Experimental Studies of the Tribological Behavior of Microelectromechanical Systems

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Experimental Studies of the Tribological Behavior of Microelectromechanical Systems

by

Hua Xiang

A dissertation submitted in partial satisfaction of the requirements for the degree of
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in
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in the
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University of California at Berkeley

Committee in charge:

Professor Kyriakos Komvopoulos, Chair
Professor Robert O. Ritchie
Professor Liwei Lin

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Experimental Studies of the Tribological Behavior of Microelectromechanical Systems

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by

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Abstract

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Doctor of Philosophy in Engineering – Mechanical Engineering

University of California at Berkeley

Professor Kyriakos Komvopoulos, Chair

Microelectromechanical systems (MEMS) are used in a wide range of applications including sensors, actuators, biomedical devices, projection systems, and photovoltaic structures. Advances in MEMS have increased the demand for more reliable microstructures. The reliability and performance of many MEMS devices depends strongly on the tribological properties of contact interface. Knowledge of the origins and evolution of the surface forces is therefore important to the design of MEMS devices with contact interfaces. In this dissertation, special microdevices fabricated by surface micromachining were used to characterize interfacial phenomena encountered under typical operation conditions of MEMS devices. Dynamic impact and sliding contact experiments were performed to determine the critical parameters of device lifetime. The evolution of static adhesion force and friction force was examined under controlled loading and environmental conditions. A criterion of micromachine failure due to excessive interfacial adhesion (stiction) was formulated based on the observed experimental trend. Surface modification was examined by scanning electron microscopy (SEM), transmission electron microscopy (TEM) and atomic force microscopy (AFM), and the observations were compared to the evolution of critical interfacial properties. The change of the interfacial force was explained in the context of nanoscale surface modification effects resulting in both physical and chemical surface changes. The deposition of self-assembled monolayer (SAM) films is one of the most promising surface chemical treatments for preventing micromachine stiction during release and operation. The tribological properties of micromachine sidewall surfaces coated with a conformal
FOTS film were also investigated in the context of goniometry measurements, adhesion and static friction tests, and X-ray photoelectron spectroscopy (XPS) results. This study extends the understanding of the effect of interfacial phenomena on the operation of contact-mode MEMS devices, and the obtained results are valuable to the improvement of MEMS reliability.
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Chapter 1: Introduction

1.1 Microelectromechanical Systems

Microelectromechanical system (MEMS) refers to integrated micro-scale systems combining electrical, mechanical or other (magnetic, fluidic/thermal/etc.) elements typically fabricated using conventional semiconductor batch processing techniques that range in size from several nanometers to microns or even millimeters. In the last two decades, this emerging technology has attracted considerable attention and shown major impact on several sectors of manufacturing industry. MEMS are used in a wide range of applications including sensors, actuators, biomedical devices, projection systems, and photovoltaic structures.

The field of MEMS evolved from the integrated circuit industry. In the 1950’s and 1960’s, several researchers in academic and industrial laboratories began to make micromechanical devices using the integrated circuit processing technology. The origins of MEMS technology can be arguably traced back to the discovery of piezoresistive effect in silicon and germanium, a crucial element of micro sensors, by Smith at the Bell Telephone Laboratories in 1954. This was followed by discoveries in the Honeywell Research Center and the Bell Labs introducing the first mechanical transducers, and strain gauges.

Interest in the application of miniature machines dramatically grew along with bulk micromachining and surface micromachining technologies. In the 1970s, diaphragm-type silicon micromachine pressure sensors made using silicon bulk micromachining were developed by IBM research laboratory. A number of pioneering companies commercialized the first silicon pressure sensor, providing the earliest commercial success of MEMS technology. Silicon micromachining technology also enabled the ink-jet printing technology by making ink-ejection nozzles extremely small. In the late 1980s, three-dimensional mechanical structures including cantilevers or membranes were made using bulk silicon or thin film silicon. The first silicon surface micromachined micro-motor driven by electrostatic forces were demonstrated by researchers at the University of California at Berkeley.

In the 1990s, the field of MEMS started to gain widespread acceptance and attract government and private funding. Several commercial MEMS devices were developed in the areas of inertial sensing and micro-optical technology. One of the most notable industrial applications for MEMS technology is the accelerometer by Analog Devices, Inc. (Analog Devices, Inc., 1996). These accelerometers are initially used in air bag
sensors for automotive safety. The accelerometer chip developed by Analog Devices, shown in Figure 1.1(a), consists of a free-moving proof mass suspended by support springs. The proof mass contains a series of comb-like protrusions. The motion of the proof mass is detected by changes in the capacitance between the proof mass and the stationary fingers. The change is detected by on-chip signal-processing electronics that release the trigger of the safety device. Another industrial breakthrough of MEMS technology is the Texas Instruments Digital Micromirror Device, shown in Figure 1.2. It consists of a light-modulating chip with more than 500,000 micromirrors in an area of about 1.5 cm². Each mirror, corresponding to a single pixel, is capable of tilting towards the light source (ON) or away from it (OFF) up to ten thousand times per second. This creates a light or dark pixel on the projection surface.

The basis of a strong and sustainable industry based on MEMS technology has been established. It is expected that MEMS will continue to find new major applications in the near future. Therefore, the importance of MEMS reliability will continue to increase as more MEMS devices enter the market place.

1.2 Tribology in MEMS

Advances in the fabrication of complicated MEMS devices have increased the demand for higher reliability and longer operation lifetime. At microscopic levels, surface forces may exceed inertial (bulk) forces by several orders of magnitude due to the increase of the surface-to-volume ratio. Therefore tribological behaviors, including adhesion, friction and wear, are the most critical reliability issues, influencing significantly the performance of dynamic microsystems with contact interfaces. A brief overview of the major issues for MEMS reliability is presented below.

1.2.1 Adhesion and friction

Excessive adhesion (stiction) is one of the primary tribological failure mechanisms in MEMS. Adhesion forces may occur due to van der Waals force, electrostatic force (trapped charge), capillary force or a combination thereof. When the adhesion force exceeds the restoring force of the system, suspended structures may adhere permanently to the substrate or other proximal surfaces, causing device failure. Failure due to excessive adhesion (stiction) may occur either during fabrication, especially the final process step of the device release or during operation, which can be referred to as release stiction and in-use stiction, respectively.

Release stiction is the adhesion of a MEMS structure to the underlying substrate following the final sacrificial layer etches. If the final step of processing involves rinsing and drying by evaporation, capillary bridges may form between components in close proximity. The capillary force can be calculated using the Laplace equation given by,

$$p_L = \frac{2\gamma \cos \theta}{d}$$  \hspace{1cm} (1.1)

where \(\gamma\) is the surface tension of the liquid, \(d\) is the film thickness and \(\theta\) is the contact angle between the liquid droplet and the solid surface. Because the surfaces of polysilicon microstructures are covered by a native layer, they are hydrophilic. The very small radius of curvature of the air/liquid interface generates a higher attractive Laplace pressure which effectively pulls the surfaces in contact.

2
The release stiction problem can be alleviated by avoiding the formation of liquid-vapor interfaces. This can be accomplished by using supercritical CO$_2$ drying$^{10}$, freeze sublimation drying$^{11}$, dry HF vapor etching$^{12}$, and polymer support$^{13}$.

In-use stiction may occur when microstructure surfaces come into contact during operation. Excessive adhesion forces can be generated at micromachine interfaces due to changes in the surface chemical behavior$^{14}$ and surface topography (roughness)$^{15}$ and the build-up of surface charges$^{16}$. When the adhesion force is higher than the restoring force, micromachine surfaces permanently adhere to each other causing micromachine failure during operation.

According to classical frictional theories, friction is proportional to the normal load and independent on the contact area and relative sliding velocity. However, because of scaling down effects, the adhesion force may be on the same order of magnitude as external forces, or even higher. The presence of the adhesion force makes the dependence of friction on the normal load more complex. A. It has been reported that the static friction coefficient of polysilicon contact-mode microdevices operated in vacuum increase nonlinearly with the apparent contact pressure, indicating that the classical friction theories do not hold as devices and loads scaled down$^{17}$. The true static coefficient of friction can be determined by considering the effects of the dominant adhesion forces (i.e., van der Waals and capillary forces) on the normal force applied at the contact interface$^{18}$. The evolution of static and dynamic friction forces at micromachine sidewall interfaces during oscillatory sliding contact showed that microdevices tend to fail due to the increase of static friction rather than exceeding dynamic friction limits$^{19}$.

1.2.2 Methods for reducing surface forces

To improve the reliability of MEMS devices, various surface treatments are used to mitigate detrimental effects of high adhesion and friction forces at micromachine contact interfaces. These treatments can be classified into two categories: physical and chemical. In physical treatments, the magnitudes of retarding surface forces are reduced by surface protrusions (dimples) to control the apparent contact area or surface texturing (roughening) to minimize the real contact area$^{14,15}$. Texturing the surface topography by elective etching may decrease apparent surface adhesion by a factor of 20$^{20}$. Investigation of the effect of 20-50 nm silicon carbide particulates on interfacial adhesion of polysilicon surfaces indicated that nanoparticles can decrease the adhesion by increasing the average separation distance$^{21}$. However, the increase in roughness may cause an undesirable increase in friction force and enhancement of the detrimental effect of abrasive wear in contact-mode devices, especially sliding interfaces.

In chemical treatments, the surface chemical composition is altered either by chemical functionalization of deposition of solid or organic films such as low surface energy plasma-polymerized fluorocarbon$^{22}$; however, to coat underneath surfaces, the thickness of the deposited film on top surfaces needs to be relatively high, which may alter the mechanical properties of the microdevices. One of the most promising surface chemical treatments for preventing micromachine stiction during release and operation is the deposition of spontaneously self-assembling molecularly thin films of low surface energy substances, known as self-assembled monolayer (SAM) films$^{23}$. It has been
demonstrated that hydrophobic SAM coatings can lower adhesive and frictional forces by suppressing capillary forces between proximity surfaces\textsuperscript{23,24}. However, mechanical and thermal degradation of SAM coatings limited their application in harsh environments\textsuperscript{25,26}. Since alcohol vapors are effective lubricants between silicon contacts\textsuperscript{27,28}, in-situ vapor-phase lubrication of silicon MEMS devices with 1-pentanol vapor, resulting in a replenishable lubricating film on the surfaces, which yielded lower friction forces and longer operational lifetime compared to chemisorbed monolayers alone, was investigated\textsuperscript{29}.

1.2.3 Wear

The generation of wear is one of the most critical issues in contact-mode MEMS device operation. The sizes of polycrystalline grains, asperities, and wear debris can be of comparable orders of magnitude with the device dimensions. Topographical changes associated with asperity removal and wear debris generation can drastically alter the tribological characteristics of the device. The primary wear effects occurring at the MEMS interfaces can be classified into two categories: chemical effects (removal of surface layers such as self-assembled monolayers, organic contaminants, or native oxide films, exposing material with a different chemical composition) and physical effects (removal of asperities leading to changes in surface roughness, real contact area, relative surface separation, and two- or three-body interactions)\textsuperscript{30}.

Figure 1.3 shows a bearing of a MEMS device before and after testing that exhibits clearly the loss of silicon material due to dynamic (impact) loading\textsuperscript{31}. Build-up of wear debris and high friction degrade the reliability and durability of micro devices. Patton et al. examined the tribological behavior of an electrostatic lateral output motor, and observed different wear mechanisms for dry air (vacuum) and humid air operation conditions\textsuperscript{32}. D. H. Alsem examined wear generation of MEMS sidewall surfaces under sliding contact using scanning electron microscopy (SEM) and atomic force microscopy (AFM) and reported a transition from adhesive wear to three-body abrasive wear with the progression of sliding\textsuperscript{33}.

1.3 Research Objectives

The reliability and performance of many MEMS devices depends strongly on the tribological properties of contact interfaces. To realize the full potential of MEMS, it is critical to understand the physical and chemical phenomena occurring at MEMS contact interfaces. Therefore, the purpose of this dissertation was to use special polysilicon microdevices fabricated by surface micromachining to examine adhesion, friction, and wear under typical operation conditions of MEMS devices. Dynamic impact and sliding contact experiments were performed to determine the critical parameters affecting the device lifetime.

In Chapter 2, the micromachines and experimental apparatus used in this dissertation are introduced. Two different microdevices for sliding and impact testing, were used in this dissertation to characterize the tribological behaviors of MEMS sidewall interface.
Sidewall contact surfaces in sliding contact are examined in Chapters 3 and 4. Chapter 3 presents a study of the changes in the adhesion behavior and morphology of sliding sidewall surfaces of polycrystalline silicon MEMS devices operated in high vacuum (~10⁻⁵ torr) and under low apparent contact pressures (0.1–18 kPa). In Chapter 4, the evolution of adhesion and friction in sliding contact-mode micromachines operated in high vacuum is considered by tracking changes in the adhesive pressure, interfacial shear strength, and static coefficient of friction with accumulating sliding cycles.

Chapters 5 and 6 explore dynamic impact contact in relation to critical parameters of the device operational lifetime. In Chapter 5, the effect of repetitive impact loading on the evolution of adhesion at sidewall contact interfaces under different conditions of contact load, ambient pressure, and relative humidity is investigated. Chapter 6 describes an experimental study of the effect of impact velocity on the adhesion characteristics of sidewall contact interfaces of dynamic micromachines.

Chapter 7 presents a study on the effect of fluorocarbon self-assembled monolayer films on sidewall adhesion and friction of surface micromachines with impacting and sliding contact interfaces. The hydrophobic behavior and structural composition of the FOTS film deposited on Si(100) are discussed in the light of goniometry and X-ray photoelectron spectroscopy measurements. The effects of contact pressure, relative humidity, temperature, and impact or sliding cycles on the adhesive and friction behavior of uncoated and FOTS-coated polysilicon micromachines are investigated under controlled testing and environmental conditions.

In this dissertation, results from the tribological examination of MEMS devices are presented and interpreted in the context of physicochemical surface changes. The results of this study provide new insight into the effects of interfacial phenomena on the operation of contact-mode MEMS devices, and have important implications in the design of more reliable and durable contact-mode MEMS devices.
References

Figure 1.1 Scanning electron micrograph of a dual-axis accelerometer by Analog Devices.
Figure 1.2 Two DMD pixels (mirrors are shown as transparent) of a Texas Instrument Digital micro-mirror device.

Figure 1.3 SEM images showing (a) an untested microdevice and (b) severe wear in a microdevice after operation\textsuperscript{31}.
Chapter 2: Experimental

2.1 Introduction

In this chapter, the techniques and testing methods used in this dissertation are described in detail. Two types of micromachines used to characterize the tribological behavior of sidewall contact interfaces of microelectromechanical systems (MEMS) are presented. The probe station used for on-chip testing of the micromachines is also described in detail.

2.2 Micromachine Test Devices

2.2.1 Impact testing micromachine

Adhesion, friction, impact, and sliding tests were performed with specially designed surface micromachines. Two types of micromachines were used in this study – impact and sliding micromachines – both fabricated by Polysilicon Multi-User MEMS Processes (MEMSCAP). All structural layers were deposited by low-pressure chemical vapor deposition and etched by reactive ion etching, which principally defined the sidewall surface topography. The structural and wiring layers consist of heavily doped n-type polysilicon. Phosphosilicate glass (PSG) and low-stress silicon nitride were used as sacrificial and electrical isolation layers, respectively. To etch the PSG layer and release the micromachines, dies with the fabricated micromachines were submerged in 49% hydrofluoric acid for 2.5 min and then rinsed with deionized water for 10 min and isopropyl alcohol also for 10 min. To avoid microstructure collapse and stiction due to the effect of capillary forces, the microstructures were released by carbon dioxide supercritical point drying, which resulted in the formation of a silicon oxide film and the deposition of a residue organic material. Further fabrication and operation details can be found elsewhere.

Figure 2.1(a) shows a scanning electron microscope (SEM) image of the polysilicon impact micromachine used in this study. Briefly, the symmetric micromachine consists of two shuttles supported by double-folded flexure suspensions that can be modeled as linear springs producing a mechanical restoring force \( F_r \) given by

\[
F_r = kx = \left( \frac{24EI}{L^3} \right) x
\]

where \( k \) is the suspension stiffness, \( x \) is the lateral displacement, \( E \) is the elastic modulus of polysilicon, \( I \) is the moment of inertia in the displacement direction, and \( L \) is the beam length. The two shuttles are driven by a set of parallel electrostatic comb-drive actuators, and can be either pushed together by activating the inside (push) comb drives or pulled away from each other by activating the outside (pull) comb drives. The comb-drive actuators generate an
The electrostatic force that can be calculated using the parallel-plate approximation. The electrostatic force \( F_{el} \) produced from a comb drive actuator is given by

\[
F_{el} = \frac{N \varepsilon_0 \varepsilon h V^2}{g}
\]  

(2.2)

where \( N \) is the number of comb fingers (= 46 in the present micromachine), \( \varepsilon_0 \) is the permittivity of vacuum (= \( 8.854 \times 10^{-12} \) F/m), \( \varepsilon \) is the relative permittivity (= 1 for air or vacuum), \( h \) is the structural layer thickness (= 2 \( \mu \)m), \( V \) is the voltage applied between stationary and moving comb fingers, and \( g \) is the gap between comb fingers (= 2 \( \mu \)m).

An extension (protrusion) on the side of one of the shuttles was used to control the apparent contact area between the sidewall surfaces. Figure 2.1(c) shows a SEM image of the protrusion at the side of shuttle (2) used to establish contact with the sidewall surface of shuttle (1). The two shuttles (initially separated by a distance of 5 \( \mu \)m) were brought into contact along their sidewall surfaces by activating the comb drives. Surface alignment in the present design has been confirmed by white-light interferometry\(^5\). Symmetric voltage waveforms were applied during testing to ensure vertical alignment of the shuttles in the presence of levitation forces\(^6\).

### 2.2.2 Sliding testing micromachine

Figure 2.1(b) shows a scanning electron microscope (SEM) image of the micromachine designed for sliding testing. Similar to the impact micromachine, microfabrication of the sliding micromachine was performed with PolyMUMPs process.

The sliding micromachine consists of two suspended shuttles, which can be actuated in orthogonal directions by comb drives, referred to as the loading and sliding shuttles. The loading shuttle is used to apply the normal contact force or to detach the sidewall surfaces, while the sliding shuttle is used to slide (shear) one sidewall surface over the other.

Similar to the impact micromachine described in Section 2.2.1, the sliding micromachine contains comb-driven shuttles that are mechanically supported by folded-flexure suspension systems. The force relationship in the comb drive actuators is given by Equation 2.2, with identical dimensions to those of the impact micromachine. The loading shuttle contains 40 comb fingers while the sliding shuttle contains 20.

The loading shuttle is supported by a symmetric folded-flexure suspension system identical to that supporting the shuttles of the impact testing device and, therefore, its mechanical restoring force is given by Equation 2.1. The sliding shuttle can be seen as a conventional comb shuttle bisected through its center along the direction of lateral movement. This shuttle possesses only a single 300-\( \mu \)m-long folded-flexure suspension. The mechanical restoring force of the suspension system in the sliding shuttle is given by

\[
F_{r}^{s} = k_{s}^{s} x = \frac{12EIx}{L^3}
\]  

(2.3)

where \( k_{s}^{s} \) and \( F_{r}^{s} \) indicate the stiffness and restoring force values of the single folded-flexure suspension of the sliding shuttle.

Similar to the impact micromachine, there is a protrusion on the sidewall surface of the sliding shuttle of the sliding micromachine to control the apparent contact area with the
opposed sidewall surface. Surface alignment of the shuttles has been confirmed by white-light interferometry\(^6\).

### 2.3 Probe Station

All of the on-chip experiments were conducted in a custom-made multi-probe vacuum station (MMR Technologies) mounted on a vibration isolation table (Newport Electronics). Eight Tungsten probe tips connected to dc power sources (Agilent, E3612A) and ac power source (Hewlett-Packard, 3310A) were used to apply voltages to the shuttles and actuator comb drives. Shuttle displacement was observed with a stereomicroscope (Olympus, SZ-CTV). Digital images were obtained with a charge-coupled device camera (Sony, DXC-390P). The chamber pressure was controlled by a mechanical roughing pump and a turbo pump. The relative humidity in the chamber was increased by placing a small container with water on the same stage with the test chip, and was decreased by placing next to the test chip a small container with desiccant and also introducing dry nitrogen into the chamber. During testing, the relative humidity and the chip temperature were monitored with a humidity sensor (Honeywell, HIH-3602-A) and a programmable temperature controller (MMR Technologies, K-20), respectively. A schematic of the probe station is shown in Figure 2.2.

### 2.4 Microanalysis Techniques

#### 2.4.1 Goniometry

The wettability characteristics of the surfaces are quantified by analysis of static contact angle measurements obtained at room temperature using a drop-shape analysis system (DSA10, Krüss GmbH). Droplets of deionized water (~6 µL) were applied to the FOTS film surface by a syringe, and the droplet configuration was captured by a camera. Then, the angle between the droplet baseline and the tangent at the water/air boundary was measured, and the contact angle was calculated as the average of the left and right contact angles.

#### 2.4.2 Scanning electron microscopy

Changes in the surface morphology and formation of wear debris on the sidewall surfaces were observed with SEM (Zeiss Supra 55VP). SEM produces images of the surfaces by scanning it with a focused beam of electrons, which interact with atoms in the sample, producing various signals that can be detected to provide information about surface topography and composition.

#### 2.4.3 X-ray photoelectron spectroscopy

X-ray photoelectron spectroscopy (XPS) was used to characterize the surface chemical composition and bonding state of self-assembled monolayer in Chapter 7. The system used is an XPS system (PHI 5400 ESCA, Perkin-Elmer), without charge neutralization or monochromator, equipped with an Al-K\(\alpha\) X-ray source of photon energy equal to 1486.6 eV. A takeoff angle of 54.7° relative to the analyzer axis and a chamber pressure of \(\sim 10^{-7}\) Torr were used to collect all the XPS spectra.
2.4.4 Transmission electron microscopy

Analytical TEM and energy-filtered TEM using a 300 kV JEOL 3010 (with LaB$_6$ filament) and a 200 kV Philips CM200FEG (field emission gun) equipped with a Gatan image filter were used to examine the horizontal cross-sections of the near-surface microstructure of sidewall surfaces in Chapter 3.

2.4.5 Atomic force microscopy

An AFM (Veeco Digital Instruments, Nanoscope IIIa) was operated in non-contact mode, using single-crystal Si tips of nominal radius of curvature equal to 5 nm (Nanoworld Pointprobe), to quantify the sidewall surface topographies in Chapter 3 and Chapter 4. Details on the principles and use of the AFM can be found elsewhere.$^7$
References

Figure 2.1 SEM images of polysilicon surface micromachines specifically designed for (a) impact and (b) sliding testing including electrical wiring for electrostatic comb-drive actuation, and (c) high-magnification SEM image of the sidewall contact region.
Figure 2.2 Schematics of the testing apparatus used for on-chip experiments.
Chapter 3: Sidewall Adhesion and Sliding Contact Behavior of Polycrystalline Silicon Microdevices Operated in High Vacuum

3.1 Introduction

Microelectromechanical systems (MEMS) fabricated by low-cost batch processing are used in a wide range of consumer products and defense/space applications, such as chemical, pressure, and acceleration sensors, optical switches, inkjet printer heads, and liquid crystal display projectors. Many of these microdevices (e.g., micromotors, microswitches, and digital micromirrors) contain components with load-bearing contact interfaces. As a result of the large surface-to-volume ratio of the microstructures comprising these systems, failure is controlled by micro/nanoscale surface damage (wear) processes. Because at these small scales surface interactions assume a dominant role, adhesion, electrostatic, and friction forces may exceed inertial forces by several orders of magnitude. Since microstructure, topography, and damage features, such as grains, asperities, and wear debris, respectively, can be of the same order of magnitude as some of the device dimensions (e.g., thickness), changes in the surface topography and formation of fine wear debris may alter significantly the tribological characteristics and, in turn, the device reliability and operation lifetime. Alteration of the adhesion, friction, and electrical characteristics by micro/nanoscale wear processes could be detrimental to the device functionality and efficiency. Despite significant progress in fabrication processes and design of agile MEMS devices, the operation lifetime of such microsystems is still limited by excessive adhesion forces, high wear rates, and rapid evolution of contact fatigue. Seizure of the microdevice operation due to surface degradation and lack of insight into the underlying failure mechanisms are major obstacles, limiting the application range of MEMS.

In contact-mode microdevices, contact stresses should ideally be relatively low to prevent surface damage during operation. In addition, to avoid detrimental environmental effects, devices must be hermetically packaged in vacuum or inert gas atmospheres. However, despite an increasing technological interest on the tribological behavior of MEMS operating under these conditions, there is only a limited amount of data available in the high-vacuum/low-contact-pressure range of operating parameters of contact-mode
microdevices. Although multi-asperity contact phenomena in micromachined polycrystalline silicon (polysilicon) have been investigated extensively using on-chip device testing under controlled contact load and environmental conditions\(^8\)-\(^{26}\), tribological studies for high-vacuum/low-contact-pressure conditions are sparse. It has been reported that wear of polysilicon in vacuum commences with the removal of the native oxide layer, followed by asperity fracture or grain pull-out of the exposed silicon surface\(^{27}\). Longer lifetime of lateral motors has been reported for operation in air than in vacuum for contact loads in the mN–N range\(^{28,29}\). The static friction coefficient of polysilicon contact-mode microdevices operated in vacuum under loads in the mN range has been reported to increase nonlinearly with the apparent contact pressure\(^{30}\). Surface micromachines suitable for studying sidewall adhesion under much lighter loads (in the nN range), resulting in mean contact pressures on the order of kPa, were used to study the contribution of contacting and non-contacting asperities to the total adhesion force\(^{31,32}\) and measure the true static coefficient, which includes contributions from both van der Waals and capillary forces to the applied normal load\(^{33}\). Insight into the wear behavior of sidewall surfaces has been obtained from transmission electron microscopy (TEM) studies of collected wear debris and by monitoring the evolution of the static adhesion force in the course of oscillatory sliding contact of devices operated in high vacuum under relatively high contact pressures\(^{34}\).

A review of the literature suggests that basic understanding of multi-asperity contact interactions in contact-mode MEMS devices operated in high vacuum and under low contact pressures is limited. Therefore, the objective of this study was to investigate the high-vacuum tribological behavior of polysilicon sidewall surfaces subjected to apparent contact pressures of a few kPa. The adhesive contact pressure was used as an in-situ indicator of the evolution of the surface morphology during reciprocating sliding of the sidewall surfaces. Changes in the surface morphology after a given number of sliding cycles and insight into the governing wear process were obtained from scanning electron microscopy (SEM), TEM, and atomic force microscopy (AFM) studies. Results and interpretations presented below are directly applicable to the design and fabrication of reliable polysilicon devices possessing contact interfaces.

### 3.2 Experimental Procedures

The test device consists of two suspended shuttles, referred to as the loading and sliding shuttles, driven laterally by electrostatic comb drives (Figure 3.1(a)). The fabrication and operation details of this device are described in Section 2.2. First, normal contact between the sidewall surfaces was established by applying a dc bias voltage to the comb drive of the loading shuttle in order to push it against a protrusion of width equal to 5, 10, or 20 μm extending from the sidewall surface of the sliding shuttle (Figure 3.1(b)). Then, oscillatory sliding contact was initiated by applying sinusoidal ac signals to the comb drive of the sliding shuttle, causing the protrusion to slide against the sidewall surface of the loading shuttle. Relationships of the forces acting during sliding contact (Figure 3.1(b)) and the true and engineering friction coefficients can be found in previous publications\(^{15,31-34}\). Adhesion and sliding tests were carried out with a multi-probe.
vacuum station (MMR Technologies) mounted on a vibration isolation table (Newport Electronics), as described in Section 2.3.

Changes in the surface morphology and formation of wear debris on the sliding sidewall surfaces were observed with a SEM (Zeiss Supra 55VP) and a dual-beam focused-ion beam (FIB) system (FEI Strata DB235) operated at 5 kV. More detailed information of the sidewall surface topographies was obtained with an AFM (Veeco Digital Instruments, Nanoscope IIIa) operated in non-contact mode, using single-crystal Si tips of nominal radius of curvature equal to 5 nm (Nanoworld Pointprobe). Horizontal cross-sections of the near-surface microstructure of sidewall surfaces were examined by analytical TEM and energy-filtered TEM using a 300 kV JEOL 3010 (with LaB$_6$ filament) and a 200 kV Philips CM200FEG (field emission gun) equipped with a Gatan image filter, respectively. AFM and TEM samples were prepared with the previously mentioned dual-beam FIB using a tungsten micromanipulator (Omniprobe) (Figure 3.2). Worn protrusions were cut from the sliding shuttles with the FIB (Figure 3.2(a)) and moved to a TEM copper grid using the tungsten micromanipulator (Figure 3.2(b)) onto which they were temporarily welded with Pt (Figures 3.2(c)–3.2(e)). FIB thinning of these protrusions (Figures. 3.2(e) and 3.2(f)) to electron transparency (~100 nm) was performed after the deposition of protective Pt layers onto the surface exposed to the ion beam. First, a Pt layer was deposited by electron-beam evaporation to protect the sample from damage during ion-beam deposition of a second thicker layer. To thin down the sample, slices perpendicular to the sliding surface were milled off, starting from the side of the sample protected by the Pt layer, i.e., opposite to the side that had been in sliding contact (Figure 3.2(f)). This was necessary to prevent ion implantation during thinning and subsequent damage of the sliding surface. The drawback of thinning from the opposite side is that some material may be re-deposited on the side of interest. For the final thickness of the TEM sample, the sampling percentage of the total apparent area of contact was 5%, which is significantly larger than the typical real-to-apparent contact area ratio, implying that any global wear within the surface layer should be visible in the TEM sample.

AFM samples were prepared by FIB sample preparation techniques similar to that of the TEM samples (Figures 3.2(a) and 3.2(b)). However, in contrast to the TEM samples for which the grids were placed horizontally, the TEM copper grid of the AFM samples was positioned vertically before the sample attachment (Figures 3.2(g) and 3.2(h)). The grid was then placed horizontally to orient the sidewall surface in the upward direction for subsequent AFM imaging. Surface height data obtained with the AFM were used to calculate the root-mean-square (rms) roughness and the power spectral density (PSD) function of sidewall surfaces scanned before and after testing. AFM images (512 × 512 pixels) of 2 × 2 µm$^2$ surface areas were analyzed by 512 line scans obtained along the sliding direction. To evaluate topographical differences in the sliding direction, PSD functions of the slid surfaces were then obtained as averages of the PSD functions determined from the aforementioned line scans. To further examine nanotopography differences between the sliding and the original surfaces, standard deviations of corresponding PSD functions were contrasted in the low-wavelength range.
3.3 Results

3.3.1 Adhesion and dynamic friction

Figure 3.3 shows the variation of the mean adhesive pressure (defined as the measured adhesive force divided by the apparent contact area between the protrusion of the sliding shuttle and the sidewall surface of the loading shuttle) with sliding cycles for mean contact pressure in the range of 0.98–17.8 kPa. The statistical results shown in this figure were obtained from 4 devices. Each point represents the mean value of 25 measurements, i.e., 5 adhesion measurements obtained from 5 different locations of the sliding track of each device operated for a given number of sliding cycles. Thus, error bars represent local experimental scatter as well as location-to-location scatter along the sliding track. The general trend is for sidewall adhesion to increase with both sliding cycles and mean contact pressure. For all contact pressures, a fairly stable adhesive behavior was observed up to ~10^5 sliding cycles (incubation period), followed by an increase in adhesive pressure by a factor of 2–4 (depending on the mean contact pressure) in the range of 10^5–10^6 sliding cycles, with the eventual seizure of the oscillatory movement (stiction) of the device sliding under the highest contact pressure (17.8 kPa).

Figure 4 shows the dimensionless stick position of 15 devices tested in the mean contact pressure range of 0.98–17.8 kPa. The dimensionless stick position is defined as the ratio of the position of the sliding shuttle at the instant that oscillatory movement seized to the oscillation amplitude at the onset of sliding under a given mean contact pressure. Two interesting trends can be identified: (a) 11 (77%) of the devices seized sliding within the center region of the oscillation amplitude (i.e., within ±30% of the dynamic amplitude measured from the rest position) and (b) variation of the mean contact pressure by more than an order of magnitude did not change the general trend of device stiction to occur within the center region of the sliding track. Plotting these data in the form of a histogram yielded a fairly symmetric distribution of devices that seized sliding due to stiction at about the zero-amplitude point, confirming the general trend revealed by the results shown in Figure 3.4.

The relation between adhesion and stick position was further examined by measuring the mean adhesive pressure at different distances from the zero-displacement point as a function of sliding cycles. This involved interrupting each test after a given number of sliding cycles, displacing the sliding shuttle from the zero-amplitude position to the desired position by applying a dc bias voltage (referred to as \( V_{\text{shear}} \)) to the comb drive of the sliding shuttle, and measuring the adhesive pressure as described above. Representative results from these experiments are shown in Figure 5. Generally higher adhesion and/or faster increase in adhesion were found within the center region of the sliding track (−2 V ≤ \( V_{\text{shear}} \) ≤ 2 V), revealing a correlation between the increase in adhesive pressure and failure (stick) position.

Among the 32 devices tested, 3 devices (~9%) did not stick even after 10^7 sliding cycles (at which instant sliding was terminated), 4 devices (~13%) failed before reaching 10^5 sliding cycles, while 25 devices (~78%) seized movement after 10^5–10^7 sliding cycles. For the latter devices, the average number of sliding cycles to stiction was equal to ~2 × 10^6, regardless of the mean contact pressure. The sliding cycles to stiction of
devices operated in the mean contact pressure ranges of 1–2, 2–4, 4–10, and 10–18 kPa varied by a factor of ~10, indicating significant device-to-device variation.

The engineering and true friction coefficients for mean adhesive pressure between 7.15 and 12.87 kPa measured before sliding and after stiction are given in Table 3.1. The data represent averages of 5 measurements obtained with the same device for a given mean adhesive pressure. Changes in the engineering friction coefficient from the onset of sliding up to the instant of stiction do not show a consistent trend. This is attributed to the significant contribution of the adhesion force to the total normal force at relatively low and moderate contact pressures, which is not included in the calculation of the engineering friction coefficient. Conversely, the true friction coefficient, which includes the effect of the adhesive force, measured after stiction is consistently lower than that measured before sliding. Although the change in the true friction coefficient indicates that sliding induced surface changes, the directly measurable parameter that dominates the true coefficient of friction is the adhesive force. Therefore the mean adhesive pressure is a suitable parameter for tracking surface changes during oscillatory sliding contact.

3.3.2 Sidewall surface morphology

SEM images of sidewall surfaces obtained before sliding revealed the presence of small amounts of surface contaminants (Figures 3.6(a) and 3.6(b)). Clearly contrasting wear debris was found on some of the sliding sidewall surfaces (Figures 3.6(d)–3.6(f)), in addition to surface contaminant particles that had survived surface rubbing (see inset in Figure 3.6(c)). This suggests that a wear debris generating mechanism was active during sliding. However, as observed by SEM, sliding did not produce any other discernible surface changes, such as pits or microcracks. The surface dimples shown in Figures 3.6(e) and 3.6(f) were also observed on the as-fabricated sidewall surfaces. Wear debris was usually only present in one or two locations and mostly in very small quantities (see insets in Figure 3.6(d)–3.6(f)). Wear debris observations made with devices tested under various contact loads (pressures) and for different durations are summarized in Table 3.2. Among the 26 devices inspected, 11 (42%) contained wear debris, with larger debris (50–140 nm) appearing to be agglomerates of smaller (10–50 nm) wear particles, not possible to determine from the SEM images if they were also agglomerates of even smaller particles.

In addition to these types of wear debris, another type of surface particles was found not only after but also before sliding (see insets in Figures 3.6(a) and 3.6(b)). These particles were likely organic contaminants generated by the release process, which also includes the removal of a photoresist layer. Energy dispersive X-ray spectroscopy (EDS) analysis of a large (several μm) cluster of this residue remotely from the sliding track and a clean area of the structural layer (reference spectrum) showed the presence of carbon and oxygen peaks only in the EDS spectrum of the contaminant cluster, indicating that it was most likely organic in nature. Although SEM imaging often results in carbon deposition onto the imaged surface, the distinct differences in the EDS spectra of the contaminant cluster and the reference surface in conjunction with the presence of oxygen are clear evidence of an organic contaminant residue.

The SEM micrographs shown in Figure 6 contain a horizontal etch line about halfway in all sidewall surfaces. The effect of this feature on the sidewall contact profile was
analyzed using FIB cross-sections. It was found that, although contact did not occur along the etch line over a region of ~170 nm in height, (apparent) contact occurred over most of the rest of the 2 μm height of the sidewall surfaces. In the mean contact pressure calculations, an upper-bound apparent contact area that includes the full sidewall height was assumed. In addition, the tapering angle of the sidewall surfaces was found to be very small (~2°).

3.3.3 Sidewall surface microstructure

Further insight into the formation of wear debris was obtained from horizontal TEM cross-section images of the sidewall surfaces. Figure 7 shows typical bright-field and energy-filtered TEM images of oxygen distribution in horizontal cross-sections of protrusions obtained before and after sliding. Table 3.3 shows a comparison between the global silicon-oxide film thickness along the full length of the protrusions of 3 devices measured after sliding and that measured over a similar reference section outside the sliding track of the same device. In both cases, the sampling size represents 5% of the total apparent area of contact. These measurements show an average silicon-oxide thickness of ~9 nm before sliding and a similar thickness after sliding. In addition, the data given in Table III indicate that the silicon-oxide film thickness was not affected by the external force (mean contact pressure) during sliding. These measurements indicate that the silicon-oxide film thickness did not undergo discernible global changes even after \((2–7) \times 10^6\) sliding cycles, despite the fact that wear debris was observed after sliding (Figure 3.6).

3.3.4. Sidewall surface roughness and spectral power density function

In addition to electron microscopy, the evolution of the sidewall surface topography due to surface rubbing was studied with the AFM. The topography of the sidewall surface slid against the protrusion of a micromachine that did not show formation of wear debris was examined (Figure 3.8(a)), and the rms roughness inside and outside the sliding track (obtained as the average of 7 and 9 roughness values, respectively, calculated from \(2 \times 2 \mu m^2\) AFM scan areas) was found equal to 11 ± 3 and 12 ± 2 nm, respectively. Two-dimensional profile traces (Figure 3.8(b)) revealed that only a few raised ridges of the sidewall surfaces participated in sliding, indicating that the real area of contact was several orders of magnitude smaller than the apparent area of contact. This implies that any possible surface alterations were confined at the tops of few raised ridges.

Despite the fact that surface sliding was confined at the highest ridges of the sidewall surfaces, AFM examination of the nanotopographies of ridges inside and outside the sliding track did not reveal any discernible differences. However, PSD plots obtained from \(2 \times 2 \mu m^2\) AFM scans revealed fine topographical differences between the sliding track and the original surface. The PSD intensity at the center region of the sliding track demonstrated a decreasing trend in the low-wavelength range (Figure 9(a)) compared to that of the original surface (Figure 3.9(b)). The decrease in PSD intensity in the low-wavelength range suggests that topography changes due to surface sliding were nanoscopic. This suggests that localized smoothening of the sliding track nanotopography was induced by a nanoscale wear process. To further illustrate that sliding indeed altered the nanotopography, standard deviations of the PSD functions shown in Figures 3.9(a)
and 9(b) plotted in the low-wavelength range (10^{-1}–10^{-2} µm) are contrasted in Figure 9(c). The higher standard deviation of the PSD function of the sliding track than that of the original surface indicates a consistently higher variability in surface feature sizes in the low-wavelength range of the sliding track topography. This is further evidence that a nanoscale wear process (not detectable even at high magnifications) was responsible for the observed nanoscopic surface changes that resulted in the increase of the adhesion force and ultimate cessation of the device movement.

3.4 Discussion

The results presented in the previous section indicate that localized changes in the nanotopography of the sliding surfaces may exhibit a profound effect on the adhesion characteristics of contact-mode MEMS devices operated in high vacuum. Nanoscale surface changes were tracked by observing the evolution of the apparent adhesion force (mean adhesive pressure) with the number of sliding cycles. The observed friction and wear behaviors of the sliding sidewall surfaces can be interpreted in the context of a mechanistic process, based on the measured adhesion force and SEM, TEM, and AFM observations.

3.4.1 Adhesion and device Failure

The increase in adhesion (Figure 3.3) correlated with the increase in sidewall surface friction (Table 3.1), indicating that surface modification occurred during sliding despite the low contact pressure, ultimately causing the device to seize movement. The adhesion increase with contact pressure (Figure 3.3) is attributed to the increase of the real area of contact, and is consistent with similar observations of an earlier study. The increase of the mean contact pressure resulted in a more pronounced increase in adhesive pressure (Figure 3.3). This can be associated with more extensive surface modification due to the intensification of surface interaction with the increase of the contact pressure. This trend is similar to that encountered in traditional tribology, i.e., the wear volume increases with the applied normal load, known as Archard’s wear law.

For most of the tested devices, operation ceased around the neutral position of the sliding shuttle (i.e., approximately zero amplitude), independent of the contact pressure applied during sliding (Figure 3.4). Moreover, the average number of sliding cycles for the device movement to cease (stiction) did not correlate with the mean contact pressure, suggesting a similar failure mechanism in all the experiments and that the highest adhesion (or adhesion increase) predominantly occurred within a relatively small distance from the neutral position. This was confirmed by observing the evolution of the adhesive pressure at various positions along the sliding track (Figure 3.5). The tendency for stiction to occur around the zero-amplitude position can be explained by considering the forces acting on the sliding shuttle at zero- and maximum-amplitude positions. At zero-amplitude position, the restoring force is zero and the tangential force (opposed by the friction force) is the maximum electrostatic force generated by the comb drives of the sliding shuttle. However, maximum restoring and electrostatic forces of equal magnitude are produced at maximum-amplitude position. Because the resulting tangential force in this case is about two times higher than that at zero-amplitude position, it is more likely
for device stiction to occur within the center region of the sliding track, as evidenced from the data of Figure 4.

### 3.4.2 Surface morphology and wear mechanisms

Despite the evidence that nanoscale surface changes were responsible for the seizure of the device operation (Figures 3.3–3.5), it was not possible to identify directly the surface morphology that governed device failure due to stiction. Although wear debris was found on the sidewalls of 42% of the tested devices (Figure 3.6 and Table 3.2), wear particles were not observed with all devices, or they were of extremely small quantities. In addition, changes in surface roughness due to sliding were statistically insignificant, and the thickness of the silicon-oxide surface layer did not show a clear trend to decrease even after \((2–7) \times 10^9\) sliding cycles (Figure 3.7 and Table 3.3). However, these results cannot accurately reveal nanoscale topography changes in a few random locations. For example, local wear of the oxide layer could have been obscured by the inherent variation of its thickness (Table 3.3). The presence of wear debris in 42% of the tested devices observed with the SEM (Figure 3.6 and Table 3.2) and the fact that any discernible changes in surface morphology were not detected by the TEM (Figure 3.7 and Table 3.3) is evidence of a random local wear process operating at the nanoscale. This conclusion is also supported by the decrease of the sliding track’s PSD intensity in the low-wavelength range (Figure 3.9(a)) and its larger variation along the sliding direction (Figure 3.9(c)), indicating that localized surface smoothening occurred by a nanoscale wear process. Furthermore, because both AFM and TEM sample preparation is a destructive process (Figure 3.2), these methods do not allow for direct comparison of exactly the same sidewall area before and after sliding. Therefore, it is extremely difficult to accurately capture changes in nanoscale surface morphology due to a random nanoscale wear process.

An important deduction from the lack of significant average wear of the silicon-oxide layer (Figure 3.7 and Table 3.3) is that wear occurred within the silicon-oxide layer and not in the polysilicon. Because of galvanic oxidation of devices containing gold-coated features during the HF release step of the PolyMUMPs process\(^{38–40}\), these oxide layers are thicker (~9 nm, as shown in Figures 3.7(c) and 3.7(e)) than typical native oxide layers of polysilicon surfaces (typically ~3 nm thick\(^{39}\)). Thus, if the oxide layer was worn off during sliding, it would have regenerated as a ~3-nm-thick native oxide layer. However, this is not the case, as evidenced by Figure 7 and Table III. Because an oxide layer thicker than the native oxide layer and wear debris were found on the sidewall surfaces after sliding, it is concluded that wear was confined at the surface of the thermal silicon-oxide layer. The absence of wear-induced surface pits and scars on the sidewall surfaces (Figure 3.6 and 3.8(a)) indicates that local nanoscale wear of the silicon-oxide layer was the governing process of material removal under the present sliding conditions. In about half of the tested devices, small wear particles (Figure 3.6(d)) were observed to cluster, forming larger agglomerates (Figure 3.6(f)). From a MEMS design point of view, this implies that for contacting polysilicon surfaces under low contact pressure, the tribological behavior of the silicon-oxide layer is of critical importance, even after a large number of sliding cycles. In addition to nanoscale wear of the silicon-oxide layer, the removal and/or smoothening (smearing) of the organic contaminant detected on the
sidewall surfaces (Figures 3.6(a)–3.6(c)) is potentially another mechanism that could have contributed to the increase in sidewall adhesion, leading eventually to device failure due to stiction. Both of these morphological effects are highly localized and differ from device to device; yet, they control the tribological behavior of the sidewall surfaces. This argument is supported by (a) the lack of measurable changes in the average surface morphology, despite the fact that wear debris was found on almost half of the tested devices, (b) the occurrence of sliding contact at only few small surface regions at the top of raised ridges on the sidewall surfaces (Figure 3.8(b)), (c) the spatial variation of adhesion within the sliding track (Figure 3.5), and (d) the relatively large scatter in the operation life of the tested devices (10⁵–10⁶ sliding cycles).

Comparing the results of this study with those of previous studies obtained in vacuum but under higher contact loads (pressures)²⁷,²⁸,³⁴, the devices of the present study show virtually no wear debris and no discernible changes in surface morphology. Despite the lack of clear surface degradation (Figure 3.6, 3.7, and 3.8(a)), it is apparent that surface modification occurred during the several million sliding cycles (Figure 9), leading to seizure of the device movement (Figures 3.3–3.5). These findings suggest that alteration of the sidewall surface morphology and, presumably, physicochemical properties, even under very mild contact conditions, may be the governing factor in the lifetime of contact-mode MEMS devices. The results of this study show that these technologically important low contact pressure conditions may lead to extremely small (nanoscopic) changes in surface morphology, which can only be detected by very detailed characterization, making large-scale routine morphological inspection of these surfaces a non-trivial challenge.

### 3.5 Conclusions

Changes in the adhesion and morphology of sidewall surfaces of polysilicon MEMS devices operated in high vacuum (~10⁻⁵ torr) and under relatively low apparent contact pressure (~1–18 kPa) were examined in the context of mean adhesive pressure, coefficient of friction, and device lifetime measurements as well as microanalysis results. Despite the low apparent contact pressures, sidewall adhesion increased during oscillatory sliding, eventually leading to seizure of the device movement (stiction), mainly within the center region of the sliding track. The adhesion force after the instant of stiction (typically after ~10⁶ sliding cycles) was found to be higher than that of the original surface by a factor of 2–4.

Very small amounts of fine debris (10–140 nm in average size) were observed on the sidewalls of about half of the tested devices without any discernible changes in surface morphology. In addition, organic contaminants from the release process were detected before and after sliding. The average thickness of the silicon-oxide surface layer (~9 nm), measured by cross-section TEM, did not show any decrease as a result of sliding, indicating that a localized nanoscale wear process was active at the surface of the silicon-oxide layer.

AFM studies of the sidewall morphology revealed that only a few raised ridges were in contact during sliding. Power spectrum density analysis showed that sliding induced nanoscale surface smoothening of those ridges. These findings indicate that a random
nanoscale wear process, confined at the surface of the thermal silicon-oxide layer, was responsible for the observed localized smoothening of the surface nanotopography. Despite the lack of any detectable wear features on the sliding surfaces, this nanoscale wear process increased the interfacial adhesion, leading eventually to device stiction. These nanoscale changes in surface morphology indicate that adhesion and wear were determined by only a few nanoscopic contacts that were strongly depended on the local topography. This explains the significant scatter in the number of sliding cycles leading to device failure (stiction) and suggests that even under very low contact pressures and high-vacuum conditions, the lifetime of contact-mode polysilicon MEMS devices may be limited by surface adhesion effects.
References

Table 3.1. Engineering and true friction coefficients, $\mu_e$ and $\mu_t$, respectively, measured before sliding and after device failure (stiction) versus mean adhesive pressure $p_{ad}$.

<table>
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<th>Mean adhesive pressure ($p_{ad}$) (kPa)</th>
<th>Engineering friction coefficient ($\mu_e$)</th>
<th>True friction coefficient ($\mu_t$)</th>
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<td>after stiction</td>
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Table 3.2. Average size of wear debris $d$ detected on the sidewall surfaces of tested devices versus external normal force $F_{ex}$, mean contact pressure $p_{ex}$, and sliding cycles $N$.

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<th>Device number</th>
<th>$F_{ex}$ (nN)</th>
<th>$p_{ex}$ (kPa)</th>
<th>$N$ ($\times 10^4$)</th>
<th>$d$ (nm)</th>
<th>Device number</th>
<th>$F_{ex}$ (nN)</th>
<th>$p_{ex}$ (kPa)</th>
<th>$N$ ($\times 10^4$)</th>
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</tr>
<tr>
<td>HV-4</td>
<td>57.8</td>
<td>1.5</td>
<td>94</td>
<td>25–75</td>
<td>HVIII-4</td>
<td>142</td>
<td>17.8</td>
<td>1335</td>
<td>Ø</td>
</tr>
</tbody>
</table>

Ø = no observable wear debris
Table 3.3. Silicon-oxide film thickness (measured before and after sliding) versus external normal force $F_{ex}$, mean contact pressure $p_{ex}$, and sliding cycles $N$.

<table>
<thead>
<tr>
<th>Device number</th>
<th>$F_{ex}$ (nN)</th>
<th>$p_{ex}$ (kPa)</th>
<th>$N$ ($\times 10^6$)</th>
<th>Silicon-oxide film thickness (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>before sliding</td>
</tr>
<tr>
<td>HI-3</td>
<td>96.5</td>
<td>12.1</td>
<td>2.92</td>
<td>10 ± 2</td>
</tr>
<tr>
<td>HIII-1</td>
<td>57.2</td>
<td>1.5</td>
<td>7.34</td>
<td>7 ± 2</td>
</tr>
<tr>
<td>HIII-2</td>
<td>90.4</td>
<td>5.0</td>
<td>3.53</td>
<td>11 ± 4</td>
</tr>
</tbody>
</table>
Figure 3.1 SEM micrographs of (a) entire test device showing the loading and sliding shuttles and (b) contact region (enclosed within a square frame in (a)) showing a protrusion on the sliding shuttle facing the sidewall surface of the loading shuttle. The viewing angle in (b) is $52^\circ$ from the normal to the top surface of the sliding shuttle. ($F_{\text{ex}}$ = external force; $F_{\text{ad}}$ = adhesion force; $F_{f}$ = friction force)
Figure 3.2 Representative SEM micrographs of the sample preparation process. TEM samples were obtained by (a) FIB cutting of the protrusion from the sliding shuttle, (b) attaching the protrusion to a tungsten micromanipulator, (c,d) transferring the protrusion to a horizontal TEM copper grid, and (e,f) thinning the sample with the protrusion to electron transparency (~100 nm). AFM samples were obtained by (g) transferring the protrusion attached to the tungsten micromanipulator to a vertical TEM copper grid and (h) orienting the sample in the horizontal direction, such that the protrusion sidewall surface to face in the upward direction.
Figure 3.3 Mean adhesive pressure versus sliding cycles for mean contact pressure in the range of 0.98–17.8 kPa. Data points represent mean values of 25 measurements, i.e., 5 measurements per location × 5 different locations on the protrusion sidewall surface of the sliding shuttle. Error bars indicate one standard deviation above and below the corresponding mean value.
**Figure 3.4** Mean contact pressure versus position of seizure of the device movement due to stiction normalized by the maximum distance (half amplitude) measured initially during oscillatory sliding contact. Data points represent mean values of 5 measurements obtained with one device at a given mean contact pressure. Error bars indicate one standard deviation above and below the corresponding mean value.
Figure 3.5 Mean adhesive pressure versus sliding cycles for different positions along the sliding track, obtained by applying different voltages $V_{\text{shear}}$ (from −10 to +10 V) to the comb drive of the sliding shuttle.
Figure 3.6 SEM micrographs of sidewall surfaces showing (a,b) surface contaminants before sliding and (c–f) formation of fine wear debris due to sliding. Contamination particles and wear debris are distinguished by rectangular frames and shown at high magnifications in the insets of figures. Sliding surfaces contain both (a) surface contamination particles and (d–f) ultrafine (10–140 nm) wear debris (bright contrasting particles).
Figure 3.7 Cross-section TEM images of original and sliding sidewall surfaces: (a) image of sidewall surface showing the polysilicon grains, cross-sections of (b) original and (d) sliding sidewall surfaces, and (c,e) energy-filtered images showing the distribution of oxygen in the (c) original and (e) sliding sidewall surfaces shown in (b) and (d), respectively.
Figure 3.8 (a) AFM image of the sidewall surface of a loading shuttle obtained after $1.3 \times 10^7$ sliding cycles, and (b) surface profile corresponding to the dashed line shown in (a) revealing the presence of only a few raised ridges that controlled the real area of contact during sliding.
Figure 3.9 Power spectral densities of (a) sliding and (b) original sidewall surfaces obtained from $2 \times 2 \ \mu \text{m}^2$ AFM scans of different surface locations, and (c) standard deviations of the power spectral densities of the sliding and original sidewall surfaces with spectra shown in (a) and (b), respectively, versus wavelength.
Chapter 4: High-vacuum Adhesion and Friction Properties of Sliding Contact-mode Micromachines

4.1 Introduction

Progress in the development of agile microelectromechanical systems (MEMS) ranging from consumer products to defense and aerospace devices have further increased the importance of reliability and longevity of these microdevices. Effective operation of contact-mode MEMS devices strongly relies on the functionality of contact interfaces. This is extremely challenging because the dominant effect of surface forces at the microscale often leads to significant variability during the operation lifetime. Variation in adhesion and friction surface forces caused by micro/nanoscale wear processes can be detrimental to the efficiency and durability of contact-mode microdevices. Therefore, insight into nano/microscale contact behavior of dynamic MEMS devices is of high technological importance.

Several previous investigations have been devoted to the evolution of interfacial adhesion and friction at contact interfaces of micromachines operated in different environments. However, understanding of the interdependence of changes in adhesion and friction at contact interfaces due to sliding contact is limited, particularly for sidewall surfaces. An electrostatically actuated surface micromachine, specifically designed for tracking changes in adhesion and friction at sidewall contact interfaces under sliding conditions typical of sliding contact-mode MEMS devices, was used to study the evolution of surface damage by tracking the temporal variation of the adhesion and friction force. Changes in the surface morphology caused by the sliding process were examined with a scanning electron microscope (SEM) and an atomic force microscope (AFM). Possible mechanisms resulting in physicochemical surface changes that affected the adhesion and friction characteristics are discussed in the context of surface force measurements and microscopy observations.

4.2 Experimental Procedures

Figure 4.1(a) shows an SEM image of the surface micromachines used in the present study. Micromachines were fabricated by the Polysilicon Multi-User MEMS Processes
(MEMSCAP, Research Triangle Park, NC), which uses n-type doped polycrystalline silicon (polysilicon) as the structural material, phosphosilicate glass (PSG) as the sacrificial layer, and silicon nitride as an electrical isolation layer. Micromachine sidewall surfaces were produced by reactive ion etching (RIE), producing a topography dominated by elevated ridges along the thickness direction. The micromachines were released by etching the PSG layer with HF (49%) for 2.5 min and then drying in a supercritical CO₂ dryer (Autosamdry 815, Tousimis, Rockville, MD). The present test micromachines include two comb-driven shuttles, referred to as the sliding and loading shuttles, which are used to apply a shear (friction) and a normal force at the sidewall contact interface, respectively. More details about the fabrication and operation procedure of this type of surface micromachine can be found in Section 2.2. Figure 4.1(b) shows a high-resolution SEM image of a protrusion extending from the side of the loading shuttle, which is used to control the apparent contact area with the opposed sidewall surface of the sliding shuttle. Surface alignment has been confirmed by white-light interferometry. All tests were performed under controlled environmental conditions of ~10⁻⁵ Torr vacuum pressure, 25°C temperature, and ~0% relative humidity.

Before the onset of oscillatory sliding, the levitation force was used to raise both shuttles to an appropriate height by applying a dc voltage to the push, pull, and shear actuators \( V_{push} = V_{pull} = \Delta V_{shear} = 40 \text{ V} \). Then, the sliding shuttle was set into a free oscillatory motion by applying a sinusoidal waveform of amplitude \( \Delta V_{shear} = 10 \text{ V} \) to the shear actuator, while the loading shuttle was actuated by applying a dc voltage that produced a normal contact force of 112 nN or an external contact pressure \( p_{ex} \) (defined as the ratio of the normal contact force to the apparent sidewall contact area) equal to 14 kPa. The driving shear force was maintained at a frequency of 25 Hz.

Changes in the adhesion and friction characteristics of the sliding sidewall surfaces were examined by periodically interrupting testing to measure the adhesion and friction forces. The adhesion force was measured by first increasing the electrostatic force until initial contact of the sidewall surfaces, then applying \( p_{ex} = 2–14 \text{ kPa} \), and, finally, gradually decreasing the electrostatic force until shuttle separation. The adhesion force at the instant of surface detachment was determined from a force-balance relationship. Similar to the adhesion experiments, an external pressure of 2–14 kPa was first applied to the contact interface and the force generated by one of the shear drives was then gradually increased until the inception of sliding. The static friction force was estimated by a force balance at the onset of sliding. For statistical analysis, each adhesion and friction force measurement was repeated five times. A total of four surface micromachines were tested under identical environmental conditions with \( p_{ex} \) varied in the range of 5–14 kPa using the same experimental procedure. In all plots presented below, data points represent averages of five tests, while error bars indicate one standard deviation above and below the corresponding average value. Despite quantitative differences, all four micromachines showed identical trends.

4.3 Results and Discussion

Figure 4.2 shows a plot of the measured adhesive pressure \( p_{ad} \) as a function of sliding cycles \( N \). Consistent with an earlier study, two phases of different adhesion...
behaviors can be observed – an incubation stage characterized by low and stable $p_{ad}$ and a surface modification stage commencing after $\sim 10^5$ sliding cycles in which $p_{ad}$ was sharply increased, indicating the evolution of surface changes due to oscillatory sliding.

Figure 4.3 shows the variation of the (average) interfacial shear strength $\tau$ (defined as the ratio of measured static friction force to the apparent contact area) with the number of sliding cycles $N$ for the same experiment of Fig. 2. Three stages of distinctly different friction behavior can be distinguished – constant $\tau$ for $N \lesssim 5 \times 10^3$, sharply decreasing $\tau$ for $5 \times 10^3 \lesssim N \lesssim 10^6$, and rapidly increasing $\tau$ for $N > 10^6$.

As reported in previous studies, adhesion forces at MEMS contact interfaces can be higher than externally applied forces by more than an order of magnitude. Thus, to obtain accurate friction measurements, it is necessary to include the contribution of the adhesion force to the total normal force exerted to the sidewall contact interface. Figure 4.4 shows the variation of the interfacial shear strength $\tau$ with the normal contact pressure $p$ (where $p = p_{ex} + p_{ad}$) and sliding cycles $N$. For all sliding stages, $\tau$ exhibits a linear variation with $p$, and the slope of each linear fit represents the true static coefficient of friction $\mu$. Figure 4.5 shows the variation of $\mu$ (corresponding to the experiments of Figs. 2 and 3) with the sliding cycles $N$. Similar to the interfacial shear strength (Fig. 3), the true coefficient of friction also demonstrates a three-stage trend, that is, high and stable $\mu$ in the low-cycle range ($N \lesssim 5 \times 10^3$), rapidly decreasing $\mu$ in the intermediate-cycle range ($5 \times 10^3 \lesssim N \lesssim 10^6$), assuming a minimum value of $\sim 0.32$ after $\sim 10^6$ cycles, and gradually increasing $\mu$ in the high-cycle range ($N > 10^6$), reaching a steady-state value of $\sim 0.37$ after $\sim 10^7$ cycles.

The experimental results shown in Figures 4.2, 4.3, and 4.5 reveal the existence of an initial sliding stage of relative low $p_{ad}$ and high $\tau$ and $\mu$, an intermediate stage characterized by opposite trends, and a final stage of dramatically intensifying $p_{ad}$ and relatively lower and less variant $\tau$ and $\mu$. These trends can be attributed to physical and chemical surface modification processes promoted by oscillatory sliding contact. Despite the profound changes in $p_{ad}$, $\tau$, and $\mu$, SEM images did not reveal any topography changes or wear debris formation on the sliding sidewall surfaces, consistent with previous studies of dry and lubricated polysilicon micromachines tested under dynamic contact conditions. To obtain further insight into possible physical changes that could have affected micromachine adhesion and friction behavior, specimens prepared by a focused ion beam method were examined with the AFM. Figure 4.6(a) shows an AFM image of a sidewall surface after $\sim 1.3 \times 10^7$ sliding cycles. The surface morphology of the sliding track is similar to that of the original topography. As mentioned earlier, sidewall surfaces are dominated by vertical etch striations (ridges), which are intrinsic surface feature of the RIE process. The cross-sectional profile shown in Figure 4.6(b), corresponding to the white dashed line shown in Figure 4.6(a), suggests that only a few raised ridges were in sliding contact, implying real contact area several orders of magnitude smaller than the apparent contact area. High-resolution AFM area scans revealed that surface regions inside [Figure 4.6(c)] and outside [Figure 4.6(d)] of the sliding track exhibited similar topographies. The root-mean-square (rms) roughness calculated as the average of seven and nine roughness values obtained from $2 \times 2 \mu m^2$ area scans inside and outside the sliding track was found equal to $11 \pm 3$ and $12 \pm 2$ nm, respectively. These statistically indifferent roughness values suggest that topographical changes must have occurred only at the top of the highest ridges. To determine if this was indeed the case, only the highest peak within a $125 \times 125 \mu m^2$ area of $1 \times 1 \mu m^2$ AFM scans
was used to calculate the rms roughness. The average rms roughness calculated from 16 such roughness measurements obtained inside and outside the sliding track was found equal to 3.3 ± 1.0 and 5.0 ± 1.9 nm, respectively. The ~24% lower rms roughness inside the sliding track suggests that localized surface smoothening of the tallest ridges occurred by a nanoscale wear process. Oscillatory sliding may lead to plastic deformation and/or high-cycle contact fatigue of interacting asperities at the top of the highest ridges, producing fluctuations in the surface contact intimacy and, in turn, changes in the adhesion and friction properties.

In addition to physical surface changes, surface chemical modification may also affect adhesion and friction behavior. The adhesion force depends on the surface energy of the interacting sidewalls and the interfacial surface energy. The polysilicon sidewall surfaces were covered by a SiO₂ film and contaminant residue produced by the supercritical point drying-release process, which has a surface energy less than that of SiO₂. Because high-vacuum sliding of polysilicon sidewall surfaces under contact pressures similar to those of the present study was not found to result in thinning of the SiO₂ film,¹⁰ it may be interpreted that the SiO₂ film was not worn off during testing. Therefore, the pronounced increase of \( p_{ad} \) after sliding for \( \sim 10^5 \) cycles may be attributed to the removal of the contaminant residue (and any airborne contaminant adsorbent) from the sliding track, leading to the increase of the work of adhesion.

According to classical friction theory,¹⁵ the friction force mainly consists of three friction (shear) force components representing asperity deformation, surface adhesion, and plowing by wear debris. Considering that wear debris and plowing marks could not be detected on the sidewall surfaces even after \( \sim 10^7 \) sliding cycles, the effect of plowing friction was negligible. Therefore, the evolution of the adhesion and friction characteristics may be attributed to adhesion and asperity deformation mechanisms. Consequently, high \( \tau \) and \( \mu \) during the initial stage of sliding (\( N \approx 5 \times 10^3 \) cycles) may be related to the dominant effect of asperity-asperity interactions (incubation stage). After this stage of sliding, both \( \tau \) and \( \mu \) decreased dramatically because nanoscale surface smoothening reduced asperity-asperity interaction. However, surface smoothening in conjunction with the removal of surface contaminants increased the adhesion friction component, resulting in the subsequent increase of \( \tau \) and \( \mu \). The competing effects of decreasing asperity deformation and increasing surface adhesion eventually reached an equilibrium stage, resulting in stable state-state friction behavior.

### 4.4 Conclusions

Summarizing, a special polysilicon surface micromachine suitable for studying adhesion and friction of sidewall surfaces under controlled loading and environmental conditions was used to investigate the variation of the adhesive pressure, interfacial shear strength, and static coefficient of friction with the number of sliding cycles. The results of this study revealed a transition from an incubation stage of low and stable adhesion and high friction (interfacial shear strength) to a surface modification stage of high adhesion and relatively low steady-state friction. SEM and AFM observations indicated that oscillatory sliding induced localized nanotopography changes. Changes in the adhesion and friction characteristics were associated with nanoscale surface smoothening and the removal of
surface contaminants, resulting from the competing effects of nanoscale adhesion and asperity deformation mechanisms.
References

Figure 4.1 SEM images of (a) sliding micromachine including the electrical wiring diagram used for electrostatic comb-drive actuation and (b) contact region showing a protrusion extending from sliding shuttle, used to control the apparent contact area with the opposed sidewall surface of the loading shuttle.
Figure 4.2 Adhesive pressure $p_{ad}$ versus sliding cycles $N$ for $p_{ex} = 14$ kPa.
Figure 4.3 Interfacial shear strength $\tau$ versus sliding cycles $N$ for $p_{ex} = 14$ kPa.
Figure 4.4 Interfacial shear strength $\tau$ versus normal contact pressure $p$ and sliding cycles $N$. 
Figure 4.5 Static friction coefficient $\mu$ versus sliding cycles $N$ for $p_{ex} = 14$ kPa.
Figure 4.6 (a) AFM image of the sidewall surface of a loading shuttle obtained after \( \sim 1.3 \times 10^7 \) sliding cycles showing portions of the sliding track and the original surface, (b) surface profile corresponding to the white dashed line shown in (a), and (c) and (d) representative AFM images of surface areas inside and outside the sliding track, respectively.
Chapter 5: Evolution of Sidewall Adhesion in Surface Micromachines due to Repetitive Impact Loading

5.1 Introduction

Microelectromechanical systems (MEMS) are used in a wide range of applications including sensors, actuators, biomedical devices, projection systems, and photovoltaic structures. Advances in the fabrication of agile MEMS devices have increased the demand for higher reliability and longer operation lifetime. At microscopic levels, surface forces may exceed inertial (bulk) forces by several orders of magnitude due to the increase of the surface-to-volume ratio. Therefore adhesion is a critical reliability issue, influencing significantly the performance of dynamic microsystems with contact interfaces.

Several studies have been devoted to the measurement of surface adhesion forces at microdevice interfaces and the implementation of approaches for minimizing undesirable adhesion effects. For example, Mastrangelo and Hsu examined permanent adhesion (stiction) of microcantilevers to the underlying substrate and determined the critical beam length for stiction. Houston et al. and de Boer and Michalske investigated in-use adhesion of microstructures and used fracture mechanics principles to calculate the adhesion energy of the contact interface. The former studies involved contact of in-plane surfaces with topographies significantly different from those of sidewall surfaces due to fundamental differences in corresponding fabrication processes. Timpe and Komvopoulos fabricated a surface micromachine specifically designed for studying sidewall adhesion and developed a procedure for predicting the contributions from both contacting and non-contacting asperities to the total adhesion force at the instant of sidewall surface separation.

Surface adhesion can be minimized by various physical and chemical modification techniques. For example, Alley et al. used a texturing method that roughens micromachine surfaces to reduce the true contact area and, in turn, the magnitude of adhesion forces. Komvopoulos and Houston et al. showed that deposition of self-assembled monolayers consisting of low surface energy substances is an effective means of preventing micromachine stiction.

Reliable operation of various contact-mode MEMS devices, such as microswitches, micromirrors, and microgears, depends on the tribological properties of contact interfaces. Surface texture modification due to repetitive dynamic contact (impact) may cause the functionality and efficiency of these microdevices to exhibit significant variability. Excessive adhesion forces can be generated at micromachine interfaces as a
consequence of changes in the surface chemical behavior\textsuperscript{4} and surface topography (roughness)\textsuperscript{9} and the build-up of surface charges.\textsuperscript{10} The increase of the adhesion force due to these effects can be detrimental to the device functionality and efficiency, and is often the main limiting factor of device lifetime. Thus, knowledge of the mechanisms responsible for the modification of surface properties in contact-mode MEMS devices is essential for determining the effective operation lifetime.

Several earlier investigations were devoted to the study of surface modification resulting from repetitive dynamic contact. For instance, Philippine \textit{et al.}\textsuperscript{11} studied the evolution of interfacial adhesion at micromachine contact interfaces operated in vacuum and observed a transition from initially low and stable adhesion to rapidly intensifying adhesion. Hook \textit{et al.}\textsuperscript{12} examined normal contact of polycrystalline silicon (polysilicon) sidewall interfaces covered by a self-assembled monolayer in a dry atmosphere and reported a twofold increase of the adhesion force with the commencement of surface degradation.

Despite important insight into surface damage due to repetitive impact in contact-mode MEMS devices obtained from previous studies, very little is known about the effects of contact pressure and environmental conditions on the surface modification and lifetime of devices subjected to cyclic impact loading. Therefore, the objective of this investigation was to use an electrostatically actuated surface micromachine, specifically designed for impact testing of sidewall surfaces, to study the effects of the externally applied contact pressure, ambient pressure, and relative humidity on the variation of the interfacial adhesion force with impact cycles.

### 5.2 Experimental Procedures

Figure 5.1 shows a scanning electron microscope (SEM) image of the polysilicon micromachine used in this study. Specific fabrication and operation details can be found in Section 2.2. An extension (protrusion) on the side of one of the shuttles was used to control the apparent contact area between the sidewall surfaces. Figure 5.2 shows a SEM image of the protrusion at the side of shuttle (2) used to establish contact with the sidewall surface of shuttle (1). The two shuttles (initially separated by a distance of 5 μm) were brought into contact along their sidewall surfaces by activating the comb drives. Surface alignment in the present design has been confirmed by white-light interferometry.\textsuperscript{8} Symmetric voltage waveforms were applied during testing to ensure vertical alignment of the shuttles in the presence of levitation forces.\textsuperscript{16}

Previous studies have demonstrated that changes in physical and chemical surface behavior and formation of wear debris can be directly correlated with the evolution of the interfacial adhesion force.\textsuperscript{18} Thus the measured adhesion force was used as an \textit{in situ} indicator of the surface modification occurring during repetitive impact loading. The experimental procedure for impact testing under controlled loading and environmental conditions comprised several steps, which are described below.

First, the adhesion force of as-processed sidewall surfaces was measured to establish a reference baseline for the micromachine adhesion characteristics. This was accomplished by gradually increasing the electrostatic forces to slowly push the two shuttles into contact along their sidewalls, applying an external electrostatic force equal
to the maximum contact force to be applied during impact testing, and, finally, decreasing the electrostatic forces in a quasi-static manner up to the instant of surface separation. The adhesion force $F_{\text{ad}}$ at that instant was determined from the force-balance relationship

$$ F_{\text{ad}} = F_r - F_{\text{el}} \tag{5.1} $$

where $F_{\text{el}}$ is the electrostatic force [Eq. (2.2)] at the instant of sidewall surface separation, and $F_r$ is the mechanical restoring force [Eq. (2.1)] generated by the suspension system. For statistical analysis, five measurements of the adhesion force were obtained after impact testing for specific durations. The effect of the very small number of quasi-static contact cycles applied during the measurement of the adhesion force on the impact behavior of the micromachines was considered to be negligible.

In general, the interfacial force includes contributions from various attractive (adhesion) and repulsive forces, such as solid bridging, electrostatic, van der Waals, capillary, and deformation forces. Because of the specifics of the fabrication process and the experimental scheme used in this study, solid bridging, electrostatic forces, and repulsive forces due to asperity deformation did not arise in the present experiments. Therefore, under the experimental conditions of this study, the measured adhesion force was mainly due to van der Waals forces (all testing environments) and capillary forces (only for testing in a humid atmosphere).

After establishing a baseline value of the adhesion force, the micromachine was operated in repetitive impact mode. To initiate impact testing, the levitation force was used to raise both shuttles to an appropriate height by increasing the voltage applied to both actuators, i.e., $V_{\text{push}} = V_{\text{pull}} = 35–40$ V. Then, the two shuttles were moved closer by decreasing $V_{\text{pull}}$, which increased the resultant electrostatic force applied to each shuttle [Eq. (2.1)]. Finally, repetitive impact was initiated by applying a 50 Hz sinusoidal waveform of amplitude $\Delta V_{\text{push}} = 8.5–10$ V. The externally applied maximum load $F_{\text{ex}}^{\text{max}}$ was calculated from the force-balance relationship

$$ F_{\text{ex}}^{\text{max}} = F_{\text{push}}^{\text{max}} - F_r , \tag{5.2} $$

where $F_{\text{push}}^{\text{max}}$ is the maximum electrostatic force generated by the comb-drive actuators of the shuttles [Eq. (2.2)] in the push direction, and was found to be in the range of 192–362 nN.

The maximum pull-off force available $F_{\text{po}}^{\text{max}}$ by the micromachine during impact testing is given by

$$ F_{\text{po}}^{\text{max}} = F_{\text{pull}}^{\text{max}} + F_r , \tag{5.3} $$

where $F_{\text{pull}}^{\text{max}}$ is the maximum electrostatic force generated by the comb-drive actuators of the shuttles [Eq. (2.2)] in the pull direction. To determine the externally applied maximum contact pressure $p_{\text{ex}}^{\text{max}}$, the forces in Eq. (5.2) were normalized by the apparent contact area $A_x$, controlled by the size of the protrusion extending from shuttle (2) (Figure 5.2), i.e.,

$$ p_{\text{ex}}^{\text{max}} = \frac{F_{\text{ex}}^{\text{max}}}{A_x} \tag{5.4} $$
Observation of the amplitude versus frequency response of the micromachine used in this study revealed that the displacement during impact testing was accurately determined from a static analysis. Based on this observation, the calculated impact velocity was found to be in the range of 492–791 µm/s.

To indirectly assess any changes in the sidewall surface characteristics, impact testing was interrupted periodically to measure the adhesion force, using the quasi-static procedure described above. Five measurements of the adhesion force were obtained after each interruption. After separating the sidewall surfaces of the permanently adhered micromachines by gradually increasing the electrostatic pull forces (i.e., \( V_{\text{pull}} \)) of the shuttles, the load was applied again to ensure the occurrence of stiction and the adhesion force was measured. To avoid complications from micromachine-to-micromachine variations in the adhesion measurements, all of the adhesion force data were normalized by the adhesion force measured before the initiation of impact testing \( F_{ad}^0 \), i.e.,

\[
F_{ad} = \frac{F_{ad}}{F_{ad}^0}
\]

The above experimental procedure was used to elucidate the evolution of surface modification at sidewall contact interfaces subjected to repetitive impact loading under different conditions of maximum contact pressure, ambient pressure, and relative humidity. Such capability is important because microscale adhesive behavior is strongly affected by variations in surface separation (contact pressure effect), testing environment (ambient pressure effect), and formation of liquid meniscus (humidity effect). All of the experiments were conducted in a custom-made multi-probe vacuum station (MMR Technologies) mounted on a vibration isolation table (Newport Electronics) as described in Section 2.3.

5.3 Results and Discussion

5.3.1 Effect of maximum contact pressure

Results from experiments performed in vacuum under different maximum contact pressures are presented first to illustrate the effect of impact intensity on the evolution of sidewall adhesion. In these tests, the chamber pressure, temperature, and relative humidity were fixed at 10^5 Torr, 25°C, and ~0%, respectively. As mentioned earlier, only van der Waals forces contributed to the adhesion force measured under these experimental conditions. The adhesion force between the protrusion and the opposed sidewall surface of eight micromachines was measured as a function of number of impact cycles and external maximum contact pressure.

Figure 5.3 shows the in-vacuum variation of the normalized adhesion force \( F_{ad} \) with impact cycles for \( p_{\text{ext}}^{\text{max}} \) in the range of 1.96–20.08 kPa. The results reveal the existence of two phases of different adhesion behaviors, consistent with a previous study of Philippine et al. The first phase, referred to as the run-in phase, displays relatively low and stable adhesion, suggesting insignificant modification of the as-fabricated sidewall surfaces. This phase was observed with all micromachines up to ~10^5 impact cycles. After the run-in phase, the adhesion force increased rapidly, indicating significant physicochemical changes on the sidewall surfaces due to repetitive impact. Therefore,
this phase is referred to as the surface modification phase. $F_{ad}$ demonstrates a nonlinear dependence on the number of impact cycles $N$, which follows a relationship of the form

$$\bar{F}_{ad} = 1 + aN^b$$

(5.6)

where $a$ and $b$ are constants. Best-fit curves following Eq. (5.6) are also shown in Fig. 5.3. Table 5.1 gives the fitted values of $a$ and $b$ in terms of $P_{ex}^{\text{max}}$ and the correlation coefficient $R^2$. The increase of $b$ with $P_{ex}^{\text{max}}$ implies shorter operation lifetime under impact conditions of high contact force (pressure). This is attributed to more extensive surface modification under intense impact conditions, leading to the increase of the adhesion force and, in turn, accelerated micromachine failure due to stiction.

The observed changes in the adhesion behavior can be related to both physical and chemical modification of the micromachine sidewall surfaces. The variation of the adhesion force shown in Fig. 5.3 is attributed to the combined effects of physicochemical processes that produced localized surface changes. Possible physical effects include fluctuations in surface contact intimacy due to plastic deformation and/or fracture of interacting asperities at top of the raised ridges of sidewall surfaces. The decrease of the mean surface separation distance due to the increase of the contact intimacy increased the real area of contact and, in turn, the adhesion force. Despite the profound increase of the adhesion force, high-magnification SEM did not show any discernible topography changes or formation of wear debris on the sidewall surfaces. Since SEM cannot reveal changes in surface features and wear debris of sizes less than ~50 nm, it may be argued that the scale of surface topography changes due to repetitive impact was less than ~50 nm. This observation is consistent with previous studies of dry and lubricated polysilicon micromachines, in which changes in the surface topography could not be detected even with high-magnification SEM, despite the significant variation of the interfacial adhesion force.

In addition to physical changes associated with the surface topography, it is likely that surface chemical modification also contributed to the evolution of the adhesion force with impact cycles shown in Fig. 3. The adhesion force depends on the work of adhesion, which is a function of the surface energies of the interacting sidewalls and the interfacial surface energy. The polysilicon surfaces were covered by an oxide film and islands of an organic residue (intrinsic of the carbon dioxide supercritical point drying process), which has a surface energy lower than that of silicon oxide. It is believed that the removal of this organic residue as a result of repetitive impact increased the work of adhesion, resulting in the increase of the adhesion force. In a previous investigation of Timpe et al. dealing with polysilicon sidewall surfaces sliding in vacuum under contact loads (pressures) in the same range with that of the present study, it was observed that sliding did not cause any discernible thinning of the oxide layer. In general, sliding is expected to produce more extensive surface modification than normal contact under similar contact loads due to the added effect of shear traction. Thus, in view of the findings of that study, it may be inferred that the oxide film on the sidewall surfaces was not ruptured during impact loading. Therefore, the increase of the adhesion force in the surface modification phase may be attributed to the removal of the organic residue from surface regions where contact occurred, leading to the increase of the work of adhesion. As a consequence, the increase of the surface affinity (compatibility) intensified the adhesion force.
The micromachine failure (stiction) region shown in Fig. 5.3 was determined by considering the maximum pull-off force $F_{\text{po}}^{\text{max}}$ of the micromachines. Stiction occurred when the curve of the normalized adhesion force intersected the lower bound of the stiction region ($F_{\text{ad}} \approx 11.4$), implying the development of an adhesion force higher than the maximum pull-off force of the micromachine. Stiction occurred for $p_{\text{ex}}^{\text{max}} = 11.78$, 14.60, and 20.08 kPa, because the adhesion force ultimately reached a value within the stiction region only for relatively high contact pressure conditions. For low contact pressure (i.e., $p_{\text{ex}}^{\text{max}} = 1.96$ and 5.19 kPa), the experiments were halted after testing for $\approx 2 \times 10^7$ impact cycles due to the excessive duration of testing and in the absence of any indication that the data might deviate from the observed experimental trend, described by Eq. (8). The critical number of impact cycles for micromachine stiction in these experiments was obtained as the intersection of extrapolated curves of $F_{\text{ad}}$ [Eq. (8)] and the lower bound of the stiction region (i.e., $F_{\text{ad}} \approx 11.4$).

Figure 5.4 shows the variation of the externally applied maximum contact pressure $p_{\text{ex}}^{\text{max}}$ with the number of impact cycles resulting in micromachine stiction $N_s$. The figure includes data from eight impact experiments for $p_{\text{ex}}^{\text{max}}$ in the range of 1.96–20.08 kPa. Five of the micromachines tested in the pressure range of 11.78–20.08 kPa failed due to stiction after $3 \times 10^7$–$3 \times 10^8$ impact cycles, because the adhesion force exceeded the maximum pull-off force of those micromachines. The three micromachines tested in the pressure range of 1.96–7.34 kPa did not fail even after testing for a very long time (>10^8 impact cycles). Therefore, for these tests, $N_s$ was predicted by the extrapolation method described above. Hence, for $F_{\text{ad}} \approx 11.4$ and $p_{\text{ex}}^{\text{max}}$ in the range of 1.96–7.34 kPa, $N_s$ was calculated to be in the range of $3 \times 10^8$–$8 \times 10^{10}$. Figure 4 shows a rapid increase of $N_s$ with decreasing $p_{\text{ex}}^{\text{max}}$, following a power-law relationship of the form

$$p_{\text{ex}}^{\text{max}} = AN_s^B$$

(5.7)

where $A$ and $B$ are constants. The curve shown in Fig. 4 is a least-squares fit ($R^2 = 0.84$) through all the data. From this fit and Eq. (9), it was found that $A = 801.9$ and $B = -0.244$. This method provides another means of predicting micromachine failure due to excessive interfacial adhesion. Because the sidewall surfaces were subjected to repetitive impact loading, it is likely that high-cycle contact fatigue of asperities residing on the raised ridges of the sidewall surfaces resulted in nanoscale smoothening, as observed with similar contact loads in a previous study, contributing to the increase of the adhesion force, ultimately leading to micromachine stiction. Therefore, the data shown in Fig. 5.4 were fitted with Eq. (5.7) because high-cycle fatigue typically follows an exponential stress-life relationship.

### 5.3.2 Effect of environmental conditions

Results from impact testing performed under different ambient pressures $P$ and relative humidity RH levels are presented next to illustrate the effect of environmental conditions on the evolution of sidewall adhesion due to repetitive impact. Figure 5.5 shows the variation of the normalized adhesion force $F_{\text{ad}}$ with the number of impact cycles $N$ for impact testing under conditions of room temperature (~25°C), $p_{\text{ex}}^{\text{max}} = 5.45$
and 25.83 kPa, $P = 760$ and $10^{-5}$ Torr, and RH = 0 and 100%. Data points represent average values of measurements obtained from five experiments performed under identical testing conditions. All experiments revealed a run-in phase during the initial stage of testing (up to $\sim 10^5$ impact cycles). Similar to in-vacuum testing (Fig. 5.3), the evolution of the adhesion force with the impact cycles may be attributed to both physical and chemical surface effects. High-magnification SEM images of sidewall surfaces obtained after testing at different environments did not show any distinguishable topography changes or formation of wear debris. Therefore, for the reasons mentioned earlier, the variation of $F_{ad}$ with $N$ is attributed to the increase of both the real contact area due to nanoscale surface smoothening (not captured under the SEM) and the work of adhesion due to the removal of the organic residue and, possibly, other airborne contaminants adsorbed onto the micromachine surfaces before testing.

Both conditions of low and high $p_{\text{ex max}}$ yielded similar trends, i.e., low and stable adhesion initially (run-in phase) followed by rapidly intensifying adhesion (surface modification phase). The effect of environmental conditions on the adhesion behavior was only apparent in the surface modification phase (i.e., above $\sim 10^5$ impact cycles), where adhesion intensified in the order of dry atmosphere (RH = 0%), high-humidity atmosphere (RH = 100%), and vacuum. For dry atmosphere, only van der Waals forces contributed to the adhesion force, whereas for high-humidity atmosphere, the adhesion force included contributions from both van der Waals and capillary forces. After $\sim 3 \times 10^7$ cycles of impact testing under $p_{\text{ex max}} = 5.45$ kPa, the adhesion force in vacuum increased by a factor of $\sim 5$ compared to $\sim 1.8$ for high-humidity atmosphere [Fig. 5.5(a)]. This dramatic difference in adhesion behavior was further exacerbated by the increase of the externally applied maximum contact pressure. Thus, after $\sim 1 \times 10^7$ impact cycles of testing under $p_{\text{ex max}} = 25.83$ kPa, the adhesion force in vacuum increased by a factor of $\sim 15.9$ compared to $\sim 1.4$ for high-humidity atmosphere [Fig. 5.5(b)].

Another important observation is that while the adhesion force increased steadily in dry atmosphere, it increased sharply exhibiting vigorous fluctuations in the presence of high humidity. This behavior can be interpreted by considering the contribution capillary forces to the adhesion force in high-humidity atmospheres. Capillary forces are due to liquid meniscus forming between proximity surfaces. Thus, the fluctuation of the adhesion force in the humid environment may be attributed to meniscus instabilities, which affected the magnitude of the capillary force.

The observed differences in the evolution of sidewall adhesion may be related to several factors. First, air damping may have decreased the work dissipated at the contact interface during impact testing under atmospheric conditions. Since air damping depends on the surface area of moving parts, damping effects become more significant at the microscale because of the increase of surface-to-volume ratio. During repetitive impact testing, the shuttles were subjected to back and forth oscillatory motion. Under atmospheric conditions, air damping in the narrow gaps of the fast moving shuttles reduced the impact intensity. This decreased the extent of surface modification, resulting in less pronounced increase of the adhesion force under atmospheric conditions than vacuum. Second, it is likely that the surface chemical behavior was affected by the testing environment. For example, the decrease of the work of adhesion during testing in
ambient air due to physisorption of monolayer films\textsuperscript{20} yielded a less pronounced increase of the adhesion force in the surface modification phase, as shown in Fig. 5. Replenishment of contaminant films during repetitive impact is difficult or impossible under vacuum compared to atmospheric conditions.\textsuperscript{21} Third, humidity intensified the adhesion force by promoting meniscus formation, resulting in the development of capillary forces. This can be observed by comparing the adhesion force responses for RH = 0 and 100\% and fixed $P$ and $p_{\text{ex}}^{\text{max}}$ shown in Fig. 5.5. As mentioned earlier, nanoscale surface smoothening and removal of low surface energy adsorbents and/or other contaminants (e.g., organic residue) during repetitive impact loading reduced the interfacial gap and increased the surface energy, respectively. Both of these effects favored meniscus formation over a larger fraction of the real contact area, increasing the capillary force and, in turn, the adhesion force at the sidewall interface. Fourth, it is well known that stress-assisted oxidation cracking of polysilicon occurs in high-humidity atmospheres.\textsuperscript{22} It is possible that this process also contributed to localized smoothening of the surface nanotopography, increasing the real area of contact and, consequently, the adhesion force. This is consistent with the much higher adhesion forces obtained under high-humidity impact conditions (Fig. 5.5).

5.4 Conclusions

A surface micromachine designed for studying the evolution of sidewall adhesion due to repetitive impact loading was fabricated and tested under different contact loads and environmental conditions. Based on the obtained results and discussion, the following main conclusions can be drawn from this study.

(1) During repetitive impact in vacuum, the interfacial adhesion force demonstrated a nonlinear dependence on impact cycles, characterized by the transition from low and stable adhesion (run-in phase) to rapidly intensifying adhesion (surface modification phase).

(2) A method for predicting micromachine failure (stiction) due to the development of an excessive interfacial adhesion force was derived based on the observed experimental trend. For in-vacuum operation, the micromachine lifetime increased nonlinearly with the decrease of the maximum contact pressure.

(3) The absence of any topography changes or wear debris from high-magnification microscopy images of the tested surfaces suggests that nanoscale wear was a likely precursor of micromachine failure due to stiction.

(4) Nanoscale surface modification comprised both physical and chemical effects. The increase of the real area of contact due to nanoscale smoothening (physical effect) and the work of adhesion due to the removal of contaminant films (chemical effect) intensified the interfacial adhesion force, eventually leading to the cessation of micromachine operation (stiction).

(5) The more pronounced increase of the adhesion force under vacuum than atmospheric conditions suggests a greater likelihood of micromachine failure due to stiction for operation in vacuum.
References

TABLE 5.1. Adhesion evolution fitting parameters versus maximum external pressure.

<table>
<thead>
<tr>
<th>Maximum external pressure $p_{ex}^{max}$ (kPa)</th>
<th>Fitting parameters</th>
<th>Correlation coefficient $R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.96</td>
<td>$6.49 \times 10^{-3}$</td>
<td>0.37</td>
</tr>
<tr>
<td>5.46</td>
<td>$4.60 \times 10^{-3}$</td>
<td>0.39</td>
</tr>
<tr>
<td>11.78</td>
<td>$9.93 \times 10^{-10}$</td>
<td>1.38</td>
</tr>
<tr>
<td>14.60</td>
<td>$1.41 \times 10^{-9}$</td>
<td>1.44</td>
</tr>
<tr>
<td>20.08</td>
<td>$1.41 \times 10^{-10}$</td>
<td>1.66</td>
</tr>
</tbody>
</table>
Figure 5.1 SEM image of a surface micromachine designed for repetitive dynamic contact (impact) between the sidewall surfaces under controlled loading and environmental conditions.
Figure 5.2 High-magnification SEM image showing a protrusion extending from the side of shuttle (2), used to control the apparent contact area with the sidewall surface of shuttle (1).
**Figure 5.3** In-vacuum variation of normalized adhesion force $F_{ad}$ with impact cycles $N$ for externally applied maximum contact pressure $p^\text{max}\text{ex}$ in the range of 1.96–20.08 kPa. (Curves represent best fits through corresponding data. Error bars equal to one standard deviation above and below the corresponding mean values are invisible because they are smaller than the symbol size.)
Figure 5.4 In-vacuum variation of externally applied maximum contact pressure $p_{\text{ex}}^{\text{max}}$ with impact cycles resulting in micromachine failure due to stiction $N_s$. 

\[ P = 10^{-5} \text{ Torr} \]
\[ \text{RH} = 0\% \]

- Measured
- Estimated

Maximum contact pressure, $p_{\text{ex}}^{\text{max}}$ (kPa)
Cycles to stiction, $N_s$
Figure 5.5 Variation of normalized adhesion force $F_{ad}$ with impact cycles $N$ under different environmental conditions for externally applied maximum contact pressure $p_{\text{ex}}^{\text{max}}$ equal to (a) 5.45 kPa and (b) 25.83 kPa. (Error bars equal to one standard deviation above and below the corresponding mean values are invisible because they are smaller than the symbol size.)
Chapter 6: The Effect of Impact Velocity on Interfacial Adhesion of Contact-mode Surface micromachines

6.1 Introduction

The development of more commercial applications of microelectromechanical systems (MEMS) has increased the importance of reliability and lifetime. Evolution of adhesive surface forces in various contact-mode MEMS applications\textsuperscript{1–3} influences profoundly durability and efficiency. High adhesion forces may arise at micromachine contact surfaces as a result of changes in surface topography and chemical characteristics during operation.\textsuperscript{4,5} The occurrence of high adhesion forces due to these effects could be detrimental to the device longevity. Knowledge of the underlying mechanisms affecting adhesion at micromachine interfaces is therefore of paramount importance to the operation of dynamic microdevices. Despite significant insight into the role of adhesion in MEMS devices obtained from earlier studies, relatively less is known about micromachine adhesive behavior under dynamic contact conditions. Previous investigations\textsuperscript{6,7} have revealed a strong dependence of micromachine lifetime on contact pressure and environmental conditions, such as ambient pressure and relative humidity. However, the effect of impact velocity on the adhesive behavior of contact-mode micromachines is sparse and mostly empirical.

Operation of several MEMS devices\textsuperscript{8,9} relies on high-frequency dynamic contact (impact) of micromachine components. A contact interface may encounter dynamic loads under a wide range of impact velocity. Very little is known about the effects of impact velocity on the evolution of interfacial adhesion and, more importantly, its correlation to device lifetime. Consequently, the objective of the work presented in this chapter was to use electrostatically actuated surface micromachines specifically designed for impact testing to study the effect of impact velocity on interfacial adhesion and its evolution with impact cycles.
6.2 Experimental Procedures

Figure 6.1(a) shows a scanning electron microscope (SEM) image of the polycrystalline (polysilicon) surface micromachine fabricated by the Polysilicon Multi-User MEMS Processes (MEMSCAP, Research Triangle Park, NC) for this study. This fabrication process uses reactive ion etching to form the sidewall surfaces, characterized by elevated ridges along the thickness direction. The suspended portion of the test micromachine was released by etching the sacrificial oxide with HF (49%) for 2.5 min, followed by supercritical CO₂ drying (Autosamdri 815, Tousimis, Rockville, MD). The micromachine includes two symmetric shuttles supported by double-folded flexure suspension systems that can be actuated by a set of parallel electrostatic comb-drive actuators. The shuttles are pushed into contact by activating the push comb drives or pulled away from each other by activating the pull comb drives. More details about the fabrication and operation procedures of this test micromachine can be found in Section 2.2. Figure 6.1(b) shows a SEM image of a protrusion at the side of shuttle (2) used to control the apparent contact area with the sidewall surface of shuttle (1). Surface alignment has been confirmed by white-light interferometry. Symmetric voltage waveforms were applied during testing to ensure vertical alignment of the shuttles in the presence of levitation forces. All tests were performed under controlled environmental conditions (~10⁻⁵ Torr vacuum pressure, 25°C chip temperature, and ~0% relative humidity).

Before impact testing, the levitation force was used to raise both shuttles to an appropriate height by applying a voltage to both push and pull actuators (V_{push} = V_{pull} = 40 V). Then, the shuttles were moved closer to each other by decreasing V_{pull} from 40 V to 30–35 V. This increased the electrostatic force applied to each shuttle, decreasing the shuttle-to-shuttle distance x to 0.08–2.62 mm. Subsequently, repetitive dynamic contact was initiated by applying a sinusoidal waveform of amplitude ΔV_{push} = 7–10 V. Impact testing was performed for driving frequency f in the range of 1–1000 Hz. Examination of the oscillation amplitude versus frequency response showed that reasonably accurate estimates of the shuttle displacement x can be obtained from a static force analysis. Thus, the impact velocity v corresponding to the above ranges of x and f was found to be between 0.01 and 12.92 mm/s. Changes in sidewall surface characteristics were correlated to adhesion force measurements obtained by periodically interrupting impact testing to obtain quasi-static measurements of the adhesion force. The adhesion force was measured by increasing the electrostatic forces up to the instant of initial contact between the shuttle sidewall surfaces, applying an external force at the established contact interface, performing impact testing for 10 cycles at a given impact velocity, and, finally, decreasing the electrostatic force gradually until the commencement of shuttle separation. The adhesion force at the instant of surface detachment was determined from a force-balance relationship, as explained elsewhere. For statistical analysis, each adhesion measurement was repeated five times. The effect of the few loading cycles applied during the measurement of the adhesion force on the impact behavior was neglected as secondary.
6.3 Results and Discussion

Figure 6.2 shows the dimensionless adhesion force $F_{\text{ad}}$ as a function of impact cycles $N$ for external maximum contact pressure $p_{\text{ex}}^{\text{max}} = 9.2$ and 20.1 kPa, impact frequency $f = 50$ and 500 Hz, and impact velocity $v$ in the range of 0.6–9.1 mm/s. To avoid complications from micromachine-to-micromachine variations, the adhesion force was normalized by the force measured before testing. Consistent with earlier studies, the adhesion behavior comprises two distinct phases: (1) incubation (run-in) phase of relatively low and stable adhesion, indicative of insignificant changes in the surface characteristics, and (2) surface modification phase demonstrating a rapid increase in adhesion, illustrative of significant physicochemical surface changes. Micromachine failure implies cessation of micromachine movement (stiction) due to the adhesion force exceeding the micromachine restoring force. The micromachine lifetime is defined as the sum of the times spent by the micromachine in the incubation and surface modification phases. The results reveal a trend for micromachine lifetime to decrease with increasing $p_{\text{ex}}^{\text{max}}$, $v$, and/or $f$, indicating that micromachine failure is due to concomitant effects of $p_{\text{ex}}^{\text{max}}$ and $v$. This can be attributed to the enhancement of surface modification due to the intensified surface interaction caused by the increase in $v$ and $p_{\text{ex}}^{\text{max}}$.

Higher $v$ and $p_{\text{ex}}^{\text{max}}$ implies higher kinetic energy and more energy dispassion at the instant of impact, respectively. Energy dissipation at the contact interface during impact loading may lead to changes in both physical and chemical surface characteristics. Physical effects may include variation in surface conformity due to changes in nanotopography resulting from plastic deformation and/or fracture of the highly stressed asperities on sidewall ridges. The decrease of the interfacial gap with the enhancement of surface conformity increases the real contact area and, in turn, the magnitude of the adhesion force. High-resolution SEM did not reveal any discernible topography changes or formation of wear particles on the impacting sidewall surfaces, consistent with previous studies of polysilicon micromachines tested under dynamic contact conditions in dry and high-humidity environments.

Adhesive behavior may also be influenced by surface chemical changes. The micromachine surfaces were covered by an oxide film and organic residue (intrinsic of the drying process) of surface energy less than that of silicon oxide. In-vacuum sliding experiments of polysilicon micromachines subjected to $p_{\text{ex}}^{\text{max}}$ in the same range as that of the present study did not show thinning of the oxide layer. Considering that sliding generally produces more extensive surface modification than normal contact because of the additional effect of surface shearing, rupture of the oxide film was precluded in the present experiments. Thus, the increase in adhesion force is attributed to the removal of organic residue from surface regions of the raised sidewall ridges where actual contact occurred. Micromachine failure (stiction) was encountered when the curve of the adhesion force intersected the lower boundary of the stiction region. For $v = 9.1, 5.1,$ and $0.9$ mm/s, failure was observed after approximately $1.3 \times 10^4$, $2.1 \times 10^5$, and $3.4 \times 10^6$ impact cycles, respectively. However for $v = 0.6$ mm/s, testing was terminated after $\sim 1.7 \times 10^7$ impact cycles, in the absence of any evidence of micromachine failure within reasonable testing time.
Figure 6.3 shows the product of $p_{ex}^{\text{max}}$ and $v$ (referred to as the pressure-velocity limit) versus number of impact cycles to failure (stiction) $N_f$ for a wide range of $p_{ex}^{\text{max}}v$. The data demonstrate that $N_f$ increases nonlinearly with the decrease of $p_{ex}^{\text{max}}v$ limit. From a total of 21 micromachines used to obtain the data shown in Fig. 6.3, 16 of those tested in the range of 3.9–187.4 kPa mm/s failed after $7.5 \times 10^3$–$2.1 \times 10^7$ impact cycles due to stiction, whereas 5 of those tested in the range of 1.5–5.7 kPa mm/s did not fail even after $>1.7 \times 10^7$ impact cycles. From a least-squares fitting, the following power-law relationship of the micromachine lifetime was obtained:

$$N_f = C[p_{ex}^{\text{max}}v]^m$$

where $C$ and $m$ are fitted constants. The curve shown in Fig. 3 represents a least-squares fit ($R^2 = 0.871$) through all data of failed micromachines. Using this fit and Eq. (1), it was found that $C = 2.84 \times 10^{10}$ and $m = -2.859$. It is interesting to note that the plot shown in Fig. 6.3 resembles a stress-life curve typical of macroscopic fatigue. Thus, in addition to Fig. 6.2, the above method provides another means of predicting the lifetime of contact-mode surface micromachines operated in a wide range of impact velocities (frequencies).

Figure 6.4 shows the dimensionless adhesion force $\bar{F}_{ad}$ as a function of impact velocity $v$ (or frequency $f$) for $p_{ex}^{\text{max}} = 5.5$ kPa. To ensure that measurements were obtained in the incubation phase, the number of impact cycles in these test was less than $10^3$. The adhesion force at a given impact velocity (frequency) was normalized by that corresponding to $f = 1$ Hz. The latter increased by only 6.9% after testing, indicating negligible surface changes, which confirmed that all of the measurements were obtained in the incubation phase. The repeatability of the results shown in Fig. 6.4 was confirmed by similar tests performed with another micromachine under identical conditions, using the same experimental procedure. Although $\bar{F}_{ad}$ for $f = 1000$ Hz was ~42% higher than that for $f = 1$ Hz, this difference in adhesion force could not be observed after separating the shuttles and consequently measuring the adhesion force by bringing the shuttles again into contact in a quasi-static manner. This reversible change in $\bar{F}_{ad}$ suggests that enhancement of the contact intimacy under high-velocity impact conditions was due to dominance of elastic deformation of the asperity contacts. Moreover, adhesion force measurements were obtained sequentially as the impact velocity was increased (forward sweep) and decreased (backward sweep) in the range of 0.01–12.91 mm/s (1–1000 Hz frequency range). The similar variation of the adhesion force observed with forward and backward sweep in the given impact velocity range, suggests that the adhesion force measured in the incubation phase does not show a dependence on impact velocity history. Thus, the variation of the adhesion force with impact velocity shown in Fig. 6.4 is attributed to elastic recovery of the deformed asperities.

Another important observation is the existence of two impact velocity regimes of different adhesion behavior in Fig. 4. In the low-velocity regime ($v \leq 1$ mm/s), $\bar{F}_{ad}$ increased rapidly with $v$, while in the high-velocity regime ($v > 1$ mm/s), $\bar{F}_{ad}$ increased slowly but steadily. This nonlinear dependence of $\bar{F}_{ad}$ on $v$ may be attributed to multi-scale roughness effects, resulting in the nonlinear increase of the density of asperity contacts with the impact intensity. The rapid increase of $\bar{F}_{ad}$ for $v \leq 1$ mm/s is attributed
to the dominant effect of the density of asperity contacts, whereas the slow increase of $\bar{F}_{\text{ad}}$ for $v > 1 \text{ mm/s}$ is inferred to the dominant effect of the size of asperity contacts.

### 6.4 Conclusions

Summarizing, a polysilicon surface micromachine suitable for impact testing under controlled conditions was used to investigate the effect of impact velocity on the evolution of adhesion at sidewall contact interfaces of MEMS devices and its implication to micromachine failure due to stiction. A transition from low and stable adhesion (incubation phase) to rapidly intensifying adhesion (surface modification phase) was observed by tracking the development of the interfacial adhesion force with impact cycles. Micromachine lifetime showed a nonlinear inverse dependence on pressure-velocity limit. The increase of the interfacial adhesion force with impact cycles was explained in the context of nanoscale surface modification effects resulting in both physical and chemical surface changes. A reversible nonlinear variation of the interfacial adhesion force with impact velocity was observed due to the dominance of elastic deformation at the asperity contacts.
References

Figure 6.1 SEM image of (a) a surface micromachine specifically designed for dynamic contact (impact) testing, including the electrical wiring diagram used for electrostatic comb-drive actuation and (b) close-up view of the contact region showing a protrusion extending from shuttle (2) used to control the apparent contact area formed with the sidewall surface of shuttle (1).
Figure 6.2 Adhesion force $F_{ad}$ versus impact cycles $N$ for $p_{ex}^{max} = 9.2$ and 20.1 kPa, $f = 50$ and 500 Hz, and $\nu = 0.6$–9.1 mm/s. (The adhesion force measured for a given number of impact cycles was normalized by that measured before testing. Data points represent averages of five tests. Error bars equal to one standard deviation above and below the corresponding average value are not distinguishable because their size is smaller than the symbol size.)
Figure 6.3 Pressure-velocity limit $p_{\text{ex}}^{\text{max}} \nu$ versus impact cycles to failure $N_f$ for $f = 50$–500 Hz.
Figure 6.4 Variation of adhesion force $\bar{F}_{ad}$ with impact velocity $\nu$ and frequency $f$ for $p_{ex}^{\text{max}} = 5.5$ kPa. (The adhesion force measured for a given impact velocity (frequency) was normalized by that measured for $f = 1$ Hz. Data points represent averages of five tests. Error bars indicate one standard deviation above and below the corresponding average value.)
Chapter 7: Effect of Fluorocarbon Self-assembled monolayer Films on Sidewall Adhesion and Friction of Surface Micromachines with Impacting and Sliding Contact interfaces

7.1 Introduction

Reliability and longevity are major limiting factors for the broader use of contact-mode microelectromechanical systems (MEMS). High surface area-to-volume ratio, weak restoring forces, and small gaps between adjacent surfaces make these microdevices highly susceptible to failure due to excessive adhesion forces. Efficient operation of numerous MEMS devices is halted by the evolution of interfacial retarding forces during dynamic impact or sliding contact.\(^1\,^2\) Changes in surface topography and chemical behavior during micromachine operation may lead to the development of high adhesion or friction forces,\(^3\,^4\) which can negatively impact the device functionality and efficiency. Irreversible surface adhesion (stiction) occurs when interfacial adhesion (attractive) and friction (shear) forces overcome available micromachine restoring forces. Therefore, control of adhesion and friction properties of contact interfaces is of paramount importance to the reliability and performance of contact-mode MEMS devices.

Various surface treatments used to mitigate the detrimental effects of high adhesion and friction forces at micromachine contact interfaces can be classified into two categories, physical and chemical. The main objective in physical treatments is to reduce the magnitude of retarding surface forces by surface protrusions (dimples) to control the apparent contact area or surface texturing (roughening) to minimize the real area of contact.\(^3\) Micromachine release by CO\(_2\) supercritical drying is a preferred drying process because it avoids the development of attractive capillary forces observed with conventional drying processes relying on liquid evaporation.\(^5\) One of the most promising surface chemical treatments for preventing micromachine stiction during release and operation is the deposition of spontaneously self-assembling molecularly thin films of low surface energy substances, known as self-assembled monolayer (SAM) films.\(^6\)

Among the various types of SAM films, long-chain fluoro carbon and hydrocarbon molecules are particularly effective in producing antistiction coatings because of their close-packed arrangement and low surface energy. A common approach is the chemical attachment of alkylchlorosilane SAM films on hydroxyl-terminated silicon surfaces through the formation of Si-O-Si bonds.\(^3\) Both liquid-phase\(^7\) and vapor-phase\(^8,\,^9\) processes have been used to deposit SAM films on MEMS devices. Liquid-phase
SAM deposition is limited by incomplete wetting of high aspect ratio structures, uncertain reaction kinetics, organic solvent waste, and high production cost. Vapor-phase SAM deposition has been shown to be a more reproducible and scalable film deposition technology. Therefore, SAM deposition from the vapor phase is considered to be one of the most effective surface treatment methods for reducing high adhesion and friction forces at contact interfaces of MEMS devices.

MEMS device operation under a wide range of environmental conditions increases the importance of the tribological behavior and stability of SAM films in different temperatures and humidity levels. The mechanical endurance of SAM-coated micromachine surfaces is critical because effective operation of several MEMS devices depends on the reliability of impact or sliding contact interfaces. Several studies have been undertaken to elucidate the effects of environmental conditions and dynamic contact loading on the adhesion, friction, and wear properties of various SAM films. For example, Klein et al. examined the effect of mechanically-induced aging on rubbing silicon surfaces coated with SAM films, and Kasai et al. used an atomic force microscope (AFM) to study the effects of humidity, temperature, and sliding velocity on the micro/nanotribological properties of silicon coated with different perfluorosilane SAM films. However, because of vast differences in the testing conditions applied in these studies, the reported results are not necessarily indicative of SAM films exposed to environments and loading conditions typical of MEMS devices. In other investigations, the tribological properties of SAM films were studied with special MEMS devices. For instance, Fréchette et al. examined the effect of temperature on in-use stiction of cantilever beams coated with fluorinated monolayers, while Hook et al. investigated degradation of fluorocarbon-coated sidewall surfaces of MEMS devices undergoing dynamic contact. However, direct evaluation of the effect of fluorocarbon SAM films on micromachine efficiency and durability cannot be obtained because a direct comparison of the performance of uncoated and SAM-coated microdevices was not performed in the former studies.

Scientific and technological demands for more reliable MEMS devices have increased the need for a basic understanding of the tribological behavior of micromachine surfaces coated with SAM films, especially sidewall surfaces exhibiting profoundly different morphologies than planar surfaces due to microfabrication process differences. Consequently, the objective of this study was to investigate the effects of humidity and temperature on the dynamic contact (impact and sliding) behavior of MEMS sidewall surfaces coated with a fluorocarbon SAM in the context of goniometry measurements, adhesion and static friction tests performed under controlled environmental conditions, and X-ray photoelectron spectroscopy (XPS) results.

### 7.2 Experimental Methods

#### 7.2.1 Deposition of self-assembled monolayer films

A tridecafluoro-1,1,2,2-tetrahydrooctyltrichlorosilane (FOTS, \( \text{CF}_3(\text{CF}_2)_5(\text{CH}_2)_2\text{SiCl}_3 \)) SAM film was deposited on single-crystal Si(100) substrates and polycrystalline silicon (polysilicon) micromachines using a molecular vapor deposition system (MVD100E, AMST). Before FOTS deposition, the samples were subjected to a 5-
min treatment with O$_2$ plasma (200 W power; 100 sccm O$_2$ flow rate; 0.28 Torr chamber pressure) to remove any organic contaminants and functionalize the silicon surfaces with –OH groups. This treatment was followed by FOTS film deposition in the same chamber. First, the precursor vapor was flown into an expansion chamber until the increase of the pressure to 0.7 Torr. Then, the chemical source was closed and the valve connecting the evacuated expansion chamber with the process chamber was opened to inject the vapor into the reaction chamber. Subsequently, water vapor was introduced into the expansion chamber and when the pressure increased to 4 Torr, it was injected into the evacuated reaction chamber. After repeating the latter process step one more time, the gases were left to react with the sample surfaces for 15 min. The reactive head-group of the FOTS molecules reacted with H$_2$O vapor to produce –OH groups, which then reacted with surface –OH groups to form Si–O–Si bonds that attached the FOTS molecules onto the silicon substrate. Reaction between the –OH groups of the attached FOTS molecules may also have contributed to the lateral bonding of the molecular chains of the SAM film. The aforementioned deposition process of the FOTS monolayer on the oxidized silicon surfaces is schematically illustrated in Fig. 6.1.

7.2.2 Goniometry

Static contact angle measurements were obtained from FOTS-coated Si(100) substrates. The wetting characteristics of the FOTS film were examined at room temperature using a drop-shape analysis system (DSA10, Krüss GmbH). Droplets of deionized water (~6 µL) were applied to the FOTS film surface by a syringe, and the droplet configuration was captured by a camera. Then, the angle between the droplet baseline and the tangent at the water/air boundary was measured, and the contact angle was calculated as the average of the left and right contact angles. For statistical analysis, ten contact angle measurements were obtained from three identical samples.

7.2.3 X-ray photoelectron spectroscopy

Elemental composition analysis of the FOTS film deposited on Si(100) substrates was performed with an XPS system (PHI 5400 ESCA, Perkin-Elmer), without charge neutralization or monochromator, equipped with an Al-Kα X-ray source of photon energy equal to 1486.6 eV. A takeoff angle of 54.7° relative to the analyzer axis and a chamber pressure of ~10$^{-5}$ Torr were used to collect all the XPS spectra. Survey spectra were acquired in the binding energy range of 0–800 eV with pass energy of 178.95 eV. For chemical bond identification, high-resolution spectra were obtained in the C1s region with a resolution of 0.05 eV. After Shirley background noise subtraction, the XPS spectra were deconvoluted by fitting 70% Gaussian–30% Lorentzian (GL) distributions of full width at half maximum equal to 2.0 eV.

7.2.4 Description of the test micromachine

Adhesion, impact, and sliding tests were performed with specially designed surface micromachines. Two types of micromachines were used in this study – impact and sliding micromachines – both fabricated by Polysilicon Multi-User MEMS Processes (MEMSCAP). Impact micromachines consist of two symmetric shuttles supported by double-folded flexure suspensions, which are driven by parallel electrostatic comb-drive actuators. The shuttles can be pushed into contact by activating the push comb drives or pulled away from each other by activating the pull comb drives. Sliding micromachines consist of two suspended shuttles,
which can be actuated in orthogonal directions by comb drives, referred to as the loading and
sliding shuttles. The loading shuttle is used to apply the normal contact force or to detach the
sidewall surfaces, while the sliding shuttle is used to slide (shear) one sidewall surface over
the other. A protrusion fabricated on sidewall of shuttle (2) (impact micromachine) and
sliding shuttle (sliding micromachine) is used to control the apparent contact area of the
sidewall surfaces. Similar to the impact micromachine, there is protrusion on the sidewall
surface of the sliding shuttle of the sliding micromachine. For both types of micromachines,
surface alignment of the shuttles has been confirmed by white-light interferometry. Further
fabrication and operation details can be found in Section 2.2

7.2.5 Testing and experimental setup
The adhesion characteristics of uncoated and FOTS-coated polysilicon
micromachines, hereafter referred to as Si and FOTS/Si micromachines, respectively,
were investigated under controlled contact loading and environmental conditions using
the impact and sliding micromachines. This was accomplished by gradually increasing
the electrostatic force up to the instant of initial sidewall contact, applying an external
normal force (pressure), and quasistatically decreasing the electrostatic force up to the
commencement of surface detachment. The adhesion force at that instant was determined
from a force-balance relationship. The adhesive pressure $p_{ad}$ is defined as the
measured adhesion force divided by the apparent contact area established between the
protrusion and the opposed sidewall surface. For statistical analysis, each adhesion
measurement was repeated five times.

The adhesive pressure was used as an in situ indicator of the surface changes
carried by repetitive impact. The experimental procedure for impact testing includes the
following sequential steps: (1) using the levitation force, both shuttles were raised to an
appropriate height by increasing the voltage applied to the push and pull actuators ($V_{push} = V_{pull} = 40 \text{ V}$), (2) the shuttles were moved closer to each other by decreasing $V_{pull}$, and
(3) a 50 Hz sinusoidal waveform of amplitude $\Delta V_{push} = 10 \text{ V}$ was applied to both shuttles,
initiating repetitive impact. From the oscillation amplitude versus frequency response of
the micromachine, it was determined that the shuttle displacement during impact testing
can be accurately obtained from a static analysis. Based on this finding, the impact
velocity was found to be equal to ~972 $\mu$m/s. Impact testing was periodically interrupted
to measure the adhesion force. Five measurements of the adhesion force were acquired
during each interruption.

To examine the tribological properties of the Si and FOTS/Si micromachines, the
measured adhesion and friction forces were used as in situ indicators of the surface
changes caused by surface rubbing. Before each sliding test, the levitation force was used
to raise the loading and sliding shuttles by applying a voltage to the push and pull actuators ($V_{push} = V_{pull} = V_{shear}^+ = V_{shear}^- = 40 \text{ V}$). Normal contact between the sidewall
surfaces was then established by applying a dc bias voltage to the comb drive of the
loading shuttle, pushing it into contact with the protrusion extending from the opposed
sidewall surface of the sliding shuttle. Finally, oscillatory sliding was initiated by
applying a sinusoidal ac signal of amplitude $\Delta V_{shear}^+ = 10 \text{ V}$ to the comb drive of the
sliding shuttle. In all sliding tests the driving frequency was set at 25 Hz.

Testing was periodically interrupted to measure the adhesion and friction forces.
The adhesion force between the sliding sidewall surfaces was measured using the same quasistatic procedure described above. The static friction force was determined by first bringing the loading shuttle into contact with the sidewall surface of the sliding shuttle, applying an external load, and then gradually increasing the electrostatic force produced by one of the shear drives until the inception of sliding. The static friction force was calculated from a force balance at the onset of sliding. The interfacial shear strength \( \tau \) is defined as the measured static friction force divided by the apparent contact area established between the protrusion and the opposed sidewall surface of the loading shuttle. For statistical analysis, each friction measurement was repeated five times.

Impact and sliding tests were performed in a custom-made multi-probe vacuum station (MMR Technologies) mounted on a vibration isolation table (Newport Electronics) described in detail in Section 2.3.

7.3 Results and Discussion

7.3.1 Wettability and composition of self-assembled monolayer film

Statistical data of goniometry measurements obtained with Si(100) and FOTS/Si(100) samples showing the efficacy of the FOTS film to modify the wettability of oxidized silicon surfaces from hydrophilic to hydrophobic are given in Table 7.1. The small contact angle (\(~25.3^\circ\) of the Si samples is indicative of hydrophilic surfaces and is attributed to the presence of the native SiO\(_2\) surface layer. The fourfold higher contact angle (\(~102.6^\circ\) of the FOTS/Si samples is illustrative of the hydrophobic character of the FOTS film. Because the contact angle of the FOTS/Si samples is >90\(^\circ\), the capillary force produced in humid environments will be repulsive, which is desirable for the release and operation of the suspended microstructures of polysilicon MEMS devices. To investigate the stability of the FOTS film under atmospheric conditions, the contact angle of FOTS/Si samples stored under ambient conditions of \(T = 25–30\) °C and RH = 20–30% was measured after 30 days. The very small contact angle change (~2.4\(^\circ\)) of the stored samples indicates the stability of the chemisorbed FOTS film under ambient conditions.

XPS analysis confirmed the deposition of a uniform FOTS film and provided information about its structure and composition. Figure 7.2 shows a comparison between XPS survey spectra of Si and FOTS/Si samples. A dominant F1s peak can only be observed in the FOTS/Si spectrum. The very weak Cls signal in the FOTS/Si spectrum reveals complete hydroxylization of the FOTS molecules on the silicon surface. Another difference is that the Si spectrum contains a small single Cls peak, whereas the FOTS/Si spectrum contains a small doublet Cls peak. The Si2s and Si2p peaks appearing in both spectra are because the sampling depth in the XPS (~5 nm) exceeds the thickness of the FOTS film.

Further information about the composition of the FOTS film can be obtained from the Cls core-level XPS spectrum shown in Fig. 7.3. The Cls peak was deconvoluted by six GL distributions (denoted by Cls-1, Cls-2, Cls-3, Cls-4, Cls-5, and Cls-6) corresponding to characteristic binding energies. The Cls-1 peak centered at 293.2 eV is assigned to \(-\text{CF}_3\), which is the end-group of the FOTS molecule (Fig. 7.1). The Cls-2 peak at 291.2 eV is assigned to \(-\text{CF}_2\), whereas the Cls-3, Cls-4, Cls-5, and Cls-6 peaks at 289.6, 288.4, 286.5, and 284.7 eV are assigned to \(\text{CF}–\text{CF}_n\), \(\text{CF}\), \(\text{C}–\text{CF}_n\) and \(\text{C}–\text{C}\), respectively. Detailed
information about the deconvolution and the interpretation of each GL distribution can be found elsewhere. The doublet C1s peak in the FOTS/Si survey spectrum (Fig. 7.2) is mainly due to the –CF2– and –CF3 distributions, which can only be attributed to the FOTS precursor. The dominant F1s peak in the FOTS/Si survey spectrum (Fig. 7.2) and the strong contribution of the –CF2– and –CF3 distributions in the C1s core-level spectrum (Fig. 7.3) confirm the chemisorption of the FOTS film onto the Si substrate.

7.3.2. Effect of external contact pressure and environmental conditions on adhesive behavior

Capillary and van der Waals forces are main contributors to the adhesion forces arising in polysilicon MEMS devices with microstructures suspended over grounded planar substrates. Hydrophilic surfaces in close proximity with each other are prone to high adhesion forces due to long-range attraction effects attributed to dipole-dipole interactions and dispersion forces and the formation of liquid menisci in humid atmospheres, as shown schematically in Fig. 7.4(a). The adhesion force due to van der Waals and capillary effects may exceed the restoring force of a suspended microstructure, rendering the MEMS device dysfunctional.3 Coating the hydrophilic surfaces of polysilicon microstructures with a conformal hydrophobic SAM film not only reduces the van der Waals forces and inhibits meniscus formation, but also changes the capillary force (wherever meniscus formation occurs) from attractive to repulsive. Consequently, a continuous and conformal FOTS film weakens dipole-dipole interactions and dispersion forces and, in particular, suppresses meniscus formation, as shown schematically in Fig. 7.4(b), enhancing the operation lifetime and efficiency of contact-mode MEMS devices. Hence, knowledge of the efficacy of hydrophobic SAM films to maintain the aforementioned beneficial characteristics under different dynamic contact and environmental conditions is critical to the design of highly sensitive (i.e., low restoring force) and durable MEMS devices with narrow-gap interfaces.

Results from adhesion experiments performed with Si and FOTS/Si micromachines under conditions of varying external contact pressures \( p_{ex} \), ambient pressure \( P \), temperature \( T \), and relative humidity RH are presented next to elucidate the beneficial effect of the FOTS film under different operation and environmental settings. Figure 7.5 shows the adhesive pressure \( p_{ad} \) of Si and FOTS/Si micromachines as a function of externally applied contact pressure \( p_{ex} \) for \( P = 10^{-5} \) and 760 Torr, RH = 0 and 100%, and \( T = 25–30 \) °C. Under vacuum conditions (RH = 0%), Si and FOTS/Si micromachines exhibit similar adhesion behaviors. Since only van der Waals forces contribute to the adhesion force in vacuum, the effect of the FOTS film is only reflected in the Hamaker constant, which is secondary to the variation of the adhesive pressure. However, under high-humidity conditions (RH = 100%), i.e., when both van der Waals and capillary forces significantly contribute to the adhesion force, the FOTS/Si micromachine demonstrates much lower adhesion than the Si micromachine through the entire range of \( p_{ex} \). Because the ambient pressure effect on the van der Waals force is secondary, the dependence of sidewall adhesion on ambient pressure is considered to be insignificant. The contribution of the capillary force to the total adhesion force can be obtained as the difference of the \( p_{ad} \) data corresponding to RH = 0 and 100%. The significantly lower \( p_{ad} \) of the FOTS/Si micromachine than that of the Si micromachine for RH = 100% and the similar \( p_{ad} \) of the FOTS/Si micromachine for RH = 0 and 100% indicate that capillary effects are negligibly small in the presence of the conformal FOTS film. The
gradual increase of $p_{\text{ad}}$ with the increase of $p_{\text{ex}}$ observed under all environmental conditions may be attributed to the increasing contributions of the van der Waals and capillary forces to the total adhesion force. The enhancement of the surface intimacy with the increase of $p_{\text{ex}}$ led to the increase of the real contact area and the decrease of the local gap between proximity (noncontacting) asperities, resulting in a higher van der Waals force and meniscus formation over a larger fraction of the contact interface, respectively.

Figure 7.6 shows the effect of the relative humidity RH on the adhesive pressure $p_{\text{ad}}$ of Si and FOTS/Si micromachines for $p_{\text{ex}} = 9$ kPa, $P = 760$ Torr, and $T = 25$–$30$ °C. The FOTS/Si micromachine exhibits stable and low adhesive pressure through the entire range of RH. However, the Si micromachine shows a similarly low $p_{\text{ad}}$ only in the low-humidity range (i.e., RH < 60%). The sharp increase of $p_{\text{ad}}$ of the Si micromachine in the high-humidity range (RH > 60%) is attributed to the significantly increased contribution of the capillary force to the total adhesion force. It appears that water condensation on the hydrophilic surfaces of the Si micromachine increased nonlinearly with relative humidity, resulting in the rapid formation of liquid menisci that greatly intensified the adhesive pressure. Because this phenomenon was not encountered in the presence of the hydrophobic FOTS film, the adhesion characteristics of the FOTS/Si micromachine were not influenced by the large humidity variations.

Figure 7.7 shows influence of the temperature $T$ on the adhesive pressure $p_{\text{ad}}$ of Si and FOTS/Si micromachines tested under conditions of $p_{\text{ex}} = 9$ kPa, $P = 760$ Torr, and RH = 100%. The adhesive pressure of the Si micromachine monotonically decreased with the increase of temperature in the range of 30–120 °C. This trend is attributed to the increasing desorption of water molecules with the increase of the temperature, resulting in a smaller contribution of the capillary force to the overall adhesion force. Through the entire temperature range examined, $p_{\text{ad}}$ of the FOTS/Si micromachine remained at low level, similar to that shown in Fig. 7.7, suggesting that the hydrophobic character of the FOTS film was not altered despite the increase of $T$ by fourfold. The slight decrease of $p_{\text{ad}}$ of the FOTS/Si micromachine with the increase of $T$ is probably due to structural and/or chemical changes in the FOTS film, reported in previous studies of SAM films.

### 7.3.3 Effect of repetitive impact and oscillatory sliding on adhesive behavior

Figure 7.8 shows the adhesive pressure $p_{\text{ad}}$ of Si and FOTS/Si micromachines as a function of impact cycles $N$ for $p_{\text{ex}}^{\text{max}} = 25.8$ kPa, $P = 760$ Torr, and RH = 100%, and $T = 25$–$30$ °C, where $p_{\text{ex}}^{\text{max}}$ is defined as the externally applied maximum load during impact divided by the apparent contact area. Data points represent mean values of adhesive pressure measurements obtained from five experiments performed under identical conditions. The results shown in Fig. 7.8 reveal two distinctly different adhesion behaviors. The adhesion behavior of Si micromachines consists of two stages—a run-in stage of low adhesive pressure encountered during the initial phase of impact testing ($N \leq 10^5$) indicating insignificant changes in the surface characteristics, followed by a surface modification stage ($N > 10^6$) of rapidly increasing adhesive pressure reflecting significant changes in physicochemical surface characteristics. The threefold increase of $p_{\text{ad}}$ of the Si micromachine (from 8.2 kPa in the run-in stage to 24.4 kPa in the surface modification stage) after $\sim 1.3 \times 10^7$ impact cycles is indicative of significant surface changes. The FOTS/Si
micromachine demonstrated insignificant changes in sidewall adhesion even after ~1.3 × 10^7 impact cycles (p_{ad} increased from 5.1 to 6.4 kPa through the entire course of testing).

Changes in the sidewall adhesion behavior of the Si micromachines may be associated with concomitant effects of chemical and physical processes. Surface chemical changes are of particular relevance given the fact that the comparison is between Si micromachines and identical micromachines coated with a hydrophobic, low surface energy FOTS film. The Si micromachines were covered with a native SiO_2 film and, presumably, islands of organic residue (characteristic of CO_2 supercritical drying) of surface energy lower than that of SiO_2. In a previous study of polysilicon micromachines sliding in vacuum under contact loads resulting in p_{ex}^\text{max} in the range as of the present study, it was observed that surface sliding did not induce discernible thinning (wear) of the SiO_2 film. Considering that sliding generally causes more extensive surface modification than purely normal contact, it may be interpreted that the SiO_2 film was not removed under the impact conditions of the present study. Thus, the increase of p_{ad} of the Si micromachine may be attributed to the removal of the organic residue. The lower p_{ad} of the FOTS/Si micromachine than that of the Si micromachine during the run-in stage (N ≤ 10^5) is due to the highly hydrophobic behavior of the FOTS film. If the FOTS film was removed from some contact regions at the sidewall interface during impact loading, the exposed hydrophilic SiO_2 film would have resulted in the increase of p_{ad}. The fairly stable and low p_{ad} of the FOTS/Si micromachine indicates that degradation of the FOTS film did not occur during impact loading even after ~2 × 10^7 impact cycles.

Surface physical changes, such as variation in surface contact intimacy due to plastic deformation and/or fracture of asperities residing at the crests of raised sidewall ridges may also influence the adhesive behavior. The decrease of the interfacial gap due to the increase of surface intimacy may increase both van der Waals and capillary forces. High-resolution SEM imaging did not yield any observable topography changes or accumulation of wear debris onto the sidewall surfaces, suggesting that the scale of physical surface changes or the size of wear debris were less than a few tens of nanometers (nanoscale wear).

The evolution of sidewall adhesion with sliding cycles can be interpreted in the context of results obtained from oscillatory sliding tests of Si and FOTS/Si micromachines. Figures 10(a) and 10(b) show the adhesive pressure p_{ad} and the interfacial shear strength \tau of Si and FOTS/Si micromachines as functions of sliding cycles N for p_{ex}^\text{max} = 1.5 kPa, P = 760 Torr, RH ≈ 35%, and T = 25–30 °C. The interfacial shear strength was obtained as the ratio of the static friction force, measured at the instant of relative movement between the sidewall surfaces, to the apparent contact area. Each data point represents the mean value of five measurements obtained under identical testing conditions. These experiments show a run-in stage of relatively low and stable p_{ad} and \tau, followed by a surface modification stage of rapidly increasing p_{ad} and \tau, revealing the commencement of significant physicochemical changes on the sidewall surfaces. Cessation of the oscillatory movement of the Si and FOTS/Si micromachines due to excessive adhesion (stiction) was observed after ~5 × 10^3 and ~5.8 × 10^4 sliding cycles, respectively. Stiction-induced failure occurred in static mode because of the relatively high static friction force that produced multiple stick-slip events, in agreement with a previous study of polysilicon sidewall surfaces sliding in a dry atmosphere.
Similar to the impact results, the evolution of the adhesion and static friction characteristics of the sidewall surfaces can be attributed to various physicochemical changes induced by surface rubbing. As mentioned earlier, the removal of the hydrophobic, low surface energy FOTS film and/or the organic residue deposited during the release process increased the surface energy, while nanoscale surface smoothening at the crests of the raised sidewall ridges caused by surface rubbing increased the real contact area and decreased the interfacial gap. All of these surface changes are conducive to meniscus formation, which increased the contribution of the capillary force to the total adhesion force and, in turn, the adhesive pressure and interfacial shear (friction) strength of the sliding sidewall surfaces. Because of the high hydrophobicity and low surface energy of the FOTS film, the FOTS/Si micromachine initially exhibited lower $p_{\text{ad}}$ and $\tau$ compared to the Si micromachine. The significant increase of $p_{\text{ad}}$ and $\tau$ of the FOTS/Si micromachine in the surface modification stage ($N \geq 5.8 \times 10^4$) suggests that localized wear of the FOTS film was initiated after $\sim 10^4$ sliding cycles, exposing the underlying SiO$_2$ surface.

Although $p_{\text{ex}}$ and RH in the sliding tests were lower than those in the impact tests, $p_{\text{ad}}$ showed premature and more pronounced changes, indicating relatively more pronounced physicochemical surface modification due to surface rubbing than impact. While only a few raised ridges of the sidewall surfaces were brought into contact during impact testing, a larger number of ridge-to-ridge interactions occurred during oscillatory sliding, implying a higher propensity for surface changes on the sliding sidewall surfaces. In addition, the simultaneous application of normal and shear traction in the sliding tests could have more dramatically altered the molecular structure of the FOTS film than the normal traction alone in the impact tests. Indeed, Monte Carlo simulations$^{22}$ and AFM studies$^{23,24}$ have shown load-induced changes in the molecular structure of SAMs and molecular reordering upon unloading. Moreover, reversible shearing is more conducive to wear of a SAM film than normal contact. The aforementioned factors may have contributed to the faster removal of the FOTS molecules during sliding, resulting in the degradation of the FOTS film sooner than impact testing. This explains the rapid increase of $p_{\text{ad}}$ and $\tau$ after fewer cycles in the sliding tests than in the impact tests.

### 7.4 Conclusions

The adhesion and friction properties of micromachine sidewall surfaces coated with a conformal FOTS film were investigated in the light of impact and sliding tests performed under controlled loading and environmental conditions. The hydrophobic character and elemental composition of the FOTS film were examined by goniometry and XPS measurements. The effects of external contact pressure, ambient pressure, temperature, relative humidity, and impact or sliding cycles on the adhesion and friction properties of sidewall surfaces with and without an FOTS film were analyzed in the context of experiments performed with surface micromachines specifically designed for impact and sliding testing. On the basis of the obtained results and discussion, the following conclusions can be drawn from this study.

1. The vapor-phase deposited FOTS film demonstrated good surface conformity, high hydrophobicity, and good stability under atmospheric conditions.
(2) The adhesive pressure increased gradually with the externally applied contact pressure. Although Si and FOTS/Si micromachines did not demonstrate notable differences in high-vacuum adhesion behavior, in high-humidity atmospheres the FOTS/Si micromachines demonstrated significantly lower adhesion than Si micromachines.

(3) While relative humidity and temperature exhibited secondary effects on the adhesion behavior of FOTS/Si micromachines, they significantly affected the adhesion behavior of Si micromachines. In particular, the adhesion of Si micromachines sharply increased with the increase of relative humidity above 60% and the decrease of temperature in the range 25–120 °C due to the strong dependence of the capillary force on relative humidity and temperature.

(4) Under high humidity, FOTS/Si micromachines demonstrated low and stable adhesion even for >10⁷ impact cycles, whereas the adhesion of Si micromachines sharply intensified after ~10⁵ impact cycles. Oscillatory sliding under ambient conditions caused the adhesive pressure and interfacial shear (friction) stress of Si micromachines to sharply increase after ~10³ sliding cycles compared to ~10⁴ sliding cycles for FOTS/Si micromachines.

(5) The evolution of the adhesion and friction behavior of micromachine sidewall surfaces were explained in terms of physicochemical surface changes, such as nanoscale surface smoothening and the removal of the organic residue (Si micromachines) or the FOTS film (FOTS/Si micromachines) during impact and sliding contact.

(6) The more pronounced and premature increase of adhesion observed under sliding than impact conditions indicates a greater likelihood for stiction-induced failure (cessation of motion) for surface micromachines with sliding contact interfaces.
References

**TABLE 7.1** Static water contact angle of Si(100) substrates with and without a FOTS self-assembled monolayer film.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Contact angle (deg.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si(100)</td>
<td>25.3 ± 2.0</td>
</tr>
<tr>
<td>FOTS/Si(100)</td>
<td>102.6 ± 0.6</td>
</tr>
<tr>
<td>FOTS/Si(100) (stored for 30 days)</td>
<td>100.2 ± 0.5</td>
</tr>
</tbody>
</table>
Figure 7.1 Chemical structure of FOTS self-assembled monolayer film and molecular chemisorption onto a hydrophilic oxidized silicon surface through the formation of Si-O-Si bonds.
Figure 7.2 XPS survey spectra of Si and FOTS/Si surfaces
Figure 7.3 High-resolution C1s core-level XPS spectrum of FOTS/Si surface.
Figure 7.4 Schematic illustration of (a) meniscus formation between hydrophilic surfaces and (b) suppression of meniscus formation in the presence of a chemisorbed hydrophobic SAM film.
Figure 7.5 Sidewall adhesive pressure $p_{ad}$ of Si and FOTS/Si micromachines tested under different environmental conditions versus maximum external contact pressure $p_{ex}$. (Data points represent averages of five tests. Error bars equal to one standard deviation above and below the corresponding average values are not distinguishable because their size is smaller than the symbol size.)
Figure 7.6 Sidewall adhesive pressure $p_{ad}$ of Si and FOTS/Si micromachines versus relative humidity RH for $p_{ex} = 9$ kPa, $P = 760$ Torr, and $T = 25-30$ °C. (Data points represent averages of five tests. Error bars equal to one standard deviation above and below the corresponding average values are not distinguishable because their size is smaller than the symbol size.)
Figure 7.7 Sidewall adhesive pressure $p_{ad}$ of Si and FOTS/Si micromachines versus temperature $T$ for $p_{ex} = 9$ kPa, $P = 760$ Torr, and RH = 100%. (Data points represent averages of five tests. Error bars equal to one standard deviation above and below the corresponding average values are not distinguishable because their size is smaller than the symbol size.)
Figure 7.8 Sidewall adhesive pressure $p_{ad}$ of Si and FOTS/Si micromachines versus impact cycles $N$ for $p_{ex}^{max} = 25.6$ kPa, $P = 760$ Torr, RH = 100%, and $T = 25-30$ °C. (Data points represent averages of five tests. Error bars equal to one standard deviation above and below the corresponding average values are not distinguishable because their size is smaller than the symbol size.)
Figure 7.9 (a) Sidewall adhesive pressure $p_{ad}$ and (b) sidewall interfacial shear strength $\tau$ of Si and FOTS/Si micromachines versus sliding cycles $N$ for $p_{ex}^{\text{max}} = 1.5$ kPa, $P = 760$ Torr, RH $\approx 35\%$, and $T = 25$–30 °C. (Data points represent averages of five tests. Error bars indicate one standard deviation above and below the corresponding average values.)
Chapter 8: Conclusions

The adhesion and friction properties of polysilicon microdevice sidewall surfaces were investigated in the light of impact and sliding tests performed under controlled loading and environmental conditions with special surface micromachines.

The effects of changes in the adhesion behavior and morphology of sliding sidewall surfaces of polycrystalline silicon MEMS devices operated in high vacuum (~10^{-5} torr) and under low apparent contact pressures (0.1–18 kPa) on the operation lifetime of the micromachines were investigated. Sidewall adhesion increased with applied contact pressure. Typically, twofold to fourfold increase in sidewall adhesion was measured upon seizure of the device operation (typically ~10^{6} sliding cycles) due to the increase of the static friction force above the restoring force of the device. Scanning electron microscopy (SEM) revealed very small amounts of ultrafine wear debris (10–140 nm) on the sidewall surfaces of about half of the tested devices, without discernible changes in the surface topography. Cross-sectional transmission electron microscopy (TEM) showed that sliding did not cause the removal of the silicon-oxide film (5–13 nm in average thickness) from the sidewall surfaces. Atomic force microscopy (AFM) indicated that sliding contact was confined at the top of a few elevated ridges on the sidewall surfaces, resulting in nanoscale wear that locally smoothened the surfaces. SEM, TEM, and AFM show that the tribological properties of contact-mode MEMS devices operating in high vacuum are controlled by only a few nanoscopic contacts, which depend on the local nanotopography of the interacting surfaces.

Knowledge of the changes in adhesion and friction at contact interfaces due to sliding contact is limited, particularly for sidewall surfaces. These changes were studied in this thesis by tracking the evolution of in the adhesive pressure, interfacial shear strength, and static coefficient of friction with accumulating sliding cycles. Low adhesion and high static friction observed in the initial stage of sliding were followed by monotonically intensifying adhesion and continuously decreasing friction until an equilibrium stage was reached at steady-state sliding. This trend revealed the existence of two regimes where asperity deformation and adhesion are dominant friction mechanisms, respectively. Scanning electron microscopy and atomic force microscopy results reveal physical and chemical surface changes induced by the sliding process. The evolution of the adhesion and friction properties is attributed to the increase of the real contact area and the work of adhesion resulting from nanoscale surface smoothening and the removal of contaminant adsorbents.

The effects of the contact pressure and environmental conditions on the surface modification and lifetime of devices subjected to cyclic impact loading were also
investigated. All micromachines demonstrated an initial run-in phase of low and stable adhesion force, followed by a surface modification phase characterized by the rapid increase of the interfacial adhesion force. The nonlinear increase of the adhesion force with impact cycles was found to be in direct correlation with the micromachine operation lifetime. A criterion of micromachine failure due to excessive interfacial adhesion (stiction) was formulated based on the observed experimental trend. Micromachine lifetime decreased non-linearly with the increase of the maximum contact pressure. The adhesion force of micromachines operated in vacuum or a high-humidity atmosphere increased faster than those operated in a dry atmosphere. Despite the significant increase of the adhesion force with impact cycles, high-magnification scanning electron microscopy did not reveal any discernible changes in the surface topography even after $3 \times 10^7$ impact cycles. The evolution of the interfacial adhesion force is attributed to the increase of the real area of contact and the work of adhesion due to nanoscale surface smoothening and the removal of adsorbed contaminant layers, respectively. Physical and chemical surface modification is interpreted in the context of results obtained for different maximum contact pressures, ambient pressures, and relative humidity levels.

The effect of impact velocity on the adhesion characteristics of sidewall contact interfaces of dynamic micromachines was also investigated in this thesis. The evolution of interfacial adhesion with contact cycles was examined for a wide range of impact velocity (frequency). Micromachine lifetime comprised two phases – incubation and surface modification. Shorter lifetime was encountered at a higher contact pressure and impact velocity. Permanent adhesion (stiction) due to the interfacial adhesion force exceeding the micromachine restoring force was found to correlate with the product of maximum contact pressure and impact velocity (pressure-velocity limit). Micromachine lifetime decreased non-linearly with the reciprocal of the pressure-velocity limit. A method for predicting micromachine lifetime was derived based on observed experimental trend. A reversible variation of the adhesion force with impact velocity, was observed in the in the incubation phase, which was explained in terms of elastic deformation of the contacting asperities. This trend also revealed the existence of two low- and high-impact-velocity regimes where the number of asperity contacts and average asperity size were dominant factors, respectively.

In another series of studies, the adhesive and friction behavior of uncoated and FOTS-coated polysilicon micromachines (referred to as Si and FOTS/Si micromachines) were investigated under controlled testing and environmental conditions. FOTS/Si micromachines yielded significantly lower and stable adhesive contact pressure than Si micromachines due to the highly hydrophobic and conformal FOTS film. Contrary to Si micromachines, the sidewall adhesive behavior of FOTS/Si micromachines showed a weak dependence on relative humidity, temperature, and impact cycles. In addition, FOTS/Si micromachines demonstrated low and stable sidewall adhesion and static friction for a significantly higher number of sliding cycles than Si micromachines. Sidewall adhesive and static friction characteristics of Si and FOTS/Si micromachines were interpreted in the context of physicochemical surface changes leading to the increase of the real contact area and hydrophobic-to-hydrophilic transition of the surface chemical character due to nanoscale surface smoothening and the removal of the organic
residue (Si micromachines) and the FOTS film (FOTS/Si micromachines), respectively, during repetitive impact and oscillatory sliding of the sidewall surfaces.

The results of this thesis provide valuable information about the effect of various material and design/fabrication parameters on the reliability and longevity of contact-mode MEMS devices.