Mechanical Durability of Fluorocarbon-based Monolayers in Normal and Sliding Contact In MEMS

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Self-Assembled Monolayers

Liquid deposited chlorosilanes or vapor deposited aminosilanes

Low surface energy reduces adhesive forces and prevents capillary adhesion

- CH_3 or CF_3 end groups result in hydrophobic surfaces

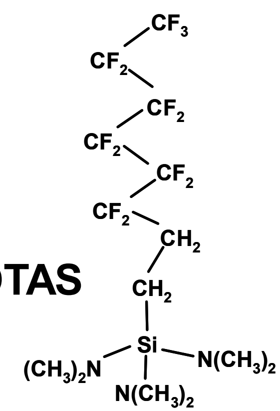
FOTAS coverage is approximately 2.5 x 10^{14} molecules/cm^2

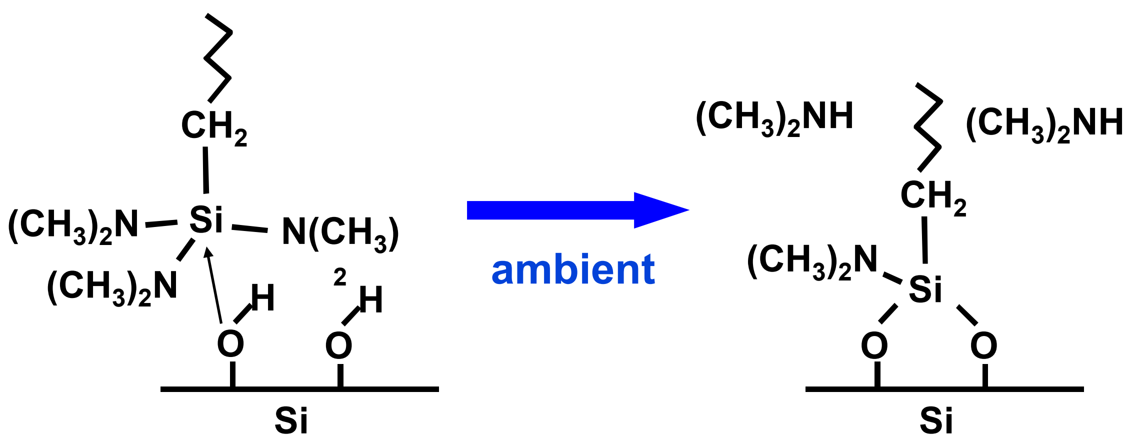
Exhibit initial coefficients of friction ~0.1

However, they are unable to heal after damage

Vapor-deposited aminosilane FOTAS: $CF_3(CF_2)_5(CH_2)_2Si(N(CH_3)_2)_3$

C8 FOTAS





Numerous Monolayers have been Explored for MEMS

Film Type	Contact Angle		Work of Adhesion	Coefficient of Static Friction	Particles	Ref.
	Water	Hexadecane	$\mu J/m^2$			
LDDMS	103°	38°	45	0.28	intermediate	[32]
VDDMS	102°	38°	62	0.35	low/none	[33]
LFDTS	111°	72°	<10	~0.10	very high	[34], [35]
VFDTS	111°	72°	3	0.12	low/none	[36]
VFOTS	≥110°	—	≤20	~0.3	low/none	[37], [38]
LOTS	110°	38°	12	0.07	very high	[32]
Oxide	~0–30°	~0–20°	~20,000	1.1	n/a	[32], [39]

W.R. Ashurst et. al, IEEE Trans Devices and Mat. Rel. vol 3 (2003) p. 173

Specimen	rms roughness (nm)	Adhesive force (nN)	Coefficient of friction		Critical normal load (wear resistance)	
			Microscale	Macroscale	Microscale (μN)	Macroscale (mN)
Si	0.20	33	0.070	0.26	N/A	N/A
PFTS/Si	0.13	19	0.024	0.12	56	100–120
ODMS/Si	0.09	26	0.017	0.14	17	40–60
ODDMS/Si	0.08	29	0.018	0.13	20	40–60
SiO_2	0.66	35	0.087			
PFTS/SiO_2	0.65	16	0.043			
ODMS/SiO_2	0.73	30	0.031			
ODDMS/SiO_2	0.55	33	0.032			
Au	0.37[a]	47[a]	0.032[a]			
HDT/Au	0.92[a]	14[a]	0.006[a]	0.26	6	10–20

Bhushan et. al, Ultramicroscopy vol 105 (2005) p. 176

- Numerous Studies of Monolayers
- Emphasis on Chain Length, Endgroup Type, and Endgroup Orientation
- All rely on Si-O reaction chemistry
- Si-O bonds susceptible to hydrolysis
- Energy imparted due to friction can be extreme
- Studies irrelevant if devices don't last

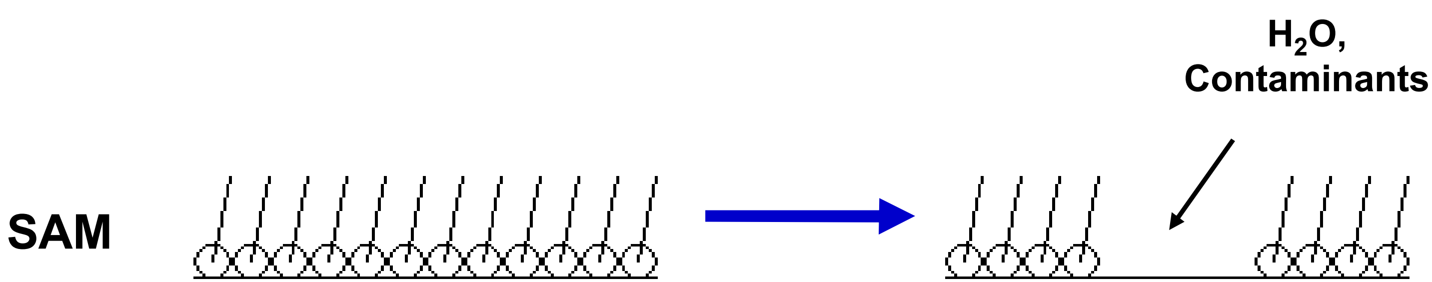
Monolayer Degradation

Thermal - increased rate of hydrolysis, or thermal decomposition

Mechanical - bond stretching and compression, physical removal

Macroscopic and AFM studies of SAM friction coefficients have alluded to wear, but do not represent contact conditions in MEMS

- few asperity contacts in apparent area
- adhesion forces are significant compared to applied forces



Dugger et al, Proceedings of SPIE Vol. 4980 Jan 2003
Frechette et al, JMEMS 15 (4): 737-744 Aug 2006
Zhuang et al J. Micromech. Microeng. 16 (11): 2259-2264 Nov 2006

Mechanical/Tribological Failure

Tribological contact occurs at both planar and sidewall surfaces, in general

MEMS have large roughness and tolerances relative to device dimensions (compared to macro-devices)

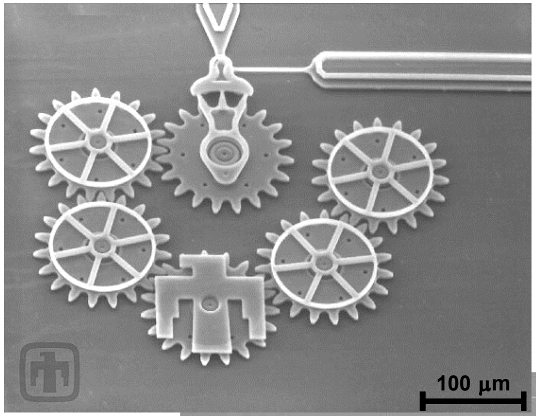
- contact locations and conditions in complex MEMS are not known a priori

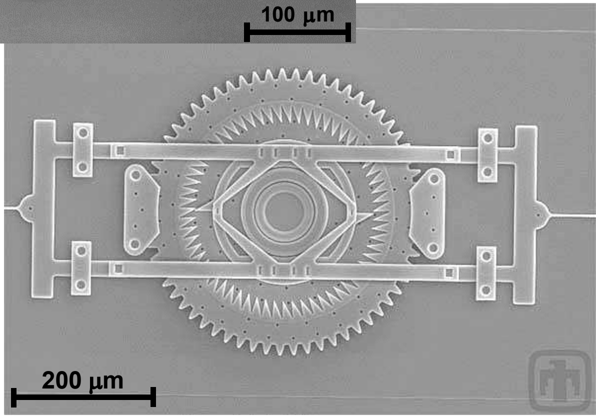
Wear is a limiting factor in MEMS reliability

- includes changes in surface chemistry as well as formation of particles and grooves

The effect of monolayer changes on adhesion and friction forces has not been investigated in detail

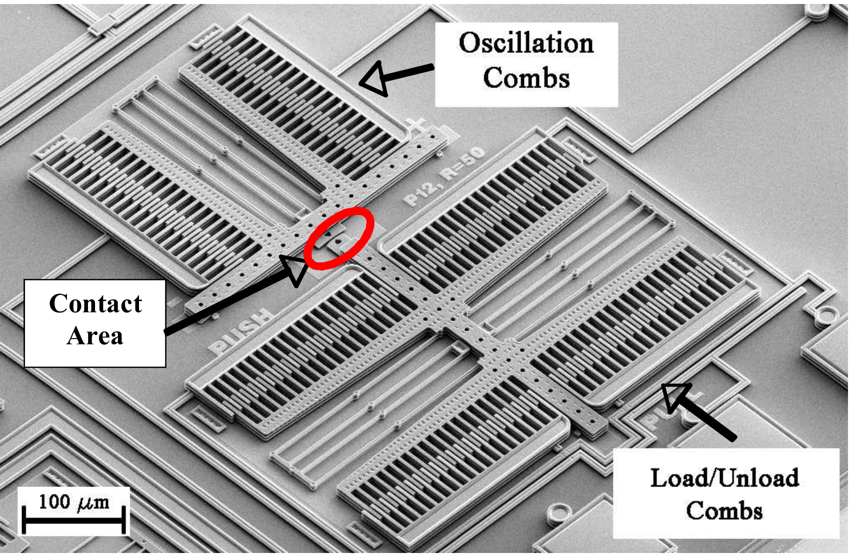
- device design requires information on monolayer wear in normal contact versus shear



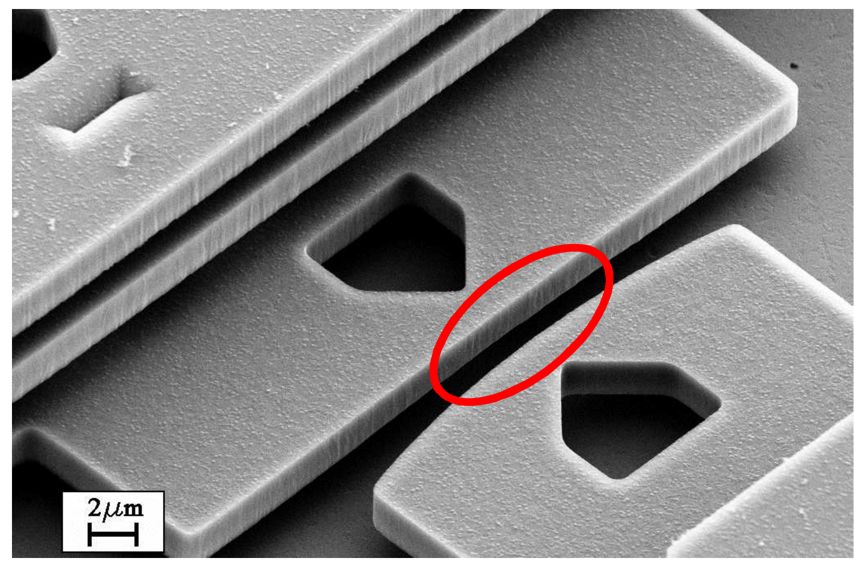


A MEMS sidewall tribometer was used to investigate monolayer wear in normal versus shear contact

MEMS Sidewall Tribometer

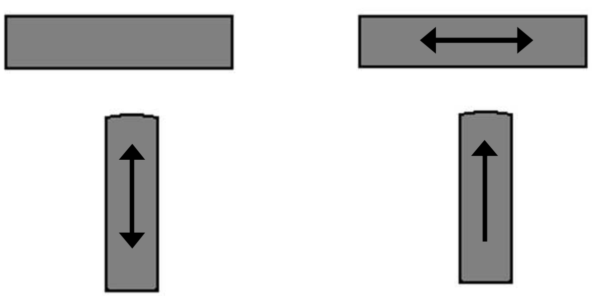


- fabricated using Sandia's SUMMiT^{TM} V process
- polycrystalline Si structures
- cylindrical sidewall contact of 50 μm radius and 2.26 μm thick
- vertical etch striations yield 20 nm rms on sidewalls



Two types of Contact Experiments:

Normal Oscillating



Normal Contact Experiment

Establish dry N_2 environment (<10 ppmv O_2, < 100 ppmv H_2O)

Perform initial adhesion force measurement

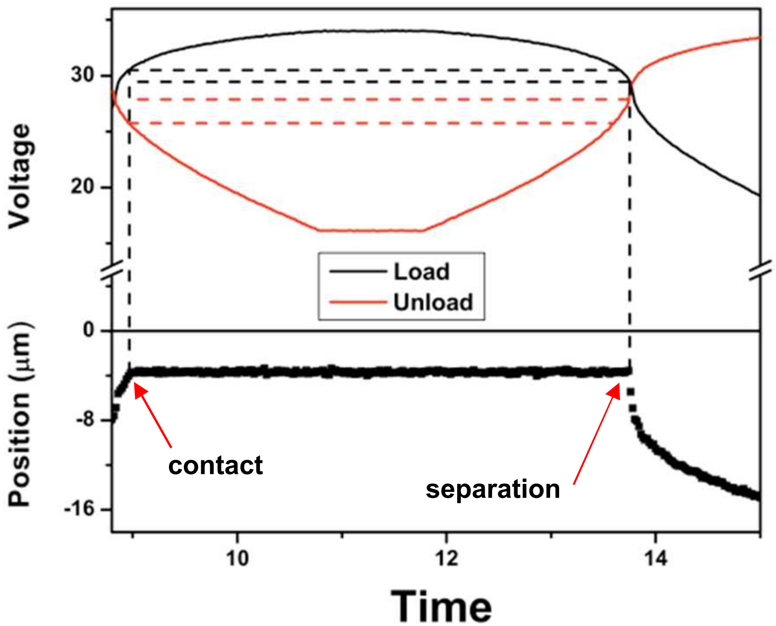
Repeat normal contact at 100 Hz, 750 nN peak load

- kinetic energy at contact = $\frac{1}{2} mv^2 = 1.46 \times 10^{-15}$ J
- Hertzian contact area = 7.4×10^{-14} m^2
- energy per unit area = 0.02 J/m^2

Stop at intervals and measure adhesion force

Continue until failure (ceased motion) or steady-state is reached

Adhesion Measurement



QuickTime™ and a
mpeg4 decompressor
are needed to see this picture.

load/unload

$$F_{ad} = F_{unload} - F_{load} + F_r$$

$$= a(V_{UL}^2 - V_L^2) + a(Vc_L^2 - Vc_{UL}^2)$$

(contact - separation) voltage permits quantification of adhesion force

- **measure after operation, environment exposure, aging, etc.**
- **reflects changes in contact surfaces due to degradation**

Shear Contact Experiment

- Establish dry N_2 environment (<10 ppmv O_2, < 100 ppmv H_2O)
- Perform initial adhesion force measurement
- Oscillate lateral force for initial motion amplitude of ~18 μm
- Engage loading actuator to give 750 nN
- Monitor oscillation amplitude as a function of cycles
- Continue lateral force oscillation until failure (ceased motion)
  - unload and reload to verify failure
- Perform final adhesion measurement

Dynamic Friction Measurement

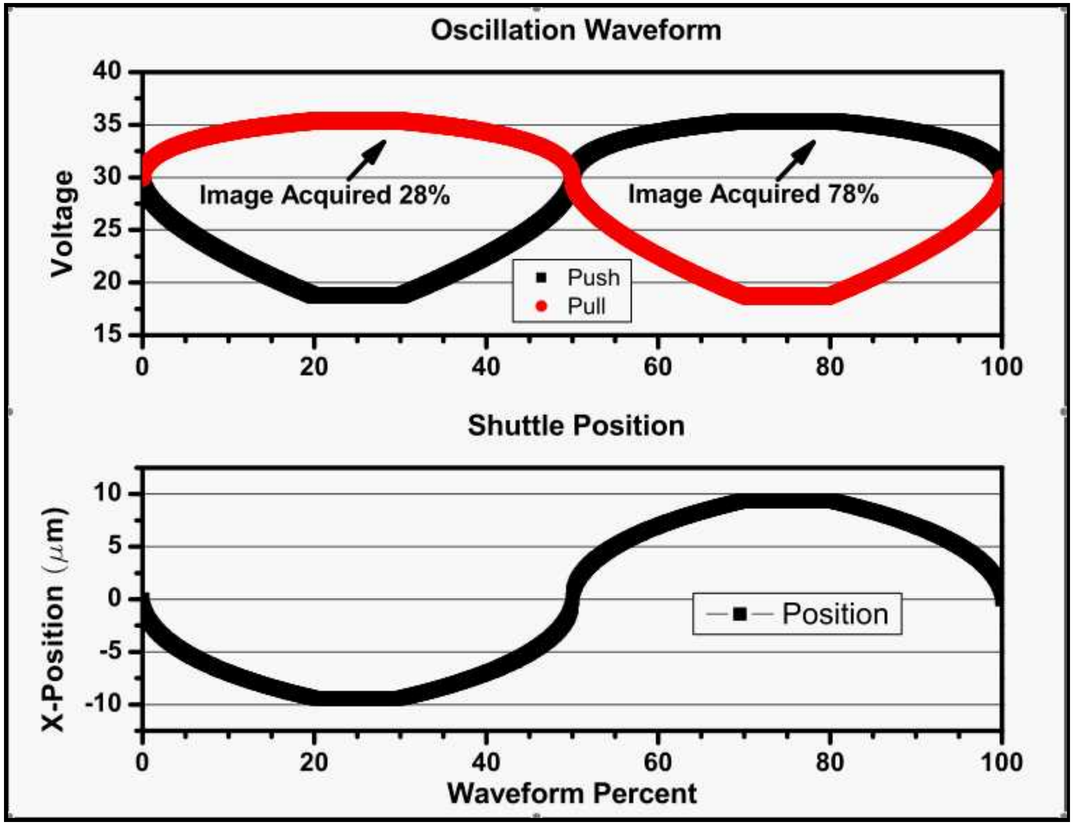


Image Capture

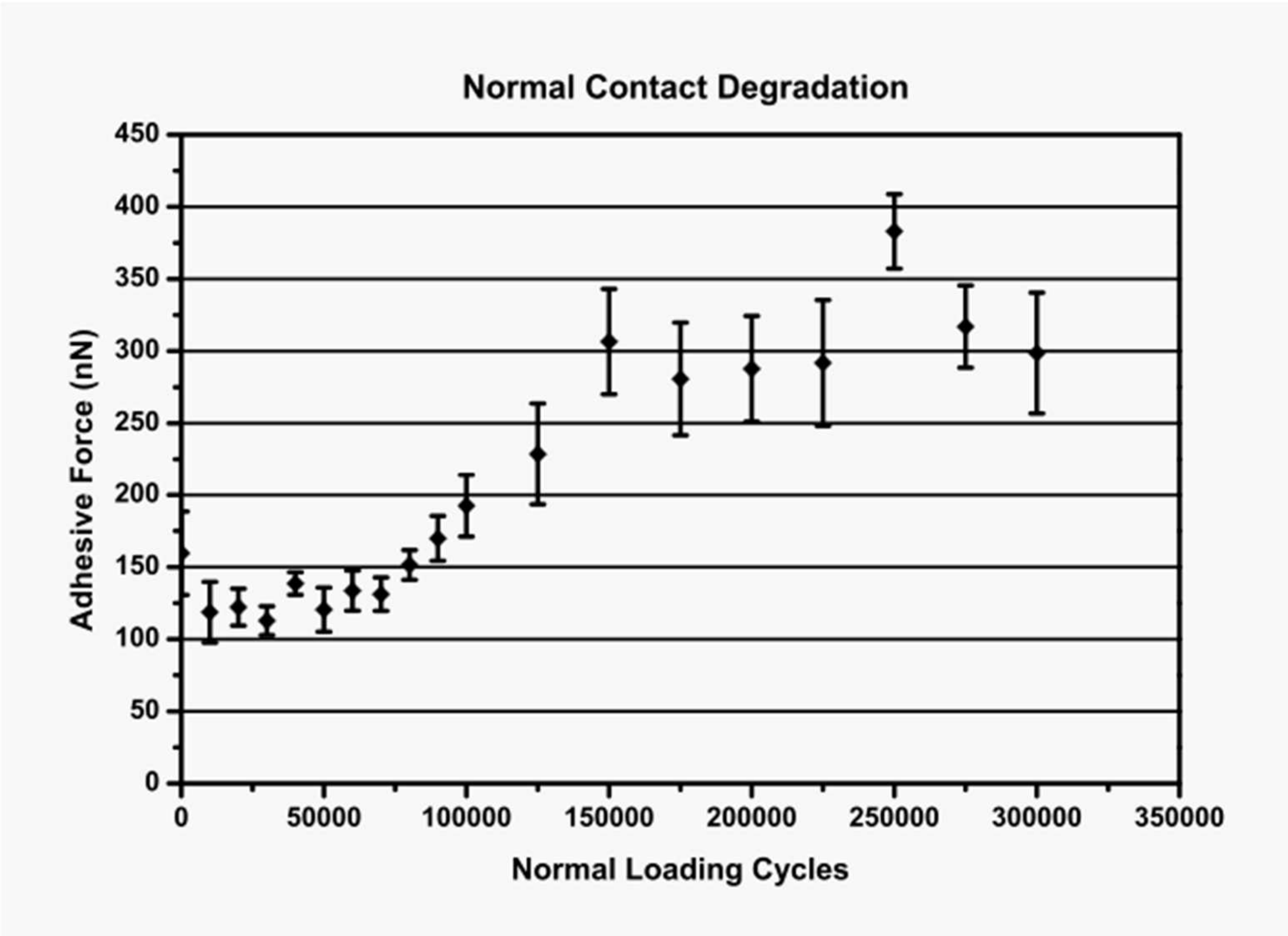
QuickTime™ and a
mpeg4 decompressor
are needed to see this picture.

↔ push/pull

Displacement amplitude is related to dynamic friction force

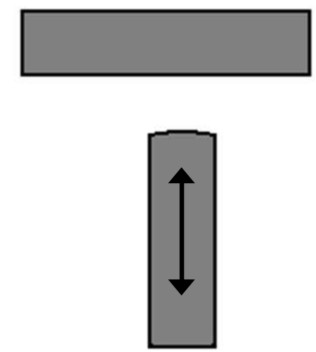
- **$F_d = k\Delta x$, where k = suspension stiffness and Δx is reduction in amplitude from non-contact**

Normal Contact Degradation



Kinetic Energy Upon Contact

$\frac{1}{2} mv^2 = 1.46\times10^{-15}$ J



10 Measurements per point

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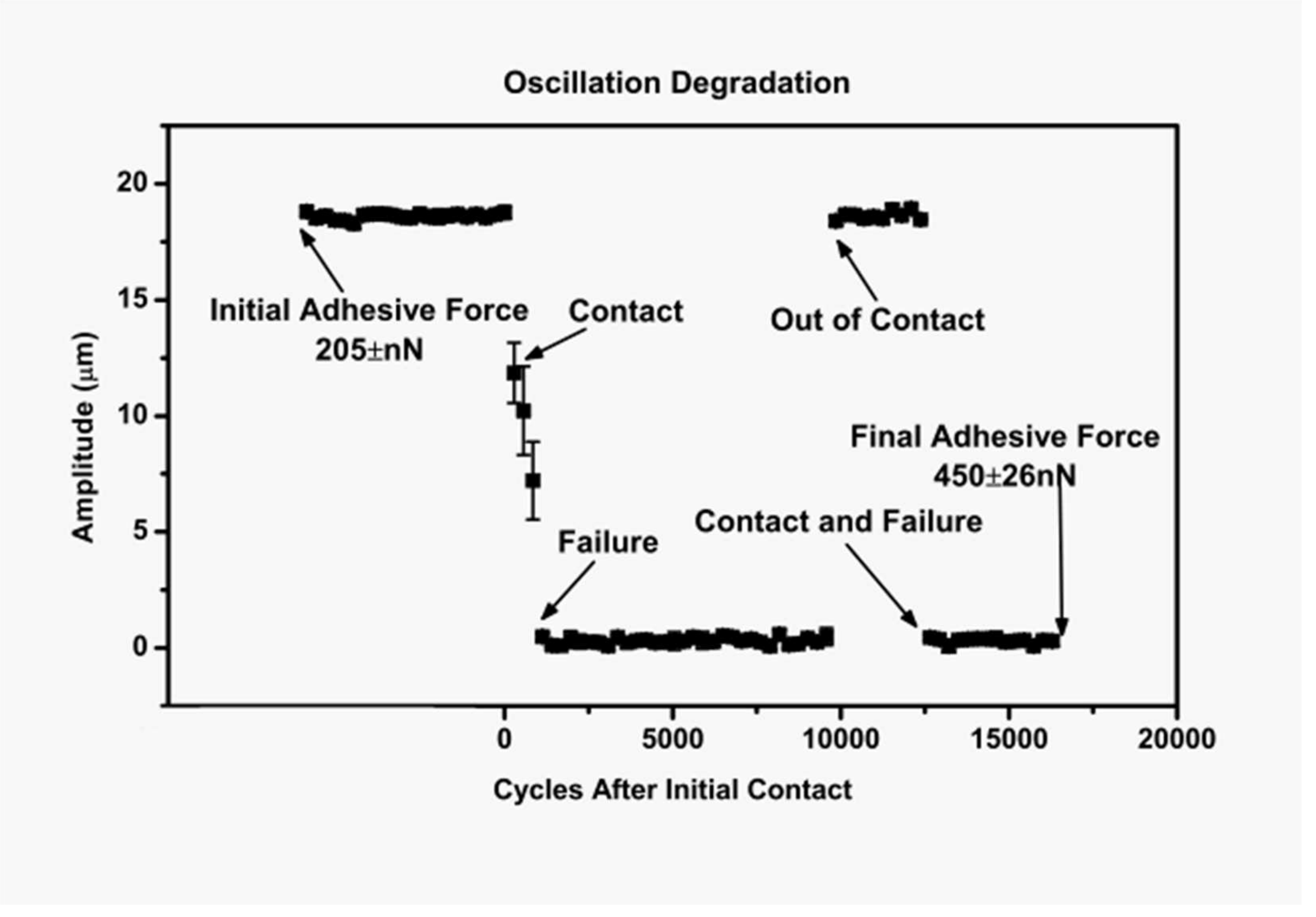
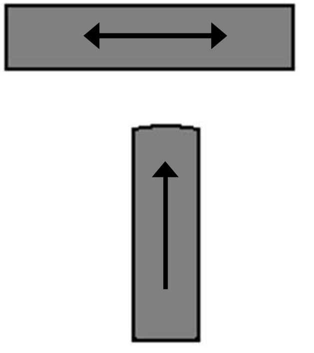
Shear Contact Degradation





National Nuclear Security Administration

Sandia National Laboratories

Contact Experiment Summary

Normal Contact

- Initial adhesion = 129 ± 13 nN
- 70000 cycles before adhesion change
- Equilibrium reached at 150000 cycles
- Final adhesion = 309 ± 35 nN
- 180 nN increase in adhesion (>2x)

Sliding Contact

- Initial Adhesion = 205 ± 20 nN
- Dynamic Friction Coeff. = 0.43 at first sample interval
  - work done by friction = 0.202 J/m^2 per cycle
- Failure at 800 cycles
- Final Adhesion = 423 ± 26 nN
- 218 nN increase in adhesion (~2x)
- Assuming 2 Si-O bonds per molecule, energy required to remove is 3.513 J/m^2

Conclusions

- **Monolayers degrade rapidly in sliding contacts**
- **Monolayers also degrade in normal contact in relatively short time**
- **Device failure can be directly related to monolayer failure**
  - increase in adhesive force leading to cessation of motion
  - wear particle generation
- **Energy imparted via sliding for 800 cycles is sufficient to break all Si-O bonds in the monolayer**
- **Energy imparted via normal contact for 70000 cycles is orders of magnitude greater than needed to break all Si-O bonds**

- **Earlier failure of monolayer is not observed due to**
  - **bond reformation**
  - **asperity contact locations evolving during sliding**
  - **energy being imparted to bulk silicon**

Acknowledgments

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