Measurement of Polyimide Interlayer Adhesion Using Microfabricated Structures

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Introduction

The adhesion of polymer layers is extremely important in many integrated circuit multilevel metal interconnect schemes. Cracks between layers can result in moisture ingression and corrosion of metal lines, leading to long term device reliability problems. In addition, catastrophic delamination (for example, induced by film stress) can result in the sudden and immediate failure of the device. In spite of the importance of interlayer adhesion, few tests are available which can measure quantitatively and in-situ the energy necessary to debond these films. Preliminary work has focused primarily on the peel test [1,2]. However, the peel test suffers from two shortcomings which limit its utility. First, loading the sample is difficult; grasping one layer of film while keeping the others adhered can be a problem. Second, and most importantly, the peel test can be tensile strength limited. If the layers to be peeled apart are very thin and also well-adhered, tearing instead of interlayer debonding may occur [3]. In this case, it is not possible to make a measurement of the debond energy of the film. We have developed a modification of the standard blister test, called the 'island blister', which can overcome this tensile strength limit, as well as allow a convenient method of loading the film. The test and its application to the problem of polyimide interlayer adhesion will be discussed below.

Island blisters

The conventional blister test for the measurement of adhesive properties of films and coatings was first reported by Dannenberg in 1961 [4]. In this test, a film is adhered to a substrate, one section of which has been removed, forming a hole which exposes the bottom surface of the film. Upon pressurizing the bottom surface, the film bulges up over the hole. If sufficient pressure is applied, the film will debond from the substrate and a 'blister' will form, growing radially outward from the perimeter of the substrate hole. By measuring the pressure necessary to sustain the blister peel as a function of the blister radius, the energy necessary to debond the film can be determined [5-7]. One critical limitation of this test is that it is often tensile-strength limited; upon pressurization, thin and/or well-adhered films burst before they debond from the substrate.

Recently, there have been a number of modifications of adhesion tests to improve the range of adhesive energies which can be measured. Most of these methods involve reinforcing the film in some manner, such as increasing the film thickness [8] or placing a plate over the top of a growing blister [9,10] in order to increase the maximum load that the film can withstand before rupture.

One modified test, the island blister test, was introduced in 1987 as a method for quantitatively measuring the debond energy of films which are too thin and/or well-adhered to be peeled in other ways [6]. The island blister test differs from the modifications to the conventional blister test described above in that the pressure necessary to sustain peel is drastically lowered. Thus, no mechanical reinforcement of the film is necessary. Since peeling can be done at low pressures, the test is ideal for the measurement of polymer film interlayer adhesion.

The island blister consists of a suspended membrane of film with an 'island' of substrate attached to the center (see Fig. 1). The island and substrate are fastened down to a rigid plate, and the film is pressurized from the back. At some critical pressure, the film will peel from the island. Modeling the island blister 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 debond energy of the film (γ_a), and the radius of film still adhered to the island (a_1) are related by:

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

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:

$$f(\beta) = \left[\frac{\beta^2 - 1}{\ln \beta} - 2 \right]^2 \qquad (2)$$
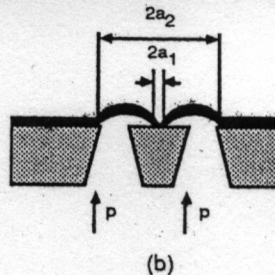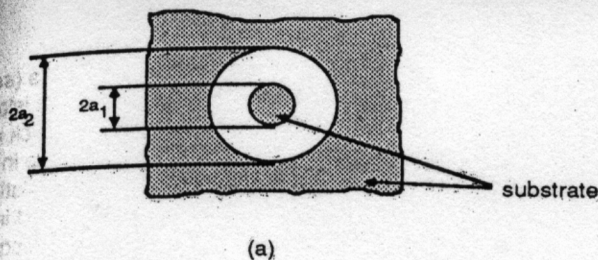
Figure 1. Island blister model. (a) Plan view (film removed); (b) Cross section (film shown), where a_1 is the radius of film still adhered to the island, a_2 is the outer radius of the membran (constant), $\beta = a_2 / a_1$, p = applied pressure

It can also be shown that the pressure necessary to peel radially inward (i.e., 'off the island') is alwa lower than the pressure necessary to peel radially outward (i.e., as in the conventional blister test) Examination of Equation 1 also reveals how the tensile strength limit has been overcome. If it is necessary to peel a system of a particular thickness and debond energy, the critical pressure p_c c always be made lower than any specified maximum pressure p_{max} (dictated by the condition that film not exceed its tensile strength) simply by making $f(\beta)$ sufficiently large. Equation 2 states that is made large when β is large, or the ratio of the outer film radius to the island radius is large. Thus tensile strength limit of the film can be overcome by manipulation of this geometric ratio.

An 'adhesion 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 debond energy. The film thickn can be determined by a surface profile measurement, and the residual stress can be measured in using a membrane load-deflection technique [11,12] which is summarized here. Once the film ha peeled, a suspended membrane of the film is formed (Figure 2). The residual stress in the film ca determined in-situ by a measurement of the load-deflection characteristics of the membrane. It ca shown that the deflection at the center of the membrane in response to the applied pressure is gi 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 th 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 [12]. Thus, the debond energy the film can be determined from a combination of island peel and load-deflection measurements.
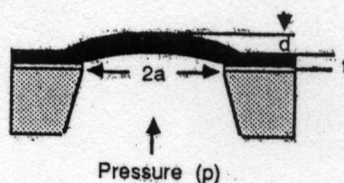

Figure 2. Structure for residual stress measurement. A square suspended membrane of thickness t and edge length 2a undergoing a deflection d at its center in response to applied pressure p.

Experimental

Description. Two types of experiments were carried out. In the first experiment, island blist using polyimide layers as both adherend and film were fabricated as described below. The first laye was 1 μm of a PMDA-ODA (pyromellitic dianhydride - oxydianiline) polyimide which had been fully cured at 400 °C for 45 minutes. The second layer was also PMDA-ODA 5.1 μm thick and cured un the same conditions. The interlayer adhesion was then measured using the island blister techniqu The second experiment was carried out to test the recent suggestion [1,2] that swelling the first lay in a good solvent (such as N-methyl-2-pyrrolidone, NMP) may improve the interlayer adhesion. Control samples were prepared as described in the previous section. Another set of samples was prepared identically to the control samples except that the first layer was swelled in NMP for 20 or 4 minutes at either 25 °C or 90 °C prior to the application of the second coat. Thus, the dependence the interlayer adhesion on the time and temperature of the NMP soak could be investigated.

Fabrication. Island blister test sites are fabricated using micromachining techniques (see Fig. 3). 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 1 μm thick film of polyimide is spin-cast with adhesion promoter on the wafer and pre-baked in order to partially imidize the film. The polyimide is then patterned lithographically into 400x400 μm squares aligned with the silicon islands using photoresist and a tetramethylammonium hydroxide developer as both the resist developer and polyimide etch. The photoresist is stripped in an acetone/methanol rinse and a low-power oxygen plasma descum, and the resulting polyimide squares (adherend pads) are cured at 400 °C in nitrogen for 45 minutes. A thin (100 Å) layer of copper (the release layer) is deposited on the wafer using an electron-beam evaporator and is patterned into squares which cover the island and adherend pad, but leave the central portion of the pad exposed. The polyimide adherend pads (forming the first layer of the interlayer peel structure) are treated in the various ways described above, and the second layer of polyimide is spin-coated and cured at 400 °C in nitrogen for 45 minutes. The 5μm silicon diaphragm is then removed using a backside SF_6 plasma etch to expose the backside of the second polyimide layer and form the island blister.

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. 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 pressure was measured by focusing the microscope on the top of the film and using a digital micrometer to measure the deflection of the microscope stage necessary to keep the film in focus.

(a)

(b)

(c)

(d)

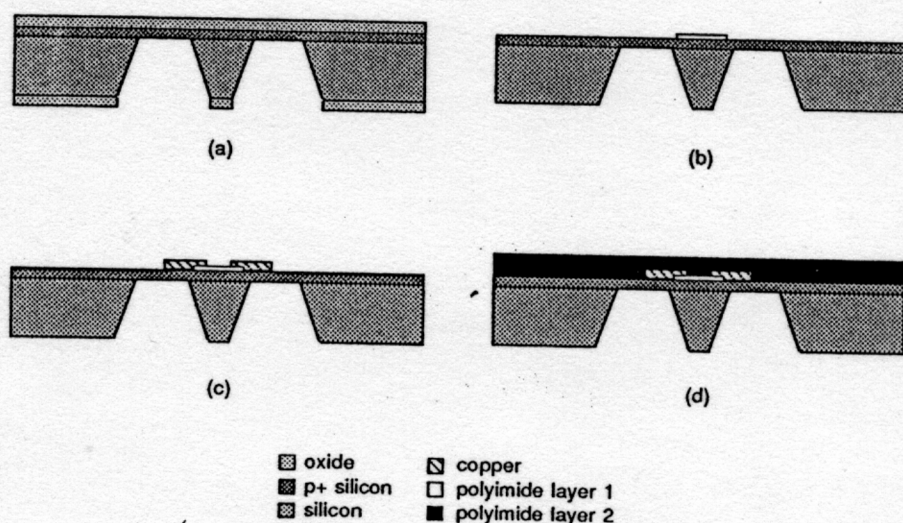|	
▨ oxide	◩ copper
▨ p+ silicon	☐ polyimide layer 1
▨ silicon	■ polyimide layer 2

Figure 3. Fabrication sequence of interlayer island blisters. (a) island pattern in silicon; (b) after patterning of first polyimide layer; (c) after patterning of release layer; (d) final structure (note: not to scale; actual dimensions in text)

Results and Discussion

The critical pressure - adhered radius data were plotted in accordance with Equation 1. A typical adhesion plot for zero soak time is shown in Figure 4. As can be seen, the data fit the theory quite well, giving a straight line through the origin. The slope of this line is determined by a least-squares method to be 257,000 Pa J/m. After peel, the residual stress was measured using the load-deflection technique and a σ_0 tproduct was determined to be 87.1 Pa-m. Combining these values yields a value for the debond energy of 94 J/m^2 for this test site. All of the sites tested obeyed the behavior predicted by Equations 1-3.
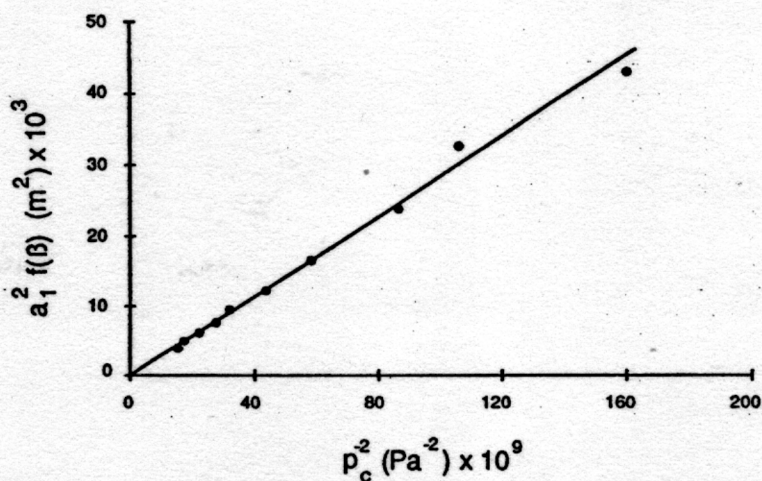
Figure 4. Adhesion plot of polyimide-polyimide interlayer peel (zero soak time)
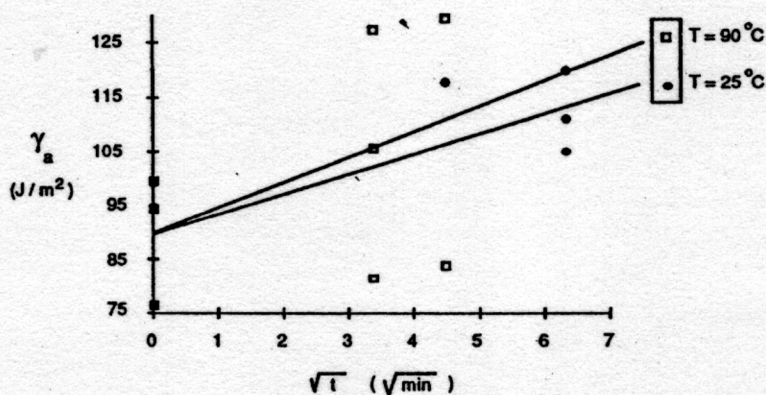

Figure 5. Debond energy as a function of NMP soak time and temperature

The data for all the experiments are summarized in Table I. The number of sites tested, conditions of pretreatment of the first layer (if any), slope of the adhesion plot, measured residual stress, and calculated debond energy are all given. For some of the sites, the residual stress was not measured; the nominal value (taken from other sites which had undergone identical deposition and curing of the second layer) was used. Figure 5 shows the data of Table I plotted as debond energy as a function of the square root of soak time (as for a diffusional process).

Although there is considerable scatter in the data, some trends are definitely observable. There does appear to be a slight increase in debond energy with soak time. For example, in the room temperature soak experiments, the highest measured value of the debond energy at zero soak time is lower than the lowest measured value of the debond energy at 40 minutes soak time. In addition, it seems as if increasing the temperature of the soak had a very small enhancing effect on the interlayer adhesion. Linear regression of the data yields a slope of 3.7 J m^{-2} min$^{-0.5}$ for the 25° soak and 3.9 J m^{-2} min$^{-0.5}$ for the 90° soak, with an intercept of 90.5 J/m^2 for both temperatures.

The recent work of Tong [2] indicates that longer soak times and/or higher soak temperatures for polyimide-polyimide adhesion enhancement are appropriate, from 2-3 days at 21 $^{\circ}$C to 30-60 minutes at 120 $^{\circ}$C. We are presently investigating these conditions using the island blister test.

Table I. Data for NMP soak

(a) T = 25 °C

Site	Soak time (min)	Adhesion plot slope (Pa J / m)	Stress - thickness product (Pa m)	debond energy (J / m^2)	
1	0	257,000	87.1	94	
2	0	277,100	85.0	99	90 ± 13
3	0	203,100	83.0	77	
4	20	325,300	86.2	118	118
5	40	303,000	90.2	105	
6	40	329,000	92.9	111	112 ± 8
7	40	397,000	103.6	120	

(b) T = 90 °C

Site	Soak time (min)	Adhesion plot slope (Pa J / m)	debond energy (J / m^2) (*)	
1	10	235,000	82	
2	10	367,000	127	105 ± 23
3	10	304,000	106	
4	20	241,000	84	
5	20	374,000	130	107 ± 23

(*) Nominal stress-thickness product of 90 Pa-m used

Acknowledgement

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