



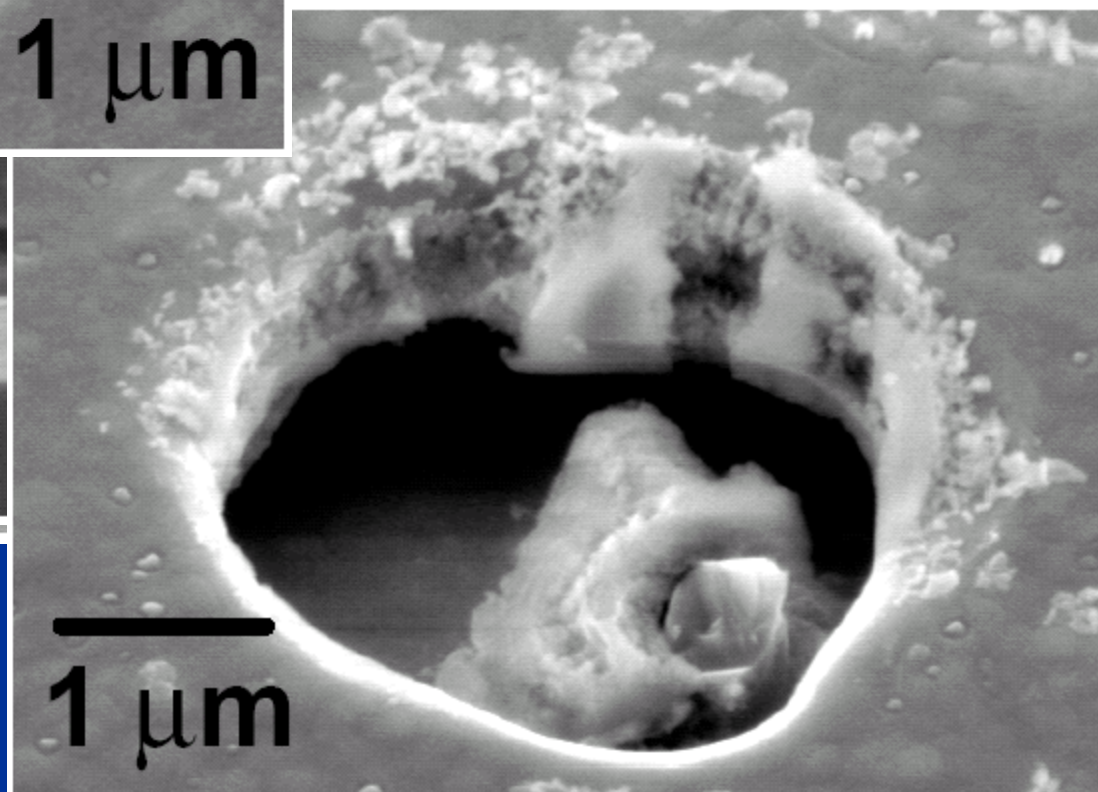
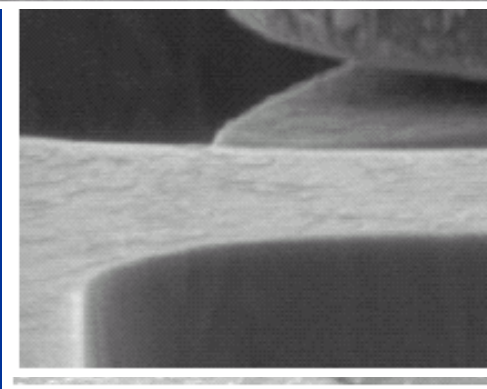
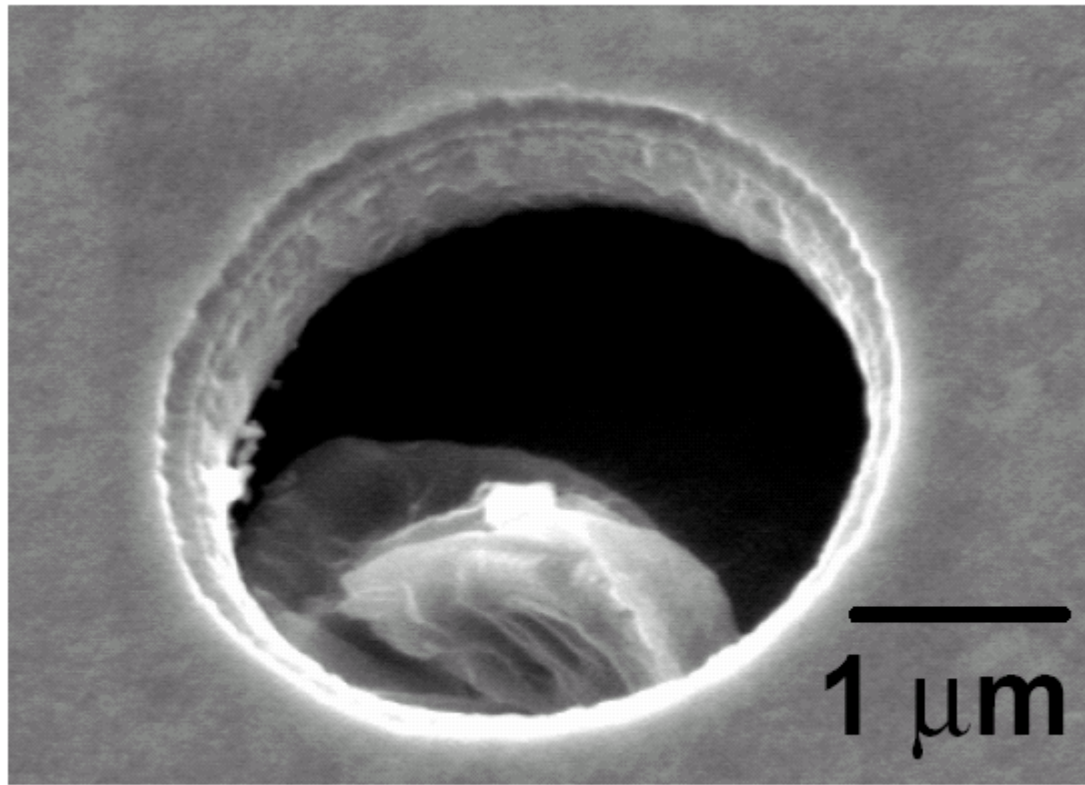
# Effects of Gas Adsorption in Nanotribology and Demonstration of in-situ Vapor Phase Lubrication of MEMS Devices

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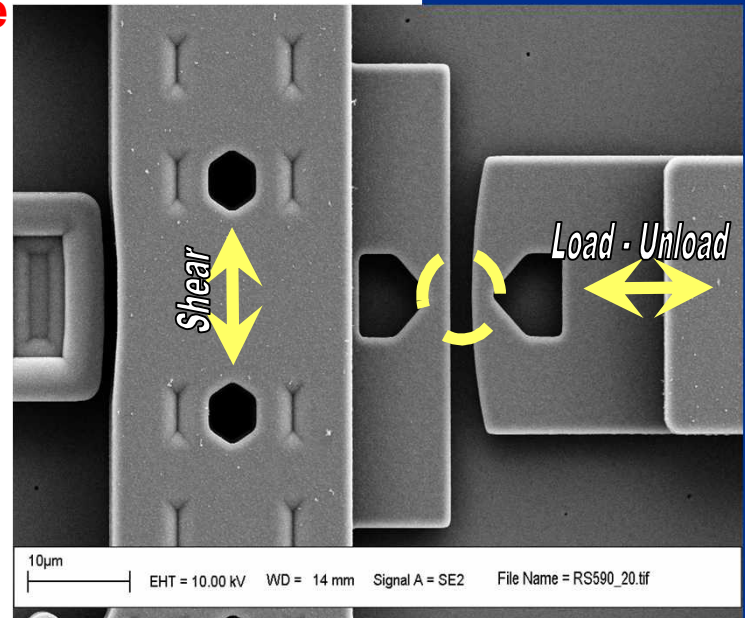
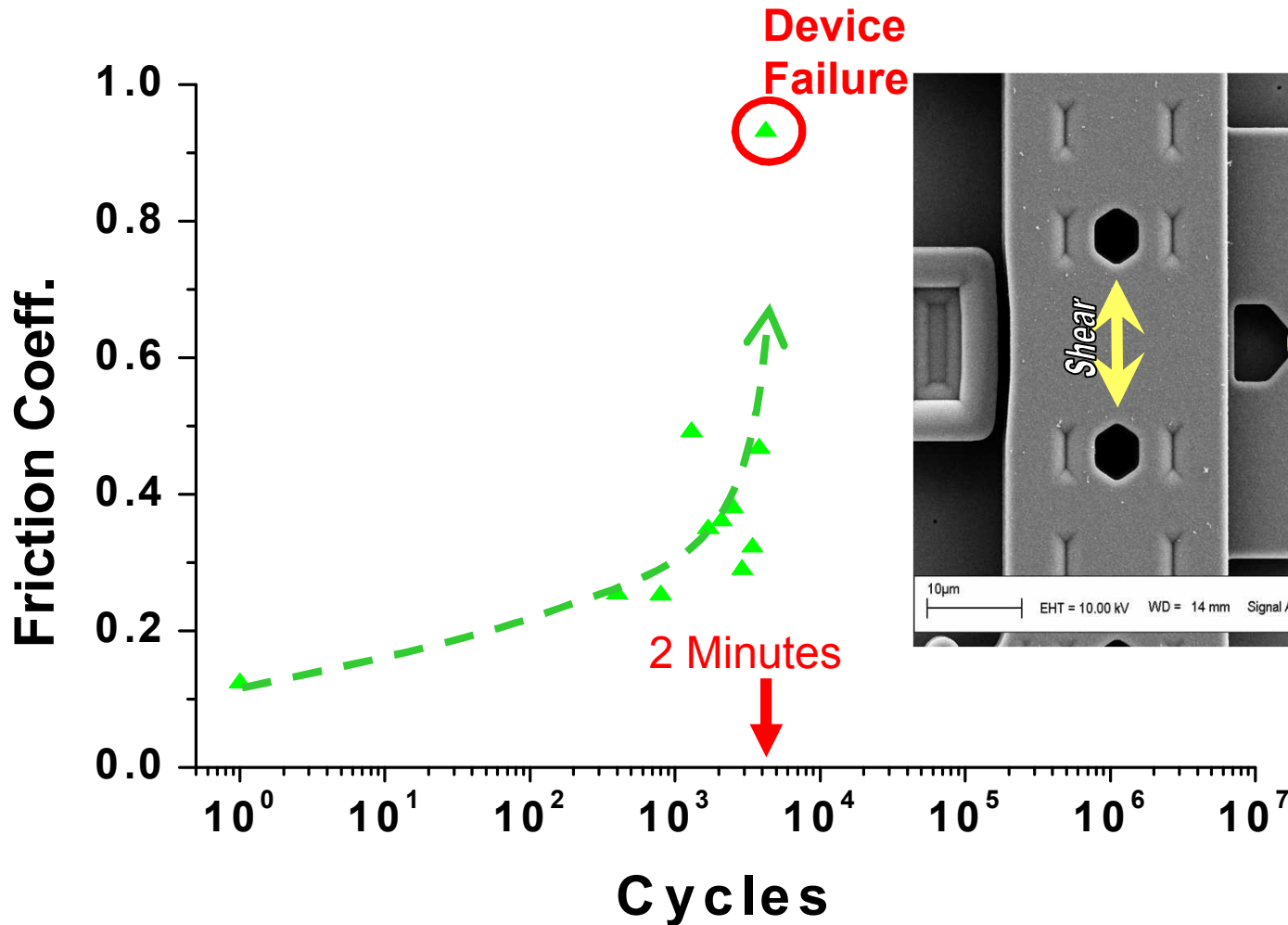


# Wear in nanoscale

$$\text{wear life} \propto \frac{\text{thickness}}{\text{erosion rate}} \propto L$$

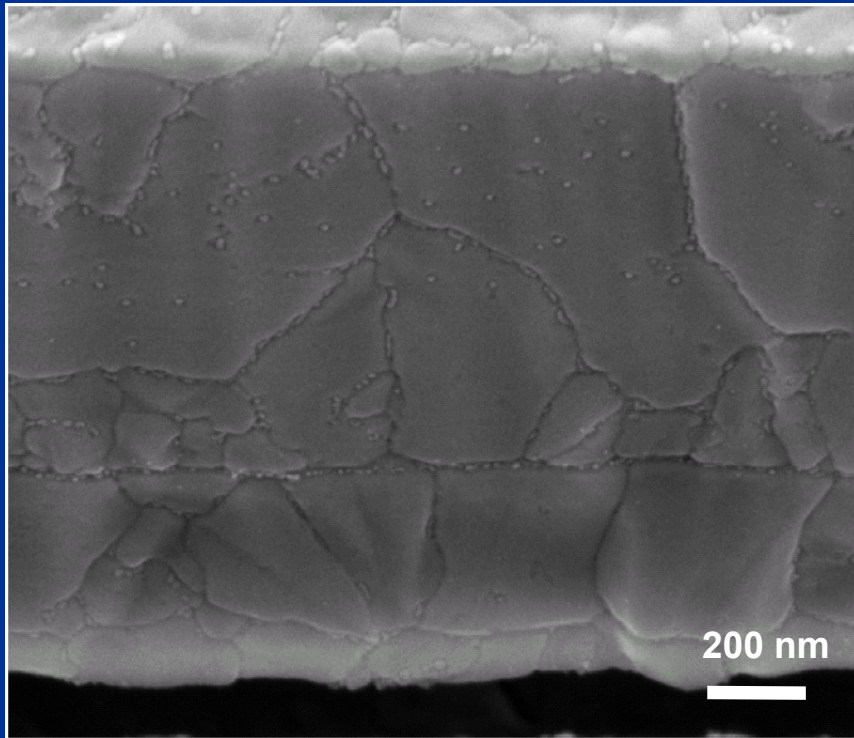
→ a centimeter-scale part having a 10 year lifetime would be expected to have a 30 sec lifetime if scaled to nanometer dimensions.

# Lifetime depends on wear...

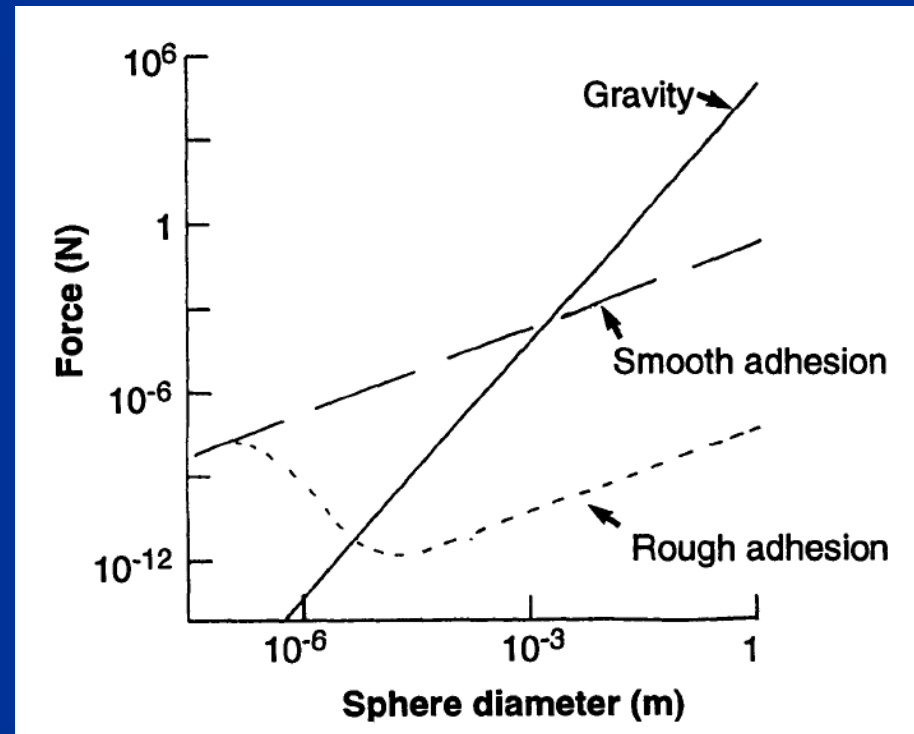


# Needs for nano-scale tribological study (adhesion, friction, wear)

Physical contacts in MEMS are composed of many nano-asperity contacts...



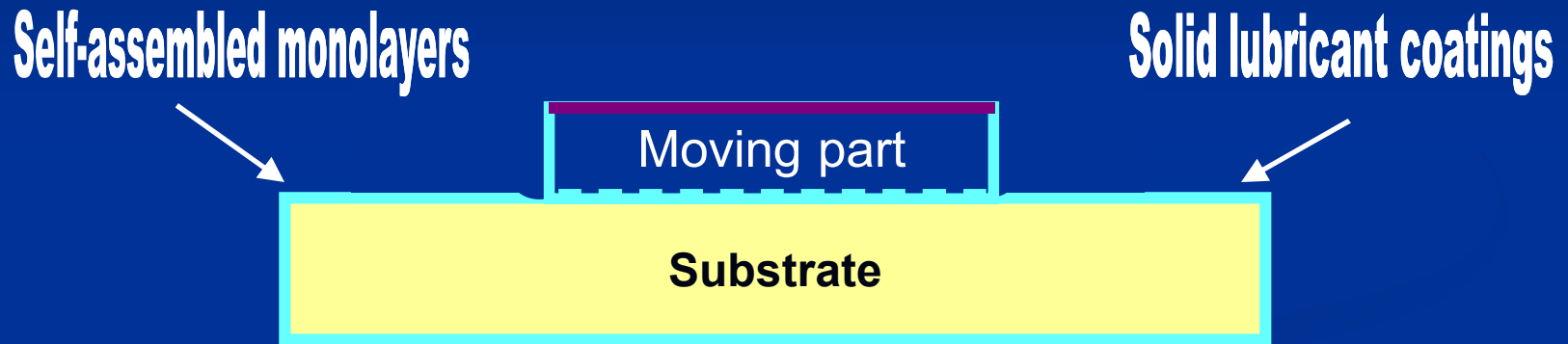
At nanoscale, forces that are negligible at the macro-scale become comparable to the actuation forces provided with on-chip actuators.



Kendall, *Science* **263**, 1720 (1994)

# Current “State-of-the Art”

## Solid lubricant (thin film coating)



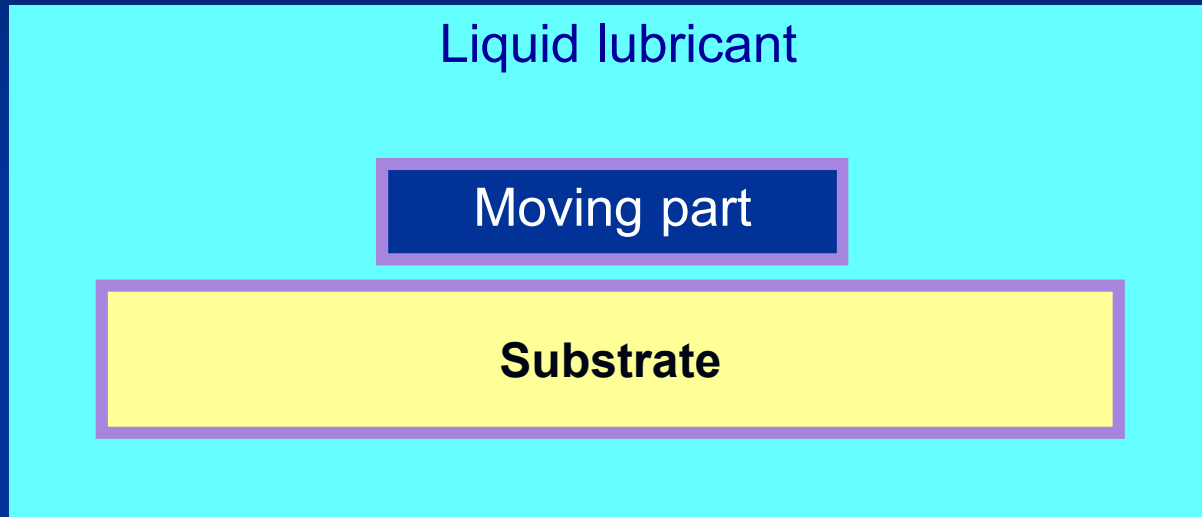
### Advantages:

- Easily incorporated into device while being fabricated
- Reduces friction

### Disadvantages:

- ✗ Not always conformal (cover all surfaces)
- ✗ Doesn't last forever → lubricant is not replenished

# Wisdom from conventional liquid-phase lubricants



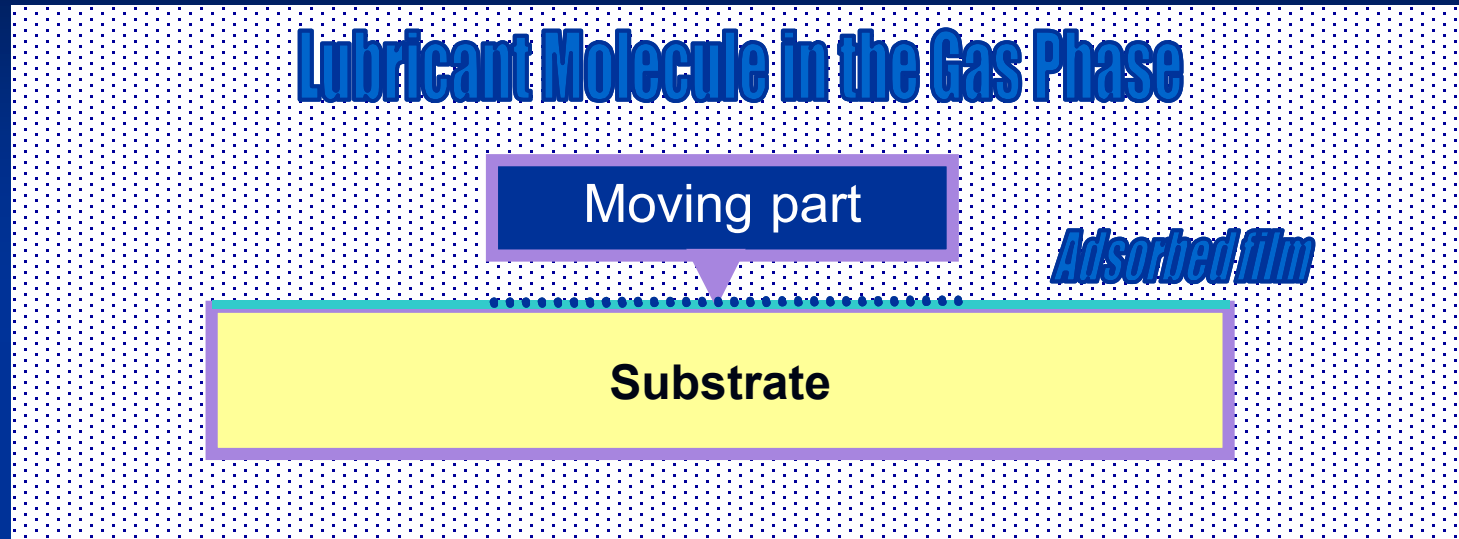
## Advantages:

- Continuously replenish lubrication film
- Liquid is fully conformal → works on buried surfaces

## Disadvantages esp. at micro- and nano-scales:

- ✗ Dragging of viscous liquid cause power dissipation problems

# *In-situ* Vapor Phase Lubrication



## Advantages:

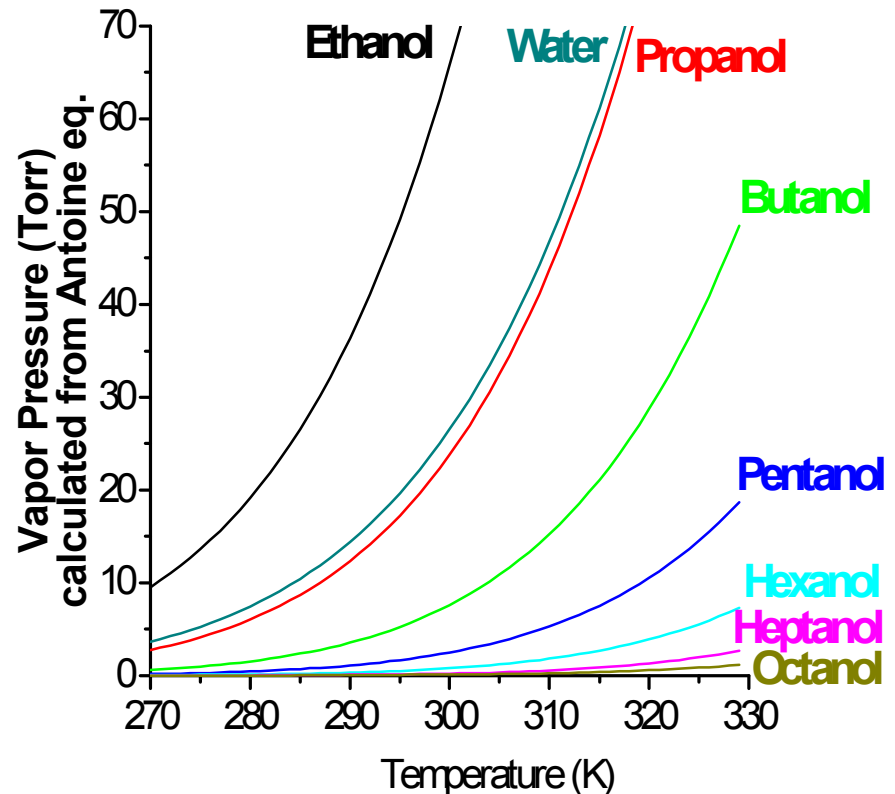
- Lubricant is continuously replenished
- Coats all surfaces exposed (Totally Conformal)
- No Power Dissipation (vs. liquid lubricants)



# Gas Phase Lubricants

## Primary Alcohols (Ethanol – Pentanol)

- Relatively high vapor pressure
- Few monolayers of water on hydrophilic surfaces
- Hydrogen bond with Si-OH surface groups
- LOW SURFACE ENERGY



### Key concept:

K. Strawhecker, D. B. Asay, J. McKinney, and S. H. Kim, *Tribol. Lett.* **2005**, 19, 17-21.

# KEY QUESTION

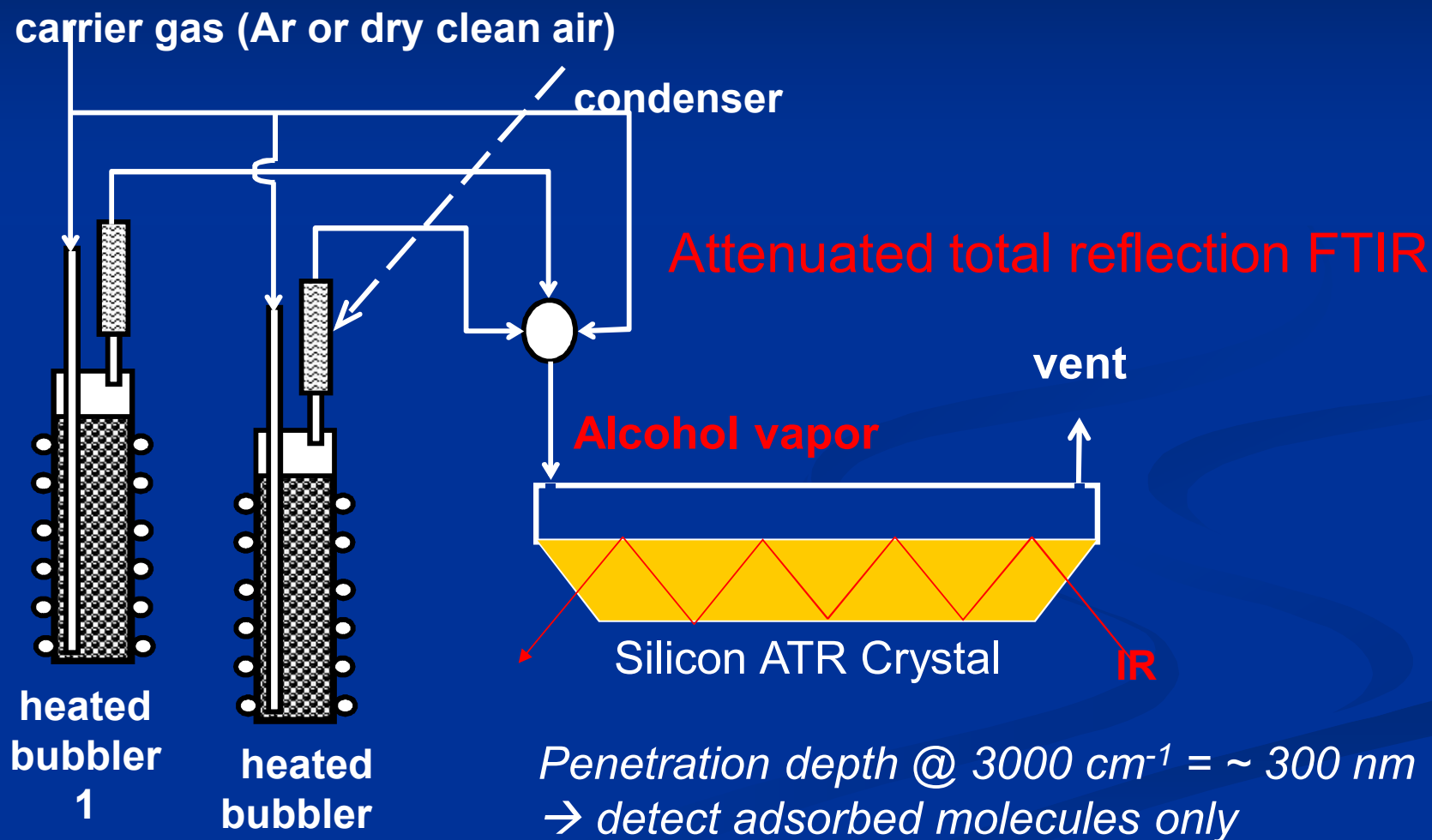
How does gas-adsorption affect the tribological properties of the surface?

- Effect on Adhesion
- Effect on Friction
- Effect on Wear

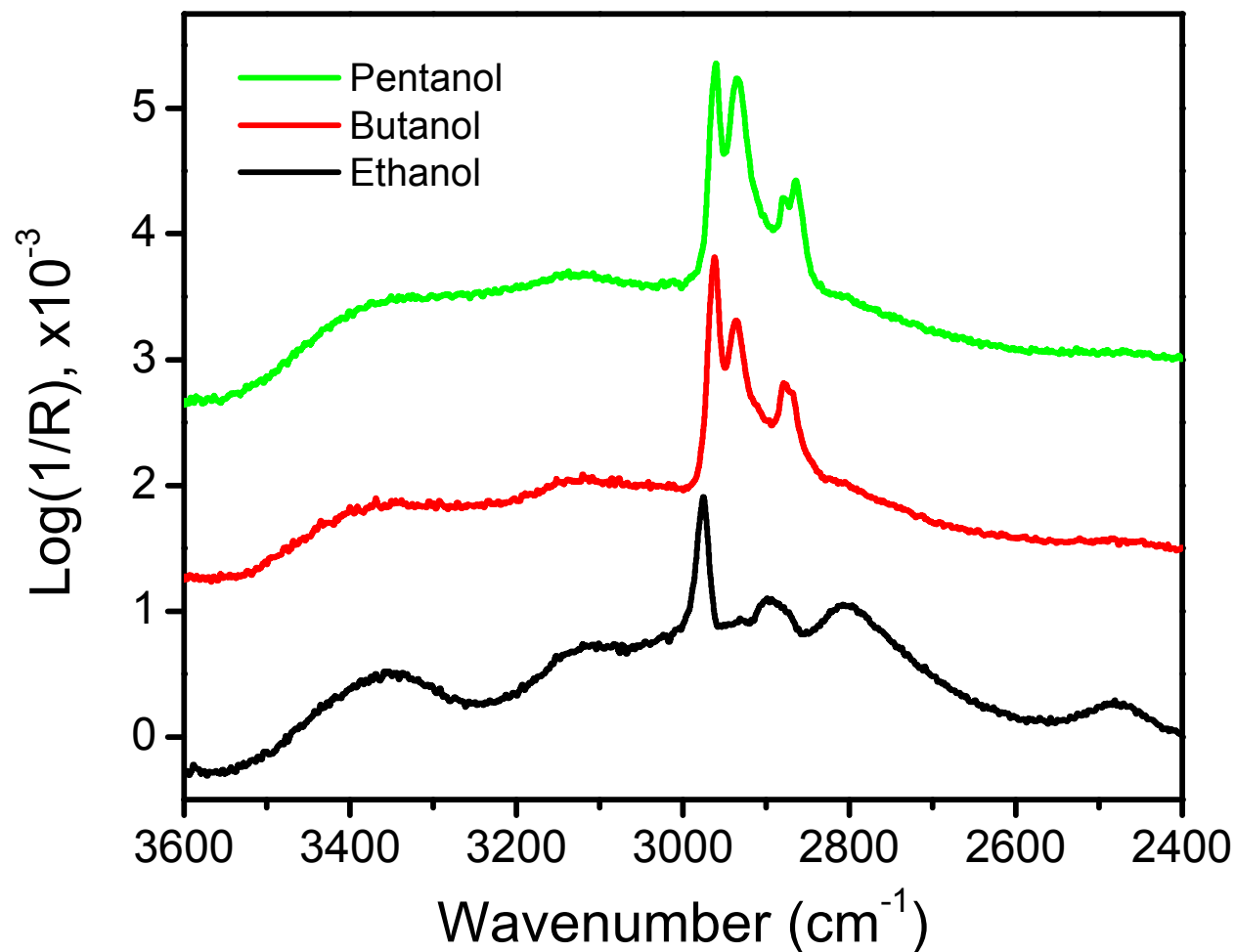
# 1. Adsorption Isotherm

How thick is the adsorbed film,  $f(P/P^{\text{sat}})$ ?

# ATR-IR $\rightarrow$ adsorption isomtherm



# ATR-IR @ 65% P/P<sup>sat</sup>

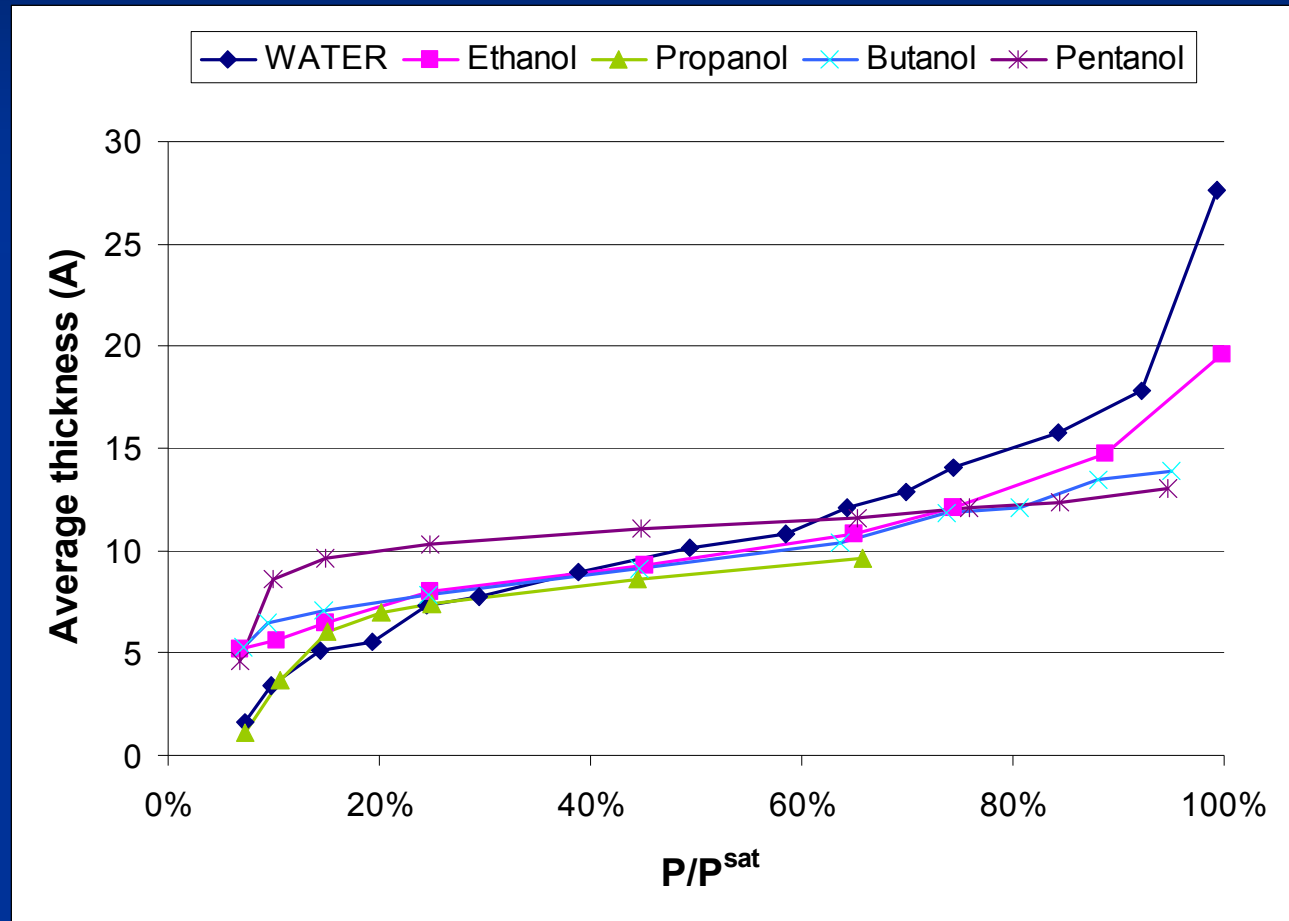


# Adsorption on Silicon

Carrier Gas:  
Argon

Temperature:  
21-23 °C

Error:  
 $\pm 2 - 3 \text{ \AA}$   
 $\pm 3\% \text{ P/P}_{\text{sat}}$



*Measured via: ATR-FTIR*

## 2. Tribological Properties

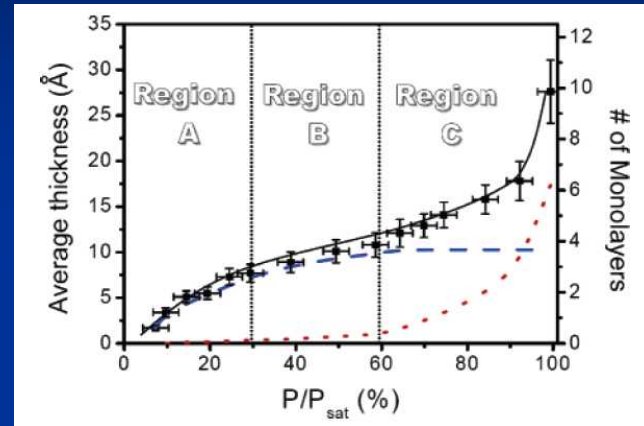
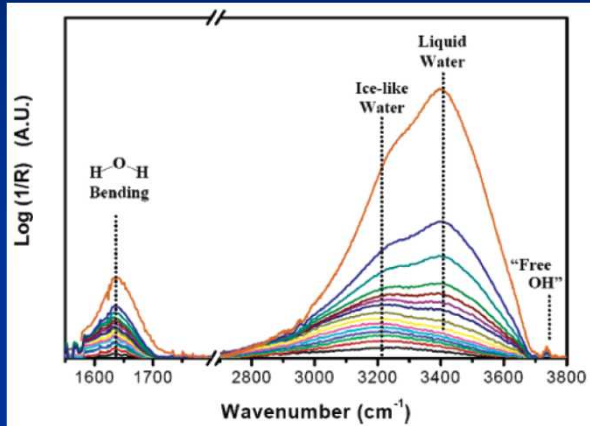
Nano-scale study : Atomic Force Microscope (AFM)

Micro-scale study : MEMS

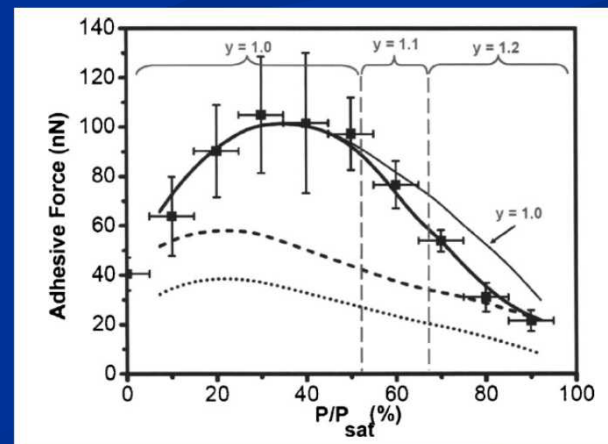
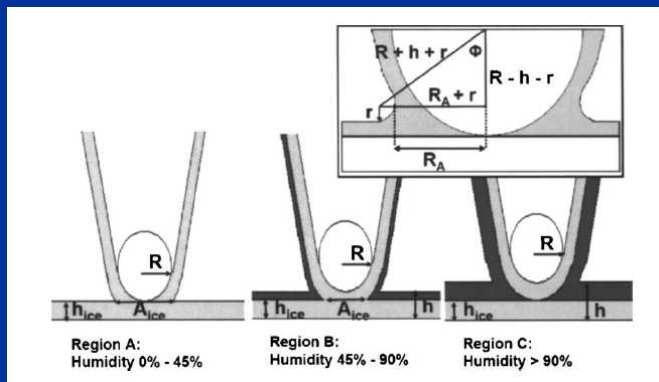
Macro-scale study: Linear Wear Test

# Effects of water adsorption

## ■ Adsorption isotherm and structure



## ■ Adhesion and structure



D. B. Asay and S. H. Kim, *J. Phys. Chem. B* **2005**, *109*, 16760-16763.

D. B. Asay and S. H. Kim, *J. Chem. Phys.* **2006**, *124*, 174712-1 - 174712-5.



# Nano-asperity adhesion in alcohol vapor environment

# Single Asperity Adhesion

Young's Equation

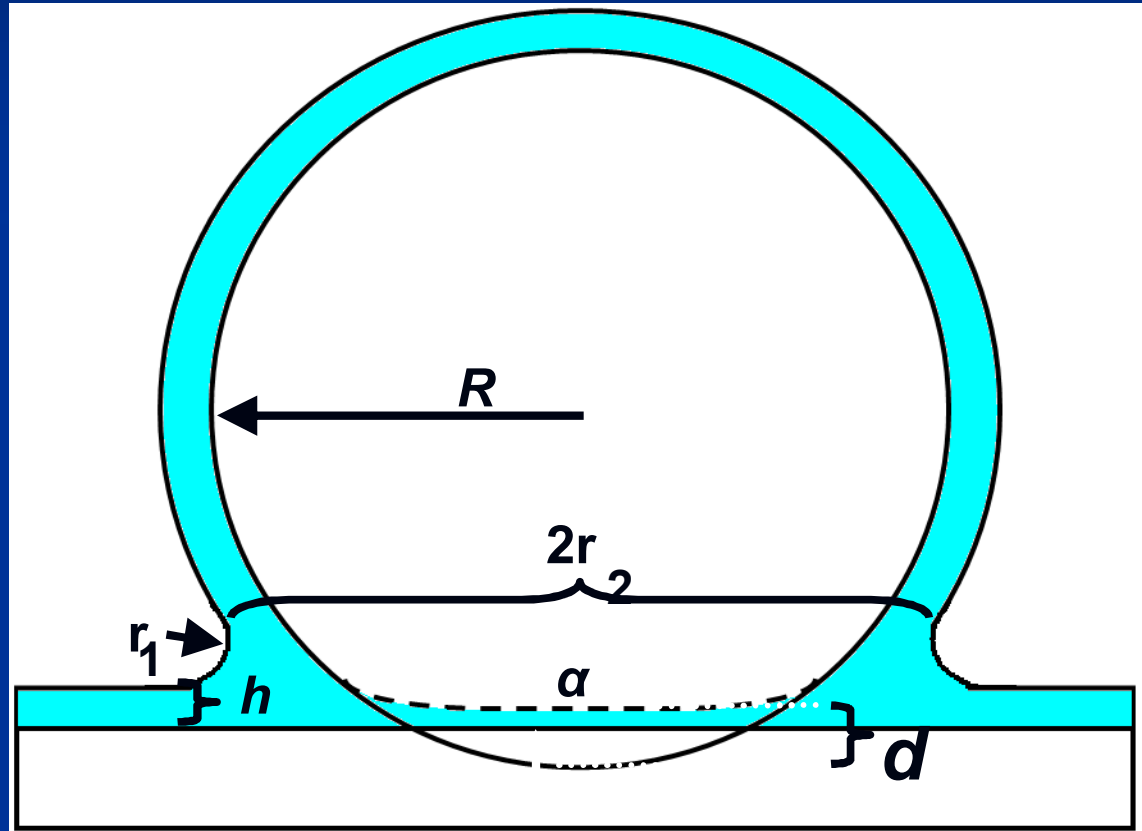
$$P_{Laplace} \equiv \gamma_L \left( \frac{1}{r_1} + \frac{1}{r_2} \right)$$

Mechanical Equilibrium

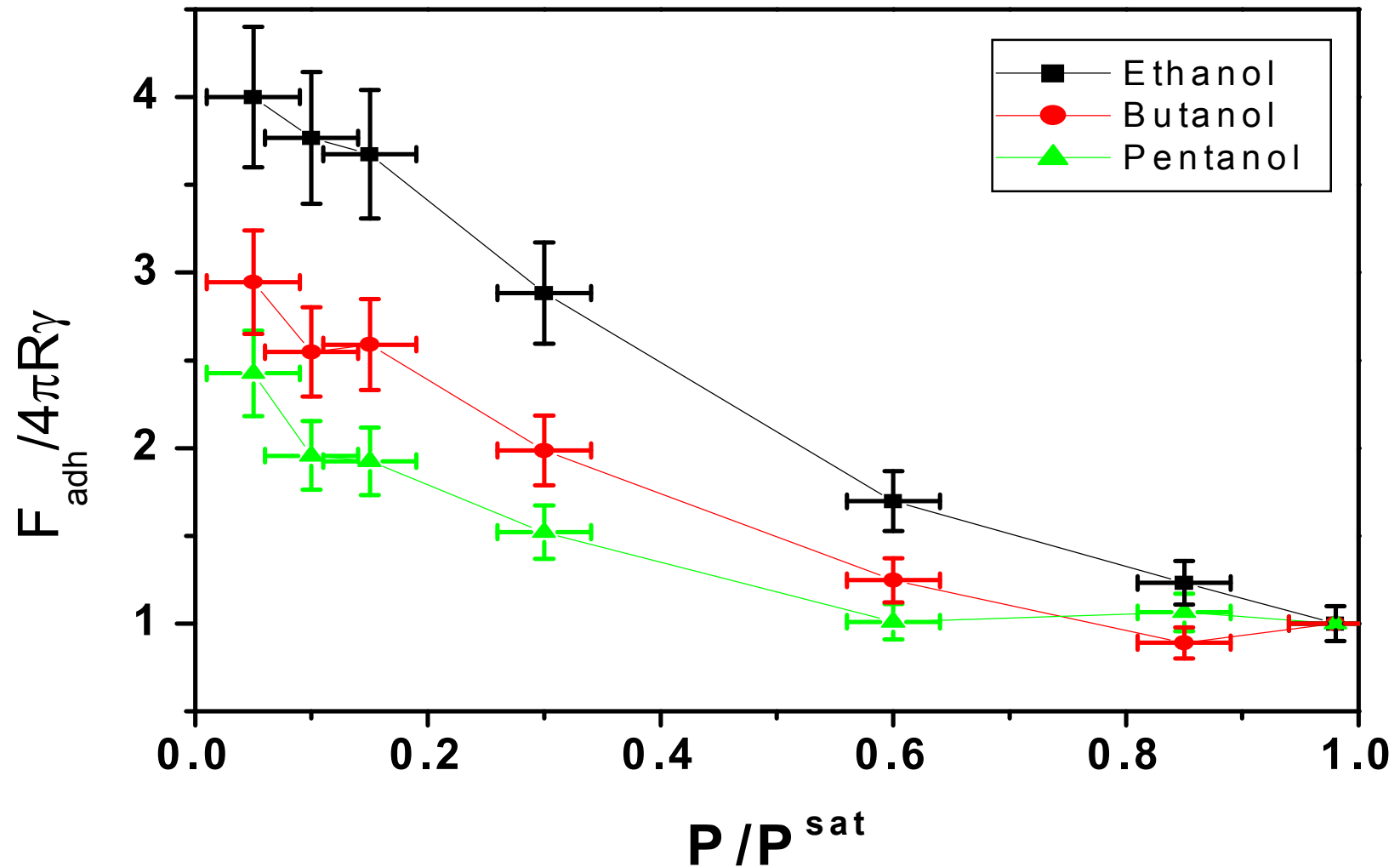
Kelvin Equation

$$P_{Laplace} \equiv \frac{R_g T}{V} \ln \left( \frac{P}{P^{sat}} \right)$$

Chemical Equilibrium



# Capillary force in alcohol vapor environment



# Why the difference?

## Macroscopic view of capillary force

→ A liquid with a higher surface tension gives a larger capillary force.

	$\gamma$ (erg/cm <sup>2</sup> )
Ethanol	22.8
1-butanol	24.6
1-pentanol	24.9

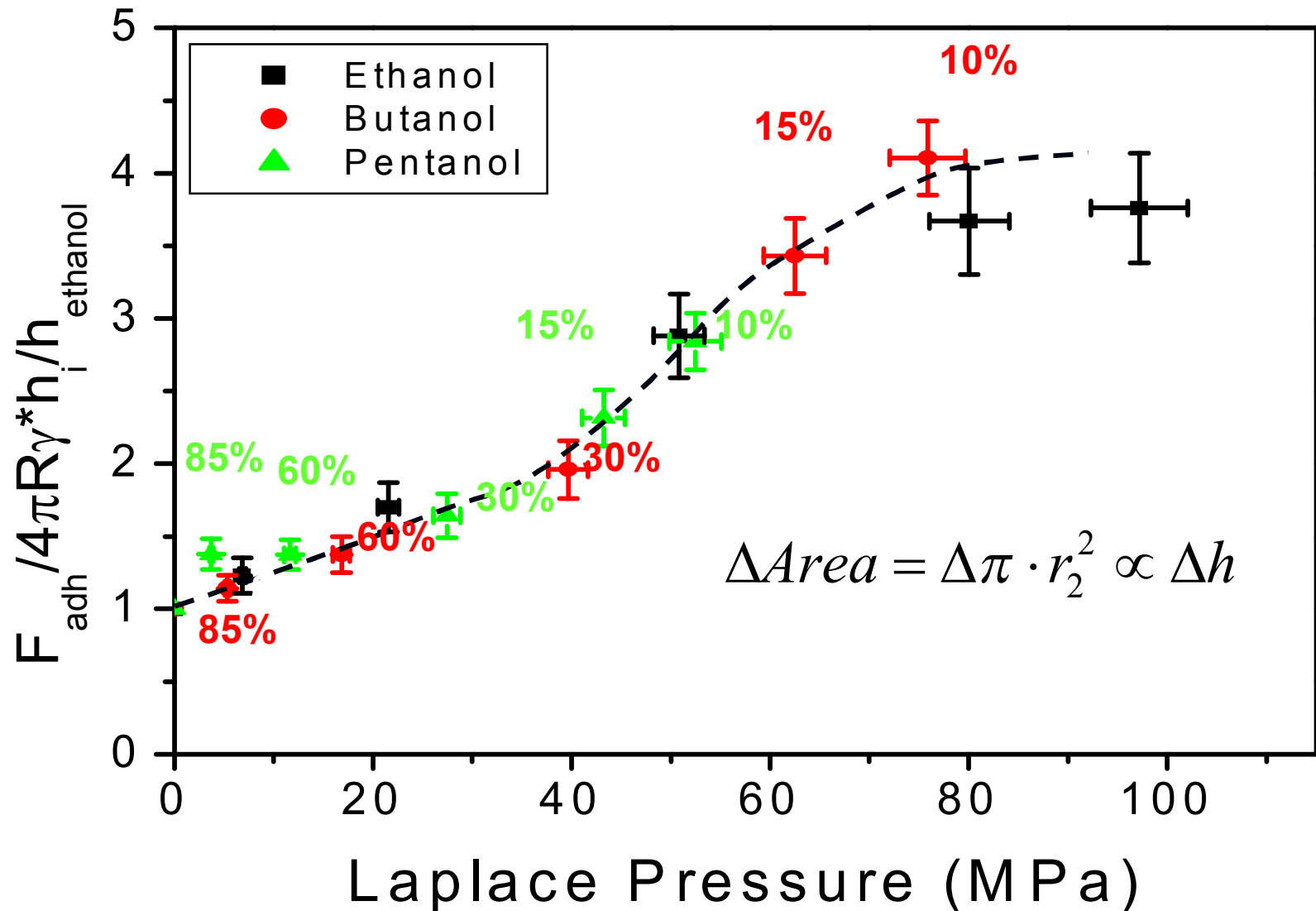
But, each alcohol has virtually the same surface tension ( $\gamma$ ) and adsorption isotherm (h)...

The difference is related to the molar volume of the condensed phase...

$$P_{Laplace} \equiv \frac{R_g T}{V} \ln\left(\frac{P}{P^{sat}}\right)$$

	V (cm <sup>3</sup> /mol)
Ethanol	58.7
1-butanol	91.2
1-pentanol	108.7

# Capillary Force vs. Laplace Pressure (Normalized for h and V)



# What's the big deal?

- Macroscopic view of capillary force due to Laplace pressure inside the meniscus:

$$F_{Capillary} = 4\pi R\gamma \cos \theta$$

- As the contact size shrinks, assumptions that are valid in the derivation for macroscopic contacts break down:

- MOLAR VOLUME

- ISOTHERM

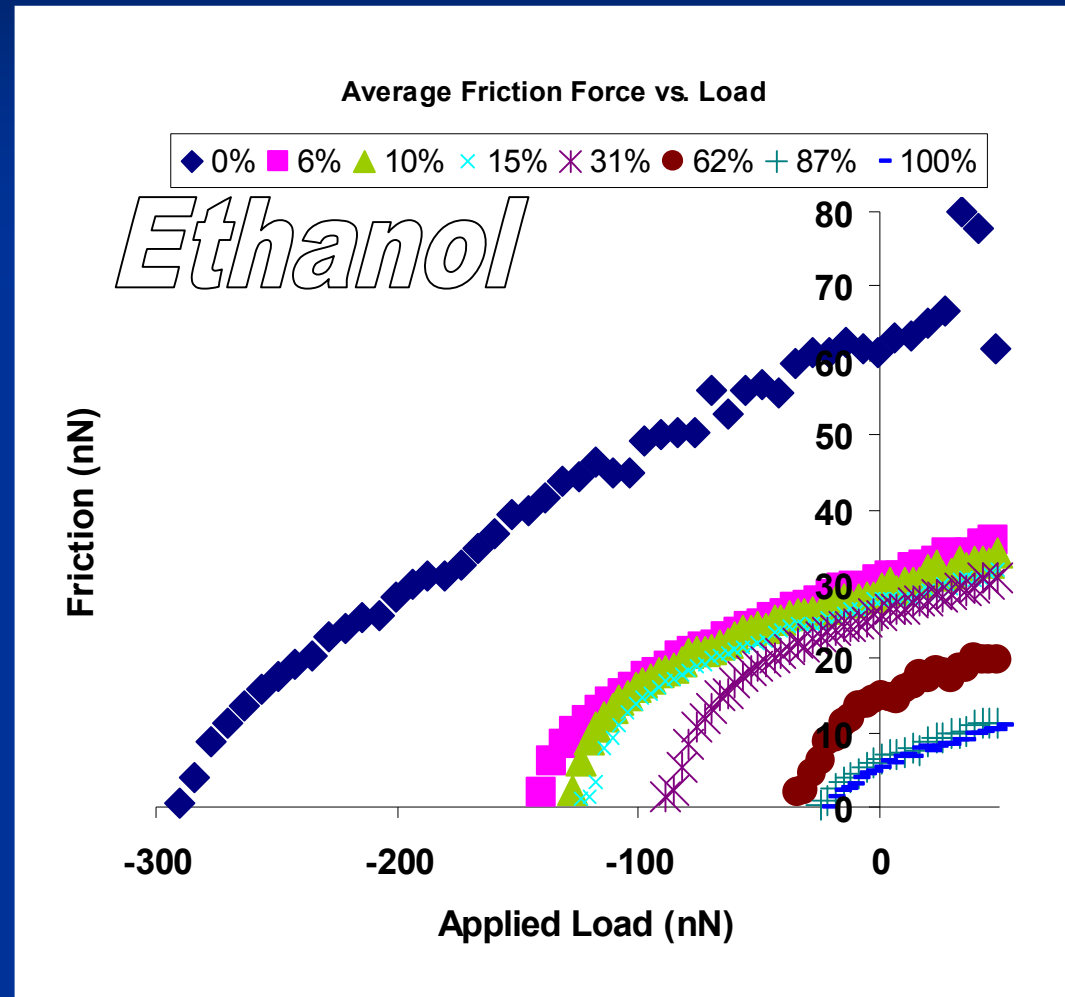
# Nano-asperity friction in alcohol vapor environment



# Friction Load Curves

## Reduction in Friction

- Reduction in Adhesion?
- Reduction in shear strength?
- Both?

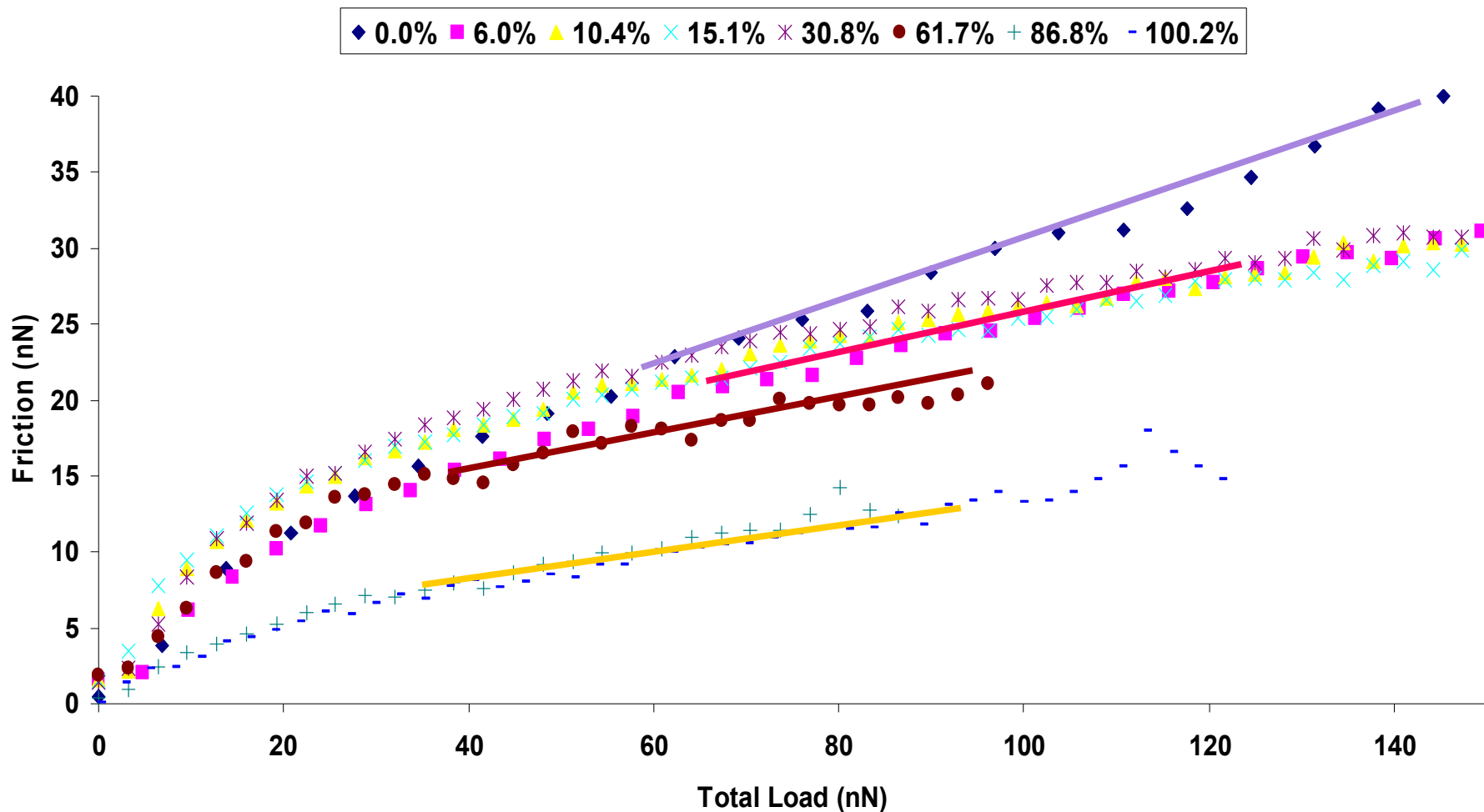


D. B. Asay and S. H. Kim  
(manuscript in preparation)

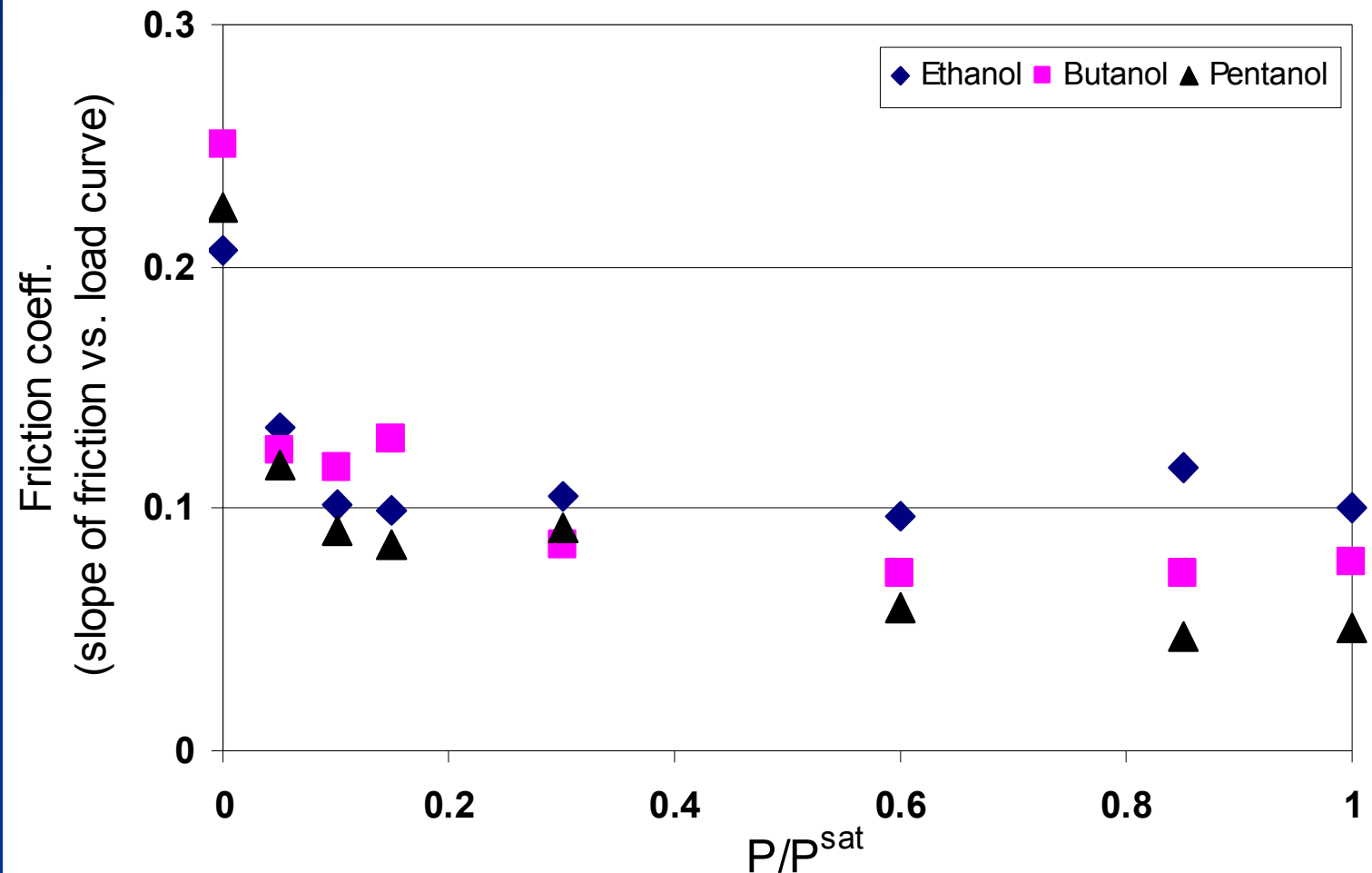
Silicon AFM tip on Silicon (100) surface at Room Temp  
speed 2  $\mu\text{m}/\text{sec}$

# Adsorbate Effect on Friction

Ethanol Friction vs Total Load



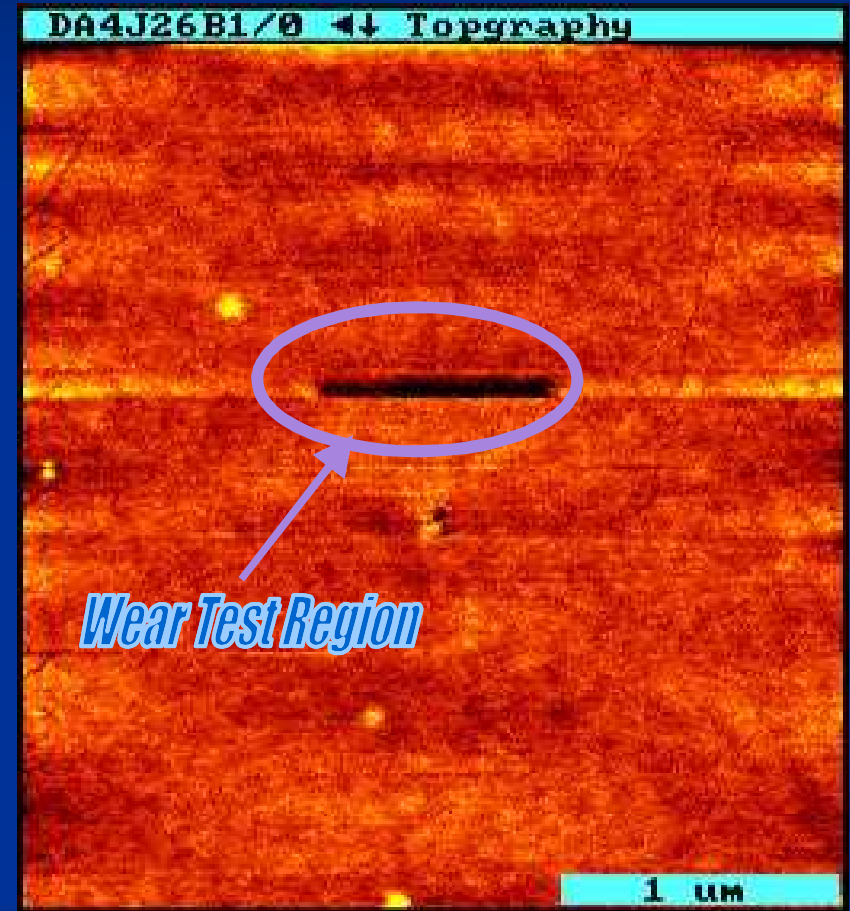
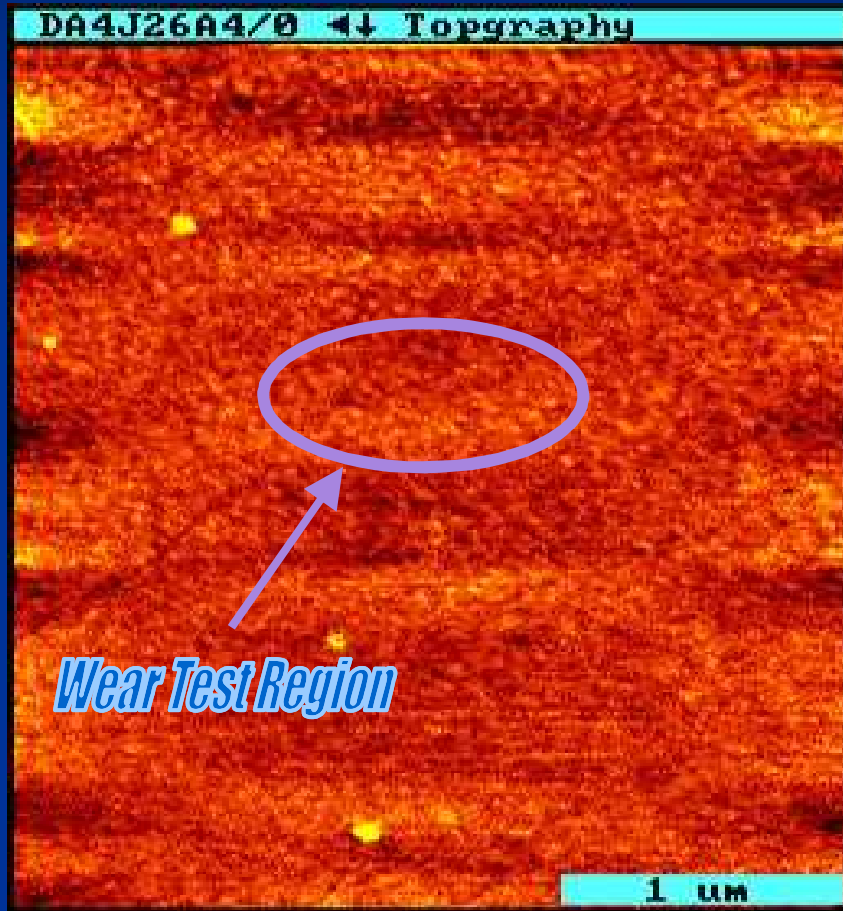
# Friction Coeff. vs. Vapor Pressure



# Nano-asperity wear behavior in alcohol vapor environment

# Single Asperity Wear

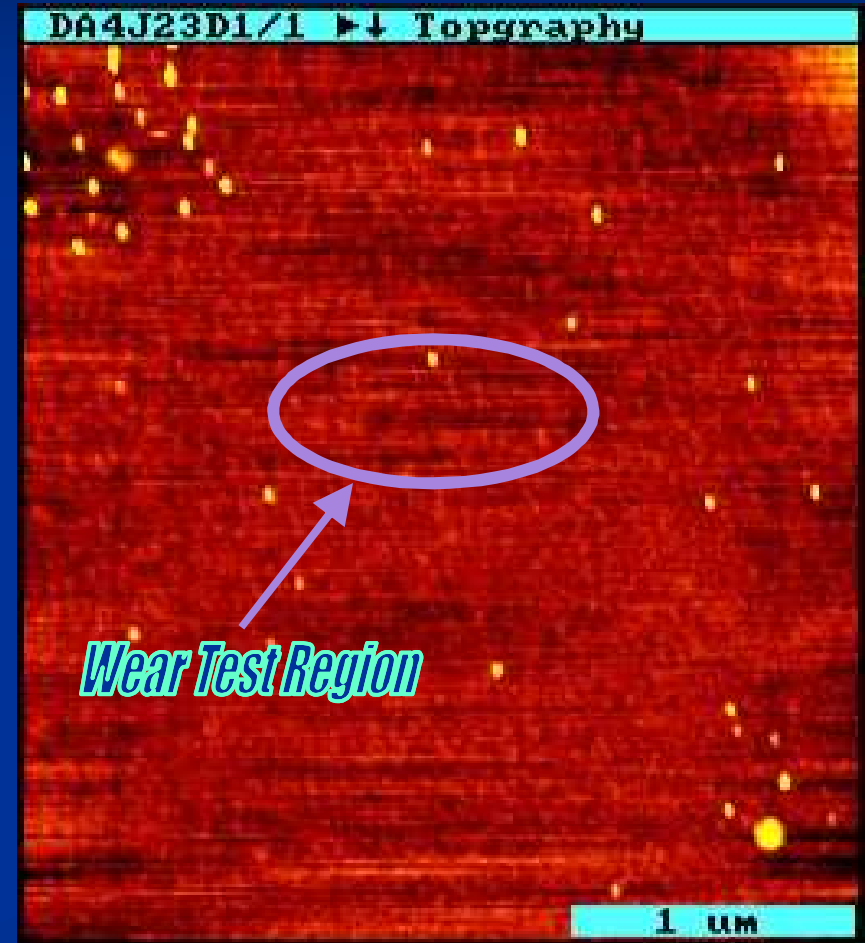
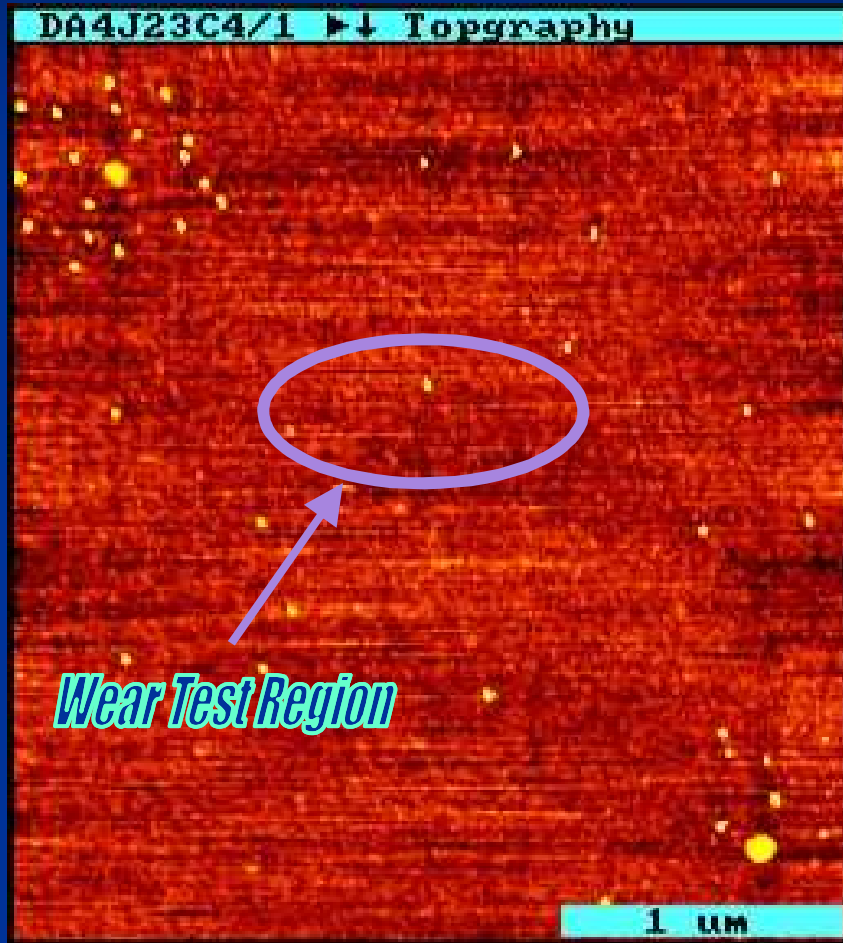
In humid air (RH = 75%)



10 Å deep trench

Full Z-scale ~1nm

# In 75% $P/P^{\text{sat}}$ n-Propanol vapor



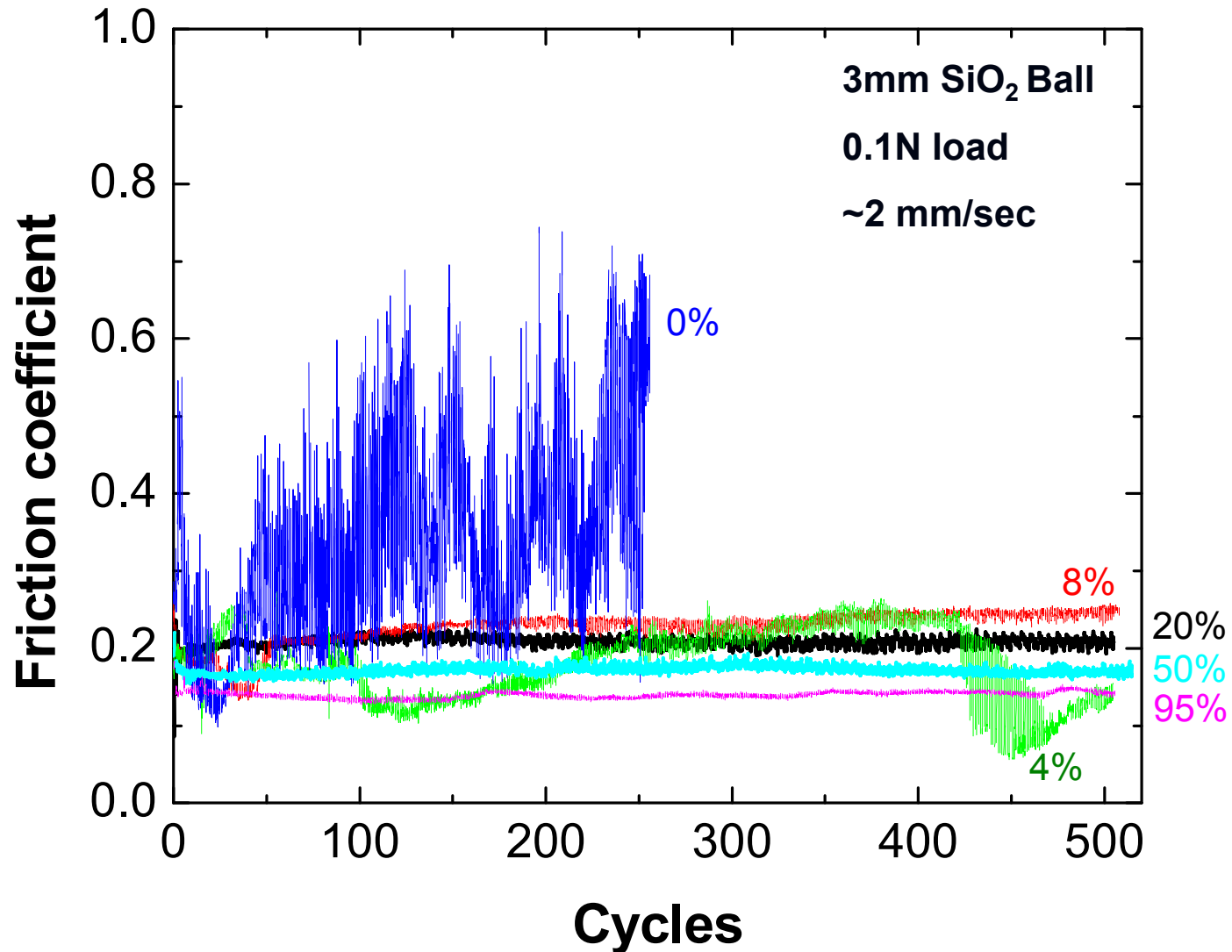
Full Z-scale ~1nm

Asay, Dugger, Ohlhausen, Kim, *Langmuir* (in press)

# Macroscopic Scale Observations

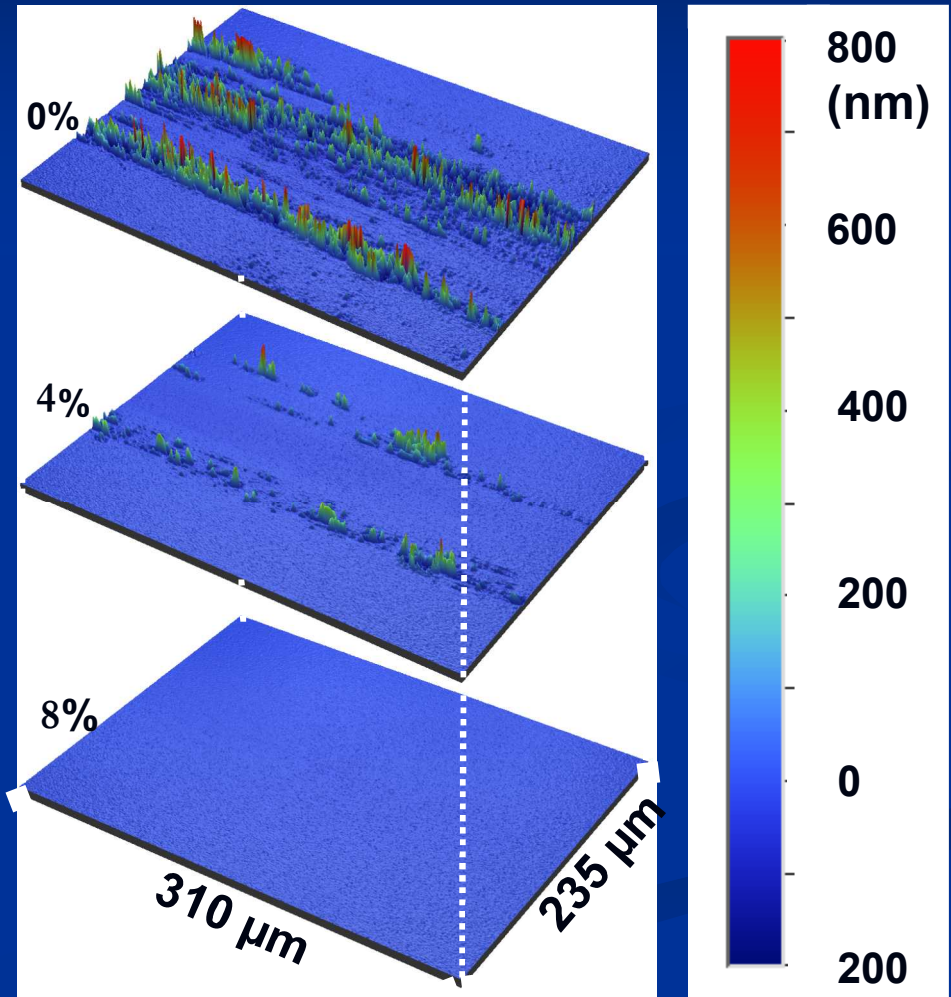
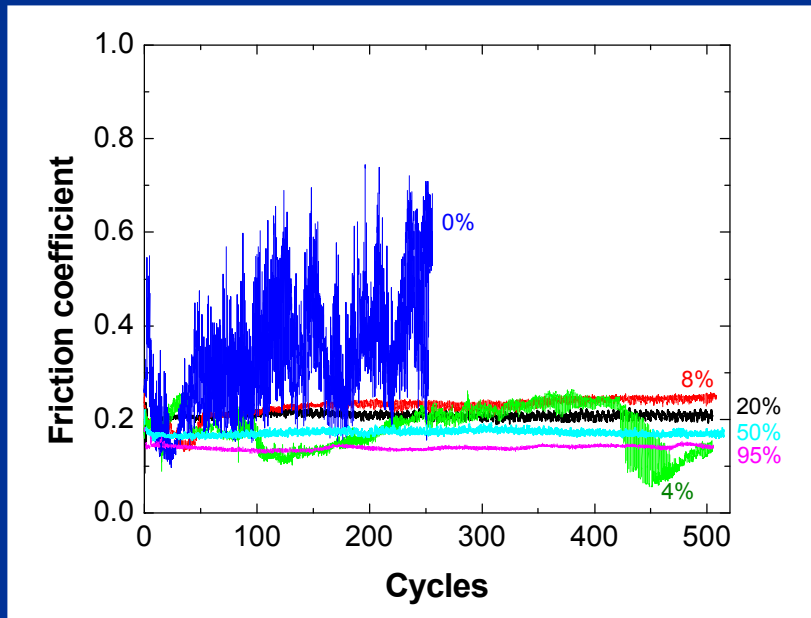
Linear wear tests in n-pentanol vapor  
environments

# n-Pentanol vapor environment



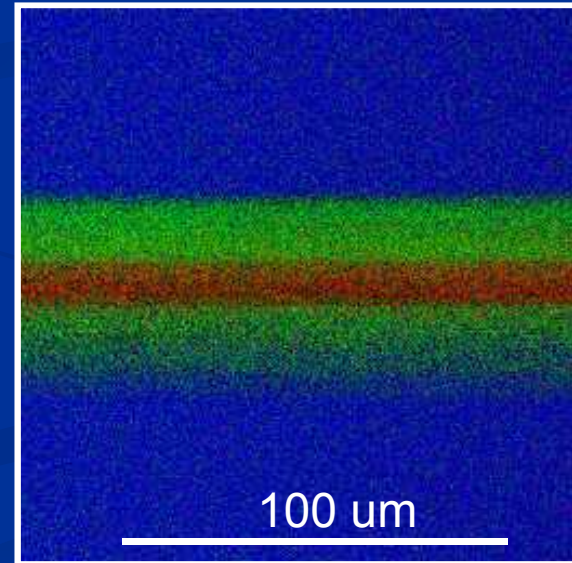
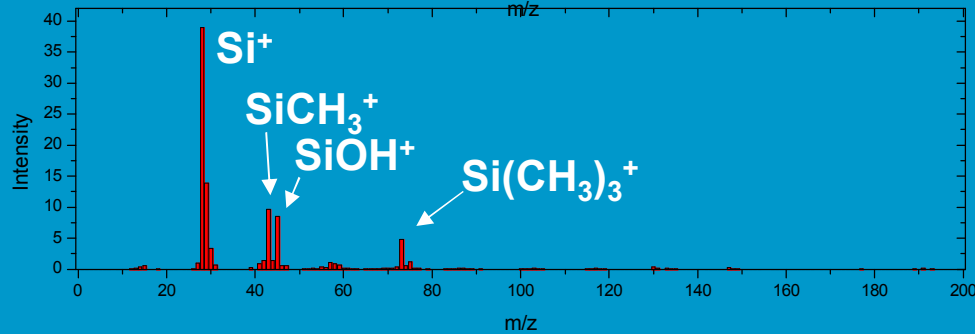
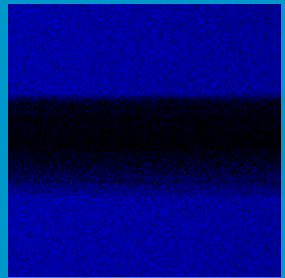
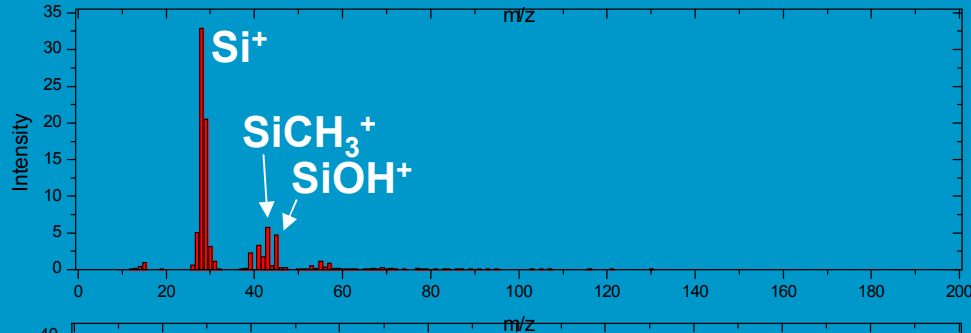
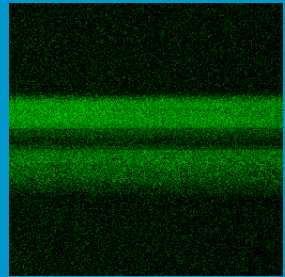
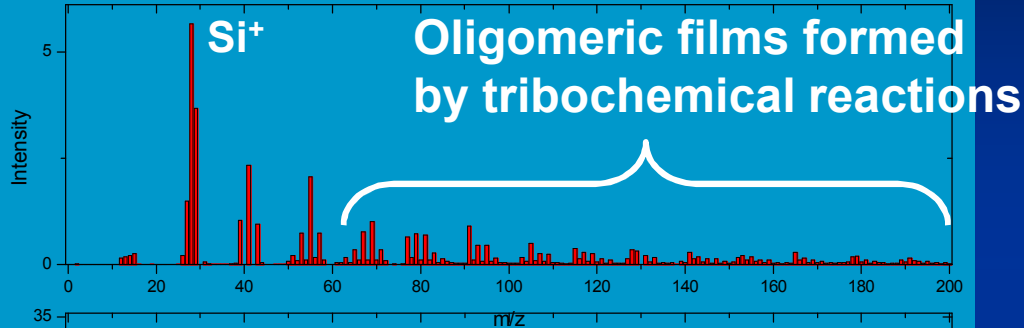
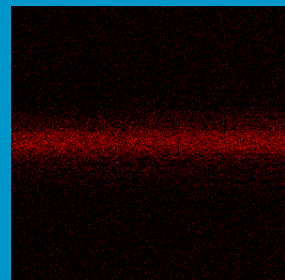


# Profilometry of wear track

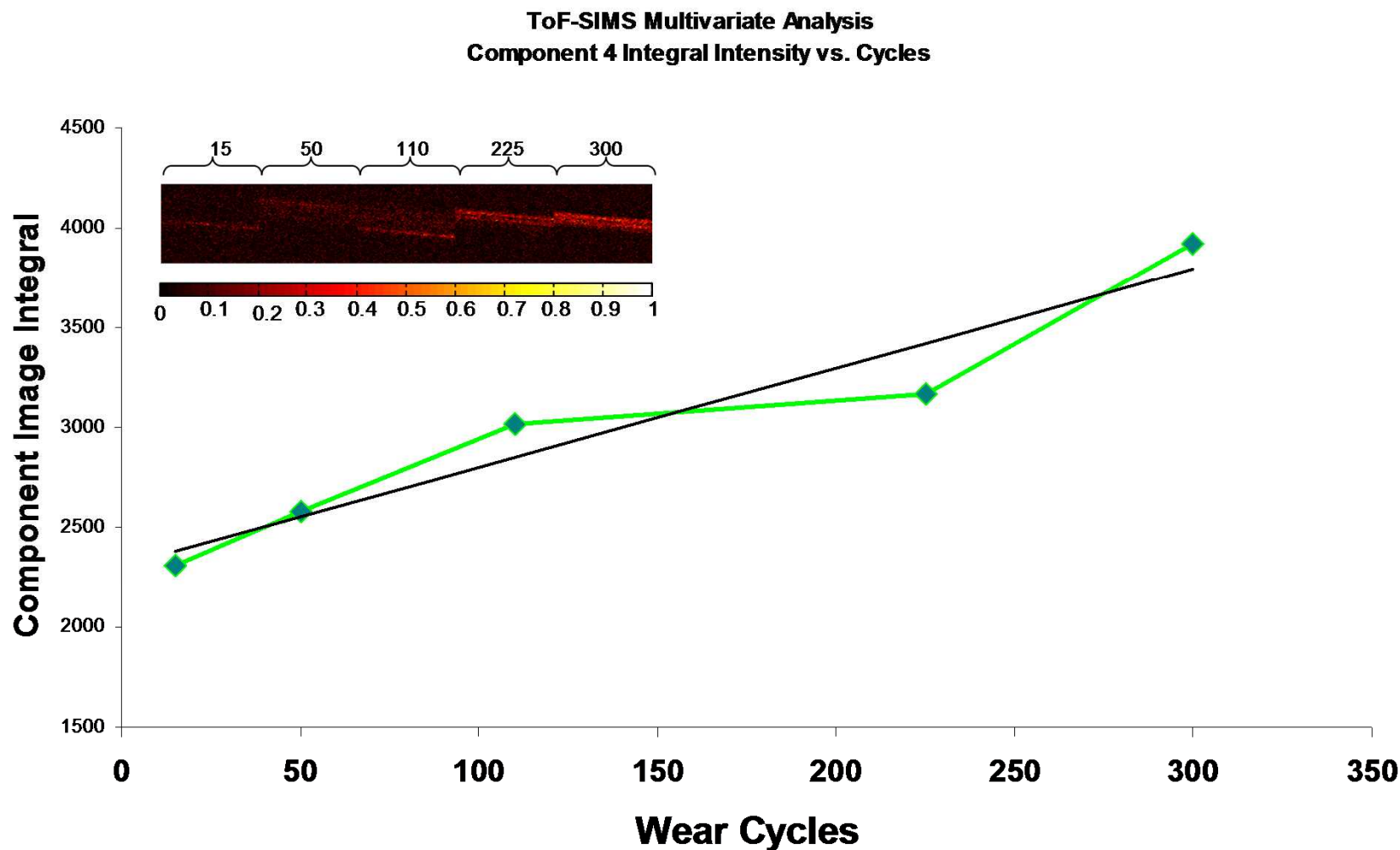


# 20% P/P<sup>sat</sup> n-Pentanol

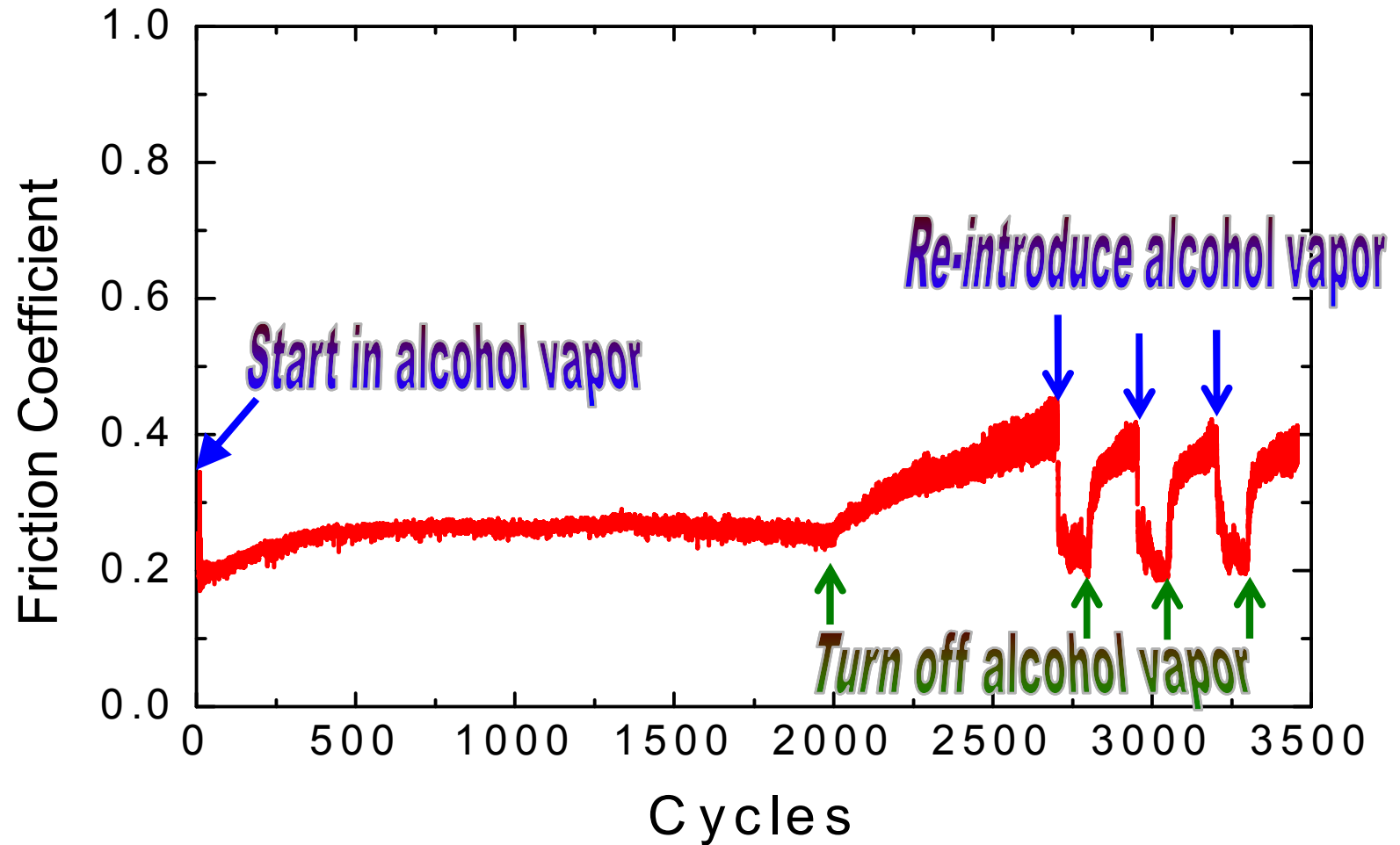
## ToF-SIMS Multivariate Image Analysis



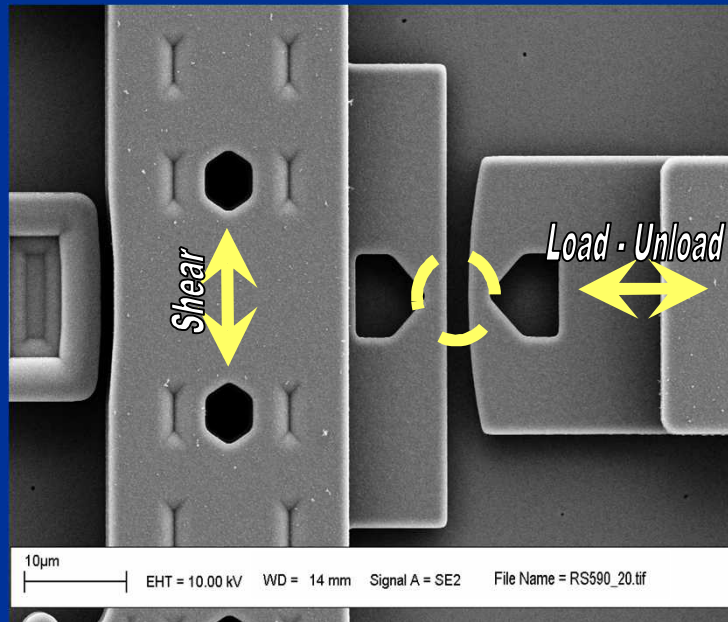
# Growth of oligomeric film



# Is maintaining the vapor environment important?



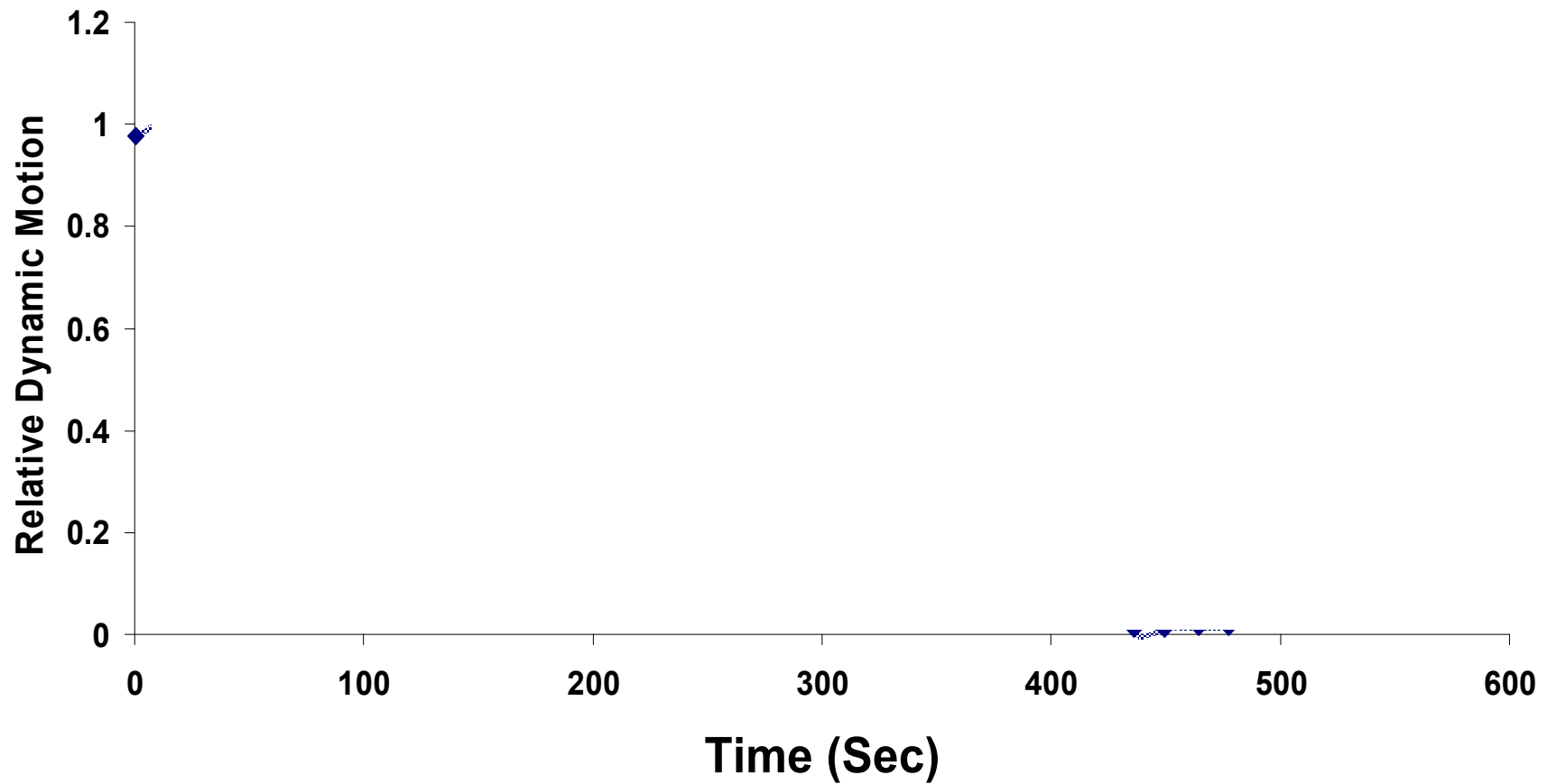
# Relevance to MEMS ?



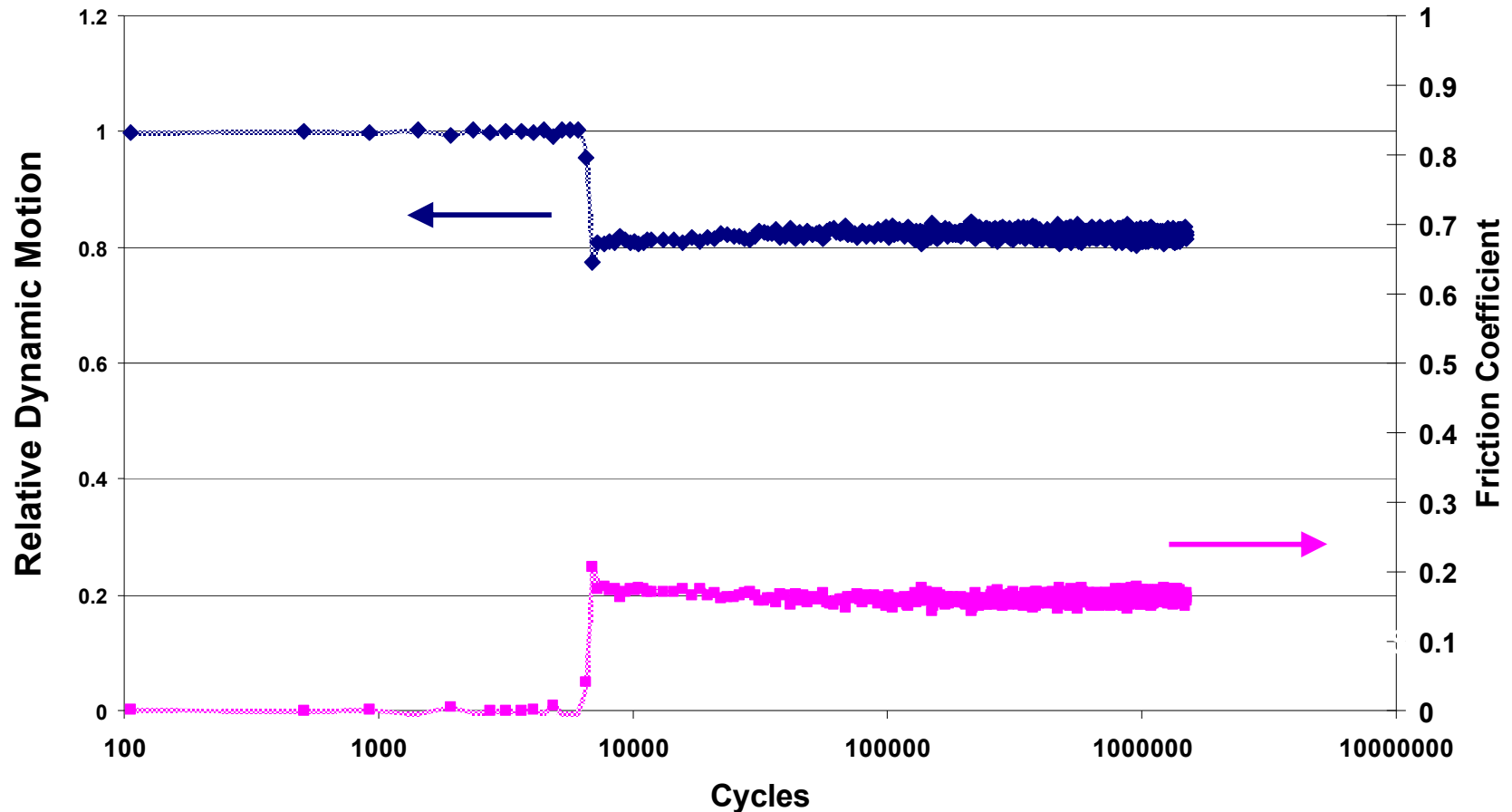
- Sidewall Friction Device
- 500 nN Load
- Oscillations at 100 Hz

# Dry N<sub>2</sub> Environment

Relative Dynamic Run



# Dynamic run in 95% $P/P^{\text{sat}}$ pentanol vapor

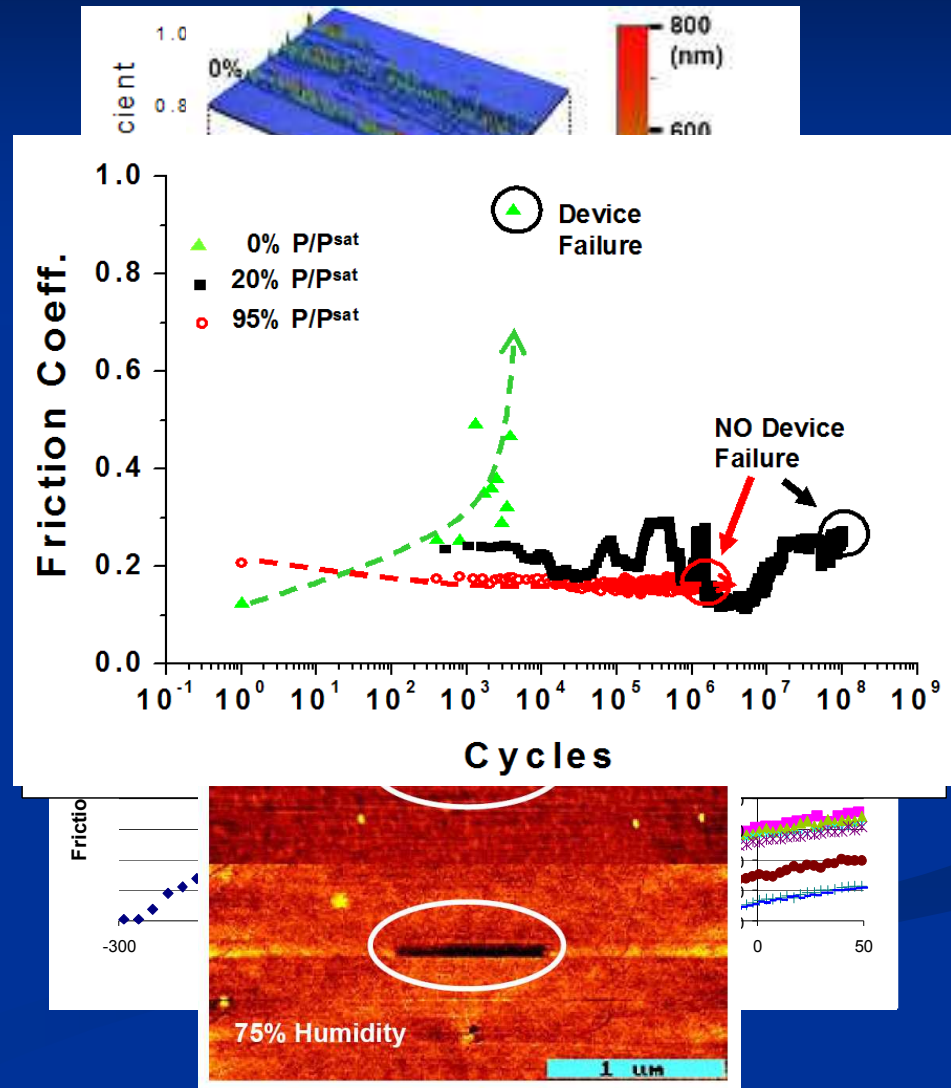


# Conclusions



# In-situ Vapor phase lubrication

- Alcohol adsorption provides a self-replenishing molecular thick lubricant layer
- Alcohol adsorption reduces Friction at all length scales
- Wear within the contact is dramatically reduced at all length scales
- MEMS failure is prevented



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Sandia National Laboratory

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