Polyimide-Metal Adhesion Measurement Using Microfabricated Structures

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Introduction

Quantitative measurement of the adhesion and mechanical properties of polymer films is a subject which has received much attention [1-3]. Recently, with the advent of polymer films in microelectronics, the need to accurately measure *in-situ* the mechanical properties and adhesion of polymer films as thin as 2 μm has arisen. Many techniques have been developed and/or adapted from thick film techniques to perform the mechanical property measurement [3-5]; however, there exist few techniques which quantitatively measure the adhesion of thin, well-adhering polymer films.

The reason for this is that thin, well-adhered polymer films often are tensile-strength limited when subjected to various adhesion tests. That is, upon attempting to remove the film from the substrate in a controlled manner, the film tears before it peels [6]. Almost every attempted solution to this problem involves strengthening the film in some way. For example, in the standard 90^{o} peel test, a very thick layer of polymer is often built up so that it can be peeled without tearing [7,8]. The problems inherent in this method are twofold. First, it is not clear that the adhesive strength (i.e., the 'interfacial adhesive strength') is the same for a thick film and a thin film of the same material. Second, the peel strength (the observable quantity in the peel experiment) may change drastically as the film thickness increases [9]. Thus, it is desirable to investigate the feasibility of a test which can be adapted to thin films without modifying them in any way.

A closely related test to the peel test is the blister test [10]. In this test, a film is pressurized through a hole in the substrate by a fluid (liquid or gas) until it begins to peel from the substrate. If the blister geometry is known, the work of adhesion can be calculated from the pressure at which peel initiates [11-13]. The blister test has several advantages over the peel test. First, it is not necessary to make mechanical contact to the film to effect peel. Second, since the peel angle in the blister test is small, it is possible that the effects of viscoelastic and plastic deformation of the film will be minimized. It has been demonstrated that blister test sites can be fabricated using materials of importance in microelectronics. Hinkley [14] has succeeded in fabricating suspended polymer films on silicon wafers using a non-lithographic fabrication process. However, the blister test suffers from the tensile strength limitation mentioned previously; if films are thin and/or well-adhering, blisters may burst before peel can be initiated. In spite of this, the blister test offers several ways around the tensile strength limit. These will be discussed below.

One method which has been proposed to overcome this limit is the 'constrained blister test' [15]. In this test, the growing blister is constrained in the vertical direction by placing a plate over it. The plate prevents large deflections in the vertical direction, allowing large pressures to be applied to the blister without tearing the film. The initial measurements using this test were done with adhesive tapes. However, it is possible that films which fail due to defect formation (for example, solvent-cast films as opposed to tapes) will still fail in the constrained blister test, which is of maximum utility for films which fail due to exceeding the maximum strain of the film. In addition, the question of failure due to stress concentration at the edge of the blister is not taken into account. Thus, an alternate method is needed.

The structure we propose to overcome the tensile strength limit is called the 'island blister' [16]. The island blister is a modification of the standard blister site in that the suspended membrane of film has an 'island' of substrate still attached at its center. The island and the substrate are both fastened to a rigid plate and pressure is applied as in the standard blister (Figure 1). Film peeling will now occur only off the center island. It can be shown [13,16] that the pressure necessary to initiate peel can be made low compared to the tensile strength of the film simply by making the center island sufficiently small. Thus, the tensile strength limit of the film can be overcome geometrically. This structure does not suffer from the drawbacks of the constrained blister test in that relatively low pressures are used to initiate and sustain peel; therefore, the issues of stress concentration and defect failure are not important. The theory and fabrication of island test sites and their application to the measurement of adhesion of polyimide films will be discussed below.

Theoretical

The island blisters can be modelled using an energy minimization approach combined with linear elastic fracture mechanics [13]. Modeling the island structure as a circular adhered film area on an island at the center of a circular suspended membrane the load-deflection behavior of which is dominated by residual stress, it can be shown that the pressure to initiate peel (p_c), the work of adhesion of the film (γ_a), and the radius of film still adhered to the island (a_1) are related by (Figure 1):

$$\gamma_a = \frac{p_c^{\,2}\, a_1^{\,2}}{32\,\sigma_0 t}\, f(\beta) \qquad (1)$$

where t is the film thickness, a_2 is the radius of the suspended film, σ_0 is the residual stress in the film, β is the ratio a_2 / a_1, and $f(\beta)$ is a function given by:

$$f(\beta) = \left[\frac{\beta^2 - 1}{\ln \beta} - 2\right]^2 \qquad (2)$$

A plot of $a_1^{\,2} f(\beta)$ as a function of p_c^{-2} should yield a straight line with slope proportional to the product of film residual stress, thickness, and work of adhesion. The film thickness can be determined by a surface profile measurement, and the residual stress can be measured as described below. Thus, the work of adhesion of the film can be obtained from the slope of the above plot.

It can be seen from Equations 1 and 2 that p_c decreases as the size of the center island decreases (i.e., as β increases). In fact, if the size of the center island is arbitrarily small, p_c can be made arbitrarily low (i.e., less than the film's tensile strength limit) independent of the work of adhesion of the film. This is the fundamental advantage of the island blister geometry.

Once the film has peeled, a suspended membrane of the film is formed (Figure 2). The residual stress in the film can be determined in-situ by a measurement of the load-deflection characteristics of the membrane [17,18]. It can be shown that the deflection at the center of the membrane in response to the applied pressure is given by:

$$\left(\frac{Et}{a^4}\right) d^3 + \left(\frac{1.66\, t\, \sigma_0}{a^2}\right) d = 0.547\, p, \qquad (3)$$

where p is the applied pressure, E is Young's modulus, σ_0 is the residual stress in the film, 2a is the site size, t is the film thickness, and d is the deflection at the center of the membrane. If a set of pressure-deflection data are taken, a plot of p/d versus $1/d^2$ is linear with slope proportional to Young's modulus and intercept proportional to the residual stress [18]. Thus, the work of adhesion of the film can be determined from a combination of island peel and load-deflection measurements.

Experimental

Fabrication. Island blister test sites are fabricated using micromachining techniques. On each die (test site), a 5 µm thick square diaphragm ten millimeters on a side with an island of silicon at the center one millimeter in diameter is etched in a (100) silicon wafer from the back using a silicon dioxide etch mask, a 5 µm p^+ diffusion of boron as an etch stop, and 50% hydrazine in water as the anisotropic etchant. The silicon dioxide etch mask is removed in a hydrofluoric acid solution, and a 1500 Å thick film of aluminum is deposited in an electron beam evaporator at a rate of 25 Å/sec. The polyimide of interest is then spin cast and cured. The 5µm silicon diaphragm is removed using a backside SF_6 plasma etch and the aluminum is removed from the membrane area using a phosphoric-acetic-nitric acid solution to form the island blister.

Application. Two types of polyimide were tested, a pyromellitic dianhydride - oxydianiline formulation (PMDA-ODA) and a benzophenonetetracarboxylic dianhydride - oxydianiline / metaphenylenediamine formulation (BTDA-ODA/MPDA). The polyimides were spun in multiple coats at 4000 rpm for 90 seconds, with a 15 minute prebake in air at 150 C between coats. The PMDA-ODA polyimide was applied in three coats and the BTDA-ODA/MPDA polyimide was applied in two coats, so as to yield an after-cure thickness of 4.5 µm for each polyimide. The final cure was carried out in nitrogen at 400 C.

Measurement. The wafer and islands were secured to a type 304 stainless steel plate using commercial epoxy, and the plate was placed in a test apparatus [19]. Pressure was applied through holes in the plate, and was measured using a silicon pressure transducer built into the test apparatus. The pressurized blisters were observed in an optical microscope and the pressure at which the film began to peel (p_c) was observed as a function of the radius of film still adhered to the island (a_1). When the film began to peel, the pressure was lowered until peel ceased. The new a_1 was then measured, and the pressure raised until the film began to peel again. In this way, a set of p_c vs. a_1 data could be measured. Once the film peeled completely from the center island, the deflection of the film as a function of pres[sure] was measured by focusing the microscope on the top of the film and using a digi[tal] micrometer to measure the deflection of the microscope stage necessary to kee[p] the film in focus.

Results and Discussion

Five test sites were measured, three of PMDA-ODA and two of BTDA-ODA/MPDA. Figures 3 and 4 present the peel data for the two polyimides. The peel data are plotted in accordance with Equations (1) and (2), with $a_1^{\,2} f(\beta)$ o[n] the y-axis and p_c^{-2} on the x-axis. From Equation 2, such a plot should be a straig[ht] line through the origin with slope equal to $32\, t\, \sigma_0\, \gamma_a$. The residual stress/thickne[ss] product was calculated by a post-peel load-deflection measurement as describe[d] above. The thickness was measured using a surface profilometer, allowing the independent calculation of residual stress. Values for thickness, stress, and wor[k of] adhesion of each polyimide are given in Table I. With one exception, the reproducibility of the measurement from site to site was good. The reason for the single discrepancy is not known.

The PMDA-ODA polyimide had consistently poorer adhesion to aluminum than the BTDA-ODA/MPDA polyimide. This is consistent with previous qualitative observations [20]. The residual stresses measured for polyimide on aluminum ar[e] somewhat lower than previously reported values for these same polyimides on silicon dioxide [18]; the effect of the substrate on film residual stress is a topic of current study.

The work of adhesion inferred from the island blister test (in J/m²) can be converted to an equivalent 90° peel strength (in g/mm) by dividing by 9.8. For PMDA-ODA, the equivalent peel strength is 11.1 g/mm, and for BTDA-ODA/MPD[A] the equivalent peel strength is 49 g/mm. These numbers are generally lower than reported 90° peel strengths obtained from thicker films [7]. It is not known whethe[r] this experimental discrepancy is due to variations in sample preparation. However, [it] must be remembered that a peel measurement incorporates dissipative effects (viscoelastic and plastic) as well as work of adhesion in the peel strength. Kim [9] h[as] shown that this dissipative term is substantial in the 90° peel test. In the island blist[er] test, not only is the peel angle close to 0° (thus straining the film less), but also the peel rate is typically 2-5 µm/s, much lower than conventional peel tests. Thus, the effects of dissipative processes are expected to be less in the island blister test, which may account for the observed discrepancy in peel strength. We are presentl[y] extending the simple peel model of Equations 1 and 2 to include both dissipative effects and the contribution of the modulus of the film to the p_c- γ_a relationship.

Conclusions

The island blister test, a method for quantitatively measuring the adhesion of thin, well-adhered films has been described. It has been shown that films which are tensile strength limited in ordinary adhesion tests can be peeled using the island blister technique. A model for the test, relating critical pressure to the work of adhesion of the film, has been developed. The test was applied to two 4.5 µm polyimide films on aluminum substrates. For a PMDA-ODA polyimide, the work of adhesion was measured to be 109±34 J/m² (3 sites), while for a BTDA-ODA/MPDA polyimide, the work of adhesion was measured to be 481±11 J/m² (2 sites).

Acknowledgement

This work was supported by E.I. DuPont de Nemours & Co. Microfabrication wa[s] carried out in the Microsystems Technology Laboratories, and in the Microelectronic[s] Laboratory of the MIT Center for Materials Science and Engineering, which is supported by the National Science Foundation under Contract DMR-84-18718. The authors wish to acknowledge helpful discussions with E. L. Yuan of Dupont on aspects of polyimide adhesion.

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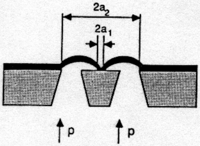

Figure 1. Cross section of island blister structure ,where a_2 = radius of film still adhered, a_1 = outer radius of membrane (constant), $\beta = a_2 / a_1$, p = applied pressure.

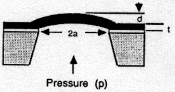

Figure 2. Structure for residual stress measurement. A square suspended membrane of thickness t and edge length 2a undergoing a deflection d at its center in response to an applied pressure p.

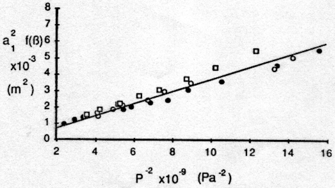

Figure 3. Adhesion plot of PMDA-ODA on aluminum (three nominally identical sites)

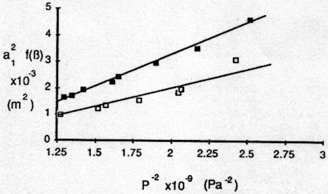

Figure 4. Adhesion plot of BTDA-ODA/MPDA on aluminum (two nominally identical sites)