. However, this most often is not the case, resulting in ambiguity between the measured data and the application.

The yielding conditions in the test and in the application need to be the same for the measured parameter to exhibit transferability from test to application. Often tests, which are convenient to perform, do not reflect the yielding conditions in the application [10].

The issue of yielding is particularly important with tests employing large strains, such as the peel test.

## 3. PEEL TEST OF ADHESION

Kim *et al.* [11 – 13] report that peel force ($p$) is a function of fracture energy (or fundamental adhesion [11, 14]), $\gamma$, and work expenditure, $\varphi$:

$$p = \gamma + \varphi \tag{1}$$

The work expenditure is caused by the plastic deformation of the adherate (adherate = the adhering film, adherend = the substrate to which the film adheres [14]), which is assumed to be primarily due to its bending during the peel. $\varphi$, therefore, is primarily the energy used for the adherate deformation. The critical factor in the evaluation of $\varphi$ is the peel strip maximum curvature, $K_B$, which is related to the (lift) base angle at the peel crack tip [15]. Figure 1 describes schematically the difference between the applied peel angle ($\theta$, often 90°) and the base angle ($\theta_B$). Kinloch *et al.* [7] point out that even if the applied peel angle were varied by about 100°, the base angle may experience only a 25° change. Park and Yu [15] describe an X-ray diffraction method to estimate the work expenditure during peeling of Cu-films off PIQ

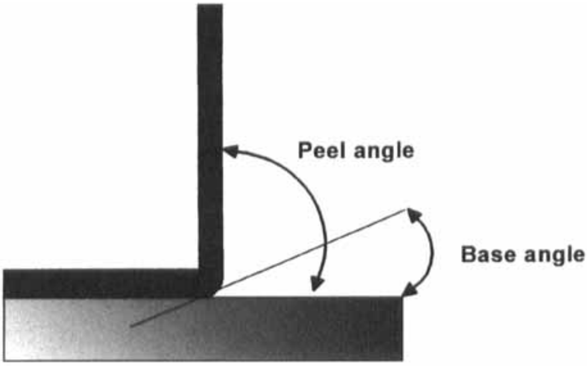

FIGURE 1 Schematic of peel test with the applied and the base peel angles.

polyimide (PI) coatings on Si-wafers. The method uses Cu(331) peak broadening and a calibration plot to estimate the plastic strain, $\varepsilon^P$, remaining in the film. The calibration plot relates the difference in the Cu(331) peak full width at half maximum (ΔFWHM) to the plastic strain. The ΔFWHM is determined as follows:

$$\Delta\mathrm{FWHM} = \mathrm{FWHM} - \mathrm{FWHM}^0 \qquad (2)$$

where

$\mathrm{FWHM} = \mathrm{Cu(331)}$ peak FWHM after uniaxial tensile testing
$\mathrm{FWHM}^0 = \mathrm{Cu(331)}$ peak FWHM at time zero (as deposited film)

The $\varphi$ estimated using the X-ray diffraction method was compared with numerically calculated $\varphi$ (Moidu, Sinclair and Spelt [16, 17]). This comparison yielded reasonably consistent $\varphi$-values in most cases [18]. Having evaluated the $\varphi$, consideration of the peel force *vs.* fracture energy (fundamental adhesion) based on Kim *et al.*'s Eq. (1) is now possible with the following results [15, 18]:

1. Fracture energies are relatively independent of the peel strip thickness ($\gamma$ should be independent of the film thickness), as shown in Figure 2. Notice: Figure 2 uses $\Gamma$ for $\gamma$ and $\Psi$ for $\varphi$.
2. The peel force correlated well with the $\Gamma$ showing $P \sim (3.5 \text{ to } 8)\,\Gamma$ dependence (Cu/Cr/PI sample).

The values of $\gamma$ reported in [15–18] are very high, in the few hundred J/m$^2$. Though the loci of failure were not reported in these

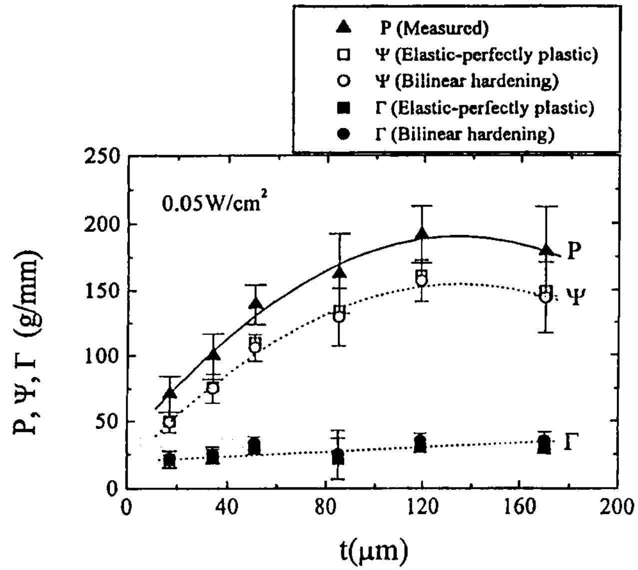

FIGURE 2 Fracture energy ($\Gamma$) *vs.* peel strip thickness ($\gamma$) in micrometers [18]. The figure also shows the peel force ($p$), and the calculated work expenditure ($\Psi$) for elastic-perfectly plastic and bilinear-hardening material as a function of peel strip thickness. $\Gamma = p - \Psi$, which is the same as Eq. (1): $p = \gamma + \varphi$. Reprinted from Materials Science and Engineering: A266, Park, Y. B., Park, I. S. and Yu, J., "Interfacial fracture energy measurements in the Cu/Cr polyimide system", 261 Copyright 1999, with permission from Elsevier Science.

studies, it is likely within the PI film as has been shown in another study of the Cu/Cr/PI system [19] and in several other metal/PI systems [20–22]. The fracture energy (the fundamental adhesion) should then be a measure of the breakage of the covalent bonds in the PI backbone. Assuming we have 2 C—C bonds ($\sim 5.7 \times 10^{-19}$ J/bond) per nm$^2$, fundamental adhesion should be in the order of few J/m$^2$, not a few hundred J/m$^2$. This suggests that the other energy-dissipating processes are not accounted for in the Park *et al.*, analyses [15, 18]. Kinloch *et al.* [7] and Kim and Aravas *et al.* [11] have suggested that the $\gamma$ determined from peel test, after corrections for work expenditure due to adherate plastic deformation, *accounts for the breaking of the bonds at the locus of failure and for the energy dissipated at the crack tip plastic or viscoelastic zone.*

As discussed earlier, there are three other possible ways to dissipate energy during the peeling process: heat, sonic emission, and fractoemission. It may be more feasible, then, to consider an effective fundamental adhesions, $\gamma_{\text{eff}}$:

> *The effective fundamental adhesion ($\gamma_{eff}$) determined from a peel test, after corrections for work expenditure due to adherate plastic deformation, accounts for the breaking of the bonds at the locus of failure, the energy dissipated locally at the crack tip plastic or viscoelastic zone, and energy dissipated as heat, in sonic and fractoemissions* [5–7].

Fundamental adhesion cannot be determined from a peel test because:

(1) The difference between $p$ and $\varphi$ is small, and gets progressively smaller as $p$ increases [11], thereby resufting in larger error in $\gamma$.
(2) It is very difficult to account for all the energy-dissipating processes during the peel experiment.

However, the correlation of peel force with fundamental adhesion is reasonable in the light of the work done by Kim *et al.* [11, 13]; Moidu *et al.* [16, 17]; Park *et al.* [15, 18] and Kinloch *et al.* [7]. The peel force appears to be a function of fundamental adhesion so that when the latter increases so does the measured peel force.

It is reasonable to consider that the local energy dissipation at the peel crack tip is a function of $\gamma$, so that when $\gamma$ increases the energy dissipation at the crack tip increases. The same can be said about dissipation of energy as heat [5]. Even though the peel test is not able to give actual fundamental adhesion it can be used for relative measurement with confidence. Using the appropriate corrections delineated in Refs. [7, 11, 13, 15–18] (removing the peel angle and film thickness dependency resulting in a geometry-independent property) results in an effective fundamental adhesion, $\gamma_{\text{eff}}$. This correction process can further be simplified: Since the peel force is a function of effective fundamental adhesion [15–18, 23, 24] so that increase in peel force indicates an increase in $\gamma_{\text{eff}}$ (Fig. 3 schematic; Gent [23] has shown this type of peel force dependence on interfacial chemical bonding with polybutadiene/silane adhesion promoter/glass structures), the *peel force* (*force required to separate the interface per unit width of the peel strip*) *itself can be used directly as a relative measure of adhesion* a **peel adhesion** measure, *if the following conditions are met*:

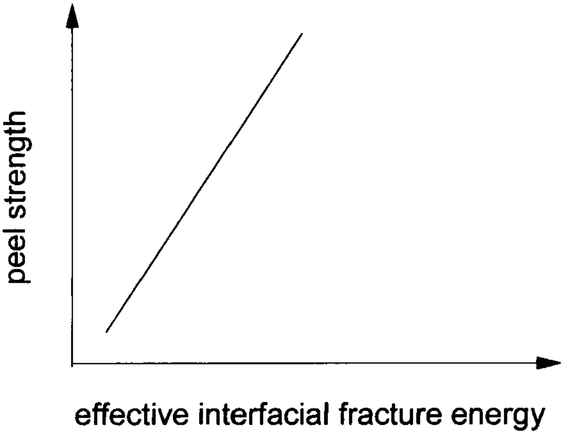

FIGURE 3 Peel force as a function of effective fundamental adhesion [50]. Effect of Interfacial Bonding on the Strength of Adhesion, A. Ahagon and A. N. Gent, *J. Polym. Sci.: Polym. Phys. Ed.*, Copyright ©1975 John Wiley & Sons, Inc.. Reprinted by permission of John Wiley & Sons, Inc..

1. Peel film thickness and substrate are constant [12, 13, 25, 26]
2. Peel rate is constant [27–29]
3. Peel ambient is controlled ($N_2$ flushed enclosure at RT adequate) [27, 29–31]
4. Macroscopic peel angle is constant [7, 32, 33]
5. The test material film thickness is insignificant relative to the peel backing material and substrate thickness so that these, not the test material, drive the mechanical properties of the sample. If this is not possible, then the film thickness within a test matrix for an interface is to be kept constant. In this case, it will be difficult to compare directly the peel force values of one polymer or metal with another since the differences in the mechanical properties will affect the results. However, improving the reliability of an interface by surface cleaning and/or by using an adhesion promoter can be safely addressed with the peel test even when test film properties in the peel strip mechanical properties cannot be ignored.

Small changes in the effective fundamental adhesion seem to result in large changes in the peel force [15, 18, 28]. This can be considered advantageous as there is then a higher level of distinction between the samples.

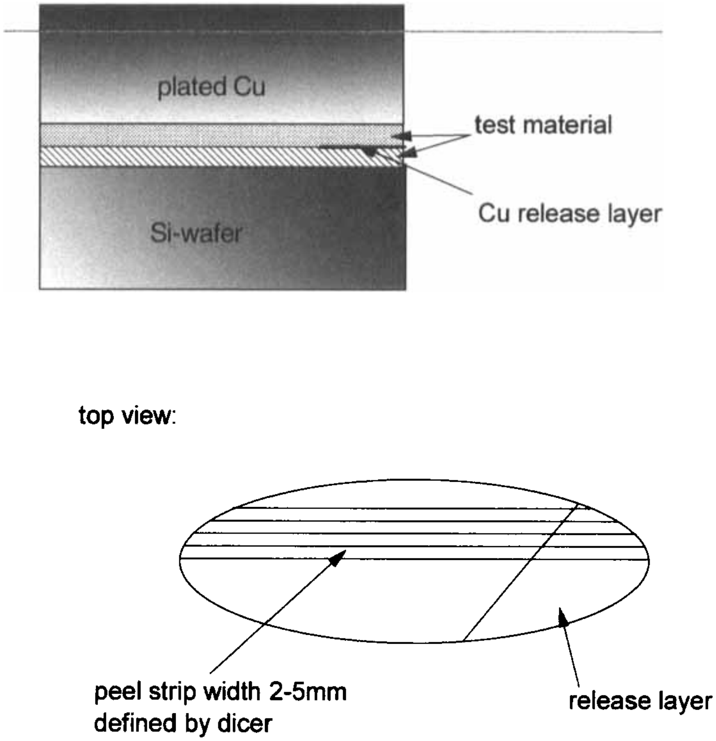

FIGURE 4 Peel test sample preparation.

A sample preparation scheme for a peel test is shown in Figure 4: The Cu-release layer has been found to be effective in initiating the peel for the testing of many interfaces. The goal of the release layer is to create a pre-crack, a weak area, where peel initiation does not require much energy. The release layer selection depends on its adhesion characteristics to either the material on substrate or the film material. A poor interface with the substrate or the film will be sufficient.

The backing material can be something other than Cu. The requirement is to be consistent within a given study. PMDA-ODA PI, for example, has been used successfully as a backing material.

One of the major issues with the peel test is yielding (*i.e.*, plastic deformation of the peel strip) as very large strains (20–30%) are not impossible. This presents a problem with yielding conditions as compared with the application. The strains in the present-day CMOS structures, for example, are miniscule – nearly zero strain conditions. The amount of strain is dependent on the peel strip properties as well as on the fundamental adhesion. Generally speaking, the higher the fundamental adhesion the larger the strain in the strip. This, therefore, should not change the trend: increased peel force, increased $\gamma_{\rm eff}$.

In the special case where the peel force (*i.e.*, fundamental adhesion) is low, the yielding is not quite as problematic since strip deformation is minimal.

The last issue of concern is the interface mode of loading during the peel testing *vs.* the interface mode of loading in the actual structures. This problem, of course, is not unique to peel but will be a question for any adhesion test used. Detailed test and application structure modeling should be done to address this issue. Most often, the mode of loading is not known in the test or in the structure. This is troubling as the mode mixity does affect the results. It has been reported that in an epoxy system where the loading was predominantly mode I, the crack propagated within the epoxy instead of the weaker interface (with aluminum, steel, or brass) [4]. Strain energy release ($G$), a measure of adhesion, is a function of the mode mixity [34]. It is, therefore, clear that mode mixity in the test should correspond to that in the application. Without this, the only way that adhesion testing can be made meaningful is to compare the test results with the actual microelectronic structure reliability.

## 4. MODIFIED EDGE LIFT-OFF TEST (MELT)

The modified edge lift-off test (MELT) is the newer one of the two tests chosen for this discussion. MELT [35] is a spin-off from the edge delamination test (EDT) proposed by Shaffer at the 1993 Materials Research Society Spring Annual Meeting [36]. The difference between these two tests is in the sample preparation: EDT utilizes lithographically-defined vias of different diameter, while the MELT uses the cut edges of the sample as the stress concentration sites for delamination [35]. MELT has gained more interest because sample preparation for MELT is simpler and quicker than it is for EDT. MELT sample preparation requires a thick (150 – 250 μm), doctor-blade-applied epoxy coating. The stored strain energy in the thick film is used to drive the delamination.

Lowering the sample temperature using liquid nitrogen cooling [37–40], as shown in Figure 5, increases the available energy stored.

A sample preparation schematic for MELT is shown in Figure 6.

After curing the epoxy coating, the wafer is either diced or snapped at about the center of the 5 mm-wide, release-layer strips into about 25.4 mm square pieces for testing. The release layer provides a pre-crack, thereby, alleviating the excessive energy associated with crack initiation so that the test is focused on crack propagation. The test may also work successfully without the release layer because of the cracks generated during dicing or snapping of the samples to proper test size. The theories to calculate the adhesion from the test results assume the presence of a pre-crack.

The MELT test equipment consists of a stage where the sample can be heated by hot $N_2$ and cooled by liquid $N_2$. The heating capability is important, as the stresses in the epoxy film will relax even at RT due to its viscoelastic nature, as shown in Figure 7 [41]. The sample is heated to temperatures above the glass transition temperature of the epoxy film, thus, normalizing the stress for all samples. This process will assure that every sample tested will have the same starting stress. To emphasize further the importance of the anneal step, Figure 8 [42] shows a schematic of possible problems if this is not done. It is obvious

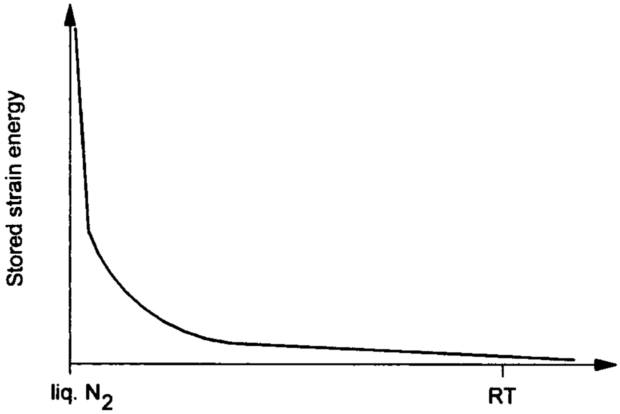

FIGURE 5 Schematic of stored strain energy in an epoxy film as a function of temperature.

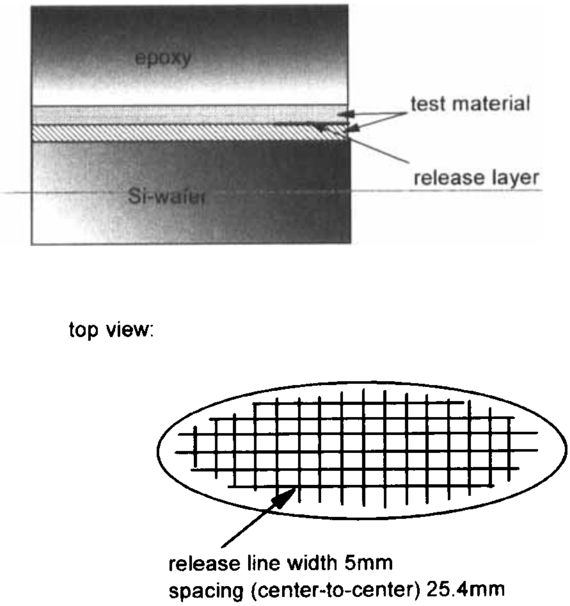

FIGURE 6 MELT sample preparation.

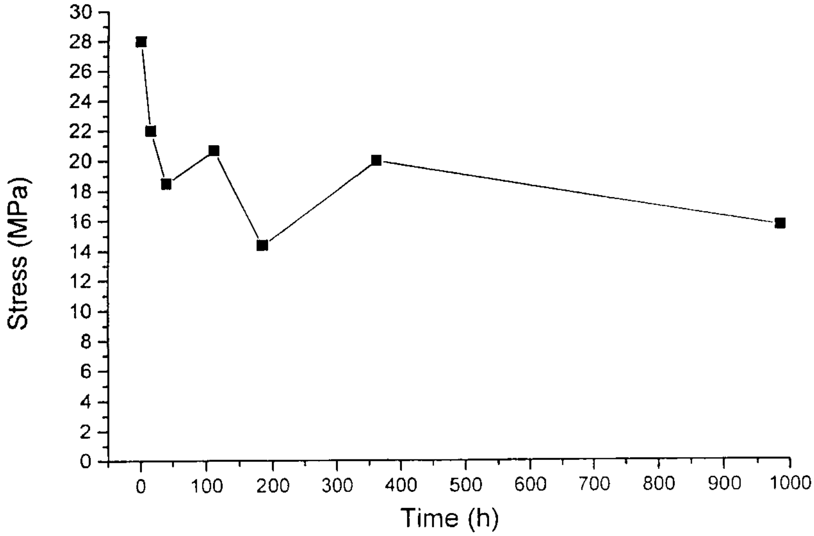

FIGURE 7 Epoxy film stress relaxation at room temperature [41].

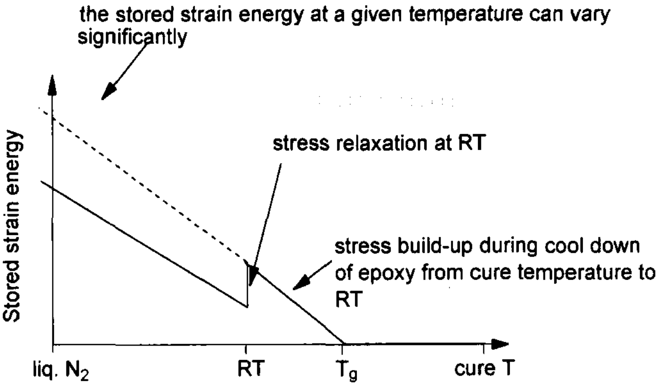

FIGURE 8 Stored strain energy in epoxy film with (a) and without (b) room temperature stress relaxation [42].

from the figure that, if the heat step is omitted, the stored strain energy in the epoxy film at a given temperature can vary significantly. The temperature at which the delamination onset is observed is used as the temperature to determine the stress level in the epoxy film from a stress-temperature calibration plot. This plot has been determined for each epoxy lot used in the test after annealling past $T_g$ and using the same cooling rate as is being used for the test itself (3 – 5 C/min).

The stored strain energy in the film is increased by cooling the sample after annealling. The cooling method is that of convection rather than conduction. Conduction cooling is dependent on intimate contact between the sample and the cooling plate, which cannot be guaranteed because the stress tends to warp the sample. With convection, the cooling rate is well controlled, but slow.

The temperature at which the film debond is initiated from the substrate is recorded. The estimation of adhesion with this test requires the knowledge of the stress of the film causing the delamination to occur. It is assumed that the mechanical properties (*i.e.*, the driving orce) of the epoxy/test film/substrate stack are driven by the thick epoxy and the thick substrate relative to the very thin ( $< 1\ \mu m$) test films. If the epoxy film thickness is 200 μm while the test film thickness is 0.5 μm then the volume fraction of the test film is only $0.25 \times 10^{-2}$. As a rough estimation it can be said that the mechanical properties of the delaminating film are that of the epoxy, providing that the test film properties are not significantly different from those of the epoxy film. More accurate estimation should include the effect of the test film on the bending stiffness [43]. This calculation, keeping the film thickness ratio as above, shows that when the Young's moduli of the test and backing materials are similar, the correction would be on the order of about one percent. However, if the test material's Young's modulus is significantly higher than that of the backing material the situation is quite different, potentially resulting in significant error. The details of the calculation can be found in the Appendix.

From the debond temperature, the residual stress, $\sigma_0$, is determined using a calibration curve (determined using the same cooling rate as in the test) for the backing epoxy layer. The effective fracture toughness of the sample is calculated from:

$$K_{\mathrm{eff}} = \sigma_0 (h/2)^{1/2} \tag{3}$$

where $h$ = backing layer thickness in meters. Equation (3) requires that an existing crack of length "$a$" be present between the rigid substrate and the coating (thickness "$h$") so that the $a/h > 0.025$ [35].

The $K_{\mathrm{eff}}$ calculated using the above equation is, strictly speaking, accounting only for the crack-opening mode. The development of the MELT [35] was done using epoxy coatings on glass. Both the compact tension $K_{\mathrm{IC}}$ values and the MELT (ELT, as it is called in Ref. [35]) values were determined. The MELT failure location was 25–50 nm into the epoxy as determined by ellipsometry. This is a cohesive failure in the epoxy. The $K_{\mathrm{IC}}$ and the MELT data correlated well, which resulted in the suggestion of pure mode I failure in this test. Whether this is necessarily true in all cases of testing may be considered immaterial from an engineering point of view. Assuming a pure mode I failure gives a minimum fracture toughness [10], thereby building in a safety factor for the manufacturing. Since the mode mixity is most often not known for the application or the test, calculating pure mode I fracture toughness is a reasonable experiment. The actual mode mixity, however, should stay constant within an experimental matrix for an interface so that reasonable data comparisons can be made. In reporting any adhesion data, the mode mixity should also be reported (this is often not done) [4].

A physically more appealing quantity for describing adhesion is the strain energy release rate, $G$, for a unit area of delaminated interface [4, 44]. The strain energy release rate and the fracture toughness are related (for a homogeneous system) [10]:

$$G = K^2/E' \qquad (4)$$

where for plane strain $E' = E/(1-\nu)$ and for plane stress $E' = E$

$E$ = Young's modulus
$\nu$ = Poisson's ratio

For a mixed mode, heterogeneous material system, the expression for $G$ is significantly more complicated [4, 10].

True fundamental adhesion cannot be determined from the MELT experiment using the above equations since they do not account for the following energy-dissipating processes:

(1) Energy dissipation in the deformation ahead of the crack tip
(2) Energy dissipated as heat, sonic emission and fractoemission
(3) Calculations assume pure mode I failure

MELT, as well as the peel test, can be used for relative adhesion measurement. MELT, however, may be preferred over the peel test for the following reasons:

(1) No large strains;
(2) Driving force for crack propagation is the strain in the film rather than externally-applied mechanical force.

However, MELT may be limited to poor adhesion cases only. If the interface or film fracture toughness exceeds that of silicon ($K \sim 0.6\,\mathrm{MPa}\,(\mathrm{m})^{1/2}$), the failure will be in the silicon. Fracture toughness is a function of not only rates of loading and thickness of the fractured section but also of temperature [10]. Therefore, the low temperature (significantly lower than actual use temperature of the structure) requirement of the test is a disadvantage of MELT, particularly with polymeric test materials.

The peel test and the MELT have been discussed above in some detail. These two tests were chosen for this publication due to the relative ease of sample preparation, data collection and creation of a large area of the locus of failure allowing utilization of surface analytical techniques for its chemical and physical characterization. Other tests, such as the four-point bending, indentation or scratch tests, were not included due to their time-consuming nature, complicated and expensive sample preparation schemes (four-point bending) or locus of failure analysis difficulties due to inadequate failure size (indentation and scratch tests), for example.

Both the peel test and the MELT can address the practical adhesion changes at a given interface. However, since the mode of loading is not known in the tests or in the structure, the results of the tests will have to be verified with actual microelectronics structure reliability results. In comparing these two techniques, the peel test may be the better choice of the two because it will not be limited in poor adhesion cases. Very high peel values can be measured [24], while MELT may be limited to samples with lower fracture toughness than that of the silicon wafer. MELT experimentation with PI/aminosilane/$SiO_2$ structures should resolve this question.

## 5. LOCUS OF FAILURE CHARACTERIZATION

The precise locus of failure (LOF) determination has been a perennial problem in the field of adhesion as it is dependent on the analytical technique used. Mittal states the following about the LOF analysis:

> "...visual inspection is inadequate to ascertain the locus of failure in a separated system, and techniques like electron spectroscopy for chemical analysis, Auger electron spectroscopy, or secondary ion mass spectrometry should be employed for this purpose..." [14].

Macroscopically (visual analysis) the LOF may appear interfacial, while spectroscopy may suggest failure in the interphasial region (*i.e.*, failure close to the interface but not at it), in one or the other of the materials making the interface, in a new component originating from one of the materials (*e.g.*, plasticizer), or in a reaction products layer. When a clear-cut macroscopic cohesive failure is observed, practical adhesion is then a measure of the cohesive strength of the material. It is not, however, often simple to decide when a true cohesive failure is observed. The macroscopic case is clear, but what about a situation where LOF characterization with surface-sensitive tools indicates failure far enough into one of the materials that no signal of the other material is detected through the residual film? Could this be identified as a cohesive failure? At the present we have opted to define cohesive failure as those cases where XPS analysis done at 35° take-off angle shows no signal of the underlying material. This is, of course, arbitrary, but may be considered reasonable, if the material properties 10–20 nm from the interface are bulk properties [45] (XPS analysis depth is about 5–10 nm).

Why is such detailed characterization of the locus of failure important? In present day microelectronic device fabrication, the film thicknesses range from several tens of nanometers to about a micrometer or so. Consider then, for example, the MELT, where a backing material must be deposited in order to perform the test. The structure of the sample may look like the one in Figure 9.

Failure loci 2, 3, 5, 6, 8 and 9 are failures in the interphasial region close to the interface but not at it. If the LOF would be one of these, XPS analysis would detect some signal from the layer below. For example: LOF 5 would show ILD (interlayer dielectric) characteristics

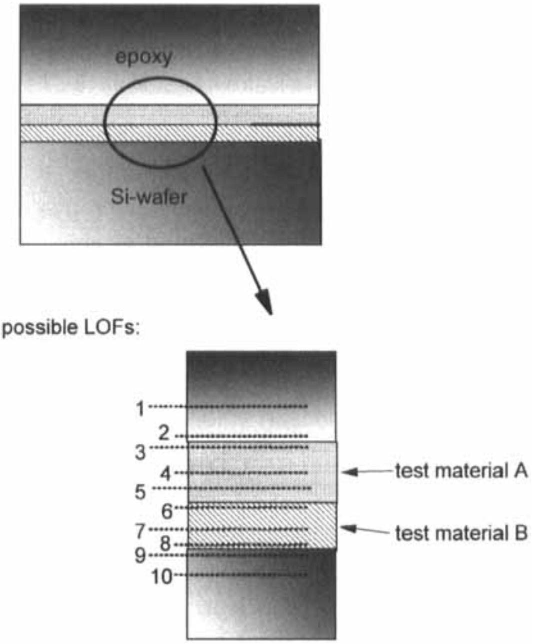

FIGURE 9 Schematic of possible failure loci in an adhesion test specimen. Example: a MELT sample.

in the XPS with some signal from $SiO_2$ below the residual ILD. If no $SiO_2$ signal is detected the failure is considered to be cohesive, *i.e.*, $SiO_2$ surface treatments would not change the adhesion properties at the mode of loading tested. Notice that the above figure does not account for interfacial, mixed mode (failure across the interface from one material to the other and back) or failure within possible reaction products layers. It is important to differentiate between cohesive and interphasial LOF. The latter is about 5–10 nm from the actual interface. With this LOF, surface treatments may improve adhesion. If the LOF is not known, it is not possible to know if the cohesive strength of one of the four materials or adhesion at one of the three interfaces/interphases was measured.

True interfacial failure seldom happens, though evidence of this kind of failure has been reported [46]. The LOF studies, which should include XPS, SEM and AFM, can give the following information:

(1) The actual LOF, which will then drive the approach for adhesion improvement:
    (a) cohesive failure will result in material change
    (b) interfacial or interphasial failure results in adhesion promotion schemes such as surface cleaning and promoter application
(2) Interfacial chemistry
(3) Cracking in the interphasial region
    Cracking perpendicular to the main peel crack path, for example, has been reported [20]
(4) Stick-slip behavior (particularly with the peel test)
    In the case of poor adhesion the stick-slip behavior is not seen. When adhesion is improved the stick-slip behavior becomes pronounced [47].

The above information will be invaluable in the quest for adhesion improvement in any given case.

## 6. SUMMARY

It is clear that the determination of fundamental adhesion from the tests described above is quite unlikely. This holds true for the other several hundred tests reported in the literature [48], since the accounting of all energy-dissipating processes has not been shown to be possible to date. Therefore, it is recommended that one report a $\gamma_{\text{eff}}$, realizing that this value is a measure of the fundamental adhesion but includes other dissipated energy that cannot simply be accounted for in the test used. If fundamental adhesion is considered imperative every effort should be made to determine the amount of energy dissipated in the processes involved in the adhesion measurement, as described earlier. The primary reason for the quest for the fundamental adhesion is modeling of the product integrity and reliability. As it does not seem feasible that fundamental adhesion could be determined with any available adhesion test, perhaps an alternative modeling approach should be considered that would make use of $\gamma_{\text{eff}}$ and not $\gamma$.

Determining the fundamental adhesion may not be of technological importance. What is needed is reliable information about the interface integrity using a simple-enough method that is practical for the research and development cycle of the product as well as adhesion problem solving at the product manufacturing level. A test that uses an interface mode of loading comparable with that in the structure and allows analytical access to the locus of failure is needed. Both the peel test and the MELT are simple in sample preparation, data collection and create large enough locus of failure areas to allow utilization of surface analytical techniques to assess its chemistry. Mode of loading comparability with the actual structure is a concern with both tests, however. Therefore, test results should be compared with actual microelectronics structure reliability results.

MELT is not as well characterized a test method as the peel test is today. There may be issues with MELT, such as limitation to poor adhesion and the possible inability to control the locus of failure in multilevel structures (more work needs to be done on this aspect of MELT) that may limit its utility.

Peel test, on the other hand, is not limited to poor adhesion cases, the locus of failure can often be controlled, and the peel force can be used directly as a measure of adhesion.

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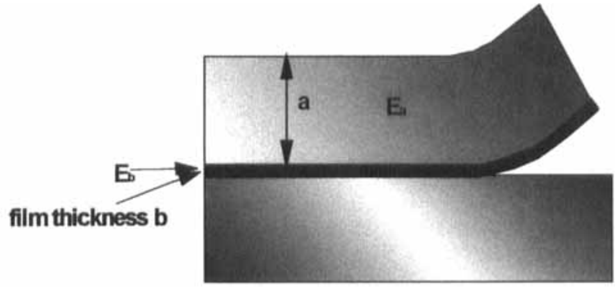
## APPENDIX [43]

### Effect of thin film mechanical properties on the delamination in a bilayer structure.

The figure below shows the structure in question:



It is known that the interface layer will dominate the physicochemical adhesion of the bilayer to the substrate. Since the coating will always bend in order to delaminate, the effect of mechanical properties arises through the bending stiffness of the bilayer. For any beam the product of the moment of inertia ($I$) and the modulus ($E$) gives $S$, the stiffness. The effective stiffness of a bilayer is [49]:

$$S = EI = [(wt_aE_at_b^3E_b)/12(t_aE_a + t_bE_b)]K_1$$
$$K_1 = 4 + 6(t_a/t_b) + 4(t_a/t_b)^2 + (E_a/E_b)(t_a/t_b)^3 + t_bE_b/t_aE_a$$

Or

$$S = wt_b^3E_b/[12(1 + t_bE_b/t_aE_a)]K_1$$

$$K_1 = (t_a/t_b)^3[4(t_b/t_a)^3 + 6(t_b/t_a)^2 + 4(t_b/t_a) + E_a/E_b + (t_b/t_a)^4E_b/E_a]$$

When $t_a \gg t_b$:

$$S = (wt_b^3E_b/12)(t_a/t_b)^3(1 - t_bE_b/t_aE_a + \cdots)(E_a/E_b + 4(t_b/t_a) + \cdots)$$

$$S = (wt_a^3E_b/12)[E_a/E_b + 4(t_b/t_a) - t_b/t_a + \cdots]$$

$$S = (wt_a^3E_a/12)[1 + 3(t_bE_b/t_aE_a) + \cdots]$$

$$= S_0[1 + \mathbf{3(t_bE_b/t_aE_a)} + \cdots]$$

where

$S_0$ = stiffness of a homogeneous beam of thickness $t_a$ and modulus $E_a$

$E_a$, $t_a$ = modulus and thickness of the thick (backing) layer, respectively

$E_b$, $t_b$ = modulus and thickness of the thin (test) layer, respectively

$w$ = mass density

the boldface text is the correction factor, the effect of the test layer on the stiffness of the bilayer structure

Assume $E_a \sim E_b$ and $t_a = 400\,t_b$

Correction factor: $3(t_bE_b/t_aE_a) = 3/400 = 0.75\%$

Assume $E_b = 100E_a$, which can be the case if test material is metal, and $t_a = 400\,t_b$

Correction factor: $3(t_bE_b/t_aE_a) = 300/400 = 75\%$.