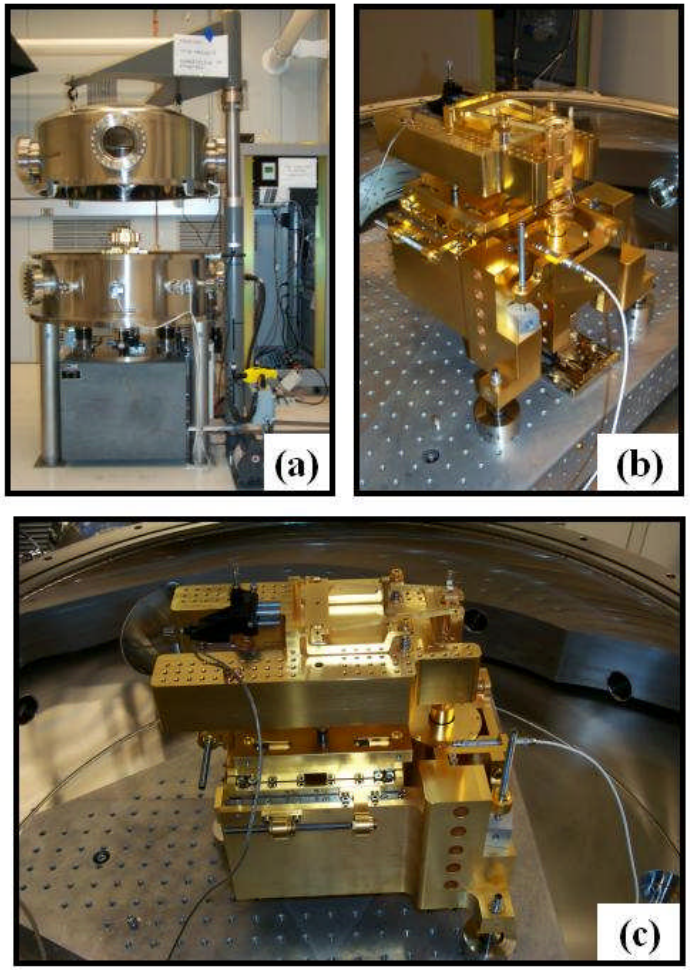
Low Force Metrology at the National Institute of Standards and Technology



Jon R. Pratt,
National Institute of Standards and Technology
Gaithersburg, MD 20899

2nd Tri-National Workshop on Standards for Nanotechnology
2/6/08

NIST
National Institute of Standards and Technology
Technology Administration, U.S. Department of Commerce

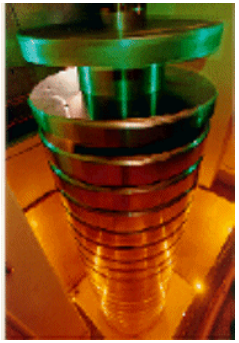
Outline

- Background
- Dissemination and calibration
- Intrinsic forces
- Summary

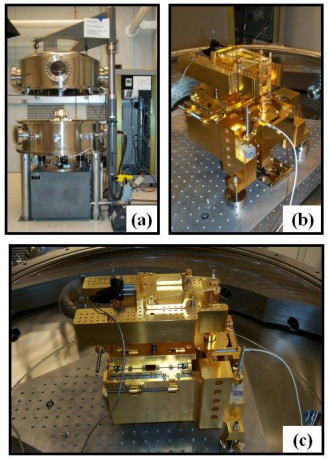
SI traceable force at NIST

- SFML traceably measures microforces using a balance and transfer artifacts achieving nN resolution
- **Molecular and atomic forces are one million times smaller**

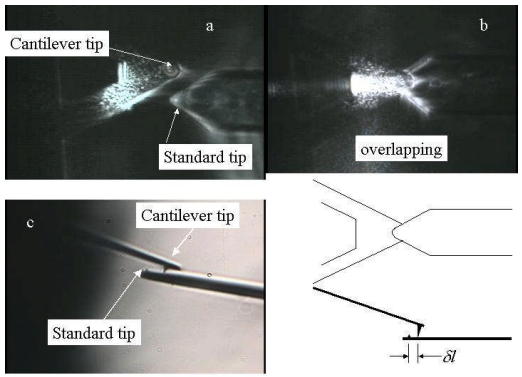
calibrated artifacts vs intrinsic forces

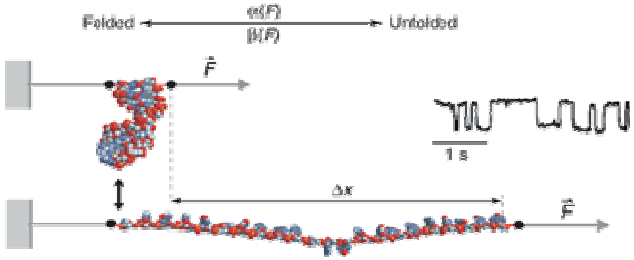


Forces
Classical mass artifact



Microforce Competence establishes SFML in FY04
shift from deadweight paradigm by linking force to quantum invariants in electrical units





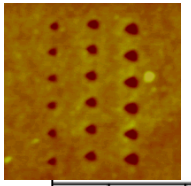
Intrinsic force project started FY06

Shift from calibrating artifacts to "calibrating" atomic and molecular interactions

Apply SI traceable electrical and optical forces directly to instruments

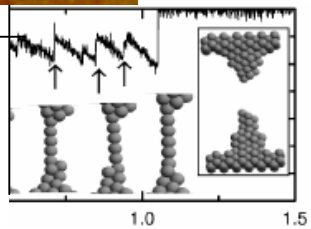
Small forces in perspective

read head "lift" forces, 10 mN

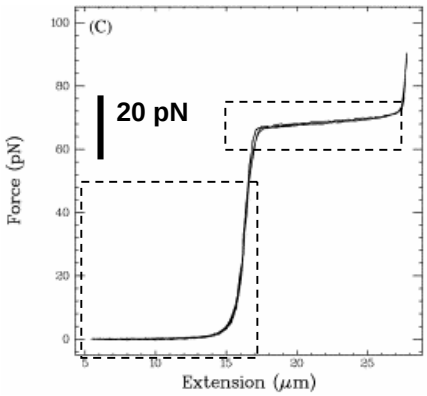


Thin film hardness tests, 1μN to 10 mN

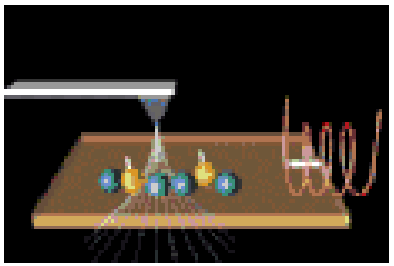
1 nN



Bond rupture experiments, 1 nN to 2 nN



Single molecule pulling, 1pN to 500 pN



Unpaired electron spins, aN's?

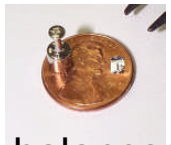
10^{-2} N

10^{-6} N

10^{-10} N

10^{-14} N

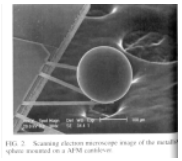
10^{-18}

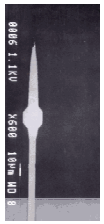


mass balances

Instrumented indentation

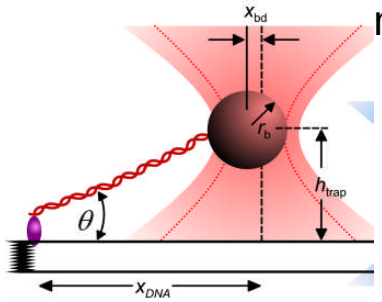
atomic force microscopy





Laser pointer, 6.6 pN

optical tweezers



magnetic resonance force microscopy

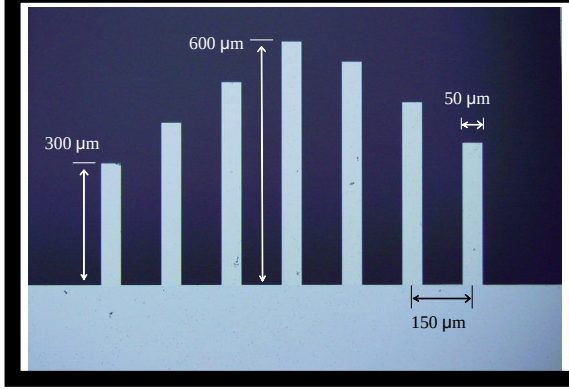
AFM force/stiffness standards

AFMs are used for imaging and materials testing

- Users want quantitative displacement *AND* force
- Manufacturers want to test their products
- Standards organizations need references
  - ISO/TC 201 on surface chemical analysis, SC 9

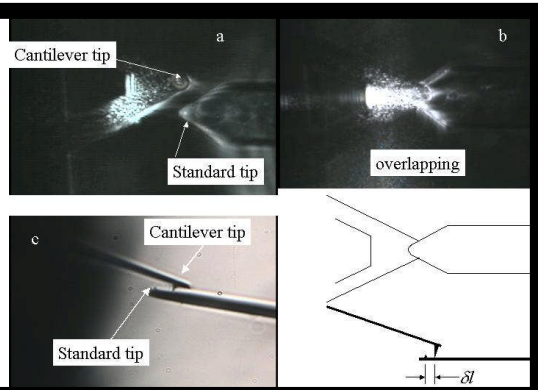
$F = k_c z$
But…
z = ?,
k_c = ?, and
F = ?

UK, Germany, and Korea:
Cumpson, et al, Nanotechnology, 2003,
Behrens, et al, J. Micromech. Eng., 2003,
Kim, et al, Metrologia, 2006



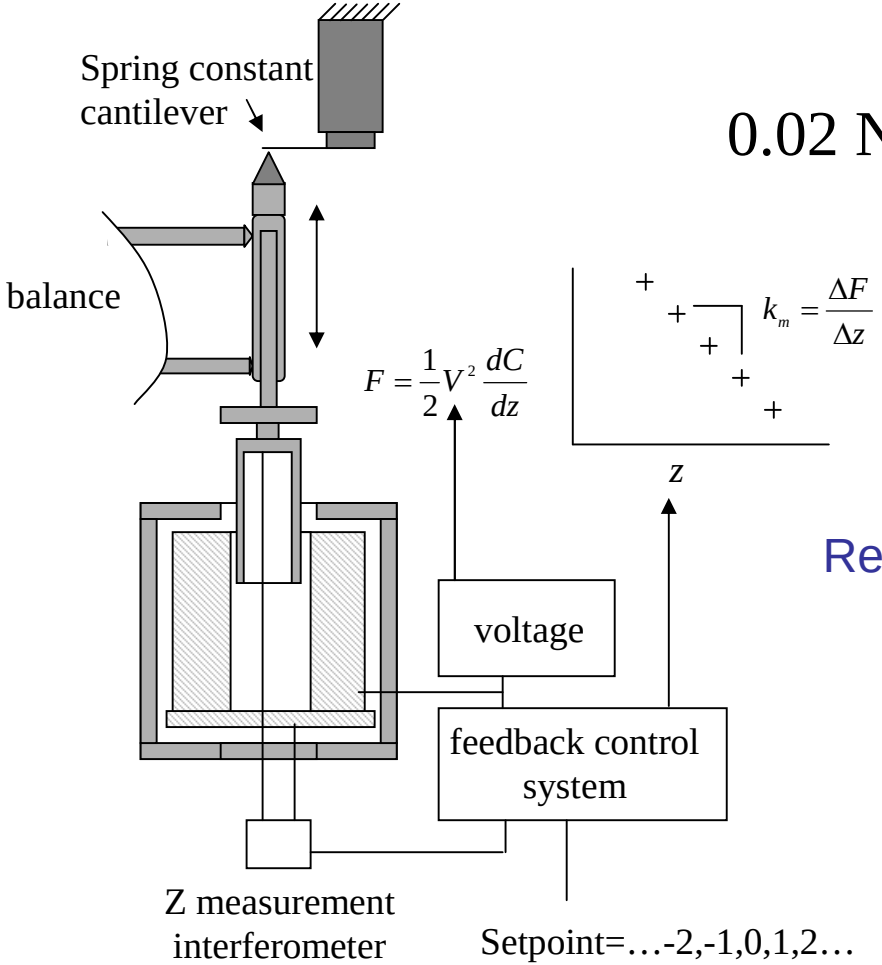
NIST traceable *k*
Gates & Pratt, Meas. Sci. Tech, 2006

NIST traceable *F*,
Pratt, et al, J. Matr. Res., 2004

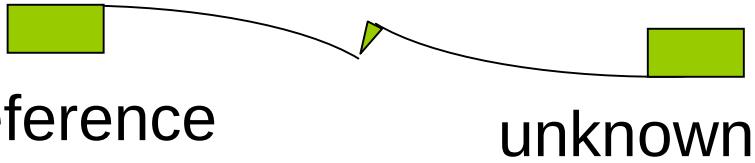


EFB as instrumented indenter

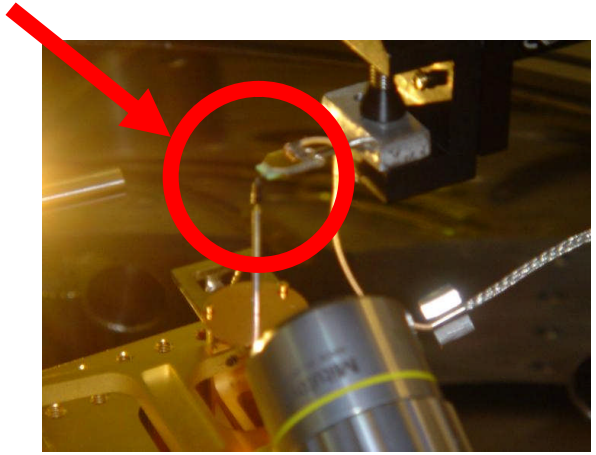
- Calibrated cantilever reference springs can be used as AFM sensing elements or as reference standards for cantilever on cantilever calibrations



$$0.02 \text{ N/m} < k < 50 \text{ N/m} \pm 0.0005 \text{ N/m}$$



Reference and EFB probe tip



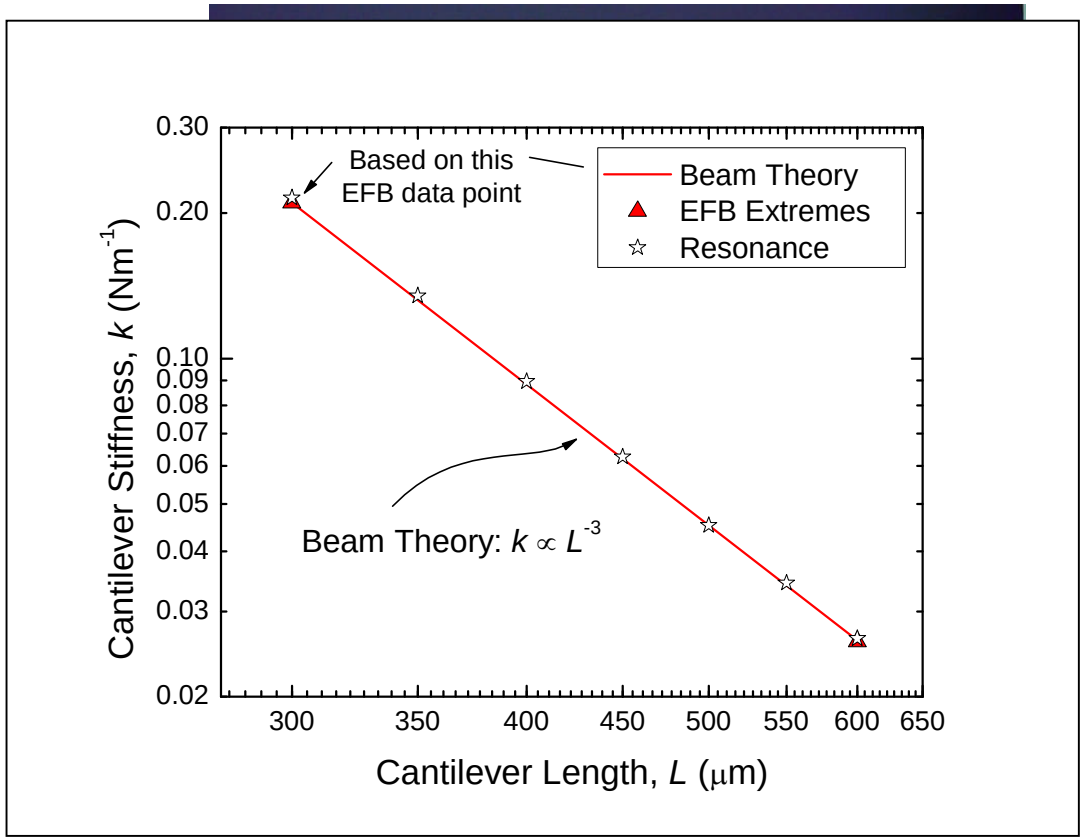
NIST reference cantilever array

- Nominal stiffness values are determined from measurements of resonance frequency, geometry, bulk value of density, and beam bending theory using

$$k = 9.585 L b t \rho f_{vac}^{2}$$

- Absolute values are checked for quality control using NIST EFB in micro-cantilever stiffness calibrator mode

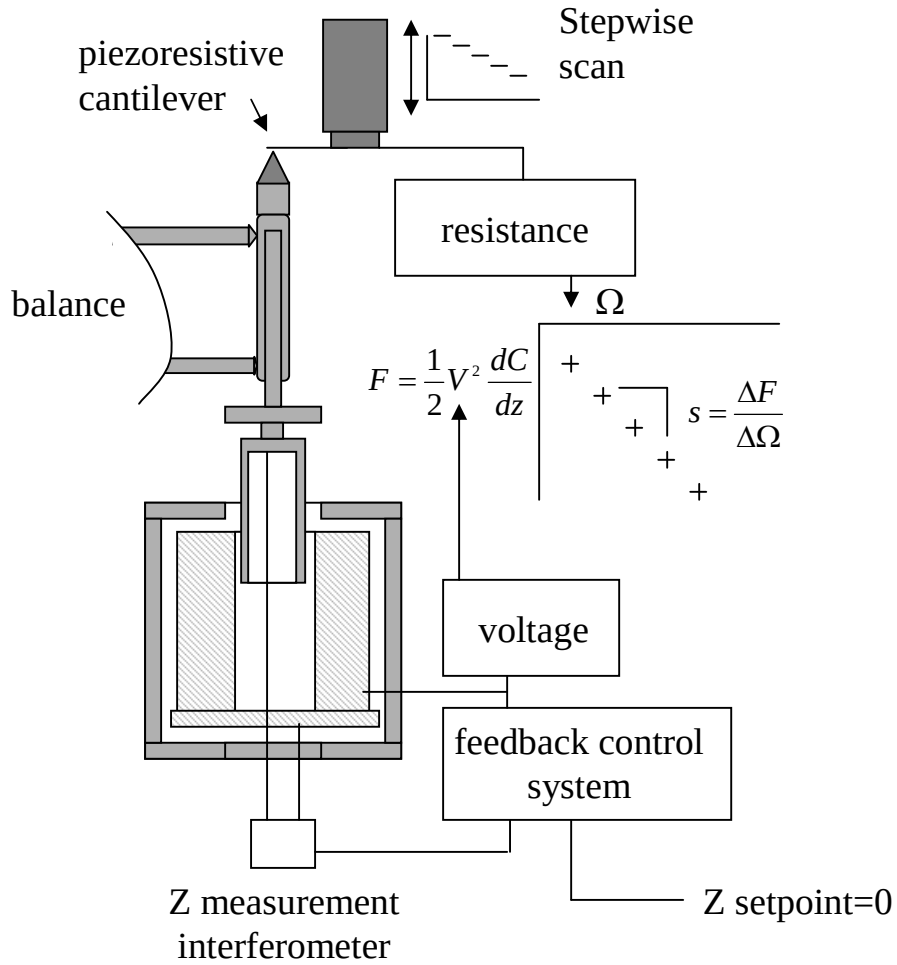
- Status: Dr. Richard Gates is developing process for fabrication at NIST. Hopeful to create SRM's within 2008.



Prototype cantilevers for SI-traceable nanonewton force measurement,
Gates & Pratt, Meas. Sci. Tech, 17, 2006

EFB as force calibrator

- Use EFB as instrumented indenter and calibrate *force sensors*, just as at macroscale

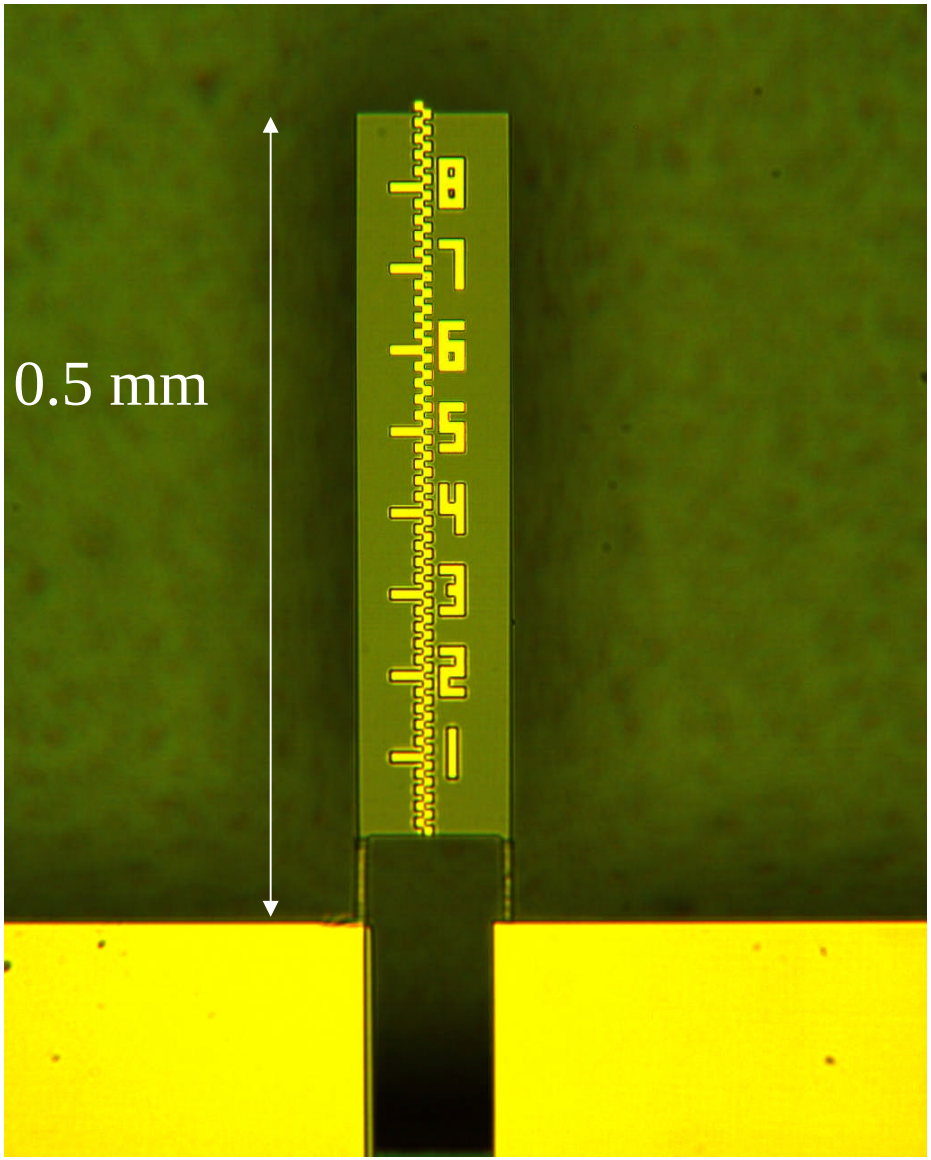


Force sensor calibration

- EFB instrumented indentation scheme measures absolute force and displacement required to move the balance suspension and any other spring in series with it

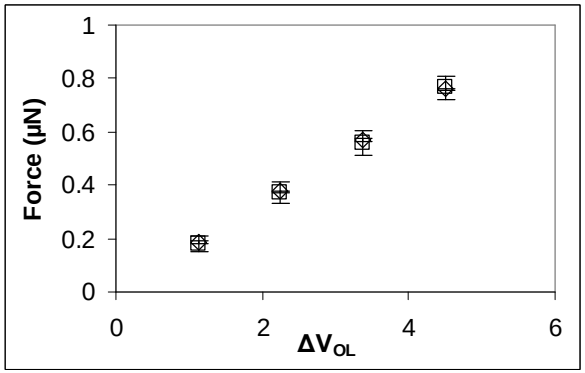
- Strain based sensors can be calibrated both as stiffness and force artifacts using the EFB instrumented indentation scheme, e.g., F vs d or F vs ohms, or F vs Volts from a bridge amplifier

- NIST has used a commercial sensor to demonstrate calibration chain ***Langlois et al, Rev. Sci. Inst., 78, 093705 (2007)***

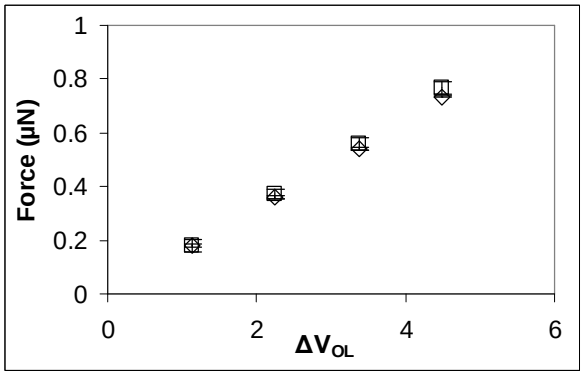


AFM calibration

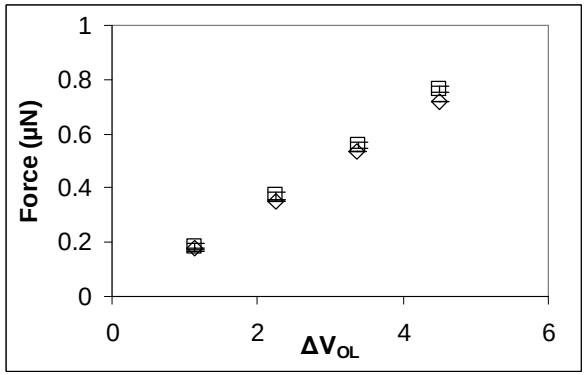
- Pressed AC mode type cantilever, 240 μm long, 55 μm wide and 4.6 μm thick with 1 N/m nominal spring constant against PFT



Fiducial 2



Fiducial 4



Fiducial 8

- AFM force sensitivity was determined from the slope of the contact force versus optical lever signal

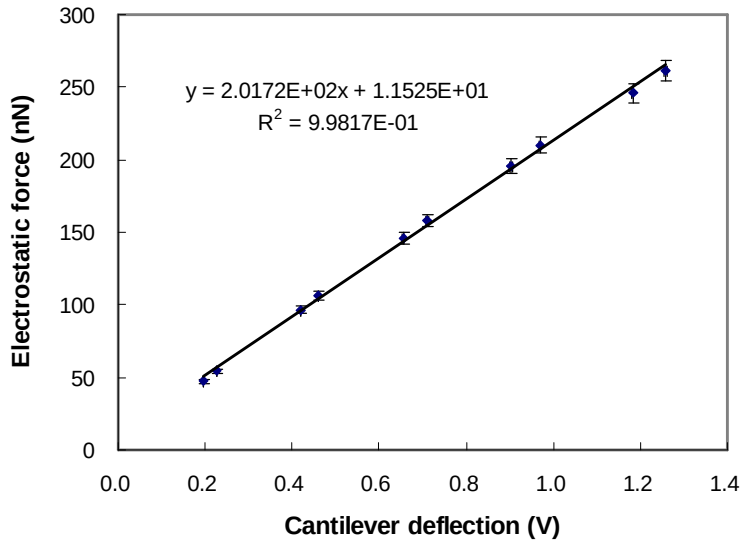
$$s_{AFM} = \Delta F_k / \Delta V_{OL} = (0.164 \pm 0.005)\ \mu N/V$$

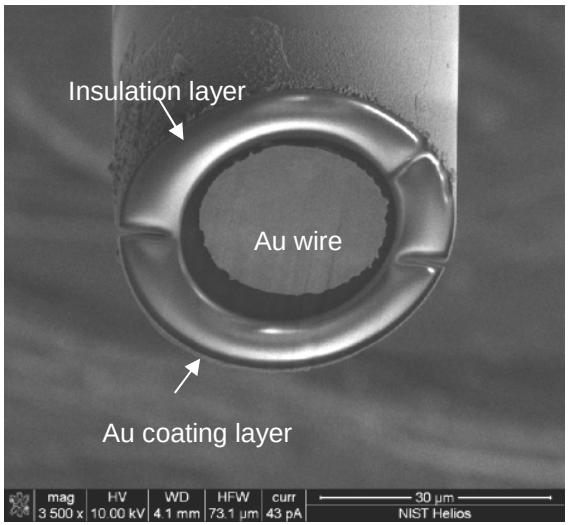
$$s_{AFM} = \Delta F_R / \Delta V_{OL} = (0.167 \pm 0.004)\ \mu N/V$$

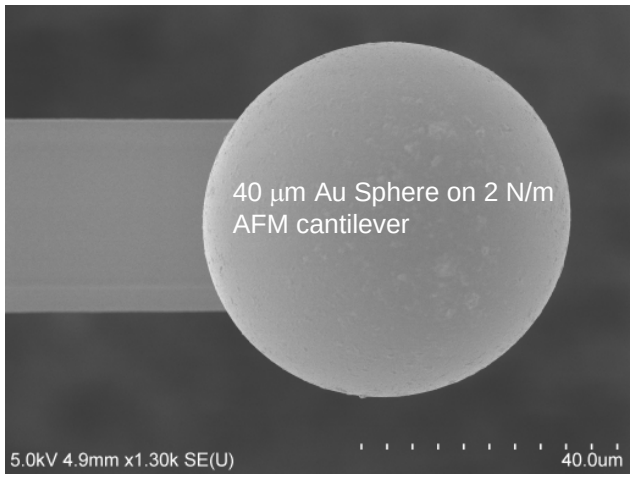
Pratt, et al, Mater. Res. Soc. Symp. Proc., Vol. 1021, 2007

Electrostatic nanonewtons applied directly to AFM sensor

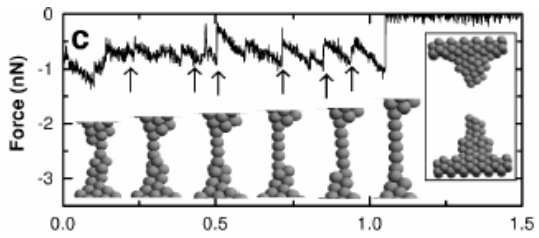
- Colloidal AFM probes find application in a variety of adhesion, friction and biological force studies, yet they are exceedingly difficult to calibrate because of their heightened sensitivity to off-axis force
- NIST has created a unique electrical probe to measure the capacitance gradient near the grounded, conducting sphere of a colloidal force sensor
  - Focused ion beam milling to create tiny coaxial conductor from a micropipette
- SI traceable electrostatic force applied directly to colloidal probe.



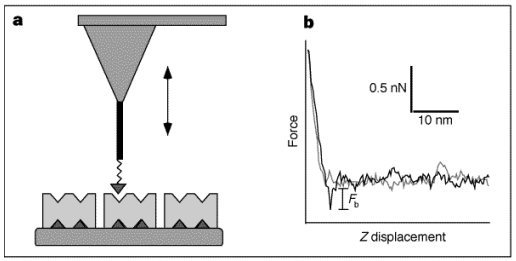




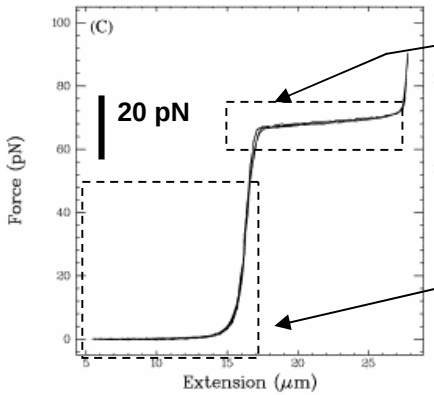
Prototype intrinsic forces from nature



Break a gold nanocontact



Pull a biotin molecule from a streptavidin binding site



Stretch DNA through a transition

Or

Simply stretch it and use as reference spring (nonlinear)

AFM

1 nN to 2 nN

Break a single atomic bond

- Highest force, most difficult experiment
- Quantum conductance at break is a plus!

100 pN to 200 pN

Rupture a binding site

- Rate dependent

35 pN & 65 pN

Change DNA structure

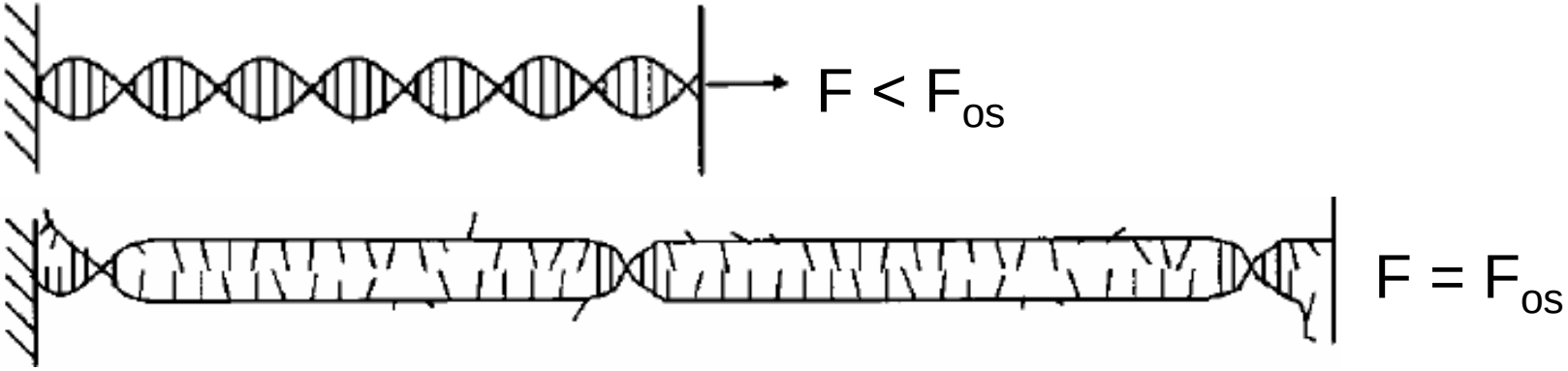
0.2 pN to 65 pN

Stretch DNA elastically

OT

DNA as a Force Standard

The overstretch transition: a partial melting of the DNA double helix in response to an applied force[REF WILLIAMS 2002]. A natural phenomenon where an increase in the length of a DNA molecule can be detected as a tensile force is applied to the DNA molecule



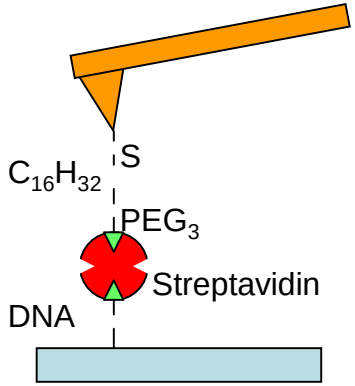
- F_{os} has been measured with AFM, optical tweezers, fluid flow, magnetic tweezers, and dielectrophoresis
- F_{os} depends on temperature, pH, ionic strength, and DNA sequence
- F_{os} is assumed to be 65 pN for most DNA at high salt concentrations, and room temperature

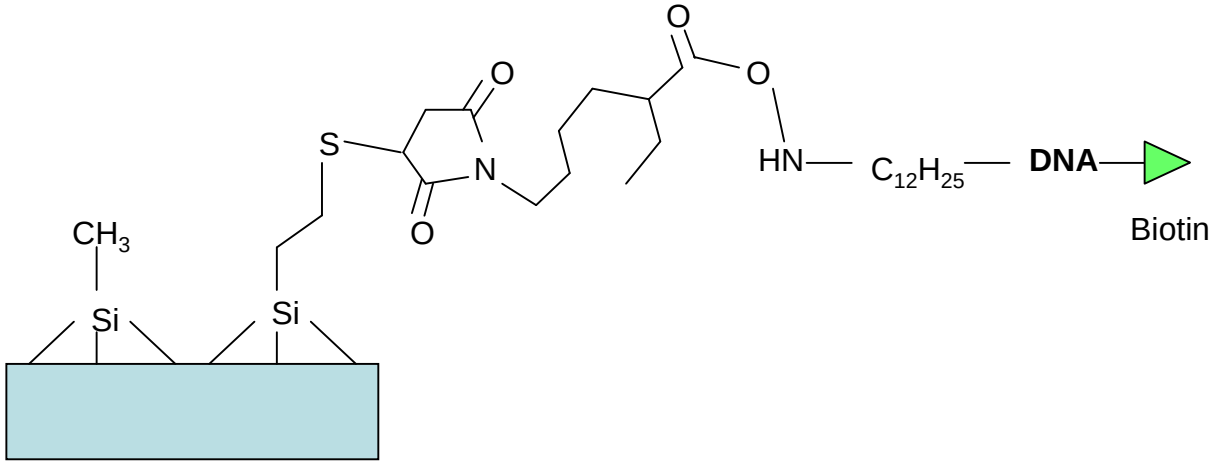
The intrinsic force project seeks to measure F_{os} in a metrological fashion traceable to the SI

DNA samples and surface chemistry

- Existing AFM DNA test protocols have been unreliable and poorly suited to standard development. Nonspecific binding is nonspecific!

- NIST is developing new AFM/DNA sample prep and testing protocol

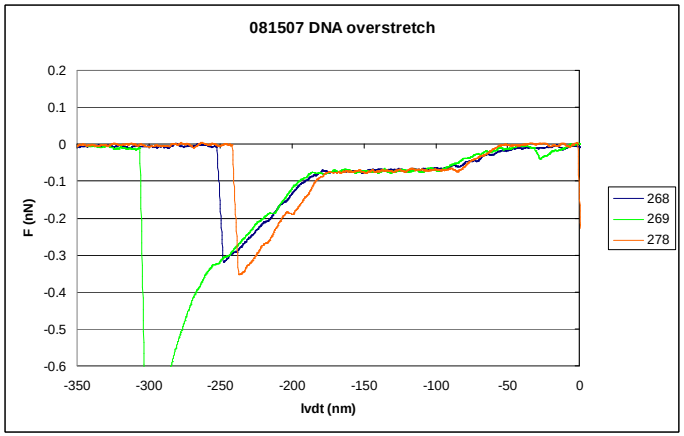


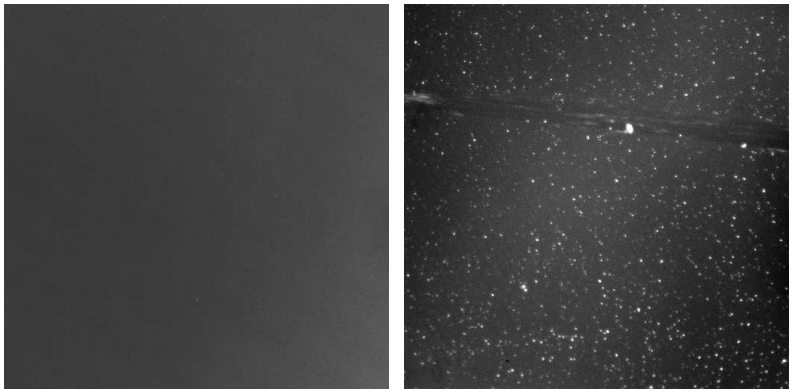


Preliminary DNA force data from AFM

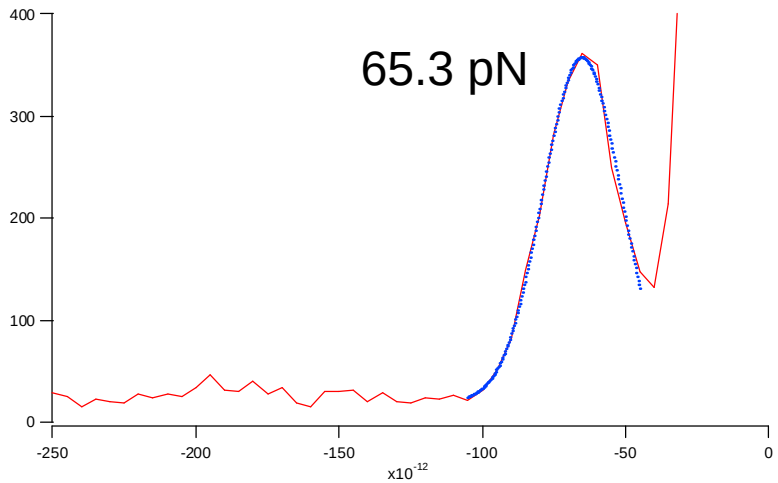
- Have attempted DNA overstretch measurements using state of the art commercial AFM in new Intrinsic force precision AFM laboratory
  - Latest NIST traceable force calibration procedures make this the most accurate force measuring AFM in the world
  - New NIST chemistry tethers DNA from the surface to minimize sensor surface interaction noise

- Single-molecule tensile data with forces traceable to SI realization of the newton





Flouresence microscope images before and after functionalizing surface with DNA. Collaboration with NIST CSTL microfluidics lab.



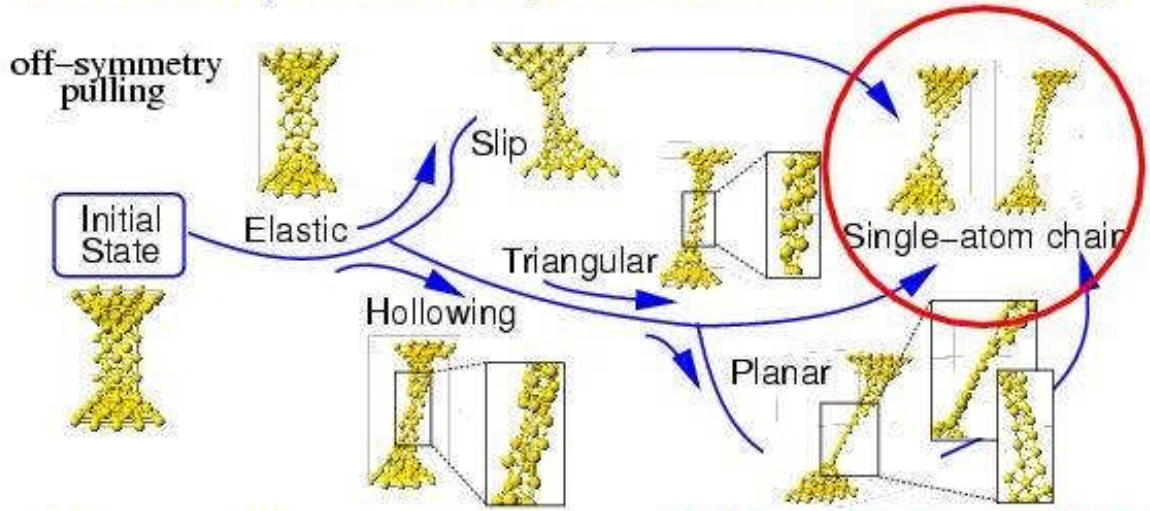
Simulations of Au bond rupture

(Preliminary) Results:

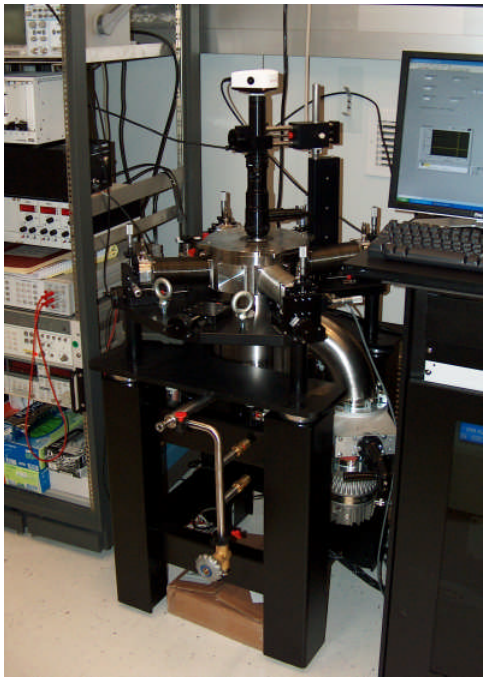
– Good news: Simulations suggest that, under real–world conditions (i.e. with symmetry broken by thermal vibrations or off–symmetry pulling, for instance), the force needed to break the NW is consistent and does NOT depend on:

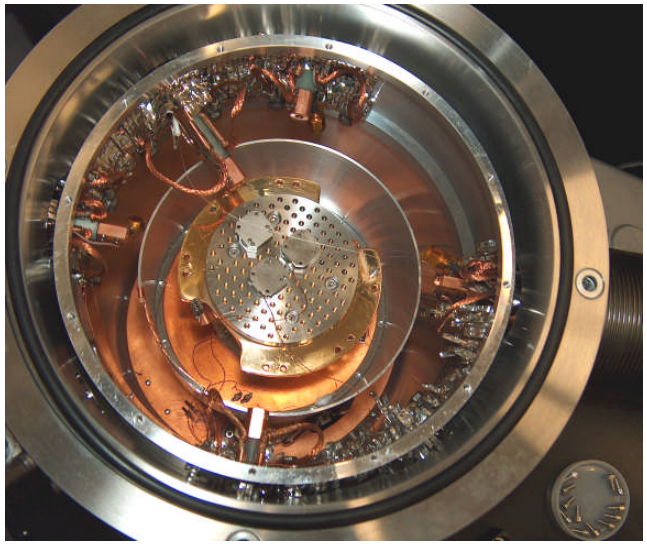
- – tensile axis (some dependence for the high–symmetry case at T=0K only);
- – stretching mechanisms;
- – strain rate;

➔ the NW always forms a single–atom chain before breaking.



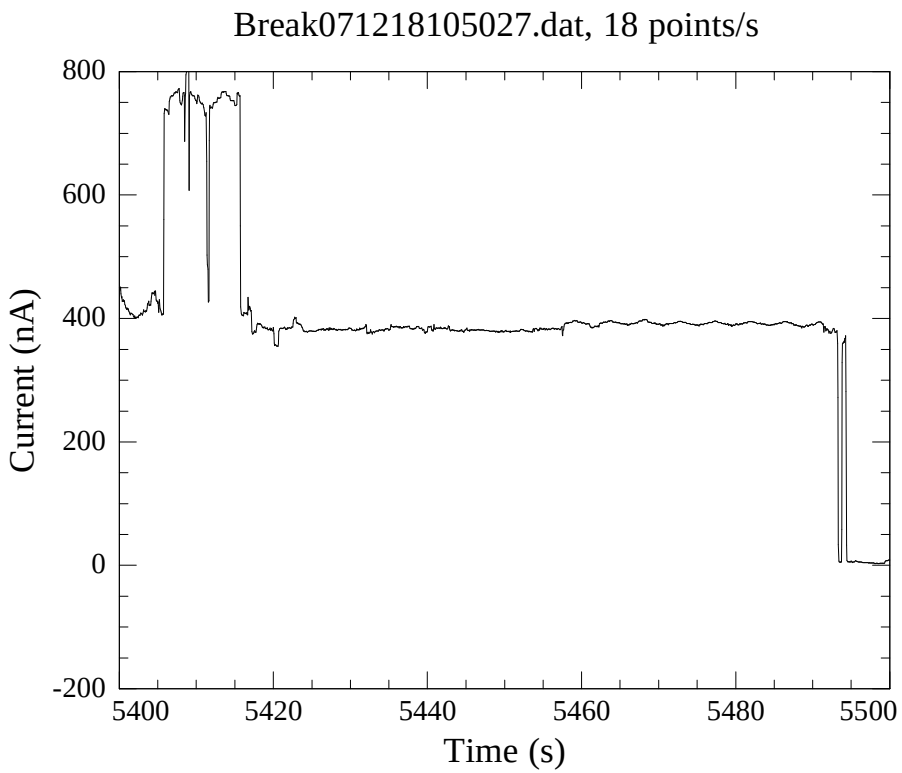
Many paths – only one final configuration! ➔ 1 breaking mechanism (one force)





Break junction test platform

- Recently measured quantized conductance of atomic gold junctions
- Does elastic response change in a quantized fashion?



Summary

- Small force measurement is a useful tool for nanomechanical characterization
- Small force metrology laboratory supports researchers in government, academia, and industry to calibrate small force measurements
  - SRM's are under development
  - We have calibrated instrumented indentation and AFM equipment
- Intrinsic standards may someday provide ready access to SI traceability for pN to nN measurements
  - DNA overstretch has been measured
  - Quantized conductance has been observed