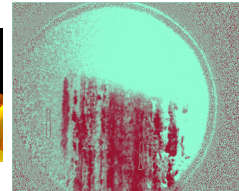
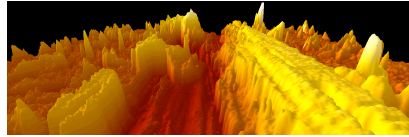


# Friction at the Nanometer Scale: Recent Experimental Advances



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## Acknowledgements

### *Nanomechanics Group:*

A. V. Sumant, Erin E. Flater, David Grierson, Andrew Konicek, Gelsomina  
"pupa" De Stasio  
U. Wisconsin-Madison

### *Silicon MEMS:*

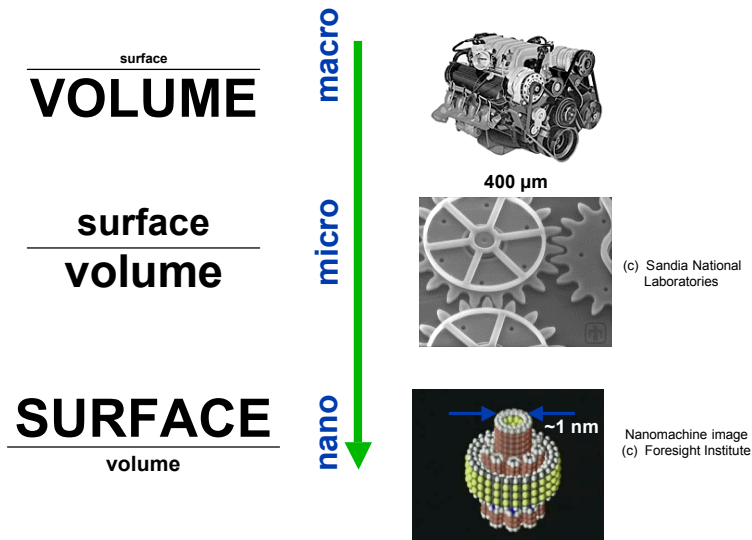
Maarten P. de Boer, Alex D. Corwin, E. David Reedy, Tom Mayer, M.  
Dugger, T. Scharf  
Sandia National Laboratories  
W. Robert Ashurst  
Auburn University

### *Ultrananocrystalline Diamond:*

John A. Carlisle, Orlando Auciello, Jennifer E. Gerbi, James Birrell  
Argonne National Laboratories

*National Science Foundation, Air Force Office of Scientific Research, Department  
of Energy, Sandia National Laboratories, Argonne National Laboratories  
Synchrotron Radiation Center (UW-Madison), Advanced Light Source (Lawrence  
Berkeley National Laboratory)*

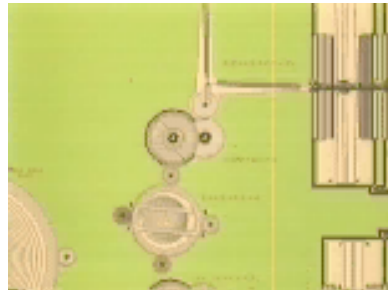
Surface forces (adhesion, friction)  
are increasingly important at small scales



## Micro-electro-mechanical systems (MEMS)

Examples:

- air bag accelerometers
- digital micromirror device (DMD)
- resonators & switches

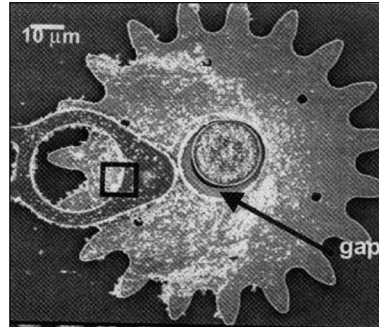
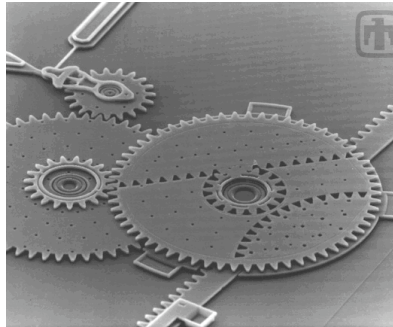


Images (c) Sandia National Laboratories

Small is beautiful.....

but complex as well

Images (c)  
Sandia National  
Laboratories

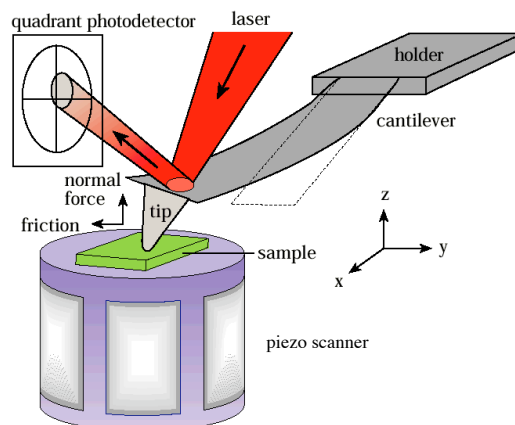


Problems with Silicon:

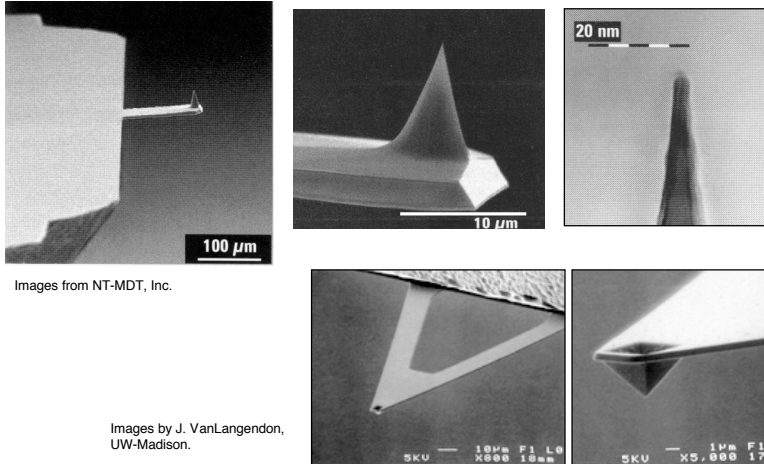
- Hydrophilic, reactive surfaces (adhesion)
  - Part stuck together after processing, during operation
- High friction
- Low fracture toughness
  - Fracture, wear

**Strategies:**     (1) *Tailor Si surface to reduce friction/adhesion/wear*  
                          (2) *Replace silicon with diamond*

The atomic force microscope senses force in  
nano-contacts at the nanoNewton level



The AFM probe is a microfabricated cantilever ( $\sim 100\ \mu\text{m}$ ) and tip ( $< 50\ \text{nm}$  radius)

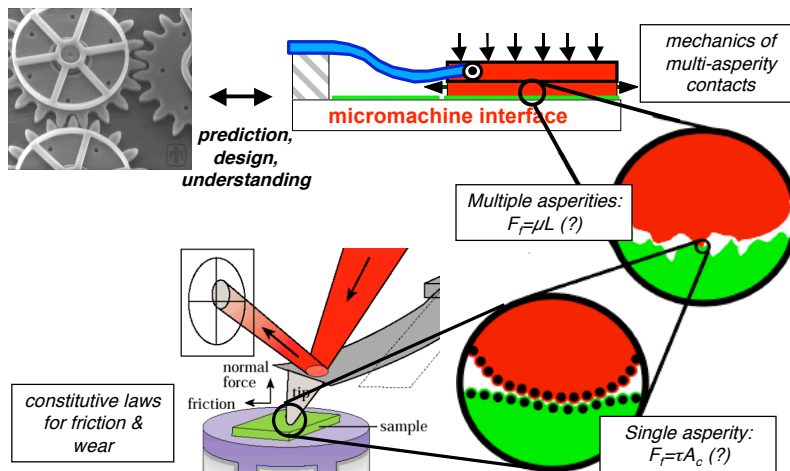


## Quantitative AFM experiments are carried out

- Si tips: uncoated, *or* with a monolayer coating prepared identically with the samples at the same time
- Normal forces calibrated using the “resonance-damping method”
  - *Sader (RSI, 1999)*
- Lateral forces calibrated using the “wedge” technique
  - *Ogletree, Carpick, Salmeron (RSI, 1996), Varenberg (RSI, 2003)*
- Tip shape checked before and after using “inverse imaging” and TEM
  - *Villarrubia (JVST, 1996), P.M. Williams (JVST, 1999)*
- Experiments repeated (back and forth between the two samples)
- Friction measured as a function of load, fit to continuum adhesive contact model with variable range of adhesion
  - *Carpick, Ogletree, Salmeron (J. Coll. Int. Sci., 1998)*
- **Note: MatLab scripts for applying our calibration methods to DI AFM measurements are available on our website**



We seek to understand MEMS friction through multi-scale experiments and modeling



### Strategy (1): Tailoring the surface of silicon

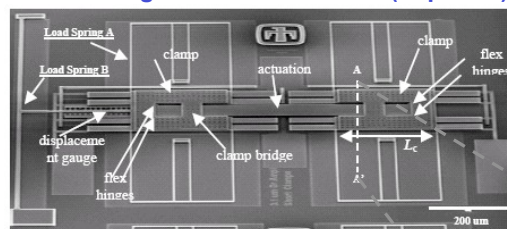
- How do tailored silicon surfaces behave in MEMS devices?
- How do tailored silicon surfaces behave at the nano-scale?

## Strategy (1): Tailoring the surface of silicon

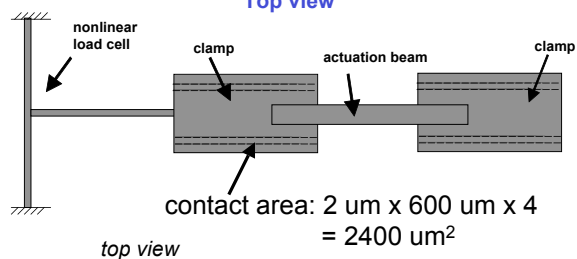
- How do tailored silicon surfaces behave in MEMS devices?
- How do tailored silicon surfaces behave at the nano-scale?

Sandia's nanotractor is designed to study friction and wear mechanisms at nanoscale

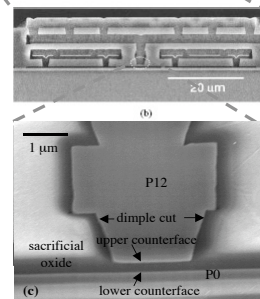
SEM image of the Nanotractor (Top view)



Top view



Cross section



de Boer M. P. *et al.*, J. MEMS 13 (1) 2004

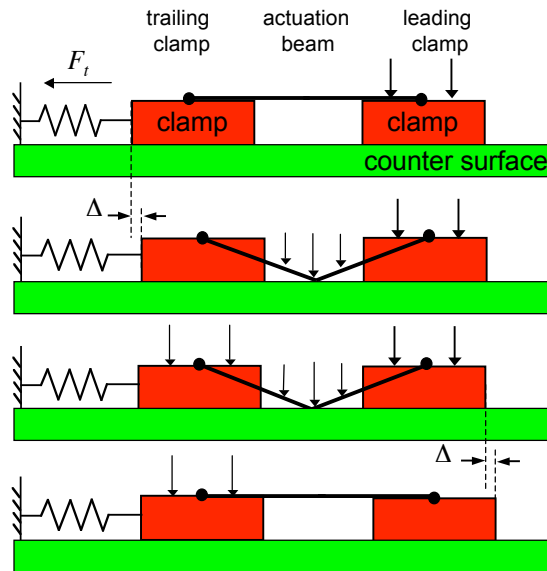
## The “nanotractor” has been designed to quantify surface forces in MEMS



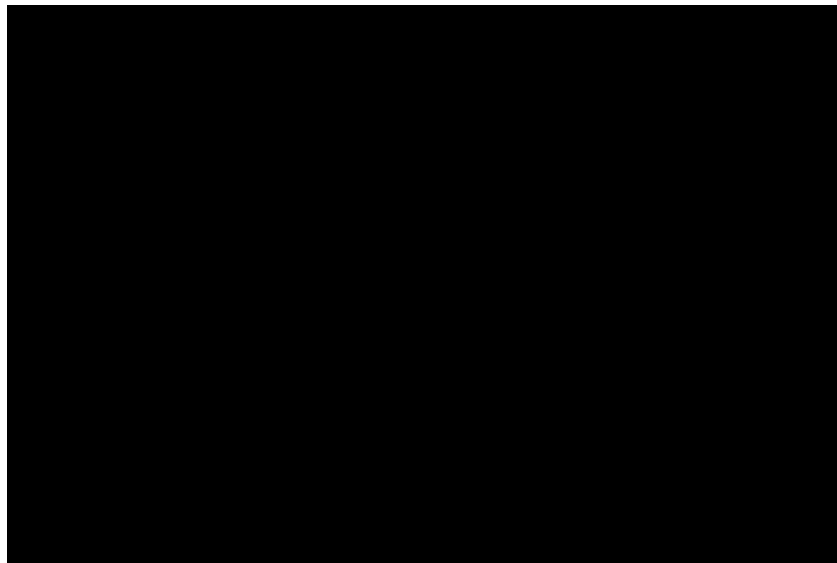
Schematic of the  
nanotractor  
(SIDE VIEW)

M.P.de Boer, D.L.Luck,  
W.R.Ashurst, R.Maboudian  
*J.MEMS* 13(1) 2004

E.E.Flater, A.D.Corwin,  
M.P.de Boer, R.W.Carpick  
*Wear* 2005 in press



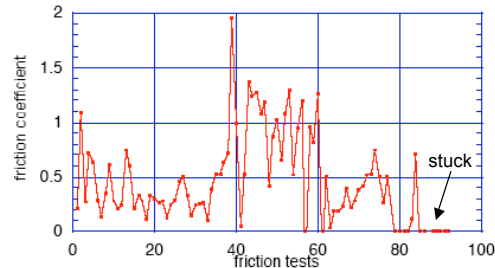
## The Nanotractor in action...



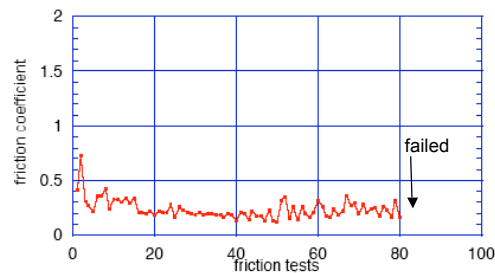
Movie courtesy of Sandia National Laboratories

## Self-assembled monolayers improve the performance and lifetime of the device

uncoated



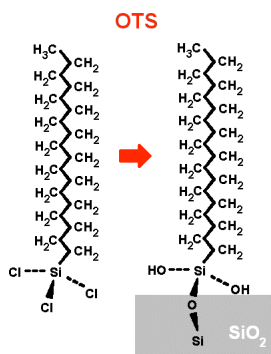
coated



E.E.Flater,  
A.D.Corwin, M.P.de  
Boer, R.W.Carpick  
*Wear*, in press (2005)

## We study two monolayers which are successfully integrated into the MEMS process flow

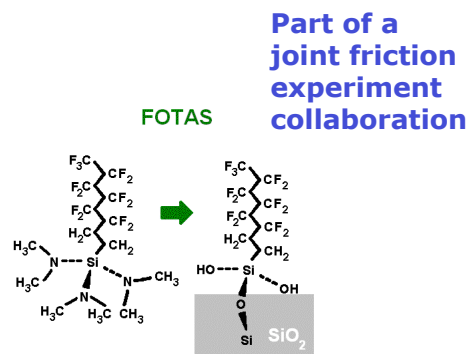
**Hydrogenated**



OTS=OctadecylTrichloroSilane

OTS prepared by W. Robert Ashurst  
(UC Berkeley, now at Auburn University)  
using solvent deposition

**Fluorinated**

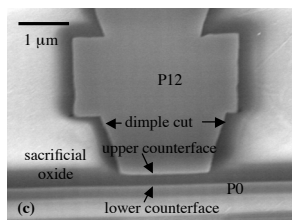
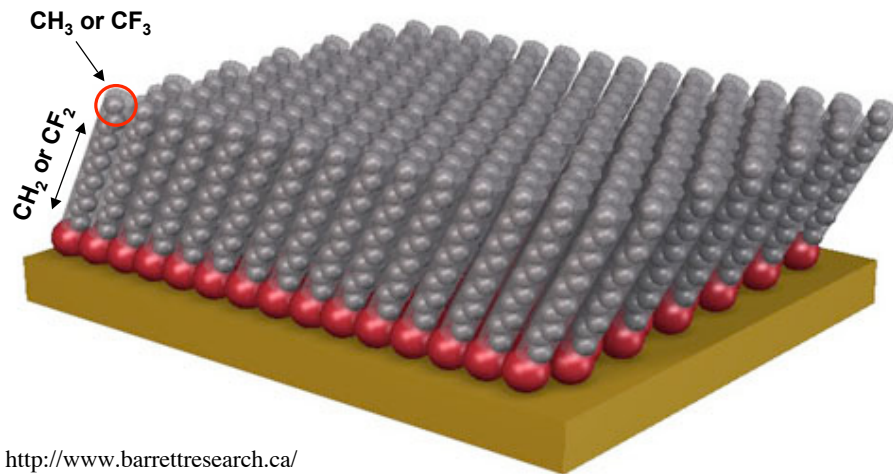


FOTAS=(tridecaFluoro-1,1,2,2-tetrahydroOctyl)Tris(dimethylAmino)Silane

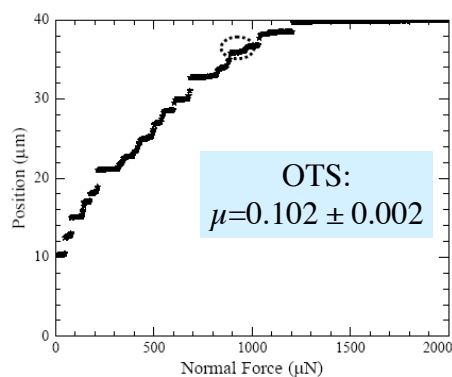
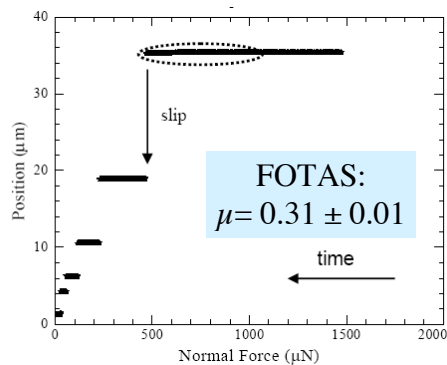
FOTAS prepared by Tom Mayer  
(Sandia National Labs)  
using vapor deposition

Part of a  
joint friction  
experiment  
collaboration

We study two monolayers which are successfully integrated into the MEMS process flow



Nanotractor friction measurements reveal contrast between OTS and FOTAS



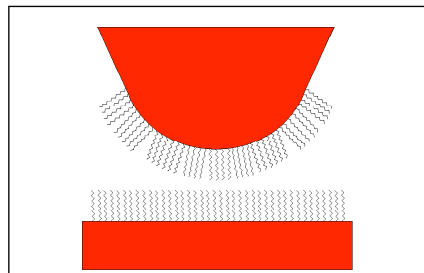
Proceedings of the 2004 ASME/STLE International Joint Tribology Conference  
A.D. Corwin, M.D. Street, R.W. Carpick, W.R. Ashurst, M.P. de Boer

**Strategy (1):  
Tailoring the surface of silicon**

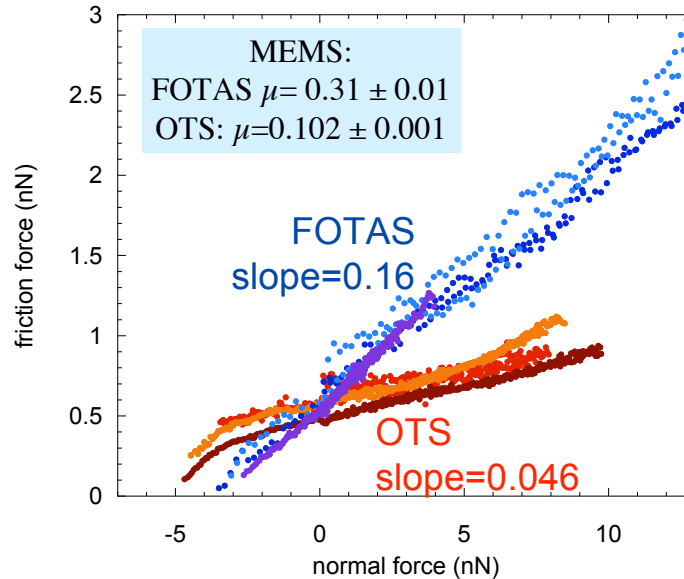
- How do tailored silicon surfaces behave in MEMS devices?
- How do tailored silicon surfaces behave at the nano-scale?

*Coated AFM tips and substrates*

- tips, flats, and MEMS devices coated at the same time with the same SAMs
    - R. Ashurst\* & R. Maboudian, UC Berkeley
    - direct comparisons with nanotractor measurements
- \*now at Auburn U.



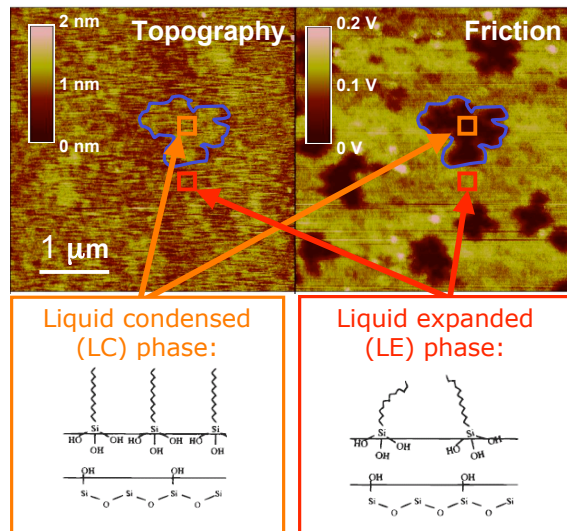
## OTS/OTS vs FOTAS/FOTAS



## Connection between nano- and micro-scale friction?

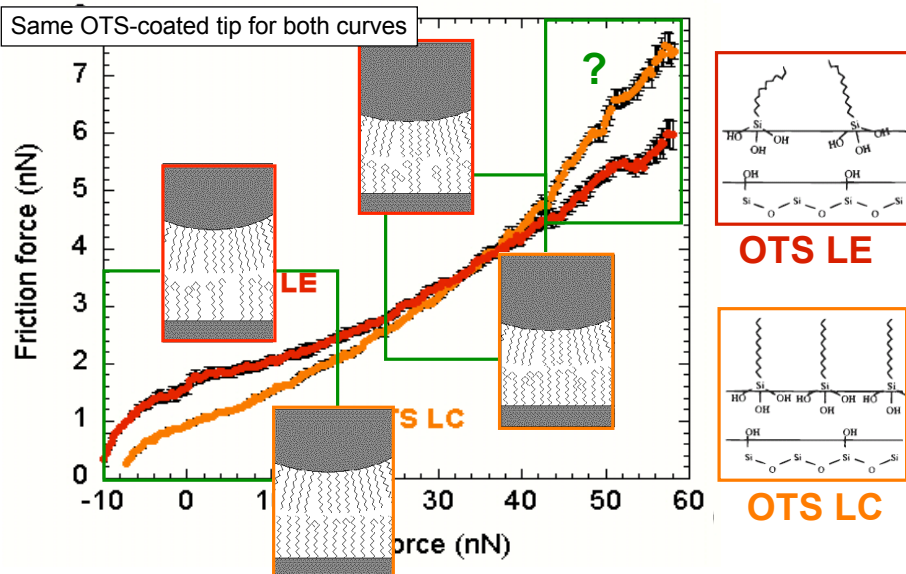
- It is not necessarily the case that the ratio of friction coefficients for rough surfaces should be equal to the ratio of friction slopes in single-asperity contacts
- Our case:
  - From MEMS, the ratio for FOTAS:OTS is  $\sim 3.0$
  - From AFM, the ratio for FOTAS:OTS is  $\sim 3.5$

## Unique two-phase surface allows for direct analysis of the role of packing density



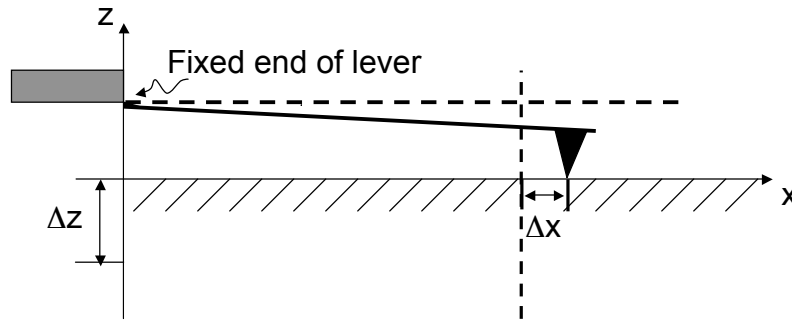
From: Carraro *et al. J. Phys. Chem. B* (1998)

## Differences in friction between the OTS phases correlate well with their packing structures



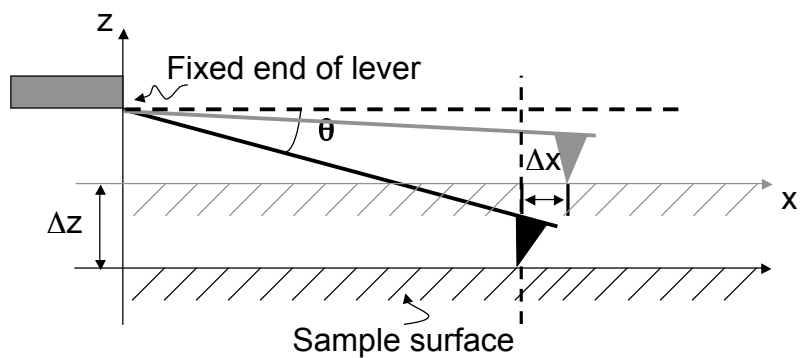


The free end of the lever displaces as the applied load varies



Cannara & Carpick *RSI*, 76(5) 2005

The magnitude of the tilt effect is a simple geometric relation

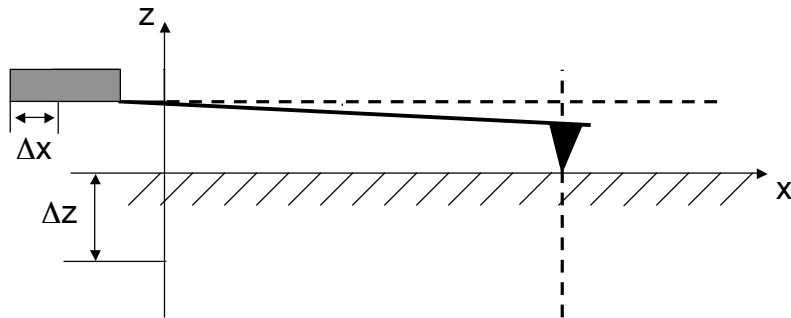


$$\Delta x = \sqrt{L^2 - (L \sin \theta - \Delta z)^2} - L \cos \theta$$

To first order,  $\Delta x = \Delta z \tan \theta$

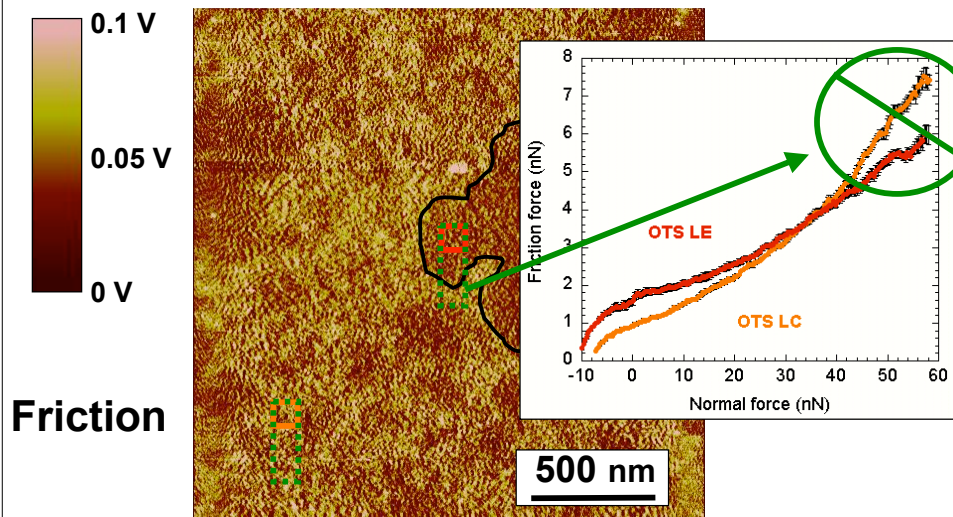
Cannara & Carpick *RSI*, 76(5) 2005

Tilt-compensation allows one to stay within the intended region of analysis

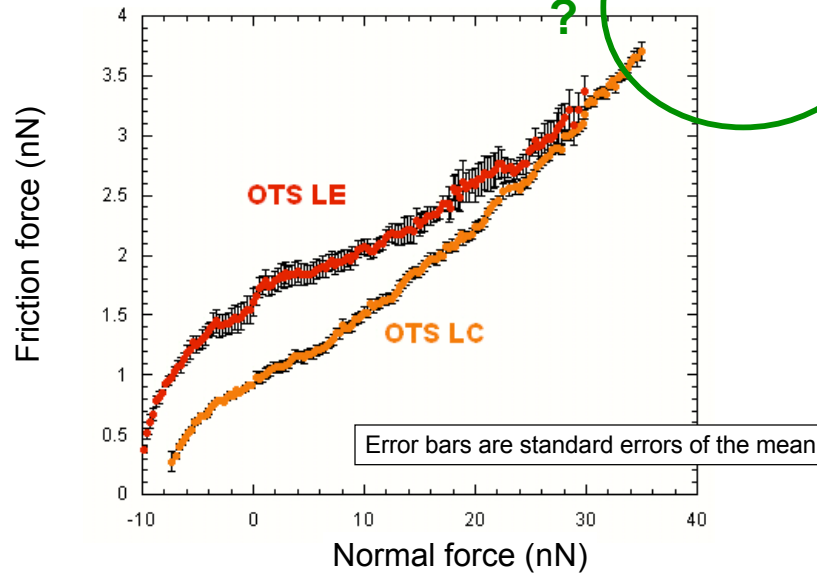


Cannara & Carpick *RSI*, 76(5) 2005

Friction measurements depend on location on sample



## Re-analysis of friction measurements does not show friction cross-over



## Is it a question of contact area?

- Junction model (Tabor)

$$F_f = \tau A$$

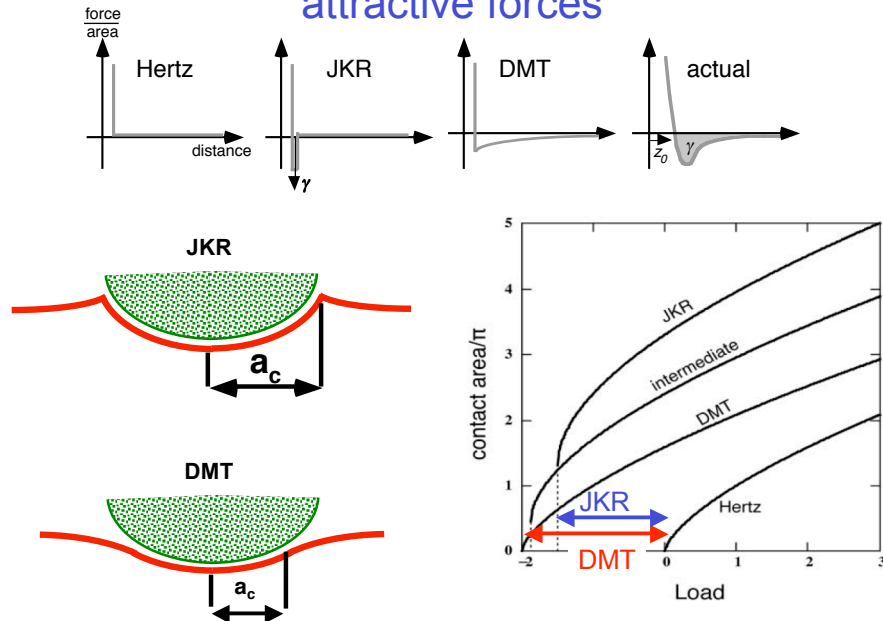
$F_f$  = friction force

$\tau$  = interfacial shear strength (units of stress)

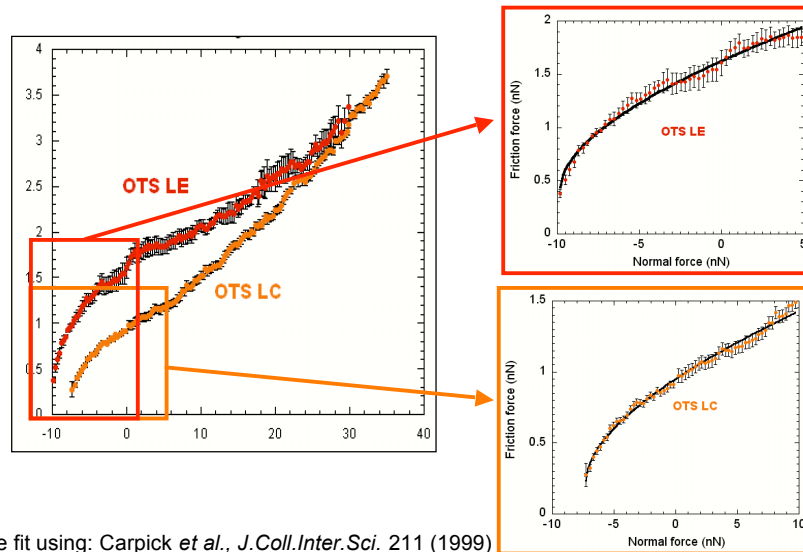
$A$  = contact area at interface

- What is  $A$ ?

## Contact properties depend on the range of attractive forces

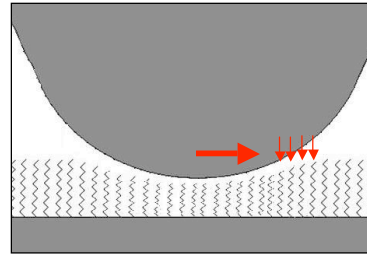
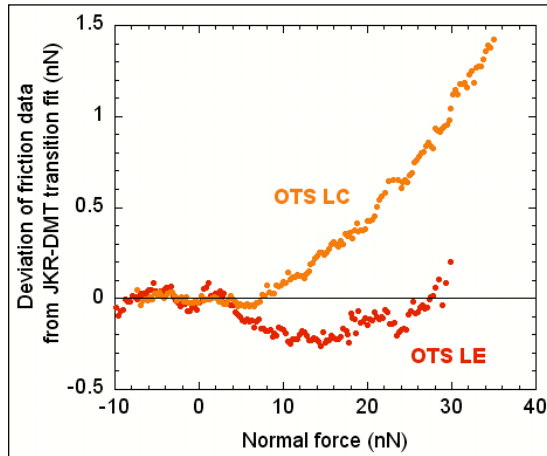


## Continuum contact mechanics fits describe frictional behavior at low loads



## Deviations from continuum models at high loads indicate “plowing” behavior

Friction data after subtracting the curve fit

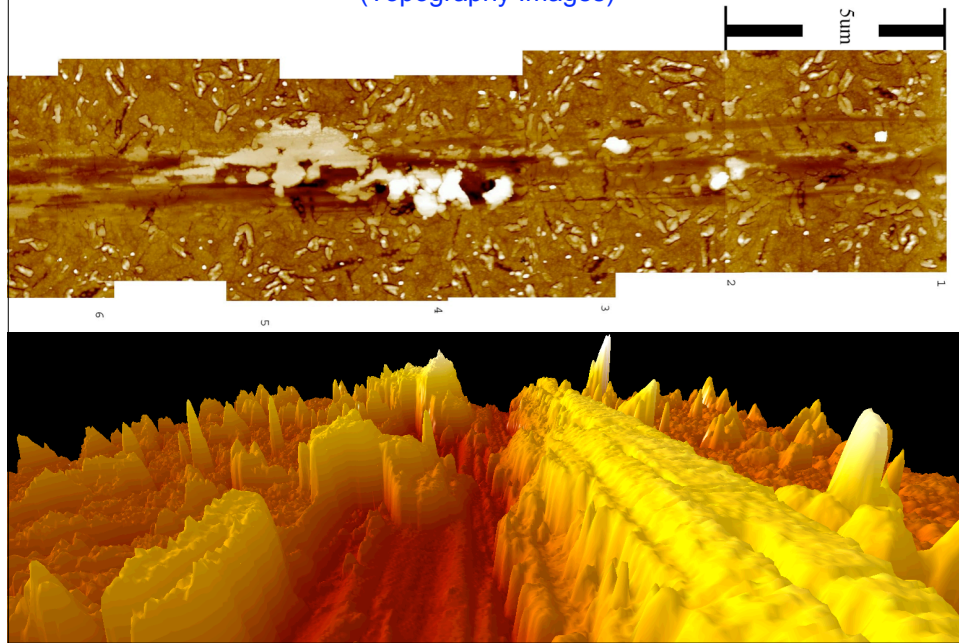


## Connection between nano- and micro-scale friction?

Hypothesis only!

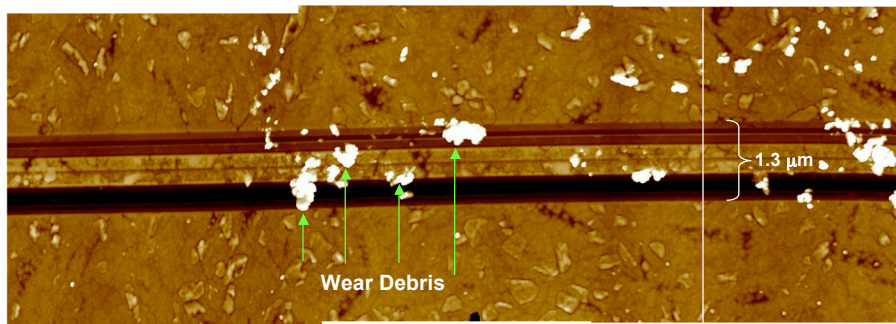
- OTS Condensed phase:**
  - Low loads: adhesive contact with a well-ordered monolayer with  $\text{CH}_3\text{-CH}_3$  groups in contact
  - Medium loads: pressure-dependent increase as plowing occurs (may include gauche defects formation)
  - Higher loads: yet to be determined
- OTS Expanded phase:**
  - Low loads: adhesive contact with a defective monolayer with many  $\text{CH}_2\text{-CH}_2$  groups in contact - more adhesion & more contact area than for the condensed phase
  - Medium loads: simply an increase in contact area, perhaps with stiffening of the layer, but not plowing
  - Higher loads: yet to be determined.
- FOTAS:**
  - Fluorinated films are stiffer - more work required to plow compared to OTS

## AFM Investigation of the wear track (Topography Images)



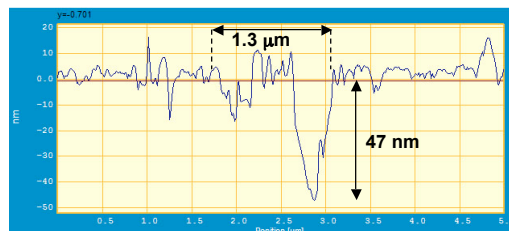
## Typical Signs of Wear: Gouging, Polishing, Debris Accumulation

Composite AFM image of the wear track



Ave. RMS roughness on  
unworn regions: 4.5 nm

Ave. RMS roughness in  
the wear track: 2.1 nm



## Conclusions

- SAM coatings substantially modify friction in MEMS, as determined by their molecular architecture
  - AFM single asperity measurements can be used to understand larger-scale friction behavior in MEMS
  - Tribochemical changes occur during wear processes in MEMS, and we need to study these further
- UNCD is a promising structural material for MEMS
  - Lower friction and adhesion than silicon at the nano-scale
  - Post growth H-plasma improves the surface chemistry and nanotribology of the bottom side. Adhesion approaches the van der Waals limit; friction is correspondingly low.  
⇒ *Is this the ideal tribological surface?*
- Tribology + imaging + spectroscopy = understanding friction?

**Thank you**