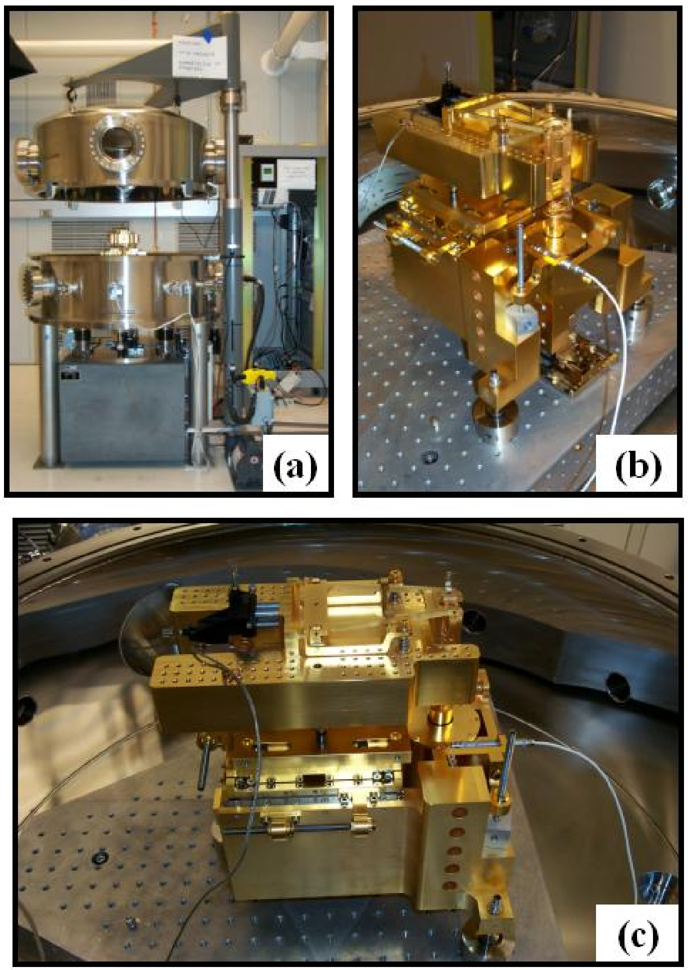
The Small Force Metrology Laboratory at the National Institute of Standards and Technology



Jon R. Pratt,
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Gaithersburg, MD 20899

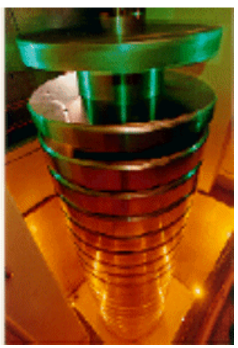
Tri-National Workshop on Standards for Nanotechnology
2/7/07

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

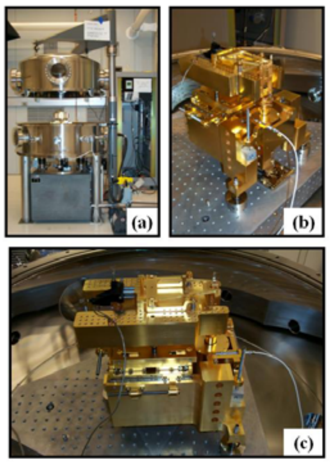
Evolution of SI traceable force at NIST

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

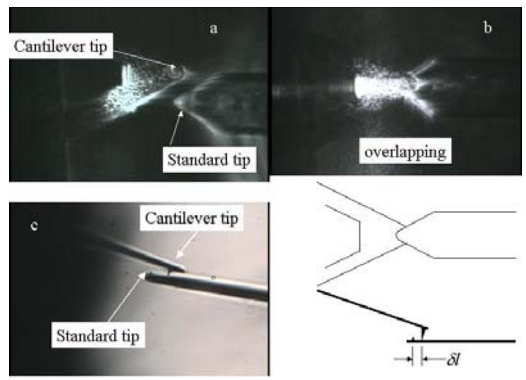
calibrated artifacts vs intrinsic forces

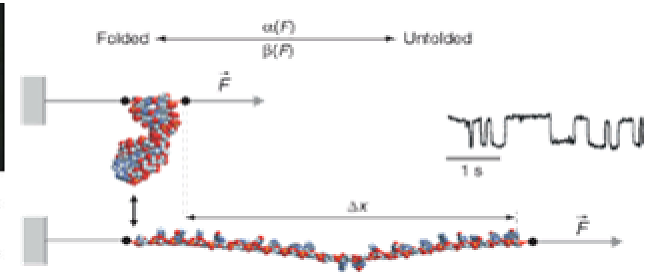


<u>Forces</u>
Classical mass artifact



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





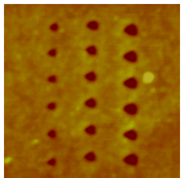
<u>Intrinsic force competence starting FY06</u>

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

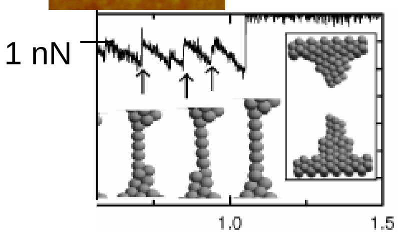
Apply SI traceable electrical and optical forces directly to instruments

Small forces in perspective

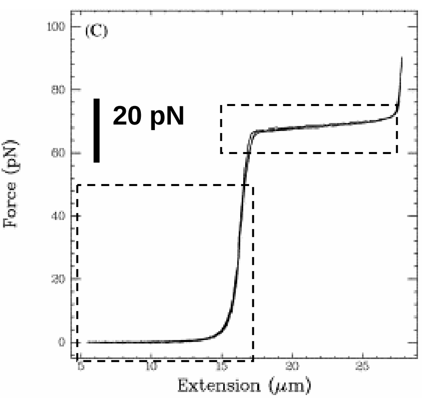
read head "lift" forces, 10 mN



Thin film hardness tests, 1μN-10 mN

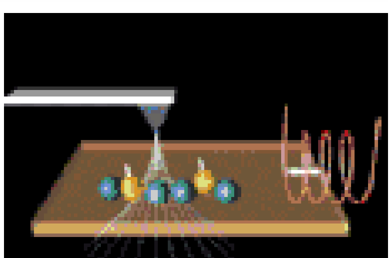


Bond rupture experiments, 1-2 nN

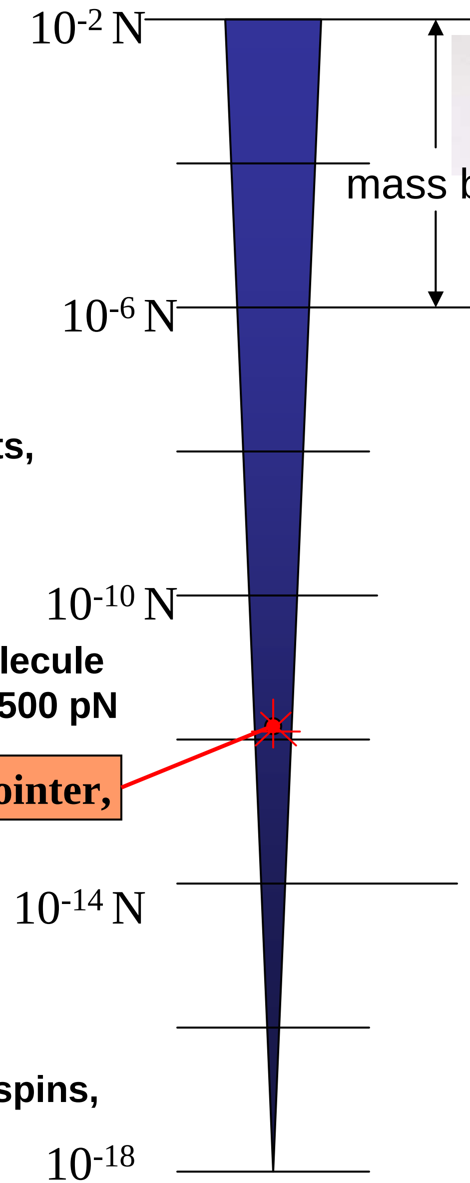


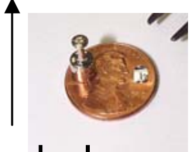
Single molecule pulling, 1-500 pN

Laser pointer, 6.6 pN



Unpaired electron spins, aN's?

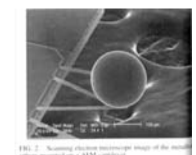




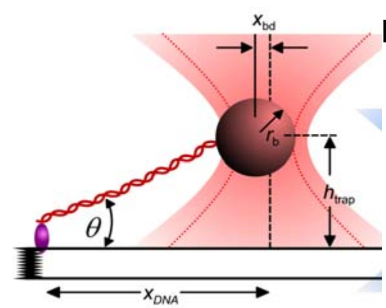
mass balances

Instrumented indentation

atomic force microscopy



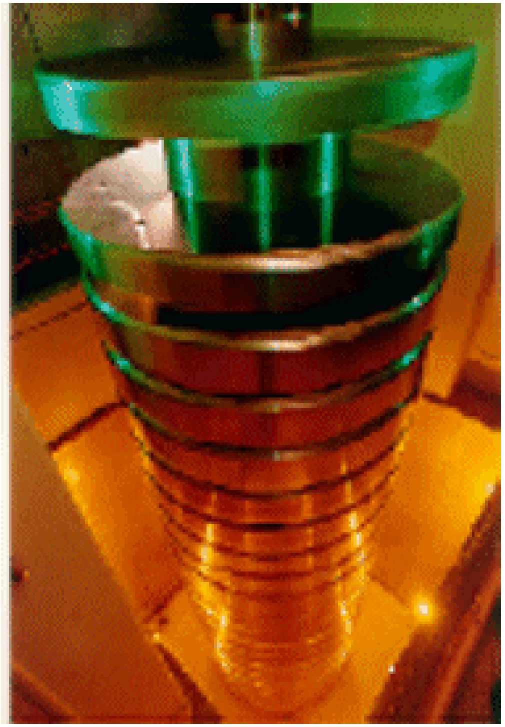
optical tweezers



magnetic resonance force microscopy

How do we calibrate a force?

- F=ma is a good start for a *deadweight* standard

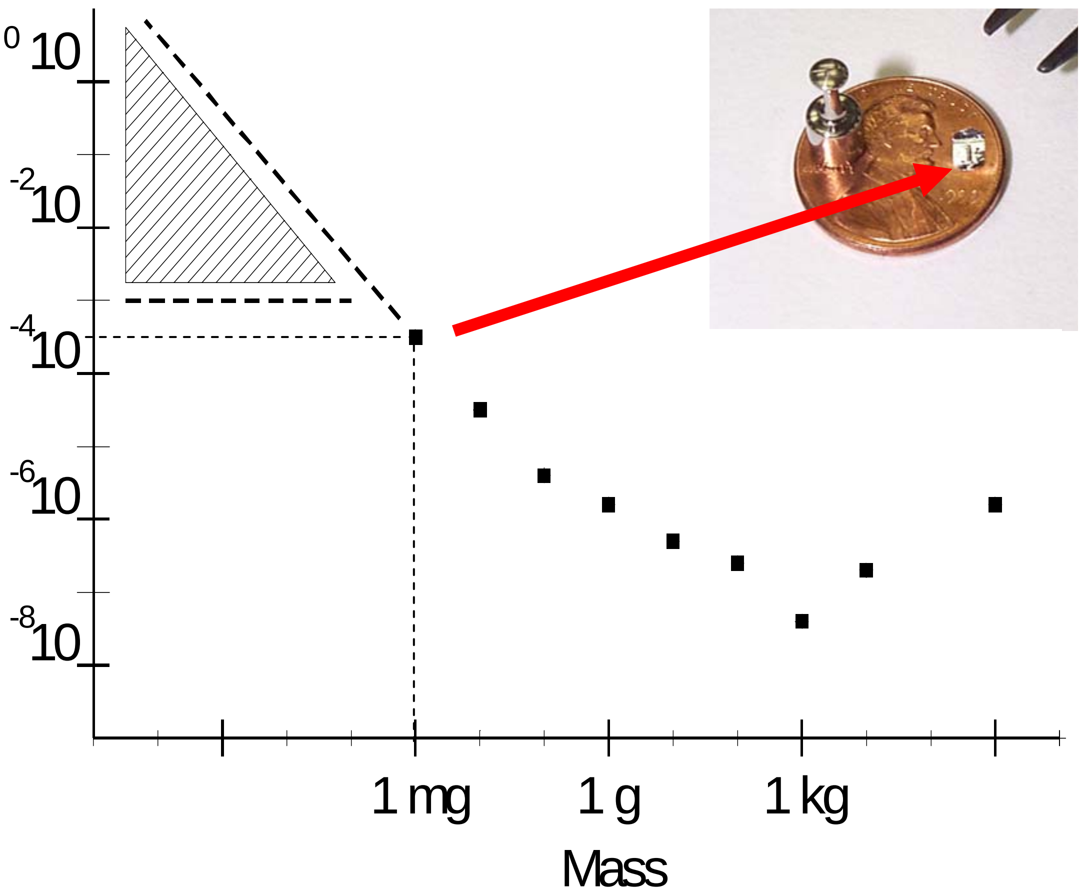
F=  X 

Mass artifacts link to Kg

Gravitational acceleration linked to sec and meter

Deadweights for small force

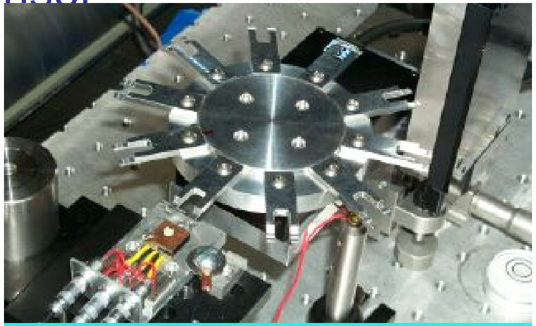
$$F = mg \sim 10^{-5}\ \mathrm{N}$$



- Deadweight force realization is simple, accurate, and reliable down to 10^{-5} N
- Accuracy: parts in 10^4
- Many sensors (even some "nano") can be calibrated by suspending wires of known mass
- **Limited by uncertainty in mass as we go smaller**

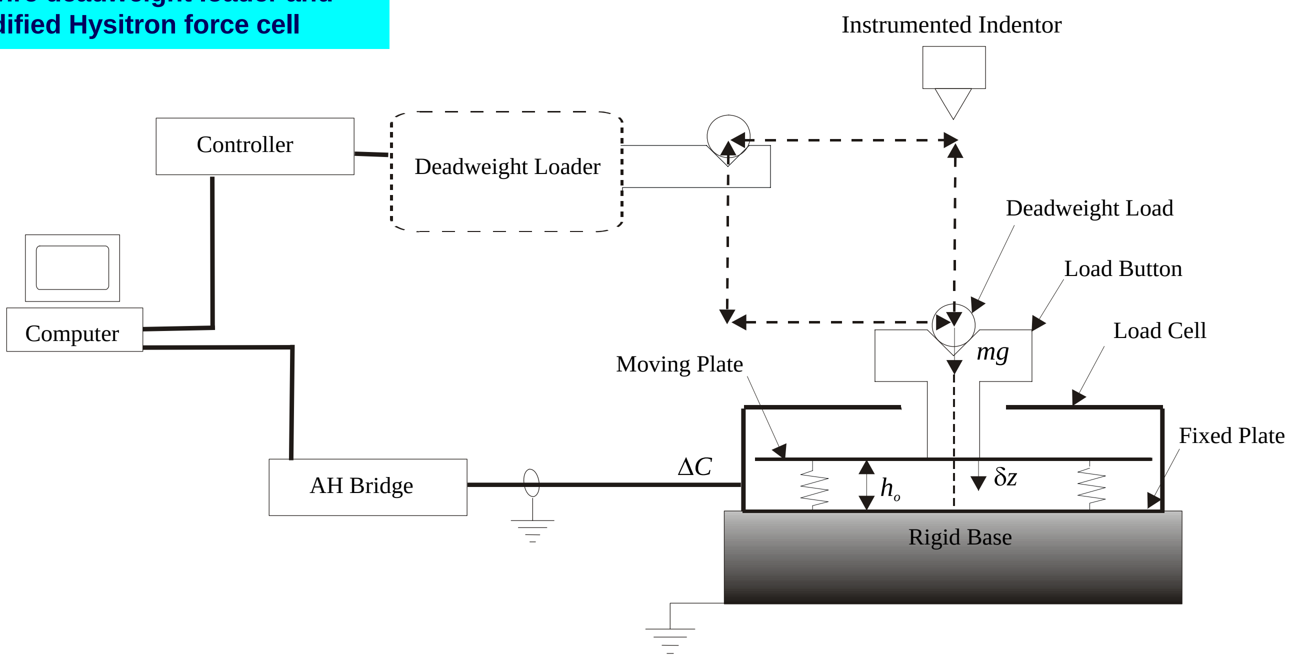
Calibrated indentation forces

- Deadweights used to calibrate standard force sensor



Traceable micro to millinewtons from NIST wire deadweight loader and modified Hysitron force cell

- Standard sensor used to calibrated indenter force
  - Discrepancies at low loads on order of few percent up to tens of percent depending on instrument



Smaller forces for AFM

- Realize forces below 10^{-5} N through the electrical units as in Watt and Volt balances

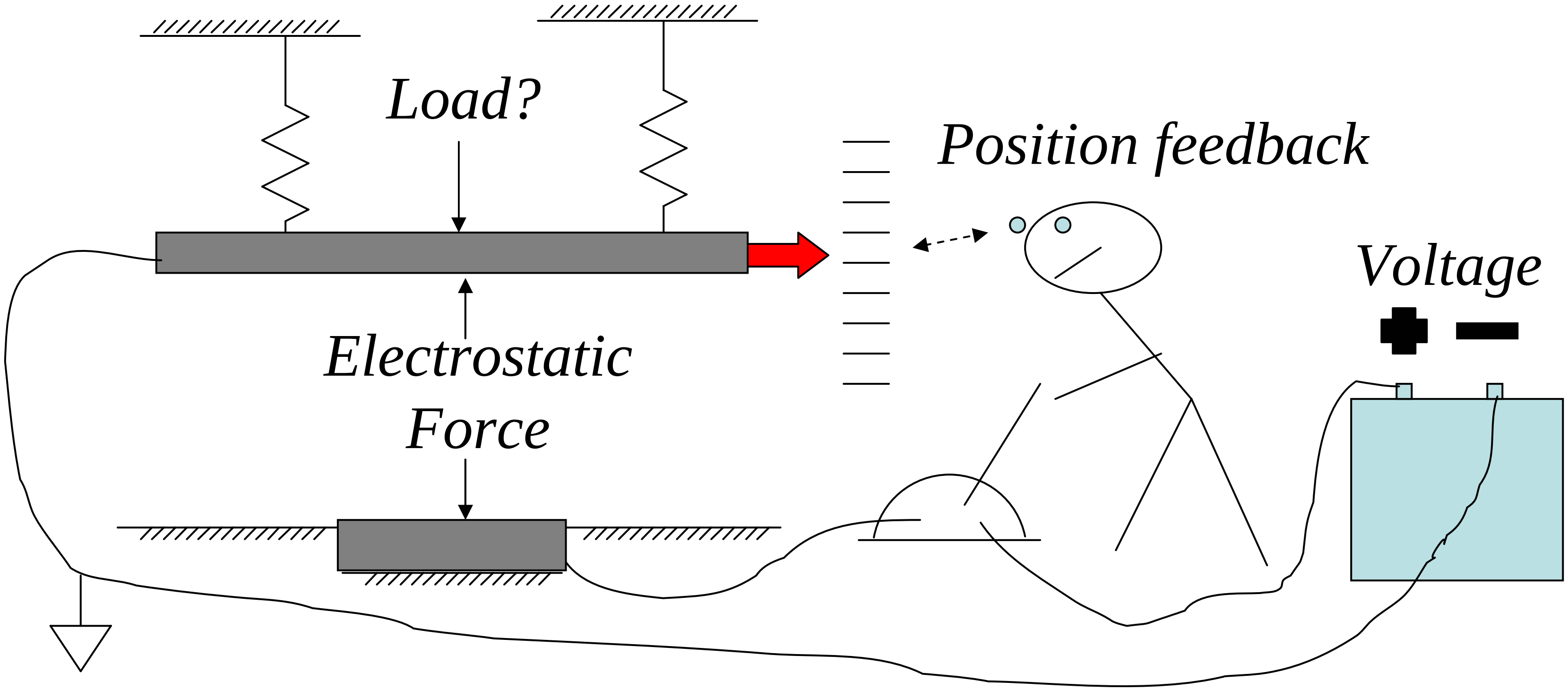
$$Electrostatic\ Force = \frac{1}{2}\frac{dC}{dz}\left(Voltage^2\right)$$

- Disseminate through a calibrated "primary standard" force balance known as an EFB
  - Range: 10 nN - 1 mN
  - Accuracy: parts in 10^4
  - Resolution: < 1 nN

What is the EFB?

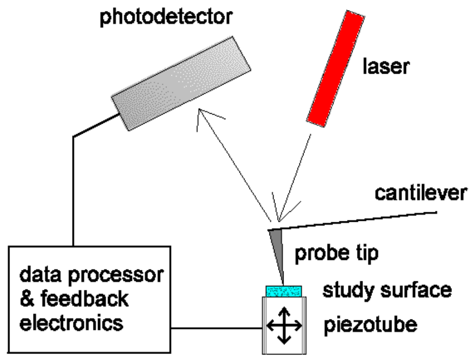
- Compares unknown loads to an SI force derived from length, voltage, and capacitance
  - Diminutive cousin of the electronic Kg experiment!

$$Electrostatic\ Force = \frac{1}{2}\frac{dC}{dz}\left(Voltage^2\right)$$



Calibrated AFM forces

- Most AFM systems use optical lever arms
- Calibrate spring constant (k) and optical lever sensitivity
- Accuracy is difficult to achieve...



Measuring Spring Constants

A word about precision:

Crappy

1-2 parts in 10 if you do everything right

Goals:
InvOLS (nm/Volt)
k (pN/nm)
Crappy or better

Asylum Research
Research tools for nanoscience and nanotechnology

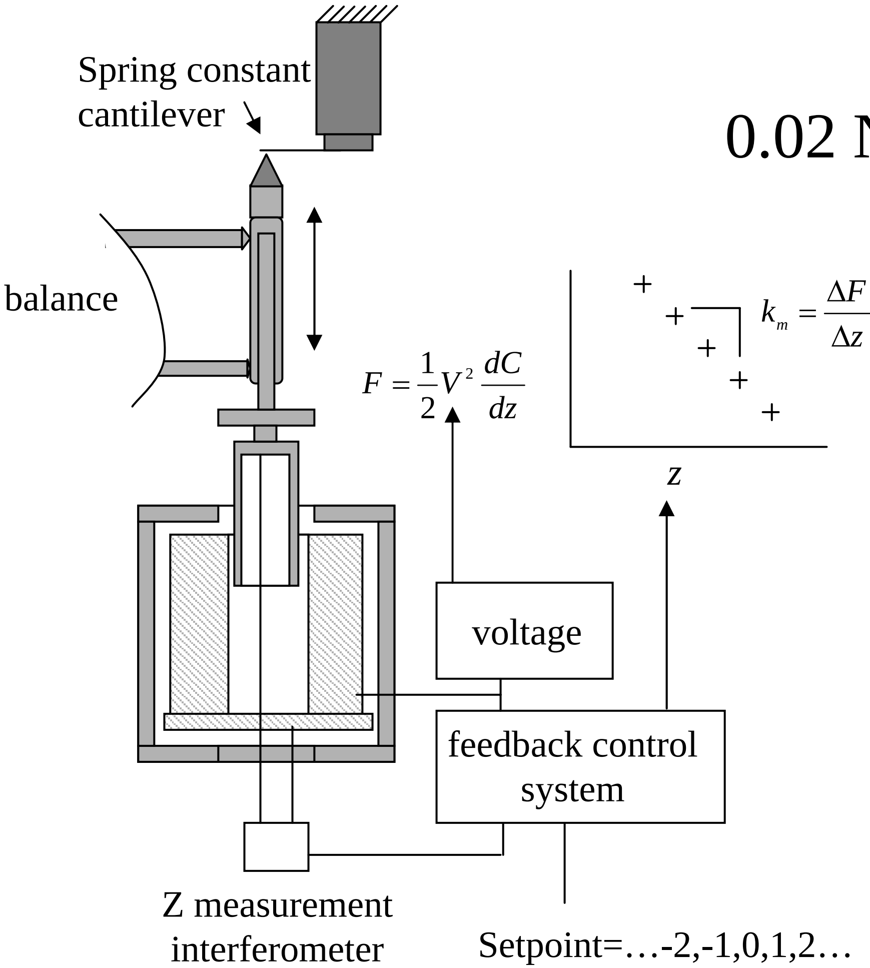
<u>Methods for determining k</u>
Thermal noise spectra
Added mass
Geometry and modulus
Nanoindentation

To date, no approaches have had traceability

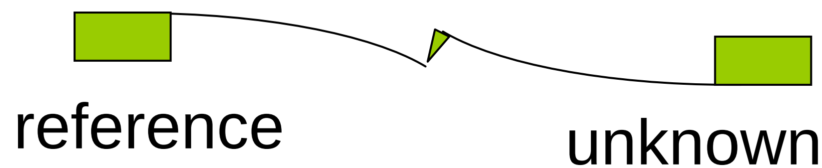
comparisons difficult

EFB as instrumented indenter

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



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

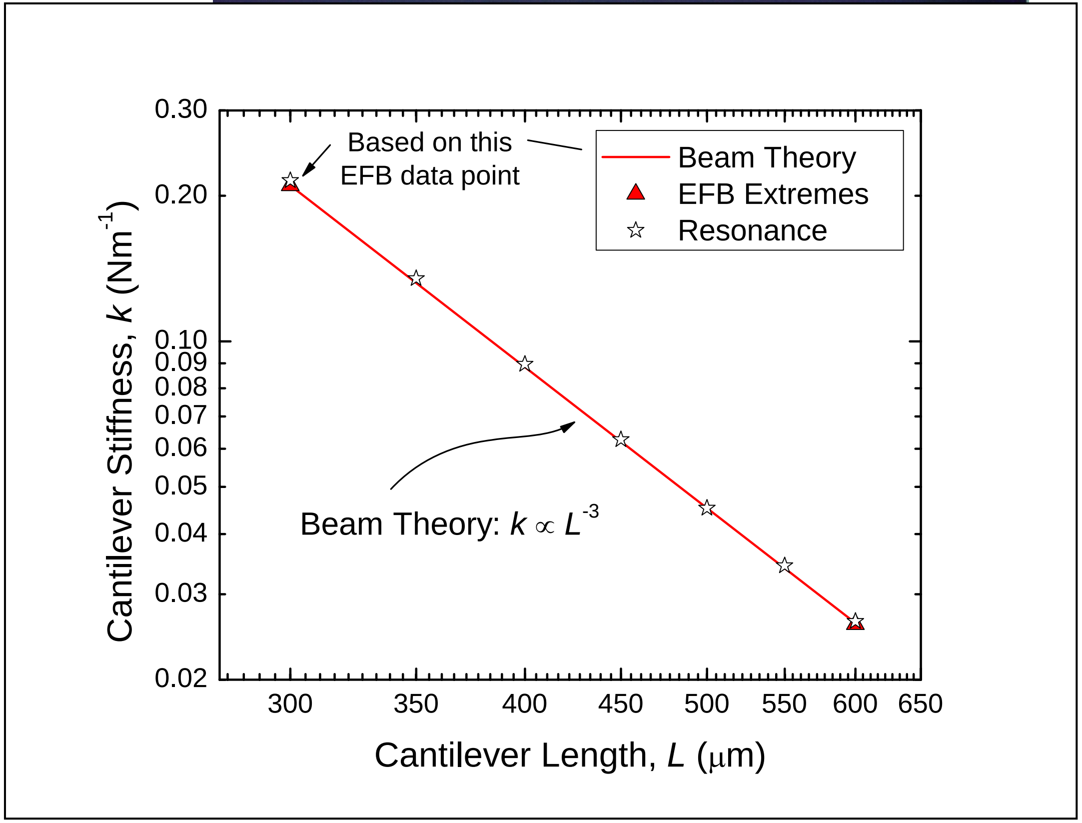


NIST reference cantilever array

- 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



Inter-comparison

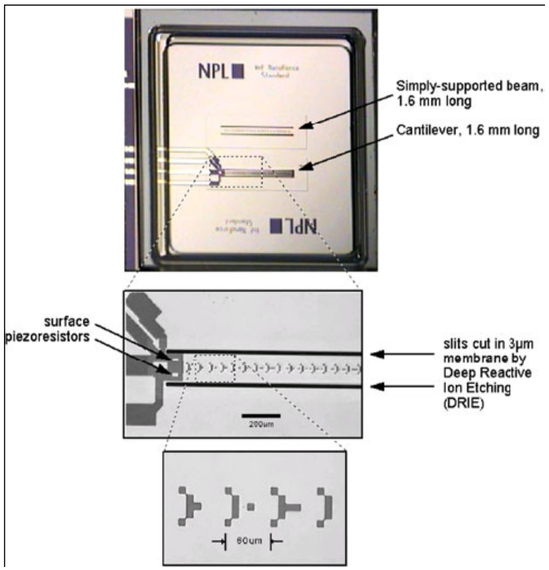


Table 2. Nominal spring constant at indicated positions along the length of the reference cantilever.

Nominal spring constant k_{ref} ($N\ m^{-1}$)	Distance from base of cantilever, L (μm)	Distance from end of cantilever, ($L_0 - L$) (μm)	Binary code, l	Code, l
26.9	92	1508	00001	1
13.8	152	1448	00010	2
7.28	212	1388	00011	3
4.15	272	1328	00100	4
2.54	332	1268	00101	5
1.66	[illegible]	1208	00110	6
1.14	452	[illegible]	00111	7
0.814	512	1088	[illegible]	8
0.601	572	1028	01001	[illegible]

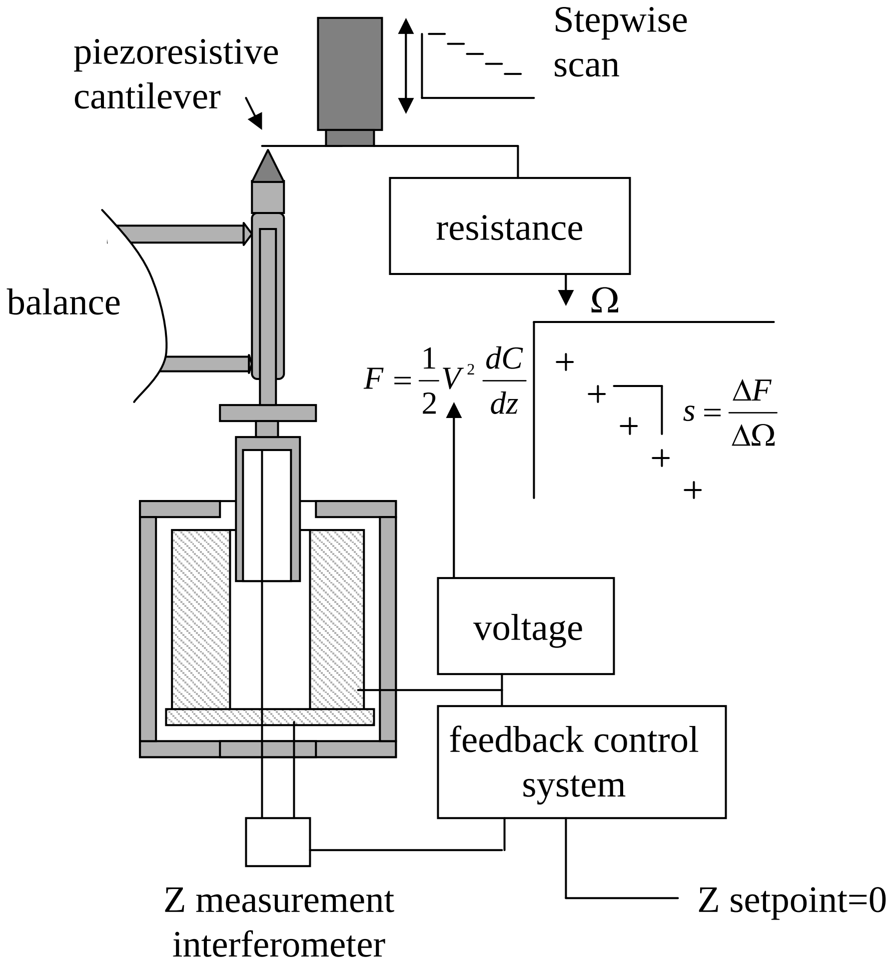
NIST value=18.6 N/m

NIST value=1.44 N/m

- **CMARS has proven well matched to EFB calibration**
  - **Size is convenient**
  - **Marks are clear and easy to hit**
  - **Access is a little difficult because of package**
- **Discrepancies exist between NPL published values and NIST EFB values**
  - **Undercut at cantilever base may be source of discrepancy**

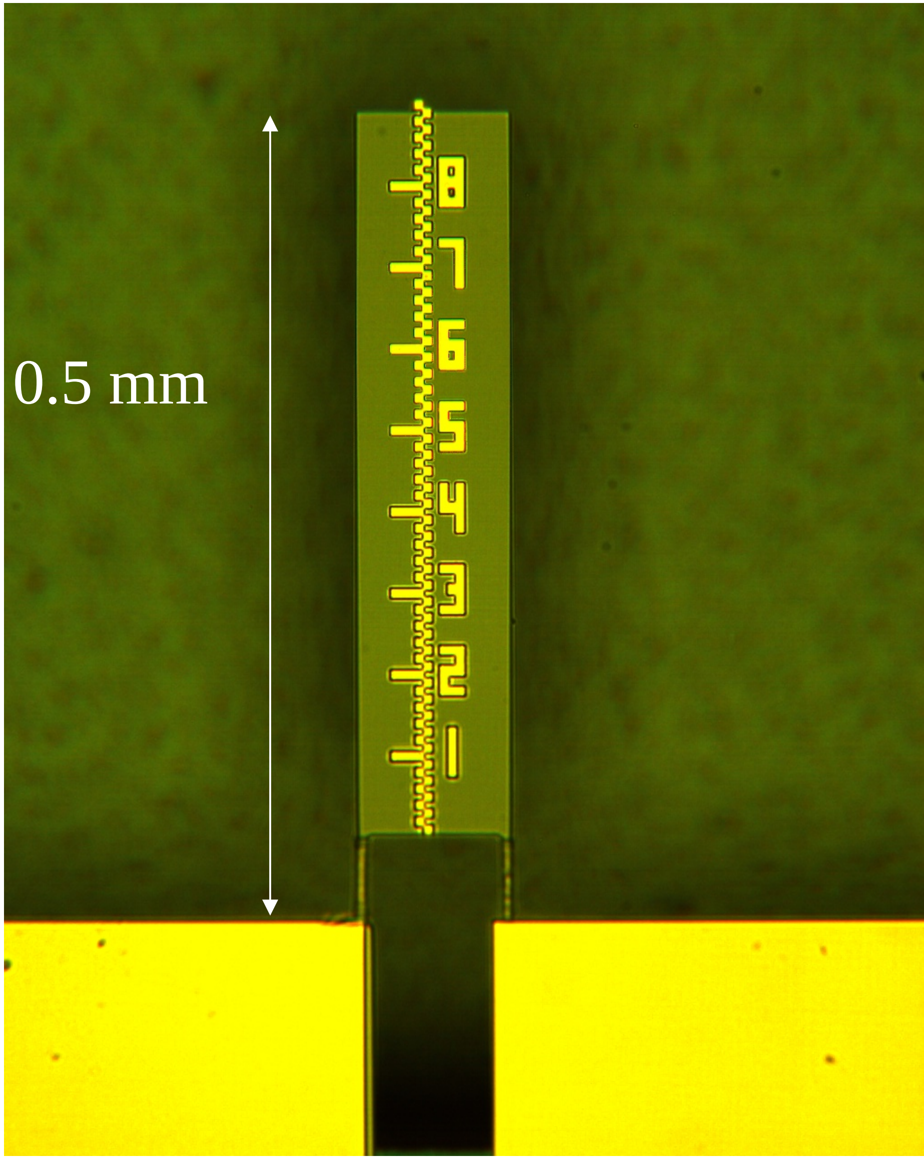
EFB as force calibrator

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

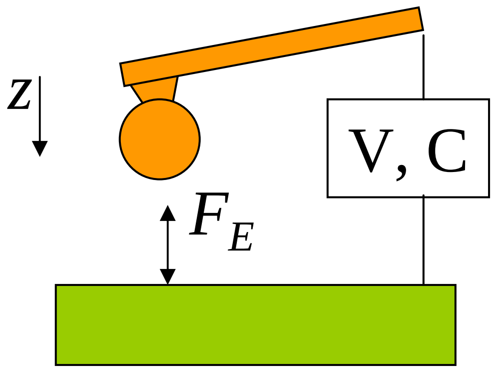


Force sensor calibration

- 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
- New NIST sensor is being developed with piezoresistive strain element for force and or stiffness calibration (0.2 N/m to 12 N/m)



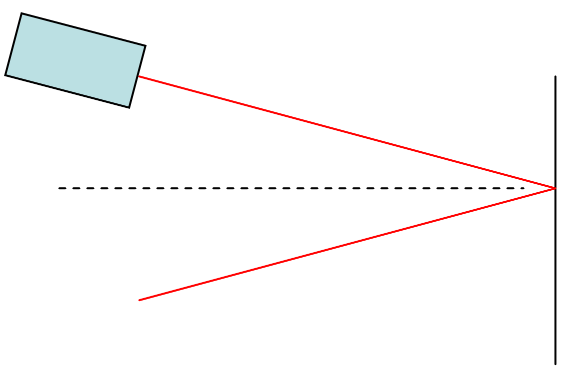
Future SI traceable picoforce realizations



$$F_E = \frac{1}{2}\frac{dC}{dz}\left(Voltage^2\right)$$

- Electrostatic force between a probe and surface (0.5 pN/mV assuming pF/mm gradient)
  - Need modestly accurate voltage, capacitance, and displacement metrology
  - IF the geometry and field is well defined

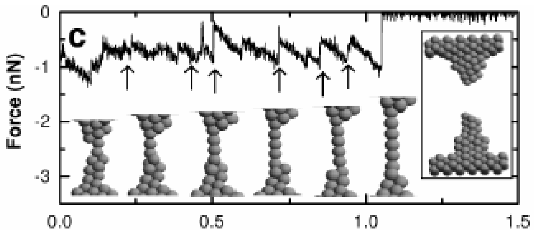
Laser with optical power P_0



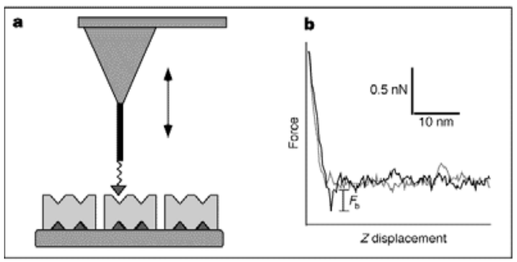
$$F_p = \frac{P_0}{c}\left(1+R\right)\cos(\phi)$$

- Photon momentum (6 pN/mW)
  - Need only modestly accurate optical power and reflectance metrology
  - IF heating and boiling off of molecules doesn't completely swamp the effect!

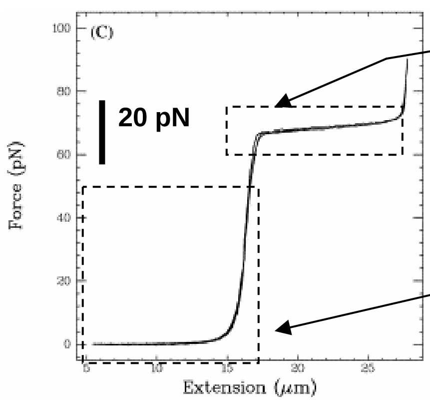
Prototype intrinsic forces from nature



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)

1-2 nN

Break a single atomic bond

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

100-200 pN

Rupture a binding site

- Rate dependent

35 & 65 pN

Change DNA structure

0.2-65 pN

Stretch DNA elastically

AFM

OT

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