

When I was in high school, I really did not know what I wanted to do with my life. I loved every subject that I came across: History, English, Chemistry, Math, and Spanish. My problem had always been that I wanted to study everything and accomplish anything. I wanted to become a doctor, a lawyer, a professor, and eventually run for Congress. There was one thing, however, I never imagined doing: engineering. The field's innovation and futuristic ideals intimidated me. Something deep down told me that I could not handle anything engineering or mechanical based. However, once I saw the goals of NANO-CEMMS, I felt that I should challenge myself and my insecurities with the Bardeen Quad. I took a chance when I applied and I now feel eternally grateful for being accepted. This past summer has stimulated my mind in so many different ways and now I finally feel like I can do just about anything if I set my mind to it and try.

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Delamination of Thin Films Using Laser Induced Stress Waves

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Abstract

A novel dynamic adhesion test is being developed to extract quantitative fracture parameters using laser-induced stress

waves. The sample preparation for adhesion tests involves patterning a weak adhesion layer beneath the test film patterns at a specified location. The thin film region above the weak adhesion layer is allowed to debond using a high-amplitude dynamic stress pulse. The energy trapped in the failed portion of the interface is diverted towards thin film delamination. Gold, carbon and octadecyltrichlorosilane (ODTS) weak adhesion layers were patterned using simple microfabrication techniques.

Introduction

The development of the microelectronics field has revolutionized the world. Usage of metallic thin films as the interconnections in

semiconductor devices is now widely accepted. Figure 1 shows a cross-section of a current generation microelectronic device consisting of several thin film layers separated by dielectric films. However, a reliability issue exists with every interface created. The term “reliability” is defined as “the ability of a system or component to perform its required function under stated conditions for a specified period of time.”¹ Though this definition implies that failure for an integrated circuit is guaranteed, there are techniques in development that can lead to an increase in lifetime of the component.

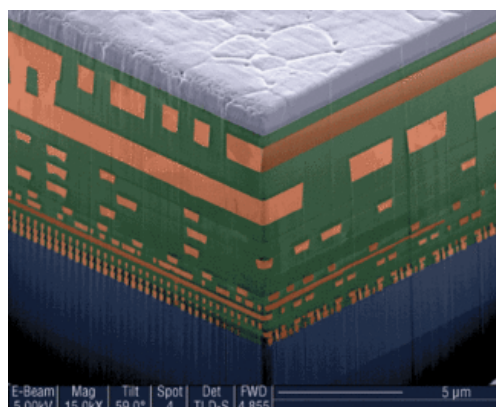


Figure 1: A cross-section of a microelectronic device showing multiple thin film stacks and interfaces.

The interface failure between a thin film and its substrate is the concentration of this research. Several tests have been developed to evaluate interface adhesion. Such tests include the scotch tape, stud pull, double cantilever, and nanoindentation tests.¹ Though these tests are commonly employed, they all involve some sort of contact with the actual thin film. As thin films continue to shrink to the feature size of a few nanometers, new techniques to measure interface adhesion of such thin films are warranted. The purpose of the research presented in this paper is to develop a technique that applies a load to the thin film in a non-contact manner to achieve thin film de-adhesion.

Failure modeling of thin film adhesion is characterized by two parameters: interface strength and interface toughness. Interface strength quantifies the applied stress necessary at the interface to achieve failure and interface toughness quantifies the amount of energy necessary to propagate a crack at the interface per unit area. It is important to note that interface strength and interface toughness are independent entities and together dictate the interface crack propagation behavior. Thus, it is essential to characterize the two individually.

Recent advancements in the laser spallation adhesion testing scheme has shown a potential to extract both adhesion parameters. While the extraction of interface strength is well established by using the laser spallation technique, the extraction of interface toughness is not well understood.

Background

Thin Film Adhesion Tests

Various adhesion tests ranging from the simple scotch tape test to complex blister tests are currently employed in industry to measure adhesion between thin films and their substrates. In the scotch tape test, a piece of scotch tape is attached to a thin film surface and lifted off. The effect of this test is the interface peeling due to its adhesion to the tape. This experiment only proves to be a good test for films with weak adhesions. The scratch test is another adhesion test where a diamond tip is moved across the thin film sample while being pressed to achieve thin film delamination. However, extracting the adhesion parameters has proven to be difficult.² More recently, bulge/blister tests and superlayer tests have been developed to quantify adhesion parameters for some thin film/substrate combinations. All of these adhesion tests are mainly quasi-static, involve direct contact, and often subject thin film to

high stress levels with deformations extending into the inelastic regime.

Laser Spallation Technique

Laser spallation technique, originally developed by Vossen and later adapted to thin films by Gupta and Junlan,² involves a laser induced, non-contact dynamic load to delaminate the thin film from its substrate. The basics of this technique are shown in Figure 2a where a Nd:YAG pulsed laser (5ns rise time) is impinged on an aluminum absorbing layer which is sandwiched between a waterglass constraining layer and the silicon substrate. A high-amplitude compressive stress wave results by the laser-induced ablation of aluminum absorbing layer, which propagates towards the free surface of the test film. After the compressive wave reaches a free surface, a reflective tensile pulse propagates in the opposite direction. If the magnitude of the tensile wave is large enough to overcome the interface strength, the thin film spalls from the substrate. Figure 2b shows a typical spallation pattern where complete failure of the test film is observed. The mechanics of the laser-induced spallation is well analyzed and extracting the interface strength is well established.

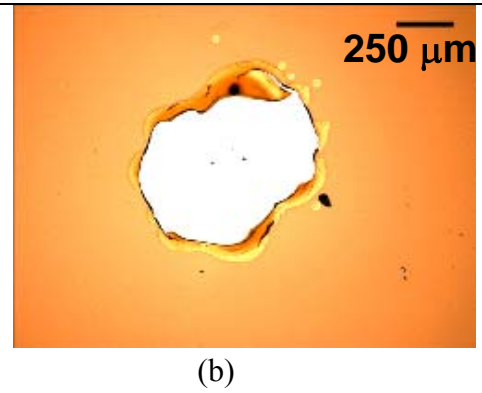
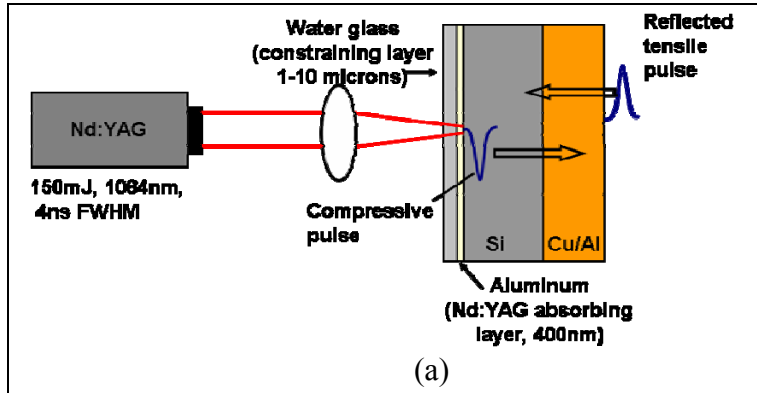


Figure 2a: Schematic of the laser spallation setup designed to measure the thin film interface strength.

Figure 2b: Optical micrograph showing thin film spallation damage.

More recently, this technique was adapted by Kandula³ to extract interface toughness. The modification involves fabricating thin film patterns with weak adhesion layers (Figure 3a) on the substrate. As shown schematically in Figure 3b, the region with the weak interface is loaded using the

laser-induced stress pulses to spall the thin film thereby creating a pre-crack at the interface. Furthermore, the energy trapped in the thin film above the pre-crack region provides the driving force to propagate the interface crack. Extraction of interface toughness involves a simple

energy balance principle where the interface toughness times the final crack extension is equated with the kinetic energy stored in the thin film over the pre-crack region.

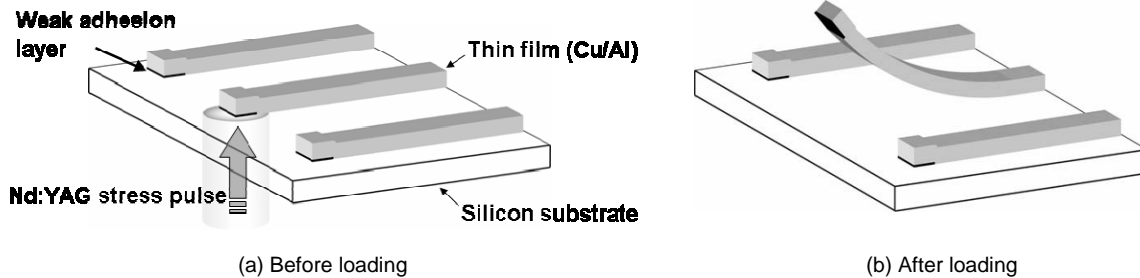


Figure 3: Schematic of the laser-induced thin film delamination designed to extract interface toughness.

The repeatability in the quality of weak interface directly translates into the repeatability of the dynamic adhesion test. In general, a weaker interface allows for more kinetic energy to be trapped in the debonded portion of the thin film. However, patterning such

extremely weak interfaces beneath the thin film poses a fabrication challenge. During photolithographic patterning of thin films, there is a possibility of film delamination above the weak adhesion regions while stripping the photoresist using ultrasonication. Improving

microfabrication procedures to pattern weak adhesion layers without causing thin film delamination was the objective of this project. In particular, three different weak adhesion layers were studied: gold, carbon and octadecyltrichlorosilane (ODTS)

to represent weak metallic, amorphous, and polymeric interfaces respectively.

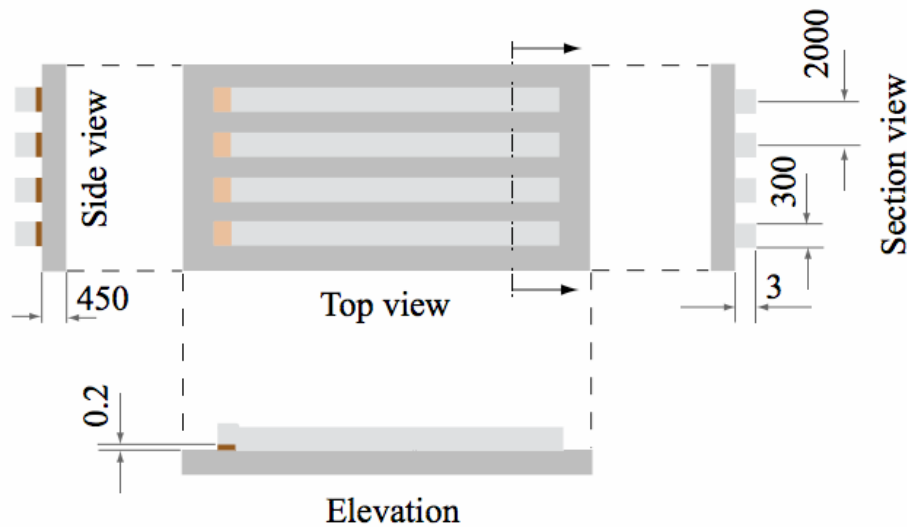
Approach

Patterning Weak Adhesion Layer Beneath Thin Film Structure

A schematic design of a representative weak interface layer patterned beneath the thin film is presented in Figure 4. Fortunately, thin film patterns

with these dimensions can be easily fabricated using standard microfabrication techniques. However, the only challenge associated with this was to ensure sufficient bonding of the thin film pattern above the weak interface region. If delaminated, the contact between the thin film and the substrate would be lost. This would result in no energy transfer to the thin film during the application of a load. A shadow mask technique was

used to pattern the gold interlayer. A combination of lithography and shadow mask technique was used to pattern carbon. Finally, a combination of lithography, soft-lithography, and shadow mask technique was used to pattern ODTS. The fabrication procedure for the three weak interfaces follows.



All dimensions are in μm

Figure 4: Details of the thin film pattern with weak interface designed for the laser-induced thin film delamination experiment.

Patterning Gold Weak Interface

A bilayer shadow mask technique was used to pattern gold beneath the thin film material. Two unique patterns of stainless steel shadow masks were used. The “thin film mask” and “weak interface mask” were designed to pattern the thin film and the weak interface respectively. Shadow masks features were chemically milled (Photoetch technologies Inc.) on

a 100 μm thick stainless steel plate and have dimensions similar to Figure 4. The technique used to pattern gold beneath the thin film material is described below.

Bi-layer shadow mask fabrication technique

1. Degrease substrate and stainless steel masks using deionized water, acetone, and isopropyl alcohol.
2. Attach alignment marker to the substrate.
3. Align thin film shadow mask with the alignment marker and attach firmly to the substrate.
4. Place the weak interface mask on top of the thin film mask and align with the alignment marker and the thin film mask.
5. Deposit gold using E-beam evaporator.



6. Remove top weak interface mask with out disturbing the thin film mask.
7. Deposit the thin film on to the top of the weak interface layer.
8. Remove the thin film shadow mask.

Examples of thin film patterns with a gold weak interface are shown in Figure 5a and 5d.

Patterning ODTS Weak Interface

To pattern ODTS beneath the thin film a combination of photolithography, soft lithography, and shadow mask techniques were used. The microcontact printing technique developed by Payne was used to transfer ODTS pattern on to the silicon substrate.⁴ Using photolithography, the weak interface shadow mask pattern was transferred onto the substrate using a positive photoresist. ODTS was then transferred onto the silicon substrate by using a flat polydimethylsiloxane (PDMS) stamp inked with ODTS solution. The resist pattern acted as a mask during the microcontact printing process. This technique results in transfer

of a 25Å tall self assembled monolayer ODTS structure onto the silicon substrate.^{4,5}

ODTS patterning technique⁶

1. Degrease silicon substrate, weak interface shadow mask with acetone, IPA, and deionized water.
2. Bake silicon substrate for 1 minute at 100 °C.
3. Spin coat AZ 5214E resist (3 ml, 2000 rpm, 45 seconds).
4. Bake sample with resist for 1 minute at 100 °C.
5. Expose using weak adhesion shadow mask (Figure 4c) for 9 seconds using I-line.
6. Develop the exposed resist in AZ 327MIF.
7. Transfer ODTS using microcontact printing.⁴
8. Deposit alignment markers.
9. Strip resist using acetone.
10. Align thin film shadow mask with the alignment markers.
11. Deposit thin film.
12. Remove thin film shadow mask.

Transferring ODTS onto the PDMS stamp

1. Make flat PDMS stamp.
2. UV expose PDMS stamp for 1 hour.
3. Spin coat PDMS stamp with ODTS solution (0.1ml of ODTS in 10ml of hexane).
4. Dry PDMS stamp coated with ODTS using nitrogen..
5. Press stamp onto silicon substrate, covering photoresist region for 30 sec.

Examples of thin film patterns with an ODTS weak interface are shown in Figure 5b and 5e.

Patterning Carbon Weak Interface

Carbon was patterned onto silicon following the steps similar to patterning ODTS except the microcontact printing step was replaced by thermal evaporation of carbon. Bi-layer shadow mask technique, similar to gold deposition, can also be used to pattern carbon. Examples of thin film patterns with a carbon weak interface are shown in Figure 5c and 5f.

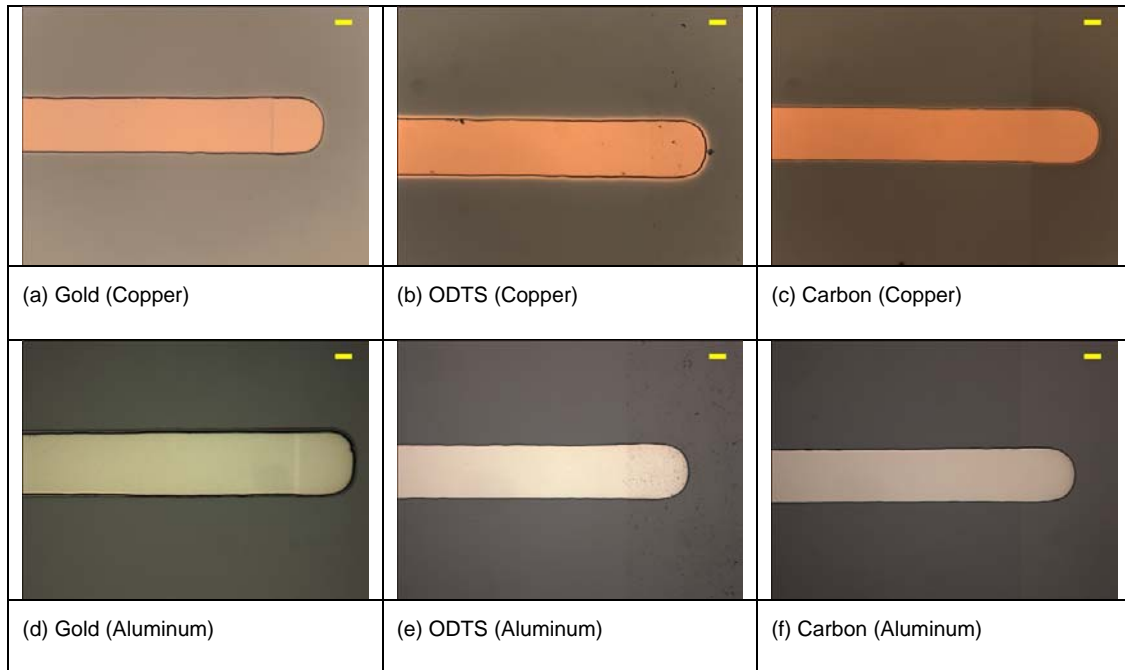


Figure 5: Top view of the thin film patterns with weak interfaces. The scale bar represents 100 μm .

Results and Discussion

Aluminum and copper thin film structures were patterned on a silicon substrate with gold, ODTS, and carbon weak interfaces following the fabrication techniques mentioned above. The thin films were deposited using electron beam evaporation (Temescal) with thicknesses of $3\mu\text{m}$ for aluminum and $2\mu\text{m}$ for copper. Thicknesses of the weak interface patterns were 200 nm, 40 nm and 25\AA for gold, carbon, and ODTS, respectively.

Dynamic peel tests were conducted by launching the stress pulses on the back side of the silicon substrate using a Nd:YAG laser. The laser energy was maintained to ensure complete failure of thin film above the weak interface. The energy trapped in the debonded portion of the thin film was channeled into interface crack propagation. Figures 6a and 6d show consistent delaminations with lengths reaching around 6 mm for thin films with gold weak interface. Repeatable

delaminations were obtained for copper thin films with ODTS and carbon monolayers seen in Figures 6b and 6c. Interestingly, for aluminum thin films with ODTS and carbon weak interfaces there was appreciable folding of the thin film instead of interface delamination as seen in Figures 6e and 6f. These observations indicate that the mechanism of interface crack propagation is dependent on the weak interface pattern and need to be analyzed in detail.

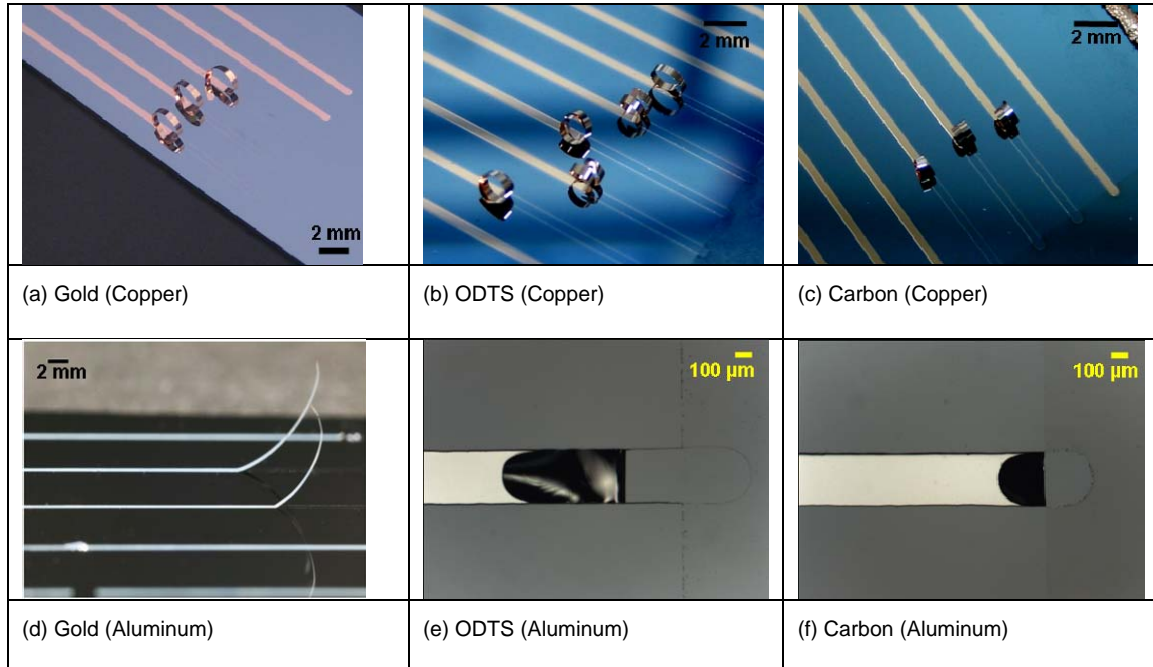


Figure 6: Images showing thin film delaminations after laser-induced pulse loading for all the three weak interface and two thin film material combinations.

Conclusion

Using a combination of shadow mask technique, photolithography, and soft lithography the gold, carbon, and ODTS weak interfaces were successfully patterned beneath thin film structures. The dynamic adhesion tests results show repeatability in the delamination lengths. The mechanism of thin film delamination seems to be very sensitive to the weak interface material and needs to be analyzed in detail.

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