National Institute For Nanotechnology

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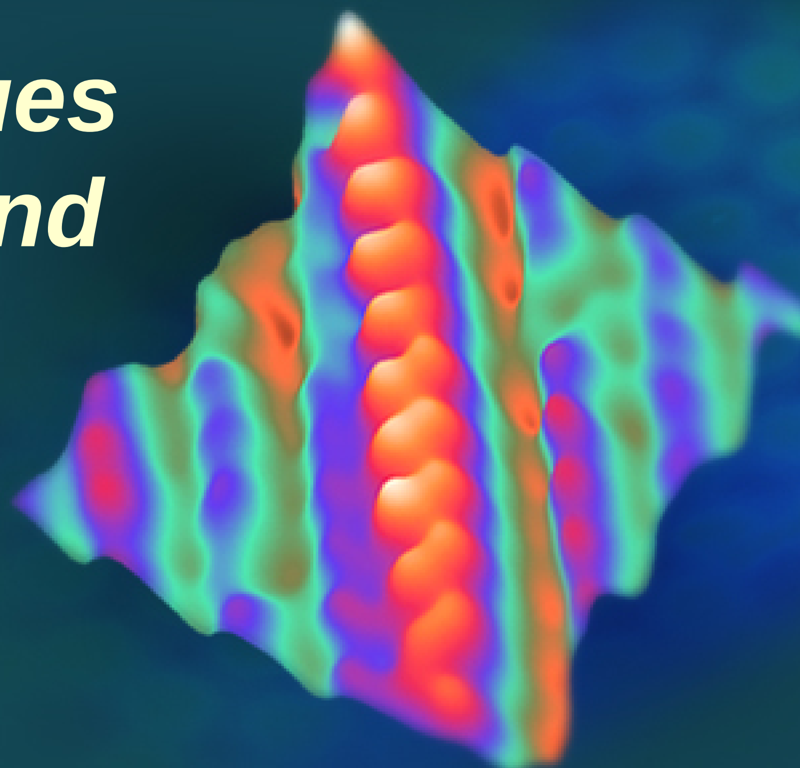
UNIVERSITY OF ALBERTA

Calibration and Standardization Issues in Scanning Force and Interfacial Force Microscopy

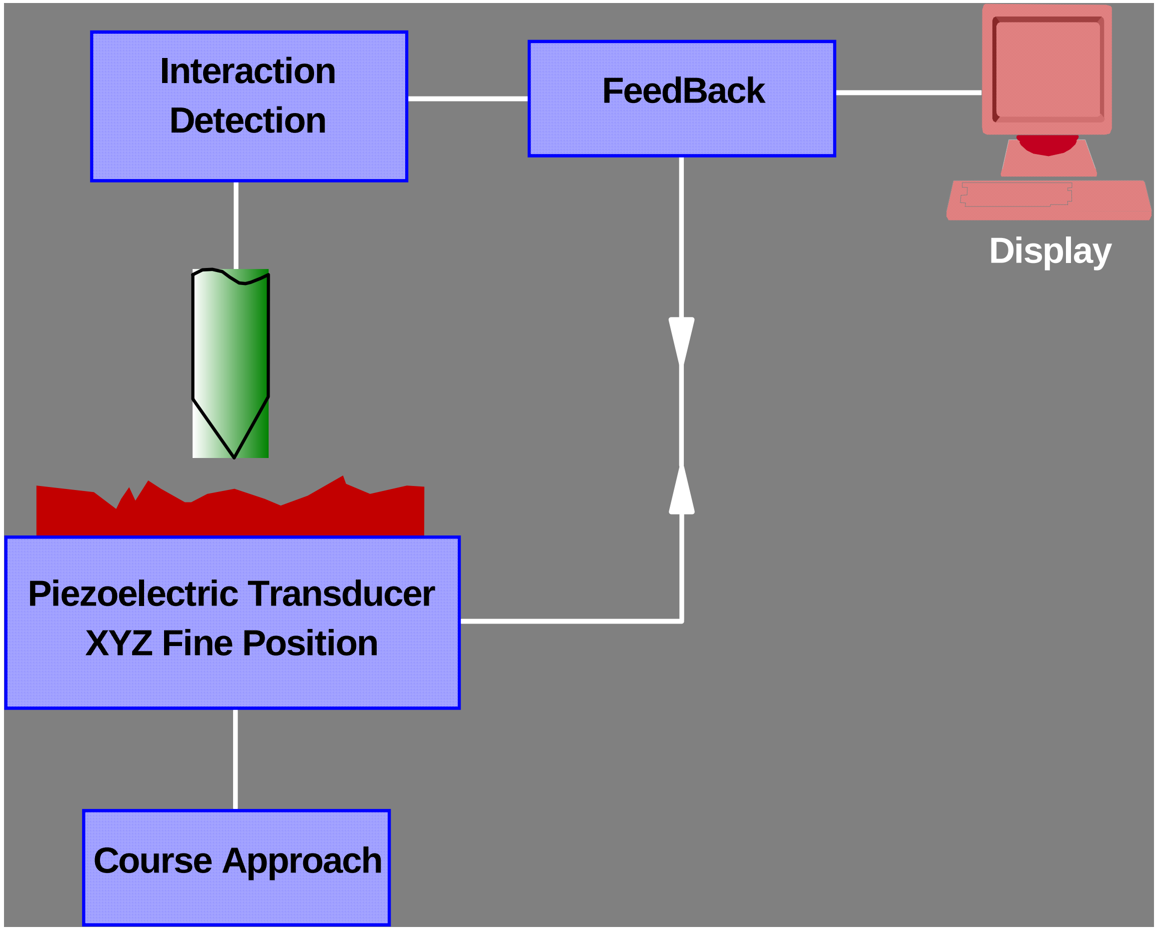


David Munoz-Paniagua
Mark T. McDermott

Alberta
GOVERNMENT OF ALBERTA

Canada

Generalized view of a scanning probe microscope (SPM)



Quantifiable Parameters in SPM

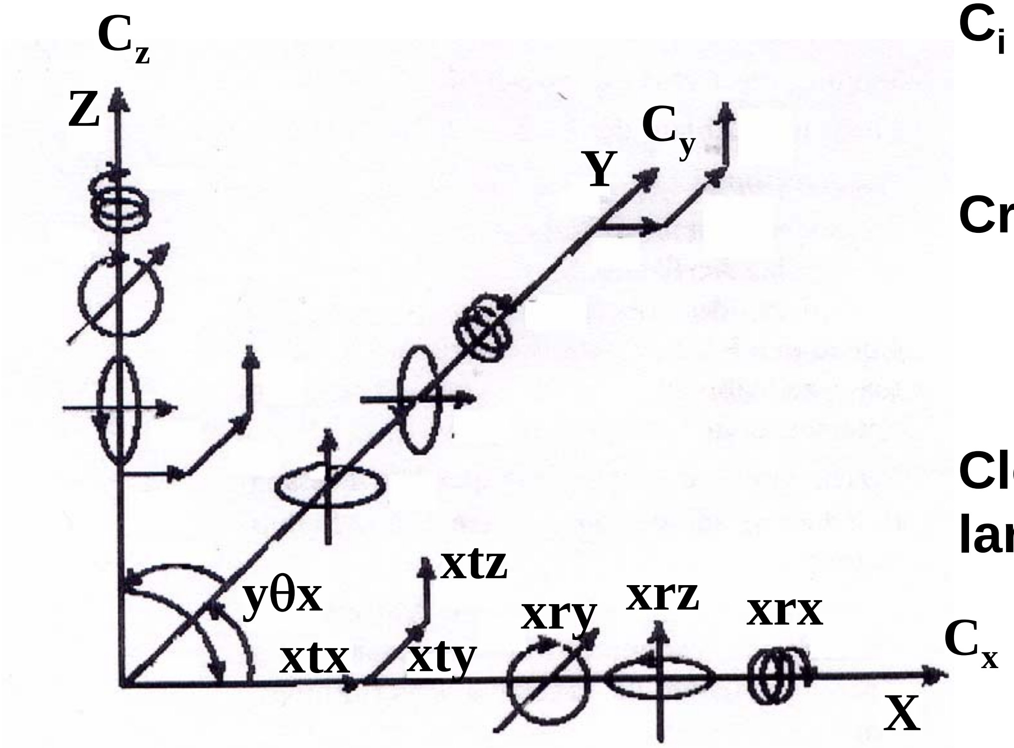
Instrumental

1. Motion in X, Y, Z
2. Tip Shape
3. Interaction parameter (current, potential, force, etc.)

Data

1. Roughness
2. Indent shape/volume

Scanner parameters that need to be characterized



C_i – piezoelectric response [nm/V]

Crosstalk - rotational (r), angular (θ) and translational (t).

Closed loop scanners correct largely for these.

Verein Deutscher Ingenieure, VDI/VDE 2656, Draft 1, Dec. 2006

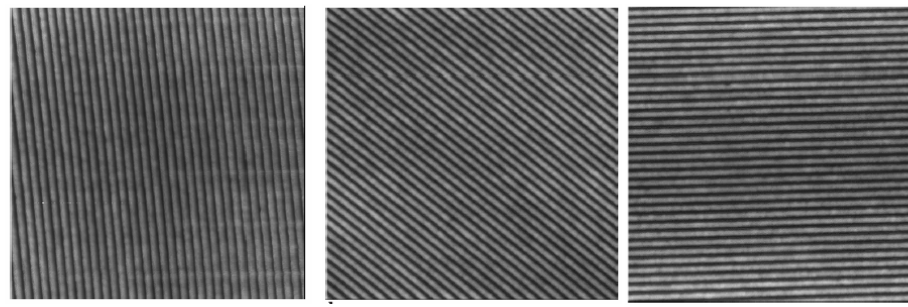
X,Y Calibration Standards

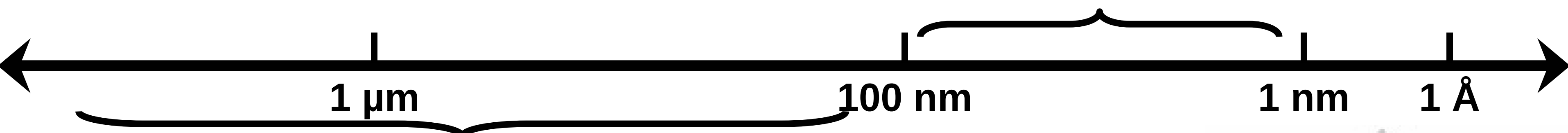
1D grating: 3 images (MBE structures, 30 nm pitch)

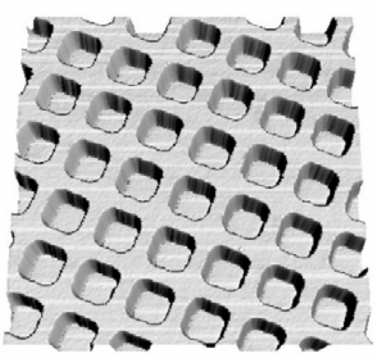
J.F. Jørgensen, C.P. Jensen

Danish Institute of Fundamental Metrology,

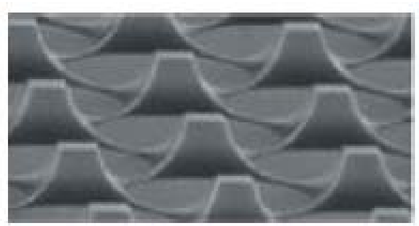
Appl. Phys. A 66, S847–S852 (1998)



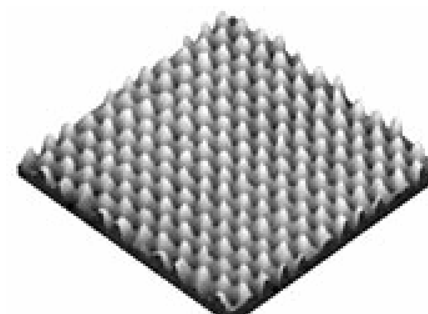




VLSI (NIST)



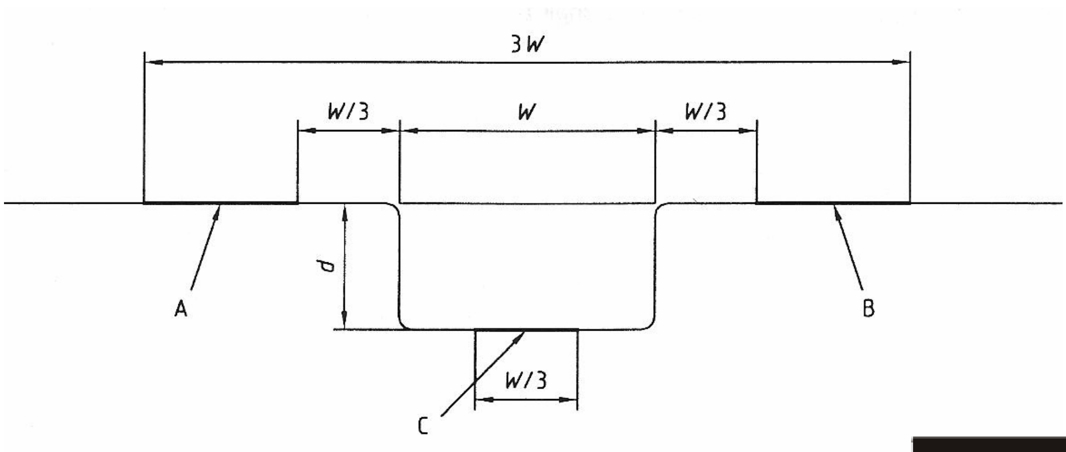
Nanoprobe (PTB)

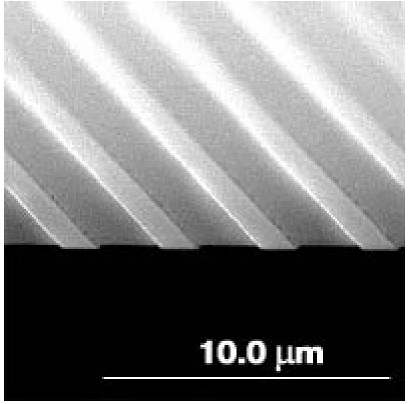


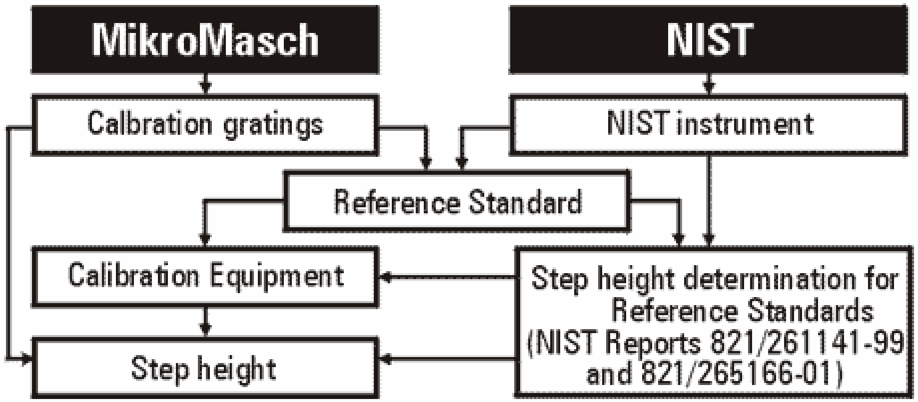
HOPG
Si<111>7X7

Example: Z Calibration Standards

From ISO 5436-1:2000 – Profile measurement

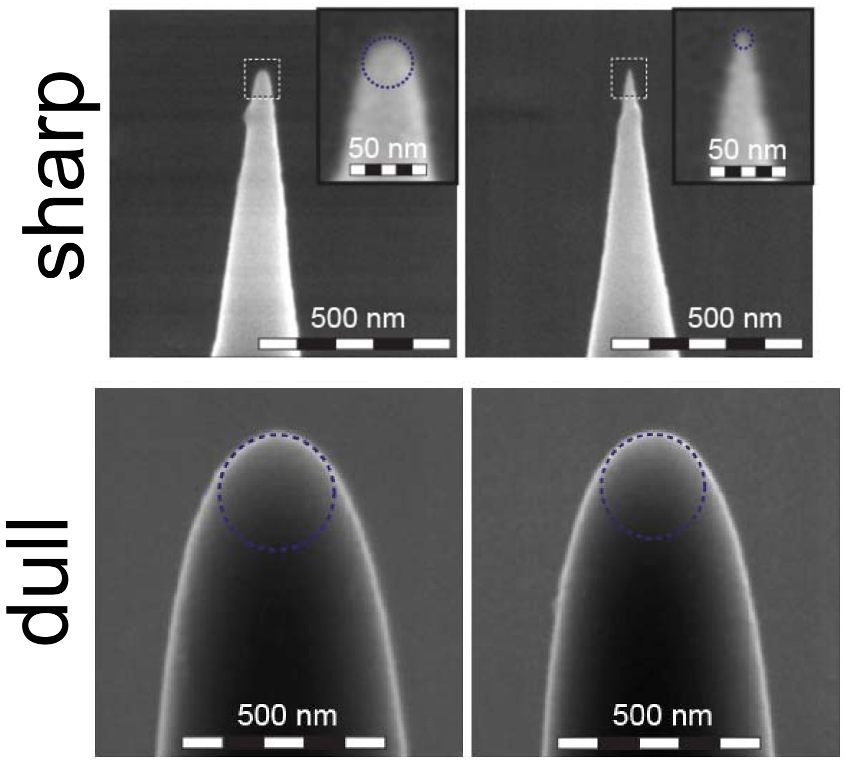


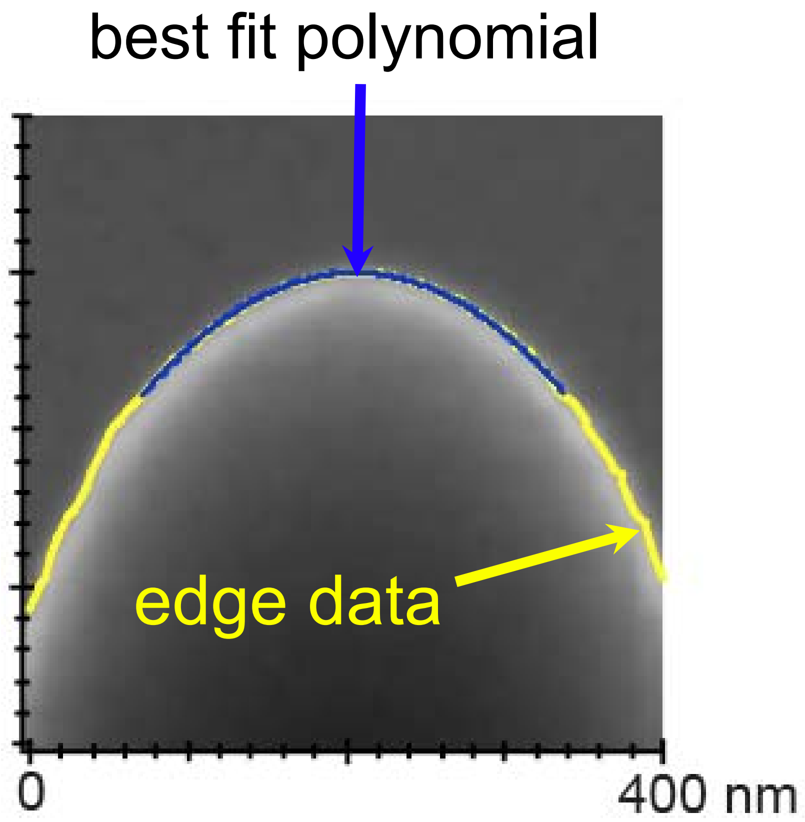




Example: AFM Tip Shape

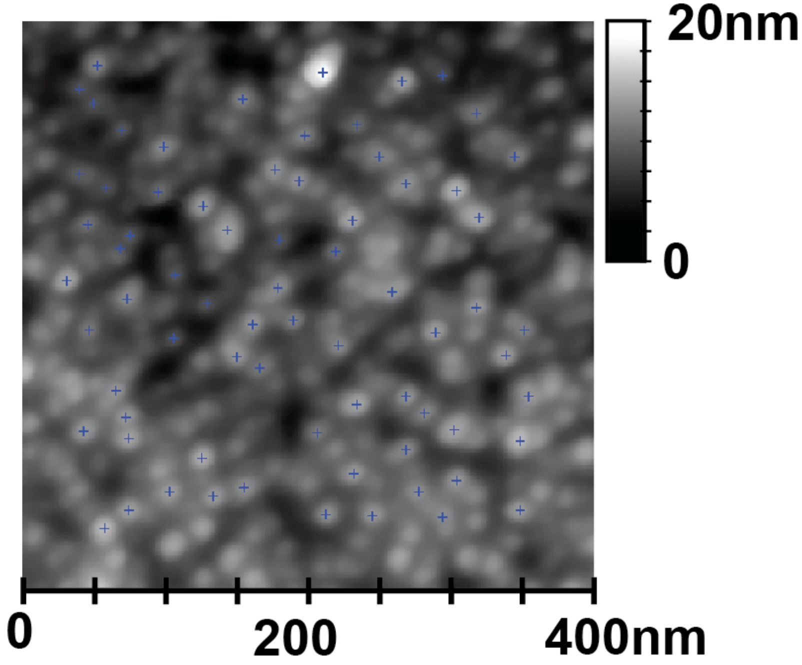
SEM imaging for tip radius estimation



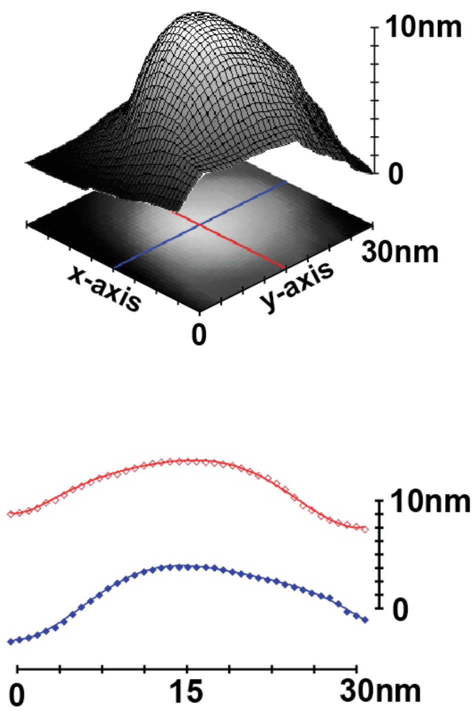


Blind Reconstruction

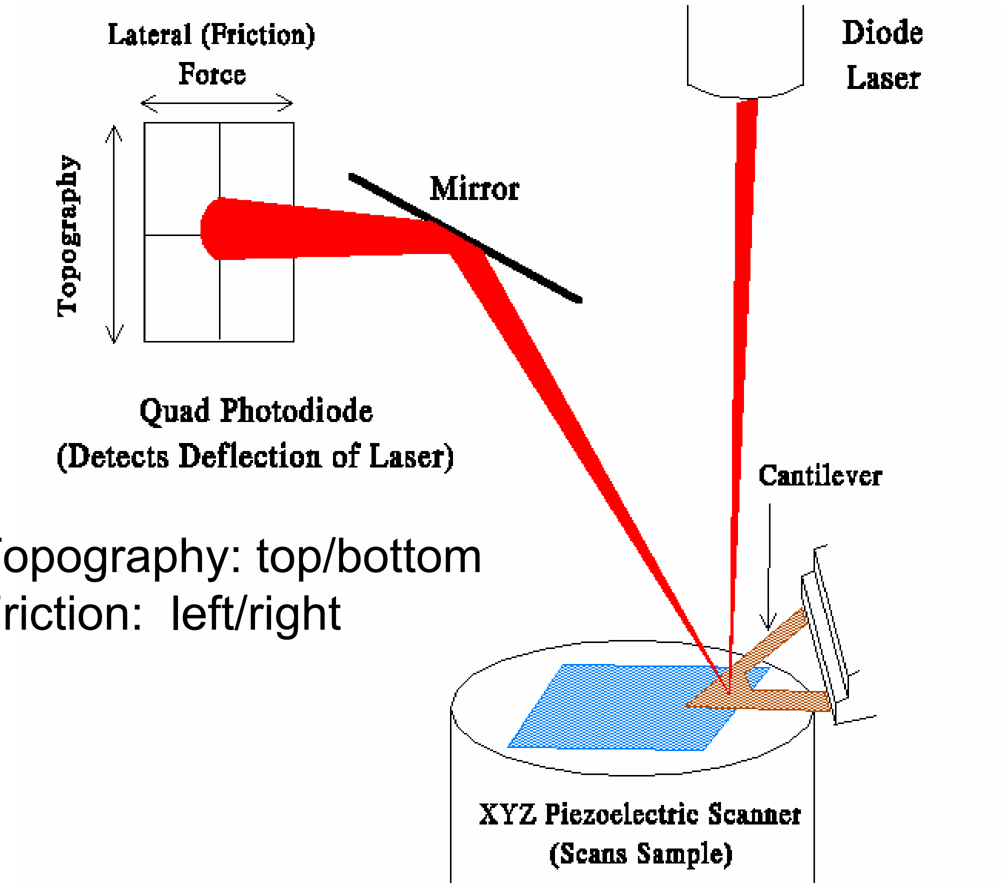
Niobium sample
MATLAB or SPIP for analysis

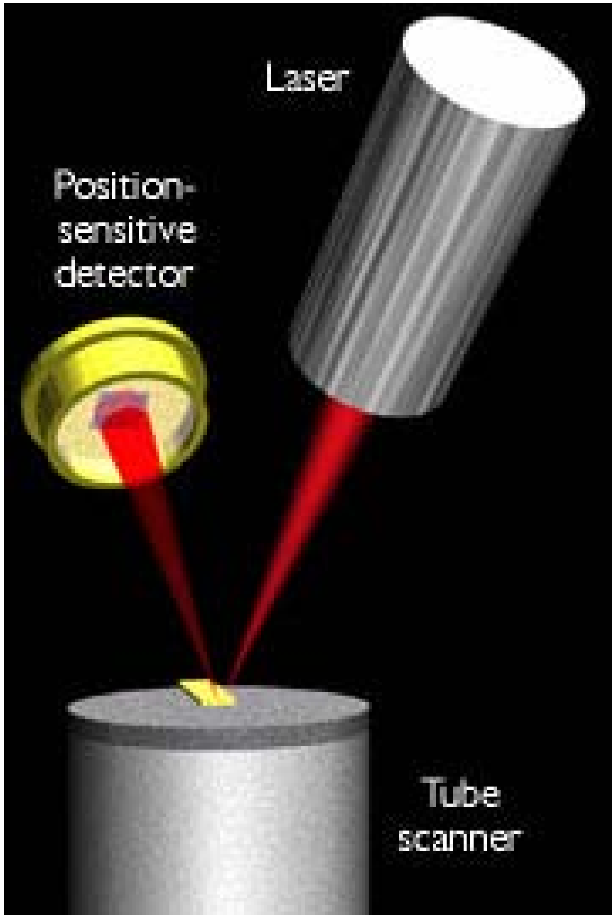


tip image



Atomic (AFM) or Scanning (SFM) Force Microscopy





Topography: top/bottom
Friction: left/right

What information can SFMs provide?

Topography – cantilever bending vs. XY

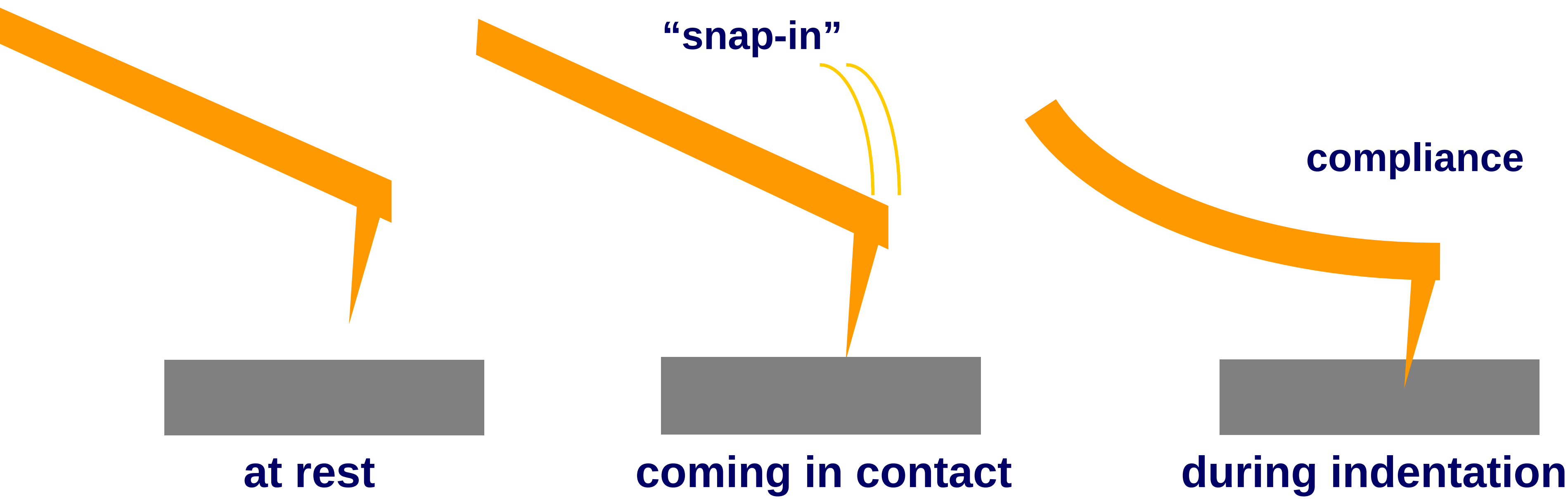
Adhesion – cantilever bending

Friction – cantilever twisting

Nanomechanical properties (e.g., elastic modulus) – nanoindentation

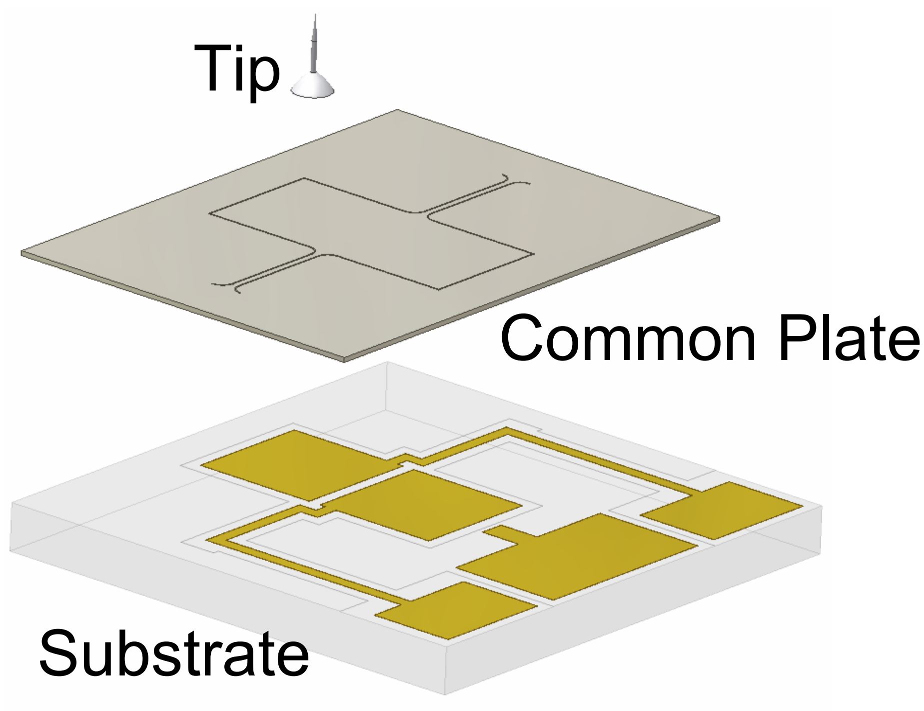
Interfacial Force Microscopy (IFM)

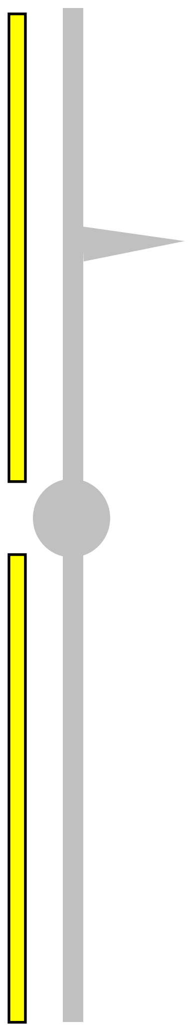
Typical cantilever behaviour



AFM is excellent for imaging but has severe limitations as an indenting tool

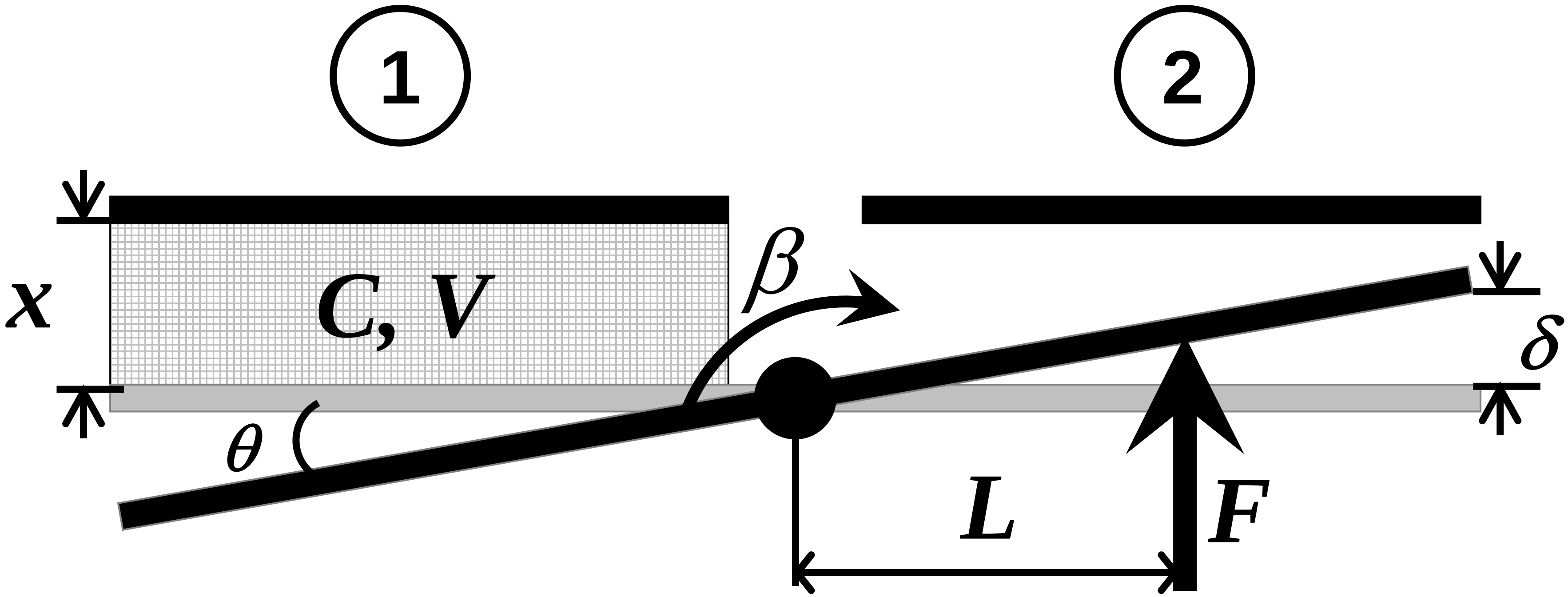
IFM Force Sensor

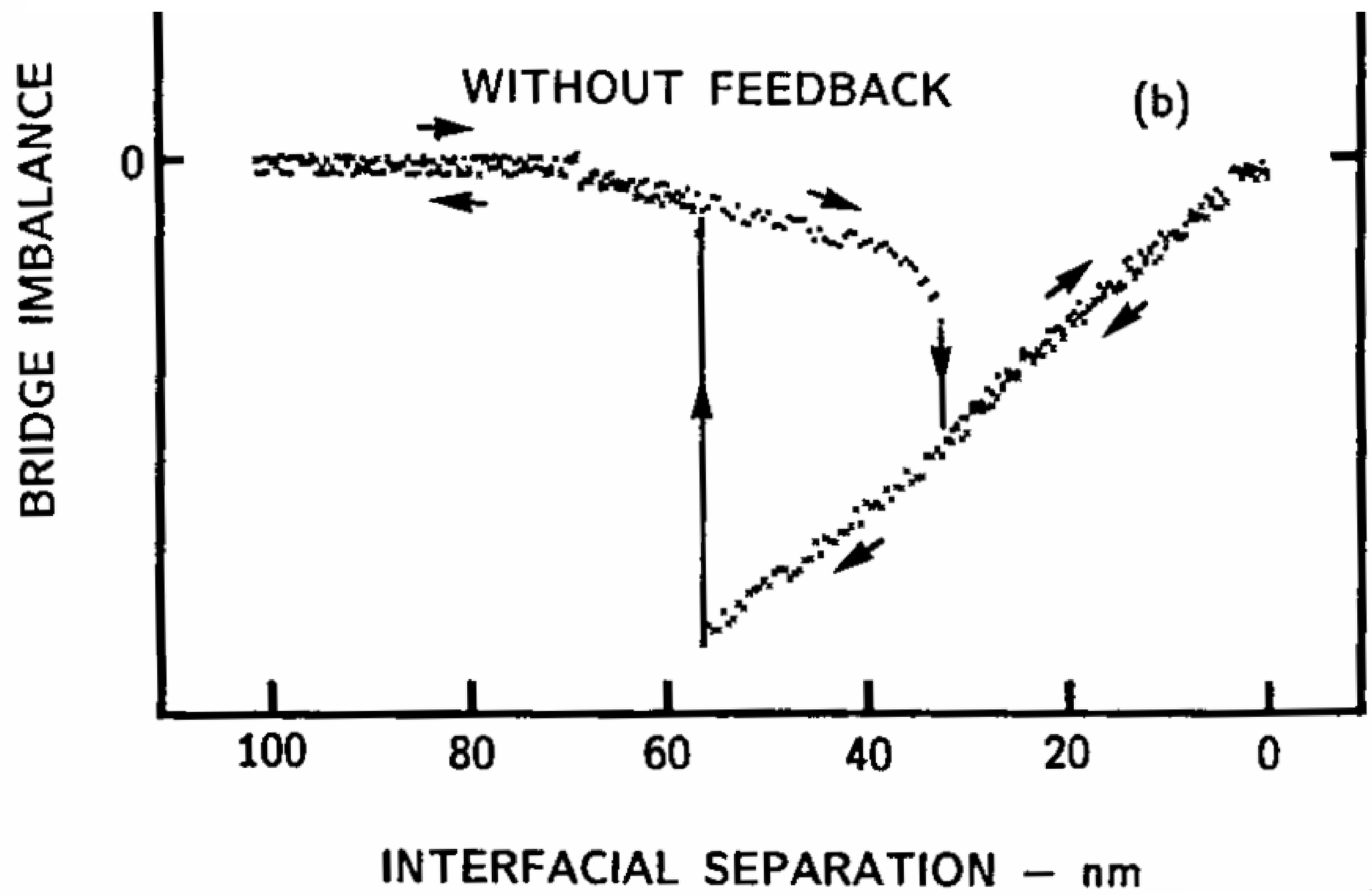




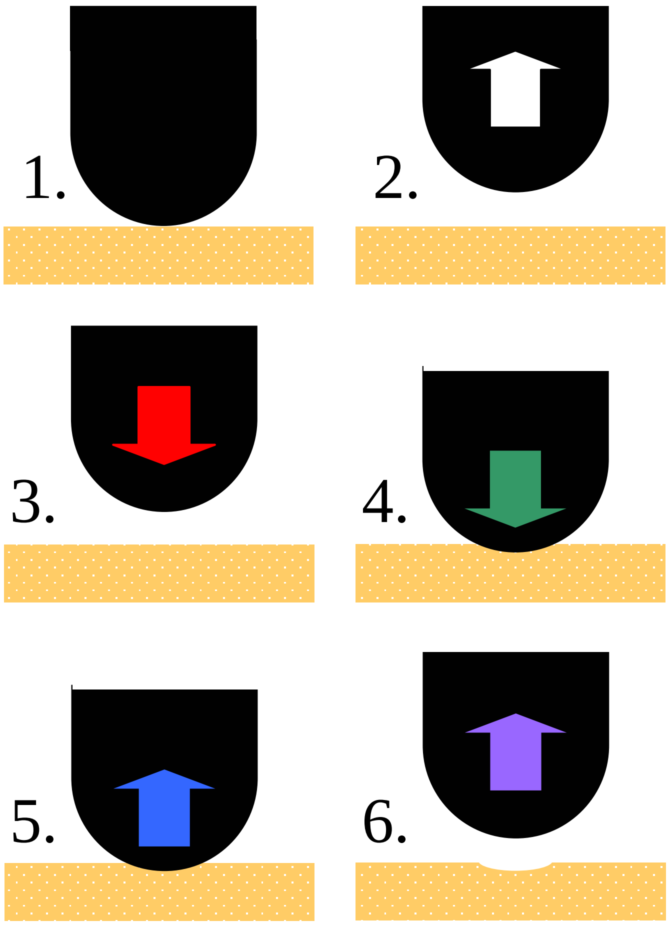
~3-5 nN force sensitivity

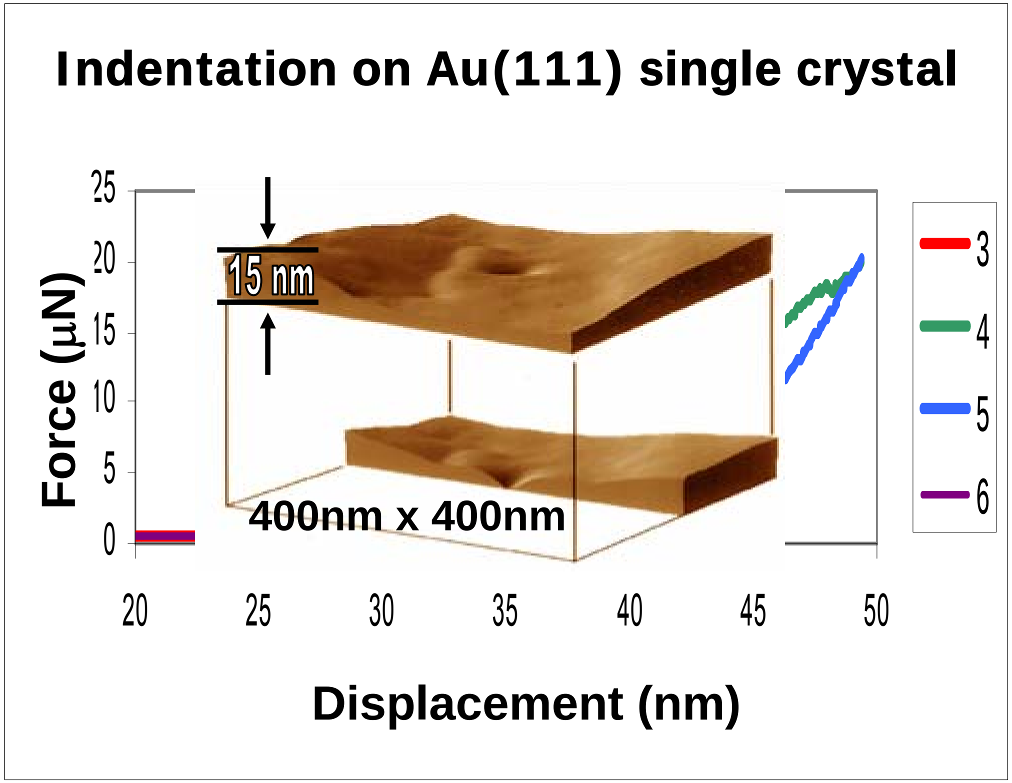
Torsion Balance



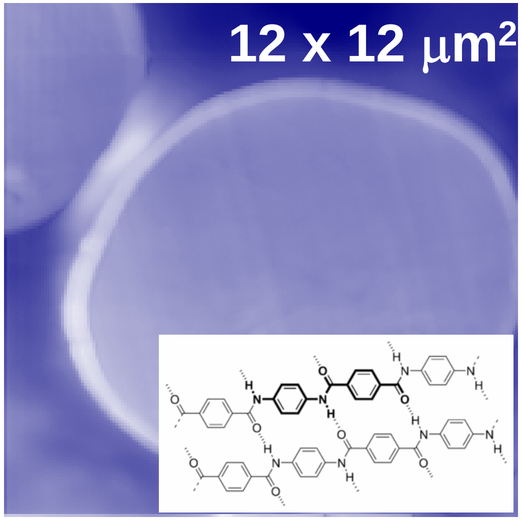

WITHOUT FEEDBACK
(b)
BRIDGE IMBALANCE
0
100
80
60
40
20
0
INTERFACIAL SEPARATION — nm

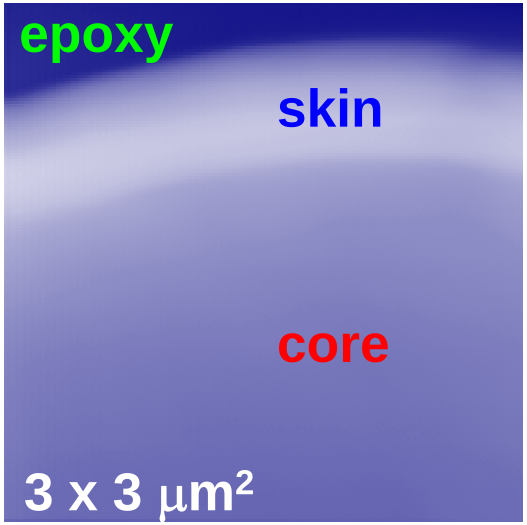
Indentation Experiments

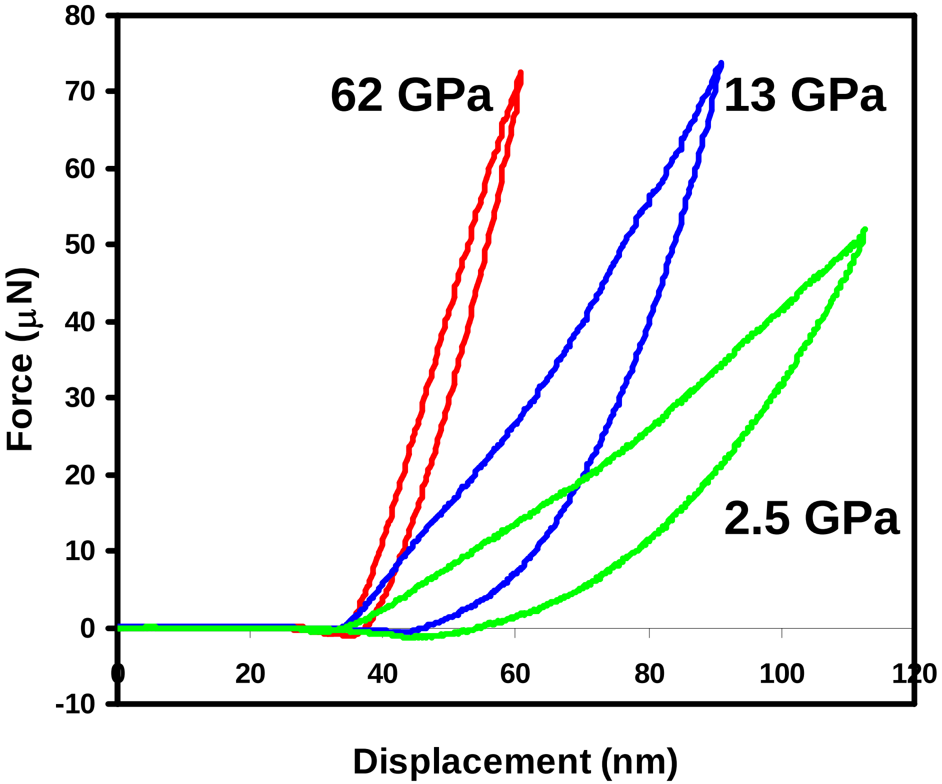




IFM Nano-indentation on Kevlar





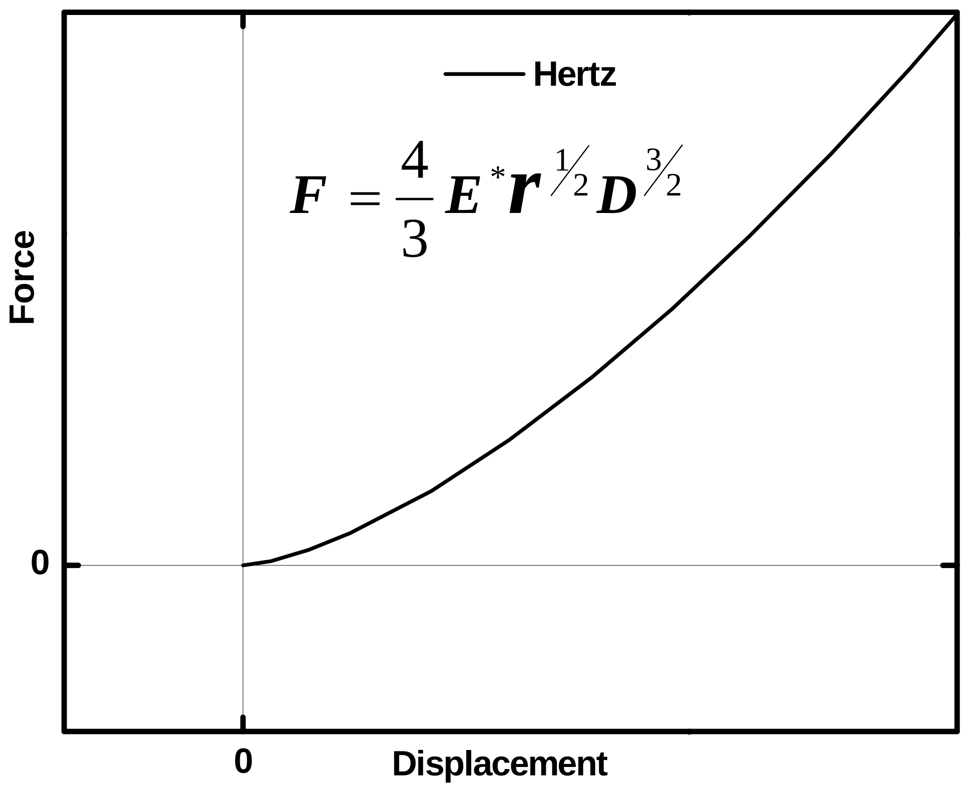


Graham, J.F., McCague, C., Warren, O.L., Norton, P.R., *Polymer* 41, 4761 (2001)

What needs to be calibrated for nanoindentation?

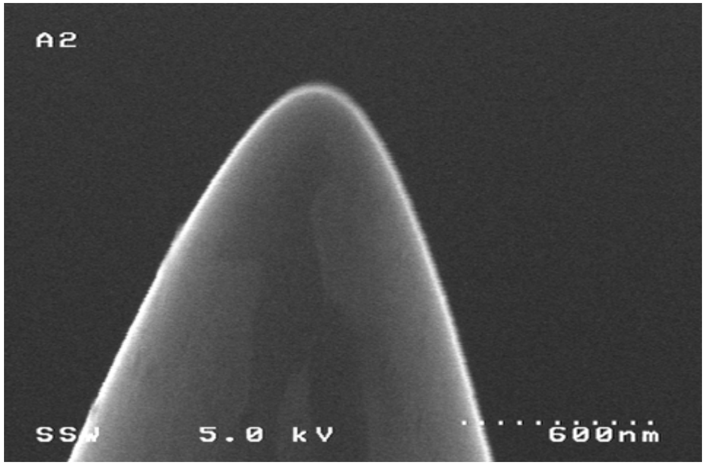
1. Tip shape/size.
2. Force
3. XY

IFM tip shape/size



SEM Tip Image

Fit shape

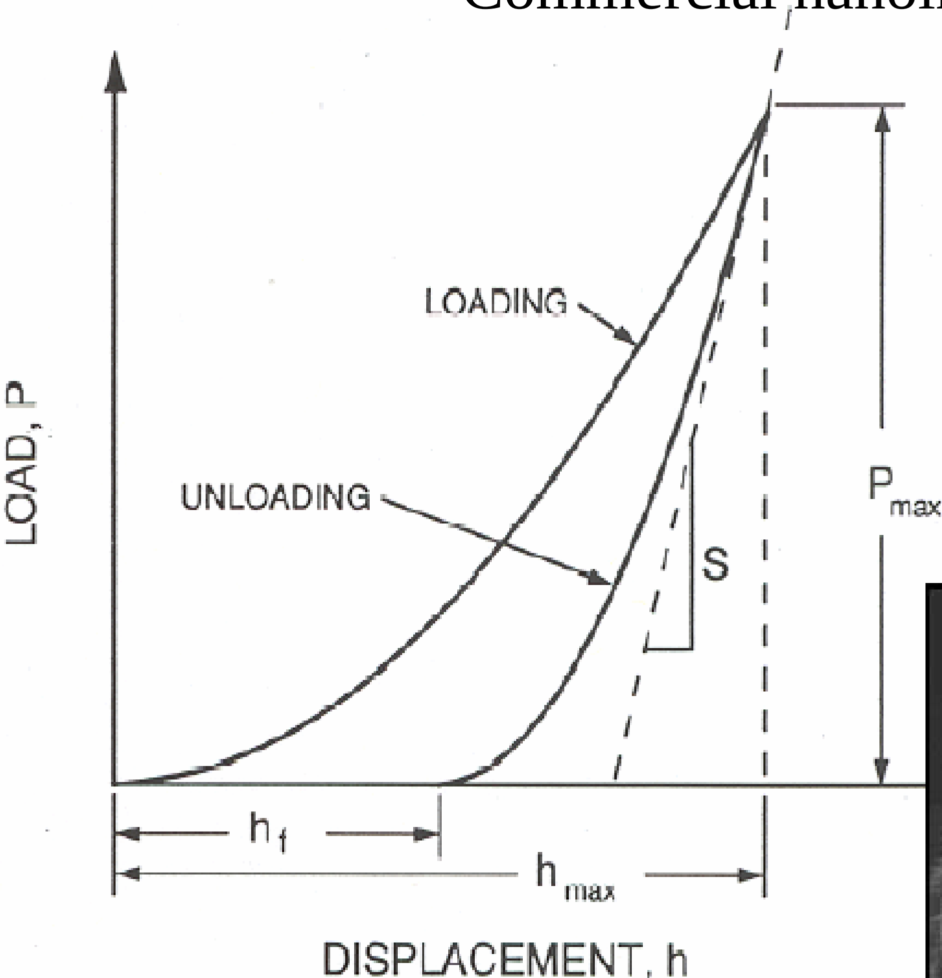


Etched W tips E ≈ 400 GPa

Parabolas.   R ≈ 100 nm

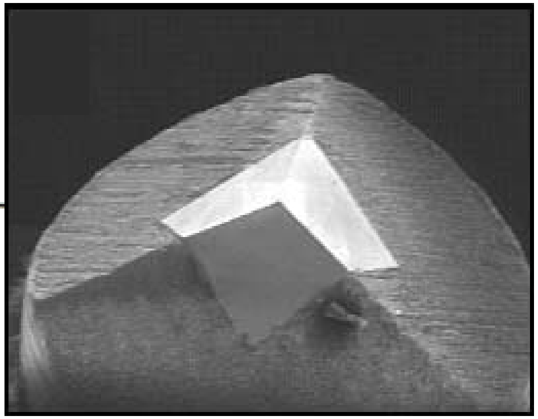
Can be: passivated, derivatized

Commercial nanoindenter - Hysitron



$$S = \frac{dP}{dh} = 2\sqrt{\frac{A}{\pi}}E^*$$

$$E^* = \left[\frac{1-\nu_s^2}{E_s} - \frac{1-\nu_i^2}{E_i}\right]^{-1}$$



Oliver, W. C.; Pharr, G. M. ***Journal of Materials Research*** **1992, *7*, 1564.**

Standard Method

Indent a standard with known Young's modulus and hardness at varying loads and estimate the cross sectional area from:

$$A = \pi \left[S \frac{1}{2\beta E^*} \right]^2$$

$$C_t = \frac{1}{S} + C_i = \frac{1}{2\beta E^*} \sqrt{\frac{\pi}{A}} + C_i$$

The instrument compliance is assumed to linearly combine with the tip-sample compliance

Problem!

Parameters become interdependent which forces iteration to ensure self-consistency

New Method (D. Munoz-Paniagua)

Acquire high resolution SPM images of the tip.

Statistically process them to determine the tip shape, radius of curvature and the transition point between a hemispherical cap and Berkovitch shape.

Use this independently determined Shape Area Function to determine the instrumental compliance.

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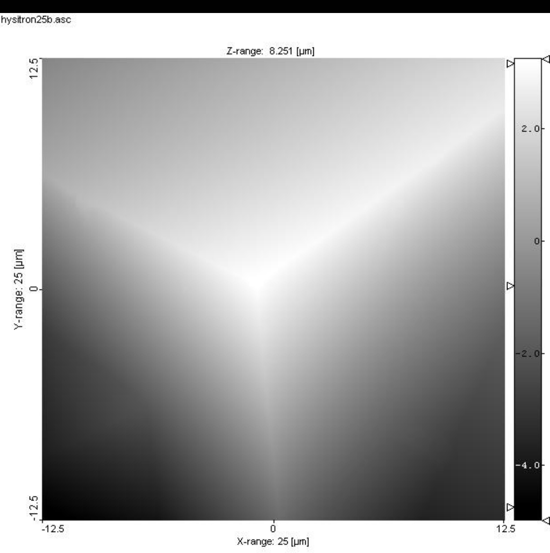
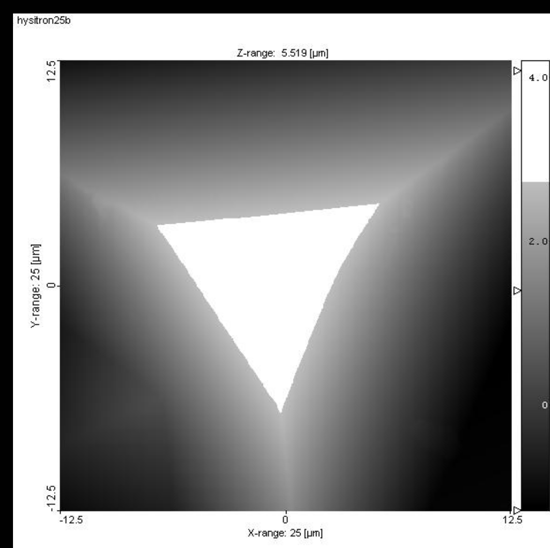
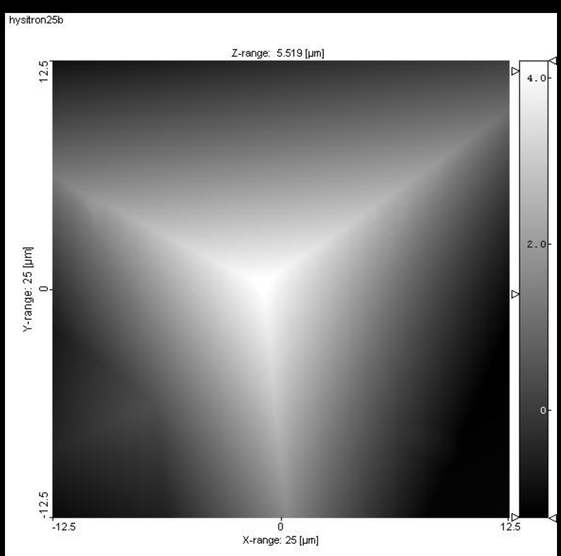
A set of different sized tip scans at maximum resolution was generated

Multiple scan with varying scan directions to check for artefacts

Saturate height scale and tilt image to ensure on-axis view



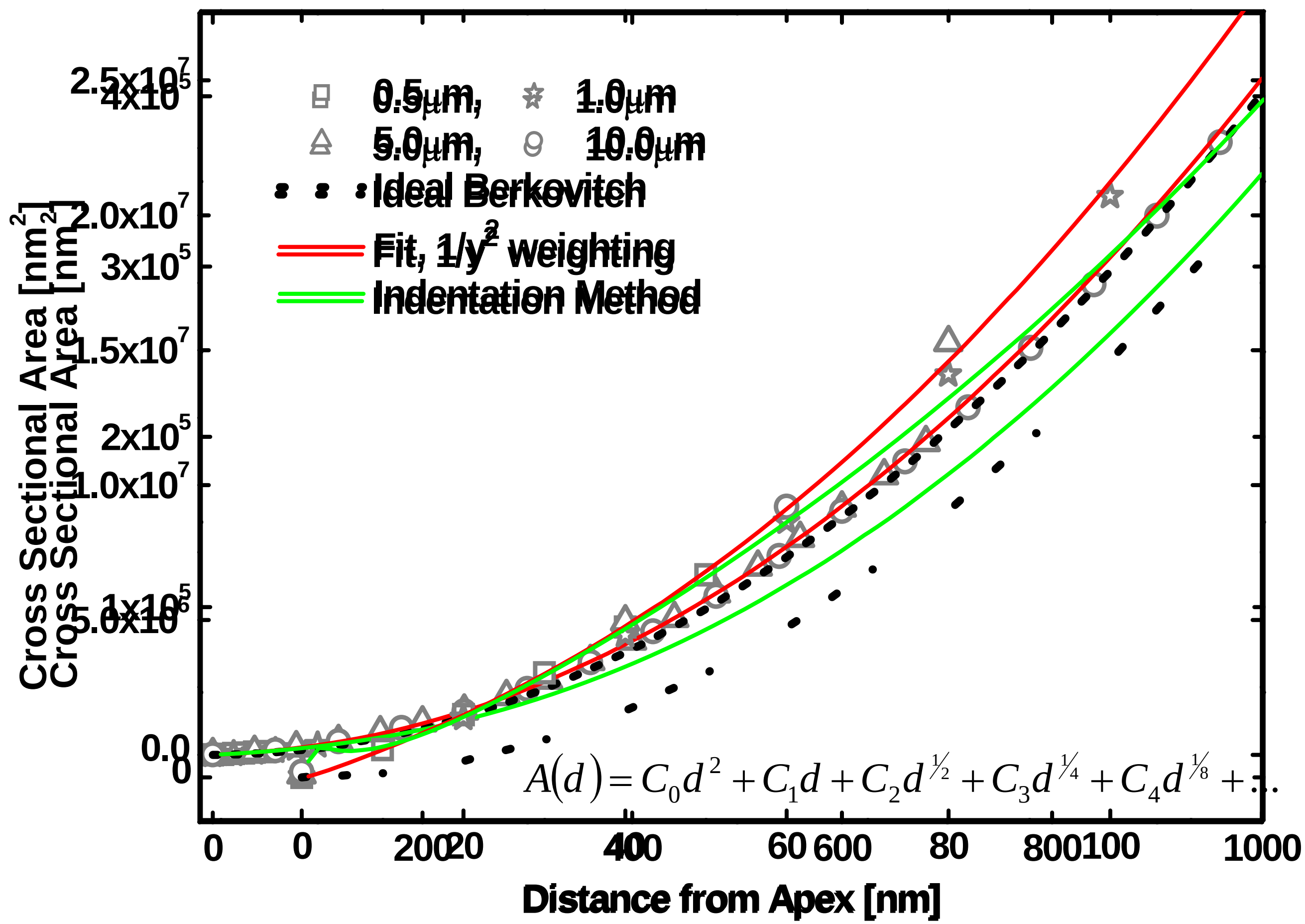




Take slices at known intervals from the apex and calculate their area

Plot these areas vs. the distance traveled from the apex and fit.

A weighting factor of y^{-2} boosts the importance of data near the apex in numerical fitting



Comparison

Fused quartz sample provided as a standard by Hysitron:

E* = 69.6 GPa, H = 9.6 GPa

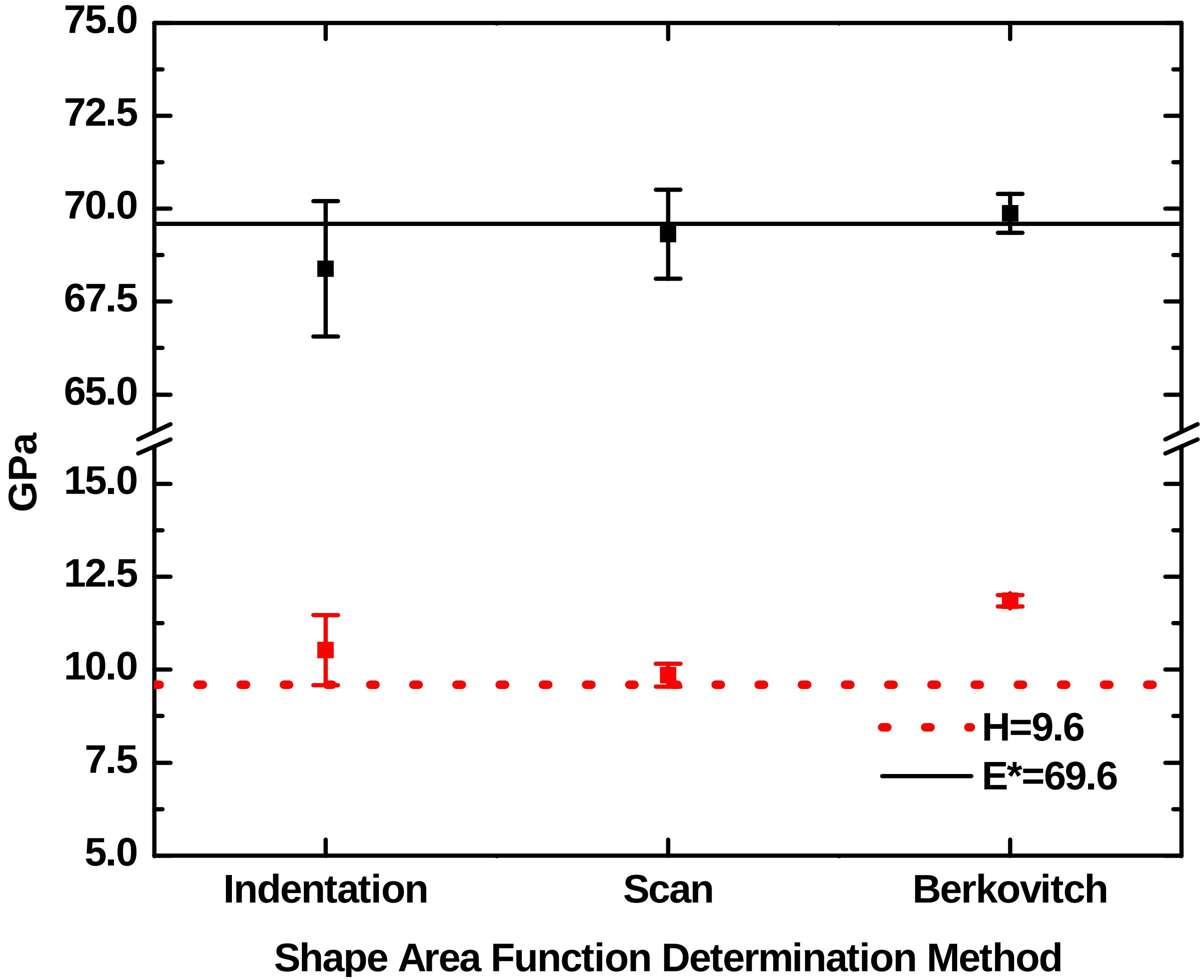
Indentation based approach (C_i = 3.2 ± 0.1 nm/mN):

underestimates E* by **1.6%** and overestimates H by **9.6%**

Scan based approach (C_i = 4.9 ± 0.2 nm/mN):

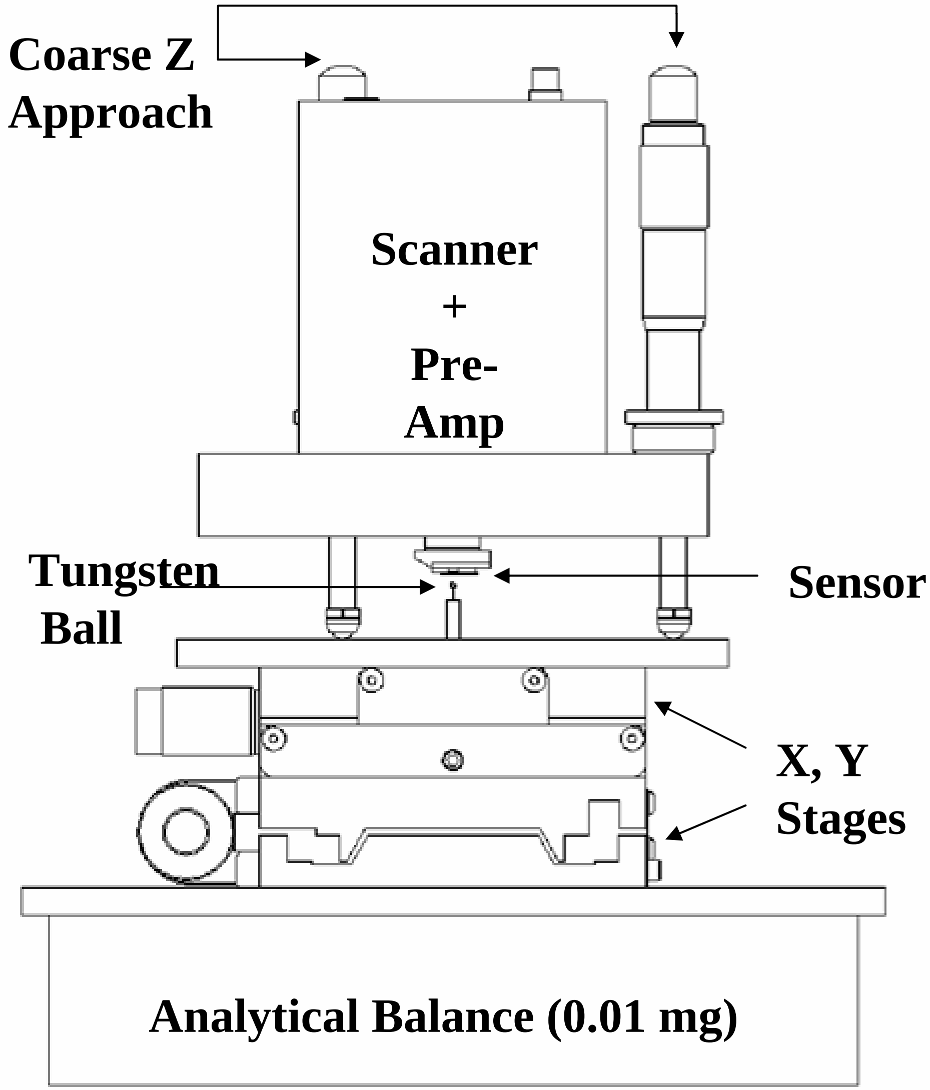
underestimates E* by **0.4%** and overestimates H by **2.5%**

Effect of shape area function determination on instrumental errors in nano-indentation experiments *(submitted Rev. Sci. Inst.)*



Sensor Calibration

Lower level ~100 nN (extrapolate to lower forces)

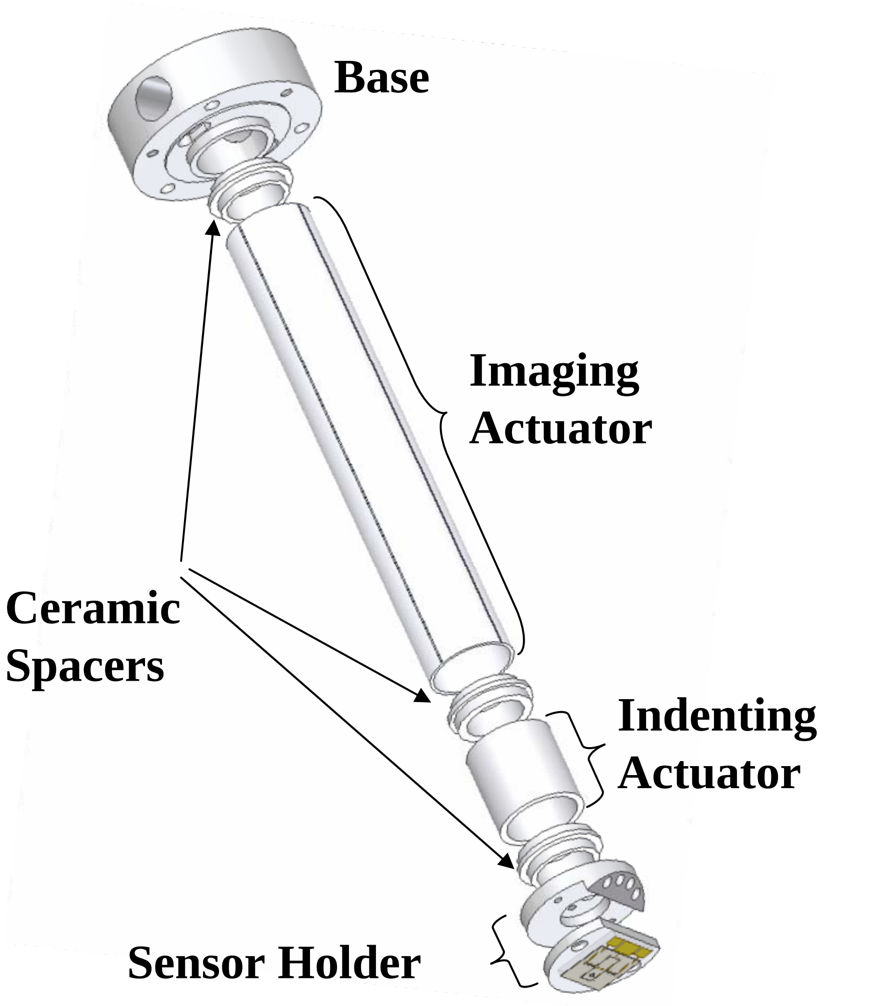


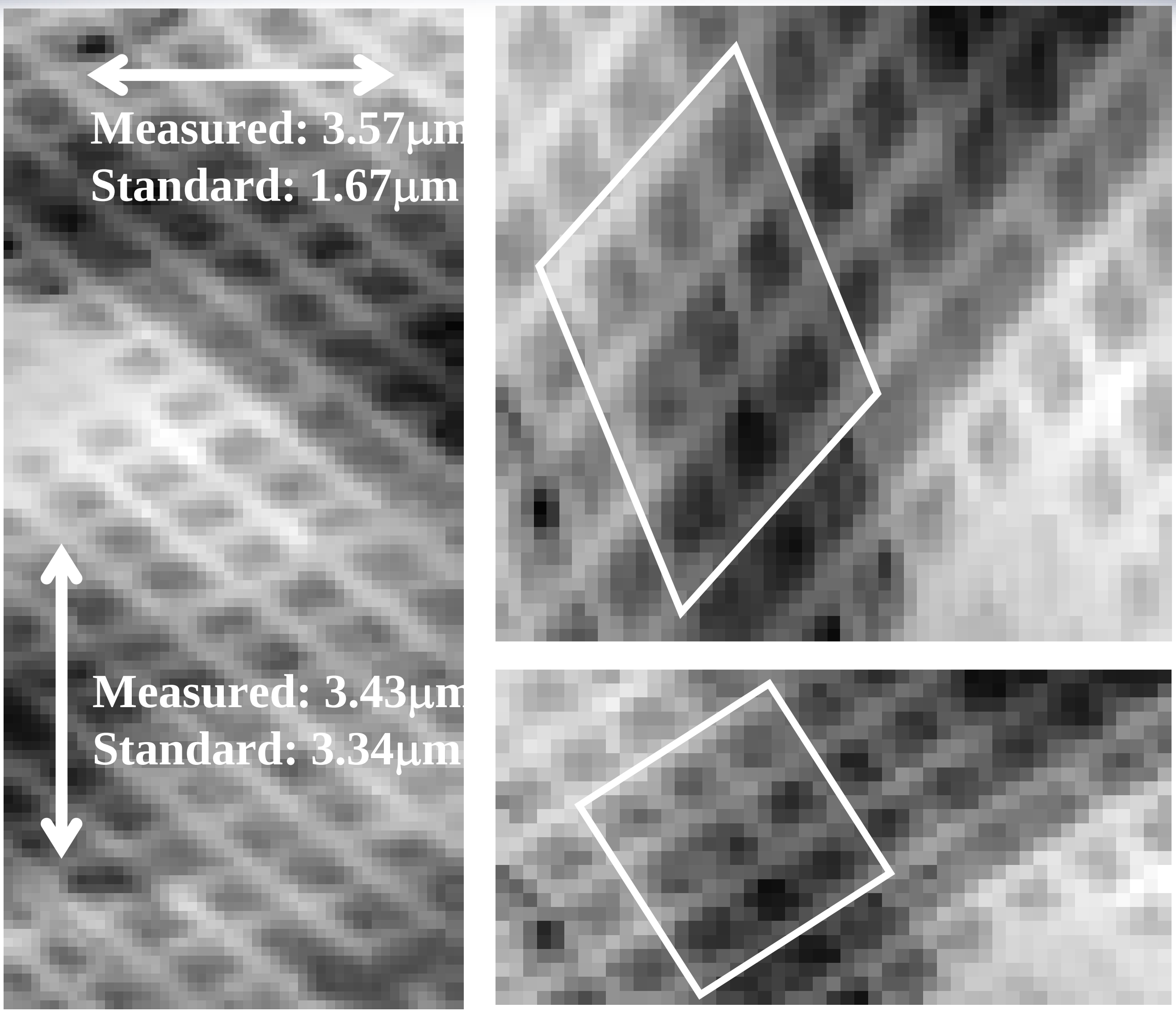
What needs to be done for high quality quantitative measurements with IFM?

1. Shape/size characterization of etched W tips

Example: widely spaced, sharp, high, vertical objects.

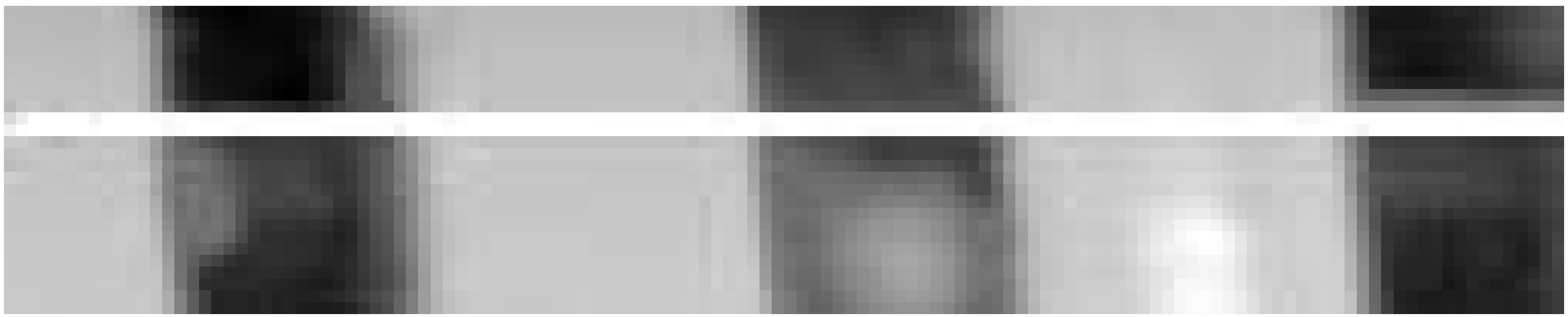
2. Force calibration (especially at lower forces)

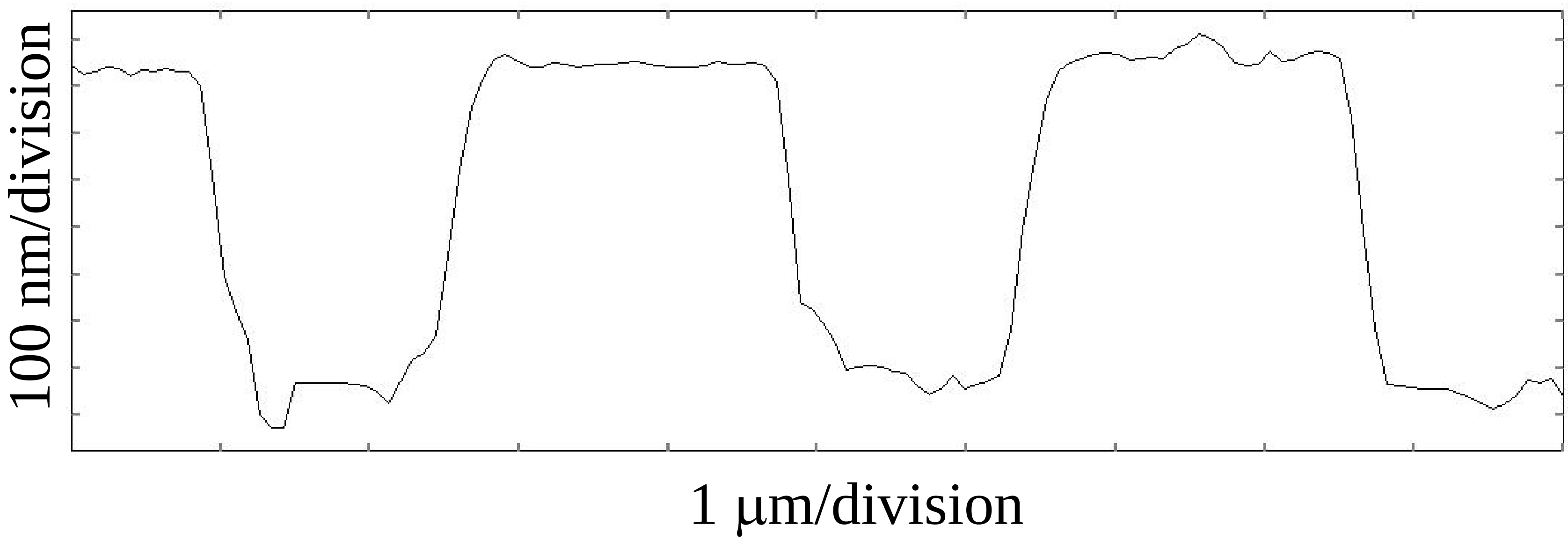

Base
Imaging
Actuator
Ceramic
Spacers
Indenting
Actuator
Sensor Holder

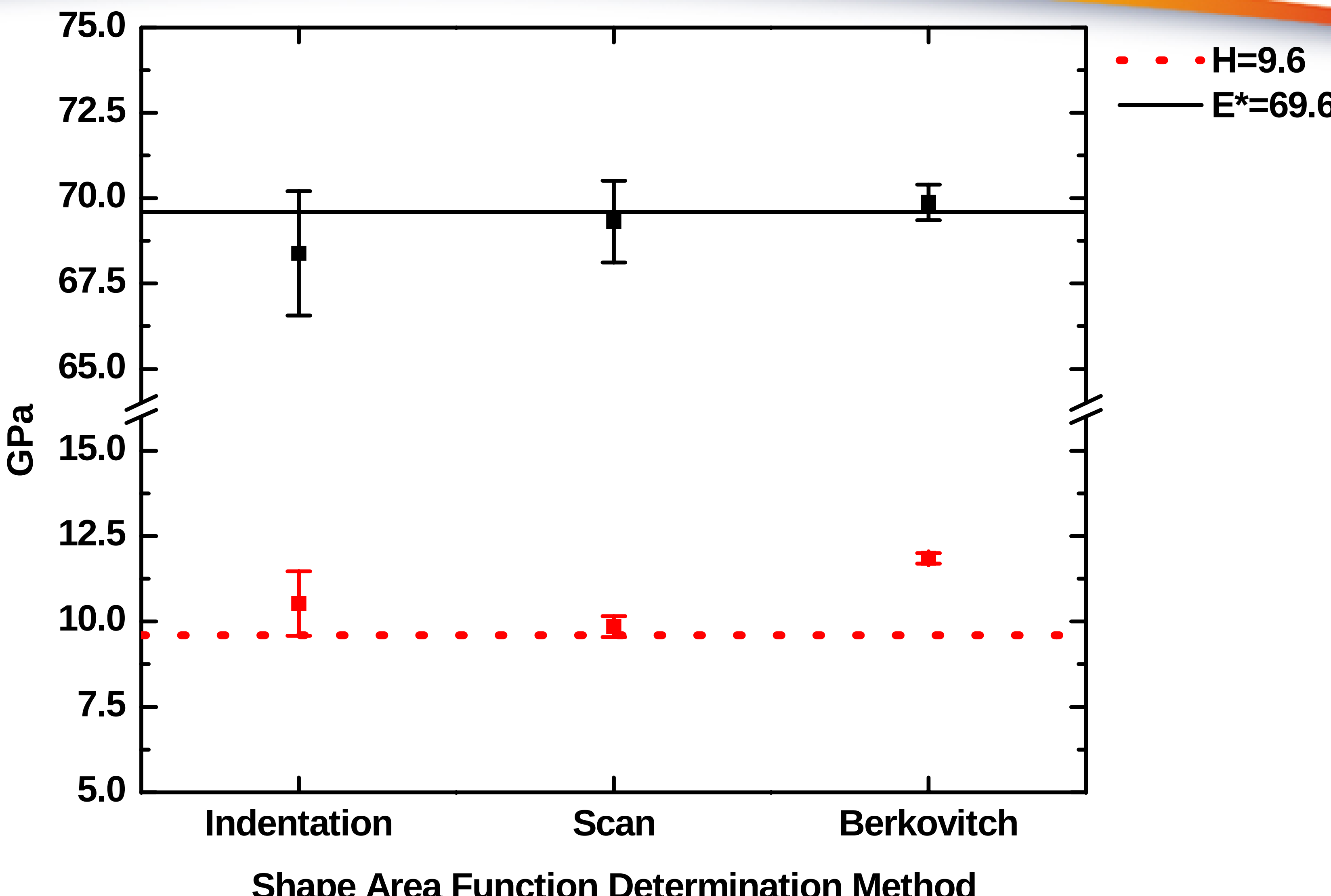


X-Y Calibration

Z Calibration

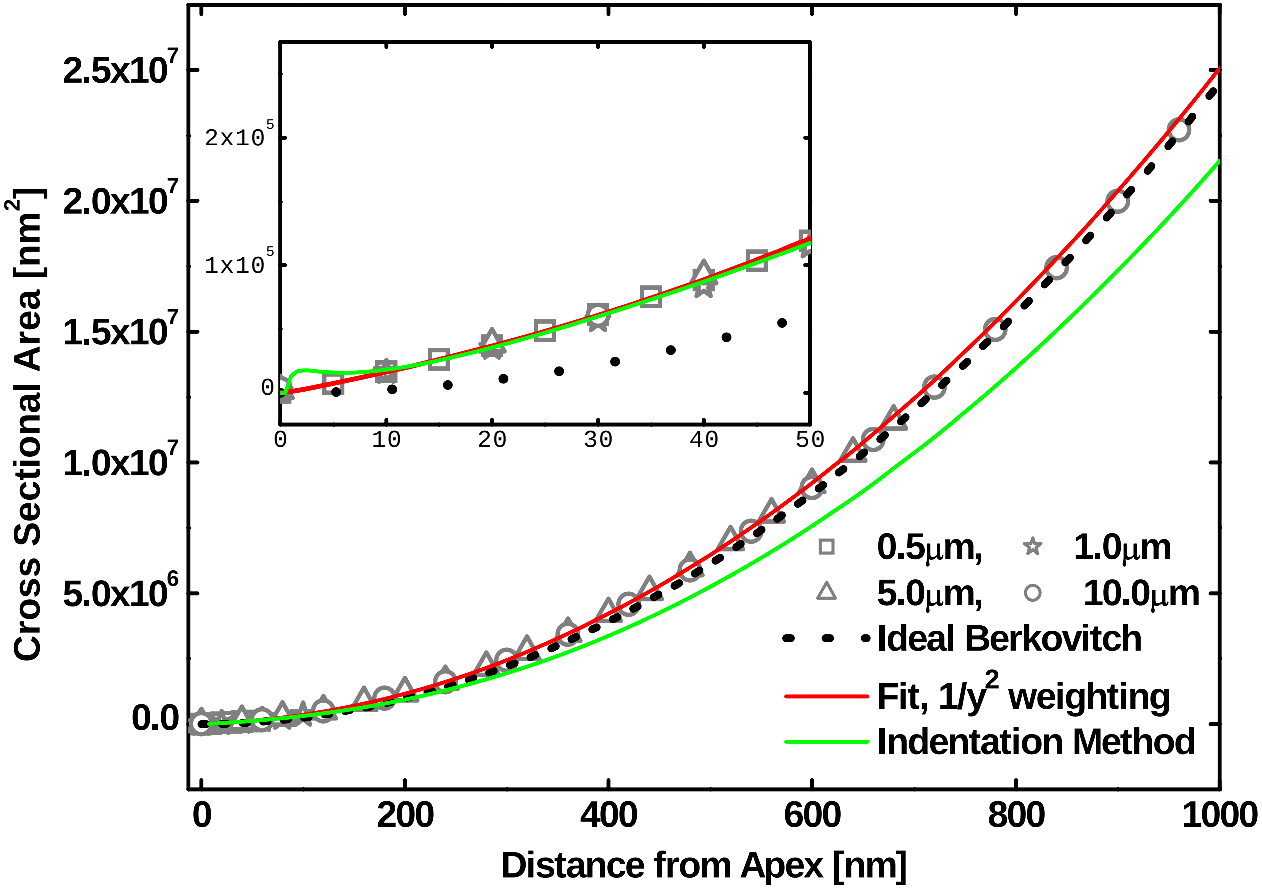






75.0
72.5
70.0
67.5
65.0
15.0
12.5
10.0
7.5
5.0
GPa
H=9.6
E*=69.6
Indentation
Scan
Berkovitch
Shape Area Function Determination Method

Effect of shape area function determination on instrumental errors in nano-indentation experiments *(for submission to Rev.Sci. Instrum.)*



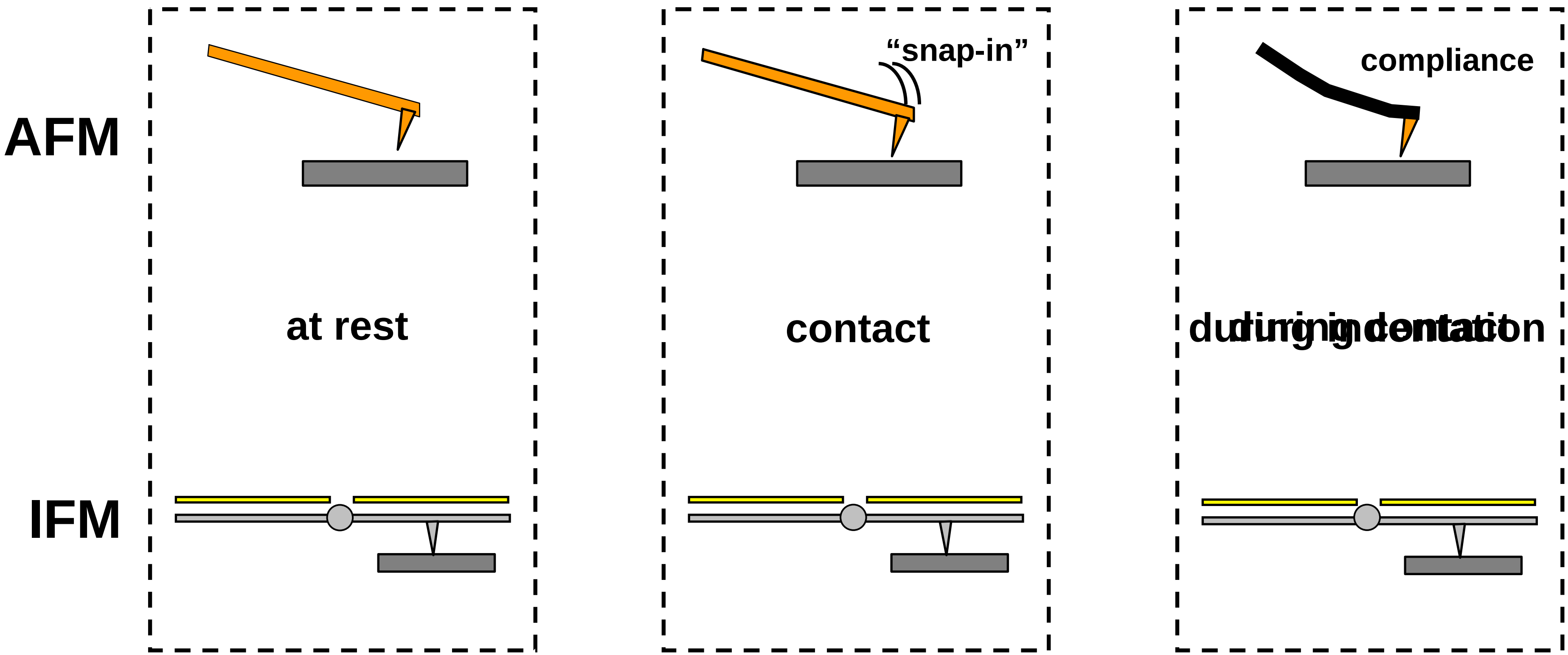
Overview

This project will construct a research tool that will quantitatively probe adhesive and mechanical properties of materials on the nanoscale.

The interfacial force microscope (IFM) technology is being transferred from the group of Professor Peter Norton, Department of Chemistry, University of Western Ontario.

We are constructing an IFM in order to implement the tool as a general usage device for routine and custom experimentation for the NINT and University of Alberta (U of A) research community.

AFM vs. IFM Force Detection



AFM is excellent for imaging but has severe limitations as an indenting tool

Progress

1. Construction began in January 2006 at UWO – base and scanner assembly
2. Main components machined and assembled by July 2006.
3. DMP relocated to NINT August 2006
4. Final components now finished (heat shielding)
5. DMP presently in London, ON doing final assembly and testing
6. Commercial SPM controller, fiber interferometer, borescope and temperature controller ordered via Capital funding.

IFM system being built for NINT

-Encoded X, Y, Z sample stage can find a volume of ~10 zL inside a 4mL envelope (formerly stepper motors)

-Updated sample heating/cooling and sensor shielding for operation ~200 to 600K

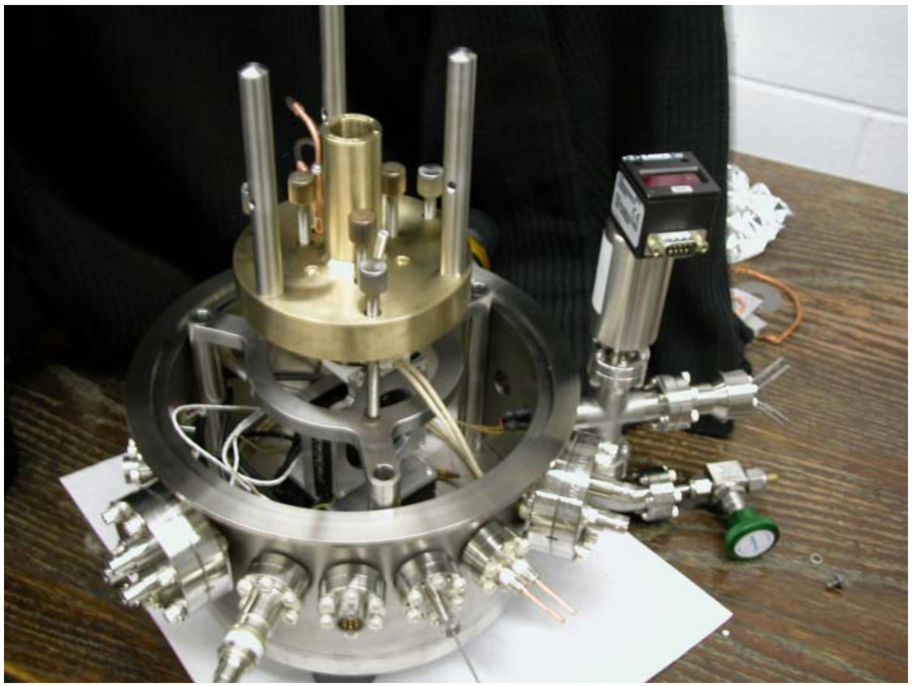
-Pressure gauge in chamber (chamber is designed to go down to roughing vacuum)

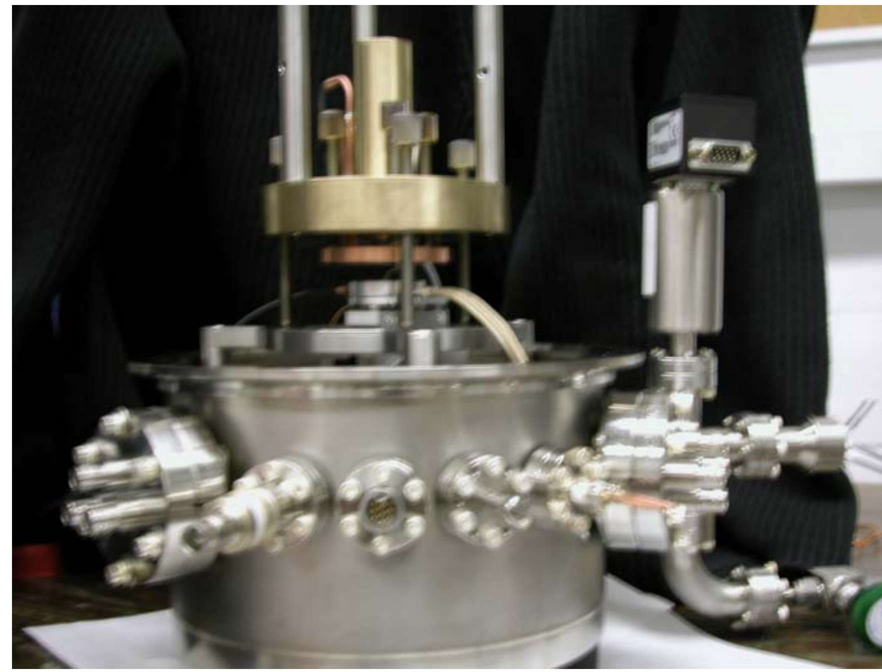
-Top viewing capabilities using a fibre inspection camera

-Isolated ground inputs for additional signals

-Interchangeable scanners

Photos from December 5, 2006 (UWO)



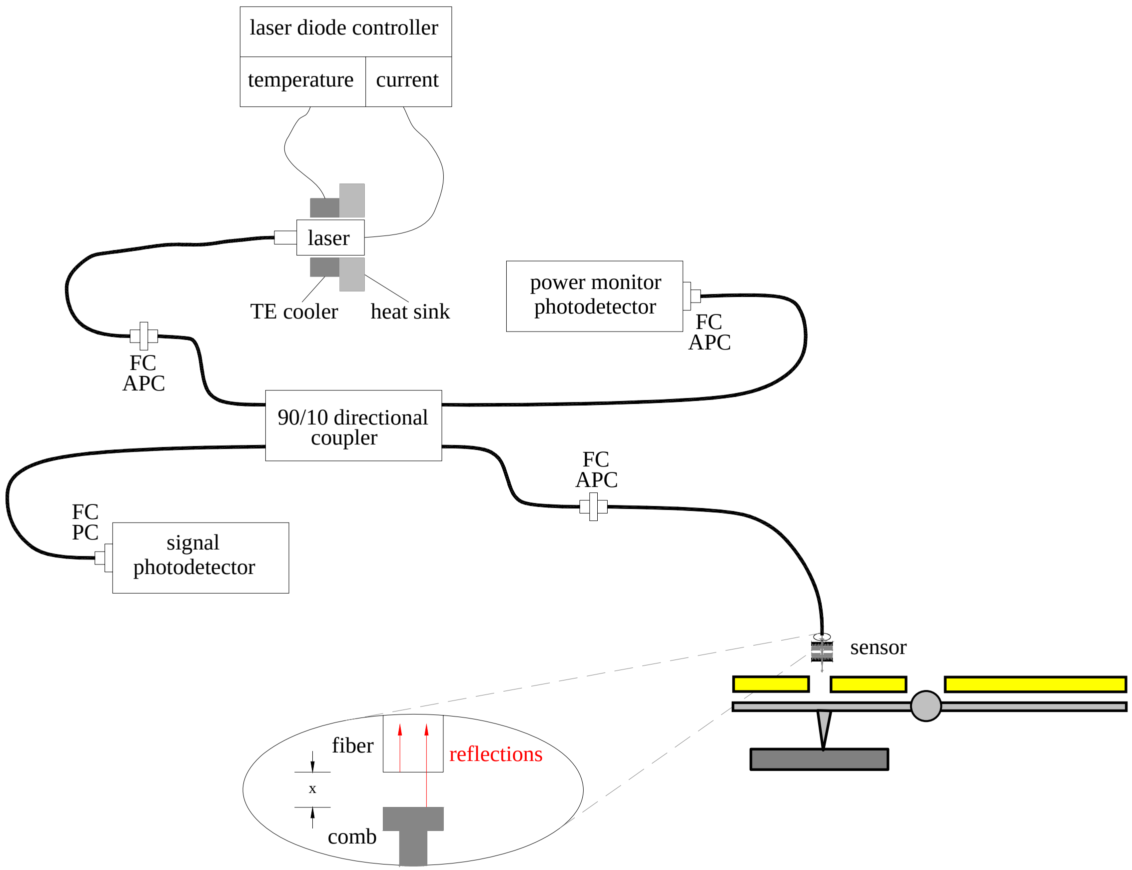


Delivered and Installed at NINT: February 2007

Operational: March 2007

Coupling laser interferometry to IFM:

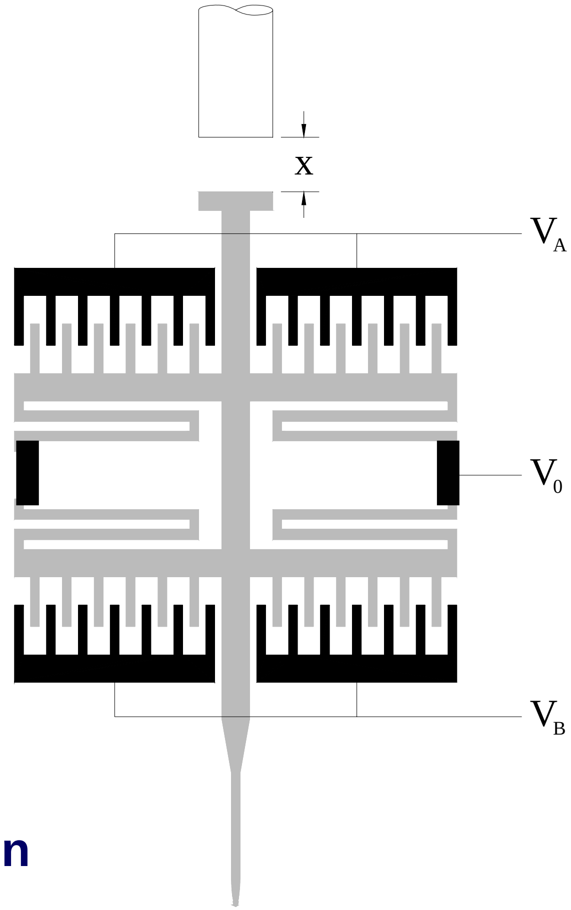
Detection of force-compensated probes



Comb Drive Sensors: pN Sensitivity

With Walied Moussa (Mech Eng)

- integrated assembly
- very small footprint
- displacement sensed optically
- displacement linear with voltage
- balanced sensor design
- pico Newton sensitivity
- retains force compensation



$$x = \frac{1}{k}\varepsilon_0 n \frac{h}{g} V_0^2$$

where: k = spring constant
n = number of comb fingers
h = height of comb finger
g = gap between comb fingers
V_0 = applied voltage

For linear motion use a push-pull mode with two opposite actuators A and B so that

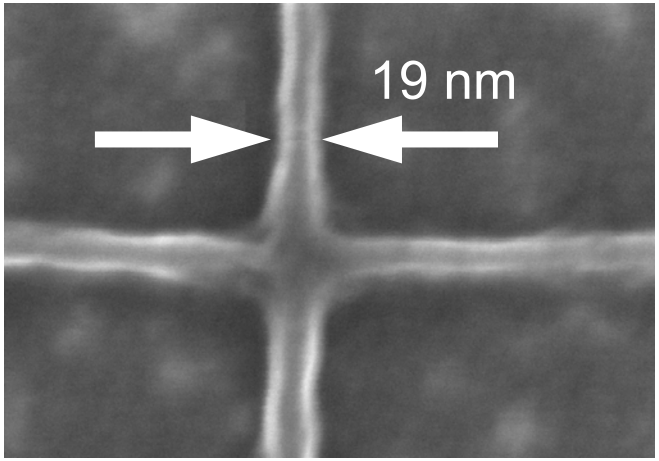
$$x = \frac{1}{k}\varepsilon_0 n \frac{h}{g}\left[(V_A - V_0)^2 - (V_B - V_0)^2\right]$$

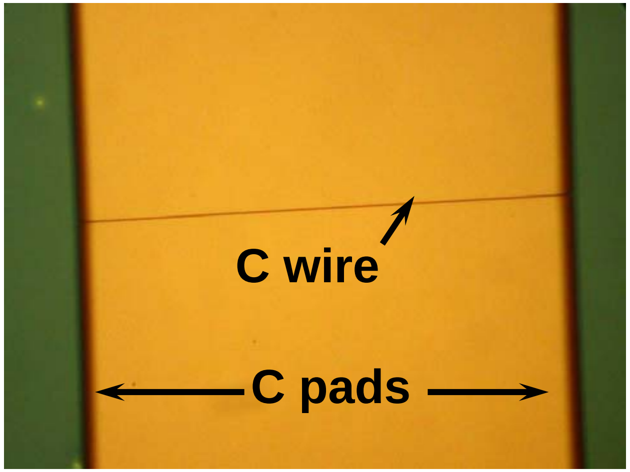
When $V_A = -V_B$ all quadratic terms cancel and

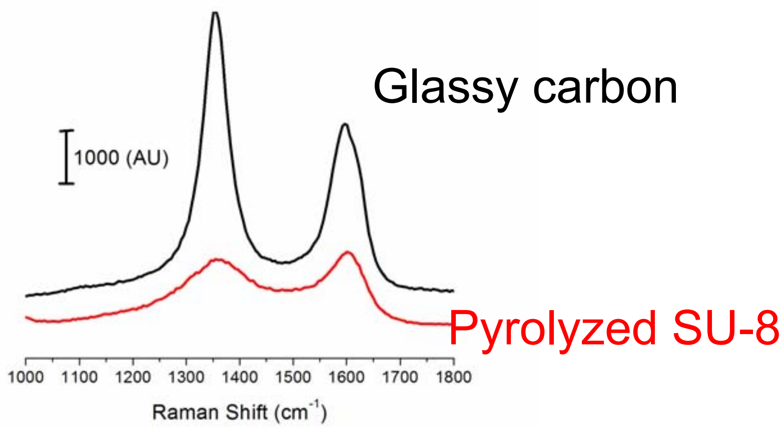
$$x = \frac{1}{k}\varepsilon_0 n \frac{h}{g} 2 V_A V_0$$

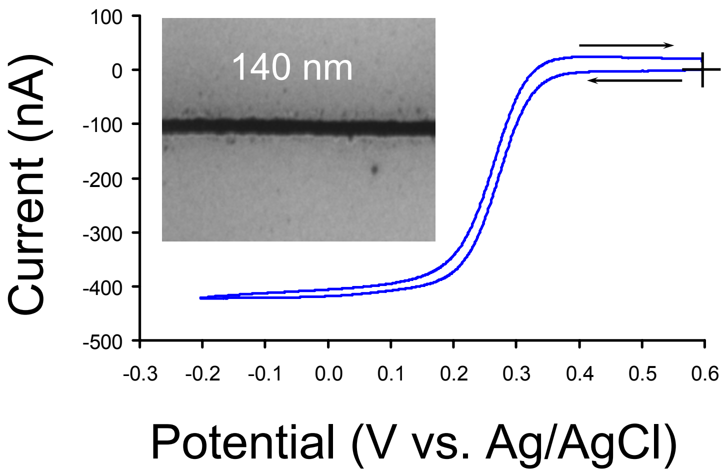
Nanoscale Graphitic Carbon Structures and Devices

Rongbing Du and Mark T. McDermott

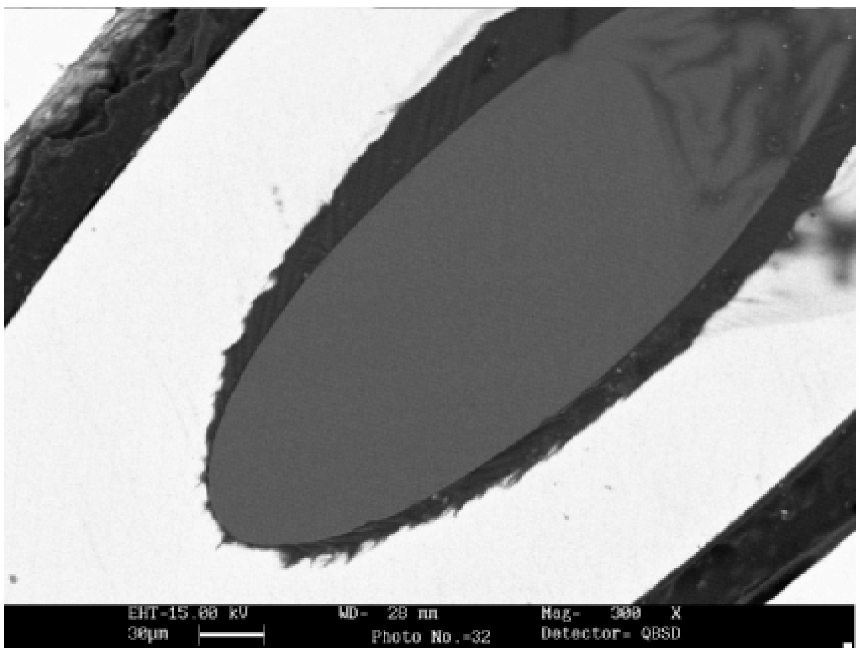








Sapphire: A viable alternative?



Harder Tips:

Diamond can be used but it is expensive and brittle