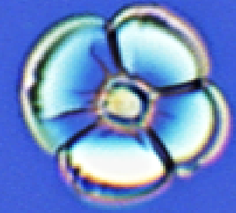
Application Note

KLA INSTRUMENTS

Film Delamination

Combining Indentation and Imaging for Detecting Critical Delamination Load and Interface Adhesion Energy



Introduction

Adhesion of dielectric thin films has been a significant challenge in interconnect integration and reliability. These devices are compromised by film layer adhesion failures, dielectric film mechanical integrity and multi-layer interfacial cracks. Underlying patterns and mismatch of the crystallographic structure generate the driving forces for debonding and delamination, resulting in the failure of dielectric layers. Understanding the failure mechanisms of interconnect structures is critical to improving manufacturing processes for enhancing interconnect reliability.

Nanoindentation is typically used for measuring thin film mechanical properties such as elastic modulus and hardness of the material. In the case of a brittle, weakly bonded film, indentation tests are also used to delaminate the film from the substrate and interfacial fracture strength is then determined from this delamination event. In this work, the KLA iMicro nanoindenter configured with a conical indenter tip was used to delaminate a low k film on a Si substrate to determine the critical delamination load. The interfacial energy released during delamination was calculated from direct analysis of loading/unloading curves.

Experimental Method

The iMicro nanoindenter equipped with an InForce 1000 actuator and a conical tip with 10µm radius was used to perform indentation testing for delamination of a ~340nm low k film on a Si wafer. Hardness and elastic modulus of the film were each measured separately using a Berkovich indenter, where the hardness was measured as 15.34GPa and the elastic modulus was measured as 2.341GPa.

Sharp cube corner or Berkovich tips are traditionally used to create fracturing in bulk hard materials. However, for brittle thin film coatings, the use of sharp pyramidal geometries results in the formation of sudden cracking at very small loads, followed by chipping. The sharp tip edges force radial cracking form at the onset of loading, followed by delamination as the load is increased. Spherical tips, on the other hand, serve to delay radial cracking at small loads such that the failure mechanism is dominated by delamination and/or interfacial cracking.

The ISO 14577 test method with a constant loading rate was used for indentation. An array of 100 indents was generated with a maximum load of 200mN, using a 0.98 decrement factor to determine the critical load at which delamination occurs. For 100 indents, the final indentation load is 27.1mN. Optical micrographs were also collected using different objectives to measure the debonded area. *Note that the NanoVision stage on the G200X can deliver similar crack length measurements by accurate imaging of the entire area of indentation and cracking – these results will be published in a different application note*.

Indentation Testing Results – Delamination

Interfacial cracking and/or delamination occurs during the indentation of low k films. In general, delamination is most likely a consequence of radial cracks that reach to the coating-substrate interface and then deflect along the interface. For a sharp pyramidal tip, the delaminated area forms a sector of a disk surrounded by radial cracks, as shown in Figure 1a. But for a spherical tip, several cracks form within the indentation area and, as the load increases, more cracks appear and grow through the coating towards the surface, as shown in Figure 1b.

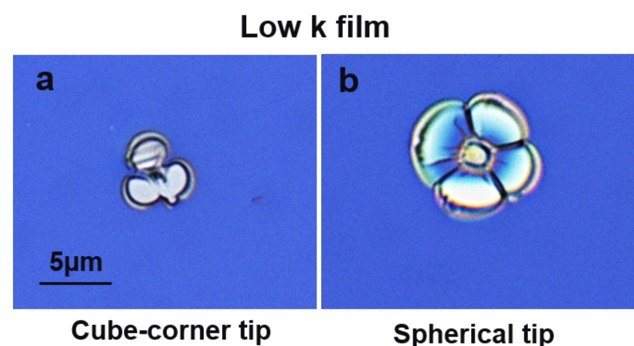


Figure 1. Nanoindentation delamination effects for (a) a sharp pyramidal tip, and (b) a spherical tip.

The delaminated film will eventually buckle due to the in-plane stresses caused by the indentation. Pyramidal tip geometries produce ill-formed film cracking for use in the adhesion model being presented here because cracks radiate from stress concentrations at the sharp edges of the indenter. Conversely for spherical tips, film cracking starts at the sub-surface interface and radiates in a symmetric pattern. For this reason, the pyramidal tip delamination is not further studied in this application note.

The critical loads for crack and delamination initiation are visible as discontinuities in the load-displacement curve shown in Figure 2. The first discontinuity or pop-in event was observed for the test ending at 47.4mN.

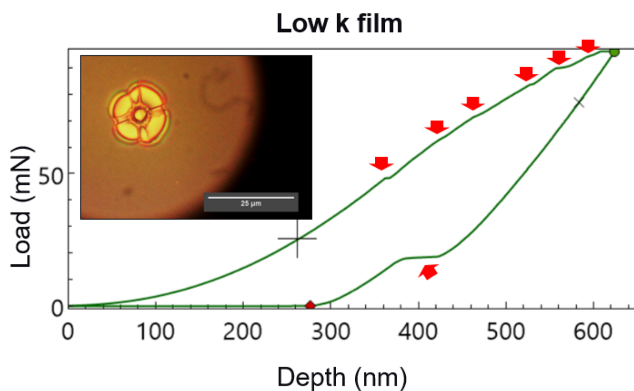


Figure 2. Critical loads that initiate cracking and/or delamination display discontinuities in the loading/unloading curve, as indicated by the red arrows.

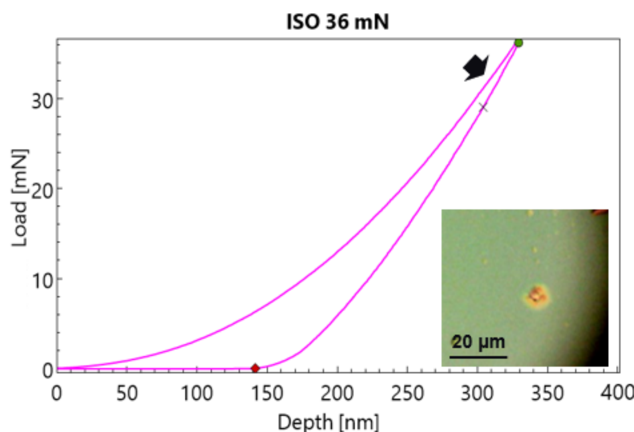


Figure 3. The load-displacement curve with a maximum load of 36mN does not show a discontinuity but does show initial cracking and delamination effects (inset). A slight change in slope, noted by the arrow, could be related to the initial cracking.

While displacement excursions and pop-in events are obvious signs of failure, initial cracking and delamination in this sample occurs at forces below 47mN, as shown in Figure 3. Importantly, the load-displacement curves show evidence of cracking through both acceleration in displacement and pop-in events at higher forces during the tests. KLA nanoindenters are sensitive enough to detect both forms of interface cracking events. Figure 4 shows the load-displacement curve for a test with a maximum load of 47mN, with a clear discontinuity observed at about 43mN and indicated by the arrow. For repeated tests above this load, a pattern of pop-in events was observed in the 47-52mN load range (top inset). These results lead to an implied load reliance for pop-ins to occur, i.e., a single load ($P_{Critical}$) where the first crack or delamination is expected. Note that the pop-in events for this sample occur at indentation depths greater than the coating thickness of 340nm; this indicates substantial elastic deformation of the film-substrate material under the spherical tip, which results in high levels of stress to drive film interface failures.

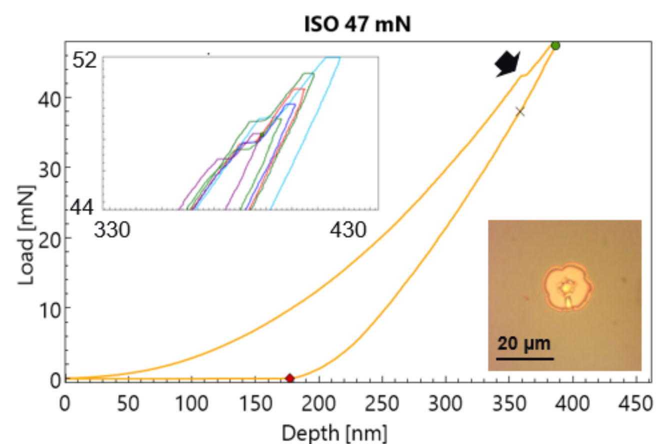


Figure 4. The load-displacement curve with a maximum load of 47mN shows a pop-in event at ~42mN. Repeated tests at higher loading (top inset) also show pop-in events at depths greater than the thickness of the low k film.

It was also observed that as the indentation load increases, additional pop-in events appear in the load-displacement curve, suggesting that the number of cracks in the delaminated area could be associated with those pop-in events.

Indentation Testing Results – Adhesion Energy

Interface adhesion energy or interfacial fracture energy is the energy required to propagate a crack along the coating-substrate interface. Using indentation, it is possible to calculate the work under the load-displacement curve, or dissipated energy, to extract the interfacial fracture toughness. In practice, it is difficult to differentiate between the cohesive failure in the coating or substrate and interfacial cracking (delamination). However, when the full fracture or first clear pop-in event

appears in the load-displacement curve, a near-constant delaminated area surrounds the indent. Following this first fracture or pop-in event, further loading results in additional radial cracking within the delaminated area. Under severe loading conditions, chipping and material ejection is possible, which leads to large damage radii and multi-mode fracture events - this excessive loading is undesirable.

Equation 1 describes the general definition of work as the total energy or work done during indentation.

$$\text{Total Energy} = \int_0^{h_{max}} P(h)dh = W_{el} + W_{pl} + W_{de} + Q \quad (1)$$

This work is defined as a function of elastic strain energy, plastic energy or yielding, interfacial cracking or delamination, with some amount of heat (Q) dissipated. Among the energy components in Equation 1, only the elastic energy is reversible, whereas other energies are not. As shown in Figure 5, the plastic work from indentation is determined by subtracting the elastic work under the unloading curve (Figure 5a) from the total work of indentation represented by the loading curve (Figure 5b). Assuming the heat dissipated during indentation is negligible, irreversible work represents the plastic work plus the work done for cracking and delamination.

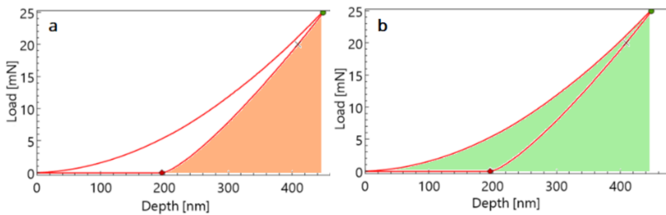


Figure 5. The elastic work (a, shaded) is recovered during unloading. The total work (b, shaded) includes both plastic and elastic work and is the area under the load-displacement curve.

In order to calculate just the adhesion energy due to delamination, two consecutive tests are considered: a test that shows a cracking (pop-in) event surrounded by full delamination; and the previous test showing elastic-plastic response with no cracking. Equation 2 describes the adhesion energy calculation. The work divided by the delamination area is used to represent the interfacial delamination energy, described in Equation 3, where *A* represents the delamination area, which can be determined from optical micrographs.

$$\Delta E_{crack} = E_{Critical} - E_{Critical-1} \quad (2)$$

$$\gamma_{crack} = \frac{E_{Elastic-Plastic-Crack} - E_{Elastic-Plastic}}{A_{Crack}} = \frac{\Delta E_{crack}}{A_{crack}} \frac{J}{m^2} \quad (3)$$

Table 1 shows the calculated interface adhesion energy values for several low k coating samples, determined using the method described above. T_{film} is the thickness of the film; $P_{critical}$ is the test load at which the first pop-in event was clearly identified; D is the radius of the conical tip used for indentation (constant for all samples); ΔE_{crack} (J) is the work or energy calculated for each sample (numerator of Equation 3); R_{crack} is the radius of the crack or delaminated area; and γ is the interfacial delamination energy.

Table 1. Interface Adhesion Energy

	Sample 1	Sample 2	Sample 3	Sample 4
T_{film} (nm)	353	359	369	371
$P_{critical}$ (mN)	47.4	50.3	44.6	50.3
$D_{indenter}$ (μm)	10	10	10	10
ΔE_{crack} (J)	$1.33E^{-9}$	$1.48E^{-9}$	$1.10E^{-9}$	$1.55E^{-9}$
A_{crack} (μm^2)	235.96	233.98	235.96	268.80
γ (J/m^2)	5.66	6.33	4.68	5.77

Results in Table 1 demonstrate that this technique is able to capture small differences in interface adhesion energy. The slight variation in calculated results could be due to an actual variation in bonding strength for different thin films during the deposition process.

Summary

KLA nanoindenters are able to measure small differences in the interface adhesion energy of thin film samples. In this work, interface adhesion energy of low k materials was measured using a KLA iMicro nanoindenter. The ISO 14577 standard method used with a decremental loading factor enabled the determination of the load required for initiation of cracking/ delamination at the film/substrate interface. Analysis of the energy dissipated during indentation was assessed for load-displacement curves near the critical load. The methodology used in this study can also be implemented for different brittle film-substrate material systems where plastic deformation of the film/substrate during indentation is negligible and most of the applied energy contributes to elastic work and interfacial debonding.

KLA SUPPORT
Maintaining system productivity is an integral part of KLA's yield optimization solution. Efforts in this area include system maintenance, global supply chain management, cost reduction and obsolescence mitigation, system relocation, performance and productivity enhancements, and certified tool resale.
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