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Characterization of thin film adhesion by MEMS shaft-loading blister testing

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A new microelectromechanical system shaft-loaded blister test was developed and demonstrated to provide stability, repeatability, and simultaneous quantitative measurements of adhesion between thin films deposited on a silicon substrate. The authors assessed adhesion of sputtered platinum, copper, and chromium/copper (300 nm) to underlaying atomic layer deposited (ALD) aluminum oxide. The average adhesion energies for thin films on ALD aluminum oxide were found to be $1.15 \pm 0.1\ \mathrm{J/m^2}$ for platinum thin films, $1.4\ \mathrm{J/m^2}$ for copper thin films, and $1.75\ \mathrm{J/m^2}$ for chromium/copper. © *2013 American Vacuum Society*. [http://dx.doi.org/10.1116/1.4801921]

I. INTRODUCTION

The performance, yield, and reliability of multilayered microelectromechanical systems (MEMS) strongly depend on adhesion properties between layers. Delamination–adhesion issues are significant, not only during the fabrication process but also during the operation of the micro- and nanodevices.[1–4]

Many different adhesion tests have been developed to measure debonding energies of thin films, including scratching, peeling, and nanoindentation tests.[5–8] These techniques are simple and easy to perform, but analysis and interpretation of results are challenging and ambiguous.[9] For macroscopic samples, obtaining quantitative, controllable, and accurate adhesion analysis can be achieved by shaft-loading blister testing (SLBT). The shaft-loading blister tests were first proposed by Malyshev and Salganik,[10] and an analytical solution was later demonstrated by Wan and Mai.[11] In the traditional set-up, the central pointed shaft is directly applied to the film and the adhesion energy is determined by measuring the delaminated area and the corresponding applied load. This technique has been applied for characterization of polymers[12,13] or relatively thick metal films in the range of several millimeters,[14] since very thin and inflexible films can undergo rupture before the delamination occurs.

The novelty of the process introduced in this paper is that it combines a noncontact shaft-loaded blister test with a microindenter in order to characterize the spot of adhesion between thin films located on a silicon substrate (Fig. 1). The microindenter pushes a microcylinder so that direct contact with thin films is avoided. The proposed technique is generic and can be applied to a variety of thin films in addition to the sputtered metals evaluated in this work.

II. EXPERIMENTAL METHODS

A. Fabrication of the test structure

The test structure consists of an array of silicon microcylinders (with diameters 1000 and 750 μm), which are

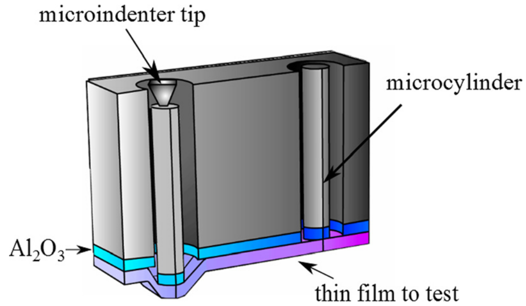

FIG. 1. (Color online) MEMS shaft-loading blister testing; delamination is initiated by the push of a microindenter tip. The chip is attached to a silicon frame (not shown) that provides space for vertical displacement.

[a)]Electronic mail: maria.berdova@aalto.fi supported by a double layer: atomic layer deposited (ALD) Al_2O_3 (alumina) and the thin film under investigation. The test structures were fabricated according to following steps:

(1) Atomic layer deposition of 200 nm Al_2O_3 layer on the top side of the double-side polished silicon (380 μm) wafers with trimethyl aluminum and water ($TMA + H_2O$) precursors at 220 °C.
(2) Deposition of 20 nm ALD Al_2O_3 on the bottom side of the wafer as an etch stop mask layer.
(3) UV lithography with standard AZ5214E photoresist on the backside of the substrate.
(4) Etching of backside Al_2O_3 in buffered hydrofluoric acid (BHF).
(5) Etching of Si annular rings through the whole wafer thickness using SF_6, C_4F_8, and O_2 gases in the Bosch process. This step also formed lanes for chip separation [Fig. 2(b)].
(6) Magnetron sputtering of 300 nm of copper, chromium-copper, or platinum thin films on the top of 200 nm Al_2O_3 film.
(7) Removal of 200 nm alumina support layer from the annular cavities by BHF for 5 min wet etching to release the film to be tested.

Processed samples are shown from top and back sides in Fig. 2. Each individual chip contains a 3×3 array of identical microcylinders. Visible corrugations in the released film are

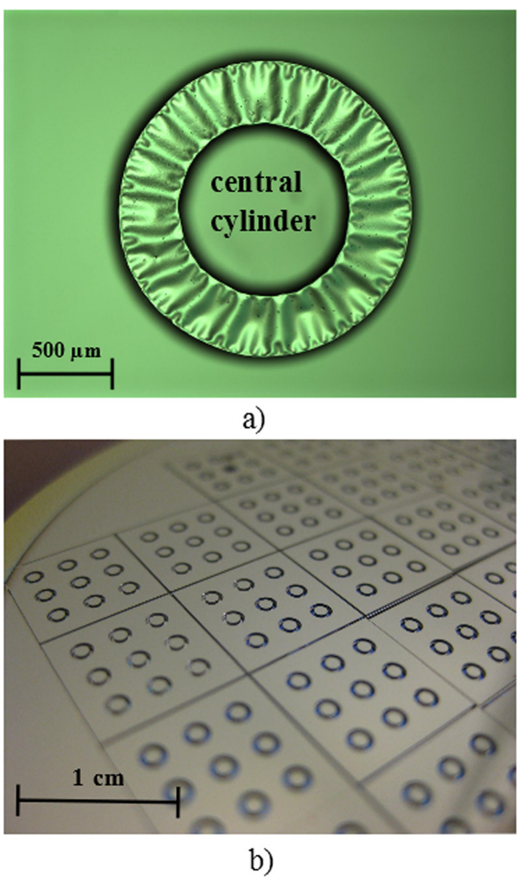

FIG. 2. (Color online) MEMS SLBT samples. (a) Top view. (b) Bottom view, showing 3×3 arrays.

due to compressive stresses generated during the low-temperature (<300 °C) sputtering.

For the indentation measurements, the chips were flipped around and fixed to a silicon frame by photoresist acting as a glue. Supporting silicon frames were fabricated by lithography and plasma etching. The frames provide free space for the central shaft to move during the indentation. Furthermore, the frames with samples were glued to steel holders using photoresist to ensure mechanical robustness during the measurements.

In the present work, we studied 11 Pt/Al_2O_3 samples with various geometries (pillar radii were 500 or 350 μm and the etched annular rings were 100, 300, or 550 μm) to quantitatively and qualitatively examine the MEMS shaft-loading blister test. Samples of Cu/Al_2O_3 and $Cu/Cr/Al_2O_3$ also were investigated to compare adhesion energies.

B. Microindentation

The samples were examined by a shaft-loaded blister test with the help of a CSM Microindenter. The Microindenter used a 20 μm radius conospherical HRC tip to apply the load to the center of the circular pillar, thereby inducing the deflection and the delamination between thin films (Fig. 1). When a microcylinder is actuated by an indenter, the thin film is strained, which causes debonding, thereby facilitating adhesion measurements. During the indentation test, the load and displacement values were measured and recorded. The load was applied to the indenter through a calibrated electromagnetic coil and displacement was measured as the change in capacitive signal (operation principle of CSM Microindenter). As a result, the load values were recorded as a function of the indentation depth in the form of loading–unloading curves.

C. Methodology

Indentation consists of several steps. Examples of the load and displacement curves and mechanical behavior of the samples are presented in Fig. 3. The first step is the approach of the indenter to the sample surface [Fig. 3(a)]. Next, the film is stretched by gradually increasing load [Fig. 3(b)] until the threshold value at which delamination starts [Fig. 3(c)]. This is observed as a significant change in the slope of the displacement curve's behavior. Then the delamination continues by further increasing the load to a preset value [Fig. 3(d)], at which the film is held for about 15 s, followed by gradual unloading.

D. Analytical solution

The traditional approach to calculating adhesion for the shaft-loading blister tests is based on the consideration that with shallow deflection angles, the films do not bend during the stretching.[11] During MEMS shaft-loading blister testing, debonding is initiated along the film/film interface via central load. Opening the interface between two thin films starts at a threshold value, after which the debonding continues. During delamination, the crack growth a_2 (instantaneous radius), deflection w_1, and load P are monotonically increasing

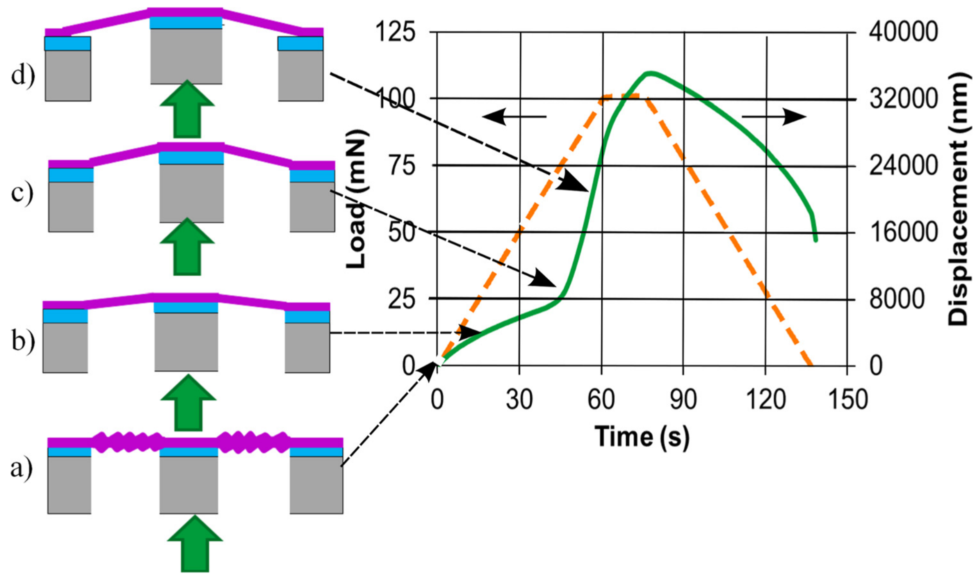

FIG. 3. (Color online) Load-displacement graph with respect to time where dashed curve is the load, and solid curve corresponds to the displacement. Interpretation of the displacement curve is indicated by arrows: (a) approach of an indenter to the sample surface, (b) stretching of a film, (c) starting point of delamination, and (d) delamination of the film.

(Fig. 4). Therefore, work of adhesion is assessed as energy released in the debonded blister area. According to the theoretical model, which has been developed and demonstrated by Wan,[11] the work of adhesion is

$$W = \left(\frac{1}{16\pi^4 Eh}\right)^{1/3}\left(\frac{P}{a_2}\right)^{4/3}, \tag{1}$$

where h is the thickness of the film, E is the Young's modulus of the thin film, and the central point load P is described by

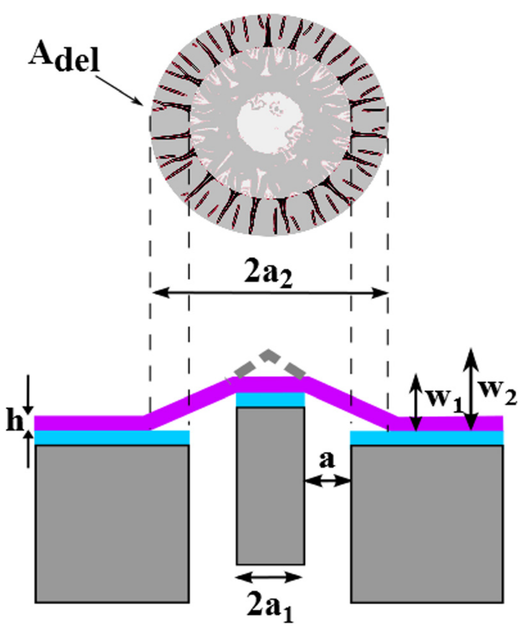

FIG. 4. (Color online) Determination of the delaminated area from an optical image and corresponding cross-section of MEMS SLBT.

$$P = \frac{\pi E h w_2^3}{4a_2^2}, \tag{2}$$

where w_2 is the central vertical deflection, which depends on blister geometry.

In the case of the MEMS test structure, the blister geometry can be assumed to be truncated, and the central deflection is $w_2 = w_1 a_2/a_2 - a_1$ (Fig. 4).

An alternative approach to measuring work of adhesion is to consider the energy dissipated per unit area upon extending a crack along the interface, or

$$W = \frac{W_d}{A_{\rm del}}, \tag{3}$$

where the delaminated area $A_{\rm del}$ is determined by an optical microscope or $A_{\rm del} = \pi(a_2^2 - (a + a_1)^2)$, and the energy dissipation W_d is defined as the hysteresis area from loading and unloading curves (Fig. 5) or

$$W_d = \int_0^1 F dl + \int_1^2 F dl + \int_2^3 F dl. \tag{4}$$

III. RESULTS AND DISCUSSION

Typical load versus displacement curves for Pt/Al_2O_3 samples of various geometries are displayed in Fig. 5. The plots justify the critical transition from elastic to plastic behavior, exhibiting the threshold points (points 1) of delamination. As discussed before, pillar radii of 500 and 350 μm and etched annular rings of 100, 300, and 550 μm were tested. The approaching speed of the tip was 4 μm/min, the loading rate was chosen as 100 mN/min, and the holding time was kept constant for the each measurement (15 s). In

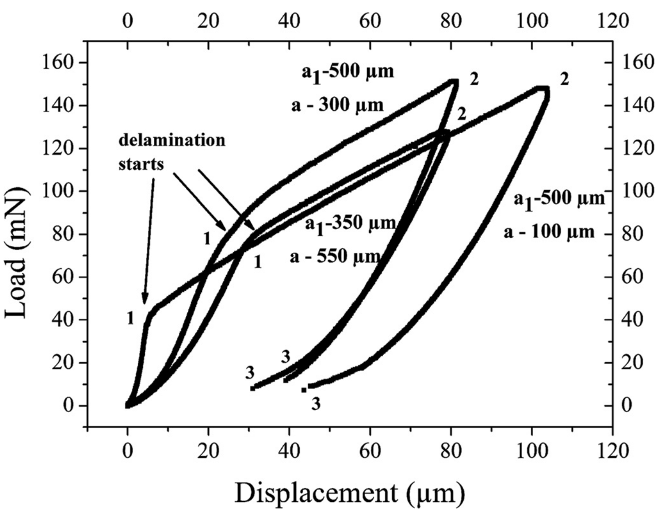

FIG. 5. Loading-unloading curves for various blister geometries of Pt/Al_2O_3 samples. Hysteresis area (1 – 2 – 3) is equal to dissipated energy *Wd*.

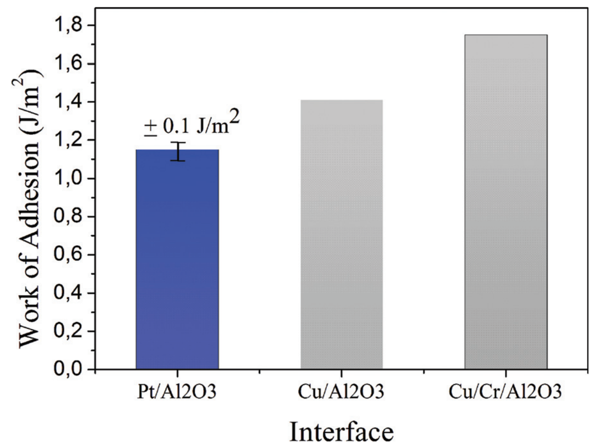

FIG. 6. (Color online) Work of adhesion of platinum, copper, and copper/chromium to ALD Al_2O_3 [evaluated from Eq. (1)].

our initial experiments, we observed artifacts arising from off-normal loading, and therefore, we aligned the tip to the center of the pillar using the indenter microscope optics.

Table I summarizes the results obtained after microindentation of the samples that have 300 nm thin film materials. The maximum displacement corresponds to the selected maximum load (100 or 150 mN), exceeding of which would lead to the breakage of thin film during the test. The error in the result is the statistical deviation of the measured values. Threshold displacement values indicate the starting moment of delamination, which can be used to qualitatively assess thin film adhesion. A thin film with lower adhesion strength starts to delaminate earlier than a film with stronger bonding energy.

Quantitative assessment of adhesion work is obtained using Eq. (1), where P and w_2 are extracted from loading-unloading curves, and delaminated area A_{del} is measured from an optical microscope image. Fig. 6 represents the average values of adhesion work for Pt/Al_2O_3, Cu/Al_2O_3, and $Cu/Cr/Al_2O_3$ samples. A number of samples were delaminated as planned. However, in several cases, the film fractured before delamination, which limited the number of successful tests. Therefore, in the present work, 11 successful Pt/Al_2O_3 samples were investigated, and a Cu/Al_2O_3, $Cu/Cr/Al_2O_3$ samples were studied for comparison of adhesion work. The obtained interfacial adhesion energies were $1.15 \pm 0.1\,J/m^2$, $1.4\,J/m^2$, and $1.75\,J/m^2$, respectively, for Pt/Al_2O_3, Cu/Al_2O_3, and $Cu/Cr/Al_2O_3$ samples. As expected, a chromium underlayer seems to improve the adhesion between copper and aluminum oxide, and the platinum/alumina interface has lower adhesion strength than copper/alumina. We have not observed the delamination for highly adhering thin films of silicon nitride and silicon carbide, as after certain displacement (7 μm for nitride, 12 μm for carbide), the films start to break with no debonding events. Therefore, further development and testing are needed for highly interfacial adhesion materials.

The alternative approach [Eq. (3)] leads to similar results. The work of adhesion for the Pt/Al_2O_3 interface is calculated to be $1.16 \pm 0.25\,J/m^2$. The result indicates that both approaches can be used. However, previous studies reported work of adhesion values of $0.3\,J/m^2$ for Pt/Al_2O_3 and $0.4–0.7\,J/m^2$ for Cu/Al_2O_3 interfaces,[15,16] where Al_2O_3 is a bulk material and Pt and Cu are sputtered. The difference in adhesion values may be due to different bonding mechanisms between bulk/thin film and film/thin film interfaces.

Platinum/alumina structures were tested to assess reproducibility and stability of the MEMS shaft-loading blister test. Identical loading/unloading curves for each repeated measurement were obtained, which justify high reproducibility of the used technique. In addition, the obtained values of adhesion energies between Pt and Al_2O_3 confirm good repeatability (91.3%). The stability of our method can be attributed to precise control of the delamination rate. The critical load per unit area remained the same, with mean and standard deviations of $33.5 \pm 0.7\,mN/mm^2$ for a variety of samples with different geometries.

TABLE I. Force-displacement data from indentation experiment (cylinder diameter 1000 μm, annular ring 100 μm).

Thin film, 300 nm	Threshold displacement value w^* (μm)	Threshold load value P^* (mN)	Maximum displacement w_{max} (μm)	Maximum load w_{max} (mN)	Delaminated area A_{del} (mm^2)
Platinum	5.2 ± 0.4	42 ± 2	70.0 ± 3.4	100.4 ± 1.5	3.56 ± 0.35
Copper	8	25	34	100	0.94
Chromium/copper	11.5	35	70	150	1.76

IV. SUMMARY AND CONCLUSION

MEMS shaft-loading blister testing was performed to quantitatively and qualitatively assess the adhesion work for Pt/Al_2O_3, Cu/Al_2O_3, and $Cu/Cr/Al_2O_3$ interfaces. The average adhesion energy for platinum thin films on ALD aluminum oxide was determined to be $1.15 \pm 0.1\,J/m^2$ by two approaches. In the first approach, adhesion work was assessed by equation $W = (1/16\pi^4 Eh)^{1/3}(P/a_2)^{4/3}$. In alternative approach, work of adhesion was measured taking into account the energy dissipated per debonded blister area. In comparison, the adhesion for the Cu/Al_2O_3 system was found to be $1.4\,J/m^2$, and the Cr underlayer acted as an adhesion promotion layer for copper deposited on alumina (adhesion energy increased to $1.75\,J/m^2$).

The developed method shows high stability, delamination control, and reproducibility. The proposed MEMS shaft-loading blister test might become a valuable tool for quantitative and qualitative assessment of adhesion between various thin films.

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