Nanomechanical measurement facility in Advanced Measurement Laboratory

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Nanometrology

Develop instrumentation, measurement methods to measure nanoscale properties

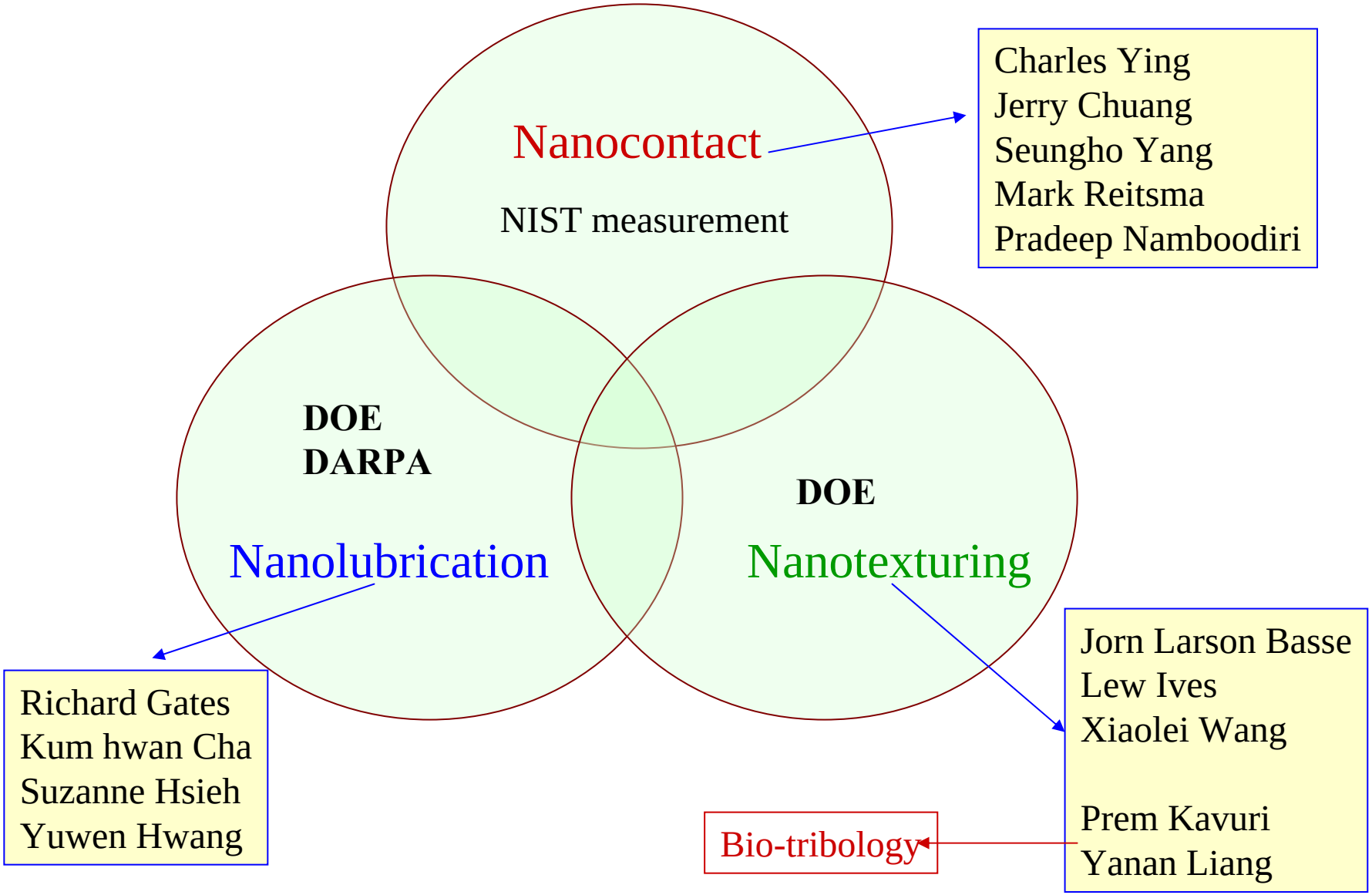
Provide methods, calibration artifacts, to teach others

- Accurate determination of size, shape, composition less than 100 nm diameter
- Accurate measurement of materials properties such as hardness, elastic modulus, surface energy
- Representative properties of a population
- Quantification of number of particles
- Determination of adhesion tendencies (stickiness, reactivity with water)

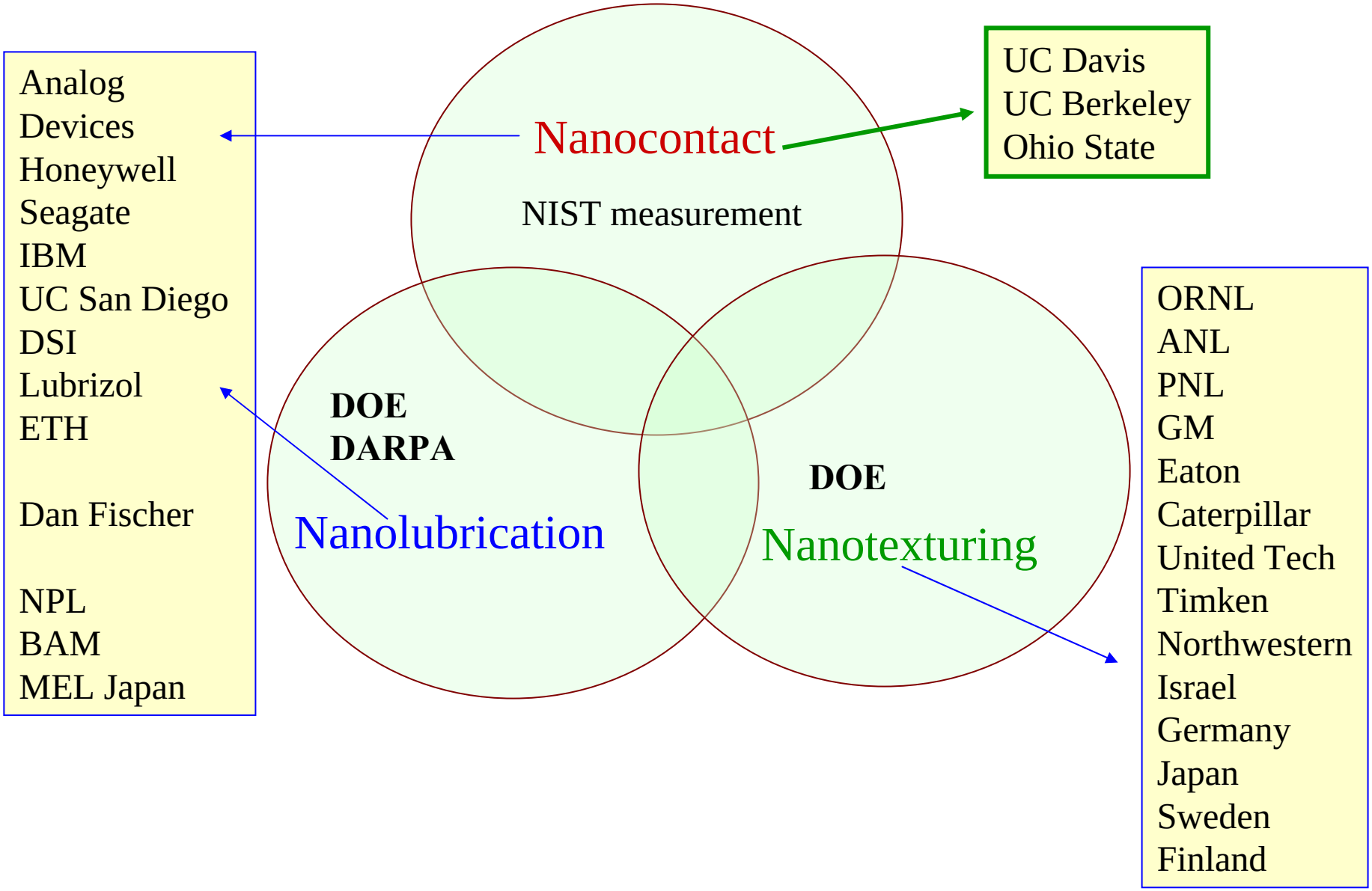
Measurement issues

- Dust particle contamination
- Vibration isolation for probe based measurement
- Temperature variations and acoustic interference
- Current measurement instrument inadequate in measuring nanoscale nanomechanical events
- Development of calibration methods & artifacts for standardization of measurement methods

Current technical activities



Collaborations and interactions



Nanoscale measurement

$$F = \mu L + \tau A + \varepsilon V + \sigma V + \nu B \pm \xi A \pm \psi d$$

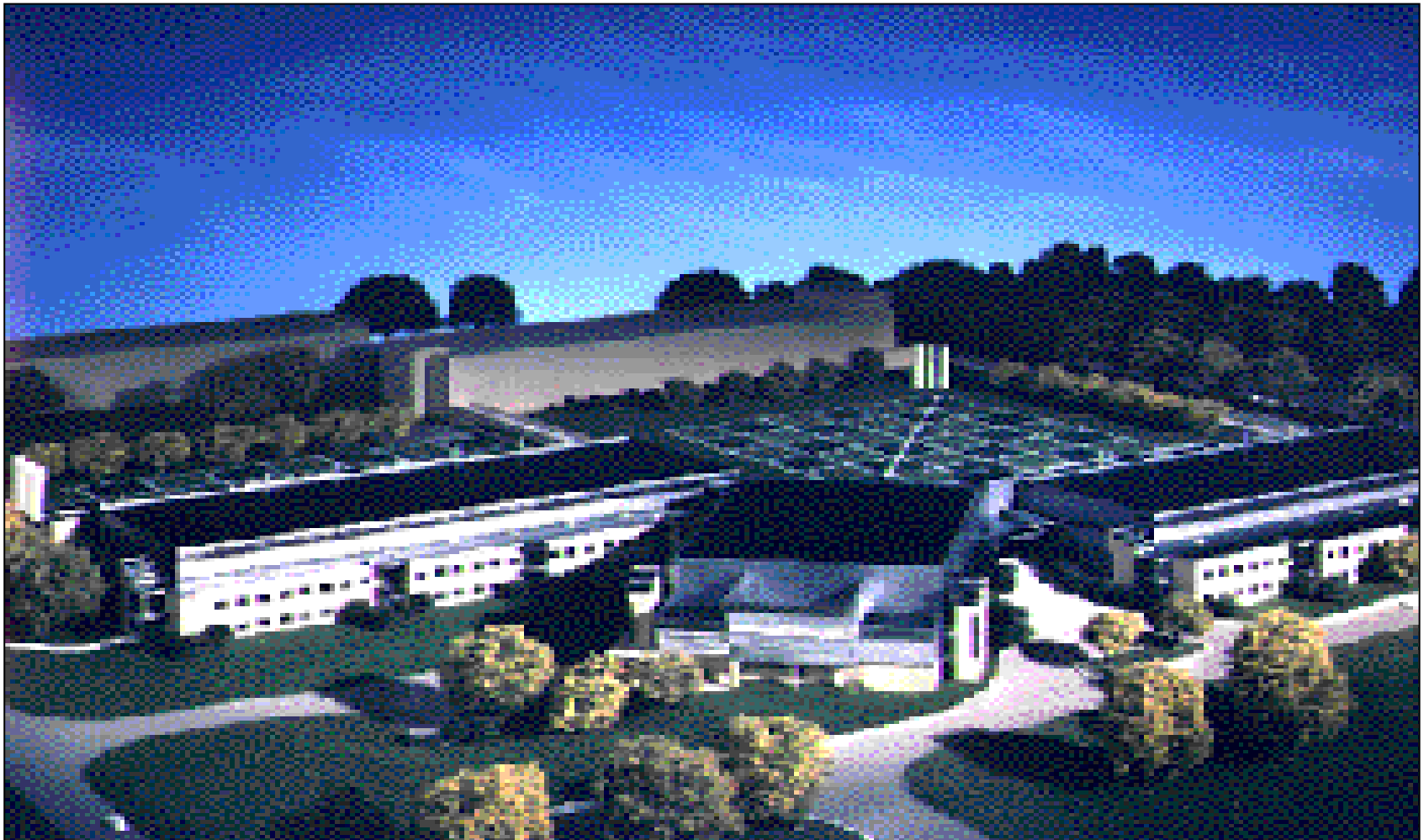
- μL= interfacial friction due to mechanical load
- τA=adhesion term
- εV=ploughing term
- σV=meniscus term
- νB=chemical bonding force
- ξA=electrostatic force
- ψd=molecular attractive or repulsive force depending on distance between surfaces

NIST Investment in Facilities

Advanced Measurement Laboratory

World's premier measurement research facility (air quality, temperature, vibration, humidity)

Completed Dec. 2004



An integrated facility

Vibration isolation, class 1000, controlled temperature

DEPOSITION

Dip coating, PVD, spin coating, LB trough

CHARACTERIZATION

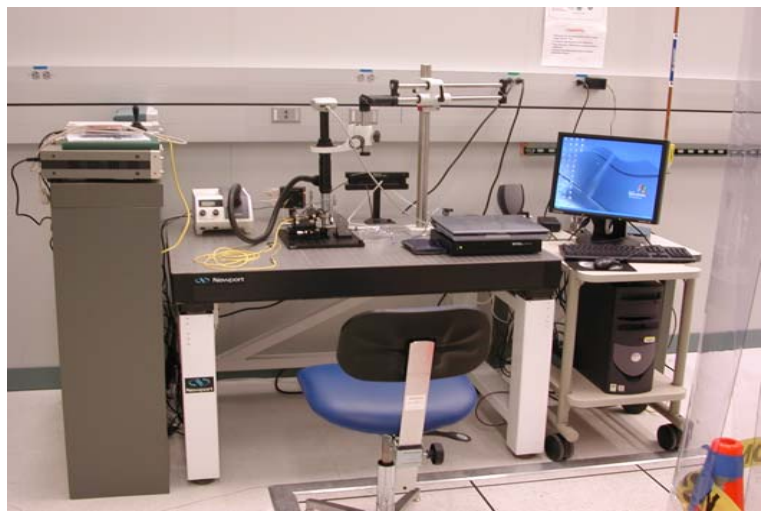
FTIR, AFM, XANES

Ultra-high vacuum STM/AFM

Veeco dual white light interferometer

PERFORMANCE

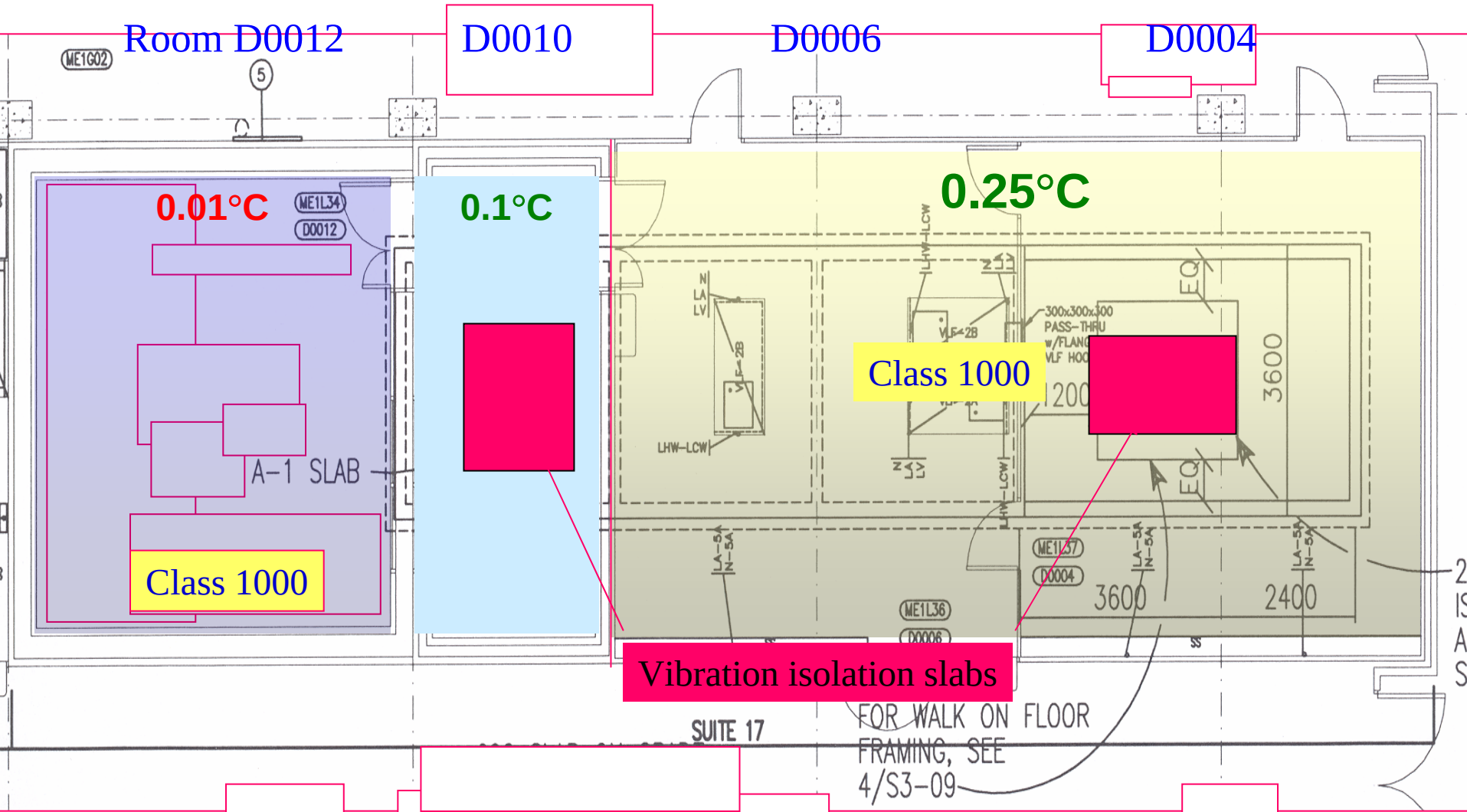
- Hysitron multiscale friction tester
- AFM Triboscope
- NIST nanoadhesion apparatus
- Film strength apparatus
- Film durability apparatus

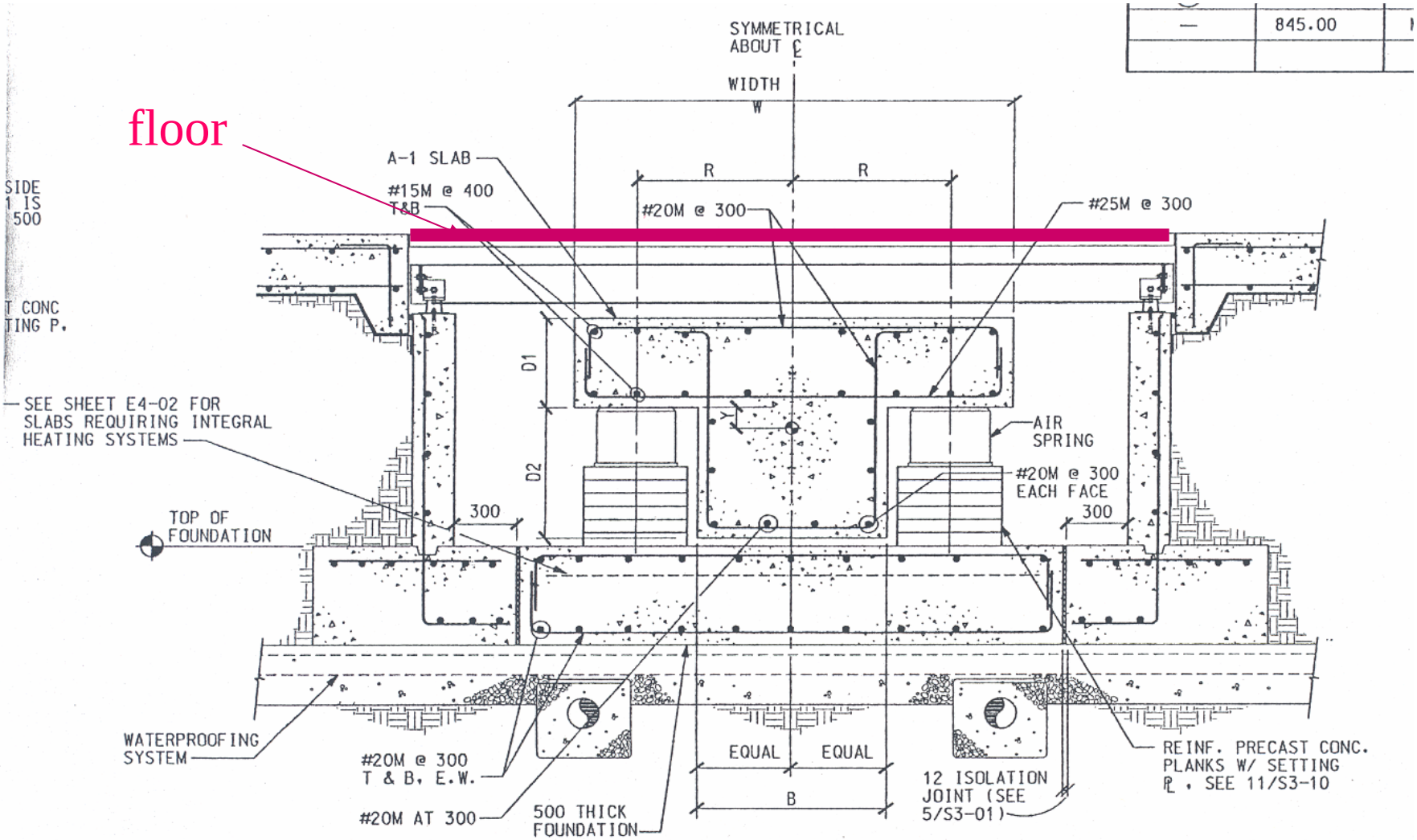


Nanotribology 6-module nanoscale measurement laboratory:

Vibration isolation, class 1000, temperature control

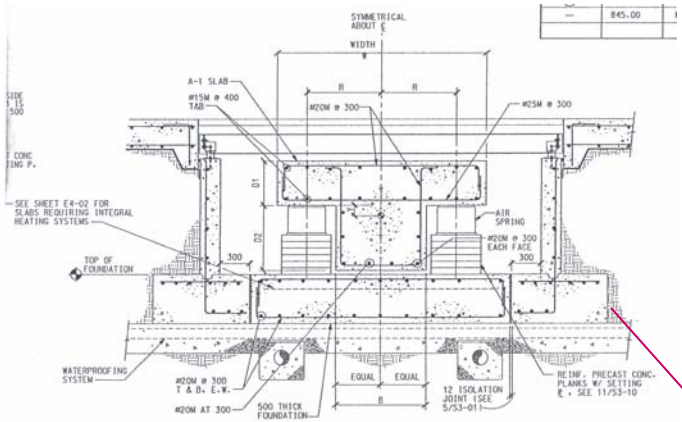
Building 218, Metrology East



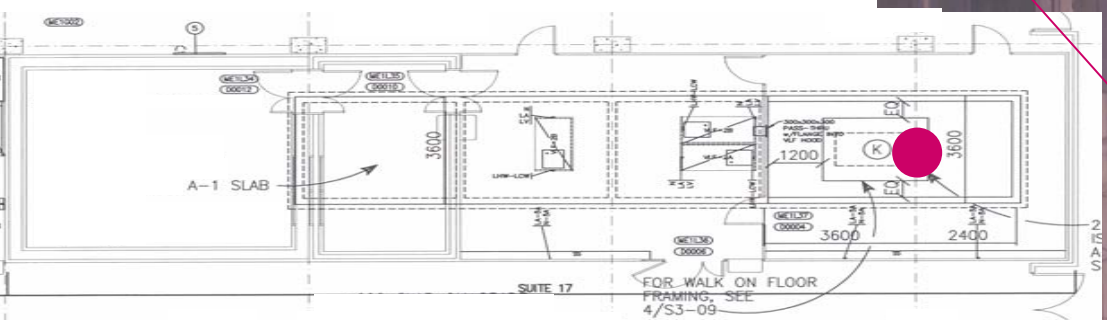


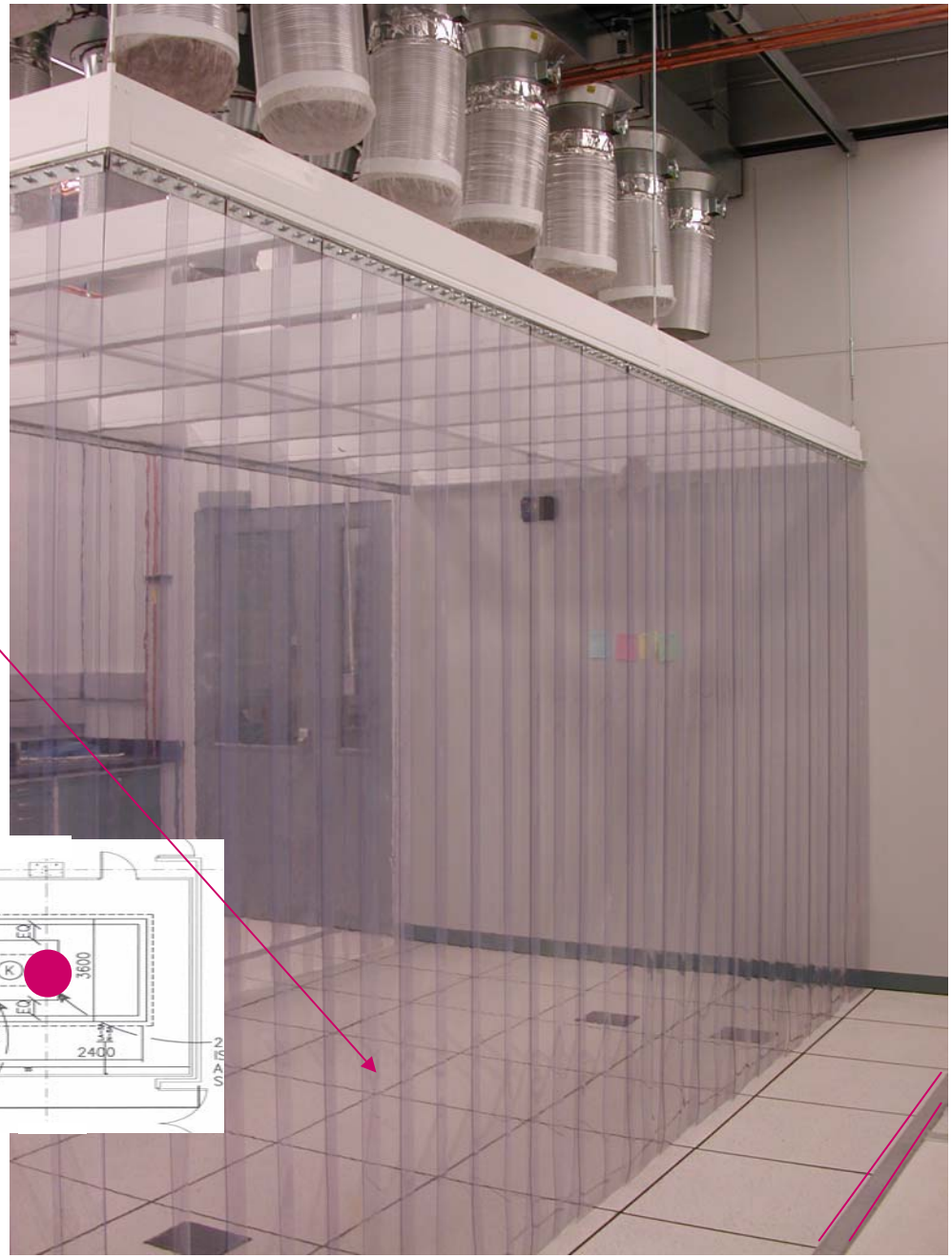
TYPICAL A-1 SLAB (W/ ONE-WAY STEM)

8 ton concrete block with air spring support



TYPICAL A-1 SLAB (W/ ONE-WAY STEM)





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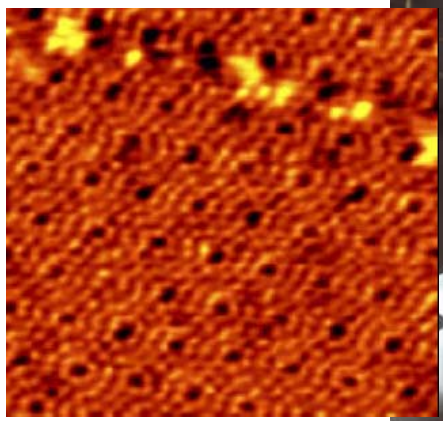
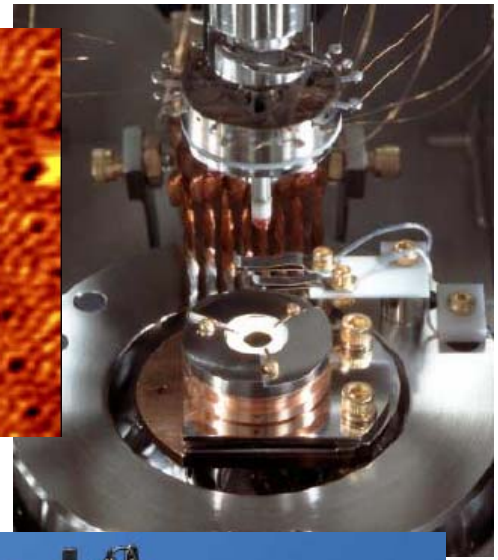
UHV 3000

UHV Variable Temperature STM & AFM

A combined scanning tunneling microscope for imaging and an atomic force microscope for manipulation & force measurement





Capabilities

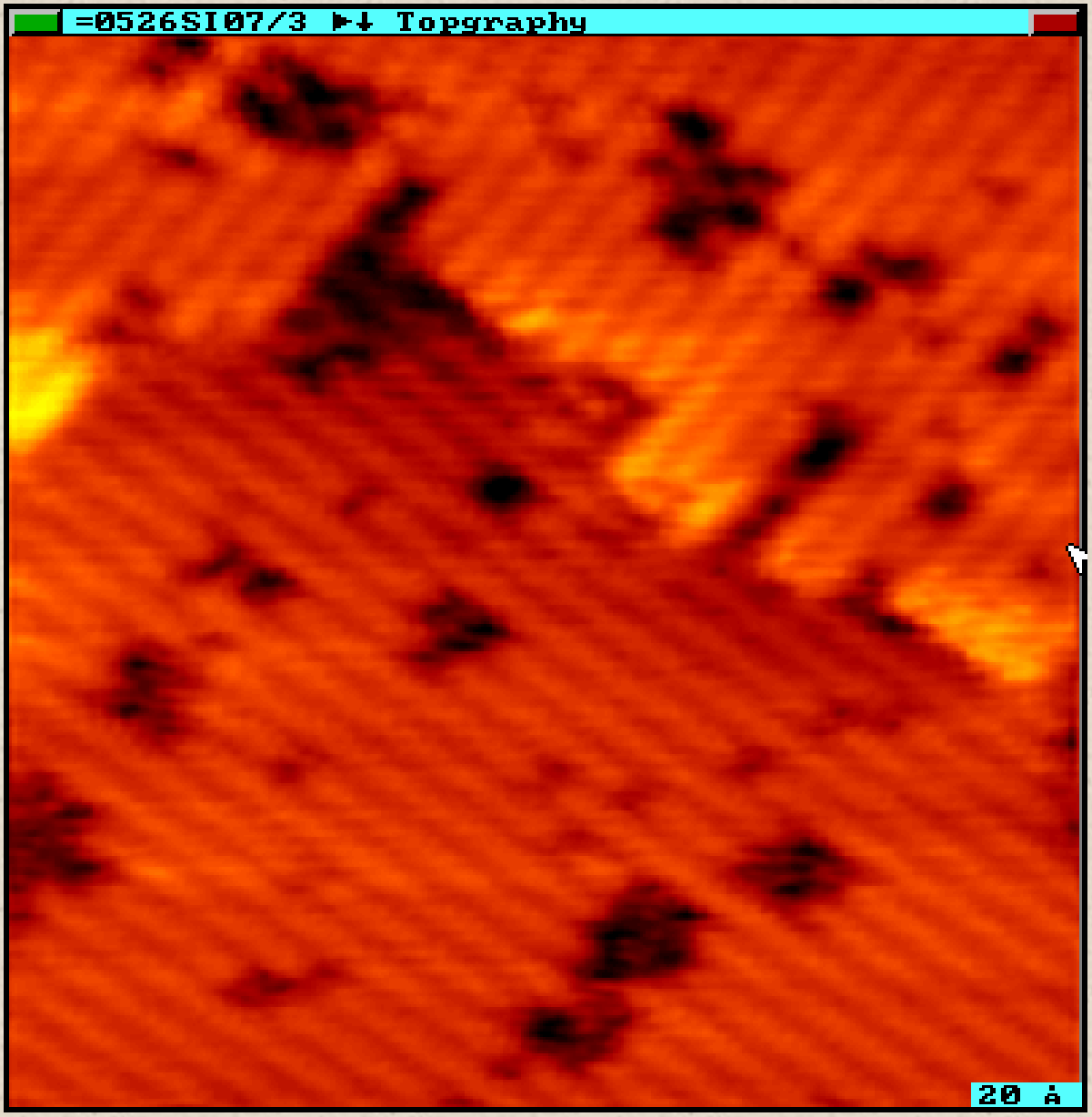
- Sample cooling and heating during STM or AFM imaging
  - 30 K to 600 K
- *z* resolution: 0.01 nm
- *z* scan range: 1200 nm
- *x-y* resolution: 0.05 nm
- *x-y* scan range: 8000 nm (8 μm)

UHV STM image of Si(100)

Si(100) surface at room temperature

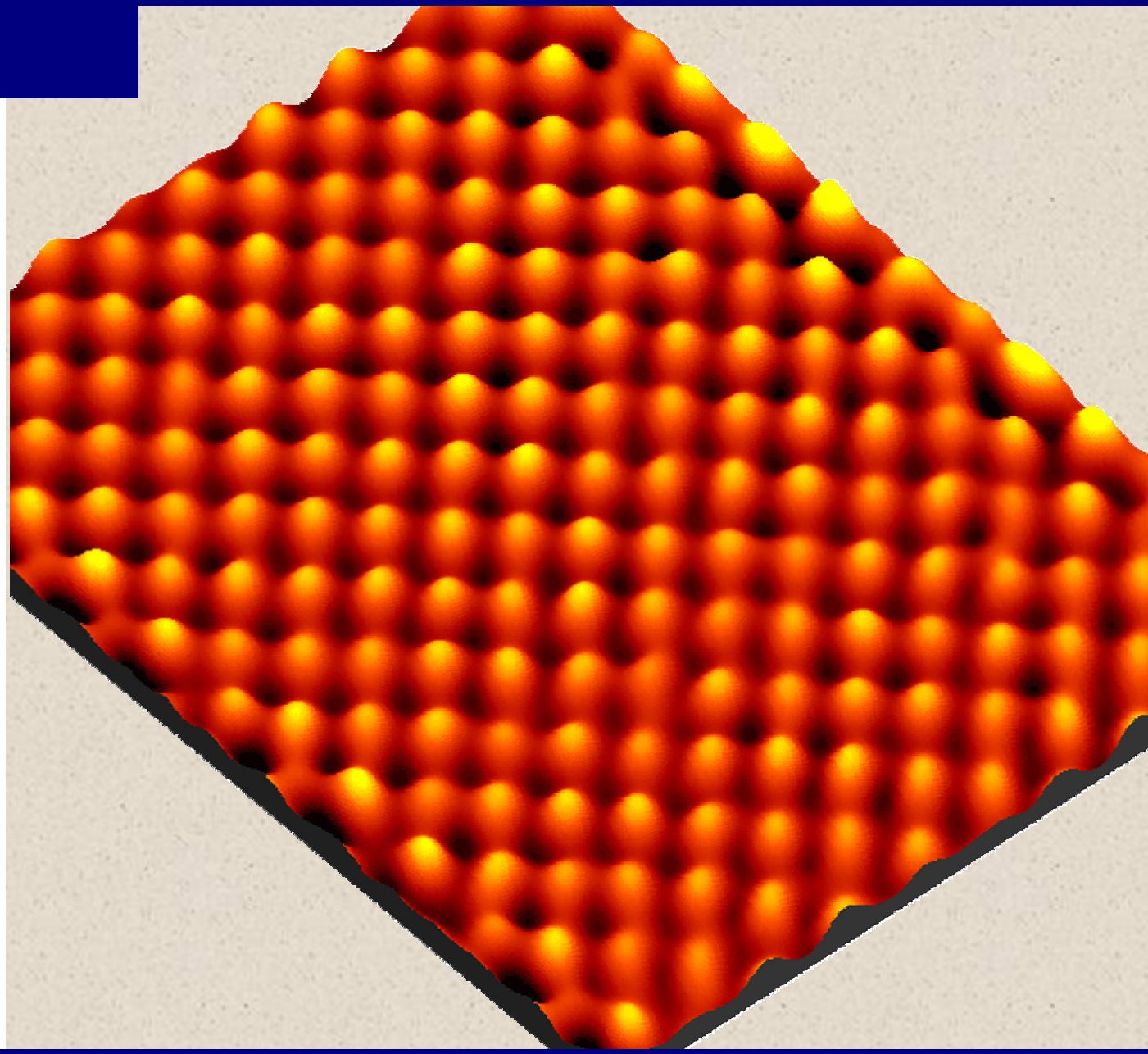
UHV350 AFM/STM operating in STM mode. Image shows the detailed structure at a step edge of the Si(100) surface with defects and/or contamination.

Measured corrugation is 50 pm orthogonal to the observed rows



Contribution by Z. Wang, RHK Technology

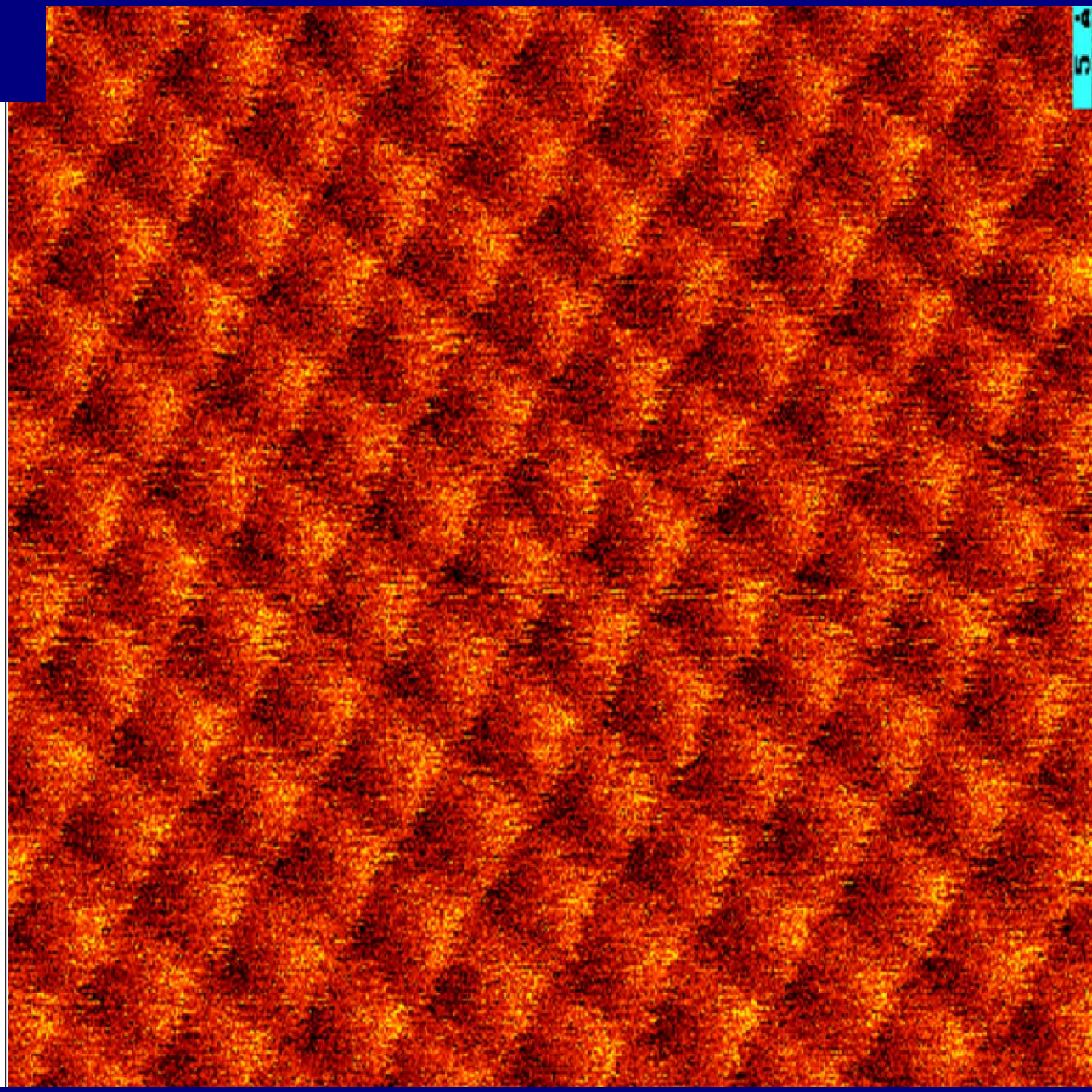
UHV AFM Image of NaCl (100)



UHV350 AFM image of a NaCl crystal, resolving the crystalline atomic structure of the 100 surface. 56 Å x 48 Å scan.

Contribution by Z. Wang, RHK Technology

AFM Friction Image on NaCl Crystal in UHV

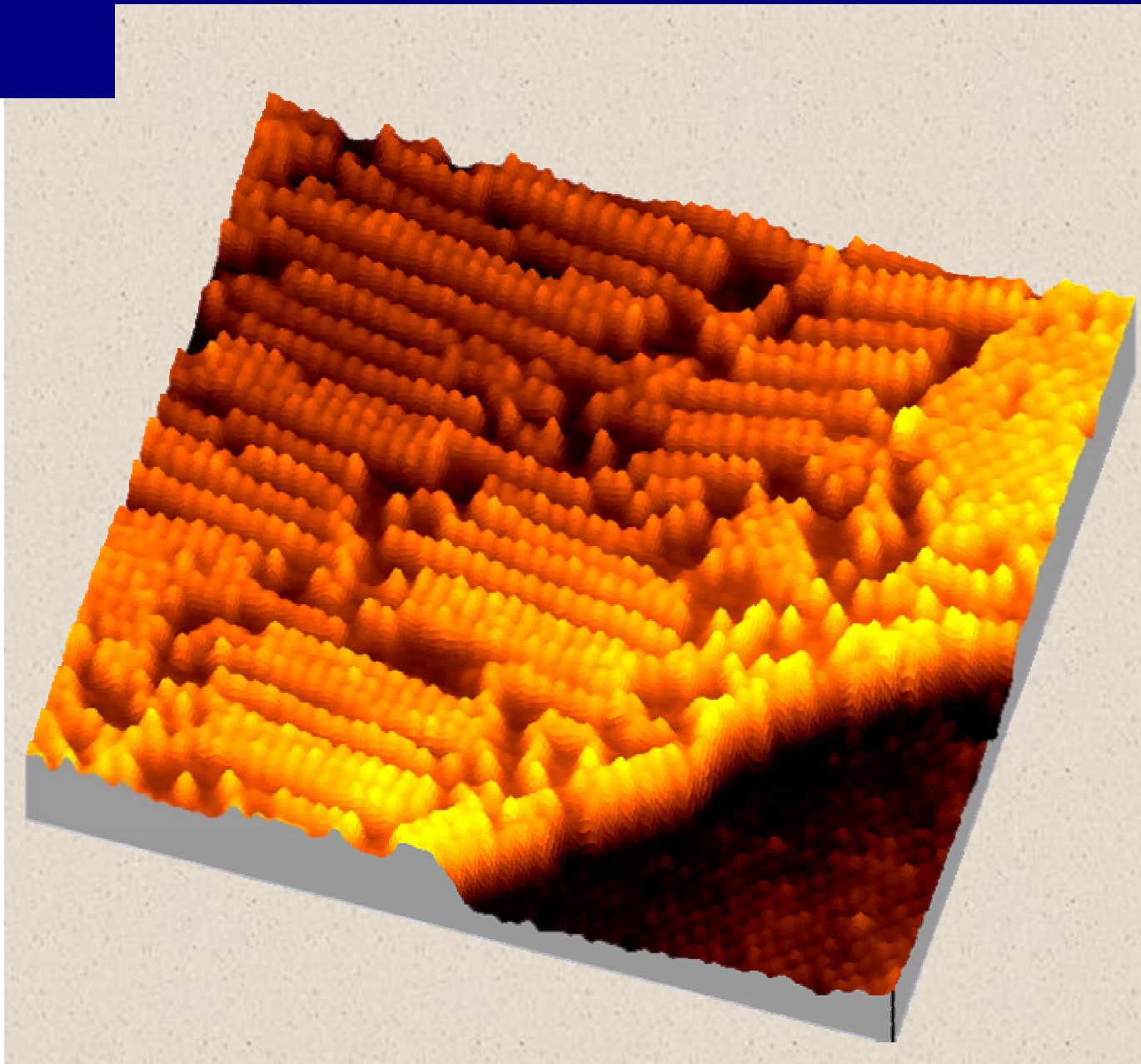


UHV 350 AFM friction image: NaCl crystal

Atomic lattice resolution on a NaCl(100) surface. Unprocessed raw data is displayed. Image was taken under UHV conditions using a silicon nitride cantilever with a force constant of 0.12 N/m, and 50 ms/line scanning speed.

Courtesy of R. Carpick and R. Cannara, Univ of Wisconsin-Madison and Z. Wang, RHK Technology

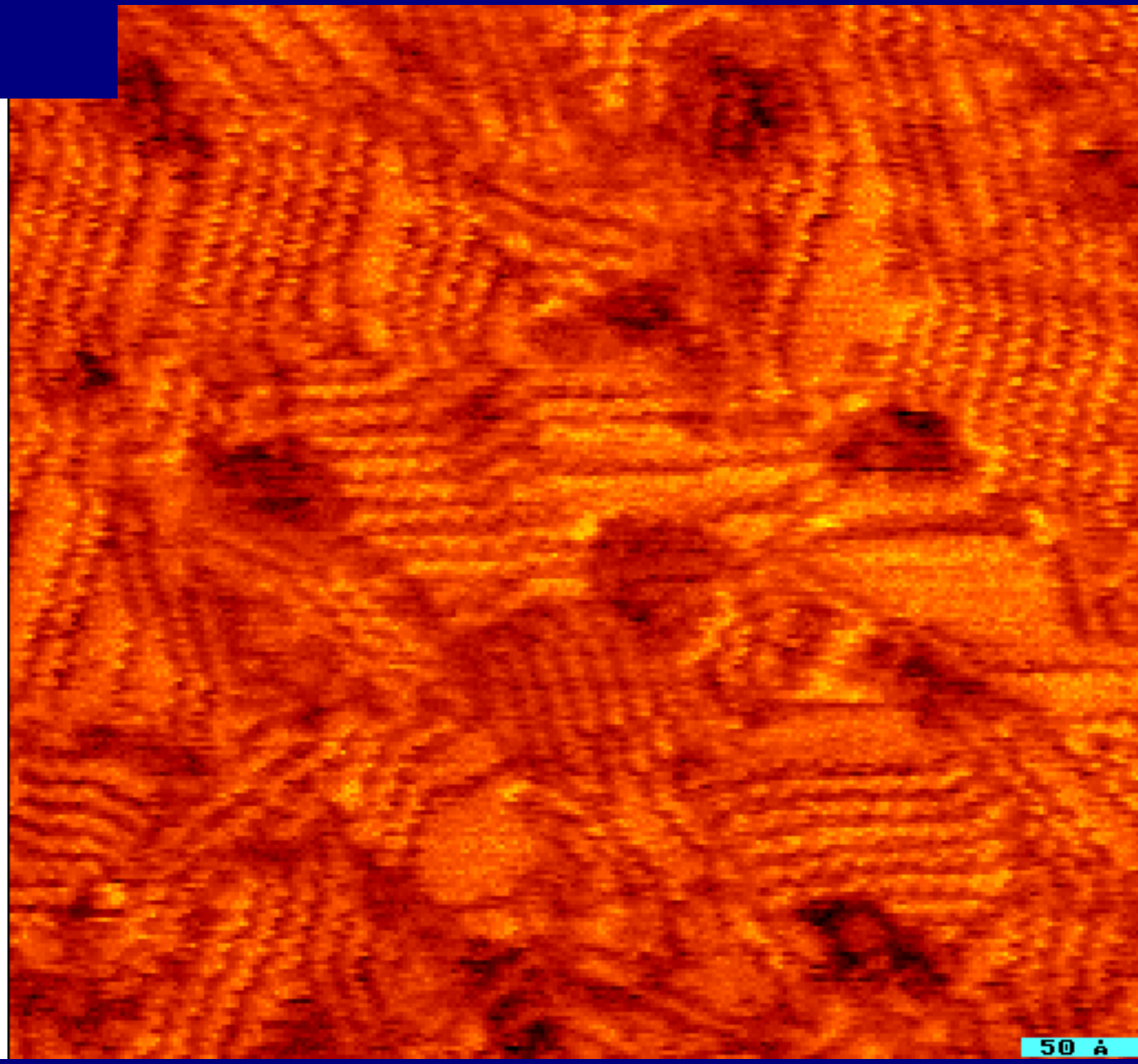
176 K - Low Temperature



Co(II) tetraphenyl porphyrin and Co(II) phthalocyanine coadsorbed on Au(111), imaged at 176K.

Courtesy of D. Barlow and K.W. Hipps, Washington State U.

Low Current: 1pA UHV STM of SAMs



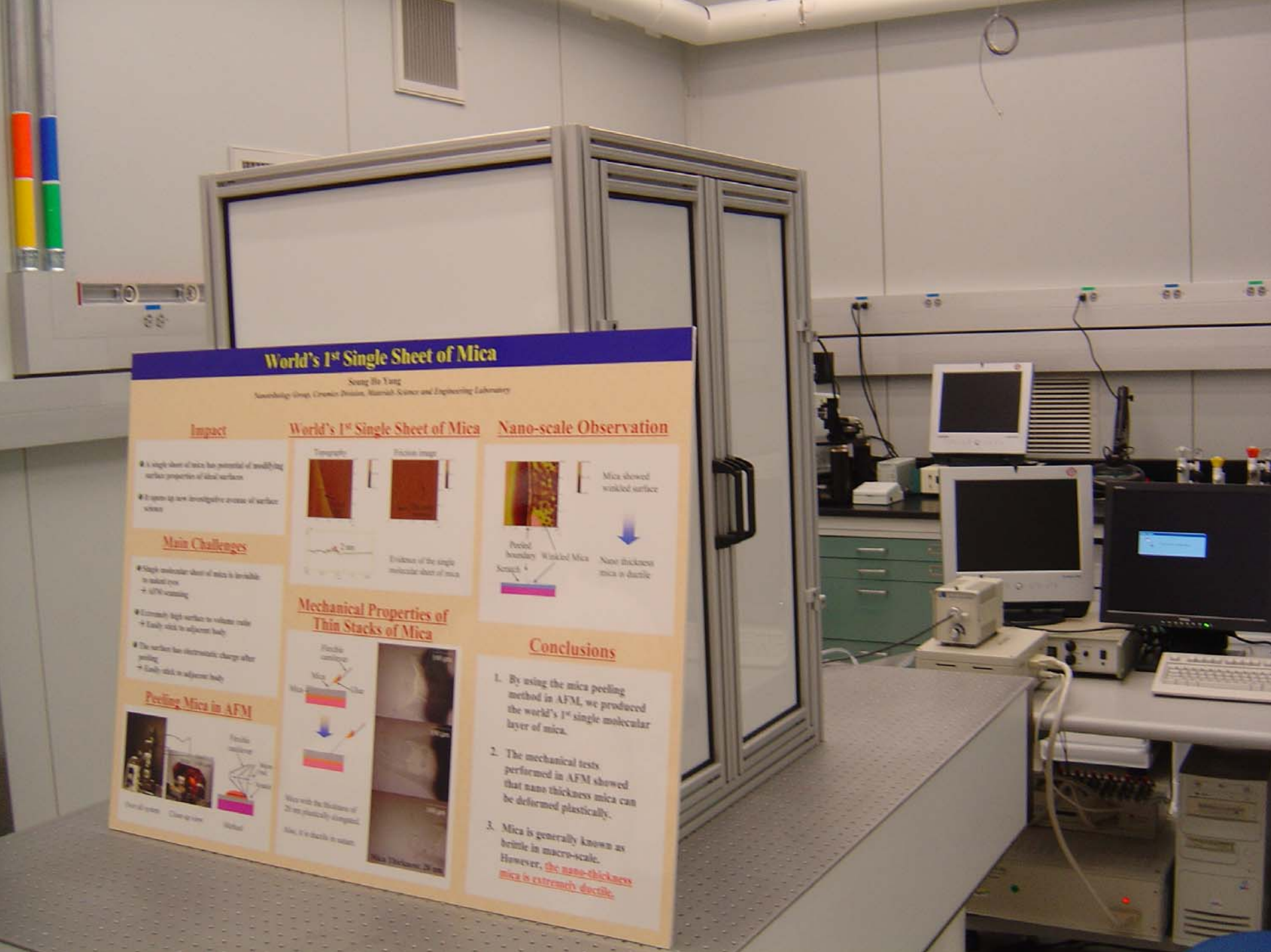
UHV STM image of an octanethiol Self Assembled Monolayer on Gold (111) acquired at 1 pA.

Courtesy Y. Qian & G.Y. Liu, UC Davis

Deposition facility
LB trough
Dip coating
Vapor deposition
Spin coating
Class 1000

Entrance into temperature controlled room

Cool light


World's 1st Single Sheet of Mica
Impact
Main Challenges
Peeling Mica in AFM
World's 1st Single Sheet of Mica
Mechanical Properties of Thin Stacks of Mica
Nano-scale Observation
Mica showed wrinkled surface
Nano thickness mica is ductile
Conclusions
1. By using the mica peeling method in AFM, we produced the world's 1st single molecular layer of mica.
2. The mechanical tests performed in AFM showed that nano thickness mica can be deformed plastically.
3. Mica is generally known as brittle in macro-scale. However, the nano-thickness mica is extremely ductile.

Atomic Force Microscope

Capabilities

- Operation in liquid & gaseous environments
- Interchangeable scanners
  - *J* head: 140×140 μm^2 (largest range)
  - *E* head: 16×16 μm^2
  - *A* head: 1×1 μm^2 (highest resolution)
- Resolution: 0.03 nm (*z*), 0.2 nm (*x-y*)
- Contact mode, tapping mode, phase imaging

Hysitron TriboScope

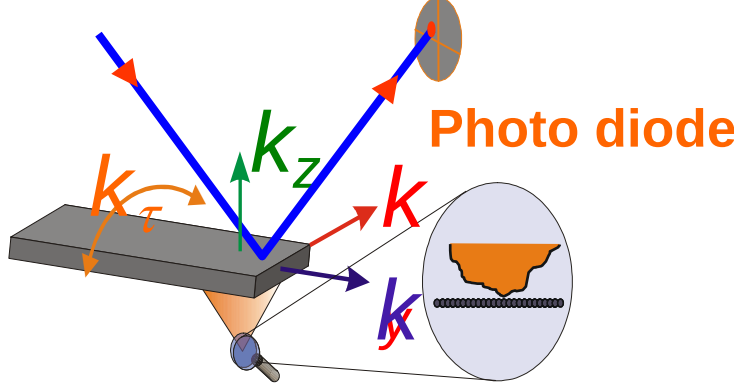
- Diamond tips
- Nanomechanical properties

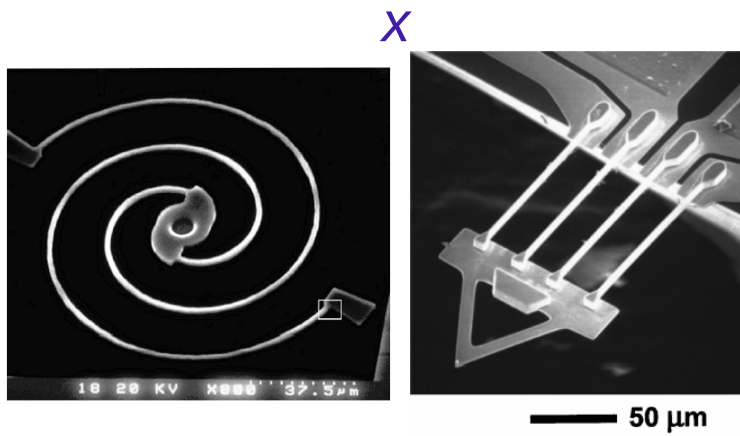
Cantilever designs & Calibrations

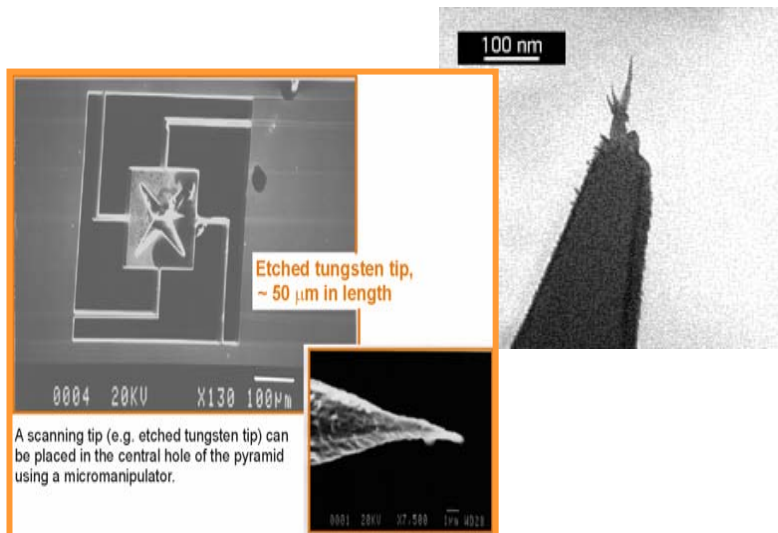
- Spring constant calibration artifacts
- Stiff springs for friction measurements
- Equal x.y spring constants

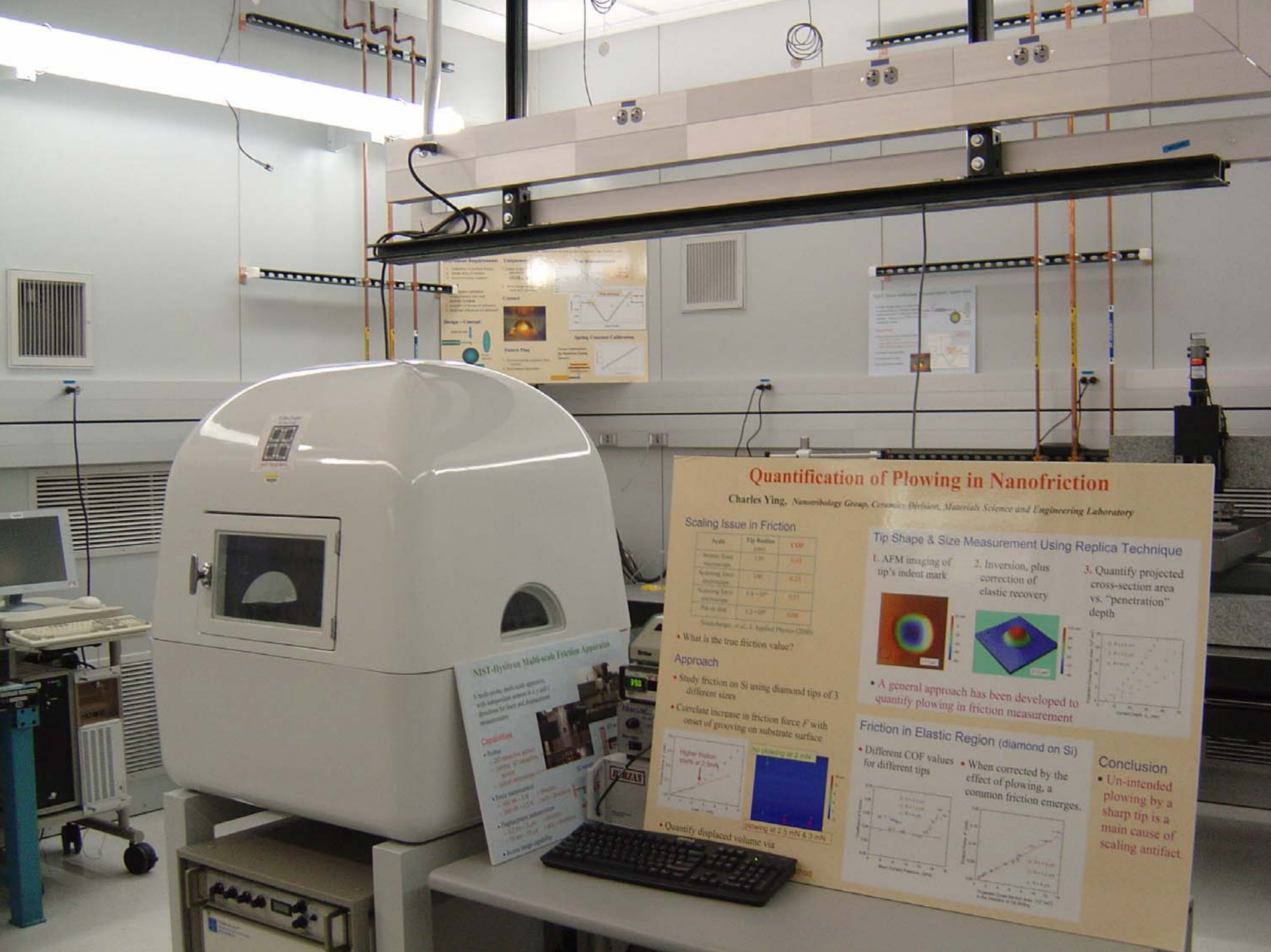
Tip fabrication & characterization

- Ultra-sharp tips
- Tip shape modification
- Sphere attachment & surface roughness control
- International round robin on spring constant cal.





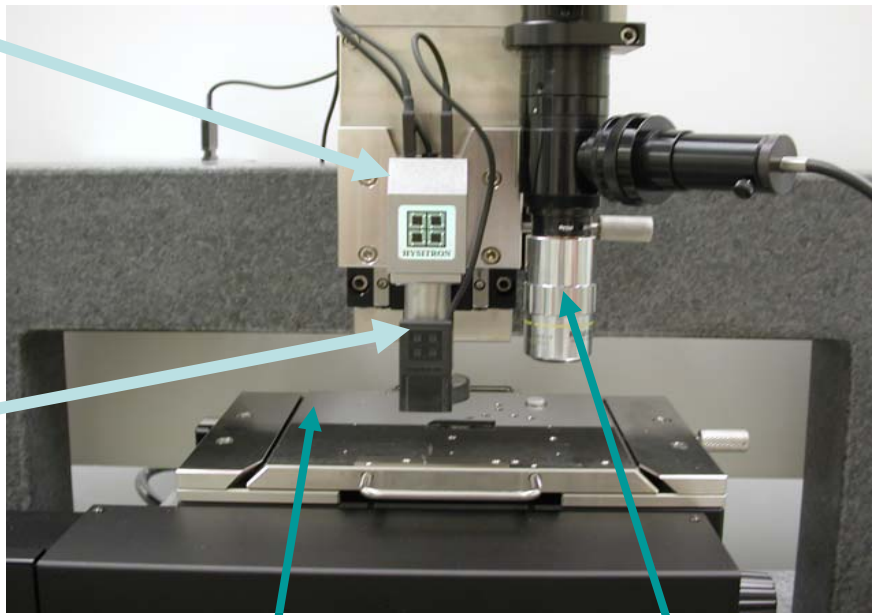



Quantification of Plowing in Nanofriction
Charles Ying, Nanotribology Group, Ceramics Division, Materials Science and Engineering Laboratory
Scaling Issue in Friction
What is the true friction value?
Approach
Study friction on Si using diamond tips of 3 different sizes
Correlate increase in friction force F with onset of grooving on substrate surface
Quantify displaced volume via
Tip Shape & Size Measurement Using Replica Technique
1. AFM imaging of tip's indent mark
2. Inversion, plus correction of elastic recovery
3. Quantify projected cross-section area vs. "penetration" depth
A general approach has been developed to quantify plowing in friction measurement
Friction in Elastic Region (diamond on Si)
Different COF values for different tips
When corrected by the effect of plowing, a common friction emerges.
Conclusion
Un-intended plowing by a sharp tip is a main cause of scaling antifact.
NIST-Hysitron Multi-scale Friction Apparatus

NIST APPROACH
Design of a new friction measurement apparatus

- **Nanoscale imaging**
  using a piezoelectric scanner

- **Quantitative & accurate friction measurement**
  using a two-dimensional force transducer



Sample platform on a *x-y* translation stage

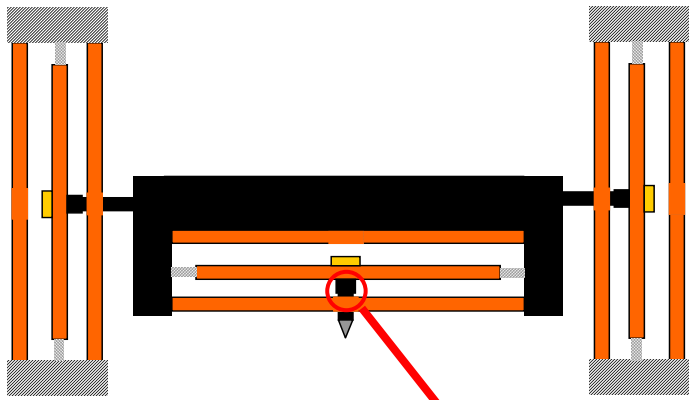
Optical microscope with CCD camera

- **Samples of up to 10 cm in size can be studied with 100-nm positioning accuracy.**

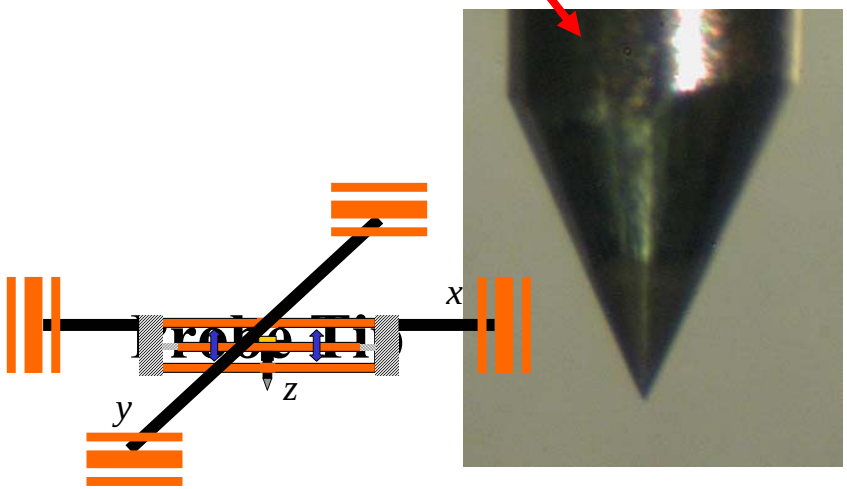
Force Transducer

Each transducer has

- **Three parallel plates with the 2 outer plates fixed**
- **A moveable center plate, attached to springs with calibrated spring constant**



- The center plate actuated via electrostatic force
- The center-plate position measured via capacitance using small AC voltage

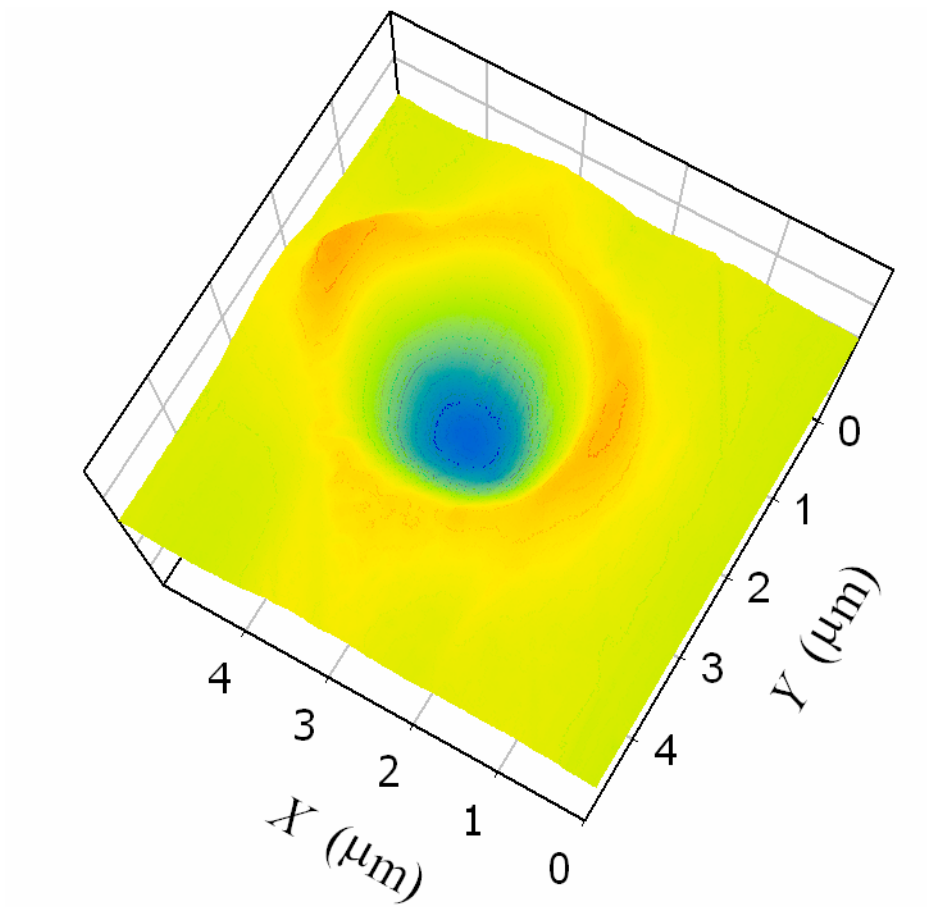


Digital reconstruction of tip

- Make an imprint of the tip on a soft material
- Surface roughness is an issue
- Depth of penetration restricted to the depth used in actual experiments

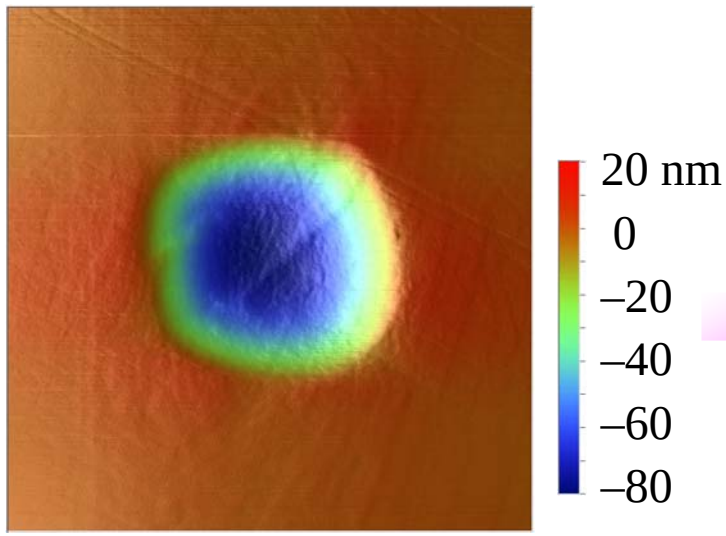
Example

- Radius $R \sim 1.5$ μm
  - After correction of elastic recovery

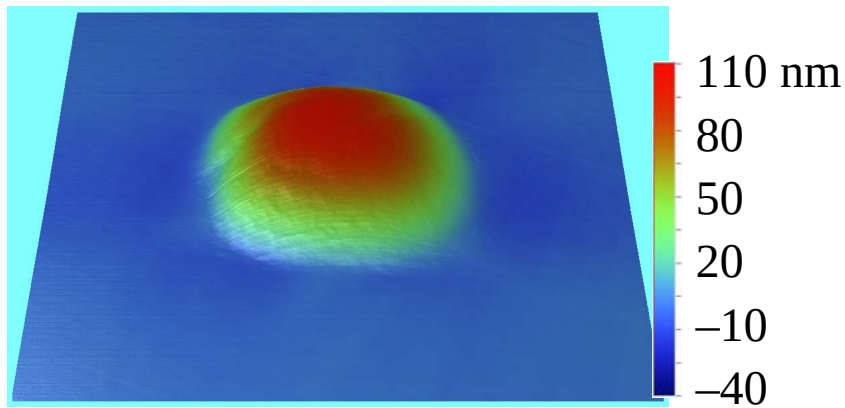


Digital reconstruction of Tip

- AFM imaging of tip's indent mark on flat surface of a ductile substrate
- Inversion of the AFM data
- Correction of small elastic recovery of the substrate



2×2 μm²



2×2 μm²

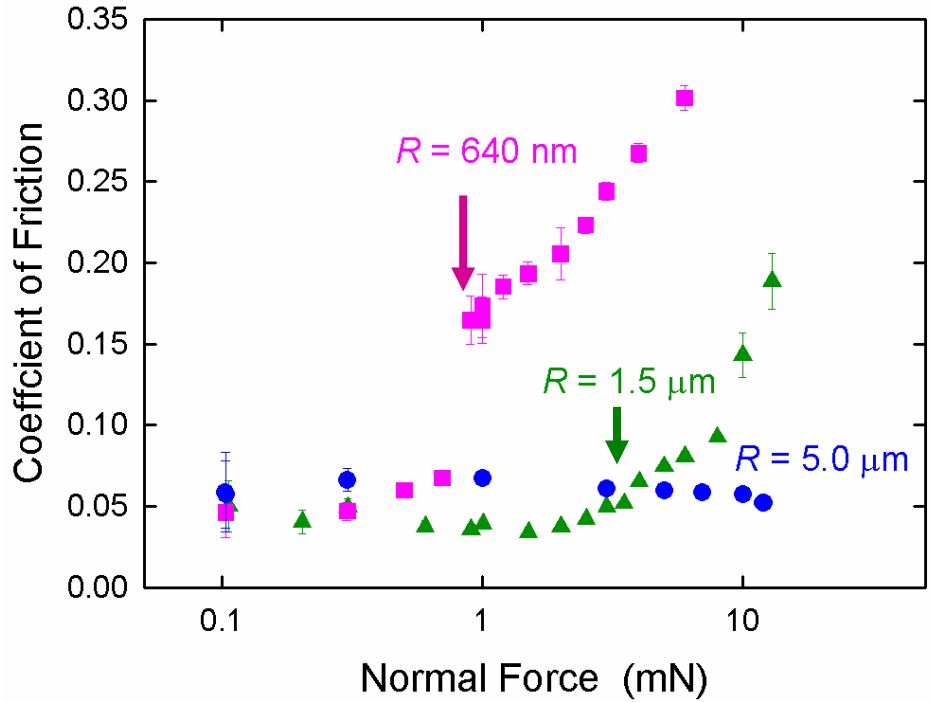
Effect of Tip Radius

- **Onset of enhanced friction**

  1 mN for R = 640 nm

  3 mN for R = 1.5 μm

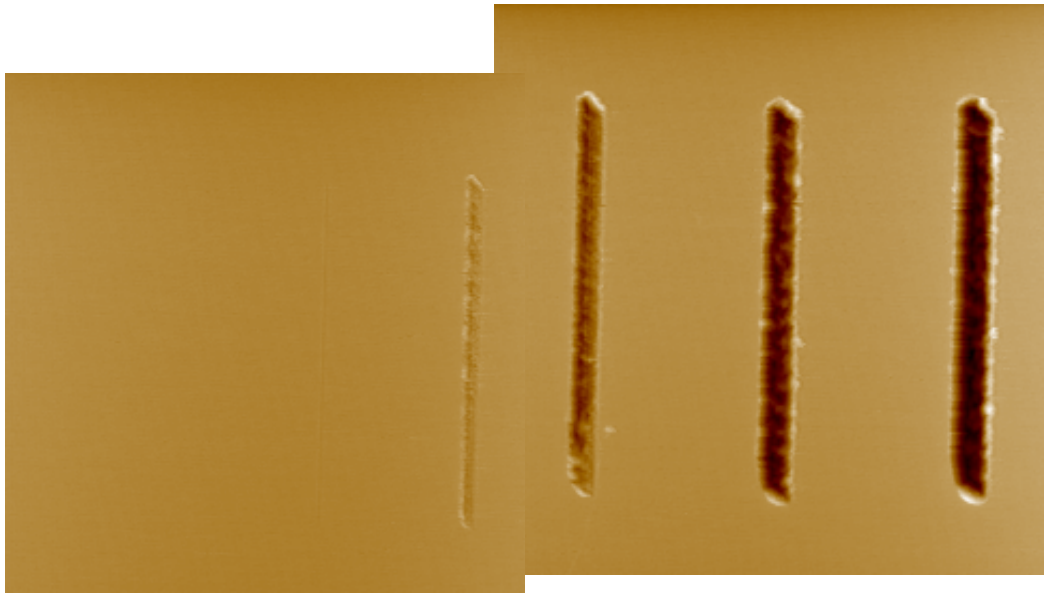
  >13 mN for R = 5.0 μm



- **Onset of enhanced friction increases with R**

Scratch Marks and Enhanced Friction

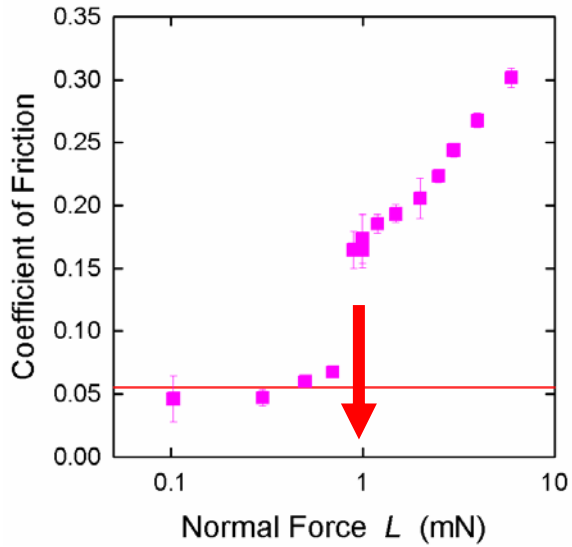
- No mark for load L = 0.1 and 0.3 mN
- Friction marks for $L \geq 1$ mN



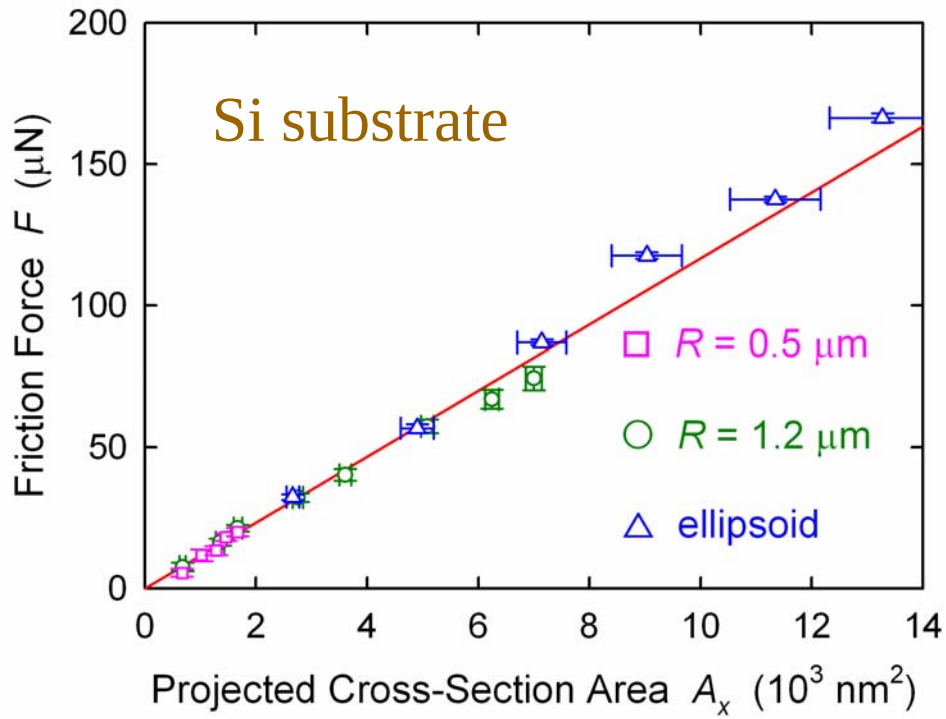
24×12 μm

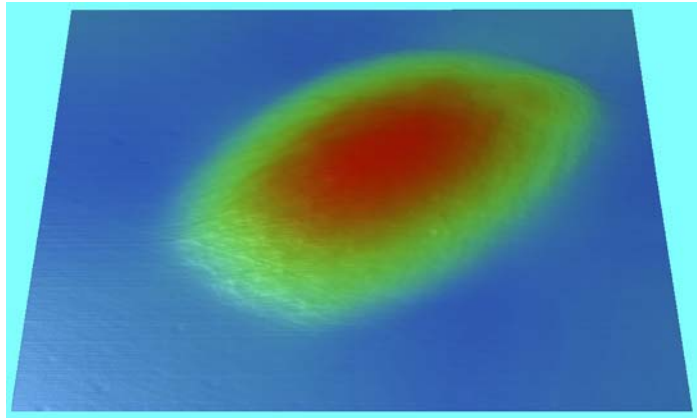
0.1 0.3 1 2 3 4 mN

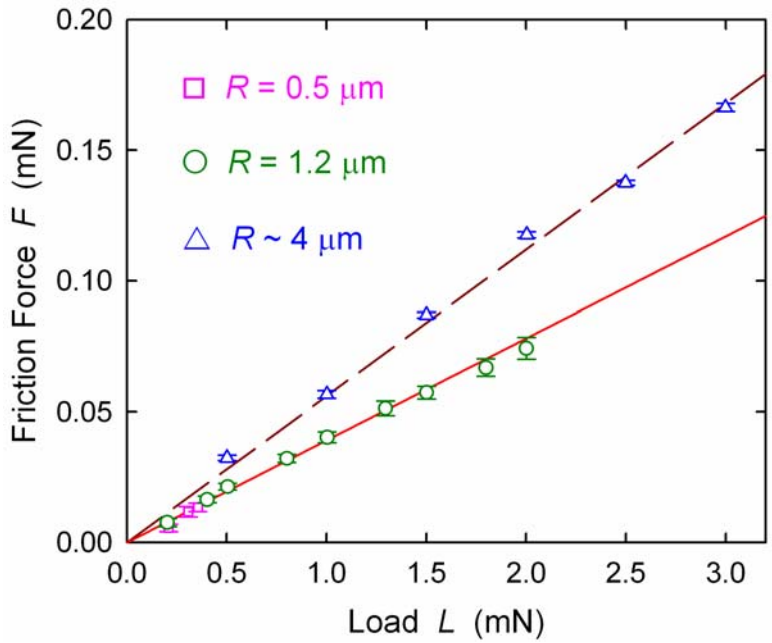
Onset



$F = sA_x$ also valid for non-spherical tips



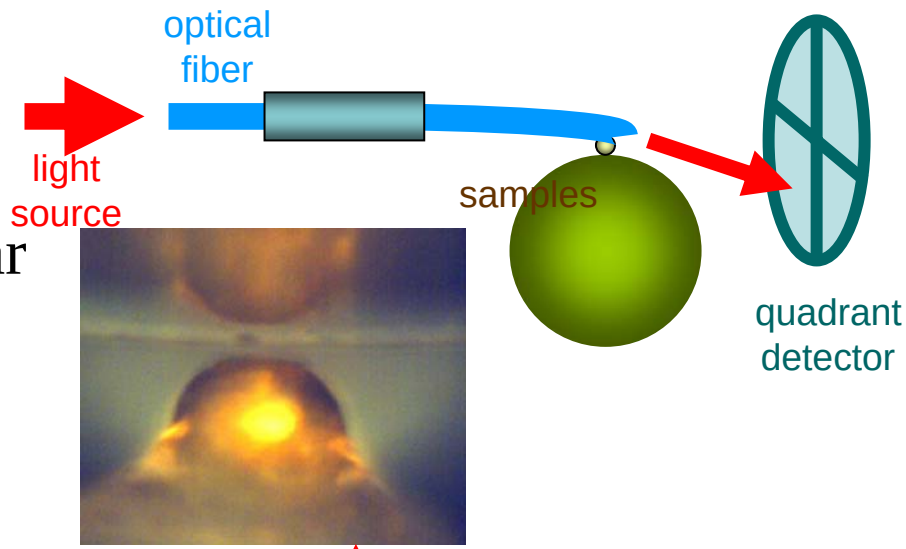


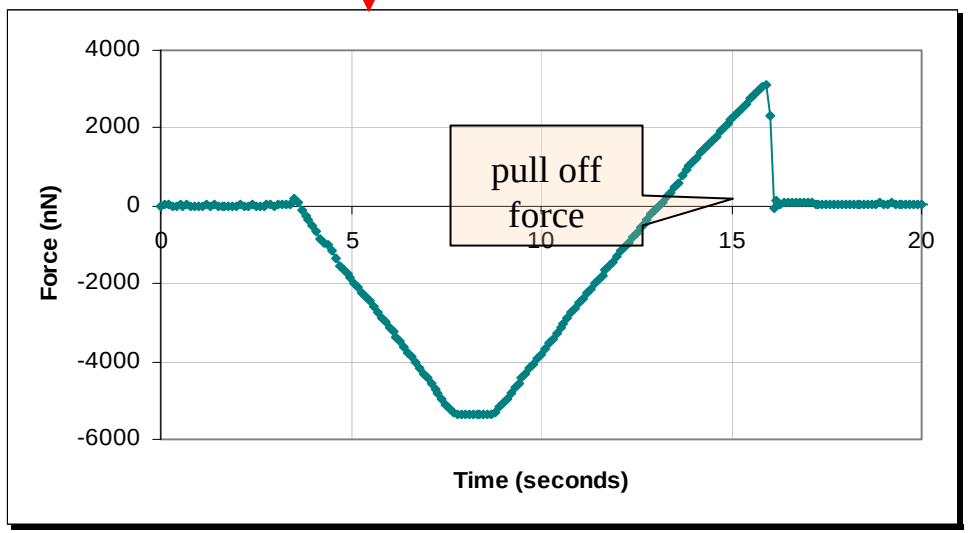


NIST Nano-Adhesion Apparatus
Instrument Requirements
Uniqueness
Test Measurements
Contact
Design – Concept
Future Plan
Spring Constant Calibration
NIST Nano-Adhesion Measurement Apparatus
TMC
TMC

NIST Nano-Adhesion Measurement Apparatus

A unique design using a variable length of optical fiber as a spring to measure displacement due to adhesion of two surfaces. Similar to an AFM's force measuring system.



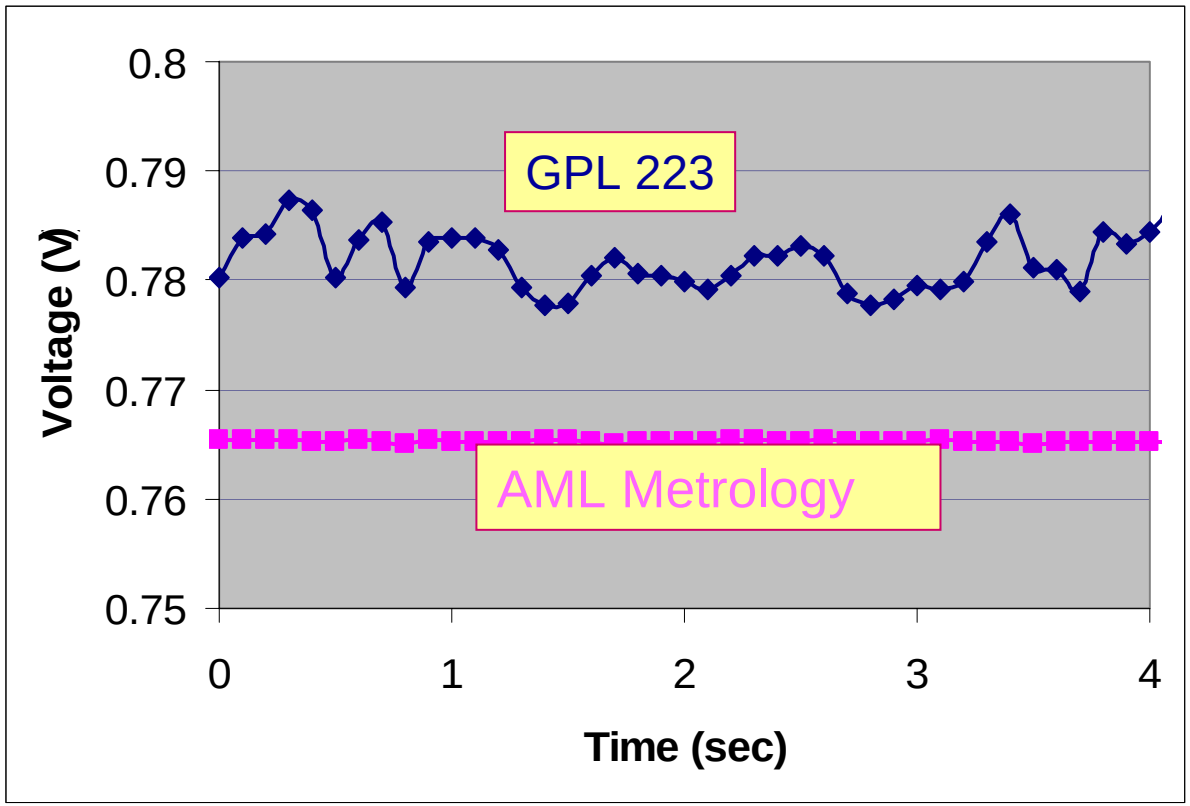


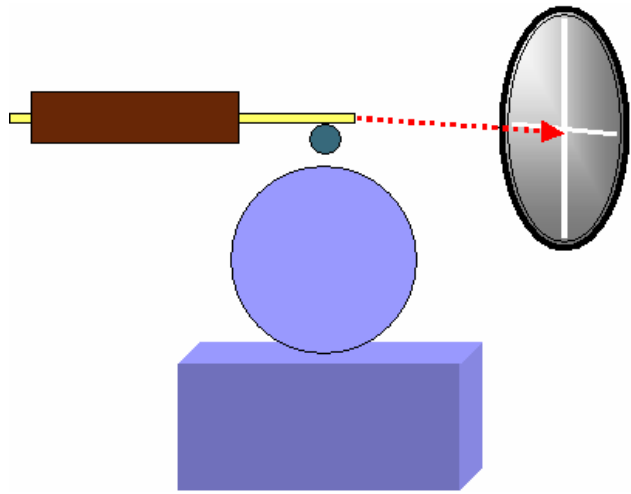
Capabilities

- Force measured by an optical fiber
  - mechanical rigidity
  - adjustable force sensitivity & range
  - wide dynamic range: 10 nN to 1 N
- External load capability
- Contact-area control
- Environmental control

Nanoadhesion force measurement baseline fluctuations

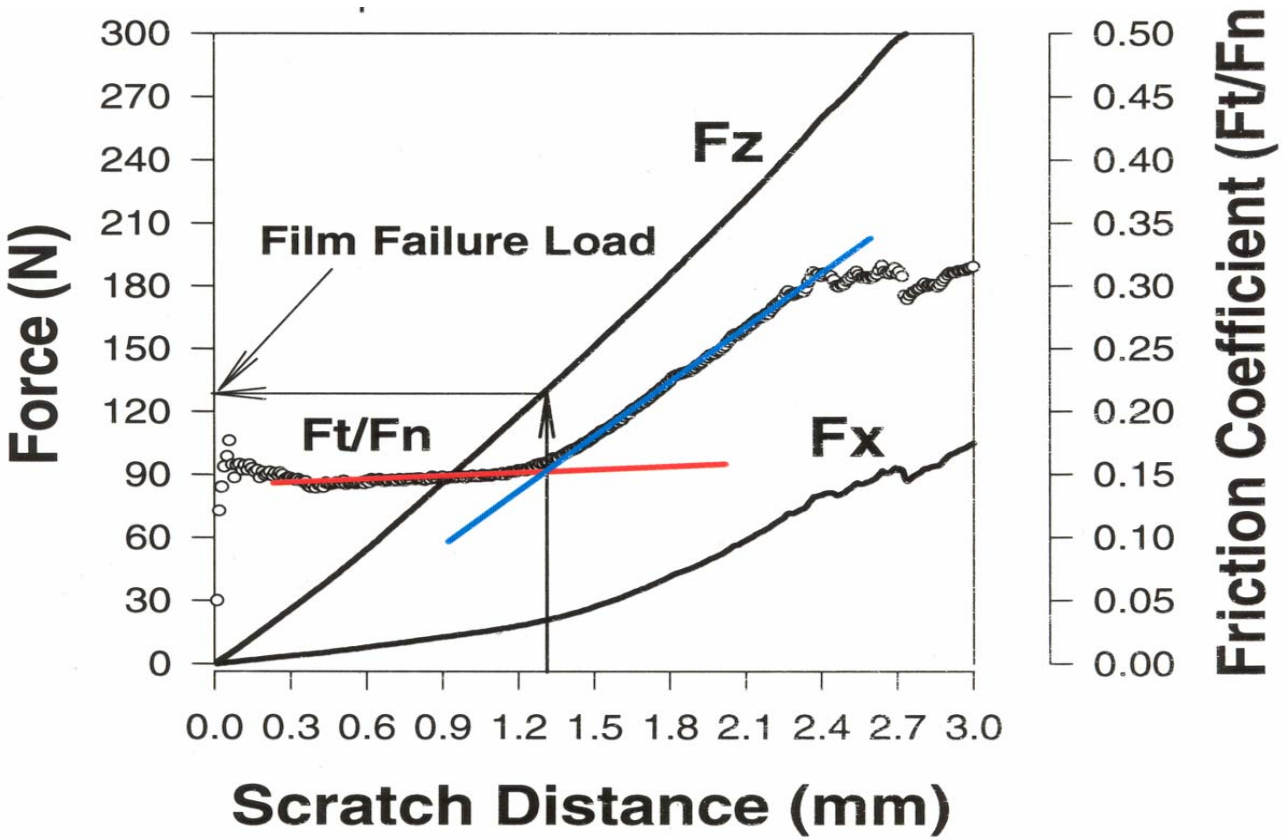
Reduction of baseline force fluctuation from ~±150 nN to ~±5 nN

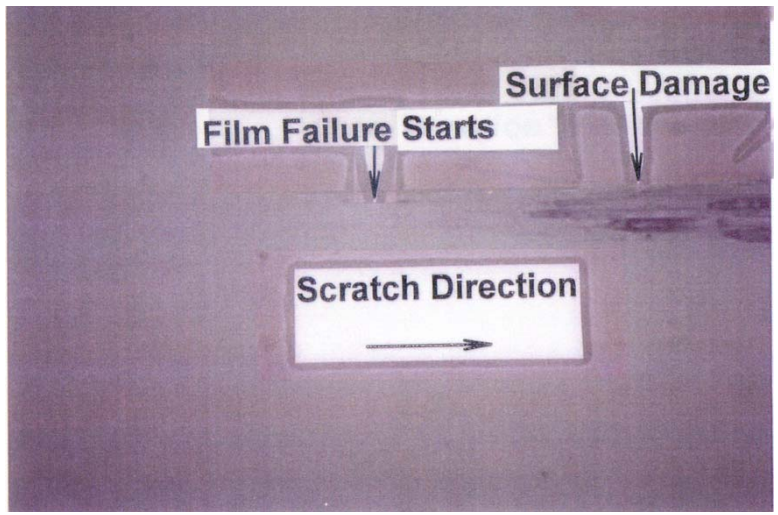


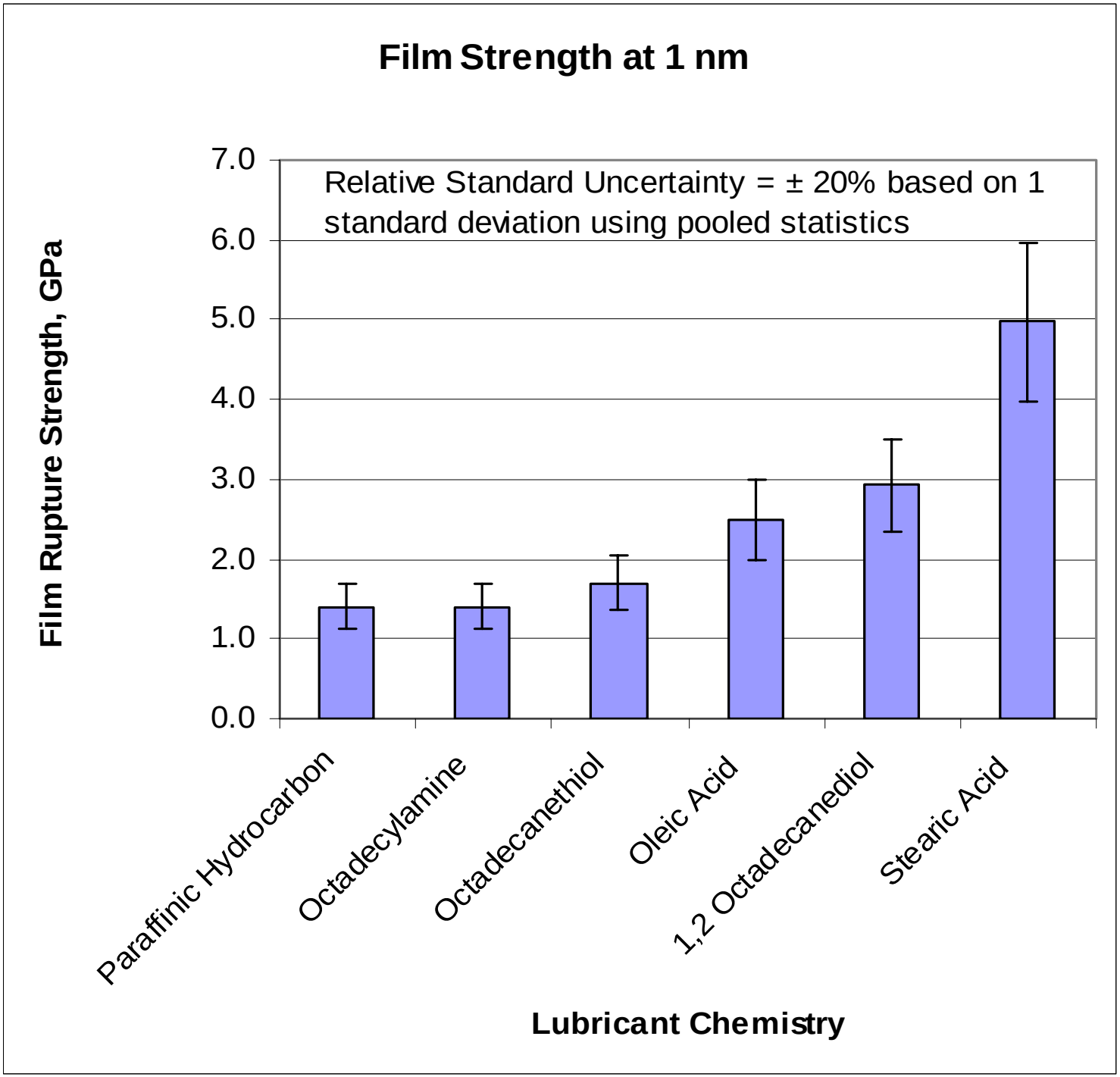


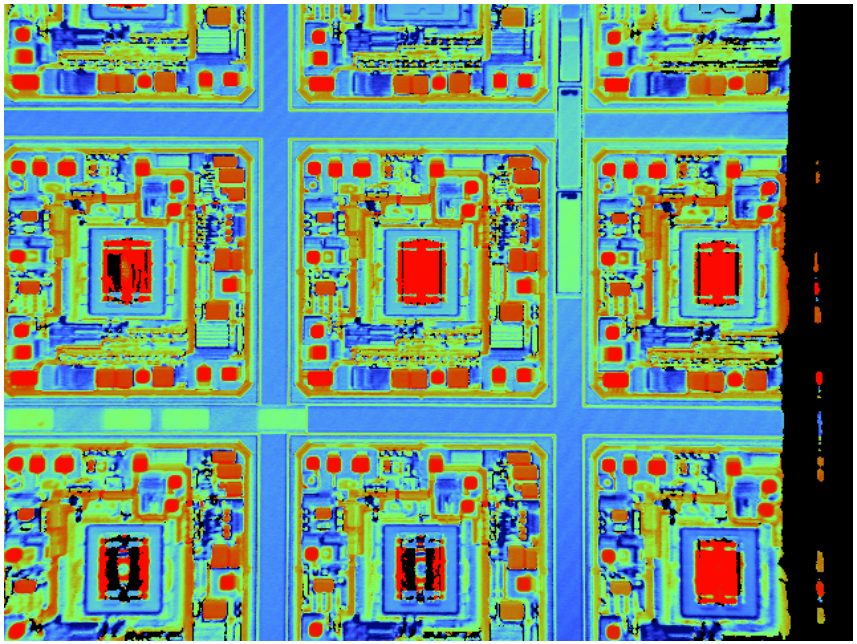
Position variation of a laser light spot on a photodiode

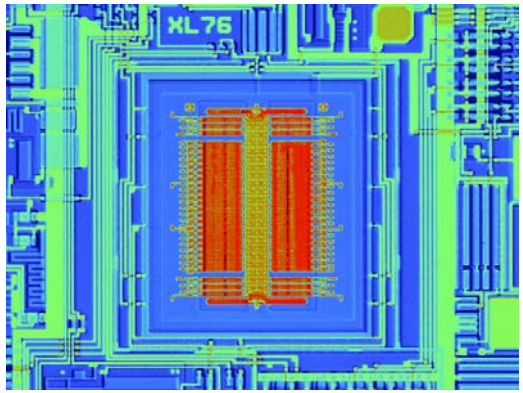
NIST Nano-Adhesion Measurement Apparatus
Capabilities

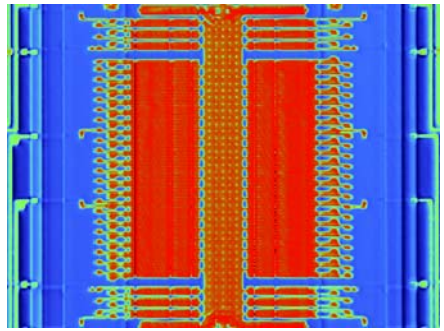

Force (N)
300
270
240
210
180
150
120
90
60
30
0
Fz
Film Failure Load
Ft/Fn
Fx
0.50
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
Friction Coefficient (Ft/Fn
0.0 0.3 0.6 0.9 1.2 1.5 1.8 2.1 2.4 2.7 3.0
Scratch Distance (mm)

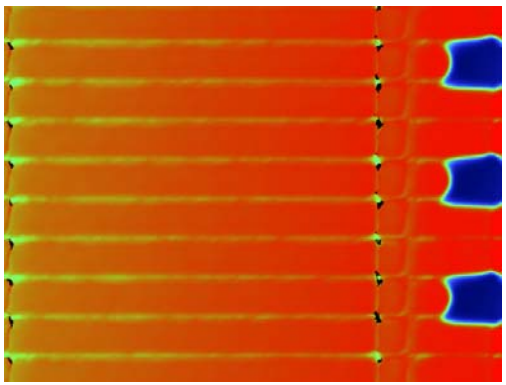

Surface Damage
Film Failure Starts
Scratch Direction

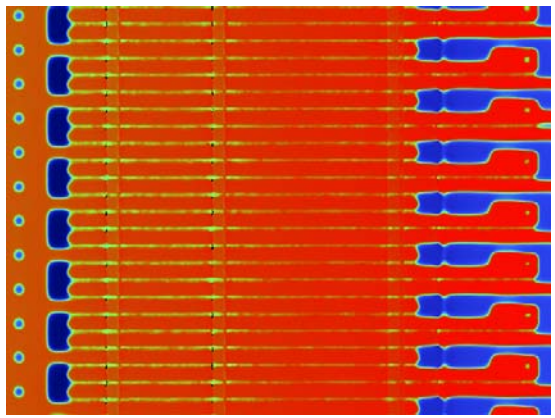

Film Strength at 1 nm
Relative Standard Uncertainty = ± 20% based on 1 standard deviation using pooled statistics
Film Rupture Strength, GPa
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
Paraffinic Hydrocarbon
Octadecylamine
Octadecanethiol
Oleic Acid
1,2 Octadecanediol
Stearic Acid
Lubricant Chemistry

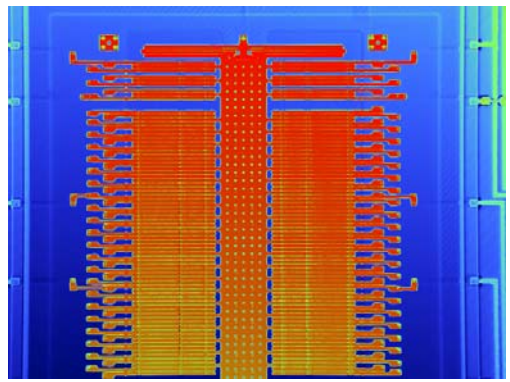



XL76









NIST MSEL Nanoscale measurement laboratory

- Unique set of integrated instruments
- World class capability & facility
- Nanoscale measurement and imaging

Joint projects welcome

Criterion based on scientific merits, relevance to NIST programs