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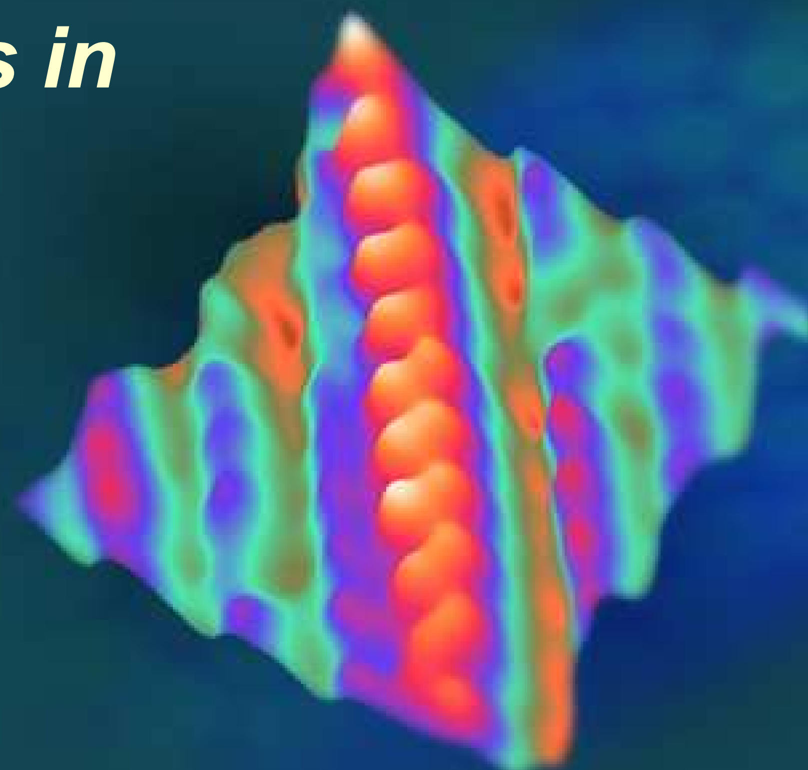
UNIVERSITY OF ALBERTA

Instrumentation Errors in Nano-Indentation



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GOVERNMENT OF ALBERTA

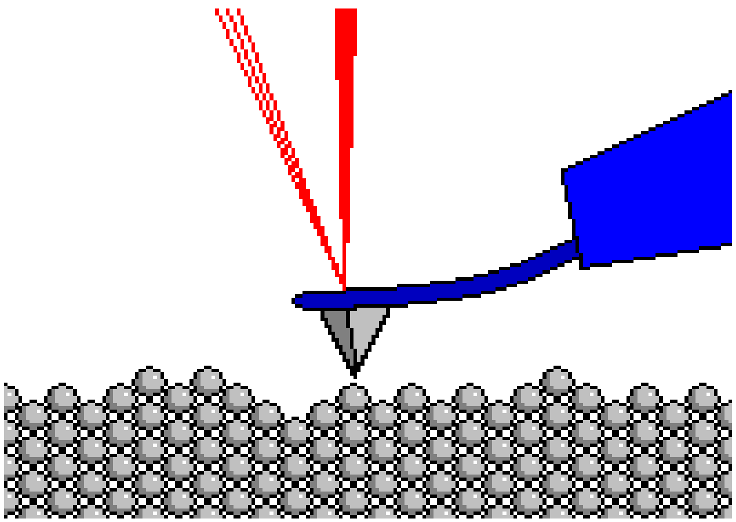
Canada

Motivation

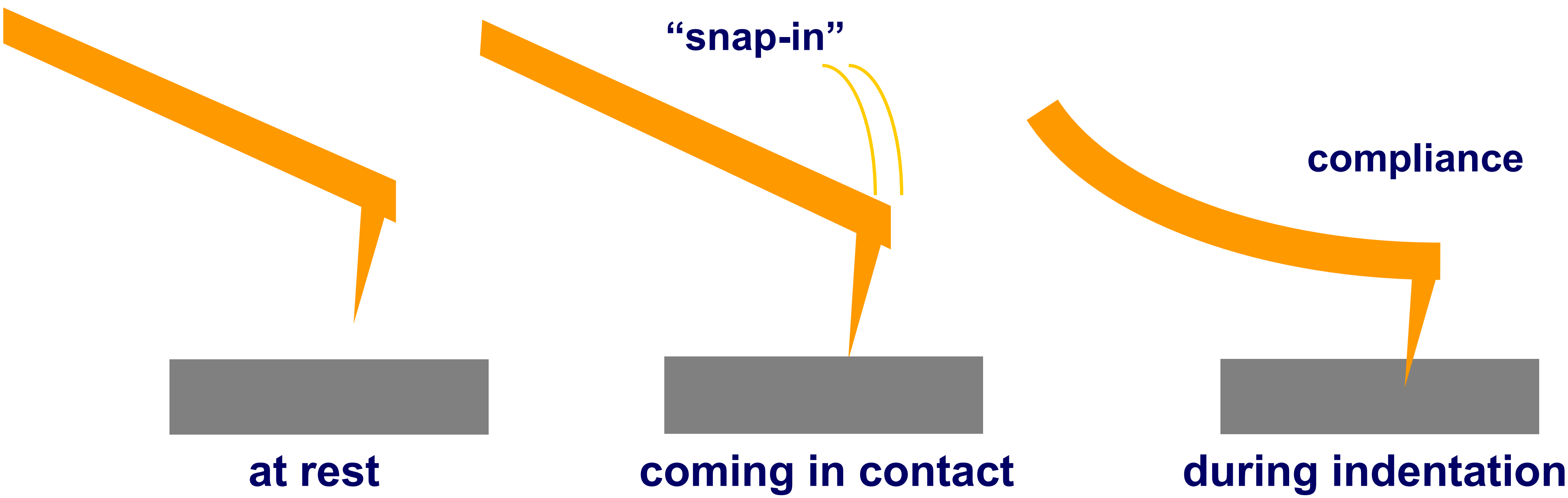
Ever increasing interest in microscopic machines demands a better understanding of the interactions of materials at sub-micron levels. This, combined with the fact that many technologically relevant materials are heterogeneous on a nanometer-micrometer length scale means that the study of the interactions of such materials requires the combination of reliable force measurements with imaging.

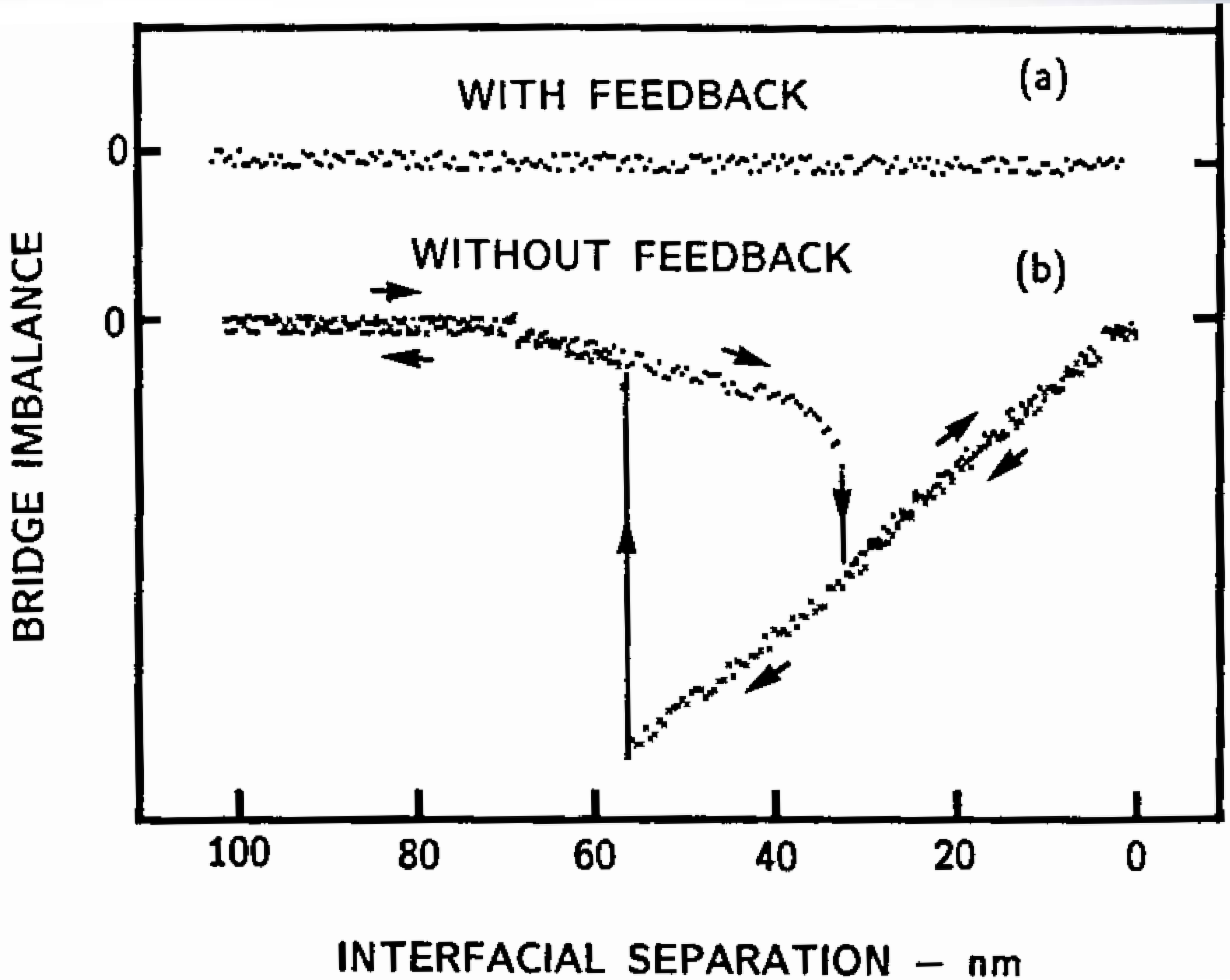
Scanning probe microscopes have been around for a number of years and are ideal for this task as they provide a relatively inexpensive way to perform high resolution imaging, can use different interactions as their input signal and can be also used for investigating the nano-mechanical properties of materials.

Topografiner -1972
Scanning Tunnelling Microscope (STM) -1981
Atomic Force Microscope (AFM) -1986
Interfacial Force Microscope (IFM) -1991



Typical cantilever behaviour



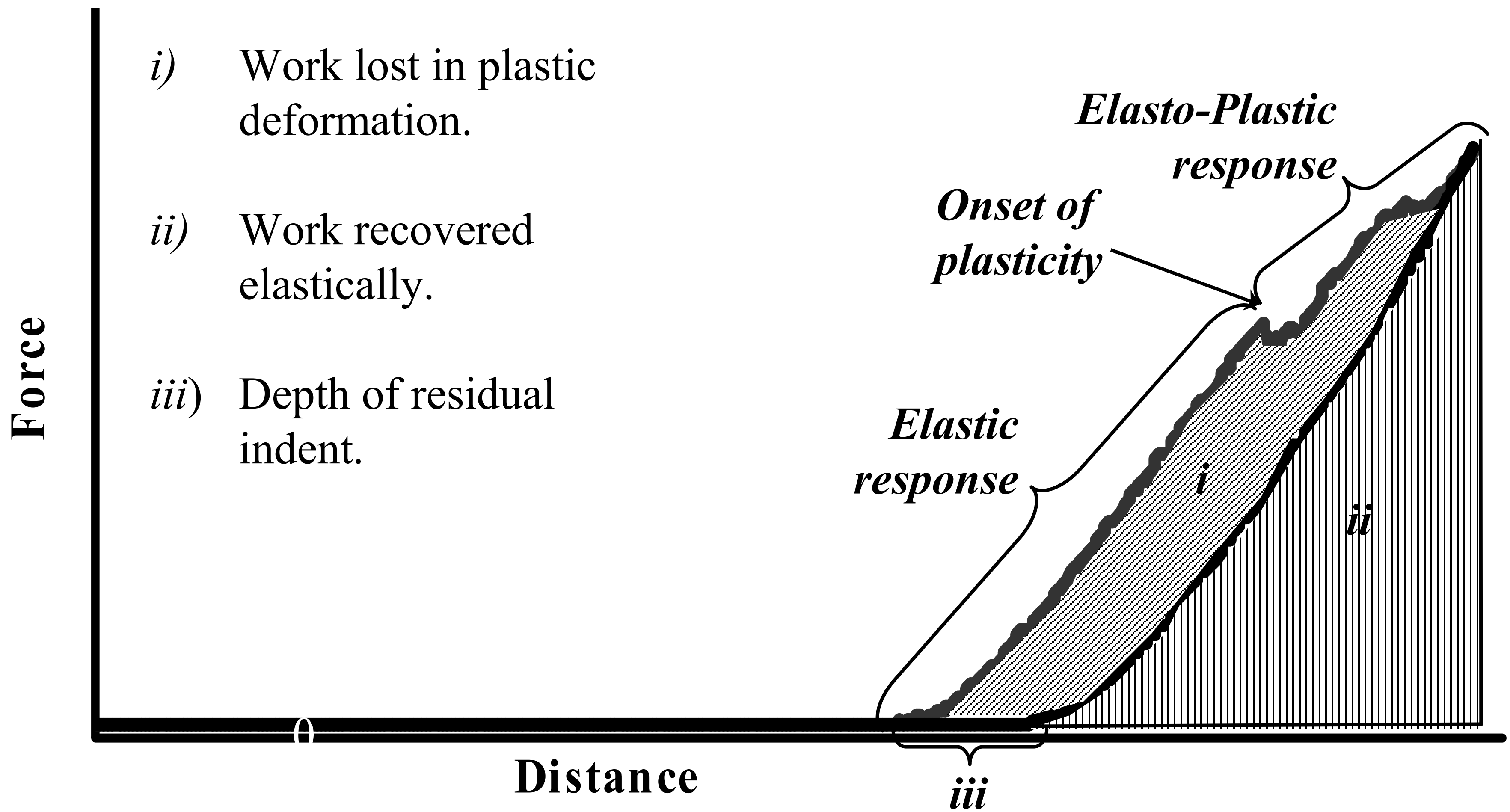

WITH FEEDBACK
(a)
WITHOUT FEEDBACK
(b)
0
0
BRIDGE IMBALANCE
100
80
60
40
20
0
INTERFACIAL SEPARATION — nm

Indentation Sequence

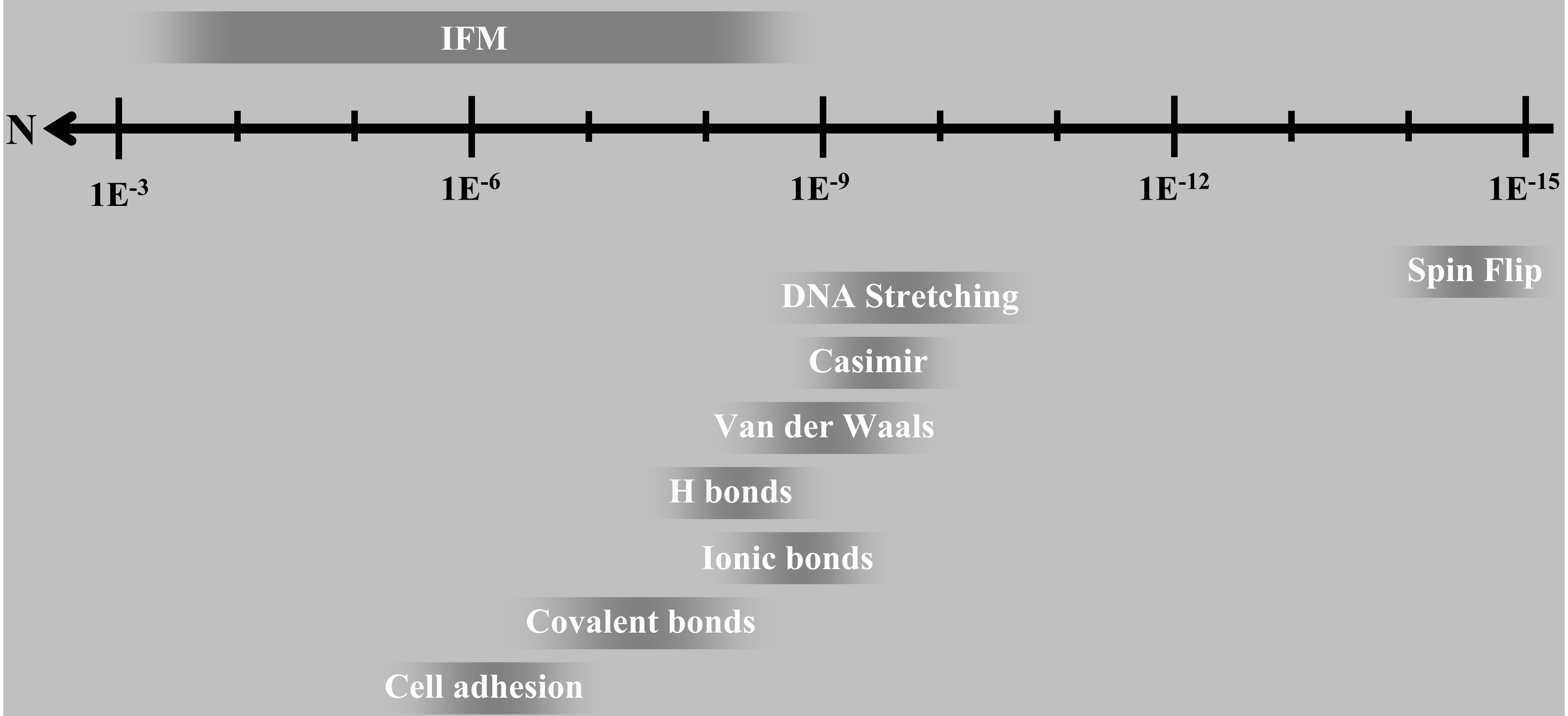
i) Work lost in plastic deformation.

ii) Work recovered elastically.

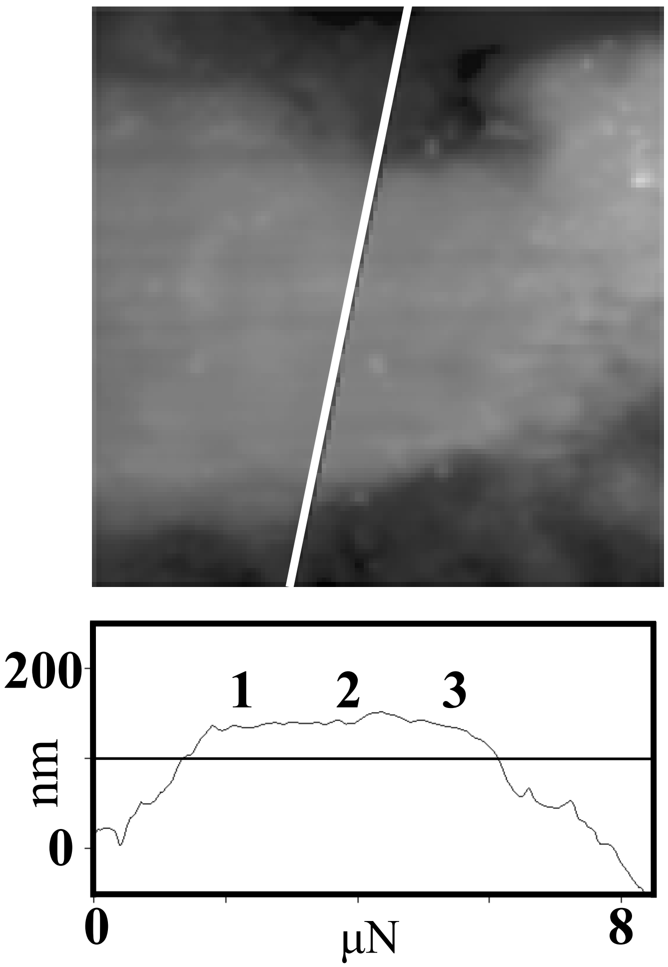
iii) Depth of residual indent.

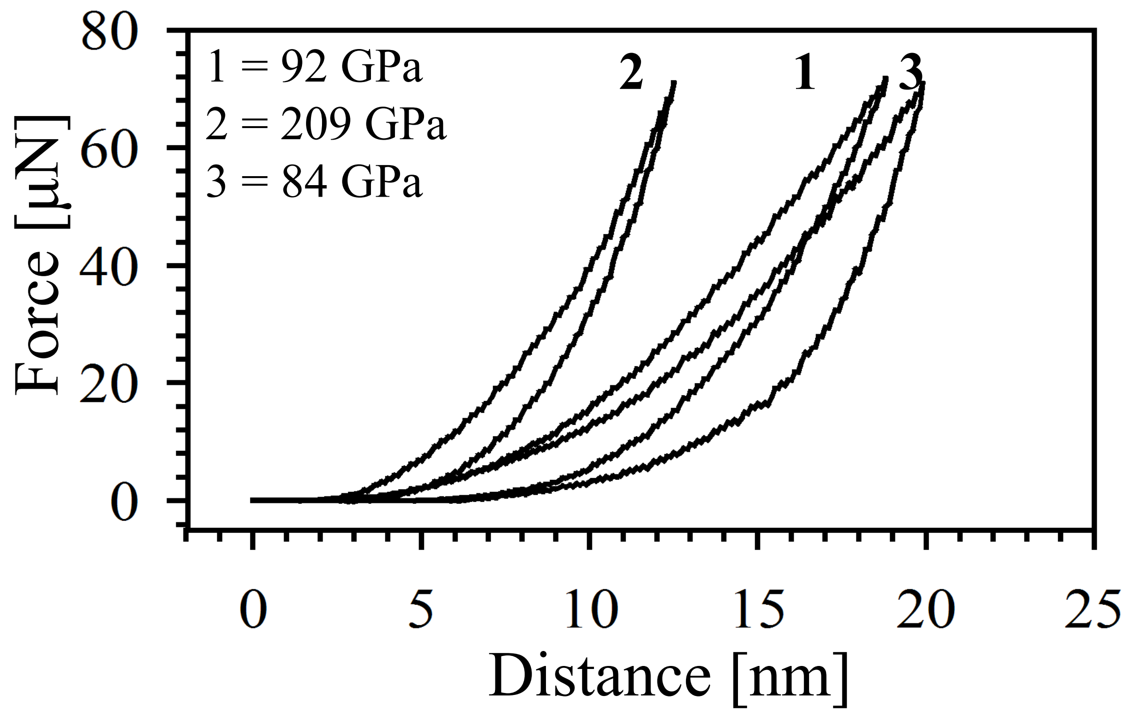


Where does the IFM force range fall?



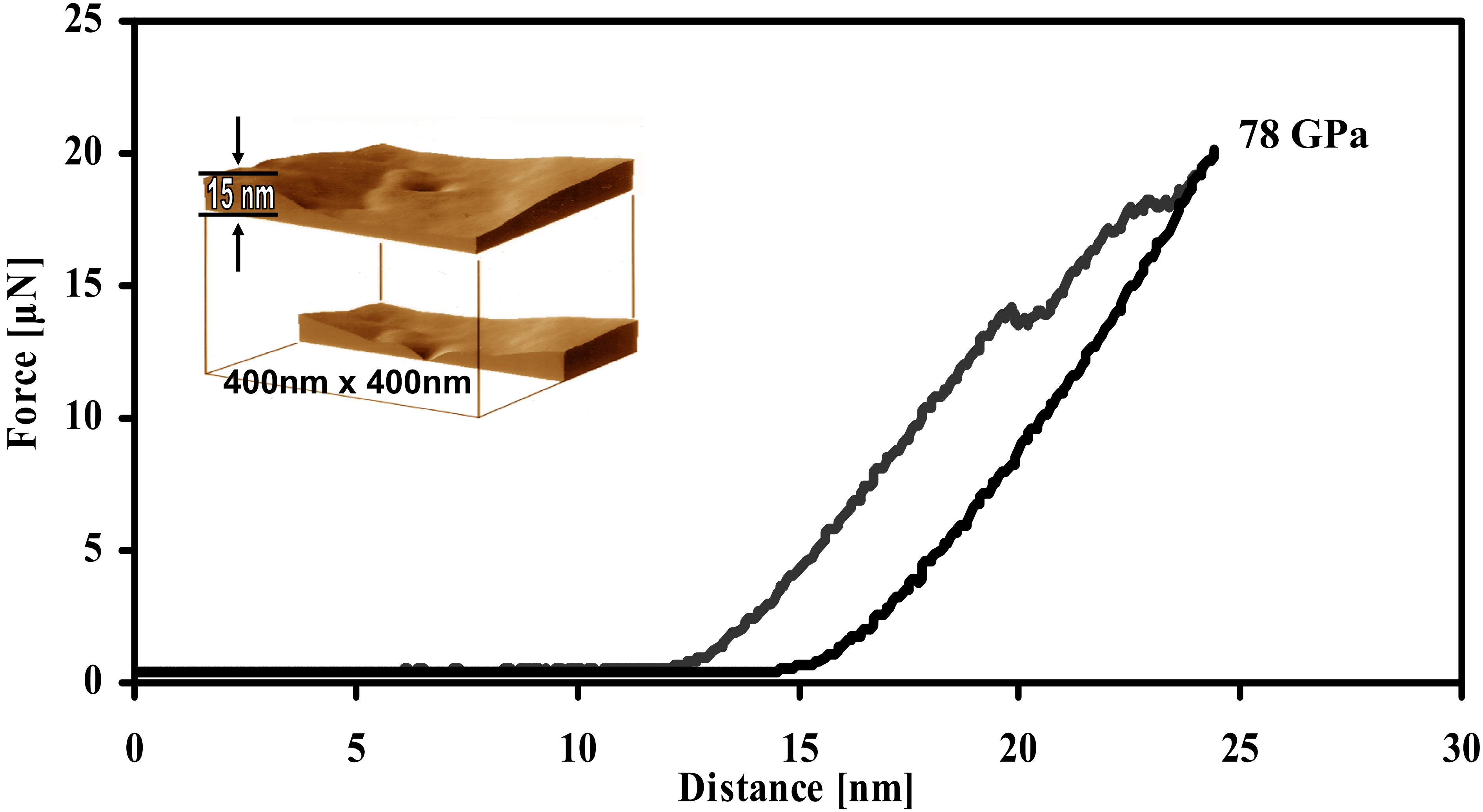
IFM Nano-indentation of Alkyl ZDDP tribofilm on steel



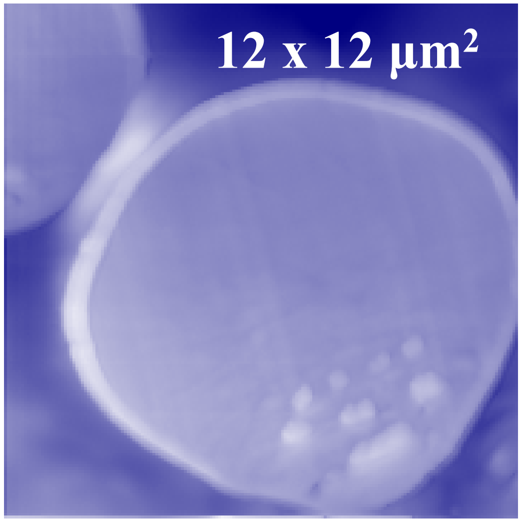


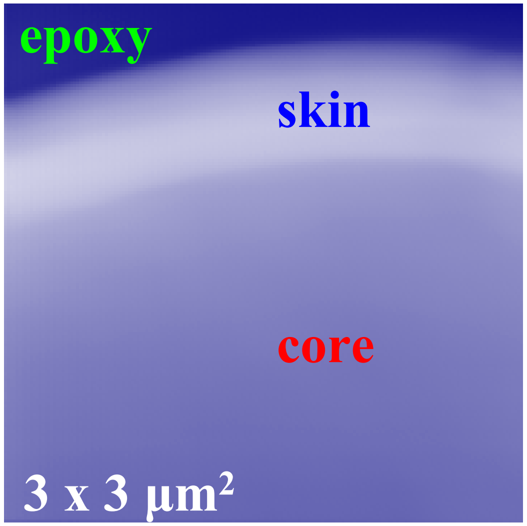
Graham, J.F., McCague, C., Norton, P.R., *Tribol. Lett.* 6, 149 (1999)

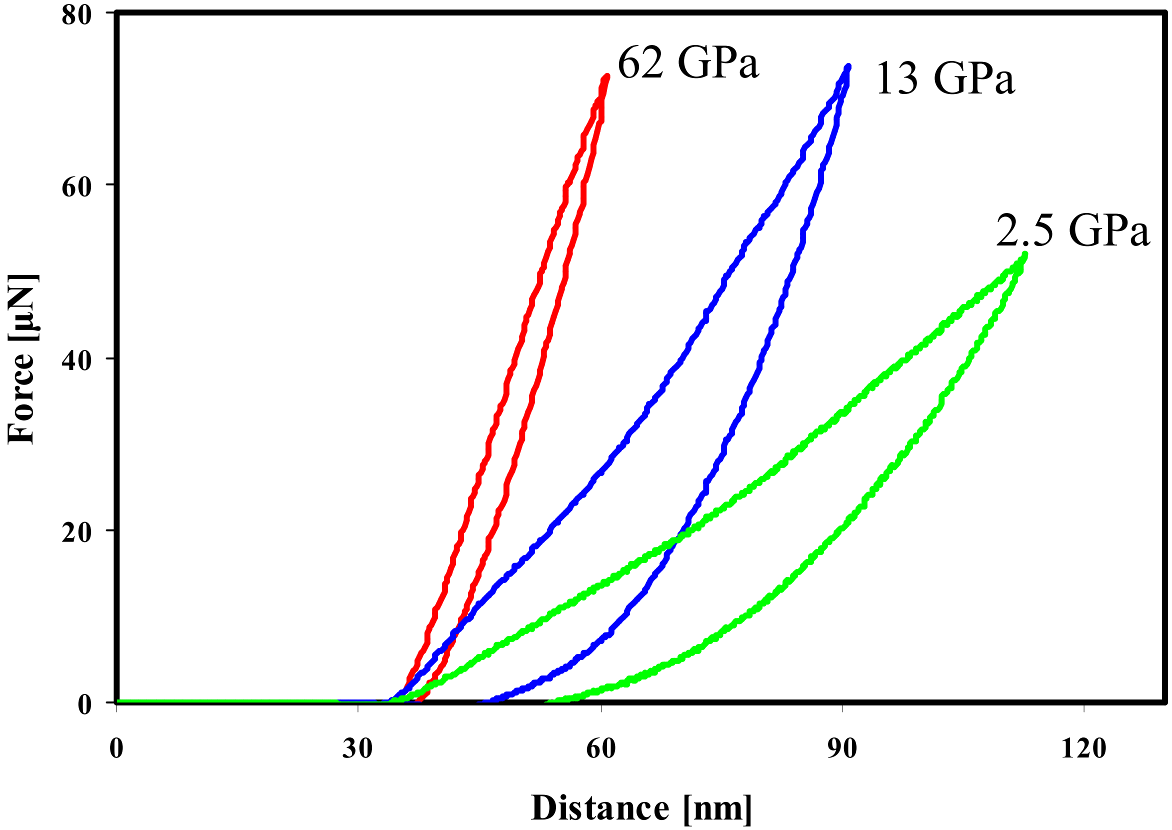
IFM Nano-indentation on Au ⟨111⟩



IFM Nano-indentation on Kevlar

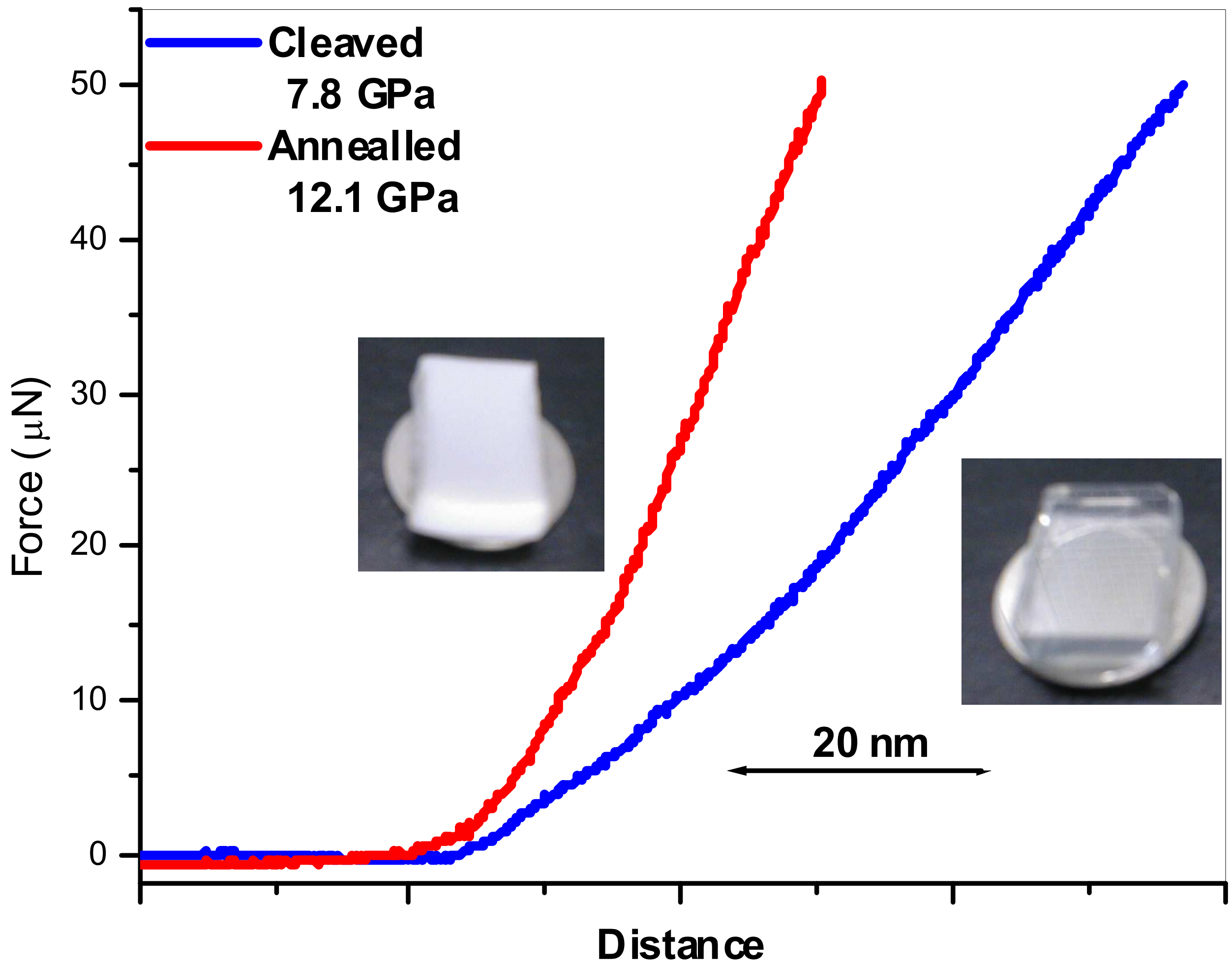




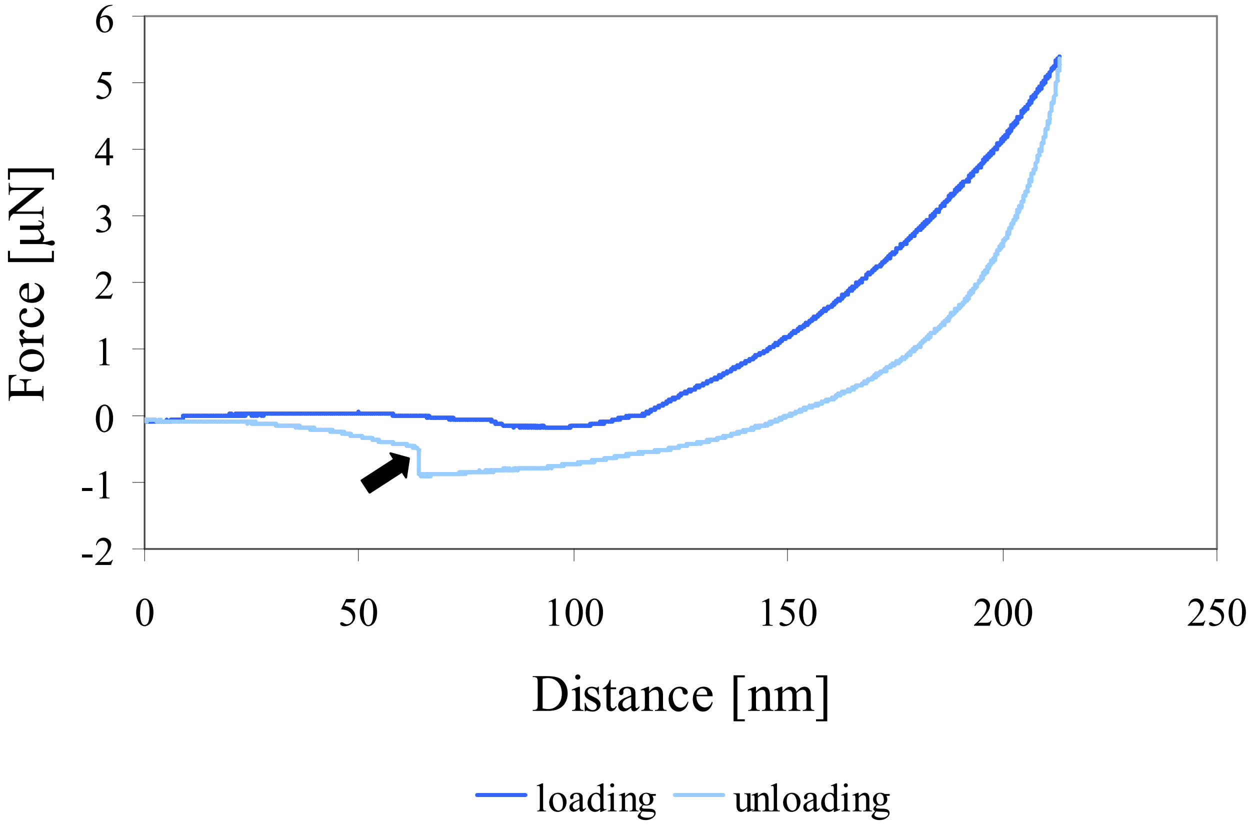


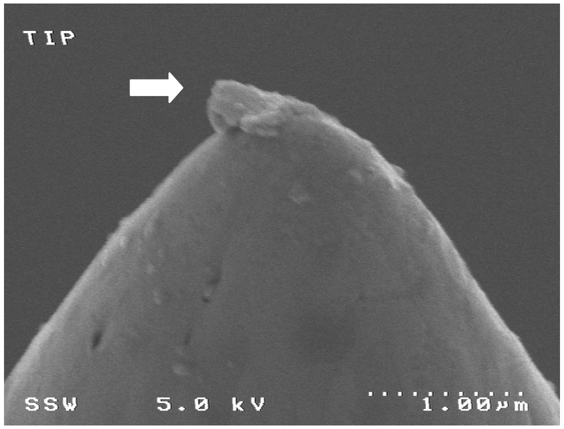
Graham, J.F., McCague, C., Warren, O.L., Norton, P.R., *Polymer* 41, 4761 (2001)

IFM Nano-indentation on PET



IFM Nano-indentation on PU

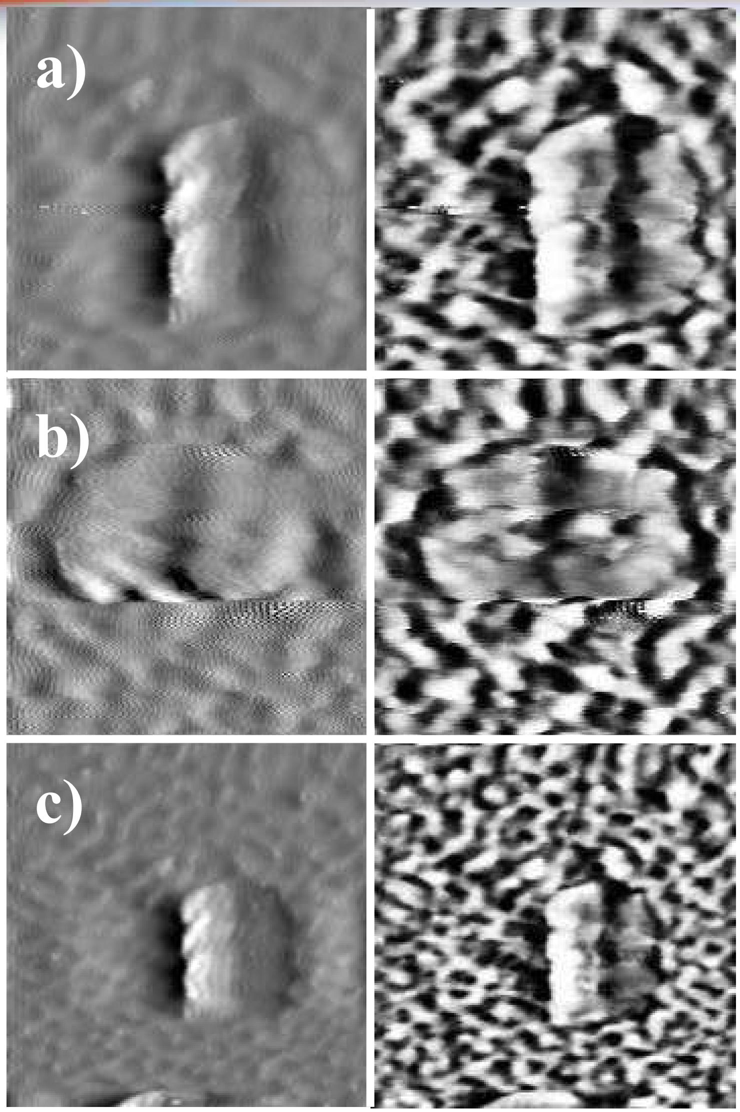




A SEM image of a tungsten probe after indentation on polyurethane.

Indentation on polyurethane showing evidence of transfer of material onto the probe.

Images taken on Polyurethane using TappingMode-IFM

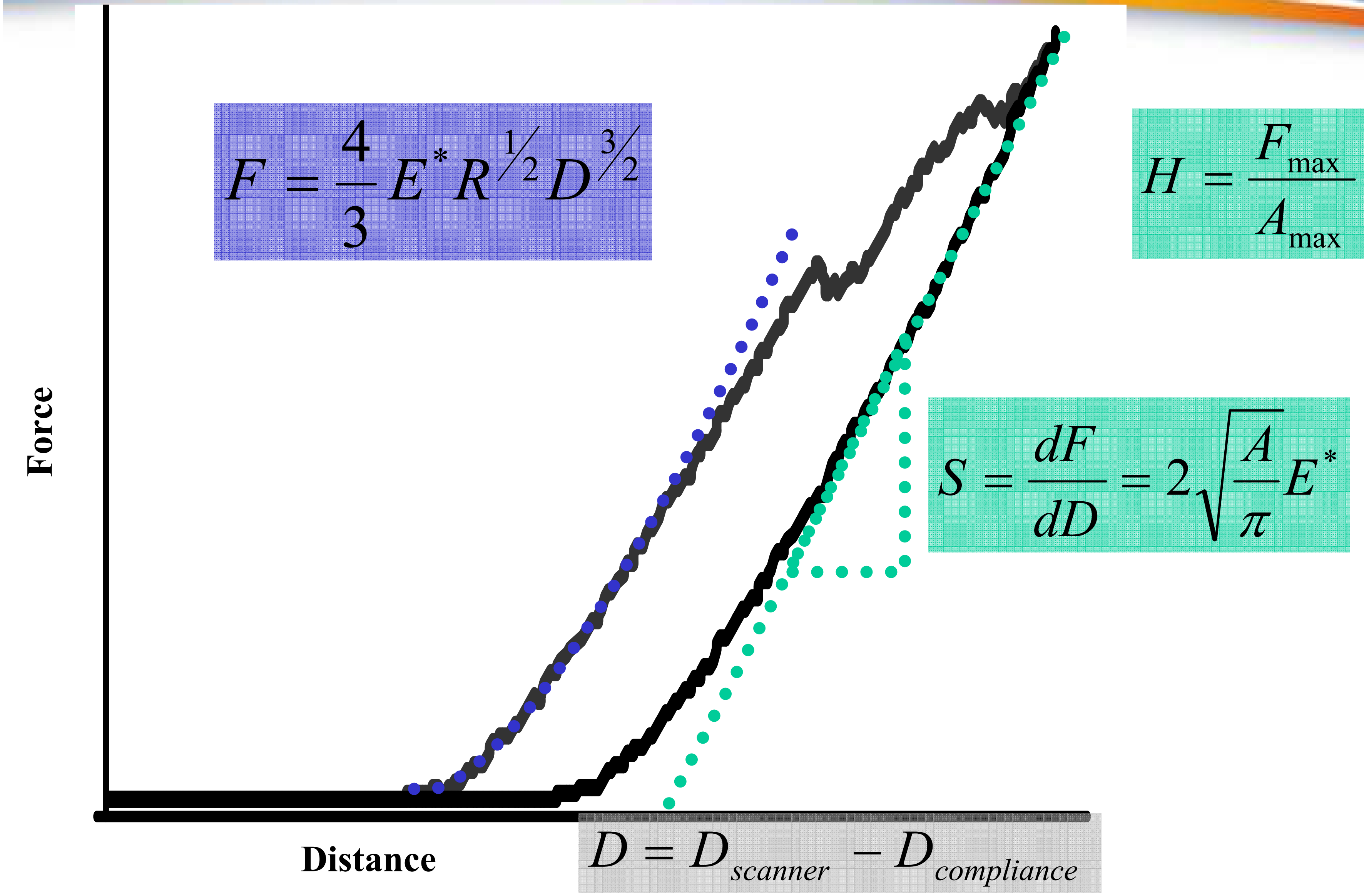


Amplitude **Phase**

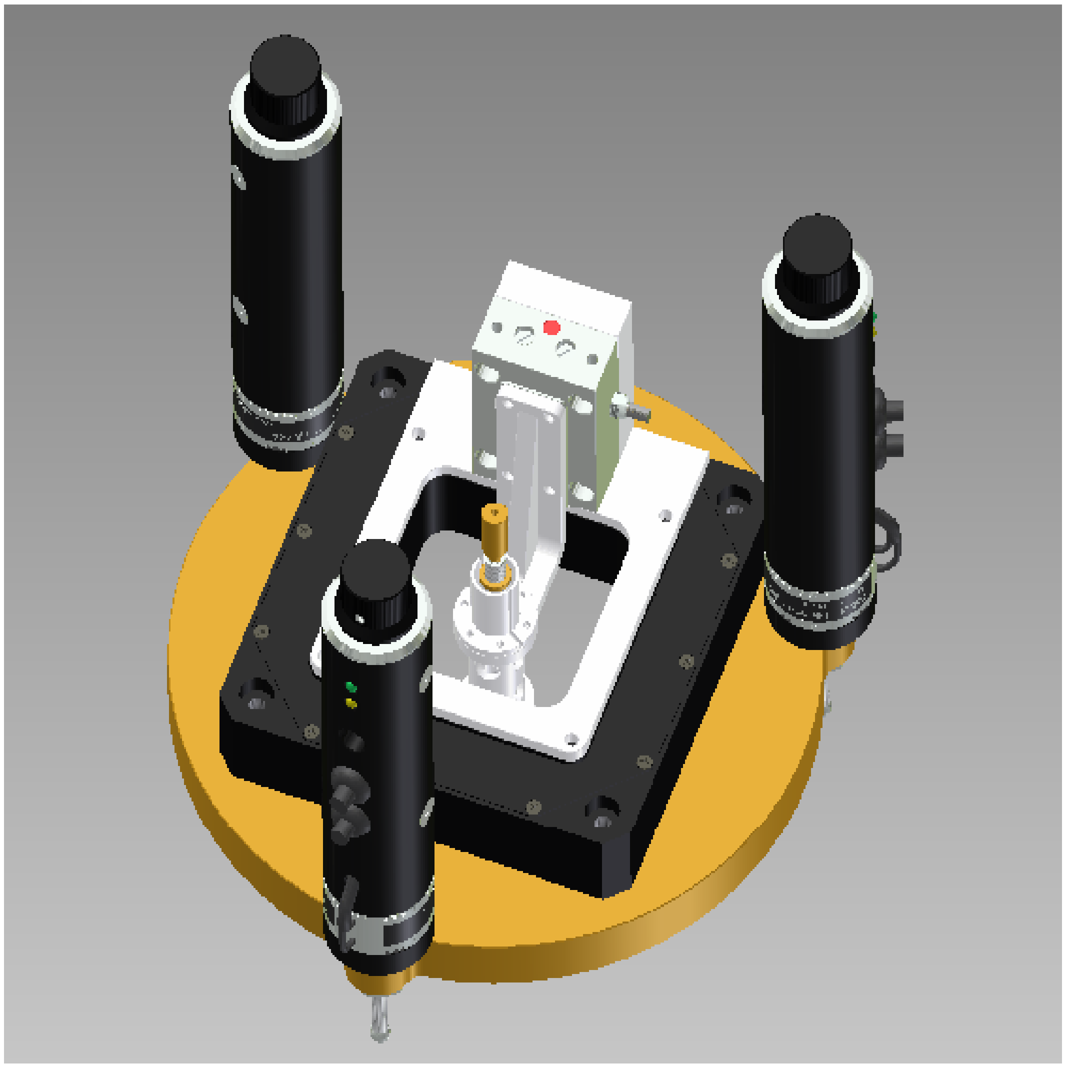
a) 6µm×6µm Tapping Mode scan after a 3µm×3µm Contact Mode scan.

b) Same spot rotated by 90°. (6µm×6µm)

c) 9µm×9µm Tapping Mode scan after several Tapping Mode scans at 6µm×6µm.


$F = \frac{4}{3} E^* R^{1/2} D^{3/2}$
$H = \frac{F_{max}}{A_{max}}$
$S = \frac{dF}{dD} = 2\sqrt{\frac{A}{\pi}} E^*$
Force
Distance
$D = D_{scanner} - D_{compliance}$

Instrument Development

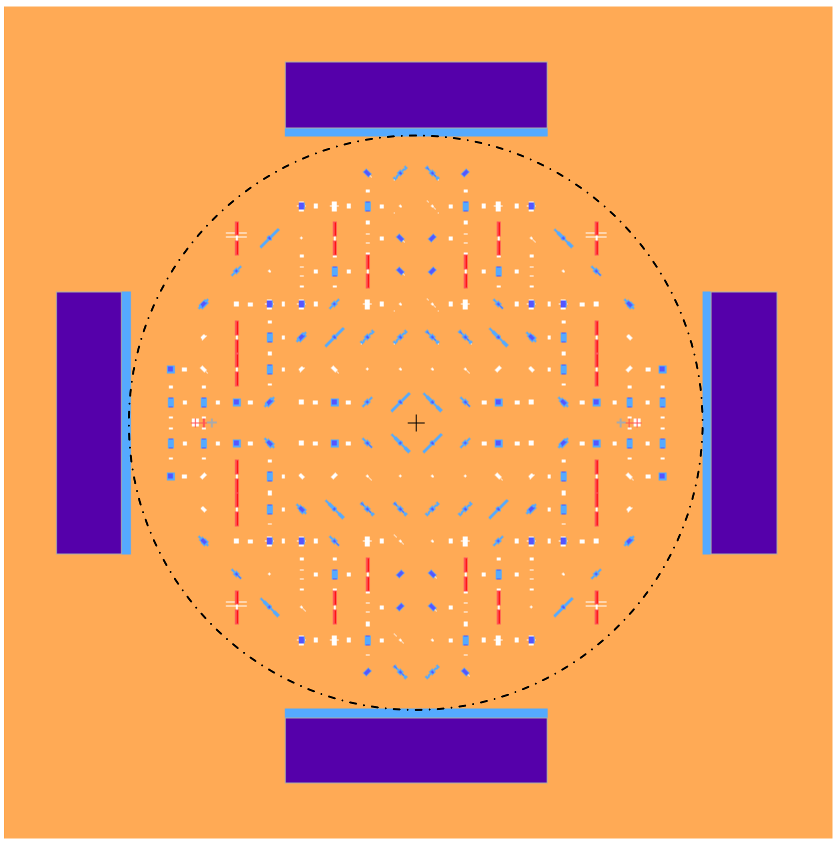


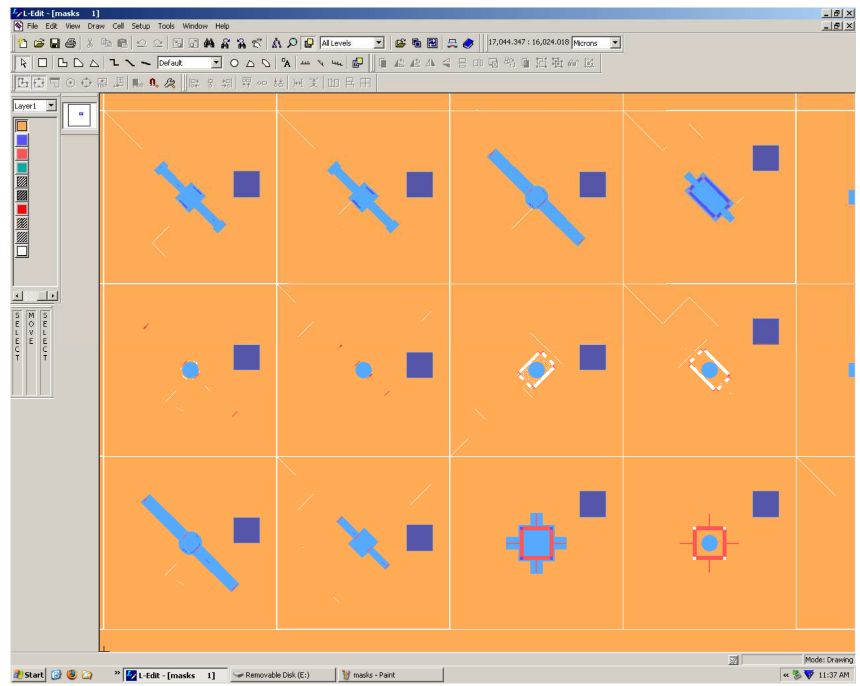
The closed loop stage design has been finalized and is been fabricated. This will give us sub-Å resolution on the distance axis.

Instrument Development

Collaboration with Walied Moussa (U of A – Mech Eng)

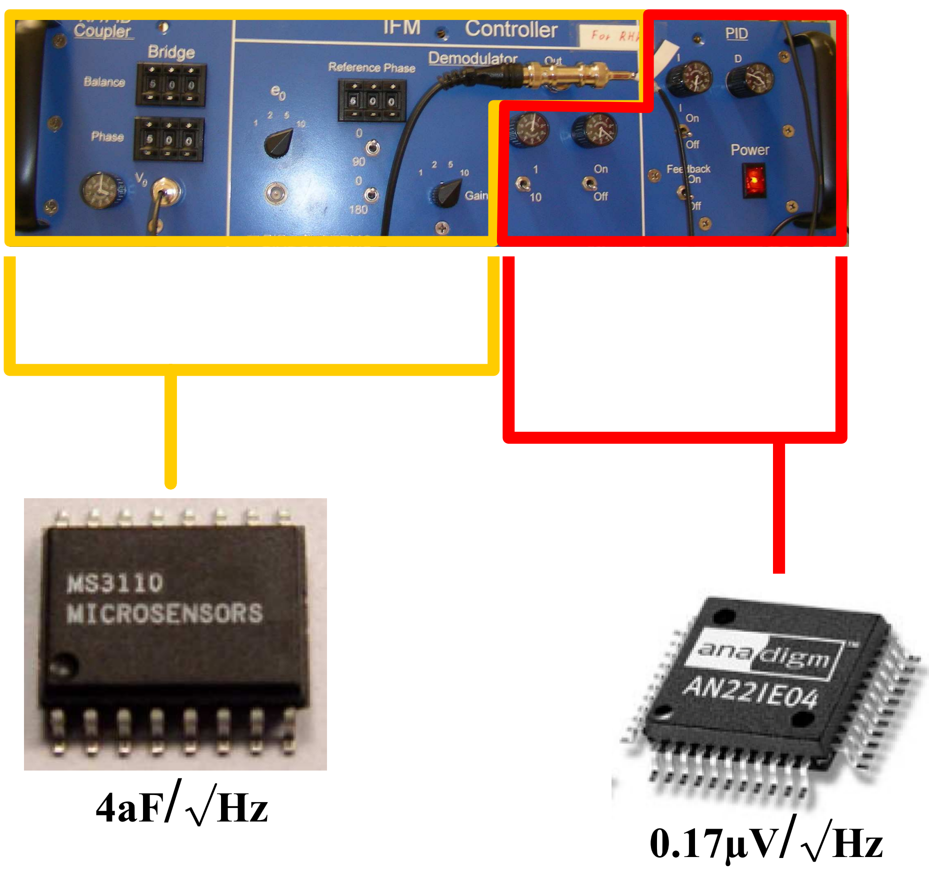
Else Gallagher is working on the first batch of prototype sensors. The mask design has been finalized as of last week. Each wafer will contain roughly 200 sensors divided between 12 separate designs.

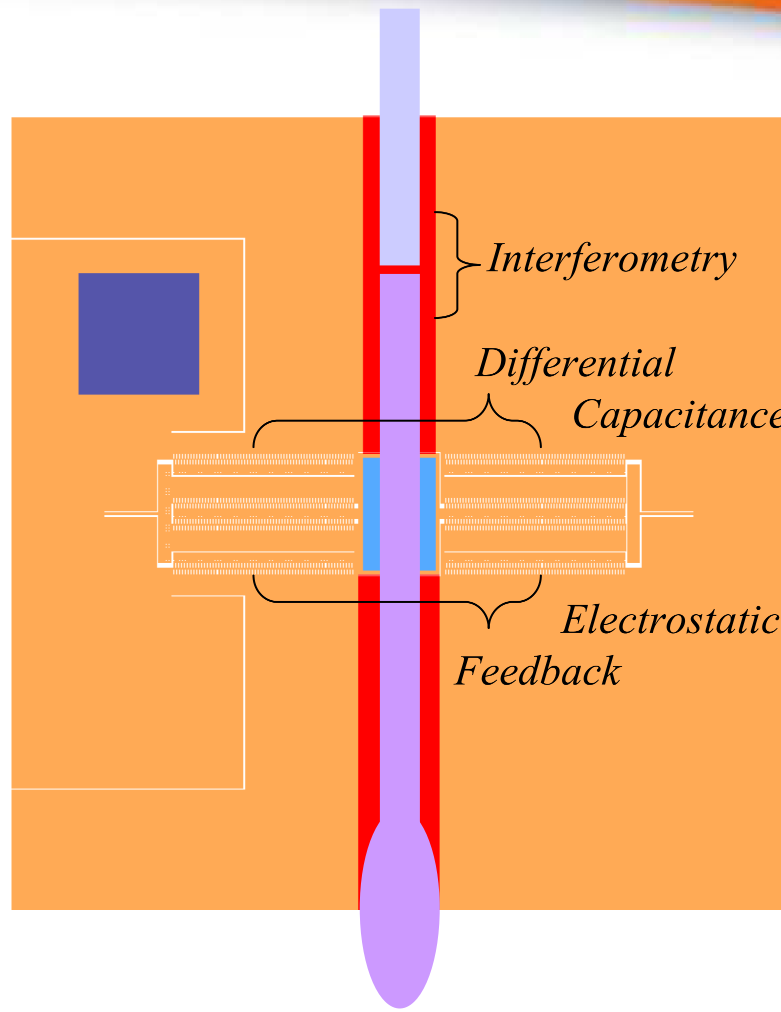




Instrument Development

We have proposed a sensor redesign that would allow us to monitor the system compliance in real time using our fibre interferometer.





Fitting the approach curve

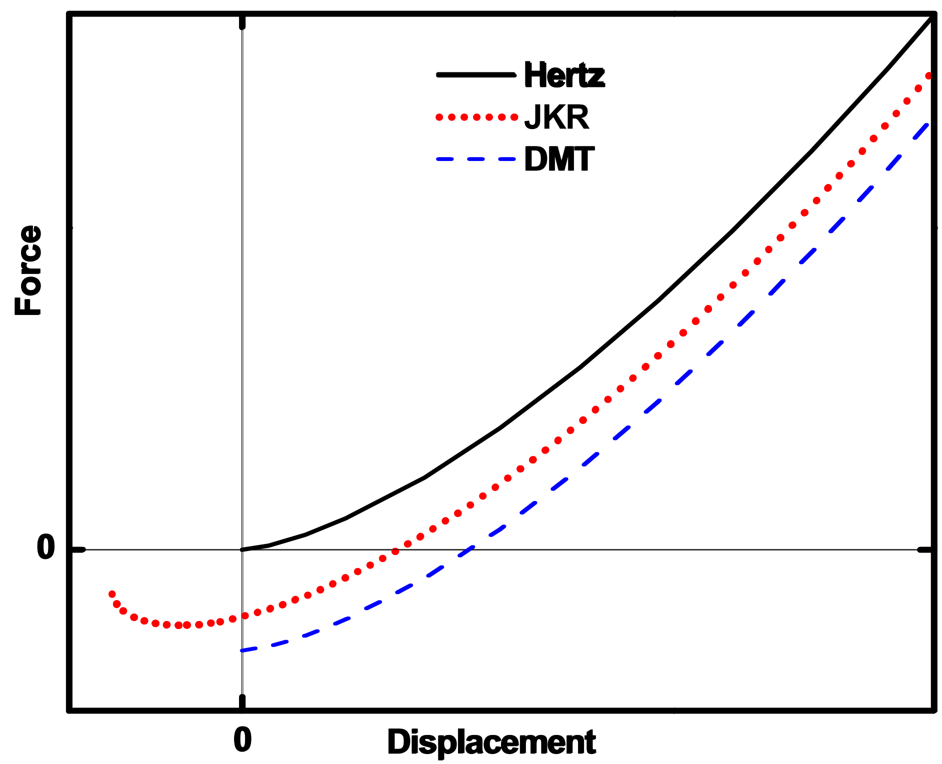
$$E^* = \left[\frac{1-\nu_s^2}{E_s} - \frac{1-\nu_i^2}{E_i} \right]^{-1}$$

$$F = \frac{4}{3} E^* R^{1/2} D^{3/2}$$

$$F = \frac{4}{3} E^* R^{1/2} D^{3/2} - 2\pi\gamma R$$

$$F(\alpha) = \frac{4}{3} \frac{E^* a^3}{R} - \sqrt{8\pi a^3 \gamma E^*}$$

$$D(a) = \frac{a^2}{R} - \sqrt{\frac{2\pi\gamma a}{E^*}}$$

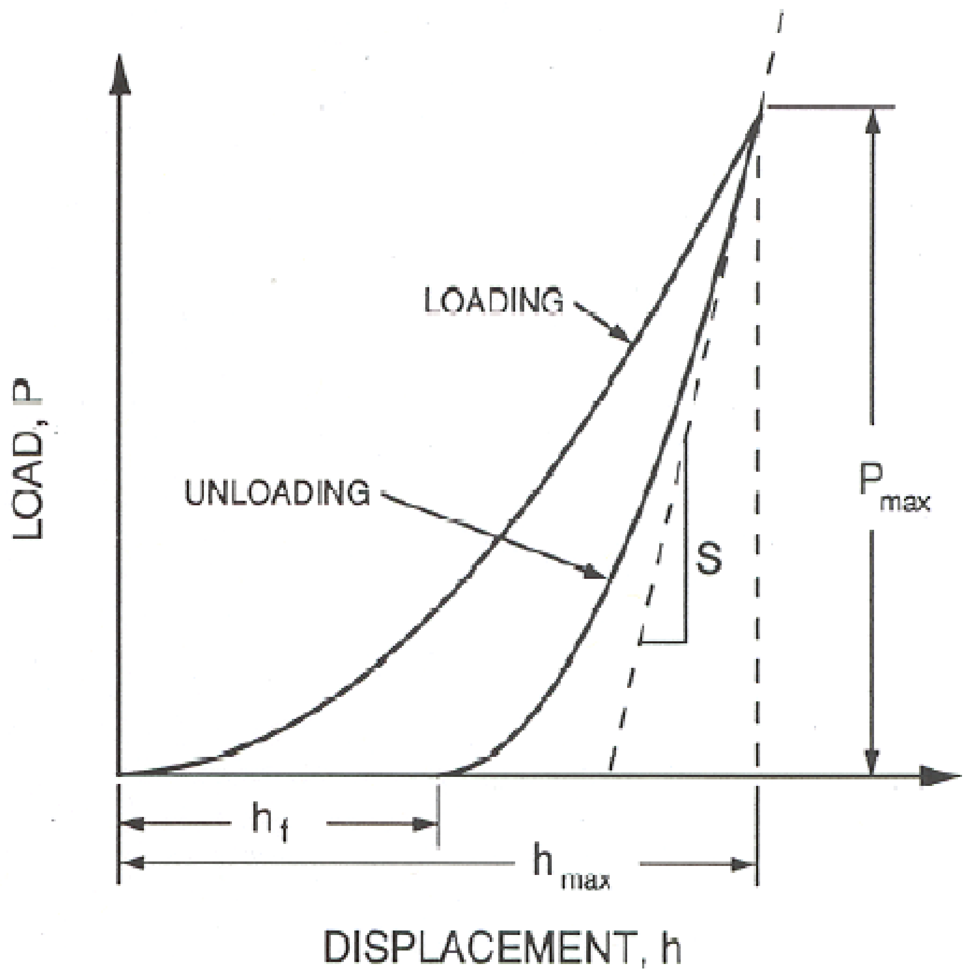


Fitting the retraction curve

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

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

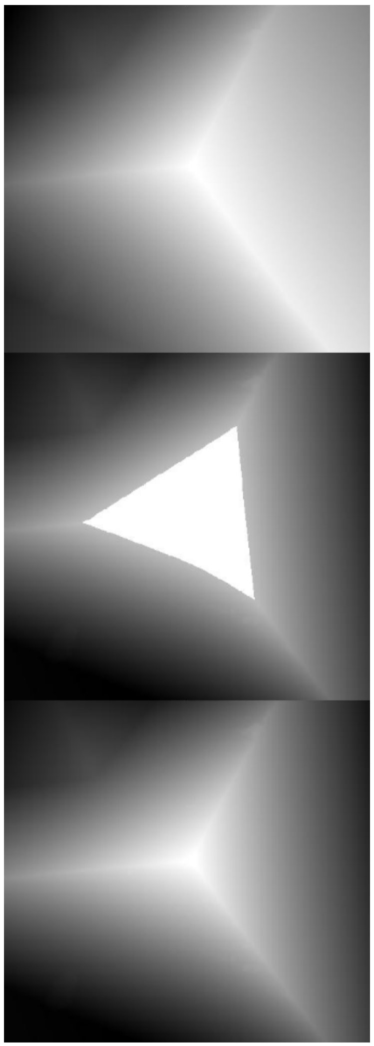
$$C_t = \frac{1}{S} + C_i = \frac{1}{2\beta E^*}\sqrt{\frac{\pi}{A}} + C_i$$



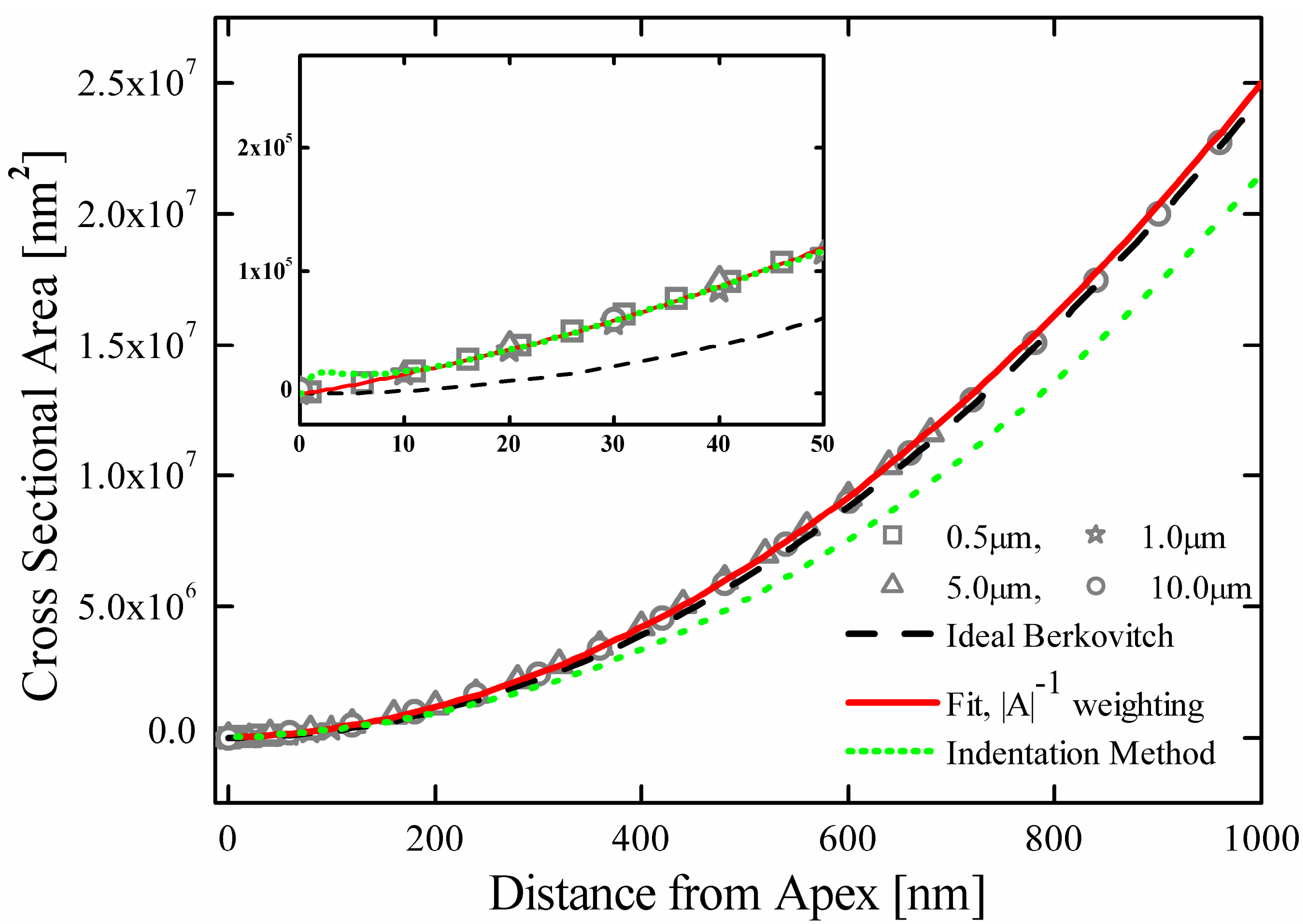
Both parameters are determined via nano-indentation experiments and are interdependent. *May not obtain a unique solution.*

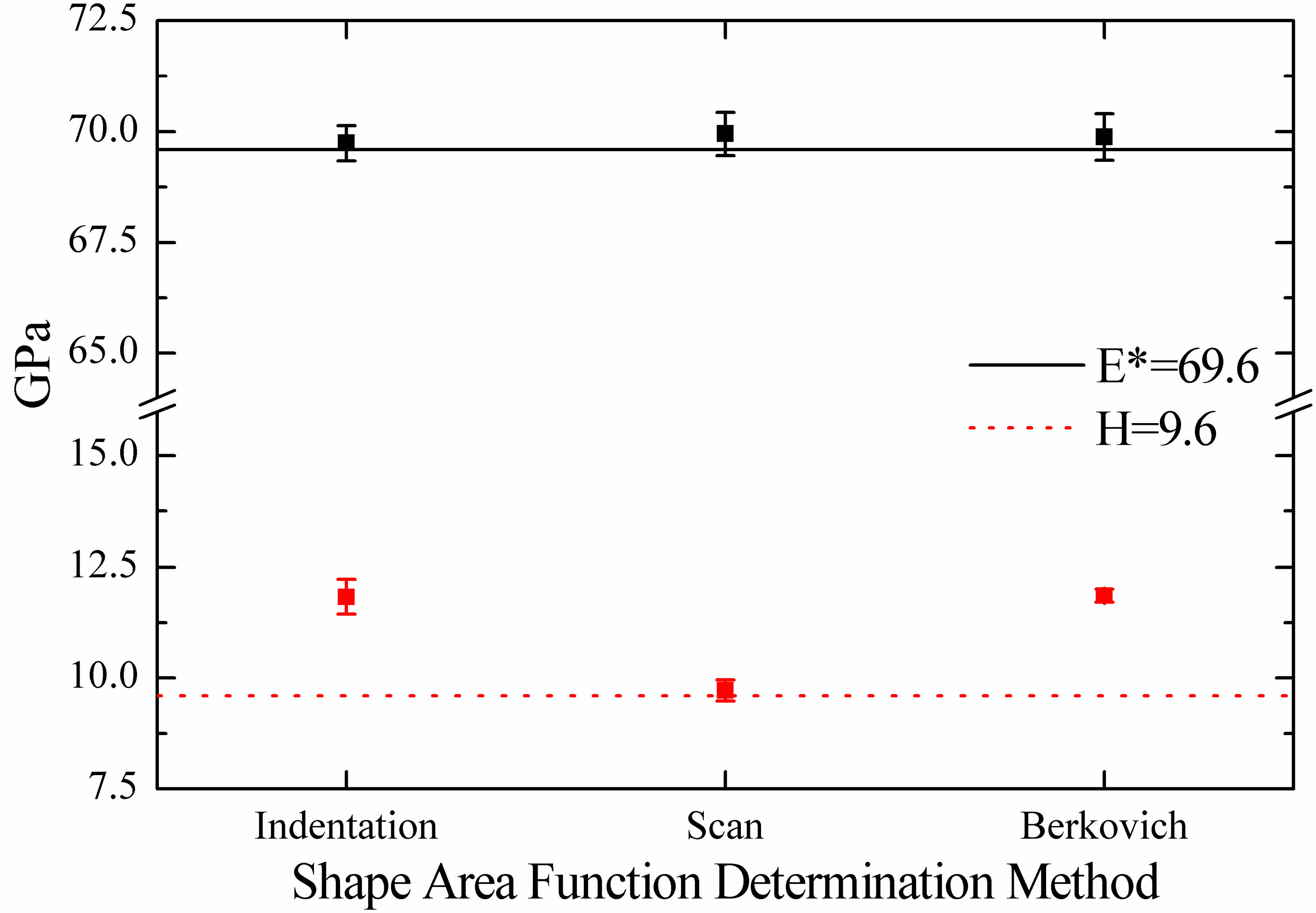
Oliver, W. C.; Pharr, G. M. *Journal of Materials Research* 1992, *7*, 1564.

Direct AFM determination of Tip Shape-Area function



Shape-area function




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

Description of a worn indenter

Cone

$$A(d)=\pi\tan^2\alpha\cdot d^2$$

Cone + Sphere (floating)

$$A(d)=\pi\tan^2\alpha\cdot d^2+2\pi r\,d$$

Cone + Sphere (tangential)

$$A(d)=\pi\tan^2\alpha\left[d+r\left(\csc\alpha-1\right)\right]^2$$

Cone + Sphere (tangential + harmonic average)

$$A(d)=\pi\tan^2\alpha\cdot d^2+\pi r\,d+\frac{\pi}{4}r^2\cot^2\alpha$$

Cone + Sphere (piecewise combination using filtering techniques)

$$A(d)=\frac{\pi}{\left(10^3 r^3\cot^{63/33}\alpha+31^3 d^3\right)^2}\cdot\left\{10^3 r^4\cot^{63/33}\alpha\sin\left(1.4\sqrt{d/r}\right)+31^3 d^3\tan\alpha\left[d+r\left(\csc\alpha-1\right)\right]\right\}^2$$

Hyperboloid of revolution

$$A(d)=3\pi\tan^2\alpha\cdot d^2+2\pi r\,d-2\pi\left(r^2 d^2+\tan^4\alpha\cdot d^4\right)^{1/2}$$

Accuracy of fit estimators for Eq. (5) to (9).

Model (Eq.)	R+r [nm]	α [°]	R^2	χ^2	μ_{RR}
Sphere + Cone (5)	195±1	70.33†	0.99998	185	-0.023±0.002
Rounded Cone (6)	288±4	70.34†	0.99979	1729	-0.131±0.089
Harmonic Average (7)	280±4	70.34†	0.99979	1729	-0.130±0.089
Piecewise Combination (8)	373±2	69.90†	0.99996	307	0.045±0.008
Hyperbolic (9)	215±2	70.33†	0.99993	603	-0.009±0.007
AFM inspection	230±5	70.38±0.02	NA	NA	NA

† Calculated error < 0.01

R^2 is the square of the correlation between the values predicted by the fit and the experimental values. χ^2 is the sum of the squares of the residuals; the relatively large value of χ^2 reflects the fact that the fits were carried over a wide range of values for the independent variable. μ_{RR} is the mean value for the relative residuals showing estimation errors approximate Gaussian distribution. Note that in all cases the value for the radius has not been corrected for the value of the AFM tip radius.

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To God Almighty

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