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Research paper

Buckle induced delamination techniques to measure the adhesion of metal dielectric interfaces

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ABSTRACT

Adhesion of thin metal films on dielectric substrates is one of the most important properties in microelectronic devices due to the fact that interface adhesion determines the device lifetime and reliability. In order to improve the lifetime of these devices, substrate treatments and adhesion layers are often utilized, making a quantitative assessment of adhesion of the utmost importance. A key example of a metal film common in many microelectronic devices is Tungsten-Titanium (WTi), which is used as a diffusion barrier and an adhesion layer. Several testing methods and mechanics-based models have been developed over the last decades to quantitatively evaluate interface adhesion of thin metal films on rigid substrates. For thin films on rigid substrates (i.e. dielectrics or silicon) the most viable methods are mechanical techniques such as nanoindentation and scratch induced delamination because of the simplicity of the test setup and sample preparation. During indentation or scratching, stresses are induced into the film system which can cause interface separation in the form of buckles. By measuring the dimensions of the buckle and employing the appropriate model, the interfacial adhesion energy can be quantitatively determined. These techniques were utilized to induce interface delamination of a WTi film on different borophosphosilicate glass (BPSG) substrates. The comparison of the results from the different techniques will provide more insight into the techniques applicability and help to better characterize the adhesion of similar film systems.

1. Introduction

In microelectronic devices a large number of interfaces between different metals and dielectrics are present. The lifetime and reliability of such devices greatly depend on the mechanical stability of these interfaces. For a better understanding of these interfaces a quantitative adhesion test method is required. Many experimental methods and theoretical models have been devised to investigate the adhesion of dissimilar materials [1–3]. Of special interest in the microelectronics industry are the interfaces between metallic conductors and dielectric glasses, because of their widespread use but generally weak adhesion [2–4]. The combination of tungsten based metals as barrier materials and borophosphosilicate glass as the dielectric are frequently used in microelectronic devices. Several chemical and mechanical studies have been conducted to investigate the interface and assess the adhesion of this kind of metal to dielectric layers which has been proven to be a weak interface [5–7].

Some of the common adhesion measurement methods are not only rather lengthy and come with extensive sample preparation but also lack the according experimental yield for quantitative adhesion assessment, such as four-point bending (4PB) [5,6,8,9] or double cantilever beam testing [7]. From a scientific and industrial point of view it is important to identify which method is suited best for the materials and the size regime in question. Of interest to this study are spontaneous buckling and stressed overlayers [2,10–12], scratch based [3,13–18] and nanoindentation [2,19–21] methods are used to quantitatively measure adhesion energies. These methods work well with hard metal films on rigid substrates to induce delamination in the form of buckles or blisters. For example, spontaneous buckles can form as a result of compressive stress in the film and either form as straight-sided or telephone cords [2,10]. Since spontaneous buckles form without any additionally applied mechanical force, they are one of the most reliable and easiest ways to measure the adhesion energy of an interface. In the case where there is not enough residual stress to cause spontaneous buckling additional mechanical force is necessary to cause delamination of the film. This can be done by scratching [13–17] or indenting the surface with a nanoindenter [2,3,11,19–21]. When scratch testing is used to evaluate adhesion, a sharp indenter tip is drawn across a surface until at a critical load film failure occurs. The nature of the failure strongly

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depends on the properties of the film and substrate and occurs in form of through thickness cracking or buckling of the film but also plastic pile-up around the scratch trace. Nanoindentation, a method originally used for mechanical property measurements, has proven to be a good technique for inducing well-defined areas of delamination to measure the adhesion of thin films. When a high enough load is applied by the indenter tip, a crack is induced at the interface which propagates to produce a delamination. The delamination can be a circular blister or a spontaneous buckle.

In the case of hard metal films and rigid glass substrates the buckling response is mostly elastic and the induced buckles or blisters can be analyzed by the model developed by Hutchinson and Suo [1]. Plastic effects due to film buckling are not considered in this model, although they become relevant in the case of softer substrates and very high film stresses [22]. The stresses and the interfacial fracture energies can be calculated using the geometry of the produced buckles. The necessary measurements are the buckle height, δ, buckle width, $2b$, as well as the thickness of the film, h and the elastic modulus, E, and the Poisson's ratio, ν of the film. In the case of spontaneous buckles the critical buckling stress, σ_b, and the driving (or residual) stress, σ_d, can be calculated by using Eqs. (1) and (2) [1],

$$\sigma_b = \frac{\mu^2 E}{12(1-\nu^2)}(h/b)^2, \tag{1}$$

$$\sigma_d = \sigma_b\left[c_1(\delta/h)^2 + 1\right], \tag{2}$$

with $\mu^2 = \pi^2$ and $c_1 = 3/4$. The Mixed Mode interfacial fracture energy, $\Gamma(\psi)$, in the following termed the adhesion energy, for spontaneous buckles is given in Eq. (3).

$$\Gamma(\psi) = \left[\frac{(1-\nu^2)h}{2E}\right](\sigma_d-\sigma_b)(\sigma_d+3\sigma_b). \tag{3}$$

In the case of indentation induced blisters the Hutchinson and Suo model for circular blisters can be used [1], under the condition that the blisters are circular, should be large compared to the indentation imprint and no radial or base cracking is caused due to the indentation [2]. Two different blister geometries can form which depend on the length of the interface crack. If the crack exceeds a certain length the film double buckles and the blister is pinned at the center, if not it single buckles and is modeled as an unpinned blister [1,2,19–21]. The formulas for the critical buckling stress and the driving stress are the same as for spontaneous buckles except for the factor μ^2, which is 14.68 for unpinned and 42.67 for pinned blisters, and $c_1 = 0.2473(1+\nu) + 0.2231(1-\nu^2)$. Using the stress calculated with Eqs. (1) and (2), the Mixed Mode adhesion energy for the indentation blisters can be calculated from Eq. (4),

$$\Gamma(\psi) = c_2\left[1-(\sigma_b/\sigma_d)^2\right]\frac{(1-\nu)h\sigma_d{}^2}{E}, \tag{4}$$

with $c_2 = [1+0.9021(1-\nu)]^{-1}$. The Mixed Mode adhesion energies calculated from Eqs. (3) and (4) are a measure for the practical work of adhesion since it depends on the phase angle of loading, ψ, which gives the relation between normal and shear forces present at the interface given by Eqs. (5) and (6) [1–3]. The phase angle of loading can be approximated for straight sided blisters by

$$\psi = \tan^{-1}\left[\frac{4\cos(\omega) + \sqrt{3}\,\delta/h\sin(\omega)}{-4\sin(\omega) + \sqrt{3}\,\delta/h\cos(\omega)}\right], \tag{5}$$

and for circular blisters by

$$\psi = \tan^{-1}\left[\frac{\cos(\omega) + 0.2486(1+\nu_1)\delta/h\sin(\omega)}{-\sin(\omega) + 0.2486(1+\nu_1)\delta/h\cos(\omega)}\right], \tag{6}$$

with $\omega = 52.1°$ and assumes no elastic mismatch in the case of a rigid substrate [1]. The knowledge of the phase angle allows for the normal mode (Mode I) adhesion energy, Γ_I, to be calculated with Eq. (7),

$$\Gamma_I = \Gamma(\psi)/\left[1+\tan^2\{(1-\lambda)\psi\}\right], \tag{7}$$

with $\lambda = 0.3$ which is a parameter defining the shear mode contribution to the interfacial fracture toughness and assumes a brittle interface [1]. The Mode I adhesion energy only considers the normal forces used for interface separation where the phase angle is 0°. This can be considered close to the true work of adhesion consisting of the thermodynamic work of adhesion and the energies dissipated in the film and substrate [3]. Over the last decades the model of Hutchinson and Suo has been advanced to include elasticity corrections [22] and utilized to study how the buckle shape influences adhesion [10,23–25]. Parry et al. [22] use Dundurs Parameters to correct for any elastic contributions experienced by the substrate. The buckle shape can also have an influence on the calculated adhesion energies. For example, Faou et al. [23] have shown that the ψ angle varies as a function of the telephone cord which would affect the Mode I values. The highest ψ angles were found at the curve of the telephone cord and the lower ψ angles near the point of inflection of the buckle. Cordill et al. [25] demonstrated that when telephone cords are measured across the point of inflection and modeled as straight sided buckles with the Hutchinson and Suo model [1] the values were the same as for straight buckles. Another model by Faou et al. [24] that could be employed to evaluate interface adhesion is to use the telephone cord wavelength. This model uses the telephone cord wavelength, λ, instead of the half buckle width, b and height, δ, as well as parameters determined from a cohesive zone simulation to assess the Mode I adhesion energy. In the following the adhesion energies of a tungsten alloy film with two differently treated dielectric substrates are investigated through the use of three different mechanical adhesion testing techniques. The experimental and calculation results are compared and their viability for the film-substrate systems at hand are discussed.

2. Materials and methods

Two sets of films were provided by Infineon Technologies Austria AG. They consisted of two 725 μm thick silicon wafers with a dielectric layer and a metal barrier film. In the first deposition step, 800 nm of borophosphosilicate glass (BPSG) was deposited on both wafers using plasma enhanced chemical vapor deposition. One of the two BPSG films was annealed at 600 °C for 160 s. The Young's modulus of BPSG lies between 60 and 70 GPa. After that a 300 nm thick Tungsten-Titanium (WTi) film was sputter deposited onto both wafers where the tungsten film had 20 at.% of Ti. The films were deposited under conditions that induced a compressive residual stress of about 1 GPa (measured by wafer bow). Scratch- and nanoindentation experiments were conducted with a Keysight G200 nanoindenter. For the scratch test a Berkovich tip was used with the sharp side of the tip being the scratch front. The scratch distance of all experiments was 500 μm and the distance between the scratches was set to 500 μm to avoid interaction with other scratches. The utilized load range was between 100 and 500 mN with a scratch velocity of 30 μm/s. For indentation induced delamination two conical diamond tips were used, 1 μm and 5 μm in diameter, with a load range between 100 and 500 mN. The indents were set about 500 μm apart from each other to avoid any interaction with other indents. Scratches and indents were examined with a light microscope to quickly identify if delaminations occurred and these examinations were repeated over a period of weeks to account for further buckle