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Fundamental Mechanisms of Micromachine Reliability

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Fundamental Mechanisms of Micromachine Reliability

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Abstract

Due to extreme surface to volume ratios, adhesion and friction are critical properties for reliability of Microelectromechanical Systems (MEMS), but are not well understood. In this LDRD we established test structures, metrology and numerical modeling to conduct studies on adhesion and friction in MEMS. We then concentrated on measuring the effect of environment on MEMS adhesion.

Polycrystalline silicon (polysilicon) is the primary material of interest in MEMS because of its integrated circuit process compatibility, low stress, high strength and conformal deposition nature. A plethora of useful micromachined device concepts have been demonstrated using Sandia National Laboratories' sophisticated in-house capabilities. One drawback to polysilicon

is that in air the surface oxidizes, is high energy and is hydrophilic (i.e., it wets easily). This can lead to catastrophic failure because surface forces can cause MEMS parts that are brought into contact to adhere rather than perform their intended function.

A fundamental concern is how environmental constituents such as water will affect adhesion energies in MEMS. We first demonstrated an accurate method to measure adhesion as reported in Chapter 1. In Chapters 2 through 5, we then studied the effect of water on adhesion depending on the surface condition (hydrophilic or hydrophobic). As described in Chapter 2, we find that adhesion energy of hydrophilic MEMS surfaces is high and increases exponentially with relative humidity (RH). Surface roughness is the controlling mechanism for this relationship.

Adhesion can be reduced by several orders of magnitude by silane coupling agents applied via solution processing. They decrease the surface energy and render the surface hydrophobic (i.e. does not wet easily). However, only a molecular monolayer coats the surface. In Chapters 3-5 we map out the extent to which the monolayer reduces adhesion versus RH. We find that adhesion is independent of RH up to a threshold value, depending on the coating chemistry. The mechanism for the adhesion increase beyond this threshold value is that the coupling agent reconfigures from a surface to a bulk phase (Chapter 3). To investigate the details of how the adhesion increase occurs, we developed the mechanics for adhesion hysteresis measurements. These revealed that near-crack tip compression is the underlying cause of the adhesion increase (Chapter 4). A vacuum deposition chamber for silane coupling agent deposition was constructed. Results indicate that vapor deposited coatings are less susceptible to degradation at high RH (Chapter 5).

To address issues relating to surfaces in relative motion, a new test structure to measure friction was developed. In contrast to other surface micromachined friction test structures, uniform apparent pressure is applied in the frictional contact zone (Chapter 6). The test structure will enable friction studies over a large pressure and dynamic range.

In this LDRD project, we established an infrastructure for MEMS adhesion and friction metrology. We then characterized in detail the performance of hydrophilic and hydrophobic films under humid conditions, and determined mechanisms which limit this performance. These studies contribute to a fundamental understanding for MEMS reliability design rules. They also provide valuable data for MEMS packaging requirements.

EXECUTIVE SUMMARY

Surface micromachining is a new technology area in which Sandia National Laboratories has demonstrated a leading technology. The advantages of MEMS are (1) very high functionality/cost ratios, and (2) the small volume these devices occupy, so that very sophisticated sensing and actuation functions can be performed in a small volume. MEMS are still in the prototyping stage of development. To take new micromachined designs from the prototype phase into the field, core competencies that need to be developed include MEMS tribology, MEMS packaging, MEMS process science (understanding the processes well enough to predict properties of components, and knowing what process windows are), and MEMS model-based design (tools that use MEMS properties in the design process so that we understand the design windows).

Tribology is important for MEMS applications in which surfaces may contact, or are allowed to come into contact. Because of the very large surface to volume ratios in MEMS, surface forces can dominate over inertial forces. In particular, adhesion (also known as stiction) can cause catastrophic failure of MEMS devices, and requires detailed characterization. The surface forces in a MEMS device depend rather strongly on the structural material, its surface roughness, whether or not a coating has been applied, the ambient it has been exposed to, and the usage it has experienced. It is very important to study and gain control over MEMS tribological properties. The focus of this LDRD was on MEMS tribology, and the main objective was to develop a metrology to quantify the major tribological properties -- adhesion, adhesion hysteresis, and friction. This was accomplished in such a way that relationships between these properties can be studied in the future. A second objective was to relate these properties to surface roughness, chemistry and ambient. We were able to make such relationships for hydrophilic and hydrophobic surfaces. The most important finding in this study is that if wetting of MEMS surfaces can be prevented, adhesion values are acceptably low.

This report is organized in self-contained Chapters. The interested reader should be able to read any given Chapter without having detailed knowledge of the other Chapters. We now summarize the main findings of each Chapter.

Chapter 1: We demonstrated that an S-shaped cantilever beam configuration should be adopted for detailed studies of adhesion forces in surface micromachining, in contrast to the previously accepted arc-shape beam. This beam configuration was not previously used because an out-of-plane metrology technique had not been applied in a quantitative manner. Applying Michelson interferometry as our metrology, we validated out-of-plane deflections predicted by beam theory. With interferometry, beam deflections are measured at the nm scale, increments in the crack length s are well resolved, and the adhesion of the S-shaped beam is accurately assessed. Using the S-shaped beam gives great latitude in measuring adhesion because the equilibrium is deep and easily attainable from either side of the equilibrium. Studies such as the effect of environment on adhesion are readily adapted to this method. The area requirement on a MEMS wafer is also much smaller, because each S-shaped adhered beam gives a value for adhesion. This is important because of the cost of real estate on a MEMS wafer.

Chapter 2: We studied and analyzed the effect of relative humidity (RH) on adhesion for hydrophilic polysilicon surfaces (which wet easily) under equilibrium conditions. The data demonstrates that adhesion increases exponentially with RH. This was a surprising result, because adhesion had been thought to be independent of humidity for hydrophilic surfaces. Our analysis of the data indicates that we can employ a single asperity model to understand the effect of RH on adhesion. At very high RH, the MEMS surfaces are fully wetted, and adhesion dominates the performance of the MEMS devices. These data and modeling improve our fundamental understanding of adhesion (e.g., stiction) in MEMS, and can lead to model-based calculations for MEMS design and packaging technology.

Chapter 3: We performed a study similar to that in Chapter 2, but for hydrophobic polysilicon surfaces (which do not wet easily) created by the application of silane coupling agent molecular monolayers. A simple macroscopic model, which predicts that adhesion should be independent of RH, works well below a threshold value of RH 95% and 80% RH for the ODTS ($C_{18}H_{37}SiCl_3$) and FDTS ($C_8F_{17}C_2H_4SiCl_3$) coatings respectively. Adhesion increased by a factor of two after prolonged exposures at >95% RH for ODTS, but by 500 times for FDTS after similar exposures. This was surprising, because FDTS exhibits lower adhesion energy at low RH than does ODTS. Ellipsometry measurements at high RH showed similar

water adsorption by the two coupling agents. This did not explain the experimental results, but the technique could not be applied over a period of several hours, which was the time scale for the adhesion increase. Atomic force microscopy performed on the actual samples after the exposure to high RH revealed agglomerated coating material, suggesting a permanent redistribution of the monolayer film into isolated vesicles. This agglomeration was more prominent for FDTS than ODTs. These findings suggest a new mechanism for uptake of moisture under high humidity conditions. At high humidities, the silane coatings can reconfigure from a surface to a bulk phase leaving behind locally hydrophilic sites which increase the average measured adhesion energy. In order for the adhesion increase to be observed, the humidity must be high and a significant fraction of the monolayer (much more than 5%) must be converted from the surface to the bulk phase.

Chapter 4: To investigate the mechanism for the increase of the hydrophobic surfaces of Chapter 3 in more detail, we developed a new experimental approach for measuring hysteresis in the adhesion between micromachined surfaces. By accurately modeling the deformations in cantilever beams that are subject to combined interfacial adhesion and applied electrostatic forces, we determined adhesion energies for advancing and receding contacts. We drew on this new method to examine adhesion hysteresis for silane coated micromachined structures and found significant hysteresis for surfaces that were exposed to high relative humidity (RH) conditions. We used contact deformation models to show that the compliance of the vesicles found in Chapter 3 could reasonably account for the adhesion hysteresis that develops as the surfaces are forced into contact by an externally applied load.

Chapter 5: We devised an alternate method for coating MEMS devices with a hydrophobic fluorocarbon film, which avoids many of the problems associated with solution-based reaction of alkylchlorosilanes. In this process we used a volatile fluoroalkylsilane as precursor for a chemical vapor deposition (CVD) process. In a vacuum chamber, controlled amounts of silane precursor and water vapor react on the surface of the sample to form silanols, and eventually siloxane linkages, with a fluoroalkyl side chain to provide a hydrophobic, low energy surface. The vapor phase coating processes have a number of distinct advantages compared to solution-based processes. We avoid a number of problems associated with solution-based coating, including wetting of high aspect ratio structures,

diffusion limited transport of reagents into confined areas, control of minute quantities of dissolved water in nonaqueous solvents, slow reaction kinetics of chlorosilanes in solution, and disposal of organic solvent waste. Vapor phase processes provide efficient transport into high-aspect ratio structures, provide good control of reagent supply, have no solvent waste, and provide for convenient in-situ cleaning of surfaces immediately prior to deposition. This CVD process does require that mechanical parts be released and dried prior to introduction to the deposition chamber, so that sacrificial oxide etching must be followed by a supercritical CO₂ or sublimation drying procedure to produce free, released parts.

Chapter 6 We describe the design, modeling, fabrication and initial testing of a new test structure for friction measurement in MEMS. The device consists of a cantilevered forked beam and a friction pad attached via a hinge. Compared to previous test structures, the proposed structure can measure friction over much larger pressure ranges, about 3 orders of magnitude, yet occupies ten to one hundred times less area. The placement of the hinge is crucial to obtaining a well-known and constant pressure distribution in the device. Static deflections on the device were measured and modeled numerically. Preliminary results indicate that friction pad slip is sensitive to friction pad normal force. The device also has a large range, about 5 orders of magnitude, over which velocity related effects on friction can be studied. In subsequent work not reported here, we were able to increase the slip to ~40 nm, so that frictional slippage will be representative of real MEMS surfaces.

In summary, we developed an infrastructure to measure tribological properties (adhesion, adhesion hysteresis and friction) in MEMS in this LDRD project. We then investigated in detail the effect of ambient moisture on MEMS surfaces. We also constructed and demonstrated a vapor phase coating process for MEMS which shows promise as a more controllable and manufacturable method to deposit coatings than the solution phase. The interferometry metrology we applied was also found to be very useful for measurement of mechanical properties in MEMS.

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1. Accurate method for determining adhesion of cantilever beams (de Boer and Michalske)

Abstract

Using surface micromachined samples, we demonstrate the accurate measurement of cantilever beam adhesion by using test structures which are adhered over long attachment lengths. We show that this configuration has a deep energy well, such that a fracture equilibrium is easily reached. When compared to the commonly used method of determining the shortest attached beam, the present method is much less sensitive to variations in surface topography or to details of capillary drying.

1. Introduction

Microelectromechanical Systems or MEMS [1] are now being used in selected commercial products including airbag accelerometers for automobiles [2] and active optical elements [3] for projection displays. Due to their potential for low cost manufacturing, many other microdevice designs and applications are currently being developed. Microrelays [4], gyros [5], optical switches [6] and security devices [7] are just a few examples. However, auto-adhesion, or spontaneous sticking between MEMS structures, remains a major limitation in bringing this new class of engineering devices to the broader market. MEMS are particularly susceptible to auto-adhesion because the structural members: (1) are constructed in close proximity to each other (within several μms), (2) are highly compliant due to their extreme length to thickness aspect ratio and, (3) have large surface to volume ratios which increase the relative importance of adhesive surface forces. If the miniature structural members are brought together by surface (capillary, electrostatic) or inertial (shock, rapid air flow) forces, they may remain adhered after the external force is removed. Auto-adhesion can lead to catastrophic failure of a MEMS device.

From a practical point of view, auto-adhesion is known to limit manufacturing yield of silicon-based, surface micromachined MEMS. In these devices, structural members are fabricated using successively patterned depositions of thin film polycrystalline silicon (polysilicon) and sacrificial oxide layers. This manufacturing approach is a direct outgrowth of silicon-based microelectronics. Auto-adhesion can occur during the final step of surface micromachine fabrication after the polysilicon structural elements are rendered mechanically free by selectively etching away the sacrificial oxide layers in a hydrofluoric acid solution, which does not dissolve the polysilicon. During the final stage of drying from the wet etch, capillary menisci form between the released beams and the substrate and can generate forces that collapse the structural member onto the substrate. Subsequently, the polysilicon material may remain adhered to the substrate after the water dries. The problem is also commonly referred to as "stiction". Numerous efforts have been directed at measuring autoadhesion and correlating to chemistry or roughness of the interface [8-19]. A supercritical drying procedure can be used to eliminate capillary forces during drying and prevent surfaces from coming into contact initially [20]. However, this approach does not address autoadhesion of surfaces that come into contact while devices are in operation. Many coating processes aimed at reducing the surface energy of polysilicon have also been explored [8, 21-23]. Even though some of these coating strategies, particularly those based on silane coupling agents, have shown some promise for improving manufacturing yield and MEMS performance, their optimization requires a quantitative approach for measuring surface energy directly on micromachined devices. The interacting effects of coating material, roughness and environmental aging on adhesion and friction must be understood in order to guarantee that the effect of surfaces forces on performance and reliability of micromachines will allow proper operation of devices over their lifetimes.

A theory and experimental method for measuring surface energy of micromachined cantilever beams has been proposed by Mastrangelo and Hsu [10-12]. They model the role of capillary forces in bringing beams into contact with the substrate and determine critical beam lengths for beam collapse [11]. As drying continues, the capillary volume diminishes leaving

only surface driven interfacial adhesion. Adhesion of the dried cantilever beam is predicted by considering the elastic energy in the deformed beam, which is attempting to pull the beam up off the substrate, and the surface energy that is promoting continued adhesion. By considering these two factors, Mastrangelo and Hsu calculated peel bounds [12] and the adhered length in the limit where the capillary volume vanishes.

Mastrangelo and Hsu [10] predict two configurations in which auto-adhered cantilever beams may be found (we will subsequently refer to ref. [10] as M&H). Long beams are adhered over a large fraction of their length, and bend into an S-shape as in Fig. 1(a). The non-adhered length from the support post to the point at which the beam comes into contact with the substrate is s , and the length of the beam L is significantly greater than s . In Chapter 1 and throughout this report, we assume a fracture mechanics formalism [24], and call s the crack length. Alternatively, short adhered beams contact the substrate only at their tip, and the beams assume an arc-shaped deformation as in Fig. 1(b). In this case, the crack length s is very nearly equal to L .

Fig. 1 (a) S-shaped beams ($m=0$) are attached over a long length d . (b) Arc-shaped beams ($m=3/2$) are attached only very near their tips.

M&H argue that either adhesion geometry can be used to extract a quantitative measure of micromachine surface energy. For S-shaped beams, the crack length s can be used to determine the apparent surface energy. However, an out-of-plane measurement technique is required to determine s . Because such a method is not commonly available in laboratories, M&H recommend using the arc-shaped beam. Then, using a high power objective on an optical

microscope, adhesion energy is determined by observing the shortest beam to remain adhered within an array of beams of various lengths. M&H demonstrated the shortest adhered beam approach on polysilicon micromachined beams having both hydrophilic and hydrophobic nature. Although their measurements yield surface energy of reasonable magnitude, they were not able to distinguish between hydrophilic and hydrophobic surfaces.

In this section, we examine the adhesion measurement approach proposed by M&H in detail. Using interferometry to measure beam deformations point by point, we extend the experimental measurement to include adhesion of S-shaped beams. By taking this approach we directly address the following outstanding questions:

- (1) Do the deformations predicted by M&H, which are subsequently used to calculate strain energy, match actual beam deflections?
- (2) What is the behavior of beams in the transition from the S-shape to the arc-shape? How are the equilibria for the S-shaped and arc-shaped beams attained?
- (3) Are the values for adhesion between S-shaped and arc-shaped beams equivalent? If not, what factors influence measured differences?

II. Mechanics of Adhered Cantilever Beams

A) Beam Deformations

The total energy of the adhered beam is a sum of (a) the elastic energy stored by bending a beam into contact with the substrate, and (b) the surface energy reduction achieved by forming a beam/substrate interface. Solving for the minimum total energy system allows one to directly determine the equilibrium surface energy. The first step in this approach is a quantitative evaluation of the stored elastic energy in an adhered beam.

In the absence of externally applied forces, the deformations for an adhered beam are given by M&H as

$$u(\eta) = h\eta^2 [(m(s)-2)\eta + (3-m(s))] \quad , \quad \eta = \frac{x}{s}, \quad (1)$$

where h is the height of the support post, t is the thickness of the beam, L is the beam length and s is the crack length (see Fig. 1). The attachment length d is the length along which the two materials are in contact, and $d \approx L-s$. The slope parameter m is defined by $\theta = m(h/s)$, where θ is the shear angle of the beam tip. From M&H, the functional form of m derive from beam shear theory is

$$m(s) = \frac{\frac{16}{5} \left(\frac{t}{d}\right)^3 \left(\frac{t}{s}\right) \left[1 + \frac{15}{32} \left(\frac{d}{t}\right)^2 \left(\frac{E}{G_s}\right)\right]}{1 + \frac{32}{15} \left(\frac{t}{d}\right)^3 \left(\frac{t}{s}\right) \left[1 + \frac{15}{32} \left(\frac{d}{t}\right)^2 \left(\frac{E}{G_s}\right)\right]}, \quad (2)$$

where E and G_s are the Young's and shear modulus respectively. $E/G_s = 2(1+\nu) \approx 2.44$ where $\nu=0.22$ is Poisson's ratio for silicon.

For the S-shaped beam, $d \gg t$. Consequently, m approaches zero implying negligible shear deformations at the crack tip s . In the limit when s approaches L , m is a strong function of d , reaching a maximum value of $3/2$ for vanishing d . The non-zero slope parameter corresponds to shear deformations induced when adhesive contact is localized to the beam tip.

The assumptions in the M&H analysis are (1) small deformations such that linear elasticity applies, (2) a rigid cantilever support post and substrate, (3) free slip of the beam over the substrate, (4) attractive forces that operate only between contacting portions of the beam and substrate surfaces, (5) no residual strain (curvature) in the beam, and (6) perfectly smooth beam and substrate surfaces.

B) Mechanical Equilibrium

In this section, we extend the analysis of M&H to investigate the mechanical equilibria of the S-shaped and arc-shaped cases. We shall find that the energy well is much deeper for the S-shaped than the arc-shaped deformation and that m can *only* take on values near 0 or near $3/2$.

Using the deformation characteristics from the previous section, the elastic strain energy in an adhered beam is given by

$$U_E = \frac{EI}{2} \int_0^s \left(\frac{d^2u}{dx^2} \right)^2 dx = \frac{6EIh^2}{s^3} \left(1 - m(s) + \frac{m^2(s)}{3} \right) \quad (3)$$

where $I = wt^3/12$ is the beam moment of inertia. Eq. (2) shows that as the crack length s decreases, the stored elastic energy increases. The surface energy U_s is

$$U_s = -\Gamma w(L-s) \quad (4)$$

where Γ is the energy of adhesion per unit area. The surface energy term is negative because energy is reduced when surfaces come into contact. For clean smooth surfaces which are reversibly separated, we expect the adhesion $\Gamma = 2\gamma$, where γ is the surface energy of the material in question. Because the beam and substrate are made of the same material, no separate interfacial energy exists and the adhesion energy Γ is simply twice the surface energy. In actual micromachined beams, factors such as surface roughness and capillary condensation must be considered when evaluating the effective surface energy.

The total system energy U_T is the sum of the elastic strain energy and the adhesive surface energy:

$$U_T = U_E + U_s = \frac{6EIwh^2}{s^3} \left(1 - m(s) + \frac{m^2(s)}{3} \right) - \Gamma w(L-s) \quad (5)$$

System equilibrium is determined by stable values of U_T defined by

$$\frac{dU_T}{ds} = 0 \quad (6a),$$

and
$$\frac{d^2 U_T}{ds^2} > 0 \quad (6b)$$

To characterize the system equilibrium, we determine stable values of U_T for various length beams assuming a fixed value of Γ . A straightforward method to solving Eq. (6) is by the graphical method. It is necessary to eliminate d from Eq. (2) using $d \approx L - s$. Then, Eq. (2) can be rewritten as

$$m(s) = \frac{A(s)}{1 + (2/3) A(s)}, \quad (7)$$

where $A(s)$ is defined in terms of known constants and variable s as

$$A(s) = \frac{16}{5} \left(\frac{t}{L-s} \right)^3 \left(\frac{t}{s} \right) \left[1 + 1.144 \left(\frac{L-s}{t} \right)^2 \right]. \quad (8)$$

Using (5), (7) and (8), we plot in Fig. 2 U_T versus s for various values of L assuming $\Gamma = 3.66 \text{ mJ/m}^2$, $t = 2.3 \text{ }\mu\text{m}$, $h = 1.8 \text{ }\mu\text{m}$ and $w = 20 \text{ }\mu\text{m}$ (reflecting the data for hydrophobic samples in section III below). The state in which the system will reside is the lowest *reachable* minimum for U_T after drying. In Appendix 1, we estimate two characteristic lengths, $L_{tip,0}$ and $L_{tip,c}$. The former is the minimum length beam which will be brought into contact with the substrate at an angle of 0° at its tip, while the latter is the shortest beam which will be brought into contact at an angle greater than 0° . The reachable minimum, after drying is complete, depends on the shortest value of $s = s_{\min}$ achieved while external forces pull the beam into contact with the substrate. As seen in Appendix 1, this depends on both the surface tension of the liquid as well as its contact angle with the substrate. In this example, we shall take $s_{\min} = L_{tip,0} = 200 \text{ }\mu\text{m}$ for S-shaped beams, reflecting a receding contact angle during the drying process of $\theta_{cr} = 80^\circ$.

Fig. 2(a) is a graph of U_T and m versus s for $L = 400 \text{ }\mu\text{m}$. For small s , U_T grows large due to bending strain energy U_E , while m is near 0. Note that m (plotted on a logarithmic scale)

Fig. 2 (a) U_T and m vs. s for $L=400 \mu\text{m}$ and $\Gamma=3.66 \text{ mJ/m}^2$. Local minima exist at s_1^* and s_3^* . The minimum in U_T is approximately -7 pJ . (b) At $L=275 \mu\text{m}$, the relative values of U_T at s_1^* and s_3^* have changed. (c) At $L=242.3 \mu\text{m}$, the extrema at s_1^* and s_2^* have merged into s_{12}^* . (d) Closeup of (c) in the vicinity of s_3^* . The minimum in U_T at s_3^* is only 10 fJ .

is not exactly 0 because the theory accounts for beam shearing at the crack tip, which exists to a small degree even for the S-shaped beam. For large s , U_T increases because surface energy U_s becomes substantial. However, as $s \rightarrow L$, m rapidly begins to increase towards $3/2$, reflecting the change from the S-shape to the arc-shape. A commensurate decrease in U_E occurs, and is manifested as a local minimum in U_T . Regarding stability, at $s_1^*=229$, $s_2^*=398.2$ and $s_3^*=399.76 \mu\text{m}$ (6a) is satisfied, but (6b) is satisfied only at $s_1^*=229$ and $s_3^*=399.76 \mu\text{m}$ (s^* refers to the local extrema). The absolute system minimum in U_T of -7 pJ is at $s_1^*=229 \mu\text{m}$ with $m=0$. It is approached and reached from the left hand side because, before drying, $s_{\min} < s_1^*$. The local

minimum in U_7 at $s_3^*=399.76 \mu\text{m}$ is not reachable. For long beams such as in Fig. 2(a), the fracture equilibrium is stable due to the relatively deep well of 8 pJ at s_1^* , and is independent of beam length. Note that if $s_{\min}$ were greater than s_1^* , as large as $\sim 390 \mu\text{m}$, the same equilibrium would be achieved as the sample dries. Then, the equilibrium is reached from the right, but the same equilibrium is found because of the depth of the energy well around s_1^* .

In Fig. 2(b) the plot is repeated at $L=275 \mu\text{m}$, where important details have changed. The value of $U_7=2.2 \text{ pJ}$ at s_1^* has increased because the relative contribution of the surface energy term $\Gamma w(L-s_1^*)$ has decreased. There are again two values of s^* for which both (6a) and (6b) are satisfied, namely at $s_1^*=229$ and $s_3^*=274.80 \mu\text{m}$. The system minimum is now at s_3^* . It is not reachable from $s_{\min}$ because of the energy barrier of about 0.8 pJ at s_2^* relative to s_1^* . Therefore, the system remains in the local minimum at s_1^* with $m=0$ when drying is complete. The energy well at s_1^* is now much more shallow than in Fig. 2(a), 0.5 pJ. Yet, if $s_{\min}$ were greater than s_1^* , as large as $\sim 270 \mu\text{m}$, the same equilibrium would be achieved from the right of s_1^* .

As seen in Fig. 2(c), the local minima at s_1^* and s_2^* merge as L decreases to $242.3 \mu\text{m}$. Now there are only two values of s which satisfy (6a), namely at $s_{12}^* \approx 233 \mu\text{m}$ and $s_3^*=242.12 \mu\text{m}$. Of these, only the latter value satisfies (6b). The value of $s_{12}^* \approx 233 \mu\text{m}$ has increased slightly above the earlier value of $s_1^*=229 \mu\text{m}$ at this transition point because the contribution of surface energy continues to diminish. The system minimum at s_3^* is now reachable, but the value of m changes dramatically from nearly 0 to nearly $3/2$. The energy well at s_3^* is now exceedingly shallow, only $\sim 0.01 \text{ pJ}$ as can be seen in Fig. 2(d).

The disappearance of the stable minimum at s_1^* implies an abrupt transition in m from 0 to $3/2$ as L decreases. Physically, it can be related to the high stored strain energy in the S-shaped beam relative to the arc-shaped beam. Consider the free body diagram in Fig. 3. To maintain the S-shape, a significant moment resulting from the adhesion over the attachment length d develops at the crack tip. When d becomes too short, the beam snaps to the arc-shape because it is energetically accessible. The required moment arm reduces to a value very near 0, and d dramatically decreases from 9.3 to $0.2 \mu\text{m}$.

Fig. 3 Free body diagram for an S-shaped beam. The force and moment are provided by the support post on the left hand side. On the right hand side, the moment and tensile force develop due to adhesive forces. When the attachment length d becomes too short, the S-shape can no longer be maintained.

As L decreases further, the beam no longer comes into contact at a slope of $m=0$ for $L < L_{tip,0} = 200 \mu\text{m}$. As shown in Appendix 1, the tip is still brought into contact for $L \geq L_{tip,c} = 115.6 \mu\text{m}$. Therefore, the beam remains adhered at its tip with $m=3/2$ after the capillary dries. However, at $L = 161.9 \mu\text{m}$, the energy well at s_3^* disappears and the attachment length d vanishes. Note that the deflections are equivalent to the case of point loading for arc-shaped beams when $m \rightarrow 3/2$. Therefore, as the capillary drop at the beam tip dries for $L < 161.9 \mu\text{m}$, the beam should pop off if the adhesion is equivalent for S-shaped and arc-shaped beams.

C) Adhesion

From a fracture mechanics perspective [24], a crack driving force is derived from the strain energy release rate, G , defined as

$$G = -\frac{1}{w} \frac{dU}{ds}. \quad (9)$$

Here, the rate refers to changing values of s . The adhesion is determined when the strain energy release rate, G , equals the crack resistance or adhesion, Γ , i.e.,

$$G = \Gamma. \quad (10)$$

This is identical to the condition (6a) above. Combining (3) and (10) results in

$$G = \frac{E t^3 h^2}{2} \left\{ (3s^{-4}) \left[\frac{m^2(s)}{3} - m(s) + 1 \right] + (s^{-3}) \frac{dm(s)}{ds} \left[\frac{2m(s)}{3} - 1 \right] \right\}. \quad (11)$$

Knowing E , t , and h , G can in principle be determined for *any* adhered beam by measuring s , m and dm/ds . As seen from the discussion of equilibrium above, m can take on values only very near 0 or very near 3/2. For beams adhered in the S-shape, m is slowly varying and hence dm/ds can be taken to be 0. Regarding beams adhered in the arc-shape, it is difficult to experimentally determine a value for dm/ds . The exception is for the shortest adhered beam in the arc-shape, where again dm/ds is 0. Therefore, practically speaking, an adhesion measurement for arc-shaped beams is only possible for the shortest adhered beam. Hence, the two limiting cases of Fig. 1 give rise to the expressions

$$G = \frac{3}{2} \left(\frac{E t^3 h^2}{s^4} \right) \text{ for } m=0, \quad (12a)$$

$$\text{and } G = \frac{3}{8} \left(\frac{E t^3 h^2}{s^4} \right) \text{ for } m=3/2. \quad (12b)$$

Here, (12a) applies to any beam of sufficient length, while (12b) applies only to the shortest adhered beam.

S-shaped beams with long attachment length d are always free to approach the equilibrium adhered state; i.e., subsequent to a perturbation on the system which forces s from its equilibrium value, s_1^* can be reached whether $s > s_1^*$, or $s < s_1^*$. In fact, the situation for large d is very similar to Obreimhof's early fracture mechanics experiment [25] in which the fracture energy of mica was determined from interferometric measurements. Note that the situation for (12b), although most commonly used to report adhesion measurements in micromachining [9, 10, 15-17, 26] or to compare calculations to data [27, 28], is precarious at best. This is because the depth of the energy well, already exceedingly small for the arc-shaped beams, approaches

zero for the shortest adhered beam. In other words, the equilibrium for small d is not always accessible; i.e., if s grows to be larger than s_3^* , the beam will pop off and an adhesive equilibrium can no longer be found. We now proceed to the measurement of deformations and equilibrium adhered lengths for a range of cantilever beam geometries.

III. Experimental Methods and Results

A) Specimen Fabrication and Surface Treatment

Our micromachined structures were fabricated with one photolithographic mask level. First, an $h=1.8\text{ }\mu\text{m}$ oxide was deposited on single crystal $\langle 100 \rangle$ oriented silicon. A $t=2.3\text{ }\mu\text{m}$ layer of polysilicon (as measured by a WYKO multi-wavelength interferometer) was deposited at $600\text{ }^\circ\text{C}$ and then annealed at $1100\text{ }^\circ\text{C}$ to relieve residual stresses. Cantilever beams of $20\text{ }\mu\text{m}$ width were defined in an array of increasing L with increments of $2\text{ }\mu\text{m}$ from 10 to $100\text{ }\mu\text{m}$, and with increments of $5\text{ }\mu\text{m}$ from 100 to $500\text{ }\mu\text{m}$. The beam support was defined by making the polysilicon wider in this area. The samples were placed in a controlled time HF acid etch such that the oxide under the beams was removed, but remained under the support posts. The samples were next transferred to deionized water, immersed in hydrogen peroxide to form a thin silica layer, and then transferred back to deionized water.

One set of samples was removed from the water and dried in air for two days or more. We shall refer to these as the hydrophilic samples. The other set was treated with a molecular coating of ODTS (octadecyltrichlorosilane, $\text{CH}_3(\text{CH}_2)_{17}\text{SiCl}_3$). We shall refer to these as the hydrophobic samples. ODTS has been investigated as a promising molecule for minimization of adhesion in MEMS [8, 22, 29, 30]. The head group of the organosilane is hydrolyzable, which facilitates the formation of covalent siloxane bonding to the substrate hydroxyl groups as well as between neighboring silane molecules to form a well attached organosilane layer [31]. Meanwhile, the CH_3 tail groups exposed to the surface exhibit low surface energy ($\sim 20\text{ mJ/m}^2$), and are hydrophobic (contact angle $\sim 110^\circ$). Following the recipe prescribed in ref. [22], the specimens were transferred from water to isopropanol to isooctane, a series of solvents for which

the next is miscible with the previous. The specimens were next submersed in a 1mM ODTs solution of 4:1 hexadecane:chloroform for 30 minutes, during which the ODTs molecules deposit on the polysilicon surfaces. The specimens were then transferred back to the solvents in reverse order, and finally removed from water and air dried.

B) Measurement Procedures

To make quantitative measurements of beam deformations and adhered length, we equipped our Leitz Orthoplan optical microscope with a Michelson interferometric attachment and green light monochromator (547 nm as characterized by spectrum photometry). To ensure minimal error due to tilt, background fringes were aligned parallel with the length of the beams using the tilt adjustment of the reference surface. Interference fringe intensity was recorded with a CCD camera and subsequently analyzed using a standard image processing program [32]. Linescan intensities along the length of adhered beams were converted into u -deflection vs. x -position data using a computer program. An absolute deflection accuracy of about 50 nm (across the entire beam), and a relative accuracy (pixels near each other) of about 10 nm resulted. The spatial resolution of our 20X objective was about 1 micron. Measurements of beam height versus distance along the beam were used to assess the adhered beam length and to provide a direct comparison with beam deflections predicted from elasticity theory.

C) Experimental Results – Hydrophilic Beams

Measured deformations and adhered length for a hydrophilic beam of length $L=365\text{ }\mu\text{m}$ are presented in Fig. 4. Fig. 4(a) is an optical interferometric image showing three such adhered beams. Intensity versus position data from a linescan taken along the beam designated by a white line in Fig. 4(a) are plotted in the graph of Fig. 4(b) (right hand y-axis). The beam comes into contact with the substrate at the point where the linescan flattens out, at $x=172.4\text{ }\mu\text{m}$, ($u=1820\text{ nm}$). From the linescan data, u deformations were computed and are plotted as a solid line referenced to the left-hand axis of Fig. 4(b). For comparison, predicted deformations for the extremes of $m=0$ and $m=3/2$, using Eq. (1), are also plotted using dashed lines. It is seen that

Fig. 4 (a) Interferogram of several long beams subjected to hydrophilic treatment. (b) Linescan across the beam adjacent to the white line of (a), and conversion to $u(x)$ deflections, which compare well with the deflections from M&H for S-shaped beams.

the actual deformations agree well with those predicted for the case $m=0$, consistent with the S-shaped deformation for beams adhered over a significant fraction of their length. Using measured values of $s_1^*=172.4 \mu\text{m}$, $h=1800 \text{ nm}$, $t=2.3 \mu\text{m}$ and assuming $E=170 \text{ GPa}$ [33] an adhesion value of $\Gamma=11.3 \text{ mJ/m}^2$ was calculated using Eq. (12a).

Adhesion measurements on various length beams were made to determine the range over which deformations matched the case $m=0$. For sufficiently long beams, the agreement was excellent. However, as the beam length approached the value of $175 \mu\text{m}$, a transition to the $m=3/2$ deformation was observed. For beams shorter than $175 \mu\text{m}$, the value of m toggled between 0 and $3/2$. An example of this behavior is seen in Fig. 5. In the interferometric image of Fig. 5(a), the $140 \mu\text{m}$ long beam designated by the white line has fringes out to its tip, indicating an arc-shaped beam geometry. Fig. 5(b) shows good agreement with the $m=3/2$

Fig. 5 (a) Interferogram of shorter beams subjected to hydrophilic treatment. (b) Deflections of beam adjacent to the white line compare well with the M&H calculations for arc-shaped beams. The beam below remains adhered in the S-shape.

deformation condition. The 145 μm beam, just below the designated beam, does not have fringes out to its tip. The measured deformation on the 145 μm beam agrees well with the $m=0$ beam deformation. All beams of less than 145 μm length remain adhered with an arc-shape.

The shortest value of L for which beams remained adhered was 58 μm . If (12b) is used to quantify Γ for this case, the result is that $\Gamma=222 \text{ mJ/m}^2$. However, as seen in Fig. 6 (for the

Fig. 6 (a) Interferogram of a short beam ($L=68 \mu\text{m}$) subjected to hydrophilic treatment. (b) Deflections which compare poorly with the M&H calculations for arc-shape beams due to significant support post compliance.

case of a 68 μm long beam), the measured deformations lie somewhere between that predicted by the $m=0$ and $m=3/2$ limiting conditions. This change in deformation behavior is due to the compliance of the step-up post, which is expected to be non-negligible for beams below 100 μm in this geometry [34]. Compliance in the step-up post reduces the strain energy that is stored in the beam and hence the calculation for adhesion according to Eq. (12b) requires a correction. Although a much smaller effect, the step-up post compliance is also non-zero for case of S-shaped beams, Fig. 4. In Appendix 2, we use the measured slope at the beginning of the beam and match the actual beam deformations to account for the step up post compliance in both the S-shape and arc-shape cases. The correction decreases Γ from 11.3 to 9.4 mJ/m^2 for the S-shaped beams, and from 222 to 91 mJ/m^2 for the arc-shape beams. Note that adhesion Γ remains a factor of ten different even after this correction!

A summary of results for untreated beams is plotted versus beam length in Fig. 7(a). The left hand axis displays values of Γ while the measured slope parameter is referenced to the right hand axis. For long beams in the $m=0$ condition, the variation in Γ reflects beam-to-beam differences in local adhesion. The average and standard deviation for measured adhesion energies for S-shaped beams are reported in Table I (row 1).

Fig. 7 (a) Adhesion Γ and slope factor m vs. beam length L for hydrophilic treatment. The value of the shortest arc-shaped beam is approximately ten times higher than the S-shaped beams. (b) Hydrophobic treatment. The value of the shortest arc-shaped beam is approximately 3 times higher than the S-shaped beams.

Table I - Γ for S-shaped beams ($m=0$)

Treatment	Γ (mJ/m ²)	σ	σ/Γ
Hydrophilic	16.5	8.2	0.5
Hydrophobic	3.4	0.5	0.16

Hydrophobic Beams

Even though our ODTS coated beams had a measured contact angle of about 105°, capillary action from drying was still able to pull beams into contact with the substrate. We are certain attachment occurred during drying because beams were observed to be free under an optical microscope just before removing from water. We believe that this result is explained by the observation that *receding* contacting angles are often observed to be smaller than advancing contact angles, a phenomenon known as contact angle hysteresis [35]. Further confirmation of this stems from an experiment in which we performed video microscopy of drying of previously adhered ODTS beams. This in-situ drying experiment was conducted without interferometry in order to allow sufficient free working distance between the water and the microscope objective. Out-of plane deflections could still be observed because green light is weakly transmitting in polysilicon, giving rise to a weak but observable contrast. Although a drying front of water moving across the surface was observed in one video frame and had disappeared in the next (30 msec later) due to its rapid velocity over the hydrophobic surface, water remained microscopically in the vicinity of the beams for a full second, and clearly pulled the beams further in. ODTS density on the substrate surface is known to be a strong function of deposition conditions [36]. We hypothesize that because the ODTS film did not attain maximum density in our deposition, the adhesion-controlling receding contact angle θ_{cr} was less than 90°.

For long ODTS treated beams, we observed again excellent agreement with deformations predicted for the $m=0$ condition yielding an s_1^* of 225 μm . This adhesion length corresponds to an adhesion energy of $\Gamma=3.9$ mJ/m² using Eq. (12a). By making a correction as reported in Table AII, the value is reduced to 3.6 mJ/m². The transition from $m=0$ to $m=3/2$ was abrupt at

$L=250$ to $245\text{ }\mu\text{m}$; no toggling between the values of m was apparent. The shortest adhered beam was at $L=120\text{ }\mu\text{m}$. Compared to the hydrophilic case, the measured deformation conformed well with the arc shape, indicating that compliance in the step-up post is small. Using Eq. (12b) results in an adhesion value of $\Gamma=12.1\text{ mJ/m}^2$. The correction as reported in Table AII now reduces Γ only to 9.5 mJ/m^2 . Note that this value remains approximately three times larger than that obtained with beams adhered in the S-shaped geometry. The results are summarized in Fig. 7(b), and the average and standard deviation S-shaped beams is tabulated in Table I. Note that the average value of Γ is approximately five times lower for the hydrophobic than the hydrophilic beams.

IV. Discussion

In this section we use our experimental results to address each of the questions that we posed in section I.

1) Are the predicted beam deflections valid for micromachined beams?

Using interferometry, we have carefully compared actual beam deformations against those assumed in the model of M&H. For adhered beams which come into contact with the substrate beyond $120\text{ }\mu\text{m}$ from the support post, we find that the deformations match those of M&H quite well. However, we find that the arc-shaped beams with hydrophilic surfaces yield deformations that are considerably different than predicted by the simple model. The explanation is that the deformation model assumes a rigid support post, while the actual micromachined beams have more complex and compliant support post geometries. In our processing approach, undercutting of the rigid support post occurs during the release etch and contributes additional compliance to the beam structure. In Appendix 2, we showed how the effect of compliance could be evaluated. Other fabrication approaches for support posts, such as that used by M&H, avoid the undercutting problem but also involve inherently compliant support post structures.

Our results show that support post compliance errors increase as the actual adhesion energy increases. This simply reflects the increased torque generated by a larger adhesive force at the adhered end of the beam. Although support post compliance is most apparent for the shortest adhered beams, close examination of Figs. 4(b) and 5(b) suggests that some support-post compliance may be responsible for the small discrepancy between the measured and predicted deformations in the S-shaped configuration. M&H suggest that the effect of support-post compliance on the measured adhesion energy can be removed by plotting the detachment length as a function of beam dimensions. In order to implement this approach one must independently vary the support post height and beam thickness. However, the processing modifications needed to independently vary the beam dimensions are difficult to achieve and may themselves lead to intrinsic changes in the curvature of the polysilicon or surface roughness that can also influence the measured adhesion energy. For this reason it is best to directly confirm the nature of deflections of the adhered beams and make corrections which account for the step up post compliance. We have carried out this procedure as outlined in Appendix 2.

2) What is the behavior of beams in the transition from the S-shape to the arc-shape? How are the equilibria for the S-shaped and arc-shaped beams attained?

The equilibrium mechanics in section IIB suggests that there should be an abrupt transition length L_{Tr} at which the transition from S-shaped to arc-shaped beams occurs. The values of L_{Tr} can be determined quantitatively by carrying out the exercise in section IIB, based on knowing the value of Γ for S-shaped beams. For the hydrophilic case, we calculate $L_{Tr}=174.5 \mu\text{m}$, while as shown in section IIB, $L_{Tr}=242.3 \mu\text{m}$ for the hydrophobic case. Fig. 7 confirms that this behavior is observed experimentally, and is in excellent quantitative agreement with the calculations for L_{Tr} . For the hydrophilic films, the transition length occurs from $180 \mu\text{m}$ to $140 \mu\text{m}$, while for the hydrophobic beams the transition occurs from 250 to $245 \mu\text{m}$. The larger range of the transition observed experimentally in the hydrophilic compared to the hydrophobic case is due to larger local differences in adhesion. Note that the ratio of standard deviation to average adhesion for S-shaped beams, σ/Γ , is 0.45 for the hydrophilic beams

versus 0.16 for the hydrophobic beams. Because the ratio approaches one-half, it is not surprising to observe that the transition length varies in the hydrophilic case. Hence, a non-abrupt value of L_{Tr} indicates that adhesion is not well controlled locally.

How are the equilibria for the S-shaped and arc-shaped beams attained? For the hydrophilic case, the shortest beam attached is 58 μm . According to the calculations outlined in Appendix 2, this beam is effectively 73 μm long when the step-up post compliance is considered. Therefore, from Table AI, the receding contact angle θ_{cr} of the drying water must be near 0° in the hydrophilic case, because then $L_{tip,c} \sim 75 \mu\text{m}$. Furthermore, from Table AI, for $\theta_{cr}=0^\circ$, $L_{tip,0} \sim 130 \mu\text{m}$. Recall that $L_{Tr}=174.5 \mu\text{m}$ for this case. We hypothesize the following occurs during the drying process: Long beams with $L > L_{Tr}$ are pulled in to a value $s_{\min} \sim L_{tip,0}=130 \mu\text{m}$ when the capillary exerts its maximum force. Because adhesion energy $\Gamma=10 \text{ mJ/m}^2$ is much smaller than $2\gamma \cos(\theta_{cr})=146 \text{ mJ/m}^2$, surface energy exerts only a weak subsequent effect and therefore $s_{\min}$ is only slightly less than $L_{tip,0}$. However, because $s_1^*=172.4 \mu\text{m}$, the equilibrium is approached from the left as the liquid dries (i.e., $s_{\min} < s_1^*$). Intermediate length beams with $L_{Tr} > L > L_{tip,0}$ are also pulled into the S-shape by capillary action. The beam gradually reverts to the arc-shape as the capillary dries, because capillary forces remaining over part of the beam will keep it in contact with the substrate. Finally, for short beams with $L < L_{tip,0}$, the beam never attains the S-shape. These remain adhered at their tip, however, as drying progresses. In effect, any beam which contacts the substrate at its tip remains in contact. This is equivalent to the assertion by Abe and Reed [13]. To confirm this hypothesis, in-situ drying experiments under interferometric conditions are required.

For the hydrophobic case, θ_{cr} is at most 82° from Table AI, such that beams of length $L=120 \mu\text{m}$ are pulled into contact with the substrate. Therefore, $L_{tip,0} \sim 210 \mu\text{m}$. Surface energy due to capillary force is now $2\gamma \cos(\theta_{cr})=20 \text{ mJ/m}^2$. This remains larger than surface energy $\Gamma=3 \text{ mJ/m}^2$, $s_{\min}$ is again only slightly less than $L_{tip,0}$. The drying sequence is qualitatively similar to the hydrophilic case for the long, medium and short length beams.

3) Are the values for adhesion between S-shaped and arc-shaped beams equivalent? If not, what factors influence measured differences?

Using corrections as outlined in Appendix 2, we were able to directly compare the adhesion energy as determined by the conventional shortest attached beam method and by our adhered length approach. We found a significant discrepancy between the results produced by the two methods, factors of 11 and 3 for the hydrophilic and hydrophobic cases respectively. Therefore, the values measured by the two methods *are not equivalent!* Before we address differences between measurement approaches, it is important to understand the absolute values obtained. For this purpose, we consider the hydrophilic samples that were treated only with hydrogen peroxide before drying from DI water. This treatment results in beam and substrate surfaces that are covered by a thin layer of hydrophilic SiO₂. In a previous report [37], we showed that the adhesion energy for silica covered surfaces is controlled by the inherent roughness of the polysilicon. For perfectly smooth, wetted surfaces one predicts and measures [38] an adhesion energy that is twice the surface energy of water ($\gamma_{\text{water}} = 73 \text{ mJ/m}^2$), or about 146 mJ/m^2 . In the case of rough surfaces, the apparent adhesion can be more than an order of magnitude lower due to the limited area of actual contact between surface asperities, and the adhesion increases exponentially with relative humidity [37]. This helps us to understand why Γ is much lower than $2\gamma_{\text{water}}$ for the hydrophilic beams. Of course, at saturation humidity conditions liquid is expected to fill the entire region surround individual asperities and lead to adhesion values comparable with smooth surface conditions.

Because surface roughness can play a significant factor in the apparent adhesion energy, one might ask whether the differences observed between measurement techniques may also be due to factors associated with surface roughness. The fact that the actual surface roughness was the same for all beams argues against this notion. However, the contact geometry is quite different for the S-shaped and arc-shaped beams. S-shaped beams make contact with the substrate over a length that is large compared with the scale of the surface roughness. Such a geometry closely approximates the parallel contact of extended surfaces that has been the topic

Fig. 8 Atomic force microscope linescan of the bottom side of a polysilicon cantilever beam.

of previous studies of rough surface contact [39]. When the area of contact is much greater than the scale of roughness, it is appropriate to use a statistically averaged measure of surface topograph. Alternatively, arc-shape beams make contact only over microscopic lengths. As seen in Fig. 2(d), a beam which has just made the transition from the S-shaped to the arc-shape is adhered over ~ 200 nm. However, the shortest arc-shaped beams

will be adhered only over a few tens of nanometers according to the M&H calculations. The atomic force microscope linescan in Fig. 8 shows that the period of major asperity peaks on these surfaces (~ 1 μm) is greater than the predicted length of contact at the tip of the beam. In this situation it is clear that statistical models for true contact area will not be reliable. Given this large difference in contact geometries for the two measurement approaches, it is not hard to believe that the actual contact in the vicinity of the crack tip will also be significantly different for each beam. This actual contact area cannot be explored through direct measurement techniques. It will be interesting in the future to apply numerical techniques such as those developed by Tian and Bhushan [40, 41] to model the actual area of contact for the two contact geometries. Perhaps such estimates could help quantify the effect of roughness on areal contact.

A second effect that may also contribute to the observed difference between apparent adhesion values is related to the drying process itself. As water evaporates from the contact region of arc-shaped beams, impurities will necessarily concentrate in the capillary drop at the tip of the beam. In the limit as the capillary volume vanishes, impurities or solubility products may actually precipitate and lead to the possible formation of a porous solid network in the vicinity of the tip of the beam. Previous fracture mechanics measurements [42] have shown that

precipitation of soluble silicates can support stress across solid silicate interfaces. In this scenario, the effective contact area can become much larger than the calculated value leading to an overestimate of the surface energy. Again, the extremely small contact region in the arc-shaped beam makes this measurement approach very sensitive to the nature and size of the contact region. Alternatively, S-shaped beams with their extended adhesive interface are expected to be much less sensitive to such effects.

While the S-shaped beam gives a more reliable value of adhesion than the arc-shaped beam, the latter is of great practical significance. This is because the shortest beam to adhere will always limit the use of MEMS devices. Two-dimensional meniscus effects on the sticking of arc-shaped beams were discussed in refs. [13] and [14]. Narrow-width arc-shaped beams are less likely to stick than wide arc-shaped beams possibly due to meniscus effects [43]. More work will be necessary to elucidate the full three dimensional nature of capillary drying and its interplay with adhesion.

V. Summary and Conclusions

We have explored in detail the deformations and adhesive equilibria of micromachined cantilever beams. Our summary and conclusions are as follows:

- (1) The deformations for adhered beams were measured by interferometry for arc-shaped and S-shaped beams, and found to agree to first order with the elasticity calculations of M&H. This verifies that for long beams ($>120\text{ }\mu\text{m}$ for our geometry), the strain energies calculated by M&H are appropriate in making adhesion calculations. Discrepancies were significant for adhered beam lengths less than $120\text{ }\mu\text{m}$, and could be explained and modeled by non-zero support post compliance.
- (2) An abrupt transition from an S-shaped to an arc-shaped beam as L decreases was predicted theoretically, and observed experimentally. This is explained by the disappearance of a local minimum at s_1^* of total system energy U_T as L is decreased. Excellent agreement between calculations and experiment for the transition length L_T .

was obtained by using the value for adhesion Γ for S-shaped beams. This suggests that the value of Γ for S-shaped beams controls the transition length, rather than the value of Γ for arc-shaped beams.

- (3) Adhesion is best measured on S-shaped cantilever beams which impose a deep energy well and a good fracture mechanics equilibrium. When compared to the method of determining the shortest adhered beam in an array of beams with a high power objective, measurement of a single S-shaped beam by interferometry permits a much smaller area to be occupied to obtain adhesion values. This is valuable because of the expense of real estate area in a MEMS device. S-shaped beams also provide much higher resolution on adhesion than the method of determining the shortest adhered beam. Adhesion statistics are obtained in a straightforward manner by measuring several S-shaped beams in close proximity.
- (4) The apparent adhesion calculated for the shortest arc-shaped beam can be different from the adhesion for S-shaped beams. The probable reason is that the attachment length d for arc-shaped beams, calculated from beam theory but impossible to confirm experimentally, is incorrect. Due to statistical variations in roughness, the contact area sampled by the crack tip of the arc-shaped beam may be smaller or larger than that sampled by the crack tip of the S-shaped beam. If the beam is dried from a liquid environment in which capillary action has brought it in contact with the substrate, the effective contact area is very likely larger than the calculated value, giving rise to an anomalously high value of adhesion.

We have demonstrated that the S-shaped cantilever beam configuration should be adopted for detailed studies of adhesion forces in surface micromachining. This method gives great latitude in measuring auto-adhesion because the equilibrium is deep and easily attainable from either side of the equilibrium. Studies such as the effect of environment on auto-adhesion are readily adapted to this method [37]. With interferometry, the crack length s is well resolved,

and therefore adhesion is accurately assessed. While surface micromachined structures were used as test samples here, S-shaped cantilever beams may be used to appropriately measure adhesion at smaller and larger scales.

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VI. Appendix

Appendix 1 Determination of characteristic beam lengths $L_{tip,0}$ and $L_{tip,c}$

Capillary action exerts negative pressure on a beam as seen in Fig. A1. A complete understanding of the drying problem requires a three dimensional analysis [13] and is beyond the scope of this work. However, a reasonable analysis for characteristic beam lengths for the case $w \gg h$ from which we can gain some insight into the strength of the capillary liquid is provided in this Appendix.

Fig. A1 Forces on the beam during the liquid drying process

For a given capillary force, we wish to determine the length of the shortest beam, $L_{tip,0}$, for which the beam tip makes a shear angle $\theta=0$ at some point during the drying process. For any beam of length greater than $L_{tip,0}$, surface energy will cause the crack length to become shorter than $L_{tip,0}$ during the drying process. Beams shorter than $L_{tip,0}$ cannot reach the S-shape configuration. We need to solve for the beam deflection when the capillary extends from the support post to the tip of the beam. In this situation, the beam is uniformly loaded by a force q , as in Fig. A1. A point reaction P at the beam tip opposes this force, Fig. A1.

From beam theory, the beam is in contact with the substrate if

$$w(L) = \frac{qL^4}{8EI} - \frac{PL^3}{3EI} = h \quad (A1)$$

and is at an angle $\theta=0$ at its tip if

$$\frac{qL^3}{6EI} - \frac{PL^2}{2EI} = 0 \quad (A2)$$

From (A2), $P = qL/3$, and from (A1), $q = 72hEI/L^4$. Therefore, the shortest beam which will be brought into contact to the substrate with an angle $\theta=0$ at its tip is at

$$L_{tip,0} = \left(\frac{72EIh}{q} \right)^{1/4}. \quad (A3)$$

Likewise, the shortest beam which will be brought into contact at its tip with an angle $\theta>0$ is at $L_{tip,c}$, where

$$L_{tip,c} = \left(\frac{8EIh}{q} \right)^{1/4} \quad (A4)$$

The uniform loading q is calculated from the Laplace pressure $q/w = \gamma/r$, where γ is the surface tension of the liquid, and r is the radius of the drop in Fig. A1. It is a simple geometrical exercise to show that $r = (h/2\cos\theta_{cr})$, where θ_{cr} is the receding contact angle of water with the solid. Therefore,

$$q/w = 2\gamma \cos\theta_{cr}/h. \quad (A5)$$

Finally,

$$L_{tip,0} = \sqrt{3} L_{tip,c} = \left(\frac{E t^3 h^2}{3 \gamma \cos\theta_{cr}} \right)^{1/4} \quad (A6)$$

In Table AI, we give some calculated lengths for $L_{tip,0}$ and $L_{tip,c}$. The values $E=170$ GPa, $\gamma=\gamma_{\text{water}}=73$ mN/m, $w=20$ μm , $h=1.8$ μm , and $t=2.3$ μm are assumed.

The analysis here provides an upper bound for $L_{tip,0}$ and $L_{tip,c}$. In reality, factors such as support post compliance and edge effects (such as the "inside meniscus" [13]) will reduce these values somewhat. Considering that in our experiments $w=11h$, the effect of the "inside meniscus" will be small.

Table AI Values for $L_{tip,c}$ and $L_{tip,0}$ versus θ_{cr}

θ_{cr}	$L_{tip,c}$ (μm)	$L_{tip,0}$ (μm)
0	74.6	129.3
30	77.4	134.0
60	88.8	153.7
80	115.6	200.3
85	137.4	237.9
89	205.3	355.7
89.9	365.1	632.4
89.99	649.3	1124.6

Appendix 2 Correcting the values of Γ for support post compliance.

Knowing the deflections for the beams, we can take into account the compliance of the step-up support post to improve the values of Γ . This is done by relaxing the constraint that the constants of integration used to derive the deflection curve Eq. (1) be zero. In these calculations, the 15 μm length of the support post was taken into account. The fact that this region was approximately twice as wide as the beam was also considered. The non-zero slope of the beam from the edge of the support post was set to the experimental value. Assigning the proper non-zero constants of integration, we matched the experimental deflections to calculated deflections. From this, the strain energy and hence the corrected values of Γ were determined. The results are given in Table AII. As expected, the values of Γ corrected for the support post compliance are smaller than the uncorrected values. For the arc-shaped beam with the hydrophilic treatment, the correction is approximately a factor of two. However, for the other cases, the correction is 30 per cent or less. This is to be expected considering that only the former deviates significantly from the ideal deflections.

Table AII - Corrected values of Γ

Treatment	Beam shape	s (μm)	uncorrected	corrected
			Γ (mJ/m^2)	Γ (mJ/m^2)
Hydrophilic	arc	58	222	91
	S	172	11.3	9.4
Hydrophobic	arc	120	12.1	9.5
	S	225	3.9	3.6

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2. Adhesion of polysilicon microbeams in controlled humidity ambients (de Boer and Michalske)

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Abstract

We characterize *in-situ* the adhesion of surface micromachined polysilicon beams subject to controlled humidity ambients. Beams were freed by supercritical CO₂ drying. Consistent adhesion results were obtained using a post-treatment in an oxygen plasma which rendered the microbeams uniformly hydrophilic. Individual beam deformations were measured by optical interferometry after equilibration at a given relative humidity (RH). Validation of each adhesion measurement was accomplished by comparing the deformations with elasticity theory. The data indicates that adhesion increases exponentially with RH from 30% to 95%, with values from 1 mJ/m² to 50 mJ/m². Using the Kelvin equation, we show that the data should be independent of RH if a smooth interface is considered. By modeling a rough interface consistent with atomic force microscopy (AFM) data, the exponential trend is satisfactorily explained.

1. Introduction

Adhesion (e.g., stiction) is a major concern in Microelectromechanical Systems (MEMS) reliability. MEMS devices are inherently sensitive to adhesion because the polycrystalline (poly) structural members are relatively compliant and only a few microns above the substrate. If a compliant member is brought into contact with the substrate by capillary, electrostatic or inertial forces, surface forces may cause the member to remain adhered after the external force is removed. The problem can occur during the final step of the manufacturing process. The devices, encased in glass, are "released" in HF acid, transferred to water, and then dried. Capillary forces are negative due to the surface tension of water on the wetted poly surface, and cause the beams to be pulled in to the substrate. Adhesion on the order of 10-100 mJ/m² is measured [1-5].

Many methods have been investigated to overcome this initial problem [6-14] resulting in initially free beams. Supercritical CO₂ (SCCO₂) drying has been shown to free long cantilever beams [14]. Devices are transferred from water to methanol, and placed in a pressure vessel. In

our laboratory, we pressurize with gaseous CO_2 in the vessel until the supercritical state is attained, and then displace the methanol [15] with SCCO_2 . Supercritical CO_2 is subsequently vented, leaving beams up to 2 mm in length free.

While initially free devices greatly improve yield, structural members may still come into contact while in use due to electrostatic or inertial forces. Their adhesion then may depend greatly on the environment they are subject to. A key concern is the dependence of adhesion on relative humidity (RH), because water may eventually penetrate even hermetically sealed devices. If the operating temperature of the device drops, RH levels can increase towards 100% because of the exponential dependence of RH on water content. After supercritical drying, adhesion of about $10 \mu\text{J}/\text{m}^2$ and $140 \text{ mJ}/\text{m}^2$ has been measured using an activation pad near the support post at an RH of about 30% and 100% respectively [16]. However, these were not equilibrium values. It is important to measure the equilibrium performance of MEMS devices subject to various humidity ambients so that worst case behavior is known for design considerations.

II. Experimental

Test devices are polysilicon beams with an activation pad located near the support post, similar to ref. [16]. A cross-section of a beam is represented in Fig. 1. In this configuration, the beams and the landing are electrically connected at the end of the beam array (not shown) such that any interaction between the beam and the landing pad is adhesive only. We take a fracture mechanics perspective [4, 5, 17] in which s is the crack length, and the attached length d must be of substantial length, $\sim 0.05 L$, where L is the beam length. The height of the support post is h and the beam thickness is t . The Young's Modulus E of the beams is assumed to be 170 GPa.

Fig. 1 Cross-section of beam geometry

The devices were fabricated at Sandia National Laboratories in the SUMMiT process [18]. The landing pad has a thickness of $0.3 \mu\text{m}$. Its roughness after fabrication was measured by AFM to be 2.1 nm (rms) . A sacrificial layer of thickness $h=1.8 \mu\text{m}$ was deposited next.

The poly beam is a thickness t of $2.25 \mu\text{m}$, as measured by optical interferometry on a WYKO multiple wavelength device. The roughness on the bottom of the poly 2 was 2.4 nm (rms) . The

devices are subjected to a 1100 °C anneal to minimize residual stress as well as strain gradient. The devices are released in 1:1 HF:HCl for 90 minutes, transferred to water and then to hydrogen peroxide to obtain a thin (~1 nm) layer of SiO₂ on all poly surfaces. The devices were subsequently transferred to water, methanol and then supercritically dried.

Fig. 2 Environmental Interferometric Microprober

In order to measure adhesion *in-situ*, we have constructed an Environmental Interferometric Microprobing Station, as represented in Fig. 2. Humidified nitrogen, introduced by bubbling through water, is mixed with dry nitrogen to control humidity. The humidity level is monitored by a Vaisala HMP234 humidity probe. The chamber is placed immediately under an optical microscope objective to which an

interferometric attachment has been fixed. Tilt on the attachment is adjusted such that fringes on the substrate are parallel to the beams. In this fashion, fringes on the beams indicate out-of-plane (z) deflections. A latex seal between the chamber and the interferometric attachment isolates the chamber from the outside world. Image information is acquired by means of a CCD camera on the microscope. Image capture and RH monitoring is controlled by LabView [19]. By taking a linescan along a given beam using NIH Image [20], fringe information is digitized, and converted to z deflections by a computer program.

Adhesion is measured on each individual beam according to the following procedure. First, the beam must be adhered over a long length d , as seen in Fig. 1. Second, the deflections are normalized and compared to the equation [1]

$$z(x) = -h(3\eta^2 - \eta^3), \quad (1)$$

where $\eta = x/s$. Third, if agreement with Eq. (1) is good, then adhesion Γ (in J/m²) is calculated according to

$$\Gamma = \frac{3}{2} E \frac{h^2 t^3}{s^4}. \quad (2)$$

We have found this methodology to be an improved method of adhesion measurement relative to measuring the shortest adhered beam in an array of beams [4, 5]. The reasons are (1) the deflection measurement validates the value for each beam, (2) the incremental area is well known, (3) the energy well is very deep, and (4) each beam gives a value for adhesion. The radius of curvature of free beams, as measured by interferometry, was 2.2 m, corresponding to 230 nm out of plane deflection for a 1000 μm long beam. Considering that these beams are nearly flat, curvature has a negligible effect on strain energy, and is ignored in the adhesion calculations.

III. Results and discussion

Adhesion results were erratic when samples were tested as-received from the supercritical drying apparatus. That is, pull-in behavior of the beams versus RH was different for each beam, even after a few days exposure time. We noticed that while test samples were fully wetted by water before entering the apparatus (i.e., a contact angle with water of $\sim 10^\circ$), the test samples had much higher contact angles ($40\text{--}70^\circ$) after the supercritical drying procedure. Apparently the apparatus deposits a thin contamination film which increases the contact angle. While in general the contamination reduces adhesion at high RH, the erratic results indicate that the process is not controllable. The source of the contamination has been traced to the input pump, and cannot immediately be fixed. In order to alleviate this problem, samples were placed in an oxygen asher after the supercritical drying. Beams remained free, and on test samples the contact angle was again reduced to $\sim 10^\circ$. We conclude that the contamination layer is organic and removable by oxygen plasma, apparently leaving a well hydroxylated surface which adsorbs water [21].

The beams were first placed in a 0% RH ambient for at least 16 hours to minimize water adsorbed from the ambient. Next, the beams are pulled into contact with the substrate via the activation pad. Because the beams are at least 1000 μm long, the beams remain in contact with the substrate over a long length d , and crack lengths are on the order of 600–850 μm , corresponding to $\Gamma=68\text{--}17\text{ }\mu\text{J}/\text{m}^2$ respectively. Next the RH was increased to 30%, and crack length s began to decrease due to capillary action at the crack tip. After 40 hours, no further decrease was observed. RH was then increased to 45, 55, 70 and 95%. At each RH, equilibrium was attained for s within 24–40 hours before increasing to the next RH.

With the oxygen ashing after supercritical drying, the results were now much more consistent, as evidenced in Fig. 3. Here a uniform decrease in s was observed as RH was increased. At RH=30% after 40 hours as in Fig. 3a, the equilibrium length for most beams is about 350 μm . At this RH, one beam continues to have very long lengths s . We take this to be due to a local barrier preventing pull-in, perhaps due to local roughness being larger. However, at RH=45%, all beams displayed nearly the same length, indicating that such a barrier is small and is overwhelmed by the capillary forces as RH increases.

Fig. 3 Interferograms of crack length s vs. RH after 40 hours exposure.

An example of validation of the adhesion measurement is given in Fig. 4. Here, the linescan from an individual beam is plotted vs. position on the right hand ordinate for an individual beam from Fig. 3a. This data is converted to z deflection vs. position data by a computer program, which is plotted on the left hand ordinate as a solid line. The dashed line represents Eq. (1). Because the data and the dashed line virtually overlap, the calculation of adhesion according to Eq. (2) is valid. In this case, the value of Γ is 0.5 mJ/m². This validation and measurement procedure was repeated for each of five beams at the five different RH values ranging from 30 to 95%, and is plotted in Fig. 5 (as indicated by "Data"), where each line represents an individual beam at different RH values. We see that the dependence of adhesion on RH is exponential.

Fig. 4 z -deflections of an individual beam

Fig. 5 Data and calculations

The Kelvin equation predicts the radius of a capillary in a narrow channel as a function of RH. We investigated two possibilities for how the Kelvin equation might explain our results. In the first model, we assume that the interface between the beams is smooth, such that a microcapillary extends across the width of the beam at the crack tip and exerts a tensile pressure according to the Laplace equation. This is schematically represented in Fig. 6. The radius of the capillary and the tensile force it exerts are then [22]

$$r_K = \frac{1.04}{\ln(RH)} \text{ nm} \quad (3) \quad \text{and} \quad q = \frac{2\gamma \cos \theta}{r_K} = \frac{0.142}{r_K} \frac{\text{N}}{\text{m}^2} \quad (4).$$

Here, r_K is the Kelvin radius in nm, $\gamma=71 \text{ mJ/m}^2$ is the surface tension of water and $\theta=0^\circ$ for this hydrophilic sample is the contact angle of water with the surface. In Eq. (3), room temperature and atmospheric pressure are assumed. Using beam theory, we can calculate the deflections of a beam subject to the boundary conditions of 0 slope at the two ends, and a distributed force per unit area q exerted at one end. Defining the length from the support post to the beginning of the capillary drop as $s_1 = s - s_2$, the deflection for $0 \leq x \leq s_1$ is

$$z(x) = -\frac{1}{D} \left\{ P \left(\frac{(s_1 + s_2)x^2}{2} - \frac{x^3}{6} \right) - qs_2 \left[\left(s_1 + \frac{s_2}{2} \right) \frac{x^2}{2} - \frac{x^3}{6} \right] \right\} \quad (5a)$$

The stiffness modulus D is $Et^3/12$, and s_2 , P and q are as in Fig. 6. P is a reaction force per unit width which develops in response to the capillary pressure at the crack tip. For $s_1 \leq x \leq s$,

$$z(x) = -\frac{1}{D} \left\{ P \left(\frac{(s_1 + s_2)x^2}{2} - \frac{x^3}{6} \right) - \frac{q}{2} \left[\frac{(s_1 + s_2)^2 x^2}{2} + \frac{x^4}{12} - \frac{(s_1 + s_2)x^3}{3} - \frac{s_1^3 x}{3} + \frac{s_1^4}{12} \right] \right\}. \quad (5b)$$

Because r_K is small, the length s_2 over which it exerts force q is short (on the order of a micron). Therefore, Eq. (5) is virtually indistinguishable from Eq. (1) when the values of P and q are properly chosen. Iteration on s_1 and s_2 , with a total deflection of h , is required to match the values of r_K and q at a given RH from Eqs. (3) and (4). This was done using at RH values of 30, 70 and 95%. The result is that the sum $s_1 + s_2 = s$ is $\sim 90 \mu\text{m}$ and is virtually independent of RH.

This balance in s is because as RH increases, r_K increases, which increases s_2 but reduces q .

Fig. 6 Assuming a smooth interface- Calc #1

Fig. 7 Assuming a rough interface - Calc #2

Because the form of Eq. (5) is indistinguishable from Eq. (1), Eq. (3) may be used to calculate the effective Γ . For a smooth interface, the calculation shows that Γ should be $2\gamma = 142 \text{ mJ/m}^2$, and independent of RH. This verifies that the expected value of $\Gamma = 2\gamma$ from thermodynamic considerations is also a direct result of the mechanics. That adhesion is $\sim 140 \text{ mJ/m}^2$ and independent of RH has been observed for silica glass [23], where the fracture plane is atomically smooth. The calculation and data are plotted as indicated in Fig. 5, and denoted "Calc #1". While the data from ref. [23] and calculation agree for a smooth interface, we conclude that the assumption of a smooth interface for polysilicon beams is poor, and that roughness must be considered. This is necessary because while the rms roughness is about 2.5 nm as reported above, the radius of individual asperities is much larger.

In the second model, we allow for roughness at the interface by considering capillary condensation at an asperity point as in Fig. 7. Because the radius of asperity tips R is large compared to r_K , the interfacial energy per wetted asperity contact can be shown to be [24]

$$W_{Asp} = 8\pi \gamma R r_K, \quad (6)$$

We make a first order assessment of Eq. (6). From AFM measurements, we obtain an approximate value of $R=50$ nm. Calling the asperity contact density n_{Asp} , inserting $R=50$ nm in Eq. (6) and substituting in Eq. (3), we obtain

$$\Gamma \approx W_{Asp} n_{Asp} = -n_{Asp} \frac{0.94 \times 10^{-16} \text{ J}}{\ln(RH)} \frac{1}{m^2}. \quad (7)$$

This relation is likewise plotted and labeled as "Calc #2" in Fig. 5, assuming $n_{Asp}=20$ per μm^2 . Eq. (7) predicts that the adhesion is approximately exponential with RH, similar to the data. Between 30 and 80% RH, the slope for the data is perhaps somewhat greater than Eq. (7). This may be because in reality n_{Asp} likely increases as RH increases, due to the increasing Laplace pressure.

The effect of roughness on real contact area has long been recognized [25]. Tian and Bhushan [26] have recently implemented 3-D finite element modeling of rough surfaces brought into contact under humidified conditions. The rough surfaces were measured by AFM and directly inserted into the model. Their calculations indicate that the simple linear relationship between meniscus area and height does not exist for rough contacts. The value for n_{Asp} can perhaps be explained by studying the details of the manner in which rough surfaces come together.

IV. Summary and conclusions

We have studied the effect of RH on adhesion for hydrophilic polysilicon beams under equilibrium conditions. The data demonstrates that adhesion increases exponentially with RH. The analysis indicates that we can employ a single asperity model to understand the effect of RH on adhesion. These data and modeling improve our fundamental understanding of adhesion

(e.g., stiction) in MEMS, and can lead to model-based calculations for MEMS design and packaging technology.

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3. Humidity dependence of adhesion for silane coated microcantilevers (de Boer, Mayer and Michalske)

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Abstract

This study examines adhesion between silane-coated micromachined surfaces that are exposed to humid conditions. Our quantitative values for interfacial adhesion energies are determined from an in-situ optical measurement of deformations in partly-adhered cantilever beams. We coated micromachined cantilevers with either ODTS ($C_{18}H_{37}SiCl_3$) or FDTS ($C_8F_{17}C_2H_4SiCl_3$) with the objective of creating hydrophobic surfaces whose adhesion would be independent of humidity. In both cases, the adhesion energy is significantly lower than for uncoated, hydrophilic surfaces. For relative humidities (RH) less than 95% (ODTS) and 80% (FDTS) the adhesion energy was extremely low and constant. In fact, ODTS-coated beams exposed to saturated humidity conditions and long (48 hour) exposures showed only a factor of two increase in adhesion energy. Surprisingly, FDTS coated beams, which initially have a higher contact angle (115°) with water than do ODTS coated beams (112°), proved to be much more sensitive to humidity. The FDTS coated surfaces showed a factor of one hundred increase in adhesion energy after a seven hour exposure to 90% RH. Atomic force microscopy revealed agglomerated coating material after exposed to high RH, suggesting a redistribution of the monolayer film. This agglomeration was more prominent for FDTS than ODTS. These findings suggest a new mechanism for uptake of moisture under high humidity conditions. At high humidities, the silane coatings can reconfigure from a surface to a bulk phase leaving behind locally hydrophilic sites which increase the average measured adhesion energy. In order for the adhesion increase to be observed, a significant fraction of the monolayer must be converted from the surface to the bulk phase.

I. Introduction

The presence of moisture can strongly affect the surface energy of materials. Microelectromechanical Systems (MEMS) [1] with hydrophilic surfaces are inherently sensitive to moisture because of their very large surface to volume ratio and small restoring forces. For hydrophilic micromachined surfaces, an exponential increase in equilibrium adhesion energies Γ [2] with relative humidity (RH) from 1 to 50 mJ/m², as RH increases from 30% to 95%, has been measured [3]. This trend was shown to agree well with a single asperity model at contacting areas of the surfaces, which exhibit nanometer scale roughness. Such energies are far too high to ensure reliable operation of MEMS devices.

The problem is greatly reduced by the application of hydrophobic silane coupling agents to the polycrystalline silicon (polysilicon) surfaces. Octadecyltrichlorosilane (ODTS, $C_{18}H_{37}SiCl_3$) or fluorodecyltrichlorosilane (FDTS, $C_8F_{17}C_2H_4SiCl_3$) have been proposed [4-6] and shown to reduce the adhesion energy to the $\mu J/m^2$ regime. ODTS has been of interest as an adhesion promoter in glass reinforced composites [7], as an agent in improving selectivity in chromatographic separations [8], as a means to lower friction coefficients of tribological coatings [9,10], and has more recently been applied to MEMS in order to reduce adhesion and friction [4,11]. However, FDTS may have greater promise for integration in MEMS, particularly because of its ability to withstand air temperatures up to 400 °C, common in the packaging process required to protect devices and for electrical connection [6].

For such hydrophobic coatings, the surface energy γ_s is reduced, and the interfacial energy γ_{ls} between liquid water and the surface coating is much larger than for the hydrophilic

Fig. 1 Nucleation of water on a hydrophobic surface is unfavored.

case. Because the surface tension of water with air γ_{la} remains the same, the macroscopic contact angle θ between water and the surface is greater than 90° and the surface is hydrophobic. In a macroscopic sense, it is thermodynamically unfavorable for water to nucleate on hydrophobic surfaces, as represented in Fig. 1. Therefore, humidity should have no effect on adhesion of micromachines coated by hydrophobic materials.

However, considering that silane coupling agents are deposited as molecular monolayers, it is important to determine their limitations with respect to severe conditions such as high temperature or high humidity. Although a large body of literature on this subject reflects the intensive study on these films [8,12-21], our understanding of the formation, structure and stability of siloxane films is rather incomplete. Nonetheless, it is clear from these studies that structural defects exist in these films and that structure is a sensitive function of the deposition process. Using micromachined cantilever beams as model test structures, we determine that in a high humidity environment, defect-driven reconfiguration of the silane coatings from a surface to a bulk phase greatly increases adhesion. The results are in agreement with infrared spectroscopy evidence that the silicon head group does not bond covalently to the oxide surface [19], and may be important for many applications where maintaining constant surface energy is crucial.

II. Experimental

Fig. 2 (a) Cantilever beam test structure after release and before actuation. (b) after actuation and relaxation of V_{pad} to 0V. Long beams remain deformed in an S-shape due to adhesion.

We designed and fabricated the polysilicon cantilever beam array test structures with an actuation pad near the support post. A cross-section of the cantilever geometry is shown schematically in Fig. 2. The surface micromachine fabrication process to produce these structures is described in detail in refs. [6] and [22]. The thickness t of the beams was $2.05 \mu\text{m}$, the height of the beams h was $2.30 \mu\text{m}$, while their lengths ranged from 100 to $2000 \mu\text{m}$. The structures are encased in glass until the final stage of MEMS processing. The cantilevers are removed from the glass matrix in hydrofluoric acid,

and coated with ODTS and FDTS according to the solvent exchange procedure described in detail in ref. [6]. On flat silicon pieces processed along with the samples, the static contact angle with water was 112° and 115° for ODTS and FDTS samples respectively. When removed from

water, the hydrophobic surface prevents capillary action from acting on the beams, and therefore the beams are suspended above the substrate. The rms roughness of the polysilicon landing pad in Fig. 2 was 2.5 nm, as measured by atomic force microscope (AFM) measurements on the landing pad area of the samples. The bottom side of the polysilicon beam exhibits a similar roughness. Samples were stored in a drybox under nitrogen flow before testing.

To measure adhesion *in-situ*, we constructed an environmental microprobing station as represented in Fig. 3. The sample is placed in a stainless steel chamber into which a controlled flow of humidified nitrogen combined with dry nitrogen is introduced. A Michelson interferometer attachment was placed on a 5X optical microscope objective, and the samples were illuminated by monochromatic green light at 547 nm. Magnification including the eyepiece is 50X, and resolution is approximately 3 μm in the plane of the sample. We sealed the chamber from the ambient with a membrane between the Michelson attachment and the chamber. RH was monitored by means of a humidity probe.

Fig. 3 Interferometric environmental microprobe station. For schematic, see Fig. 2 of Chapter 2.

As represented in Fig. 2(a), the beams were initially free from the substrate after the monolayer ODTS or FDTS coating process. To actuate the beams for adhesion measurements, a voltage of 160V was applied to the actuation pad to bring the beam into contact with the substrate. At 160V, the crack length $s \approx 300 \mu\text{m}$ as represented in Fig. 2(b). The beams as well as the landing pad are electrically grounded, ensuring that only adhesive forces exist between the beam and the substrate once the actuation pad voltage is relaxed to 0V. (The complete actuation pad voltage cycle was 0, 50, 100, 120, 140, 160, 140, 120, 100, 50 and 0V, with approximately 30 seconds between each voltage increment. Looking over this sequence allows us also to study adhesion hysteresis [23]). After the actuation pad voltage cycle, the crack length s represents a mechanical equilibrium between mechanical energy in the beam and surface energy in the interface between the beam and the substrate [24].

At 0V, each beam adhered over a long attachment length d is configured in an S-shape, and adhesion can be measured using deflections calculated from the interferogram. This is done as follows: First, background fringes are aligned parallel to the length of the beams. Then, fringe information along the length of the beam is digitized and converted to deflection data by a computer program, resulting in approximately 10 nm out-of-plane resolution with a data point at each pixel (approximately every $2.75 \mu\text{m}$ at the 50X magnification used). The measured deflection is compared to the calculated deflection from elasticity theory [25] under the assumptions of the JKR model [26] (i.e., adhesive force exist only in the contact zone). An example will be given in the results section.

On a given cantilever beam array, we first verified that the beams were free from the substrate, and then characterized their radius of curvature R (due to stress gradient through the thickness of the film) using interferometry. The beams were then placed in the environmental chamber, and a 24 hour dry nitrogen purge was carried out to minimize water adsorption. Next, the beams were brought into contact with the substrate by applying the pad voltage actuation cycle described above. An interferogram was recorded at each voltage. This cycle was repeated at intervals of 1/2, 4, 7 and 10 hours at a given RH. After the test was carried out at 0% RH, the RH was increased to levels of 30, 60, 80, 90, 95 and 99%, and the adhesion test was repeated at each RH. At high RH levels, the samples were exposed up to 48 hours to determine long term effects. The timing between voltage actuation cycles, the application of the pad voltage, the

taking and storage of interferograms and the monitoring of the RH were all carried out under computer control. The computer-stored interferograms were later analyzed to measure adhesion.

Each sample test chip contained multiple arrays of cantilever beams. This allowed us to determine that the exposure to high RH levels, rather than repeated contact cycles, was the main contributor in the adhesion effects we observed.

III. Results

A) Adhesion versus humidity

An interferogram of an ODTS-coated cantilever array of 1500 μm length beams after the silane coating procedure is presented in Fig. 4(a). The fringes are attributed to a residual stress gradient through the thickness of the structural polysilicon film, giving rise to beam curvature. In this case the beams bend up [27]. ODTS-coated beams were initially suspended above the substrate after coating to a length of 2000 μm . Likewise, FDTS-coated beams were free to the maximum length of 2000 μm , however they only very slightly curved up, as seen in Fig. 4(b).

Fig. 4 (a) Interferogram of ODTS sample before actuation. Beams are 1500 μm long and are curled up due to stress gradient in the polysilicon film. The apparent shift in fringe location from top to bottom is due to a small tilt of the reference plane relative to the substrate. (b) Interferogram of FDTS sample before actuation. The beams are 1500-2000 μm long and very nearly flat, as indicated by the lack of fringes.

Fig. 5 Curvature of 1000 μm beams from interferograms. The data matches very well with simple beam theory assuming an external moment (due to the strain gradient) is applied at each end of the beam

The polysilicon film curvature is sensitive to minor annealing temperature variations in the final step of thermal processing before removing the glass matrix, and is readily determined by converting the fringe intensity data to deflection data as described above. This was done on beams of less than 1000 μm length so that gravitational effects could be ignored. The result is shown in Fig. 5, where we see that the radius of curvature is 0.21 m for the ODTS coated beams, and 1.6 m for the FDTS coated beams.

We display an interferogram of an ODTS-coated adhered beam array at RH=0% after the first pad actuation cycle in Fig. 6(a). In this array, the beam lengths are from 1000 to 1500 μm . At lengths greater than a transition length $L_{Tr}=1200$ μm , most beams remained adhered over a significant attachment length d , as can be seen in Fig. 6(a). Such beams are S-shaped. As described in ref. [24], shorter beams of length $L < L_{Tr}$ are adhered at their tips only and are arc-shaped until they decrease to a length $\sim 0.6 L_{Tr}$, when they free themselves from the substrate to assume their unadhered shape. Some exceptions to this exist, e.g., the longest beam at the top of Fig. 6(a). For such beams, the local adhesion is apparently very low, and they remain adhered at their tips only. Also, some of the other longest beams, such as the second from the top of Fig. 6(a) continue to show fringes along the length of the beam. Although they are adhered over a reasonably long length d , they delaminate from the substrate beyond the contact zone due to their curvature. This could be determined from the deflection data – such beams were clearly configured in the S-shape at a value of s significantly less than L .

An example of the quantification of the beam deflections is carried out in Fig. 7 (for the 6th beam from the top of Fig. 6(a)). The linescan across the beam from the top is plotted against the right hand axis. The deflection data is plotted against the left hand axis, where it is compared to the equation [24]

$$u(\eta) = h(\eta^2)(3 - 2\eta), \quad \eta = x / s, \quad (1)$$

Fig. 6 (a) Array of 25 ODTS-coated beams after first actuation. Beam lengths in this array increase by 20 μm increments from 1000 to 1500 μm . The transition from arc shaped beams adhered at their tips to S-shaped beams adhered over a significant portion of their length occurs at $L_{tr}=1200 \mu\text{m}$. (horizontal compressed 2.7X relative to vertical). (b) Array of FDTS-coated beams after first actuation. Beam lengths in this array increase by 20 μm increments from 1500 to 2000 μm . $L_{tr}=1700 \mu\text{m}$. (horizontal compressed 2.7X relative to vertical)

where u is the deflection, h is the height of the support post and s is the crack length as defined in Fig. 2(a). Because the data and theory for the deflections agree well, the equation

$$\Gamma = \frac{3}{2} E \frac{h^2 t^3}{s^4}, \quad (2)$$

can be used to determine adhesion Γ of the beam to the substrate [24], where t is as defined in Fig. 2(a), and $E=170 \text{ GPa}$ [28] is Young's Modulus. For Fig. 7, $s=1069 \mu\text{m}$ can be applied to determine a value of adhesion $\Gamma=8.9 \mu\text{J}/\text{m}^2$ for this beam. This procedure was repeated for each of eight beams adhered over a long length in Fig. 5(a), with the results reported in the first row of Table 1. The average adhesion for the ODTS-coated beams at 0% RH is $\bar{\Gamma}=6.7 \mu\text{J}/\text{m}^2$ and standard deviation $\sigma=1.8 \mu\text{J}/\text{m}^2$.

In Appendix 1 we demonstrate that a correction to the adhesion calculation Eq. (2) to account for the stress gradient is not necessary for S-shaped beams. This non-intuitive result is very useful because it simplifies the adhesion calculation, quite different from the case of beams adhered only at their tips [29].

Due to their lower adhesion, we chose an array of beams from 1500-2000 μm in length for the adhesion measurements of the FDTS-coated beams. After the first pad actuation voltage cycle at RH=0%, the initial crack lengths were longer than for the ODTS-coated beams, and $L_{Tr}=1700 \mu\text{m}$ is observed in Fig. 6(b). These are indications of lower adhesion for the FDTS-coated structures. For eight beams measured with $L > L_{Tr}$, $\bar{\Gamma}=2.4 \mu\text{J}/\text{m}^2$, and $\sigma=1.1 \mu\text{J}/\text{m}^2$.

The values of $\bar{\Gamma}=6.7 \mu\text{J}/\text{m}^2$ for ODTS and $\bar{\Gamma}=2.4 \mu\text{J}/\text{m}^2$ for FDTS coated beams found here are in reasonable qualitative agreement with previous results by the cantilever beam array

Fig. 8 Adhesion of 8 individual beams vs. RH for ODTS and FDTS coatings.

Fig. 7. Linescan of fringes (right-hand abscissa) and deflections (left-hand abscissa) of the 1400 μm long ODTS-coated beam (6th beam from top of Fig. 6a) after initial actuation. The measured deflection shape agrees very well with the shaped calculated from theory, Eq. (1), as represented by the dashed line.

technique where values of 30 and 8 $\mu\text{J}/\text{m}^2$ were determined for ODTS and FDTS respectively [6]. The reason for the lower adhesion of the FDTS coated samples was attributed to the lower surface energy of the fluorinated compared to the hydrogenated tail group. The low values in the $\mu\text{J}/\text{m}^2$ range can be attributed to the low surface energies of the coatings acting in concert with the roughness of the surfaces which causes the true contact area to be much smaller than the apparent contact area.

As humidity was increased to 90% RH, the adhesion remained at approximately $7 \mu\text{J}/\text{m}^2$ for the ODTS-coated beams. At 95% RH, a small increase in adhesion to $10 \mu\text{J}/\text{m}^2$ was noted. After a 48 hour exposure at 99% RH, the adhesion was observed to increase to approximately $18 \mu\text{J}/\text{m}^2$. The results are plotted in Fig. 8 and tabulated in Table 1.

Table I - Adhesion after exposure to increasing RH

RH (%)	Time (Hrs)	ODTS $\bar{\Gamma}$ (σ) ($\mu\text{J}/\text{m}^2$)	FDTS $\bar{\Gamma}$ (σ) ($\mu\text{J}/\text{m}^2$)
0	0	7 (2)	2 (1)
80	12	7 (2)	2 (1)
90	4	7 (2)	4 (2)
90	7	7 (2)	273 (395)
95	10	10 (5)	790 (781)
99	40	18 (10)	1008 (645)

In Fig. 9, we show interferograms of FDTS-coated beams subjected to high humidity levels increasing from 80 to 95%. Similar to the ODTS, adhesion was unchanged over a large range of RH from 0 to approximately 80%, as can be seen by comparing Fig 9(a) to Fig 7(b). However, at 90% RH, adhesion began to increase with exposure time. Relative to Fig. 9(a), the effect is small, yet noticeable in Fig. 9(b). Counting from the top of Fig. 9(b), s for beams 7, 8, 9 and 11 has decreased noticeably, and the same can be said for several of the beams near the bottom of the interferogram. Furthermore, the transition length L_{Tr} has decreased from $\approx 1610 \mu\text{m}$ to $\approx 1570 \mu\text{m}$. After the 4 hour exposure at 90% RH, the average adhesion increases to $\bar{\Gamma}=3.5 \mu\text{J}/\text{m}^2$ and $\sigma=1.8 \mu\text{J}/\text{m}^2$ as graphed in Fig. 8.

A far more dramatic effect is observed in Fig. 9(c), where the exposure time at 90% RH has increased to 7 hours. The crack lengths for various beams has decreased from 1200 to 1600 μm in Fig. 9(b) to 300 to 700 microns in Fig. 9(c). This corresponds to a large increase in adhesion according to the $1/s^4$ relationship of Eq. (2). The standard deviation in the data is now larger than the average value with $\bar{\Gamma}=273 \mu\text{J}/\text{m}^2$ and $\sigma=395 \mu\text{J}/\text{m}^2$, indicating some non-uniformity in the mechanism causing the adhesion increase. At 95% RH $\bar{\Gamma}$ continues to increase, while $\sigma/\bar{\Gamma}$ decreases to less than unity after a 30 hour exposure at this RH. From this, one may infer that the mechanism for adhesion increase eventually effects a more uniform

change across the entire surface. In Fig 8, the increase in Γ as well as the sharp increase and subsequent decrease of $\sigma/\bar{\Gamma}$ for the FDTs-coated beams is clearly seen.

Fig. 9 FDTs-coated beams after actuation and (a) RH=80%, 10 hour exposure. (b) RH=90%, 4 hour exposure (c) FDTs coating, RH=90%, 7 hour exposure (d) RH=95%, 30 hour exposure

Beam arrays on the same FDTs sample test chip which had been exposed to the high RH ambient but not actuated remained free from the substrate. At high RH levels, we actuated such beam arrays, and they exhibited the same large values as the beam array of Fig. 9. This indicates that the adhesion increase mechanism is caused by exposure to the high RH, rather than by a cycling phenomenon due to multiple contacting episodes. Other beam arrays were tested after the RH was subsequently reduced to 0%. These showed no apparent effect due to the exposure, while the previously adhesion-degraded beams tested at high RH remained adhered.

B) Investigation of the degradation mechanism

Fig. 10 Ellipsometry results for water layer thickness vs. RH (10 minute equilibration time)

As discussed in the introduction, there is a strong barrier to nucleation of water on the surface given the hydrophobic nature of the coatings. However, for the FDTS films above 80% RH and ODTS films above 95% RH, this barrier is apparently surmounted. To begin to understand this phenomenon, we conducted *in-situ* ellipsometry measurements of single crystal silicon samples subjected to increasing RH levels. Three types of surface were studied: (i) oxidized hydrophilic substrates (ii)

ODTS-coated substrates and (iii) FDTS-coated substrates. Ellipsometry measures the product of the index of refraction and thickness of the layers on the substrate. This can be converted into an approximate value of film thickness, and is plotted in Fig. 10. Above 50% RH, the SiO₂ surface exhibits multilayer absorption of water. The ODTS-coated surface exhibits small but significant water uptake above RH levels of 50%. Although the water absorption of the FDTS-coated substrate is somewhat smaller than the ODTS coating, there is still a detectable amount of water absorbed. The measurements were taken after approximately a ten minute equilibration period. After longer exposure times, drift in the ellipsometer measurements make the results unreliable.

These results gives us information on the average amount of water on the surface, which we interpret in terms of a classical BET adsorption isotherm [30]. For the hydrophilic SiO₂ surface below 50% RH, a sub-monolayer film of water exists on the surface. Above 50% RH, multiple layers grow as the driving force for water to nucleate on itself increases. For the ODTS and FDTS coated surfaces, approximately a monolayer of water is adsorbed by the film when the RH reaches 100%. For this relatively short equilibration time of 10 minutes at each RH, there is not a significant difference in water absorption between the ODTS and FDTS coatings. This is qualitatively in agreement with adhesion results for the cantilever beams – a noticeable adhesion increase was only observed after 4 hours at a high humidity levels.

We next compared the landing pad areas (see Fig. 2(a)) of samples which had been exposed to high RH to the same area of those which had been kept in air (in RH generally <50%) by atomic force microscopy (AFM). A Digital Instruments (Santa Barbara, CA) Nanoscope IIIA

(a) ODTS coating exposed to high RH (b) ODTS not exposed to high RH

Fig. 11 (a) Contact mode AFM image ($10 \times 10 \mu\text{m}^2$) of ODTS-coated sample (polysilicon landing pad area) revealing mounds of agglomerated material after exposure to high RH. The mounds are easily displaced by the tip during the scanning (left to right), producing several streaks in the image. (b) Same as (a), but sample was not exposed to high RH. The contrast is due to the roughness of the substrate. FDTs-coated samples that were not exposed to high RH appeared the same.

was used with silicon nitride cantilevers. The AFM was conducted in air after the samples were removed from the high RH environment. Results are shown in Figs. 11 and 12 for the ODTS and FDTs samples respectively. A number of agglomeration sites (mounds) are seen for samples exposed to high RH for both ODTS (Fig. 11(a)) and FDTs (Fig. 12(a))—coated samples, relative to the unexposed sample (Fig. 11(b)). For the ODTS-coated sample, exposed to 99% RH for 48 hours, the density of the mounds is approximately 0.05 to 0.1 per μm^2 density, their heights are typically 5-15 nm, and the diameters are typically 400-800 nm. As seen in Fig. 11(a), the ODTS mounds are easily displaced by the AFM tip, even at low applied loads, producing streaks in the image along the fast scan direction. This effect is reduced but not eliminated by using an intermittent-contact mode imaging. This implies that the mounds are extremely weakly bound, in a physisorbed manner, to the substrate. The FDTs mounds are distinct in several ways. They are not displaced by the tip, demonstrating that they are bound much more strongly to the surface than the ODTS mounds. The density is approximately 0.1 to 0.2 per μm^2 density, which is twice the density of the ODTS mounds. The heights are typically 10-25 nm, but can be as large as 65 nm, and the diameters are typically 500-1000 nm. A linescan of a relatively large mound is

Fig. 12 (a) Contact mode AFM image ($10 \times 10 \mu\text{m}^2$) of FDTS-coated sample (polysilicon landing pad area) revealing mounds of agglomerated material after exposure to high RH. Without exposure to high RH, the samples appeared the same as Fig. 11(b).
 (b) Linescan across a mound as designated by the gray line near the bottom of (a).

shown in Fig. 12(b). For both ODTS and FDTS films, smaller mounds may be present but are difficult to distinguish from the somewhat rough substrate.

IV. Discussion

Both the ODTS and FDTS coated films behave remarkably well for micromachining applications – up to a threshold value of 95 and 80% RH respectively there is very limited susceptibility to humidity. This implies that well coated micromachine structures do not require special storage or packaging in most situations. Even when the FDTS degrades, the maximum adhesion observed at 95% RH is only 1 mJ/m^2 , much smaller than the value of 50 mJ/m^2 measured for uncoated hydrophilic beams at the same RH level [3]. The adhesion result for threshold RH levels up to 95 and 80% for ODTS and FDTS respectively is in agreement with the detailed attenuated total reflectance infrared ATR-IR study of Angst and Simmons on ODTS [13]. They report that at humidity levels up to 80% RH, monotonically increasing water absorption in the film occurs at silanol sites at the silica-silane interface. The ellipsometry results of Fig. 10 also agree qualitatively with Angst and Simmons' work, who measure approximately a monolayer of water is adsorbed at high RH in the interfacial sites. Because the

monolayer tail group remains as the exposed surface, their results would predict that adhesion energy should not change with RH. This agrees with our results up to the threshold level. However, at very high levels of RH and long exposure times, the ODTS and FDTS-coated films in this study clearly exhibit an increase of adhesion on humidity, an effect explained neither by Angst and Simmons nor by our ellipsometry results.

Our adhesion results on the silane coating agents are also in contrast to the adhesion behavior of fatty acids on atomically smooth mica surfaces at RH levels from 0 to 100% studied in the surface force apparatus (SFA) [31,32]. There, water penetration lead to uniform swelling of monolayers increasing monotonically with RH, with concomitant lateral mobility, overturning of molecules within the monolayer, and monotonically increasing adhesion with RH. The resistance to humidity of the silane coupling agents is probably due to the tendency (albeit weak) towards cross linking of the head groups [8,19]. This cross linking tendency does not exist in the fatty acid films, and is the reason that water can penetrate at any RH level. Neither the results of Angst and Simmons [13], nor of Israelachvili and coworkers [31,32] helps us to understand the mechanism for adhesion increase at high RH levels we observe in the silane coupling agents. The fact that a threshold humidity value for adhesion degradation is observed for the silane coupling agents, as well as the highly non-uniform onset of the adhesion degradation mechanism, implies that a defect mechanism is more likely in the present situation. This notion is consistent with the appearance of mounds as seen in the AFM images of Figs. 11 and 12.

How might the mounds initiate in the silane coupling agents at high RH values? From AFM observations on ODTS after various stages of growth, films are known to contain a variety of defects and domain structures with different density [15,16]. Two types of defects which must be present are low density regions with disordered tail structure (liquid expanded - LE) and domain boundaries of high density ordered regions (liquid condensed - LC). Steric factors and the degree of ordering on the tail groups may both play a role in determining the film density. Wirth [8] and Stevens [12] have argued that close packing of tail groups in films formed from ODTS cannot be accommodated over large areas due to steric constraints. The Si-O-Si bond distance of 0.32 nm is smaller than twice the van der Waals radius of the hydrocarbon chain, 0.4 nm, thus close packing of the tail groups would lead to unacceptable strain in the polymerized head groups. This would imply that disordered regions of less than theoretical density are likely to form. From these various observations it is clear that the experimental

measurement of a large contact angle, 110° to 120° , does not necessarily indicate a densely packed array of tail groups, or the complete passivation of the surface toward water adsorption.

Such regions of low density and structural defects may serve as sites where water can penetrate the film and adsorb or react at the substrate interface. This allows water islands to

Fig. 13 Schematic of water nucleation in FDTD films

emerge on the surface of the film at high RH levels, as seen in Fig. 13. The very small apparent film thickness observed by ellipsometry in Fig. 10 could alternately be viewed as a formation of many small water droplets nucleating at defect sites on the surface. This local, defect controlled adsorption of water may be the incipient mechanism for the formation of the mounds.

The relatively large height of the mounds suggests that there is a further evolution of the film structure beyond the

water nucleation suggested in Fig. 13. Monolayer degradation can initiate at a site as shown in Fig. 13. If we suppose that the headgroups are cross linked by hydrogen bonding rather than covalently bonding as has been suggested in ref. [19], it would be favorable for molecules to detach from the interface at local water islands and rearrange themselves into a bulk phase. We can imagine two mechanisms by which the large mounds form. In the first, head groups might roll up and expose themselves to the ambient, forming a structure analogous to rolled sod. This seems possible because of the low energy interface which would form when water wets the exposed silicon atoms. Also, it is reasonable that an accumulated rolling process would result in such large mounds. However, there are two arguments against this notion. First, phase diagrams for surfactants [33] indicate that hydrophilic head groups would prefer to remain oriented towards the inside of the water droplets, leaving the hydrophobic tail group exposed. Second, if the tail groups were hydrophilic as in the rolled sod model, we would expect spontaneous healing of the cracks due to strong capillary forces as is observed for uncoated hydrophilic beams [3]. In contrast, electrostatic actuation was required to heal cracks in our experiments, even at a 97% RH level. Therefore, the mounds likely are hydrophobic, and perhaps diffusional transport is the most likely mechanism for their formation. A schematic representation is shown in Fig. 14. The

details of this formation process, as well as the micellar, vesicular or lamellar nature of the mound structure remains to be elucidated.

Although both films are expected to contain structural defects, the large difference in behavior between the ODTS and FDTS coated beams is surprising. One reason may be the density achieved by the respective films. The density obtained during deposition is likely related to both the deposition dynamics as well as steric constraints between closely spaced molecules. Previous studies of ODTS deposition have measured packing densities

of 4.2 to 4.8 per nm^2 [20,21] compared to a theoretical maximum packing density of 7.2 per nm^2 based on the van der Waals radius of the molecule and the assumptions of all-trans configuration and close hexagonal packing of the tail group [34]. This implies that the ODTS film achieves 60% of its theoretical density. For FDTS films, a density of 3.3 per nm^2 has been reported [21], compared to a theoretical maximum by this same calculation of 4.4 per nm^2 , giving the FDTS film a 75% theoretical density. Although neither film achieves theoretical density levels, the absolute density of the ODTS films is higher.

The performance difference between the films can be attributed to the two following considerations. First, because the 18 carbon ODTS chains are longer and denser than the 10 carbon FDTS chains, interchain bonding forces are greater, suggesting that the driving force for molecular alignment is larger for ODTS. Second, because the ODTS hydrocarbon chain is of smaller diameter than the FDTS fluorocarbon chain, one would expect that the strain induced by head group polymerization is larger in the FDTS case, also inducing a greater degree of structural imperfections. This has been observed for the case of thiols on gold [35]. Although water intercalates into both films, defects in the FDTS films are more open, allowing greater water penetration.

Although the ODTS adhesion increase was much less than FDTS, the AFM results of Figs. 12 and 13 suggest that the mechanism for the increase is similar for the two films. If we assume that the mounds consist mainly of the original coupling agent at the average of the

Fig. 14 Possible mechanism for formation of agglomerated mounds.

density, diameter and height reported above, and that the mound density measured by AFM is the same as that while the sample is exposed to high RH, we can calculate the percentage of the original monolayer which has been converted into the bulk phase. For the FDOTS films, the volume of the mounds is then approximately 40% of the volume of the film on a uniformly coated flat surface. Migration of coupling agent from within a 900 nm radius of the original defect can then explain the mound size. Under the same assumptions for the ODOTS films, the volume of defects is approximately 5% of the volume of the film on a uniformly coated flat surface. With the smaller average mound size, migration of approximately 500 nm is required. This implies that adhesion of the cantilevers at high RH is sensitive to only a large distortion of the silane coupling agent.

Fig. 15 Schematic of crack tip for FDOTS surface after exposure to high RH

As seen in Fig. 14, locally hydrophilic areas will be left behind when the hydrophobic mounds form. A factor which works counter to the adhesion increase is that the mounds are slightly higher than the surrounding hydrophilic areas. To explain the result that adhesion energies are observed to increase only upon actuation of the electrostatic pad, it is necessary to consider that compressive forces exist very near the crack tip. These forces can quantifiably account [23] for sufficient deformation of the mounds that the hydrophilic mechanism is engaged, as suggested in Fig. 15. Support for this picture will be presented in the next Chapter.

V. Summary and implications

We constructed an environmental microprobing station with interferometric capability to measure the effect of humidity on adhesion of micromachined cantilever beams in-situ. We verified our adhesion values by measuring individual beam deflections which compared favorably with elastic theory calculations. A correction to account for the effect of strain gradient was shown to be zero for S-shaped beams (e.g. Appendix 1).

A simple macroscopic model, which predicts that adhesion should be independent of RH, works well below a threshold value of RH 95% and 80% RH for the ODTS and FDTS coatings respectively. This is in good agreement with the results of Angst and Simmons, who report that for RH up to 80%, who concluded that up to a monolayer of water is adsorbed at the hydrophobic film/oxide interface, even though the hydrocarbon outer surface of the film remains hydrophobic. For ODTS-coated beams, adhesion is independent of RH for levels up to 95%, and increases by a factor of two after a prolonged exposure of 48 hours at 99% RH. For the FDTS-coated beams, adhesion remains constant at RH levels below 80%, and increases significantly by a factor of 500X after a 48 hour exposure at 95% RH. The adhesion increases with exposure time on the order of hours for the FDTS beams. However, the FDTS coated films exhibit 50 times lower adhesion at 95% RH relative to uncoated surfaces [3] even after they have begun to degrade.

Ellipsometry measurements indicate that water is present on the surface for both ODTS and FDTS films, but do not explain the increase in adhesion after long exposure times at high RH levels. Results on fatty acids using the surface forces apparatus [31,32] also do not explain the behavior of the silane coupling agents. We propose a new mechanism for water uptake at very high RH levels for silane monolayers. Namely, water adsorption at silanol sites at the interface between the FDTS coating and the oxidized substrate leads to islanding of water at high RH levels, as in Fig 13. This leads the film to reconfigure from a two-dimensional to a more stable three-dimensional phase at high RH levels, leaving behind locally hydrophilic areas which cause the adhesion increase, as in Figs. 14 and 15. The ODTS film is susceptible to a similar phenomenon, but better resists conversion from the surface to the bulk phase because it is denser and has a longer tail length. The conversion from surface to bulk phase is on the order of a monolayer for the FDTS, but only about five percent of a monolayer for the ODTS, indicating that only large scale reconfiguration of the monolayer film can cause the adhesion increase.

This work adds further insight into the question of which molecule, ODTS or FDTS, is more appropriate in packaged MEMS applications. Previous work [6] suggested that FDTS might be favored because it is less susceptible to oxidation at temperatures up to 400 °C. However, even hermetically sealed packages may eventually allow penetration of water, and this possibility should be considered when assessing the long term reliability of MEMS devices. The present results may also be important for durability of MEMS coatings subject to sliding friction.

Namely; energy supplied by the sliding motion may significantly lower the threshold humidity or time for coating degradation.

The adhesion increase does not occur spontaneously, in contrast to the case of hydrophilic beams where capillary action at the crack tip does work on the beams to decrease the crack length s [3]. Rather, actuation of the beams after prolonged exposure to high RH is required to observe the adhesion increase in the present case of hydrophobic beams. If operation of MEMS devices at low RH can be assured, then the degradation of the coupling agent may not cause reliability problems.

In-situ interferometric observation of cantilever beams subject to various environments is a very useful tool for evaluating the stability of coating materials. The method can be extended from the static to the dynamic regime for greater insight into adhesion related mechanisms of surfaces.

VI. Appendix

Stress gradient contribution to G is zero for S-shaped beams

Micromachined cantilever beams often exhibit curvature due to an internal stress gradient through the thickness of the film. In order for a cantilever beam to configure itself in the S-shape indicative of a fracture mechanics adhesion equilibrium, strain energy must be expended to overcome this internal strain gradient. We calculate this energy, and then take its derivative in order to find the strain energy release rate G .

A free beam with stress gradient has a radius of curvature $-\rho$ (with ρ a positive number), which is negative for a beam curved up as in Fig A1(a). By applying an external moment M_o , where

$$M_o = -\frac{D}{\rho} \quad (A1)$$

the initially curved beam will be flattened as in Fig. A1(b). After external forces are applied to bring the beam into contact with the substrate over a long length, sufficiently long beams will remain adhered in the S-shape as in Fig. 2(b). Deflections of the S-shaped beam are unaffected by the strain gradient, however an additional internal moment $-M_o$ exists in the beam as shown in Fig. A1(c). The S-shape deflection is

$$u(x) = 3h\left(\frac{x}{s}\right)^2 - 2h\left(\frac{x}{s}\right)^3 \quad (A2)$$

Over the unadhered region of the beam $0 \leq x \leq s$, the total moment in the beam is the sum of the moments due to the S-shape deflection and the internal moment M_o

$$M = M_s + M_o = -D \frac{d^2 u}{dx^2} + M_o = \frac{6Dh}{s^2} \left(\frac{2x}{s} - 1 \right) + M_o \quad (\text{A3})$$

while the adhered portion of the beam $s \leq x \leq L$ has a moment $-M_o$.

The strain energy due to bending in the beam is

$$U_E = U_{E1} + U_{E2} = \frac{1}{2D} \int_0^s (M_s + M_o)^2 dx + \frac{1}{2D} \int_s^L M_o^2 dx \quad (\text{A4})$$

which reduces to

$$U_E = U_{E1} + U_{E2} = D \left[\frac{6h}{s^3} + \frac{s}{2\rho^2} \right] + D \left[\frac{L}{2\rho^2} - \frac{s}{2\rho^2} \right] = \frac{D}{2} \left[\frac{12h^2}{s^3} + \frac{L}{\rho^2} \right] \quad (\text{A5})$$

The strain energy release rate G is then

$$G = -\frac{dU}{ds} = \frac{18Dh^2}{s^4} = \frac{3}{2} \frac{Et^3 h^2}{s^4}, \quad (\text{A5})$$

identical to the result with zero strain gradient assumed [25]. This is a non-intuitive result, because one might expect that the excess energy in the beam would give rise to a larger G . As can be seen from (A5), however, the s dependence of this excess energy over the interval $0 \leq x \leq s$ is cancelled out by the s dependence over the interval $s \leq x \leq L$.

Therefore, once the crack length s is determined for an adhered S-shaped beam, adhesion can be accurately determined *without needing to measure the initial curvature* in the beam. This is in striking contrast to adhered arc-shaped beams in which this must be accounted for [29]. Two major advantages are clear: (1) the calculation is simple and (2) even if all the beams are initially adhered in the S-shape such that the strain gradient cannot be measured, the value G for is in any case unaffected. If the compliance of the boundary is taken to be non-zero, this result will be affected slightly. For the relatively low adhesion energies considered in this work, this effect will be very small.

Fig. A1 (a) beam after release with curvature due to strain gradient. (b) beam is straightened by applying an external moment M_o . (c) S-shaped beam and associated moment diagrams with 0 strain gradient (solid line) and with an internal strain gradient (dashed line).

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4. Adhesion hysteresis of silane coated microcantilevers (de Boer, Mayer and Michalske)

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Abstract

We have developed a new experimental approach for measuring hysteresis in the adhesion between micromachined surfaces. By accurately modeling the deformations in cantilever beams that are subject to combined interfacial adhesion and applied electrostatic forces, we determine adhesion energies for advancing and receding contacts. We draw on this new method to examine adhesion hysteresis for silane coated micromachined structures and found significant hysteresis for surfaces that were exposed to high relative humidity (RH) conditions. Atomic force microscopy studies of these surfaces showed spontaneous formation of agglomerates that we interpreted as silanes that have transformed from uniform surface layers at low RH to isolated vesicles at high RH. We used contact deformation models to show that the compliance of these vesicles could reasonably account for the adhesion hysteresis that develops as the surfaces are forced into contact by an externally applied load.

I. Introduction

Adhesion and friction are significant tribological concerns in addressing the reliability of Microelectromechanical (MEMS) devices [1,2]. This is because surface to volume ratio of structures grows as dimensions shrink. The structural members in MEMS are only a few microns thick and a few microns apart, are constructed of polycrystalline silicon (polysilicon) and exhibit nanometer scale root mean square (rms) roughness. The surface properties of MEMS devices are strongly related to their processing, as well as their environmental exposure. For example, polysilicon roughness depends on its deposition temperature [3]. Likewise, the surface energy of the polysilicon depends on the final surface treatment process. If supercritical drying [4] is performed to release the parts, the surfaces are high energy and hydrophilic. The surfaces can instead be rendered hydrophobic by applying monolayer lubrication layers [5]. Adhesion can be locally affected by contamination or by deposition kinetics in the surface treatment process resulting in non-uniform films [6-9]. Therefore, it is important to develop in-situ tribology techniques on as-fabricated MEMS parts. These will eliminate the question of whether measured results from macroscopic tribological tools reflect real properties of MEMS structures. Furthermore, issues of coating stability, aging and wear, important in micromachined products [6], can be directly addressed.

Knowledge of adhesion hysteresis values may be valuable in developing a fundamental understanding of MEMS tribology. The surface forces apparatus (SFA) [10] is commonly used to study adhesion and friction of monolayer coatings. A link often made between these properties is adhesion hysteresis, the difference between the adhesion energy measured when two surfaces are brought into contact versus when they are being separated [11]. The connection is that the static friction coefficient is related to the adhesion values of propagating cracks, while the dynamic friction coefficient is related to adhesion hysteresis values. Friction experiments on sliding surfaces of micrometer scale roughness have also led to the notion that the age of contact is critical in determining coefficients of friction [12], suggesting that hysteresis is also an important key to understanding friction laws of rough surfaces.

In this Chapter, we develop an in-situ technique for measuring adhesion hysteresis of an as-fabricated MEMS test structure, and report experimental values of hysteresis at low and high humidity levels for a film coated with perfluorotrichlorosilane ($C_8F_{17}C_2H_4SiCl_3$, FDTs), a monolayer lubricant. The work establishes an important element in the MEMS tribological

toolbox we are developing. The hysteresis measurements also allow us to determine the local uniformity of adhesion, as well as the effect of contact history on adhesion in MEMS. Furthermore, they are accomplished in a small area ($15 \times 2000 \text{ micron}^2$ for each measurement), important because of the expense of real-estate on a MEMS wafer.

II. Mechanics for measurement of adhesion in the presence of an externally applied load

1) Analytical equations

In our experiments we employ cantilever beams to study the adhesion due to interfacial forces. Adhesion hysteresis experiments require application of a load to heal and propagate cracks. We provide an externally applied load by means of electrostatic forces to move the crack, as in

Fig. 1. From our interfacial fracture mechanics perspective, the non-adhered

region from the cantilever support post to the point of attachment is considered to be a crack of length s . Note that the beam and landing pad are electrically grounded, such that only adhesive forces and no electrostatic forces operate in the contact zone d . Voltage V_{pad} is applied to the actuation pad far away from the crack tip. Increasing V_{pad} heals the cracks, while decreasing V_{pad}

Fig. 1 A cantilever beam subject to combined interfacial and electrostatic forces.

propagates the cracks, corresponding to advancing and receding contacts.

Fig. 2 Adhered cantilever beam with uniform distributed load q applied for development of the analytical model

We first develop an analytical model to gain insight into the mechanics of this geometry. Consider as in Fig. 2 a cantilever beam of length L , thickness t and height h adhered to the substrate over a distance $d=L-s$, subject to an externally applied uniform distributed load q . For $q=0$, with the JKR assumption [13] (adhesion forces

only in the contact zone over the attachment length d as represented by the cross-hatches), assuming free interfacial slip, an elastically rigid support post and a rigid substrate, its deflection profile is [14]

$$v(x) = h \left[3 \left(\frac{x}{s} \right)^2 - 2 \left(\frac{x}{s} \right)^3 \right]. \quad (1)$$

This applies for S-shaped beams such that the slope of the beam at the point of contact with the substrate is zero [14]. The effective energy of adhesion Γ (J/m^2) of the beam to the substrate can be measured when the energy release rate in the absence of an applied external load, G_o , has reached a static equilibrium. Then

$$G_o = \frac{18Dh^2}{s^4} = \Gamma \quad (2)$$

where $D = EI/w = Et^3/12$ (E is Young's Modulus, w is the width of the beam and $I = wt^3/12$ is the moment of inertia of the beam, and t is the thickness).

If now we apply q over the interval $0 \leq x \leq a$ as in Fig. 2, the crack length s will tend to decrease. To determine adhesion in the presence of an external load q , we must first calculate the deflection curves of the beams for a given crack length. Knowing this, we can then find the

Fig. 3 Deflections for increasing values of q and a constant value of $s=1000 \mu\text{m}$. ($q - \text{N/m}^2$)

energy release rate in the presence of the applied load q , G_q . The important results are outlined next, and the details are given in the appendix.

The deflection curves for $v_1(x)$ over the intervals $0 \leq x \leq a$ per Eq. (A1) (see the appendix) and for $v_2(x)$ over the interval $a \leq x \leq L$ per Eq. (A2) are shown in Fig. 3 for a fixed value of s and decreasing Γ (J/m^2). Here we choose

$a=60 \mu\text{m}$, $t=h=2 \mu\text{m}$, Young's Modulus $E=160 \text{ GPa}$ [15], hold $s=1000 \mu\text{m}$ constant and increase q . These values are typical for micromachined polycrystalline silicon (polysilicon). (Note that downwards corresponds to a positive deflection according to standard beam theory conventions). In Fig. 3 as q increases, the deflections increase and the adhesion Γ decreases because s is held constant. The value for q_{max} corresponds to $\Gamma=0$, as discussed below.

Per Eq. (A12), the result for G_q is

$$G_q = G_o + \frac{a^6 q^2 (4s - 3a)^2}{288 s^4 D} - \frac{qa^3 h}{144 s^4} (288s - 216a), \quad (3)$$

Fig. 4 Dependence of crack length s on adhesion Γ in the presence of an externally applied uniformly distributed load q .

where $D=Et^3/12$ is the beam bending stiffness.

Inserting the numbers above, $G_o=7.68 \mu\text{J}/\text{m}^2$ (i.e., microjoules per square meter) from Eq. (2), while $G_q=1.67 \mu\text{J}/\text{m}^2$ with $q=10 \text{ kN}/\text{m}^2$ from Eq. (3).

Referring to Fig. 4, in which s is plotted versus q using Eq. (3) for different values of Γ and assuming a , t , h and E as above, we see that crack length is very sensitive to Γ for small values of q , but that the sensitivity diminishes as q grows. From Fig. 3 however, we see that the adhesion is sensitive not only to the crack length but also to the

beam deflection profile. We take advantage of this feature in our experiments by measuring point by point beam deflection profiles, so that even at high applied loads our sensitivity to adhesion is significantly better than Fig. 4 would imply. Nonetheless, the adhesion resolution depends on crack length as well as applied force. Assuming 10 nm out of plane resolution from the interferometry, at $q=10 \text{ kN}/\text{m}^2$ and $s=1665 \mu\text{m}$, the resolution is approximately $0.03 \mu\text{J}/\text{m}^2$, while at $q=70 \text{ kN}/\text{m}^2$ and $s=300 \mu\text{m}$, the deflections are dominated by the applied force and the experimental resolution is about $20 \mu\text{J}/\text{m}^2$.

Eq. (3) applies for $G_q \geq 0$, or for

$$q \leq q_{\max} = \frac{72hD}{a^3(4s-3a)} \quad (4)$$

At $q = q_{\max}$, $G_q = 0$ and beam mechanics (ignoring adhesion) describe the deflections. As seen in Fig. 3, $d^2(v_2(s))/dx^2 = 0$ for $q = q_{\max}$. At the crack tip, the moment due to adhesive forces then vanishes, and a pure negative (upwards) shear force is imposed by the substrate. For $q > q_{\max}$ the deflections $v_{2q}(x)$ penetrate the substrate and therefore no longer apply. If in fact the adhesion energy were negative (corresponding to repulsive, e.g., magnetic forces), an additional negative (counterclockwise) moment can be added to the constants of integration (Eq. A3) to ensure that

that the deflections remain avoid dipping into the substrate.

With the JKR assumption of adhesion forces operating only over the contact zone, the beam theory equations reveal a point reaction and moment at $x=s$ which are imposed by the rigid substrate. Beyond the crack tip ($x > s$), the beam deflection is constant at $v(x)=h$, and there is no loading over the attachment length d for $x > s$. The reaction force applied by the substrate at the crack tip is equal and opposite to the shear

Fig. 5 Reaction force P_{tip} near the crack tip for a 10 μm wide beam

force $V(s)$ and is given by

$$V(s) = -D \frac{d^3}{dx^3} (v_2(s)) = -\frac{qa^3}{s^2} + \frac{(qa^4/2 + 12hD)}{s^3} \quad (5)$$

In Fig. 5, we plot this relationship. Here Γ is held constant, q is found with Eq. (3), and Eq. (5) is then applied to find the reaction force $P_{tip} = wV(s)$ (calculated for the experimental beam width

$w=10\text{ }\mu\text{m}$). We see that for small values of q , the reaction force is tensile and increases with Γ because there is more tension at the crack tip for larger adhesion. For large values of q , the force becomes compressive, because q now dominates over adhesion. The shear force diagram in the vicinity of the crack tip is shown in Fig. 6(a).

Fig. 6 (a) Shear force loading diagram under (a) JKR assumption, (b) schematic elastic solution with surface force law at the crack tip

The loading situation as shown in Fig. 6(a), in which the compressive force is maximized very near the crack tip, is quite different from that in surface force apparatus adhesion hysteresis experiments on crossed cylinders, in which the compressive force is maximized at the center of the contact zone. We shall see that this compressive loading near the crack tip can cause an adhesion increase during crack healing experiments, a signature that is not observed in spherical geometry adhesion hysteresis experiments.

Besides the JKR approximation, another well-known model for adhesion in contacting bodies is that of Derjaguin, Muller and Toporov [16], often referred to as the DMT model. In the DMT approximation, surface forces only outside of the areas of contact are considered in calculating adhesion, but these forces have no effect on the elastic deflections of the bodies. This approximation tends to be valid when the bodies are elastically hard [17]. For the necessarily compliant beams in this study, the DMT approximation is not physical. If we assume that surface forces act outside the contact zone, then only one shape is possible in order to balance forces. This shape can be solved for by iterative calculation, as described in the next section.

2) Finite element modeling

While the analytical model provides helpful insight, it is inadequate for the quantitative reduction of our experimental data. There are several reasons for this. First, in Fig. 1, the distributed load applied by electrostatic forces is not uniform, because the gap between the beam

and the substrate changes as a function of distance x from the support post. The electrostatic force at each point along the actuation pad is

$$q(x) = \frac{\epsilon_o}{2} \left(\frac{V_{pad}}{h - v_1(x)} \right)^2, \quad (6)$$

where V_{pad} is the applied voltage and ϵ_o is the permittivity of air. In this more complicated situation, an iterative solution is required to solve for the local applied forces and deflections. The solutions were found using the ABAQUS [18] finite element method (FEM) code, with the electrostatic force law implemented by means of a subroutine. Note that in Figs. 4 and 5, the x -axis is plotted in two ways. The lower x -axis corresponds approximately to the applied voltage in our experiments below. The correspondence is not exact because the load is not uniform (the correspondence was calculated at $x=55 \mu\text{m}$) and also because the adhesion affects the deflections, which in turn affect the force. The second reason FEM analysis is required to reduce the data is that non-idealities such as support post compliance and fringing fields at the edges of the beam could also be incorporated into the FEM modeling, as discussed further in the results section. The third reason is that FEM allows incorporation of surface forces near the crack tip, as discussed in the next paragraphs.

The adhesion was calculated by FEM in two ways. First, clamping the beam to the substrate at small increments of s , we checked that the FEM modeling agreed with the analytical results. Second, a force law between two smooth surfaces which can approach to within an approach distance D_o was assumed. While this is a reasonable approximation for our surfaces in most cases as shown below, the details of the effect of the roughness on local adhesion deserve further consideration beyond the scope of this work. Also, we shall not consider here the details of the stress field at the crack tip, such as the mode mixity and its relationship to interfacial slip, or its interplay with roughness. We implemented the surface force law in a general fashion, with integration of the force per unit area from D_o to infinity defining the surface energy. D_o is established by the roughness of the surfaces as seen below. A simple ramp force law over an effective range of 5 nm was used for most of the data reduction. Other more physical force laws such as a $(1/\text{distance})^3$ dependence (corresponding to van der Waals forces for two parallel plates) were also investigated but gave identical results as long as the active distance was

approximately the same. The deflection of the beam was then calculated as a competition between elastic restoring forces in the beam, and surface forces at the crack tip. When coupled with the experimental data, the FEM allows us to accurately evaluate adhesion energies.

One effect of the surface forces at the crack tip is to cause further flexure of the adhered beam compared to the JKR model. Thus, for a given surface force law, the FEM deflections are accurate within the limitations of the element size (we used 1-D beam elements every $2.5\text{ }\mu\text{m}$, and checked for good convergence with 2-D mesh elements and more closely spaced elements down to $0.5\text{ }\mu\text{m}$). In ref. [19], we shall detail the differences in the flexures when the surface force laws are incorporated. The main result is that for small and large q , the deflections are very similar, but for intermediate q and relatively large values of Γ , there is a small difference because of the long distance over which the beam is close to the substrate, as can be seen in Fig. 3. A second effect is that implementing the surface force law more closely reflects the real tractions in the crack tip area than does the JKR assumption. As seen in the schematic Fig. 5(b), surface force tractions reduce the beam shear force to the left of the crack tip. Also, by going beyond simple beam theory, a distributed pressure reaction force rather than a point reaction force exists beyond the crack tip in the attachment region $s \leq x \leq L$. There is a peak in the pressure near the crack tip, and for $x > L + t$, the reaction is equal to the pressure due to the surface forces. To solve accurately for the reactive pressure in the substrate requires a highly refined mesh in that regime.

III. Experimental

We fabricated polycrystalline silicon (polysilicon) cantilever beam test structures with an actuation pad near the support post. Their fabrication is described in detail in ref. [20], and a side view is shown schematically in Fig. 1. The thickness of the beams was $2.00\text{ }\mu\text{m}$ and their height h was $2.28\text{ }\mu\text{m}$. The length of the actuation pad in Fig. 1 is $50\text{ }\mu\text{m}$, beginning at $13\text{ }\mu\text{m}$ and ending at $63\text{ }\mu\text{m}$ from the support post. The rms roughness of the polysilicon is approximately 2.5 nm with approximately a $0.5\text{ }\mu\text{m}$ distance between local asperities, as measured by atomic force microscopy [21]. Before the cantilevers are rendered free standing, they are encased in a matrix of silica as a result of the fabrication process. The samples are then dipped in hydrofluoric acid, which removes the silica but does not perturb the polysilicon. After transferring from HF to water and through a series of miscible solvents, they were coated with

perfluorodecyltrichlorosilane (FDTS, $C_{10}H_{17}F_{17}SiCl_3$) in isooctane according to the procedure described in ref. [5]. FDTS is a silane coupling agent which nominally is deposited in monolayer form, with hydrophobic fluorinated tail groups remaining exposed [22]. However, in octadecyltrichlorosilane (ODTS, $C_{18}H_{37}SiCl_3$), a better studied monolayer lubricant, questions remain about the nature of the bonding of the silicon atom to the substrate (covalent versus hydrogen bonded) [23]. Also, the degree of polymeric cross linking between siloxane bonds in the interface may be limited by the strain resulting from the steric constraint that the hydrocarbon chain van der Waals radius is greater than the siloxane bond length [24]. The cross linking in FDTS may be further limited, because the fluorocarbon tail diameter is larger than the hydrocarbon diameter.

After the FDTS treatment, the structures were transferred from the solvents in reverse order to water. Because the contact angle of the treated surface with water is greater than 90° (115° was measured), capillary action does not pull the beams into contact with the substrate when removed from water. Using interferometry, we confirmed that the cantilevers were free standing after drying up to the maximum fabricated length of $2000\ \mu\text{m}$. This implies that the coverage of the FDTS film was very good. The freestanding cantilevers were curved up very slightly due to a residual stress gradient through the thickness of the film, with a radius of curvature of approximately $1.6\ \text{m}$, as measured by interferometry. This corresponds to an out-of-plane displacement of $300\ \text{nm}$ for a $1000\ \mu\text{m}$ long beam.

To measure adhesion hysteresis and its dependence on relative humidity (RH), an environmental microprobing station was constructed, schematically represented in Fig. 7. A Michelson interferometer is attached to a 5X microscope objective, and enclosed by a latex membrane in a humidity controlled environment. Magnification including the eyepiece is 50X, and resolution is approximately $3\ \mu\text{m}$ in the plane of the sample. The samples were illuminated by monochromatic green light at $547\ \text{nm}$ as characterized by spectrum photometry. Tilt of the reference surface in the Michelson interferometer was arranged such that background fringes on the substrate were parallel to the long axis of the cantilever beams. Then, fringes on the cantilever beams directly indicate out-of-plane displacements. Interferograms were recorded on a computer controlled charged coupled device (CCD) camera.

Humidity was introduced by flowing N_2 gas through a stainless steel water vessel heated to 50°C . Relative humidity (RH) was monitored using an in-situ humidity probe. Electrical

Fig. 7 Environmental microprober

current between the actuation pad and ground was monitored on a picoammeter. The current generally remained in the pA regime up to 80% RH. There was no evidence of electrostatic breakdown between the actuation pad and the beams in spite of the high electric field (0.8 MV/cm) in the gap, consistent with the observations in ref. [25]. Even at 160 V and 99% humidity, the current did not exceed 100 nA. The mechanism for the current flow is likely surface conduction between the actuation

pad and the landing pad or the support posts.

On one sample, tests were conducted at RH=0%, and subjected to thousands of actuation cycles. No qualitative effect on the adhesion or adhesion hysteresis was observed. However, after increasing the RH, after several hours an increase in adhesion was noted at 90% RH. Another sample was then inserted and this phenomenon was investigated more carefully. This sample was first tested in a dry nitrogen (RH=0%) ambient, while being viewed *in-situ* under interferometric conditions. Initially and at timed intervals of 1/2, 4, 7 and 10 hours thereafter, V_{pad} was stepped through values of 0, 50, 100, 120, 140, and 160 V for “crack healing” and subsequently through 140, 120, 100, 50 and 0 V for “crack propagation”. During the voltage stepping, the time between images was approximately 30 seconds. At 160 V, the distributed force is large and the crack lengths are about 300 μm long. This sequence was repeated at increasing RH levels of 0, 30, 60, 80, 90 and 95%. At each value of V_{pad} , an interferometric image was recorded and stored for future analysis. From the stored interferograms, linescans along individual beams were extracted using an image analyzer program [26]. These contain fringe information representative of out-of-plane $v(x)$ deflections, and were interpolated to nm-scale deflection data along the length of the beam by a computer program.

IV. Results

The initially suspended beams were brought into contact with the substrate by applying voltage to the actuation pad at RH=0%, and were then adhered with crack lengths $s \sim 1400 \mu\text{m}$ after the voltage was relaxed, corresponding to an adhesion of $2.4 \mu\text{J}/\text{m}^2$. The voltage step cycles described above were then begun. Comparing the measurement at $V_{\text{pad}}=0 \text{ V}$ before and after a given voltage stepping cycle, adhesion of the beams was not observed to change significantly from 0 to 80% RH. However, after four hours exposure at 90% RH, an adhesion increase to $4 \mu\text{J}/\text{m}^2$ was noted, and a significant increase to as large as $400 \mu\text{J}/\text{m}^2$ was observed after a seven hour exposure [21]. We shall focus the present adhesion hysteresis analysis on the cycle taken initially at RH=0%, and on the cycle at RH=90% after the seven hour exposure. As discussed in ref. [21], beams coated with ODTS showed only a very small adhesion increase due to exposure to humidity levels up to 99%.

There was no apparent difference in interferograms of the cantilevers at increasing and decreasing values of voltage at RH=0%. This is a qualitative indication that at this low RH value, there is no adhesion hysteresis. However, even when there is no hysteresis, adhesion changes along the length of the beam should be evaluated to determine the degree of adhesion non-uniformity due either to surface roughness or FOTS coating non-uniformities. Interferograms of the cantilever beam array exposed for 7 hours at 90% RH show significant adhesion hysteresis, as seen in Fig. 8. Comparing fringe patterns at 100 V, the fringes end closer to the support posts for the decreasing voltage than for the increasing voltage. The effect is much more pronounced at 50 V and 0 V, qualitatively indicating strong hysteresis. The beams in Fig. 8 are configured in an array of increasing length from bottom to top. This is to help in the assessment of the release process to create the free standing beams. Here, we measure adhesion only on only those beams which are attached over a long length d , per. ref. [14].

A few comments on the experimental observations deserve mention: (1) The results at high RH were qualitatively the same on the sample which was subjected to many contact cycles at low and the sample which was not. Cantilever beam arrays which were exposed to high RH, but had not been actuated, were tested at high RH and showed an adhesion increase similar to

Fig. 8 Interferograms of adhesion hysteresis observed at RH=90% after 7 hour exposure. The images are compressed by a factor of 3 in the horizontal direction relative to the vertical. The beams run horizontal, are 10 μm wide, and spaced 5 μm apart. Their lengths are from 1500 μm (bottom of each image) to 2000 μm (top). Beams longer than 1700 μm are cut off because of the CCD camera field limit. The voltage was increased to 160 V before decreasing.

those which had been actuated many times. These observations indicate that the adhesion increase is primarily related to exposure of the surface to high RH, rather than to the cycling of the samples. This is supported by the atomic force microscope (AFM) images shown below. (2) Note that in Fig. 8 the behavior of individual beams is quite different, especially as voltage is relaxed. We believe that this is a signature of the non-uniform nature of the adhesion increase mechanism, as described in the discussion section. (3) Cantilever beam arrays which were exposed to high RH, but had not been actuated, were also tested at low RH. These displayed an adhesion very similar to beams which had not been exposed to high RH.

To quantify the adhesion along the length of the beams accurately, we found that it was important to incorporate several non-idealities into the modeling. These include (1) the compliance of the support post, which was very important in obtaining measurements at high actuation pad voltages. It was determined directly by measuring the difference between

Fig. 9(a) Measured vs. modeled deflection data during the "crack healing" process.

Fig. 9(b) Measured vs. modeled deflection data during "crack propagation"

deflections of 1000 μm long cantilever beams (not yet touching the substrate) with increasing actuation voltages applied, and comparing to varying modeled support post compliances until good agreement was found. Other non-idealities accounted for in the FEM calculations include (2) the initial curvature of the beams due to stress gradient (caused by non-uniform stress

Fig. 10 Average rms per pixel curves versus adhesion at applied voltages of 0V, (b) 100V and (c) 160 V. The adhesion is taken as the minimum in each curve.

through the thickness of the film [27]), (3) the electrostatic fringing field at the outside edges of the beams [28], and (4) a linear correction to account for a small degree of angular offset between the background fringe direction and the beam direction. With accurate characterization of these non-idealities, adhesion of beams contacting the substrate at various loads applied was extracted by minimizing the difference between modeled and measured $v(x)$ values at a given applied voltage, as described next.

In Figs. 9(a) and (b) we show measured and modeled deflections of an individual beam from Fig. 8 at increasing and decreasing voltages respectively. The modeled deflections represent an optimum fit to the data in which the only free parameter is the adhesion. The best fit is determined by choosing the minimum in average root mean square error per pixel between measured and modeled deflections as shown in Fig. 10(a)-(c). In these plots, we utilize the full length of the measured deflection curve for error minimization. The minimum error is on the order of uncertainty from the interferometry measurements (< 10 nm). This indicates that

the modeling is good and that the important physical mechanisms have been incorporated and quantified well. In Fig. 10, the depth of the minimum successively decreases with increasing applied voltage, as is to be expected from the discussion on sensitivity versus applied load. We shall discuss the fitting procedure and results in more detail in ref. [19].

Fig. 11 Adhesion vs. voltage for two beams at RH=0.

Using this methodology, we plot the results of the adhesion hysteresis measurements versus applied voltage in Fig. 11 for two beams at low RH and for four beams in Fig. 12 at high RH. (The beam numbers are counted from the top of the image). For the beams at low RH in Fig. 11, the adhesion is almost independent of applied voltage, indicating that the adhesion is uniform along the length of the beam, with values ranging from 0.5 to 3 $\mu\text{J}/\text{m}^2$. In fact, adhesion was observed to be in the range of 1-10 $\mu\text{J}/\text{m}^2$ and uniform along the beam length up to RH levels of 80%, indicating that the FDTs film is uniformly deposited and relatively inert to water adsorption up to these levels. The beams in Fig. 12 at 90% RH were chosen to examine the range of adhesion increase during the actuation cycle, from 30 to 400 $\mu\text{J}/\text{m}^2$. In Fig. 12, we see that the adhesion begins to increase with increasing voltage above 100 V. Although the uncertainty is relatively large for the values of voltage above 100 V ($\sim 20 \mu\text{J}/\text{m}^2$ at $V_{\text{app}}=160 \text{ V}$), the large difference between the low and high humidity cases of Figs. 11 and 12 indicates that the adhesion increase with increasing voltage is a real effect, and not an artifact of sensitivities in the adhesion extraction algorithm.

Fig. 12 Adhesion vs. V_{pad} for four beams at RH=90%, 7 hour exposure. (The beam numbers are counted from the top of the cantilever array of Fig. 8)

To begin to understand the physical mechanism for the adhesion increase, we compared the landing pad areas (see Fig. 1) of the samples which had been exposed to high RH to the landing pads of samples which had been kept in air (in RH generally $\leq 40\%$) by atomic force microscopy (AFM). A Digital Instruments (Santa Barbara, CA) Nanoscope IIIA was used with silicon nitride cantilevers. The AFM was conducted in air after the samples were removed from the high RH environment. Results are shown in Fig. 13, where a number of agglomeration sites (mounds) are seen for samples exposed to high RH (Fig. 13(b)). Such mounds were not observed in samples not exposed to high RH (Fig. 13(a)). The density of the mounds is approximately 0.1 to 0.2 per μm^2 , their heights are typically 10-25 nm, but can be as large as 65 nm, and the diameters are typically 500-1000 nm. A linescan of a relatively large mound is shown in Fig. 13(c). Smaller mounds may be present but are difficult to distinguish from the 2.5 nm rms substrate roughness. The presence of these mounds was reported in ref. [21], and their origin is discussed further below.

Fig. 13 (a) Landing pad roughness at low RH.
(b) agglomerated mounds of FDTS
after exposure to high RH.
(c) linescan across a large mound.

V. Discussion

No adhesion hysteresis is observed up to 80% RH.

It is clear from the experimental interferograms that there is minimal adhesion hysteresis for the FDTS-coated surfaces up to 80% RH. That is, these interferograms look identical for increasing and decreasing voltages. Yet, hydrocarbon and fluorocarbon-tailed monolayer films deposited by the Langmuir-Blodgett technique exhibit adhesion hysteresis in surface force apparatus experiments [29,30], as well as in similar experiments between polydimethyl siloxane and fluoroalkylsiloxane coated mica [31]. The proposed mechanisms for the hysteresis are chain interdigitation when surfactant films are in an amorphous state [29], and molecular as well as submolecular changes in a fluorocarbon surfactant due to the bulky fluorocarbon groups [30]. Due to the atomically flat surfaces, the advancing contact (corresponding to crack healing) adhesion in those experiments is tens of mJ/m^2 (i.e., millijoules per square meter) while the

receding contact (corresponding to crack propagation) adhesion can reach values several times larger.

In the present experiments, the surfaces exhibit a 2.5 nm rms roughness as measured by AFM (atomic force microscopy). This is a fairly smooth surface, and one might expect that there is still sufficient contact area that adhesion hysteresis would manifest itself. Indeed, as mentioned in the mechanics sections, our experimental resolution with respect to adhesion hysteresis at low voltages is such that a difference of $0.03 \mu\text{J}/\text{m}^2$ between healing and propagating crack energies should be detectable, well beyond the resolution for adhesion hysteresis in the surface forces apparatus.

The theory of Fuller and Tabor [32], which uses the JKR assumption, is one way to calculate the expected adhesion and hence the expected adhesion hysteresis of rough surfaces. (A theory for the adhesion of rough surfaces with the DMT approximation has also been developed by Maugis [33]. However, this theory considers reversible van der Waals forces only near asperities in contact, and therefore would predict zero adhesion hysteresis). In the Fuller-Tabor theory, the force of adhesion depends on the true area of contact at a given applied load. By calculating the ratio of true to apparent area and then multiplying by the surface energy for advancing and receding contacts, we can determine what adhesion hysteresis value the JKR theory predicts for a given rough surface. In principle, the contact area is larger when the crack is propagated, because asperities which have been brought into contact during crack healing are then stretched. However, because of the relatively high modulus of the silicon and the small radius of curvature of the asperity tips ($R \sim 100 \text{ nm}$), this difference in contact areas is negligible for our surfaces.

To make first order calculations for what the adhesion hysteresis should be at $V=0 \text{ V}$ and $\text{RH}=0\%$, we must recognize that there is a self-loading due to van der Waals forces as predicted by the Hamaker equation for two flat surfaces. From the average measured adhesion energy of $2.4 \mu\text{J}/\text{m}^2$, the average separation D_o due to the surface roughness can be determined from

$$\Gamma = \frac{A}{12\pi D_o^2} \quad (7)$$

where A is the Hamaker constant. A ranges from 0.5×10^{-19} to 2×10^{-19} J for a variety of surfaces [34]. Using $A = 0.5 \times 10^{-19}$ for fluorocarbon material, we find $D_o = 23$ nm. From the derivative of Eq. (7), an average force due to the van der Waals interaction in the attachment region is $q = 200$ N/m². Carrying out the calculations according to the Fuller-Tabor theory, the real contact area is approximately only 2×10^{-8} of the apparent contact area. Assuming crack healing and propagation energies of 10 and 30 mJ/m², respectively, for areas which actually achieve intimate contact, an adhesion contribution from contacting asperities of 0.2×10^{-3} μJ/m² for healing cracks, and an adhesion hysteresis of 4×10^{-4} μJ/m² are expected.

Because these values are only ~0.01% of the measured adhesion, the assumption just made that van der Waals forces are the main contributors to the adhesion (e.g., Eq. (7)) is justified. Secondly, even if adhesion hysteresis exists, it would contribute $\sim 4 \times 10^{-4}$ μJ/m², a factor of 100 times beyond our experimental resolution at 0 V. Therefore, due to the large equilibrium separation between the surfaces of $D_o = 23$ nm, effectively only the reversible van der Waals forces are probed in our experiments, and indeed no adhesion hysteresis is expected at 0 V at low humidity. Rather than resorting to the mathematically complex theory of Fuller and Tabor, a much simpler theory, described by Eq. (7), applies for the adhesion of our micromachined surfaces. This is because the rms roughness is far smaller than the average separation between the surfaces. Said differently, only the highest asperity peaks control the average separation between our surfaces.

As discussed in the results section, there is also no apparent increase in adhesion at high values of voltage. Reasoning similar to that just described for $V = 0$ V can be used to explain this result. Although the expected values of adhesion hysteresis increase somewhat because the load very near the crack tip increases above the self-loading from van der Waals forces, the experimental resolution on adhesion decreases with shorter crack lengths. At the highest applied voltage of 160 V, our calculations show that the expected adhesion increase due to increasing pressure near the crack tip (e.g., Fig. 4) grows by ~50 times to ~ 0.02 μJ/m², but cannot be detected because at 160 V the experimental sensitivity decreases to approximately 20 μJ/m². The higher pressure near the crack tip decreases the average spacing D_o to ~15 nm from 23 nm, from which we would expect an increase in adhesion from 2.5 to 6 μJ/m² according to Eq. (7). This effect is also not detectable. From this we can conclude that at low RH, the

adhesion is constant to within $\sim 10 \mu\text{J}/\text{m}^2$ along the length of the beam. As the crack tip samples a relatively short zone on the order of a micron or less in length, variations in adhesion within this range can be attributed to local differences in roughness, which can affect the average separation between the surfaces.

Large adhesion hysteresis is observed at high RH

At high RH values, two major effects were observed. First, the adhesion hysteresis is large, with $\Delta\Gamma$ from 30 to $400 \mu\text{J}/\text{m}^2$ as seen in Fig. 12. Second, the adhesion increases both as the crack heals and then repropagates. The agglomerated mounds observed in Fig. 13 suggest that these observations correlate strongly with a structural change in the film. As discussed in ref. [21] and described briefly here, we believe the mounds are actually isolated vesicles formed by a restructuring of the surface monolayer phase to a lyotropic bulk phase at high humidities. Lyotropic phases form between surfactant molecules and water, and exhibit viscoelastic properties. (See for example ref. [35]). Essentially, very soft and large radius of curvature asperities have developed on the surface as a result of this restructuring at high RH. The vesicles in Fig. 13 are three dimensional, and their volume is approximately that of the original monolayer. Because this process occurs over the course of hours, we surmise that a diffusional process leading to a more stable bulk phase consisting of the vesicles has taken place. The vesicles are surrounded by lower lying areas which are likely hydrophilic because they are no longer covered by the coupling agent..

The vesicles themselves are likely hydrophobic, because if the adhesion increase at high RH were uniform along the length of the beams, the cracks would heal spontaneously. However, even at 90% and greater RH, this was not observed – crack lengths decreased only as a result of a pad actuation cycle. This is in striking contrast to the case of hydrophilic beams, where we observed spontaneously decreasing crack lengths with increasing humidity over a range from 30 to 95% RH [36]. In that case, nanometer size capillaries at the crack tip do work against the strain energy of the beam to heal the crack. Here, the protruding vesicles likely remain hydrophobic, while the areas which are uncovered become hydrophilic. We can think of the surface in Fig. 13 as hydrophobic islands rising above a hydrophilic sea. This leads to a non-uniform adhesion on the surface, with the scale of the non-uniformity being microns as defined by the distance between the vesicles. We now explain the main characteristics of the adhesion hysteresis curves for the four adhered beams at high RH shown in Fig. 12.

A) Crack healing (increasing voltage) at high RH

As V_{pad} is increased from 0 to 50 V in Fig. 12, Γ decreases from ~ 3 to $\sim 1 \mu\text{J}/\text{m}^2$.

Analysis of the data indicates that the crack length remains very nearly the same from 0 to 50 V, with the concomitant decrease in adhesion. The increase from 0 to 50 V provides only a small crack healing force. If adhesion were constant at $3 \mu\text{J}/\text{m}^2$, the crack would heal approximately 100 μm . This indicates that in the vicinity of the crack tip, surface forces are not locally available to do work on the beam. We can understand this in the following sense: if the protruding vesicles are hydrophobic, they will resist crack healing. As seen from Fig. 5, pressure which might compress the vesicles is very small (or tensile) at 50 V, and therefore it is difficult for the cracks to heal beyond the first vesicle encountered.

When pad voltage is increased to 100 V, sufficient driving force exists to force the cracks to heal some 200 μm , and the apparent Γ sometimes increases and sometimes decreases relative to the values at 0 and 50 V. If the crack tip reaches an area of locally high adhesion within the hydrophilic sea, surface forces will do more work on the beam to pull it further in, resulting in a larger value of adhesion. On the other hand, if it reaches a hydrophobic island it does not heal further, a lower value of adhesion is inferred. Note from Fig. 5 that the compressive force applied near the crack tip remains small at $V_{\text{pad}}=100$ V.

Γ increases monotonically as applied voltage is increased from 120 V up to 160 V. At higher voltages, compression near the crack tip may act to deform the compressible viscoelastic vesicles seen in Fig. 13(b). This would decrease the separation between the surfaces in the vicinity of the crack tip, allowing lower lying hydrophilic areas to come into contact, and causing the monotonically increasing adhesion with increasing voltage.

We can test this idea quantitatively. Recalling the discussion for Fig. 5, the magnitude of force at the crack tip is on the order of hundreds of nanonewtons at the highest voltage. Near the crack tip, this load will be borne by an individual vesicle because as seen in Fig. 13 they are spaced microns apart. From simple Hertzian mechanics, the deformation of the vesicles will be

$$\Delta = \left(\frac{P_{\text{tip}}^2}{K^2 R} \right)^{1/3} \quad (8)$$

where P_{tip} is the compressive load applied by the beam to the vesicle, $K=(4/3)(E/(1-\nu^2))$ is proportional to the modulus of the vesicle, and R is the radius of curvature of the vesicle. At $V_{pad}=160$ V, the value of q from Eq. (5) will be approximately 70 kN/m^2 , resulting in $V(s)=-550$ nN per Fig. 5. The radius of curvature of the polymeric vesicles is $a_o^2/2\delta$, where a_o is the vesicle radius and δ is the vesicle height. From the results section, we have $a_o=0.2 \text{ }\mu\text{m}$ and $\delta=20 \text{ nm}$, resulting in $R=1 \text{ }\mu\text{m}$. Estimating $K=1 \text{ GPa}$ for the polymeric vesicles (typical polymers have modulus values of 1-3 GPa, but we might expect a more compliant structure here), and inserting these values into Eq. (8) results in $\Delta=6.7 \text{ nm}$. Furthermore, the contact area is $\pi R\delta$, and the average pressure will be 25 MPa. This is beyond the hardness of many polymeric materials. Because of this high pressure and because the vesicles are likely viscoelastic, subsequent deformation beyond $\Delta=6.7 \text{ nm}$ is to be expected. This brings to mind the notion that the adhesion is pressure sensitive in this system.

The above calculation supports the notion that the adhesion increase during crack healing is in fact due to increasing contact area under the applied load near the crack tip. Once the hydrophobic vesicles are sufficiently deformed, locally hydrophilic areas can bridge across the surfaces as seen in Fig. 14, which will subsequently significantly increase the measured

Fig. 14 Schematic of crack tip for FDTD surface after exposure to high RH

adhesion. The monotonic increase in adhesion with voltage from 120 to 160 V indicates that more sites come into contact as the load is increased. The result is strikingly different from adhesion hysteresis experiments for spheres come into contact, which exhibit adhesion increase only upon crack repropagation [29-31]. The reason is that in the case of two spheres coming into contact, the pressure maximum is at the center of the contact area, while in the present case the maximum is at the contact edge. The emergence of the low modulus hydrophobic islands in the hydrophilic sea at the high RH

levels, as well as the compression very near the crack tip, is the mechanism for the increase in adhesion during crack healing in the present work.

After prolonged exposures of 40 hours at RH=95%, adhesion was observed to continue to increase up to 0.5 to 1 mJ/m² [21]. Hydrophilic surfaces approach 140 mJ/m² at these humidity levels [36]. This maximum value for the hydrophobic surface can be understood in terms of Fig. 5. There we see that for large values of Γ , compressive force can no longer be generated in the vicinity of the crack tip at the maximum value of V_{pad} . Consequently, adhesion cannot increase beyond the mJ/m² range for the restructured surfaces in these experiments.

B) Crack propagation (decreasing voltage) at high RH

In Fig. 8 we see that the adhesion is non-uniform from one beam to the next at the end of the actuation cycle. We attribute this to a statistical distribution of the vesicles. If there is a locale in which locally fewer vesicles and more hydrophilic surface area is present, this will give rise to greater adhesion. This will be observed not only from beam to beam, but within a beam as well, and helps to explain the characteristics during crack propagation.

As V_{pad} is lowered from 160 V, the adhesion initially decreases or remains constant as seen in Fig. 12. Analysis of the deflection curves shows that the crack propagates in this region, indicating there is sufficient restoring force in the highly deformed beam to move past regions of high adhesion. However, as voltage decreases to 100 V or less, examination of the FEM results indicates that crack propagation ceases. The crack tip, which samples the adhesion continuously along the length of the crack during crack propagation, stops propagating at a site of high adhesion energy. The pinning of the crack gives rise to an apparent increasing adhesion as the applied voltage continues to decrease. This explains why the energies appear to increase as V_{pad} is lowered below 100 V. Also, at $V_{\text{pad}}=0$ V, the apparent adhesion value is a lower bound. The exception is for beam 10 (Fig. 12c), which did not propagate at all as V_{pad} was lowered from 160 V. This beam exhibited the highest adhesion of the four beams in Fig. 12. For this beam, the crack tip probed a site of high local adhesion at $V_{\text{pad}}=160$ V.

The observation that beams exposed to high RH, and tested at low RH showed no adhesion increase is consistent with the picture of the hydrophilic sea. Namely, at low RH, the hydrophilic areas will no longer be wetted, and therefore will not act to increase the adhesion.

VI. Summary and Conclusions

We have developed the mechanics to investigate adhesion hysteresis of cantilever beams adhered to a substrate, both by an analytical approach as well as by finite element modeling (FEM). A key result of the mechanics is that for sufficiently high applied loads, a compressive load must be supported near the crack tip where the beam contacts the substrate. This is different from SFA adhesion hysteresis experiments in which the pressure is maximized away from the crack tip. From this we have some reason to suspect a different adhesion hysteresis characteristic than has previously been measured if the material near the crack tip is compressible.

A silane coupling agent (FDTS) monolayer was applied to the silicon microcantilevers in order to minimize susceptibility of adhesion to various RH ambients. To determine performance limits of the monolayer, multiple contact (up to tens of thousands of cycles) and increasing humidity experiments were conducted. The RH experiments showed a significant adhesion increase, and this effect was studied in detail. Adhesion was tested at RH levels increasing from 0 to 95%, with adhesion hysteresis test cycles conducted periodically up to 24 hours at a given RH level. FEM was required to quantify the adhesion at high loads because of the gap-dependent load calculation. Furthermore, FEM allowed us to take into account such non-idealities as the support post compliance and the fringing fields from the electrostatic loading. Also, we were able to incorporate more physical surface force laws than can be achieved with the JKR assumption.

At low RH values up to 80%, negligible adhesion increase with applied load, as well as negligible adhesion hysteresis was observed. This is explained by the average separation between the surface being large enough that reversible van der Waals forces dominate the adhesion. Adhesion values did not increase significantly with voltage because the contribution to adhesion of increasing real contact area is very small, and because the reduction in average surface separation is small.

A substantial change in the adhesion and adhesion hysteresis was noted after a seven hour exposure at 90% RH. The adhesion increase is observed only upon actuation of the beams, rather than by spontaneous crack healing. Atomic force microscopy indicated that the silane coupling agent surface phase reconfigured into a bulk phase consisting of ~20 nm high polymeric mounds, which we interpret to be vesicles. Formation of the vesicles leaves behind

locally hydrophilic areas, but the vesicles themselves probably are hydrophobic and protrude above the surface, leading to a picture in which hydrophobic islands are surrounded by a hydrophilic sea. FEM quantification revealed that an adhesion increase is observed upon crack healing at high applied loads, in contrast to SFA adhesion hysteresis measurements. By calculating the applied load near the crack tip, we showed using Hertzian mechanics that the polymeric vesicles deform significantly, allowing lower lying hydrophilic areas to come into contact. When this occurs, capillaries can bridge the hydrophilic areas of the two surfaces, and the adhesion increases. This indicates that the restructured surface displays a pressure sensitive adhesion characteristic.

We have demonstrated that adhesion hysteresis of cantilever beams is a powerful tool for understanding aspects of reliability in surface micromachining. These experiments have enabled us to connect mechanics and chemistry at the microscopic level, and indicate that if wetting of MEMS surfaces can be prevented, adhesion and adhesion hysteresis can remain reasonably low.

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VIII. Appendix

G in the presence of an externally applied distributed load q

Consider Fig. 1 in the main body of this Chapter. A cantilever beam of length L , thickness t and height h is adhered to the substrate over a distance $d=L-s$. If now a distributed load q is applied over the interval $0 \leq x \leq a$, the crack length s will decrease (if the adhesion is uniform along the length of the surface). In the absence of adhesion, simple beam mechanics can be used to determine where the beam will contact the substrate. However, because of the adhesion forces in the vicinity of the crack tip, the actual crack length s will be somewhat shorter. Knowing the value of s , we can determine the adhesion according to the following beam mechanics. We use the JKR assumption in these calculations.

Over the interval $0 \leq x \leq a$, the equation for the deflections $v_{1q}(x)$ is

$$\frac{d^4}{dx^4} [v_{1q}(x)] = \frac{q}{D} \quad (\text{A1a})$$

where $D=EI/w=Et^3/12$ (E is Young's Modulus, w is the width of the beam and $I=wt^3/12$ is the moment of inertia of the beam). Integrating four times, we have

$$v_{1q}(x) = \frac{1}{D} \left(\frac{1}{24} qx^4 + \frac{1}{6} C_{1q} x^3 + \frac{1}{2} C_{2q} x^2 + C_{3q} x + C_{4q} \right) \quad (\text{A1b})$$

Over the interval $a \leq x \leq L$, the equation for the deflections $v_{2q}(x)$ will be

$$\frac{d^4}{dx^4} [v_{2q}(x)] = 0 \quad (\text{A2a})$$

Integrating again four times,

$$v_{2q}(x) = \frac{1}{D} \left(\frac{1}{6} (qa + C_{1q}) x^3 + \frac{1}{2} C_{5q} x^2 + C_{6q} x + C_{7q} \right) \quad (\text{A2b})$$

Here we have used $qa + C_{1q}$ as the constant of integration in (A2) because the shear force in the beam at $x=a$ must be greater by qa than at $x=0$.

For rigid support post and substrate, the boundary conditions are:

$$v_{1q}(0) = 0, \frac{d}{dx} [v_{1q}(0)] = 0, v_{2q}(s) = h, \frac{d}{dx} [v_{2q}(s)] = 0. \quad (\text{A3a-d})$$

Continuity at a requires that

$$v_{1q}(a) = v_{2q}(a), \quad \frac{d}{dx}[v_{1q}(a)] = \frac{d}{dx}[v_{2q}(a)], \quad \text{and} \quad \frac{d^2}{dx^2}[v_{1q}(a)] = \frac{d^2}{dx^2}[v_{2q}(a)]. \quad (\text{A3e-g})$$

where the final boundary condition is because the moment cannot change instantaneously.

Solving for the constants of integration in terms of known quantities gives

$$C_{1q} = -qa + qa^3/s^2 - (qa^4/2 + 12hD)/s^3,$$

$$C_{2q} = qa^2/2 - (2qa^3)/(3s) + (qa^4/4 + 6hD)/s^2,$$

$$C_{3q} = C_{4q} = 0,$$

$$C_{5q} = (-8qa^3s + 3qa^4 + 72hD)/(12s^2),$$

$$C_{6q} = qa^3/6, \quad \text{and} \quad C_{7q} = -qa^4/24 \quad (\text{A4})$$

Inserting these values into (A1b) and (A2b), choosing $a=60 \mu\text{m}$, $t=h=2 \mu\text{m}$, $s=1000 \mu\text{m}$, $E=160 \text{ GPa}$, and various values of q , we see the deflections in Fig. 2 in the main body. As expected, Eqs. (A1) and (A2) agree with Eq. (1) from the main body for $q=0$.

In the absence of external force, the adhesion Γ is as according to Eq. (2). To find the energy release rate G_q in the presence of the external force, we must recognize that there are contributions both from work performed by external forces F , as well as from elastic energy U_E . Therefore,

$$G_q = \frac{dF}{ds} - \frac{dU_E}{ds} \quad (\text{A5})$$

The incremental work performed per unit width of the beam is

$$\Delta F = q a \overline{\Delta v_1} \quad (A6)$$

where $\overline{\Delta v_1}$ is the average differential displacement over the interval $0 \leq x \leq a$, is defined by

$$\overline{\Delta v_1} = \frac{1}{a} \int_0^a [v_1(x, s + \Delta s) - v_1(x, s)] dx \quad (A7)$$

In the limit where $\Delta s \rightarrow 0$,

$$\frac{dF}{ds} = \frac{q^2 a^6}{144 D s^4} (4s - 3a)^2 + \frac{q a^3 h}{144 s^4} (216a - 288s) \quad (A8)$$

The strain energy per unit width of the beam is

$$U_E = U_{E1} + U_{E2} = \frac{1}{2D} \left[\int_0^a (M_1(x))^2 dx + \int_a^L (M_2(x))^2 dx \right] \quad (A9)$$

where $M_n(x) = -D \frac{d^2}{dx^2} [v_n(x)]$. Solution to (A9) yields

$$U_E = \frac{-80a^6 s^2 q^2 + 60a^7 s q^2 - 15q^2 a^8 + 8640h^2 D^2 + 36q^2 s^3 a^5}{1440s^3 D} \quad (A10)$$

The strain energy release rate term reduces to

$$-\frac{d}{ds} U_E = \frac{18Dh^2}{s^4} - \frac{a^6 q^2 (4s - 3a)^2}{288 s^4 D} = G_o - \frac{a^6 q^2 (4s - 3a)^2}{288 s^4 D}, \quad (A11)$$

where G_o is as defined by Eq. (2). Substitution of (A11) and (A8) into (A5) gives

$$G_q = G_o + \frac{a^6 q^2 (4s - 3a)^2}{288 s^4 D} - \frac{q a^3 h}{144 s^4} (288s - 216a) \quad (A12)$$

For the present example, $G_o = 7.68 \mu\text{J}/\text{m}^2$, while $G_q = 4.16 \mu\text{J}/\text{m}^2$ with $q = 5 \text{ kN}/\text{m}^2$. Eq. (A12) is plotted in Fig. A1 (below) for decreasing values of s .

The solution (A12) applies for $G_q \geq 0$, or for

$$q \leq q_{\max} = \frac{72hD}{a^3(4s - 3a)} \quad (A13)$$

The insight gained from this analysis is described in the main body of this Chapter.

Fig. A1, $G_q (=G_{Tot})$ vs q

IX. References

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5. Chemical vapor deposition of fluoroalkylsiloxane monolayer films for adhesion control in microelectromechanical systems (Mayer, de Boer and Michalske)

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Abstract

We have developed an alternate method for coating MEMS devices with a hydrophobic fluorocarbon film, which avoids many of the problems associated with solution-based reaction of alkylchlorosilanes. In this process we use a volatile fluoroalkylsilane as precursor for a chemical vapor deposition (CVD) process. In a vacuum chamber, controlled amounts of silane precursor and water vapor react on the surface of the sample to form silanols, and eventually siloxane linkages, with a fluoroalkyl side chain to provide a hydrophobic, low energy surface. Vapor phase coating processes have a number of distinct advantages compared to solution-based processes. We avoid a number of problems associated with solution-based coating, including wetting of high aspect ratio structures, diffusion limited transport of reagents into confined areas, control of minute quantities of dissolved water in nonaqueous solvents, slow reaction kinetics of chlorosilanes in solution, and disposal of organic solvent waste. Vapor phase process provide efficient transport into high-aspect ratio structures, provide good control of reagent supply, have no solvent waste, and provide for convenient in-situ cleaning of surfaces immediately prior to deposition. This CVD process does require that mechanical parts be released and dried prior to introduction to the deposition chamber, so that sacrificial oxide etching must be followed by a supercritical CO₂ or sublimation drying procedure to produced free, released parts.

I. Introduction

In this Chapter we present an outline of the coating process and reactor, details of an in-situ plasma cleaning process we have incorporated, in-situ ellipsometric measurements of the film deposition, and characterization of the film by contact angle measurements, atomic force microscopy, and preliminary adhesion measurements using a cantilever beam test structure.

II. Experimental

The chemical vapor deposition apparatus is shown schematically in Fig. 1. It is a simple vacuum system; pumped by a turbomolecular pump and a mechanical roughing pump. The fluoroalkylsilane and water precursors enter the reactor through high precision leak valves from separate reservoirs. Also included is a microwave cavity surrounding a quartz flow tube, which

Fig. 1 Apparatus for chemical vapor deposition of fluoroalkylsiloxane films, including in-situ spectroscopic ellipsometer

can be used to excite a gas for cleaning substrates prior to the film coating. The wafer susceptor contains a resistive heater, which allows heating to $>400^{\circ}\text{C}$. Windows are also included for in-situ ellipsometric measurements of the sample during processing.

The fluoroalkylsilane precursor used in this study is 1,1,2,2 tetrahydrofluorooctyltrichlorosilane ($\text{CF}_3(\text{CF}_2)_5(\text{CH}_2)_2\text{SiCl}_3$), which we label FOTS. This compound has a vapor pressure of approx. 0.3 torr at room temperature, and is thus convenient for use as a CVD precursor. We have also investigated the 10-carbon chain analogue, however its significantly lower vapor pressure leads to considerably longer process time. The silane precursor is vacuum distilled prior to use.

Deionized and ultra filtered water with resistance of $>18\text{ M}\Omega$ is used. The water is outgassed on the vacuum system prior to use. Both precursor reservoirs are maintained at room temperature.

Flat Si test samples as well as cantilever beam samples obtained from the MDL had previously undergone release etch, reoxidation in H_2O_2 , and supercritical CO_2 drying. In-situ sample cleaning is accomplished using a downstream microwave discharge in O_2 or H_2O vapor. After evacuation and prior to coating, a discharge is ignited in flowing O_2 or H_2O at approx. 0.1 – 0.3 torr, at 30 W forward power. The effectiveness of the cleaning process is measured by observing the water contact angle on flat samples before and after cleaning. After the supercritical CO_2 drying process parts are initially somewhat hydrophobic, with contact angle of $60 - 70^{\circ}$. Separate x-ray photoelectron spectroscopic measurements have confirmed that parts typically have a high level of carbon-containing contamination. As shown in Fig. 2, cleaning in H_2O vapor discharge for >10 min. results in a surface with a contact angle of $<5^{\circ}$ with no measurable carbon contamination. Similar results are obtained for cleaning in O_2 discharges. Although these results are given for flat surfaces with line-of-sight exposure to the discharge flux, we assume that the underside of mechanical structures and confined areas of high aspect ratio structures also receive sufficient reactive flux to remove organic contaminants. Later test measurements on cantilever beam structures will show this assumption to be warranted. This cleaning process then provides us with a clean, hydrophilic surface on which to deposit the fluoroalkylsilane film.

The film coating process then begins by closing the pumping valve and introducing FOTS to a static gas pressure of approx. 0.15 torr. (This takes about 10 minutes because of the low vapor pressure of the FOTS, and small conductance of the leak valve used.) Upon reaching the desired

Fig. 2 Contact angles measured on SCCO₂ dried SiO₂ surfaces after microwave plasma cleaning in H₂O vapor discharge

pressure, the leak valve is closed, and the H₂O leak valve is opened. Water vapor is introduced to a total pressure in the chamber of 0.6 – 0.8 torr, when the H₂O valve is closed. The gases are then left to react with the wafer surface under static conditions for approx. 15 minutes. Growth of the film during this time can be monitored by in-situ ellipsometry, shown in Fig. 3. Initial adsorption of the FOTS to the surface is confirmed by a small decrease in the ellipsometric measurable, Δ , however coverage is low, corresponding to an equivalent film thickness of 0.1 – 0.2 nm. Moreover, this layer is not tightly bound to the surface, and will desorb upon pumping away the FOTS precursor. A stable film is formed only after introduction of water vapor, also shown in Fig. 3. The reaction is reasonably rapid and seems to terminate at an equivalent film thickness of approx. 1 nm. Repeated cycles of FOTS and H₂O exposure result in only minor additional film growth. A fully dense, ordered, two-dimensional FOTS film with extended alkyl chains should have a thickness of approx. 1.2 nm, so it appears that the limiting thickness of FOTS film formed in this process corresponds to a reasonably well packed monolayer film. We believe the self-limiting behavior is caused by the inability of the precursor molecules to stick to the fluoroalkyl-terminated surface, giving self-limiting film growth.

Fig. 3 In-situ ellipsometric measurement of FOTS film growth at room temperature, showing sequential cycles of FOTS and H₂O vapor exposure

III. Results

Film thickness and water contact angle as a function of FOTS + H₂O reaction time and deposition temperature are shown in Figs. 4 and 5. All films are hydrophobic, with contact angle >100°. For a given reaction time, films formed at room temperature are thicker and have higher contact angle. This is presumed to be a result of decreased sticking probability of the precursors with the surface at elevated temperature. Atomic force microscopy measurements of these films show an apparently continuous film, with no obvious indication of gross defects or partial surface coverage. RMS surface roughness of the films is typically approx. 2.5 nm over a field of view of 10 micrometer, which is also characteristic of the initial substrate roughness. In short, the films appear continuous, reasonably dense, and hydrophobic.

Fig. 4 Thickness and contact angle for FOTS films grown at room temperature as a function of deposition time.

Fig. 5 Film thickness and contact angle for FOTS films grown at various temperatures. Deposition time was 30 minutes

Fig. 6 (a) Uncoated beams after exposure to >90% humidity for >24 hr. All beams are adhered along their entire length. (b) FOTS coated beams after exposure to >90% humidity for >24 hr. Most beams are stuck only at the tip.

Adhesive properties of surfaces coated by these films have been measured using the cantilever beam adhesion test structure described earlier. Adhesion of coated and cleaned, but uncoated, beams was measured at ambient humidity levels of 30 – 90% relative humidity (RH) at room temperature. At low RH, neither coated nor uncoated beams adhere upon bringing them into

contact. (This is typical behavior for this batch of beam test structures.) The humidity was then increased and the beams repeatedly brought into contact over an extended period of time. After exposure to increasing humidity levels up to 94% over a period of three days, the uncoated beams are nearly all tightly adhered, shown in Fig. 6a. The coated beams did not adhere, however, even after this high humidity, long time exposure, shown in Fig. 6b. AFM measurements after high humidity, long-time exposure also revealed no obvious film degradation.

IV. Summary

In summary, we have developed an alternate fluoroalkylsilane coating process, based on a chemical vapor deposition approach, which yields monolayer, hydrophobic films. Growth is self-limiting at approximately 1 monolayer, and films display superior adhesion and stability properties in high humidity environments. An in-situ surface cleaning process has also been incorporated into the deposition process to ensure clean, hydrophilic surfaces prior to coating with the fluoroalkylsilane film.

6. A hinged-pad test structure for sliding friction measurement in micromachining

(de Boer, Redmond and Michalske)

Abstract

We describe the design, modeling, fabrication and initial testing of a new test structure for friction measurement in MEMS. The device consists of a cantilevered forked beam and a friction pad attached via a hinge. Compared to previous test structures, the proposed structure can measure friction over much larger pressure ranges, yet occupies ten to one hundred times less area. The placement of the hinge is crucial to obtaining a well-known and constant pressure distribution in the device. Static deflections on the device were measured and modeled numerically. Preliminary results indicate that friction pad slip is sensitive to friction pad normal force.

1. Introduction

In Microelectromechanical Systems (MEMS), surface forces can play a relatively large role compared to gravity and inertia, which are the dominating forces at the macroscale. Because surface forces are not well characterized and often difficult to reproduce in MEMS structures, most commercial applications avoid contact between structural members. Examples include accelerometers and gyroscopes [1-3]. An important commercial device in which *adhesive contact* between moving structures and the substrate is allowed is the Digital Mirror Device [4]. For this product, adhesion (e.g., stiction) and adhesion hysteresis (changes in adhesion over time or due to environment) are important factors in determining device reliability. While great reliability improvement has necessarily been achieved through testing of full scale devices [5], these surface properties can also be studied through the use of test structures [6-8]. From such studies, we hope to understand and control surface forces in micromachine structures, which may permit us to take advantage of surface characteristics in micromachine design.

If *sliding contact* between structural members is allowed, a large new class of devices can be brought to product realization in MEMS. Examples include gears for mirror rotation [9], pop-up mirrors and linear racks [10], and shuffle motors [11]. However, relatively little is understood regarding the friction laws of such devices in contact, such as how friction may scale with

apparent pressure[†] or sliding velocity. These parameters can vary by orders of magnitude: 0.1 to 100 MPa in apparent pressure and 0.1 to 1000 $\mu\text{m}/\text{sec}$ in velocity, depending on the application. In larger scale devices, friction can depend strongly on these factors [12]. Information on friction of horizontal surfaces in MEMS has been obtained using reciprocating comb drive structures [13- 14]. According to these data, the coefficient of friction (*cof*) ranges from about 2-5 for oxide coated polycrystalline silicon (polysilicon) devices to 0.1 for polysilicon devices coated with hydrophobic films. Applied pressures were in the tens of kilopascals. Because the force generated by comb drives is on the order of micronewtons, only a small normal pressure range can be studied for a given test structure. Further, the comb drive consumes a large area to obtain the friction data. In this Chapter, we consider cantilever beams as potential friction devices. Forces generated can vary from the micronewton to the millinewton level. Hence, a much larger pressure range can potentially be explored at the expense of slip range. Also, with their small footprint, such devices have the potential to serve as parametric test structures. That is, they can provide valuable information without occupying significant real estate on chips, acting as *in-situ* diagnostic process monitors.

II. Conceptual evolution of the test structure

Fig. 1 Slip induced by out-of-plane deflection of amplitude A .

For an inextensible beam, out-of-plane force applied to a cantilever beam induces slip Δ , according to

$$\Delta = c \frac{A^2}{L}. \quad (1)$$

Here, A is the amplitude of the deflection, L is the length of the beam, and the constant $c \approx 2$ to first order. The slip Δ is small for such a design; we shall see that it is on the order of 15 nm. An average contact radius between asperities of 10 nm has been calculated for the rms roughness typical of our polysilicon [15]. Therefore, while the slip is small, it should be sufficient to disengage contacting asperities, and engage asperities at new contacts. Hence, it seems reasonable to expect that a properly designed cantilever structure can incur losses typical of frictional processes in micromachines.

Our goal is to design a friction device which meets the criteria of having independent pressure and velocity control. Although the slip for a cantilever beam is small, the force generated can be very large. Therefore, it should be possible to apply a large range of pressures if the device is properly designed. Velocity can be controlled through the frequency of the applied force to the driver beam.

Fig. 2a First concept for friction test structure. This structure fails because the slider block cannot be uniformly maintained in contact with the substrate as driver force P_d cycles.

Our first attempt at building a test structure is conceptually represented in Fig. 2a. The driver beam is initially flat and suspended above the substrate, being cantilevered from the support post at the left end. The friction pad is brought into contact by the distributed load q . Oscillating force P_d will cause the slider to move back and forth, with contact area controlled by the design length of the friction

pad. Both q and P_d are generated by electrostatic forces.

Because of the restoring force of the cantilever, the force q applied to the slider pad cannot immediately cause uniform contact. Beam theory was utilized to understand the deflections in the slider pad area as a function of P_d . For this test structure design, we found that the slider pad could not be maintained in contact along its length as P_d varied, even for large values of q . At $P_d=0$, this was physically traced to the connection between the end of the cantilever and the friction pad. To keep the pad in contact along its entire length, the slope must be 0 at this connection. Enforcement of this condition requires that a moment exist at the connection. However, as the pad becomes flat with increasing q , the moment vanishes. For

Fig. 2b Improved friction test structure. By connecting to the friction pad via a hinge, a virtually uniform pressure is achieved for relatively small values of q .

example, when applying 40V to generate a q of $7,000 \text{ N/m}^2$, the length along the friction pad which is not touching is approximately 30 microns for $P_d=0$. As P_d increases, the pad eventually does come perfectly into contact. But for subsequent increase of P_d , the friction pad rocks about its left bottom corner, leaving only a point contact.

We improved the design as seen in Fig. 2b. Relative to Fig. 2a, the structure differs only in its connection between the sliding friction pad and the driver beam, in that now the connection is made by a hinge. The hinge placement allows the contact area of the pad to be virtually independent of P_d . This is for two reasons: First, at $P_d=0$, the force applied by q on both sides of the hinge allows the slope at the connection point to approach 0 at relatively low values of q . Second, as P_d increases for a given value of q , rocking is resisted because the hinge placement allows the friction pad to extend to the left of the connection between the driver beam and the pad.

The layout and design of the structure is shown in Fig. 3. Fig. 3a is a top view, where it is seen that the hinge is formed by forking the driver beam around the friction pad. In the present work, the length of the driver beam L_d is 500 μm , the length of the friction pad L_p is 200-600 μm , and the length of the hinge L_h is 10 μm . This hinge length is sufficient to be "stiff", that is, it twists only a small amount due to its high polar moment of inertia. L_h can be shortened to 2 μm such that the hinge becomes flexible. This further reduces the change in contact area vs. P_d . However, according to our calculations, axial compliance of a flexible hinge can be a nm or more for large cof . Because the slip $\bullet$ is only about 15 nm, this can reduce the actual slip of the friction pad. The width of the driver beam W_d is 50 μm to maximize the axial force developed by the driver beam. The width of the hinge W_h is 5 μm to minimize its torsional and lateral compliance.

Fig. 3a Top view of the hinged friction pad
P3 - poly 3, P0 - poly 0.

Fig. 3b Cross-section through AA' of Fig. 3a.
P - poly, SO - sacrificial oxide.

Fig. 3b shows a cross-sectional schematic view after poly 3 etch of the friction pad area through section AA' of Fig. 3a. Four levels of polysilicon (poly), designated as P0, P1, P2, and

P3 are used to form this part of the structure. Sacrificial oxide layers are designated as SO1, SO2 and SO3. Voltage applied across the poly 1 pad to the electrostatic wings in poly 3 provides normal distributed force q to bring the friction pad into contact with poly 0 at a few volts. Once this occurs, the friction pad acts as a stop, and subsequent voltage applied to poly 1 supplies controllable normal pressure. In this structure, poly 1 is not a necessity; poly 0 could be used instead. However, because of the inverse square relationship of electrostatic force with gap distance, the use of poly 1 allows much higher normal forces to be applied at a given voltage. By taking advantage of conformality in polysilicon deposition, fabrication of the friction pad with a $6\text{ }\mu\text{m}$ thick beam is possible. This creates a highly uniform pressure across the width of the sliding friction pad because its flexure is extremely small when loaded by the electrostatic wings.

III. Analytical modeling

To derive quantitative trends regarding controllability of the contact area, distributed loading q was assumed in the friction pad region, and point loading P_d was applied to the driver pad, as shown in Fig. 4a. The point loading allows simple analytical modeling to be utilized, and deformation trends can be predicted. In the actual experimental situation of Fig. 3a, distributed electric field loading is applied across the driver beam – this will be numerically modeled in the testing section below.

Initially the friction pad is suspended above the surface. As q is increased, it comes into contact with the substrate. Referring to Fig. 4a, reaction across the length of the friction pad is composed of uniform pressure for $x < L_d - a_1$ and $x > L_d + a_2$, while point reactions P_1 and P_2 act at $x = L_d - a_1$ and $x = L_d + a_2$ respectively. To calculate the deflections of the driver beam, these loads can be reduced to an effective load and moment P_h and M_h acting at the hinge, as shown in Fig. 4b.

Fig. 4a Beam model used to quantify deflections.

Fig. 4b Transformation to model driver beam.

Friction pad voltages corresponding to the loads required for a given q are represented by V_p . Electrostatic instabilities [16] in the friction pad region are neglected here. They are not catastrophic because the friction pad acts as a stop. The bending stiffness of the friction pad beam is approximately doubled by the narrow thick poly 2/3 stack at its center (Fig. 3b). This doubling was incorporated in the simulations, but changed the quantitative trends only in detail. To solve for the deflections, the four governing equations for the left and right side of the driver beam and friction pad respectively were expressed. Then, at a given loading, boundary conditions were applied. This gave four equations with four unknowns, which were solved iteratively. Inextensibility of the driver beam was assumed in the calculations.

III.1 Effect of friction pad loading, q

Two simulations were carried out. In the first, the friction pad was loaded with increasing values of q , as represented in Fig. 5. Fig. 5a shows the deflections of the driver pad as well as the friction pad. Fig. 5b shows a close-up in the vicinity of the friction pad. At $q=20$ N/m² (2V), the friction pad just touches the substrate at its right extreme. As q is increased to 350 N/m² (8V), the slope of the right hand side of the friction pad becomes 0, but the left hand side is not yet in contact. The large increase in q from 20 to 350 N/m² is because large bending resistance in the friction pad must be overcome. Increasing q to 600 N/m² (10V), the left hand side of the friction pad comes into contact with the substrate. At $q=1300$ N/m² (15V), the slope of the friction pad becomes 0 at its left end. Observe also that the contact in the right hand of the friction pad has increased considerably - a_1 has decreased to 29 μ m. Further increases in q reduce both a_1 and a_2 . At $q=5100$ N/m² (30V), a_1 has decreased to 49 μ m and a_2 to about 14 μ m.

Fig. 5a Deflections for increasing q with $P_d=0$

Fig. 5b Close-up near friction pad

Not only do a_1 and a_2 decrease substantially, but the deflection of the friction pad over the substrate becomes extremely small. For reference, a typical 2 nm rms roughness (from atomic force microscopy measurements [17]) of the poly 0 layer is superimposed in Fig. 5b. Considering that for $q=5100 \text{ N/m}^2$ the maximum deflection of $\sim 1 \text{ nm}$ is smaller than the rms roughness of the lower poly layer, the pressure of 5100 N/m^2 decreases only slightly over the curved portion of the friction pad. In the smooth beam theory, point load reactions P_1 and P_2 act at $x=L_d-a_1$ and $x=L_d+a_2$. However, because the friction pad does not lift off the rough surface, these loads will tend to distribute themselves over the sliding interface as well. The effect is opposite to the pressure lowering due to the uplifting of the friction pad. This net cancellation implies that the pressure modulation in the region $L_d-a_1 < x < L_d+a_2$ is small. Therefore, we have nearly constant pressure along the length of the friction pad for sufficiently high q . Contact mechanics of rough surfaces [18] can be invoked to understand the pressure modulation in more detail.

III.2 Effect of driver pad loading, P_d

In the second simulation, the effect of the driver beam force P_d on driver and friction pad deflections at $q=5100 \text{ N/m}^2$ was studied. The results are graphed in Fig. 6. In Fig. 6a, the maximum deflection occurs at $P_d=284 \text{ mN/m}$. This is because for $P_d=0$, the center point of the driver is $(6-1.04)=4.96 \mu\text{m}$ above the substrate. It can deflect one third of this distance before destructive electrostatic instability, i.e., $1.66 \mu\text{m}$. Therefore, the maximum deflection is $(1.04+1.66)=2.70 \mu\text{m}$ at $x=L_d/2$. This limits the total slip of the device, according to Eq. (1).

Fig. 6a Deflections for increasing P_d (mN/m) with $q=5100 \text{ N/m}^2$

Fig. 6b Close-up in friction pad area

In Fig. 6b, a close-up of Fig. 6a in the friction pad area, a_1 is greater than a_2 for $P_d < 90$ mN/m because the driver beam rotation at $x=L_d$ is positive. At $P_d=90$ mN/m, both a_1 and a_2 are zero (independent of q). Effectively only a small point load operates at $x=L_d$; the moment vanishes. For $P_d > 90$ mN/m, a_1 is less than a_2 as the driver beam rotation at $x=L_d$ rotates counterclockwise to become negative. Note that the pad deflections continue to be less than the rms roughness, except at the largest value of $P_d=284$ mN/m. Therefore, the modulation of pressure as P_d cycles continues to be small, according to the above discussion. In order to minimize the effect of the modulation, most devices were built with pad length $L_p=600$ μm rather than 200 μm . This assures that the pad has uniform distribution over at least 520 μm , i.e. 87% of the area is contacting at pressure $q=5100$ N/m² during any part of the cycle.

III.3 Relative slip and total friction load

Fig. 7 Relative Slip and Total Normal Load vs. P_d

Assuming inextensibility of the driver beam, the relative slip of the friction pad vs. P_d can be calculated from the arc length formula, and is plotted in Fig. 7 for $q=5100$ N/m². The slip increases with the square of P_d , as expected from Eq. (1). The total frictional (normal) load consists of the distributed load q times the area of the electrostatic wings, and the sum of point reactions P_1 and P_2 times the width of the electrostatic

wings. Their sum must increase as P_d increases. However, variation of the total frictional load is less than 5% because the normal loading is dominated by q , which is independently applied. At $P_d=284$, the maximum slip is approximately 13 nm. This is the maximum slip possible, and is nearly attainable for reasonably low axial force.

The axial load which the driver beam must develop in order to cause slip of the friction pad is the product of the coefficient of friction and the total frictional load. While the slip is small, the driver beam can develop very high axial loads, on the order of several millinewtons. Therefore, it can induce slip of the friction pad over a wide range of coefficient of friction as well as normal load. With $q=5100$ N/m², the pressure on the slider pad is $5100 \cdot (50/3) = 0.085$ MPa ($W_p=50$ μm , and the width at the bottom of the friction pad is 3 μm). In principle this can be

increased; with a pad voltage of 200V, the applied pressure would be 4 MPa. Another order of magnitude increase in pressure may be possible by reducing the Sac Ox 3 thickness, or by designing the friction pad such that it comes into contact with poly 0 only occasionally along its length.

To summarize, modeling results indicate that with the hinged pad structure, above a reasonably small value of q , the normal load is controllable, nearly evenly distributed and nearly constant along the length of the friction pad as the driver beam oscillates through its cycle. The slip is small, but perhaps sufficient to allow measurement of the *cof*. In principle, this device can be tested both statically and dynamically, yielding *cof* data over a wide experimental pressure and velocity space [19].

IV. Fabrication, testing and simulation

The devices were fabricated according to the Sandia multi-level polysilicon surface micromachining process [20]. An SEM cross-section of the friction pad is shown in Fig. 8. By comparing to Fig. 3b, the structure appears to have been successfully realized. In Fig. 8, note that the poly 3 is flat, in spite of the complicated topography beneath it. This can be achieved because our process includes chemical mechanical polishing (CMP) of the Sac Ox 3 layer. The width of the region where the slider pad makes contact with the poly 0 is 3 μm . This is dictated by the 2.0 μm minimum width of the Sac Ox 3 cut, as well as alignment tolerances between poly 3 and poly 2.

Fig. 8 SEM cross-section of the friction pad. (Compare to Fig. 3b)

Devices were released in 1:1 HCl:HF, transferred to H_2O_2 and freed by supercritical CO_2 drying [21]. After the H_2O_2 treatment, the surface is nominally hydrophilic, and a 1 nm oxide covers the surface. Electrical actuation performed under an interferometric microscope

Fig. 9 Pulling down the friction pad with V_p

(monochromatic green light with $\lambda=547$ nm), such that deflections could be assessed. In Fig. 9, we show results of the effect of pad voltage on the deflections, with 0V on the driver beam. With no voltage applied, we see that the structure is not flat – each fringe represents $\lambda/2=274$ nm. By focusing up and down, we could determine that the structures are curved up slightly. This is due to the stress gradient in the polysilicon. The driver beam shows small curvature from the stress gradient, while the curvature in the friction pad is larger. In the friction pad area, a conformal poly 3 deposition fills in 4 of the 6 μm height. (Poly

2 has negligible strain gradient, as could be determined by observing cantilever beams). Apparently due to the poly 3 being distributed over a 4 μm thickness, the strain gradient in the friction pad is larger than in the driver beam.

By applying 3V, the friction pad is brought into contact with the substrate, Fig. 9b. At 10V, it is flat along most of its length, as seen in Fig. 9c. Finally, at 25V, the beam is substantially flat. These results are in good qualitative agreement with the spirit of the

Fig. 10 Driver beam deflections for two different pad voltages

calculations presented in Fig. 5.

The main reason for the discrepancy in comparing the detailed deformations is due to the strain gradient of the poly 3.

By now inducing driver beam deflections at different driver pad voltages, we can test whether the friction pad induces resistance to slip. For these tests, pad voltage was first applied.

Then driver voltage was

increased until the driver beam was near pull-in. After a given test, both voltages were relaxed. Usually, the friction pad freed itself from the substrate – adhesive forces were relatively low. In Fig. 10, beam deflections for $V_d=58$ and for $V_p=10V$ and $40V$ respectively are shown. Here, the length of the friction pad is $200\text{ }\mu\text{m}$ rather than $600\text{ }\mu\text{m}$ as in Fig. 9. Note that in Fig. 10a, there are approximately eleven fringes from the support post on the left hand side, while in Fig. 10b, there are only ten fringes. Therefore, comparison of the inteferograms in Figs. 10a and 10b suggests qualitatively that there is a difference in the slip for the two cases of $V_p=10V$ and $40V$, because the same driver voltage produces dissimilar deflection profiles.

Fig. 11a Measurement and Abacus modeling for data in Fig. 10a

Fig. 11b Schematic of model to match measured deflections

To obtain a quantitative value of the slip, the deflections are first deduced from the fringe data. Linescans were taken along the driver beam from the pad to the slider, and also along one of the forked sections. Because the last segment of the driver beam near the slider and the beginning segment of the fork overlap in x , these two sections were superposed to obtain the total deflection, as shown in Fig. 11a.

Numerical simulation is required to model the deflections because of charge redistribution along the length of the driver beam. The situation modeled is shown in Fig. 11b, where the axial load due to the friction is represented by F_p . To infer slip distance, nonlinear finite element beam models were developed in Abaqus [22] to match the experimental data. The distributed electrostatic force was accurately modeled as a nonlinear deformation dependent capacitive loading, including Osterberg's fringing field correction [23].

In order to obtain reasonable agreement with the data, the compliance of the support post and the hinge could not be ignored. Rather than estimating this compliance from the geometric structure, the slope near the extremes was measured and included in the modeling boundary

conditions. Driver voltage V_d was then applied, and force F_p was increased until the deflections were matched. We see in Fig. 11a that excellent agreement between the simulation and the data is achieved.

Table 1- Calculated parameters from the modeling

V_p (V)	Slip (nm)	F_p (μ N)	F_n (μ N)	cof
10	-4.56	350	45	7.8
40	0.73	390	127	•3.1

As seen in Table 1, the modeling results indeed indicate there is a difference in slip of approximately 5 nm between the two cases. The values are relative to the slip after the pad has been brought into contact by pad voltage, and are negative for slip to the left. Slip to the right is not expected, therefore the value of 0.73 nm (which may be slightly off due to experimental error) for $V_p=40$ V probably indicates that the pad has not initiated slip. The values for F_p do not differ greatly. This apparently indicates that for $V_p=10$ V, $F_p \bullet 350$ μ N is required to initiate slip, while for $V_p=40$ V, a value of $F_p=390$ μ N is insufficient.

Normal force F_n in Table 1 is computed from sum of the point reaction force, 40 μ N, and the distributed force. For $V_p=10$ V, most of the contribution is from the point loading, and the distribution of pressure is rather non-uniform. The pad is in contact only to the left of the hinge, therefore the approximate pressure is ~ 150 KPa. However, over small variations in pressure, the cof is not generally expected to vary greatly. Keeping this in mind, the apparent $cof \sim 8$ is somewhat high compared to previous results. For $V_p=10$ V, $cof=7.8$. This is a lower bound, because it is possible that F_p must increase to move the pad further. The value compares to $cof=2$ measured for a supercritically dried surface in ref. [13], and $cof=5$ in ref. [14]. Further study is required to understand this high value. At $V_p=10$ V, $V_d=58$ V is very near the pull-in voltage of the device.

For $V_p=40$ V the pad is in contact along its entire length, and the value of $cof=3.1$ is a lower bound because the slip apparently has not yet initiated. If the cof is truly 8, no slip is expected before pull-in at $V_p=40$ V. Such a high coefficient of friction limits the usage of this device as currently designed. Greater slip amplitude is possible by the principle of leveraged

bending [24], and has been implemented in our next design. Calculations indicate the potential slip can be doubled at the expense of greater driver voltage. Also, we will apply molecular coatings [25] to these devices to lower the coefficient of friction in subsequent measurements.

A problem encountered in our current design is in the symmetry of the friction pad. As seen in Figs. 3b and 8, the pad is loaded symmetrically, which theoretically will give equal loading from each wing. However, a small difference due to geometry after etch or misalignment of poly 3 to poly 0 will cause the force to be unequal on the two sides, giving rise to positive feedback. This results in toppling of the device when the torsional resistance of the driver beam is overwhelmed. Depending on the device, the voltage on the friction pad has been limited to 30-50V maximum. Our follow-up design will employ a more stable friction pad configuration.

V. Summary and conclusions

We have designed, modeled, fabricated and performed initial test measurements on a new structure to measure friction in micromachining. The device consists of a cantilevered forked beam and a friction pad attached via a hinge. The placement of the hinge is crucial to obtaining a well known and constant pressure distribution in the device. Compared to comb drive configurations, the current structure has a larger pressure range and occupies a much smaller area at the expense of range in slip. Furthermore, it has a very large velocity range [19]. Static deflections on the device were measured and modeled using Abaqus. With the oxide coating, slip of 5 nm was inferred shortly before pull-in. The observed $cof \sim 8$ limits the slip range due to stretching of the driver beam. The cof can be reduced by applying molecular coatings, enhancing the usefulness of the present device. To improve on the device, increased slip by leveraged bending and a more stable friction pad configuration will be implemented.

VI. Acknowledgements

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