Nano Indenter XP System

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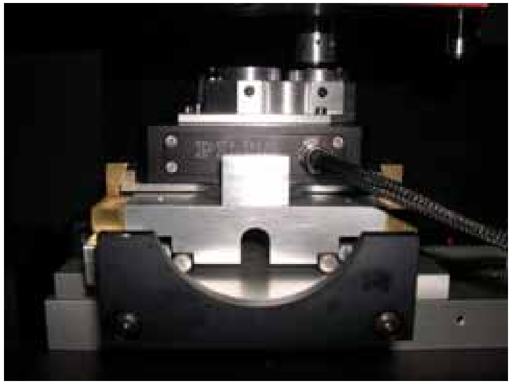
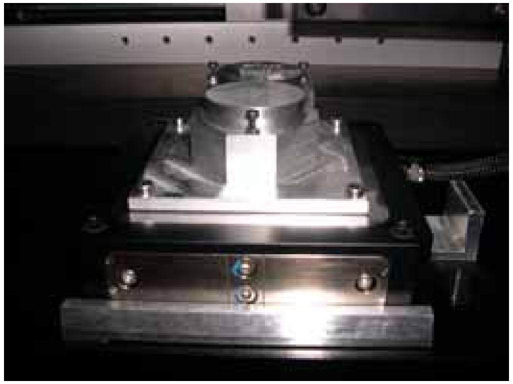
2005.06.15

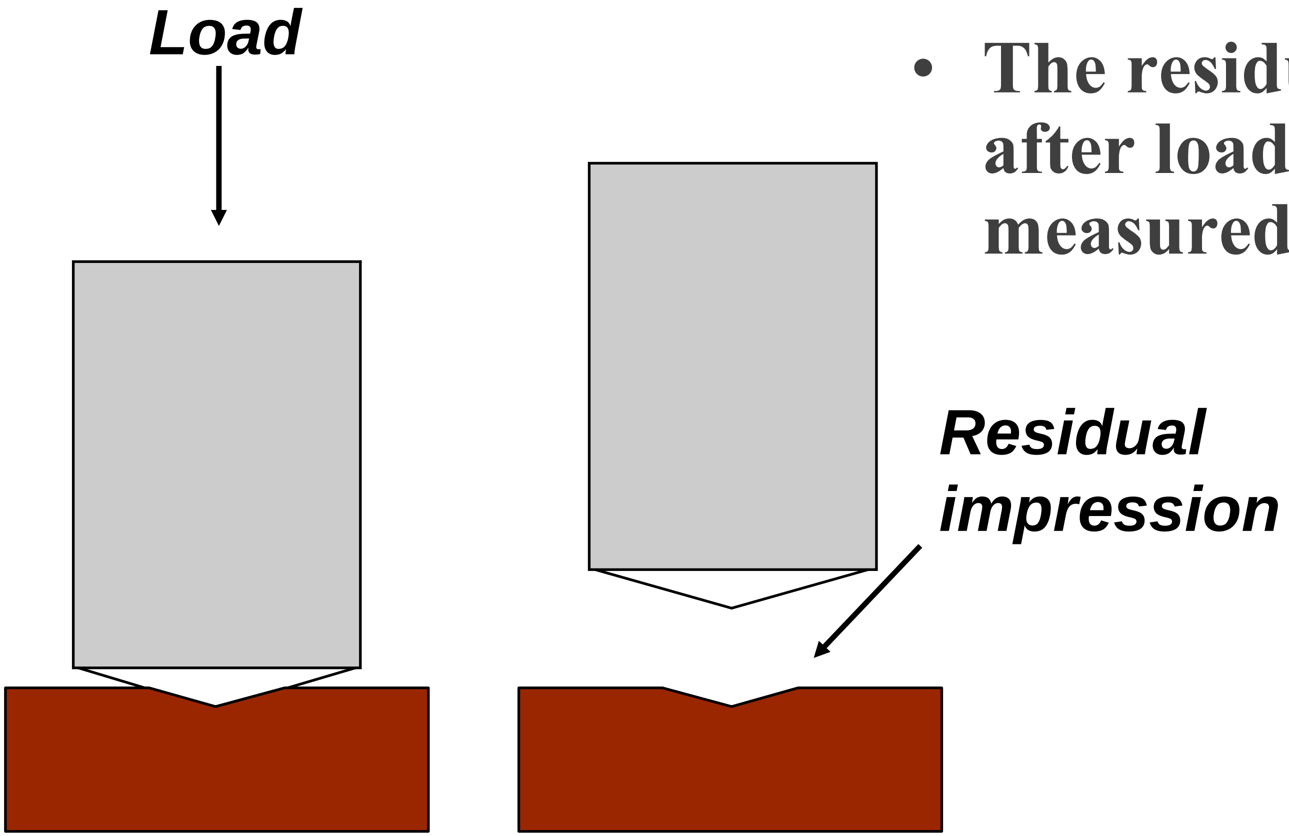
Nano Indenter Introduction





Theorem

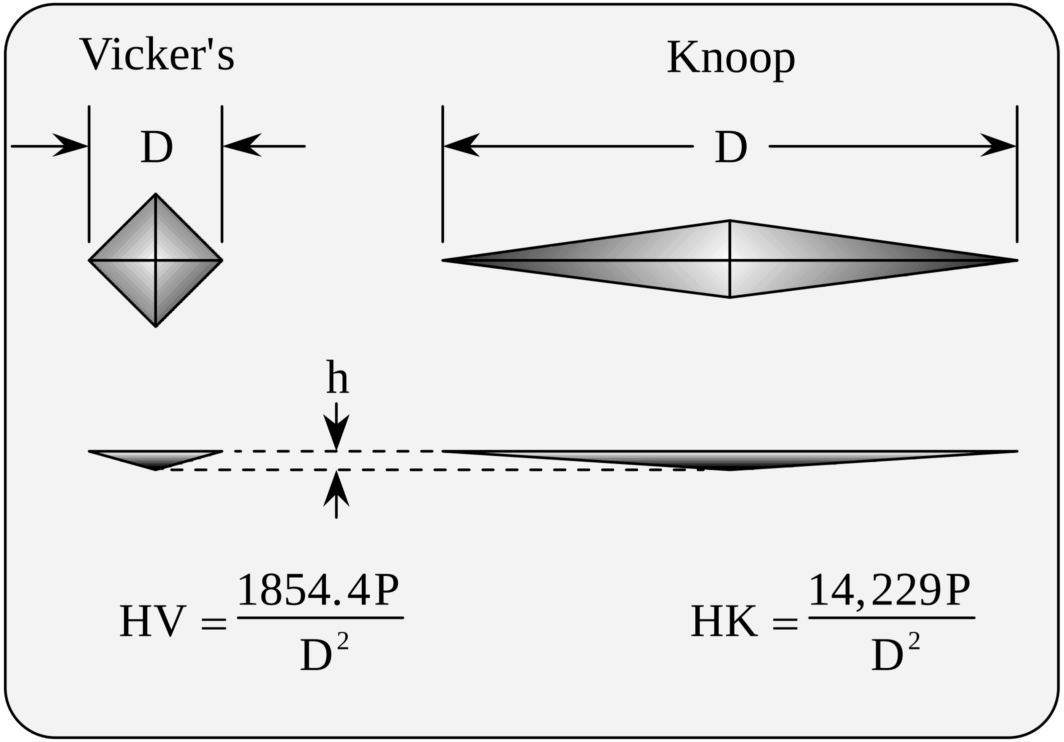
Conventional Microhardness



- The residual impression after load removal is measured as hardness

Conventional Microhardness

- **Diagonal, *D*, is measured optically after removal of load**
- **Vickers: $D = 7h$**
- **Knoop: $D = 30.5h$**

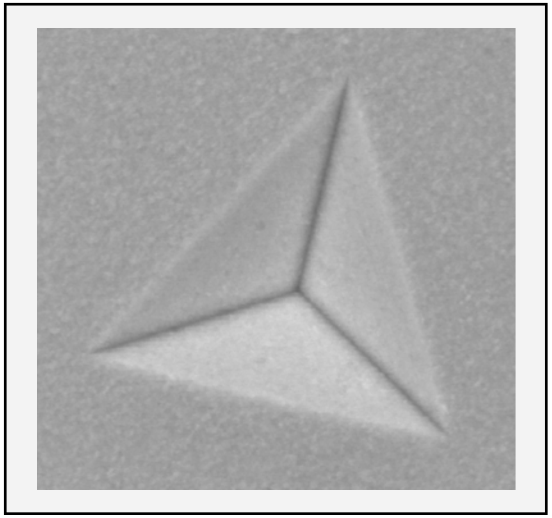


Berkovich Geometry Diamond Tip

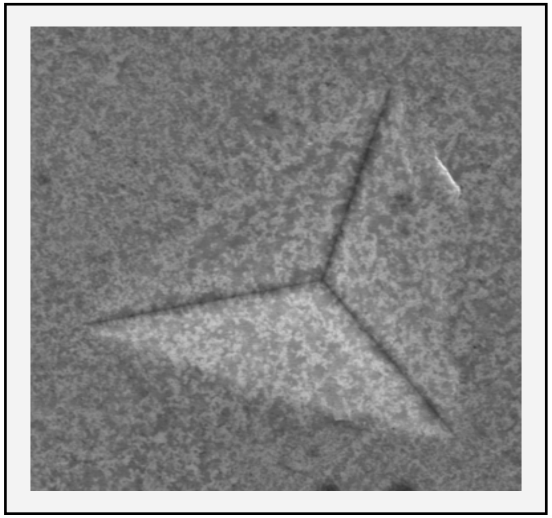
- Three-sided geometry gives a sharp tip
- Residual indentation shape is material dependent

Fused Silica

Nickel



Aluminum alloy



What is Depth-Sensing Indentation

- Apply a specific,quasi-static or dynamic load-time history on a diamond indenter
- Measure the displacement-time response
- Ues the data to extract certain mechanical properties based on analytical models

Hardness & Young's Modulus

- Hardness is the mean pressure the material will support
- Young's modulus is calculated from the composite response modulus, E_r
- Though not shown explicitly here, both H and E require load, depth *and stiffness* for calculations

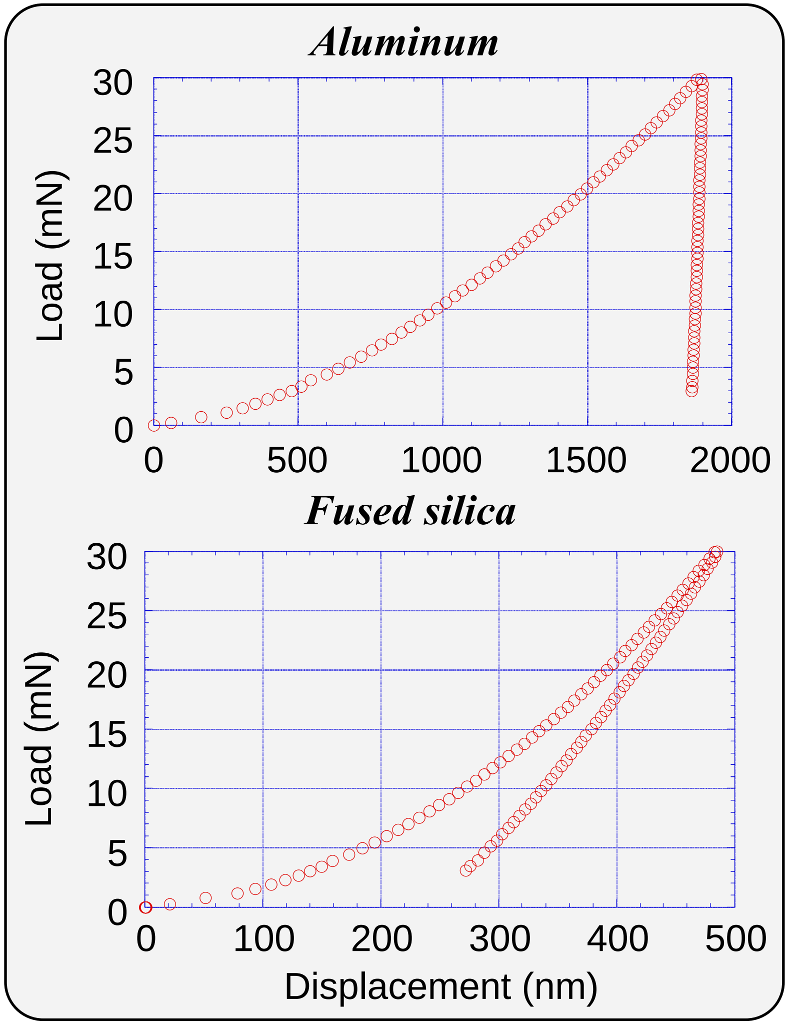
$$H = \frac{P}{A}$$

$$E_r = \frac{S\sqrt{\pi}}{2\beta\sqrt{A}}$$

Load-Displacement Behavior

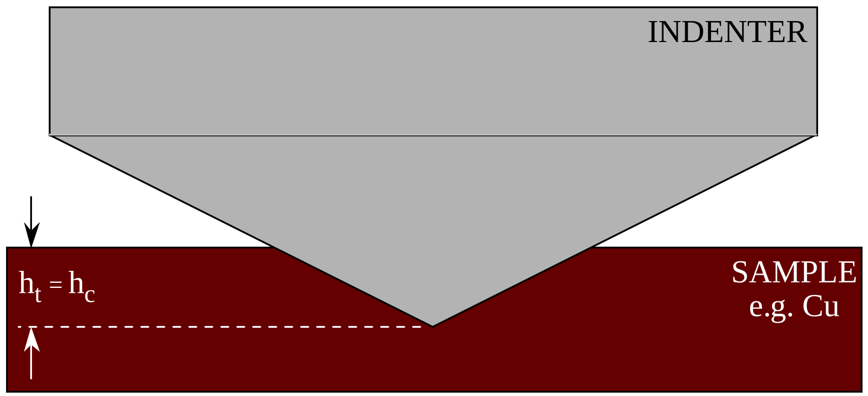
- **Aluminum, typical of soft metallic behavior, shows very little displacement recovery upon unloading**

- **Fused silica, typical of ceramic behavior, shows large elastic recovery upon unloading**

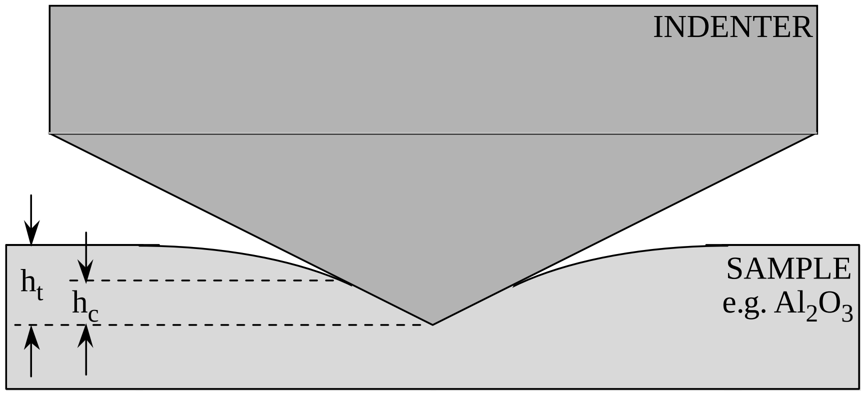


Elastic/Plastic Indentations

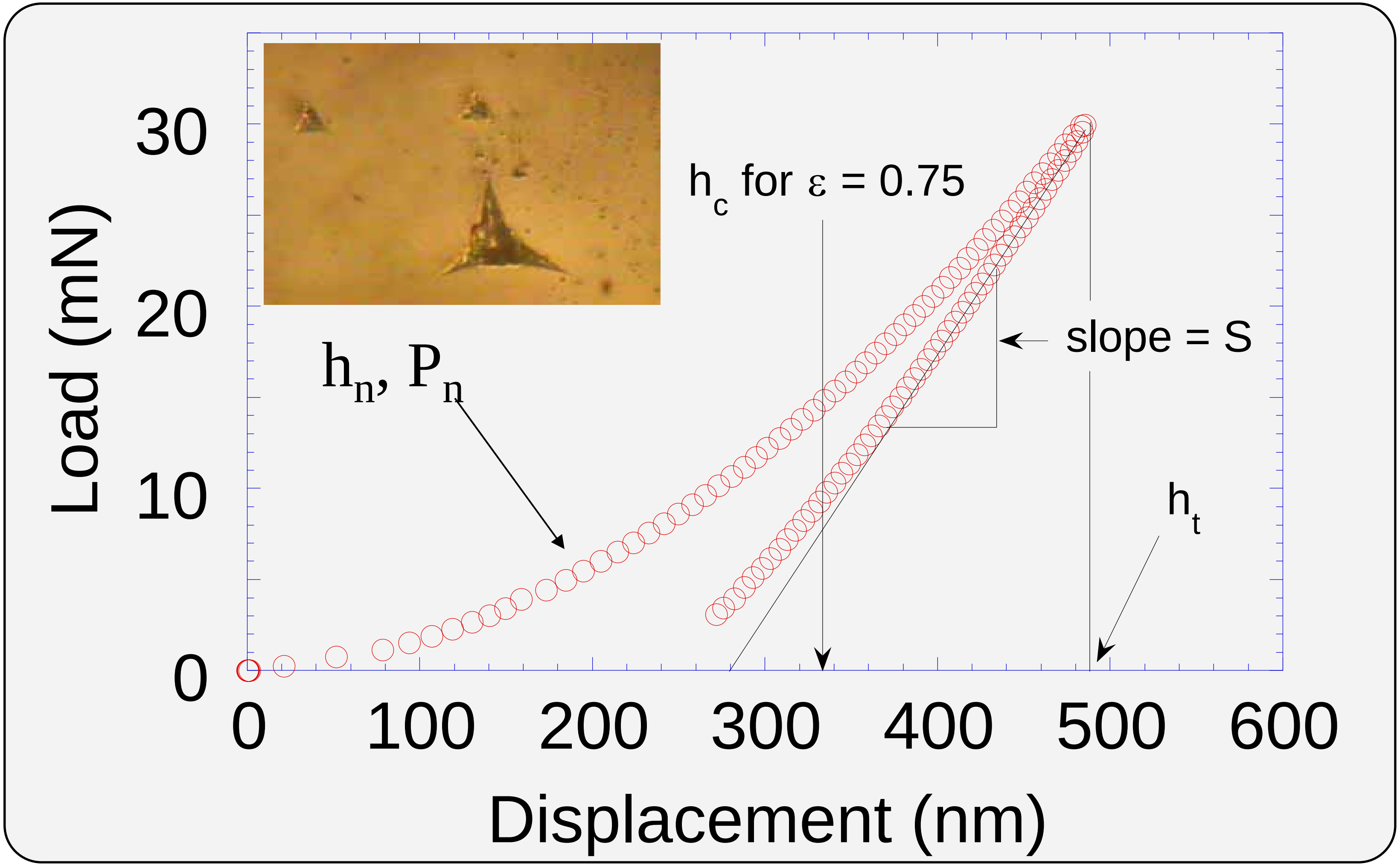
- **For an ideally plastic indentation, $h_c \approx h_t$**



- **For an elastic/plastic indentation, $h_c < h_t$**



Stiffness From Unloading



Contact Depth and Stiffness

- **The unloading curve follows a power law**

$$P = \alpha h_t^m$$

- **Contact stiffness is the slope of the unloading curve**

$$S = \left.\frac{dP}{dh_t}\right|_{P_{\max}}$$

- **Contact depth is determined from the displacement, load, and contact stiffness**

$$h_c = h_t - \varepsilon \frac{P_{\max}}{S}$$

Contact Area (the tip function)

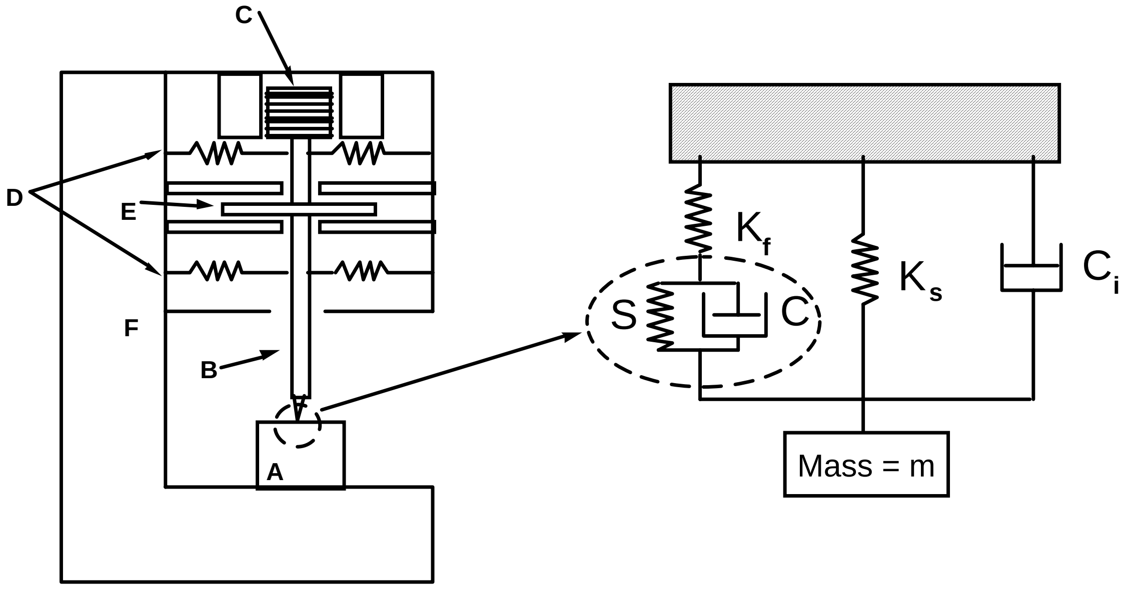
- The "tip function" for the ideal Berkovich tip

$$A = 24.56 h_c^2$$

- Experimental tip function
  - Arbitrary form Coefficients determined experimentally

$$A = 24.56 h_c^2 + \sum_{i=0}^{7} C_i h_c^{\frac{1}{2^i}}$$

Dynamics Mode

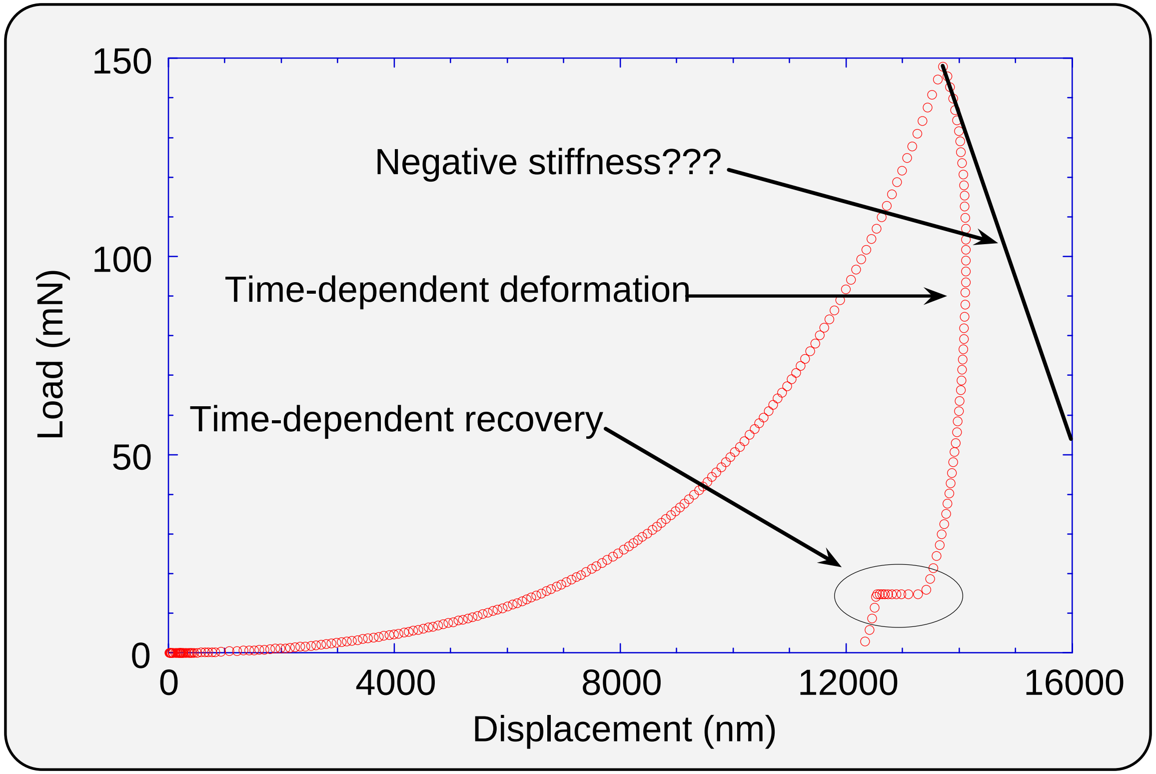


A. Sample
B. Indenter Column; mass=m
C. Load Application Coil
D. Indenter Support Springs; Stiffness=K_s
E. Capacitive Displacement Gauge; Damping Coefficient=C_i
F. Load Frame; Stiffness=K_f=1/C_f

Problems with Time-Dependence

- "Conventional" stiffness determination unreasonable
- Large amounts of time-dependent deformation
- Large time-dependent recovery

Polymer thin film



CSM

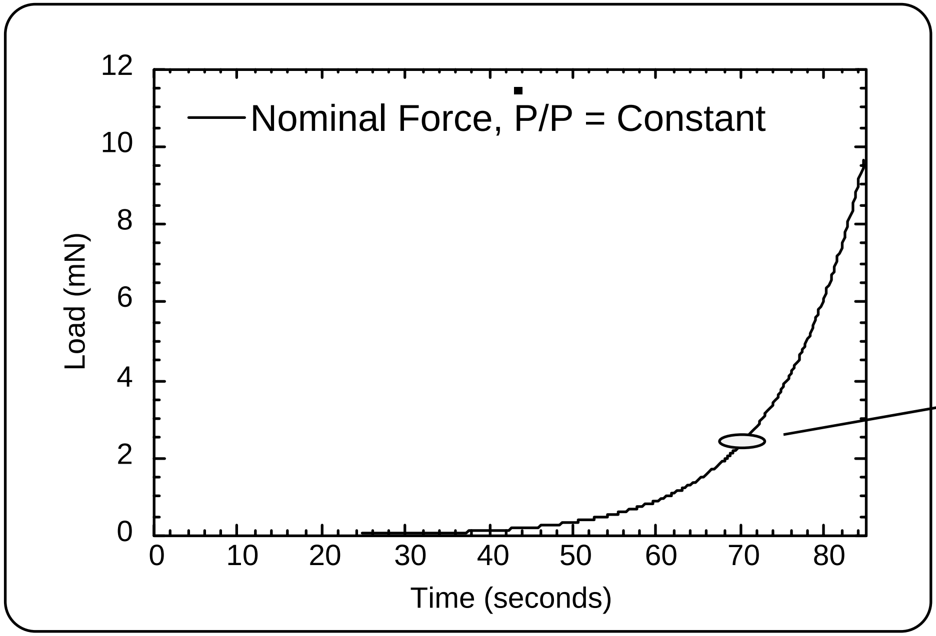
CSM

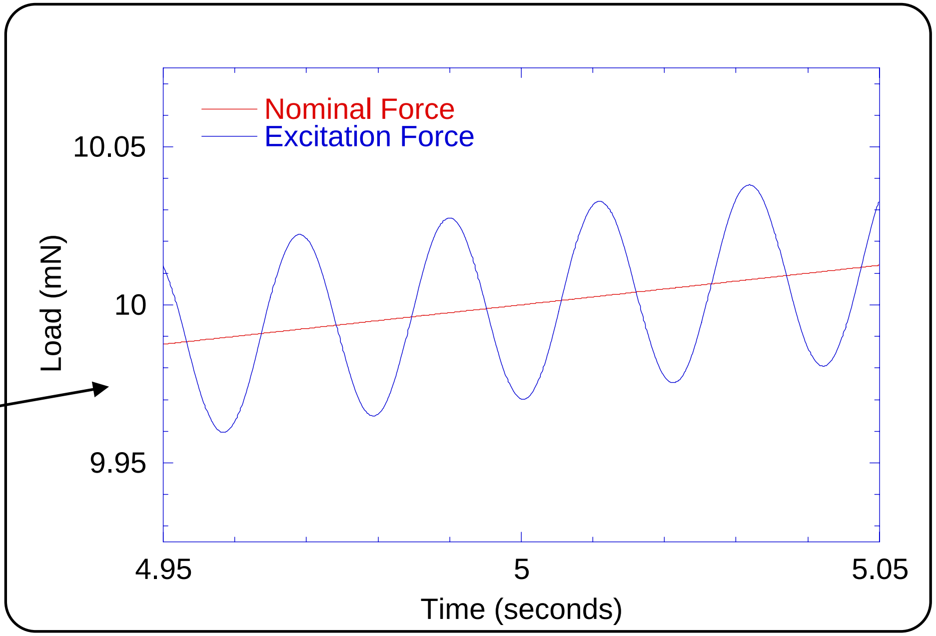
- **Patented Method for the Continuous Determination of the Elastic Stiffness of Contact Between Two Bodies**
- **“Frquency - Specific, Depth-Sensing Indentation”**

Benefits of CSM

- Properties vs. depth — avoiding substrate effect
- Controllable strain rate
- Surface contact determination
- Viscoelastic materials and properties

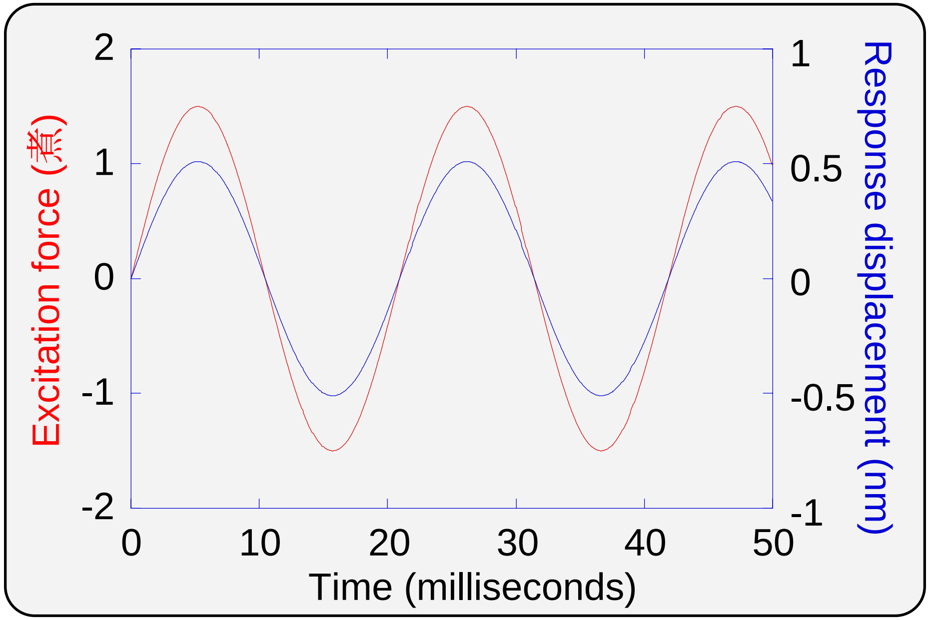
CSM - Force Oscillation



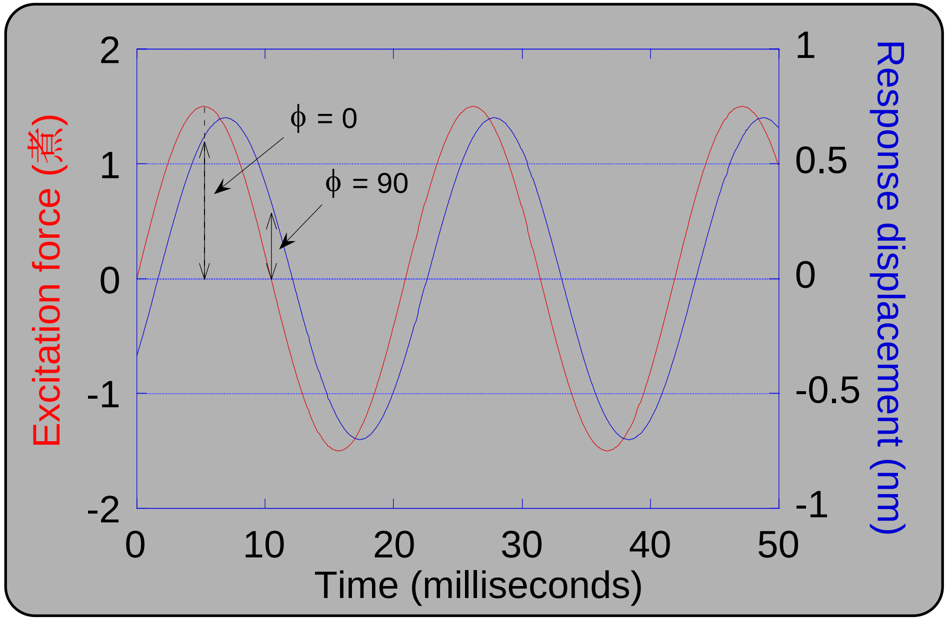


CSM - Linear Elastic Materials

Elastic



Viscoelastic



CSM Calculations

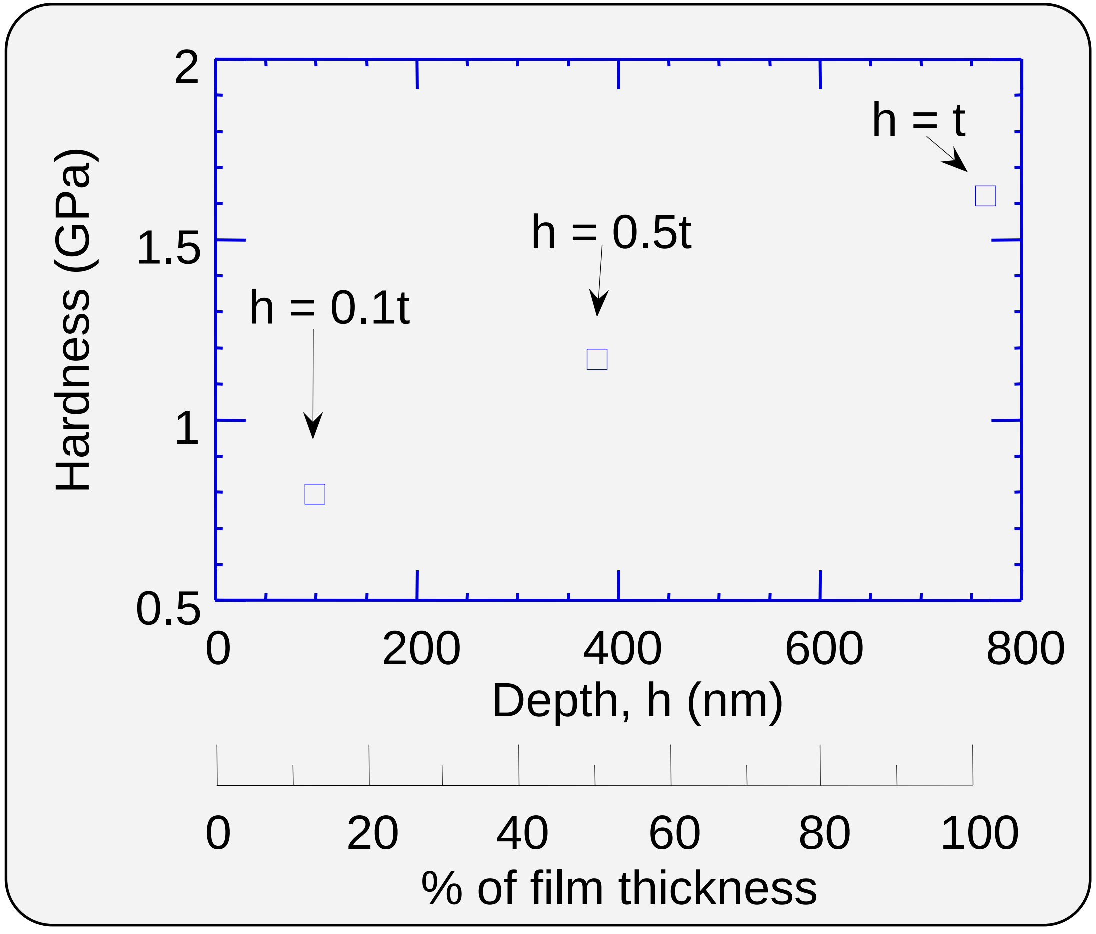
- **The phase and amplitude of the material's response to the force oscillations are characteristic of the stiffness of the contact as well as the damping of the material.**

$$S = \frac{F}{h}\cos\phi$$

Thin Film Testing without CSM

- **Unloading data**
  - **Unloads at 0.1, 0.5 and 1.0 times thickness**
  - **Values imply film hardness varies linearly with depth**

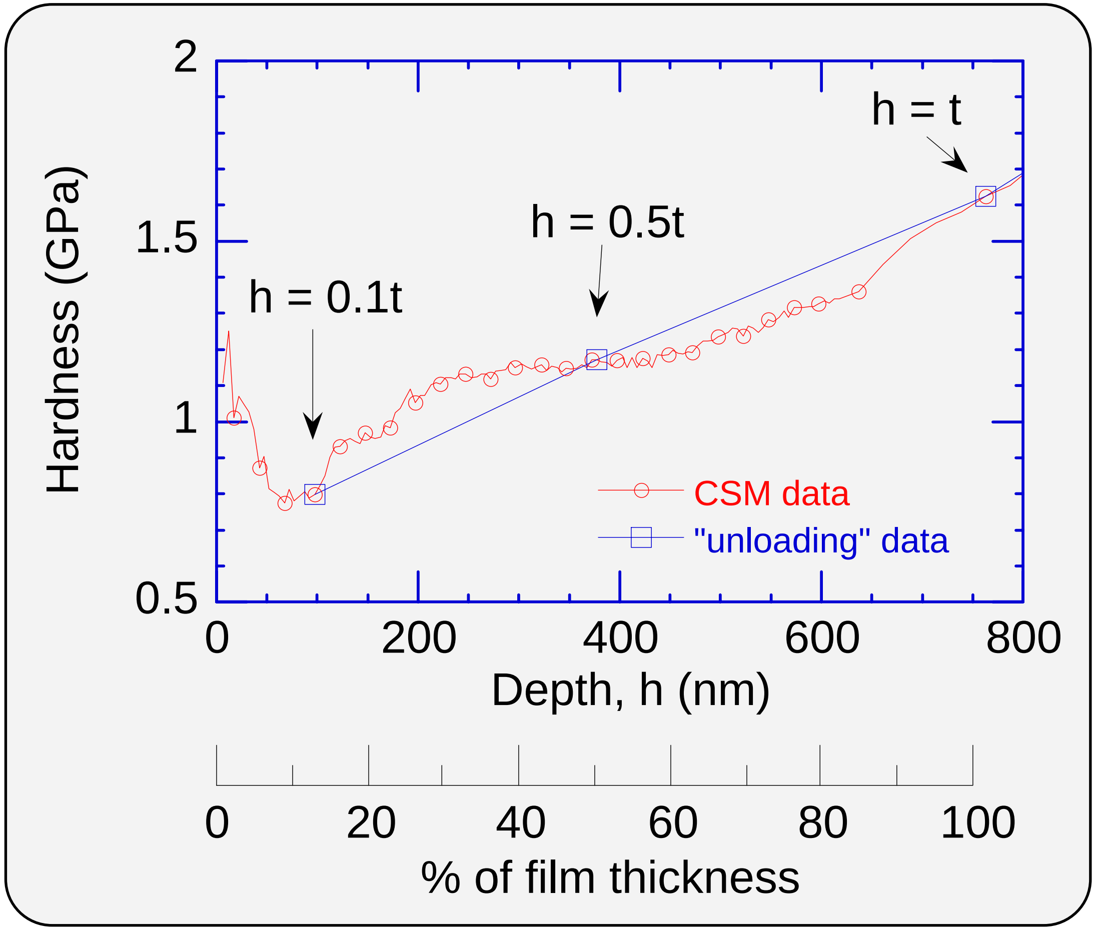
Al film on glass



Thin Film Testing with CSM

- **CSM data**
  - **Continuous data with thickness (i.e., depth) shows true character of film**

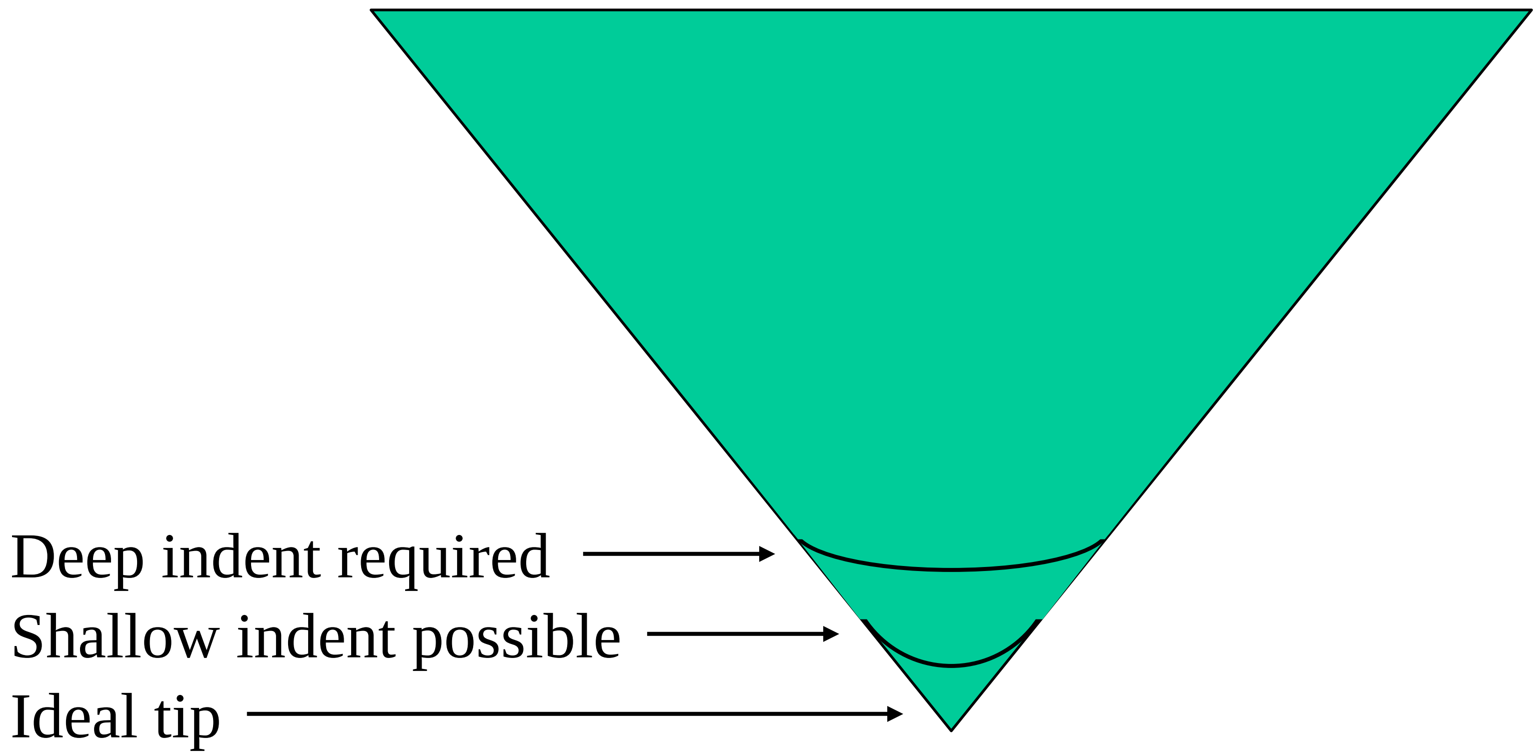
Al film on glass



Berkovich Diamond Tips

AccuTip™ Berkovich Diamond Tips

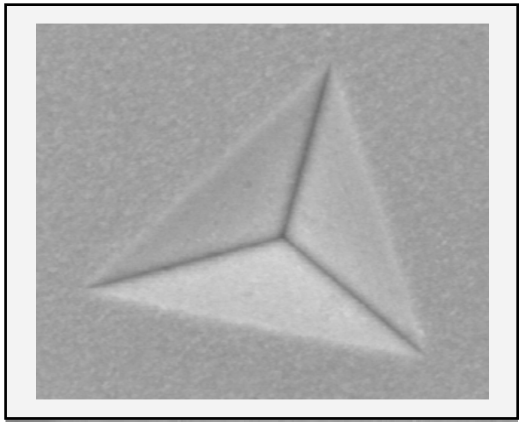
- **The sharper the tip, the shallower an indent can be made to give reliable hardness values**

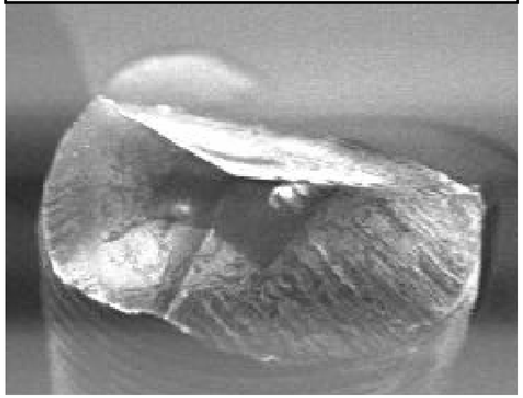


AccuTip™ Berkovich Diamond Tips

- **Old diamond tips**
  - **Face angles fairly consistent, but not known with any precision**
  - **Tip radius typically ~ 100 – 150 nm**
- **New AccuTip™ Berkovich diamond tips**
  - **Face angles known to ± 0.025°**
  - **Tip radius ≤ 50 nm (typically ~ 40 nm)**

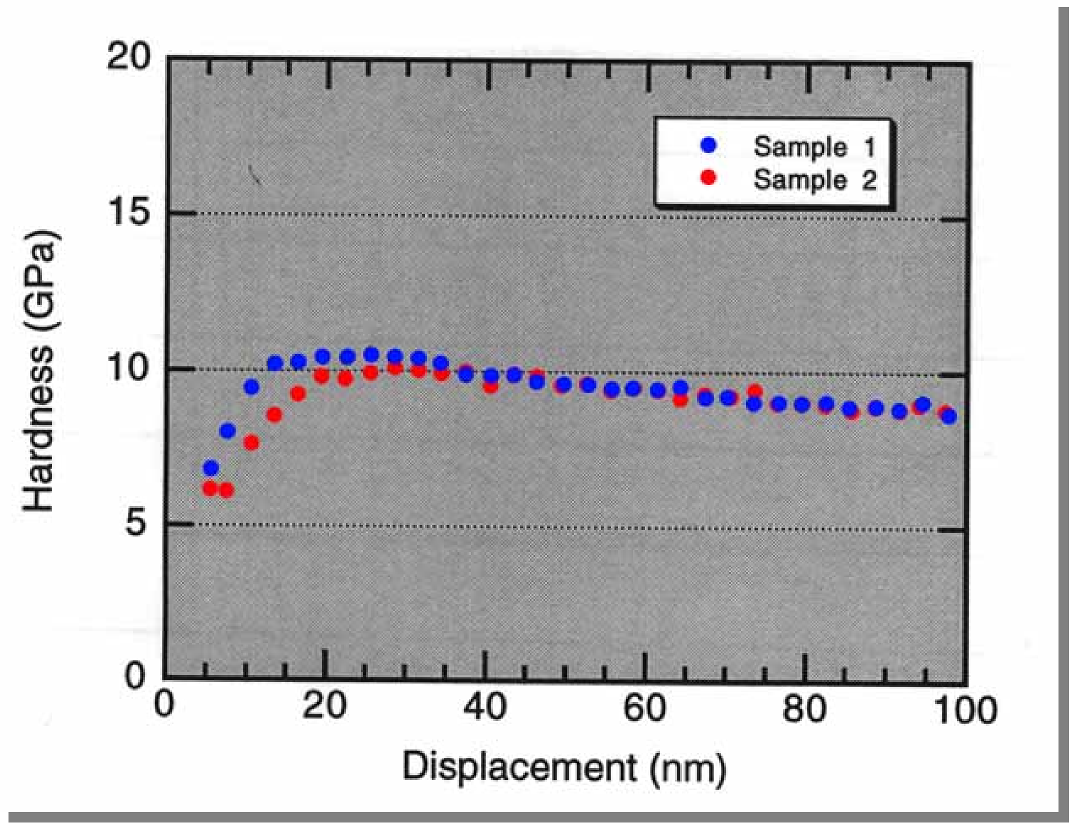
Nickel





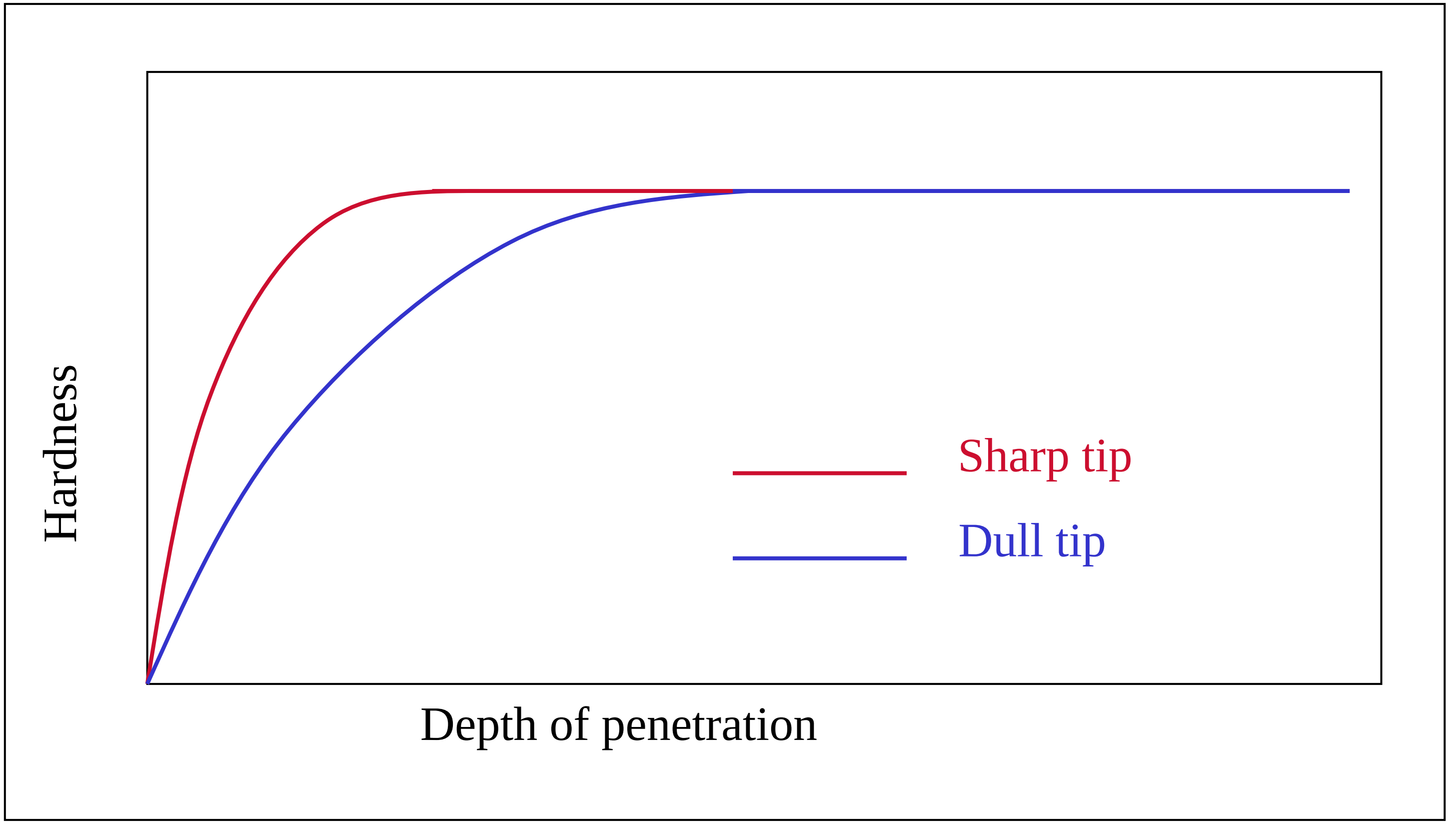
Old Berkovich Diamond Tip

- Two different DLC films, both 20 nm thick
- No significant difference between them measured
- Plasticity does not begin at a shallow enough indent depth to see a significant effect of the film in the measurement



Tip Sharpness

- **A sharp tip is required for making hardness measurements at very shallow indentation depths**



Systems

Nano Indenter® XP

Nano Indenter® XP Specifications

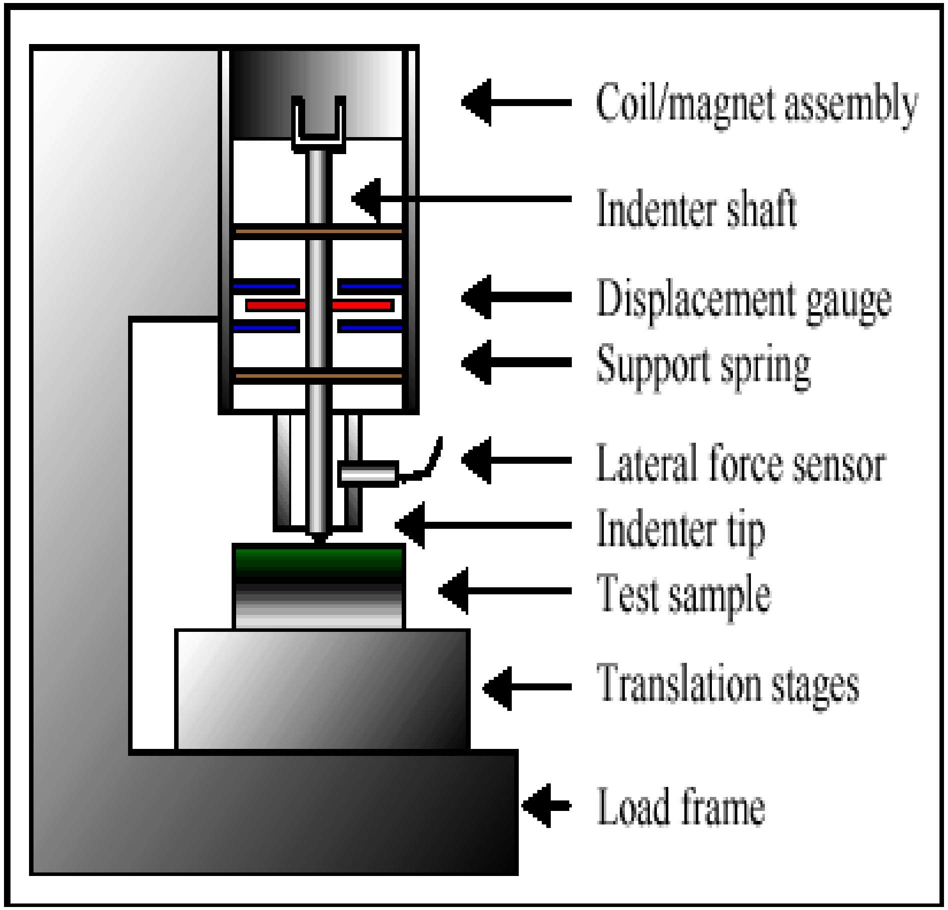
Displacement resolution…………...	<0.01 nm
Total indenter travel …….………..	2 mm
Max indentation depth …………..	>500 μm

Loading Capability

• Maximum load ………………..	500 mN (50.8 gm)
• Max load - high load option …	10 N (1 kg)
• Load resolution ……………….	50 nN (5.1 μgm)
• Load resolution – high load …	50 nN (5.1 μgm)
• Contact force …………………..	<1.0 μN
• Load frame stiffness…………...	≈ $1x10^7$ N/m

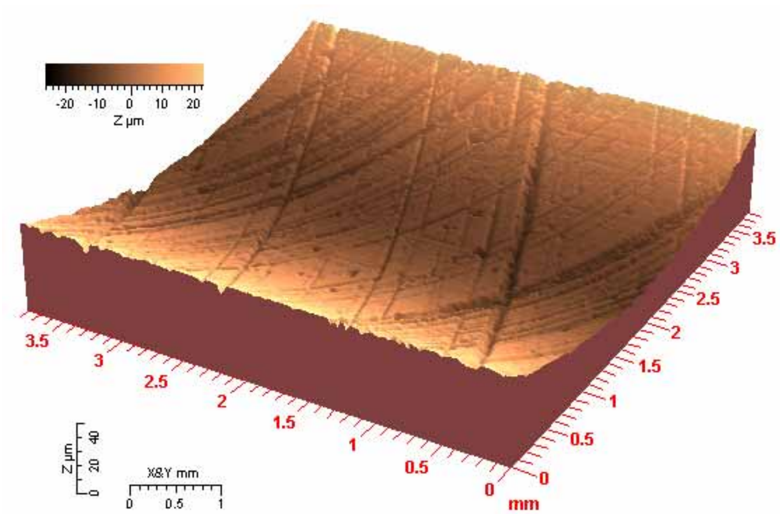
Hardware Options

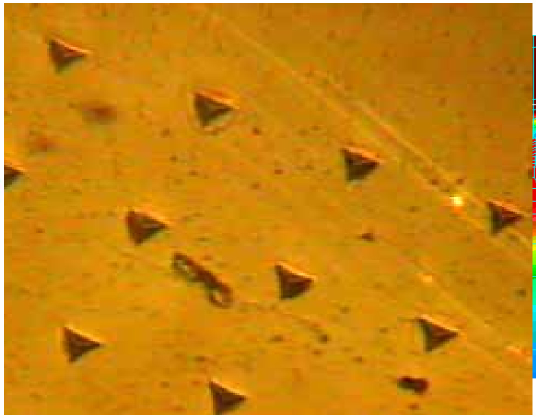
- DCM
- CSM
- LFM
- Tips
- AFM
- Nano Vision
- 100x Objective
- 150x Objective

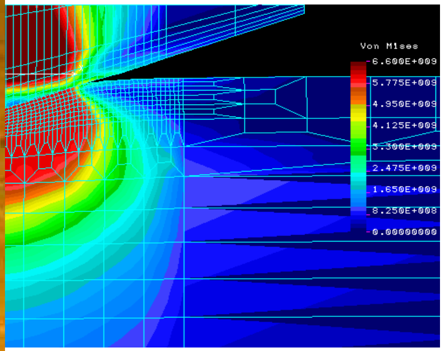


Software Options

- Explorer software for Testworks v. 4
- 3D software
- Virtual Indenter : FEM modeling

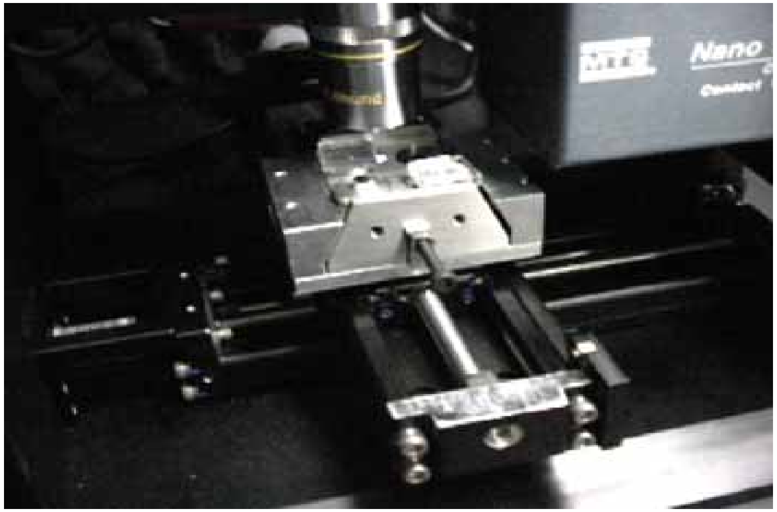


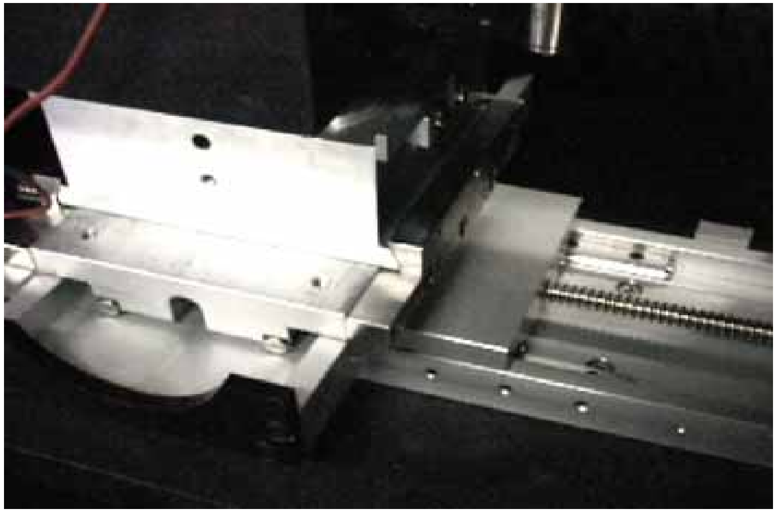




Motion System Options

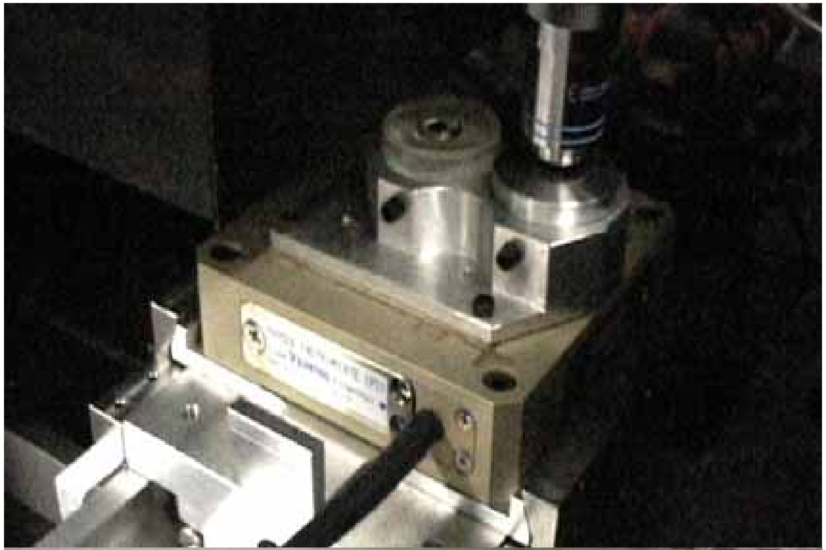
- High stiffness, rotary encoders, low profile, large footprint (crossed roller slide w/ rolled lead screw & anti-backlash nut)
- Travel:
  - XP : 90 mm x 100 mm
  - XPW: 225 mm x 300 mm
  - SA2: 35 mm x 30 mm
- Resolustion (X-Y Direction)
  - XP : 0.5 mm
  - XPW: 0.5 mm
  - SA2: 0.5 mm
- Accuracy: 1mm





More on the Nano Positioning Stage

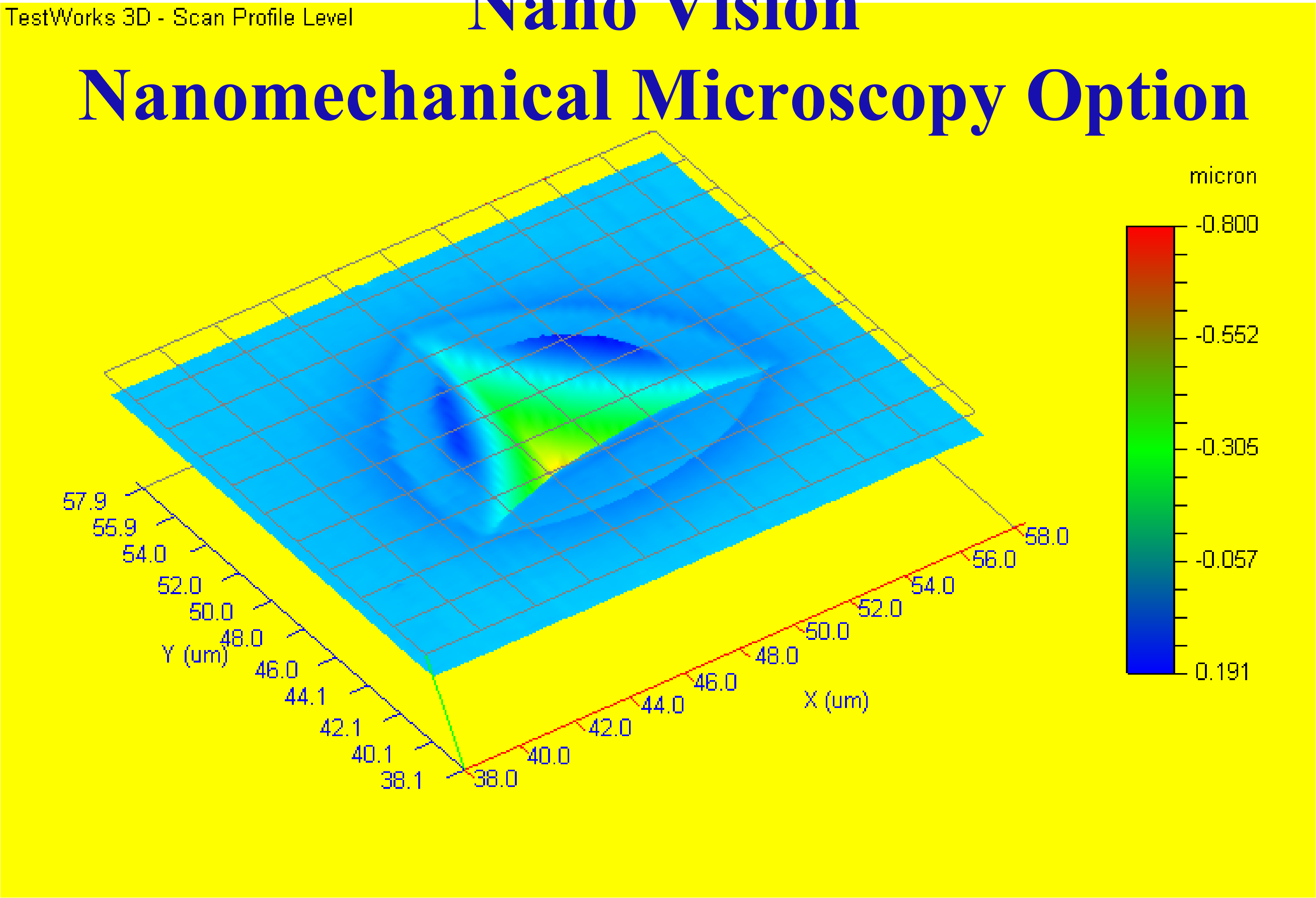
- Integrated nano positioning stage, “Piggyback” on standard XP motion system
- Two samples can be mounted, both accessible
  by XP
- Travel: 100 mm x 100 mm
- Resolution: 2 nm
- Accuracy: 0.01 %



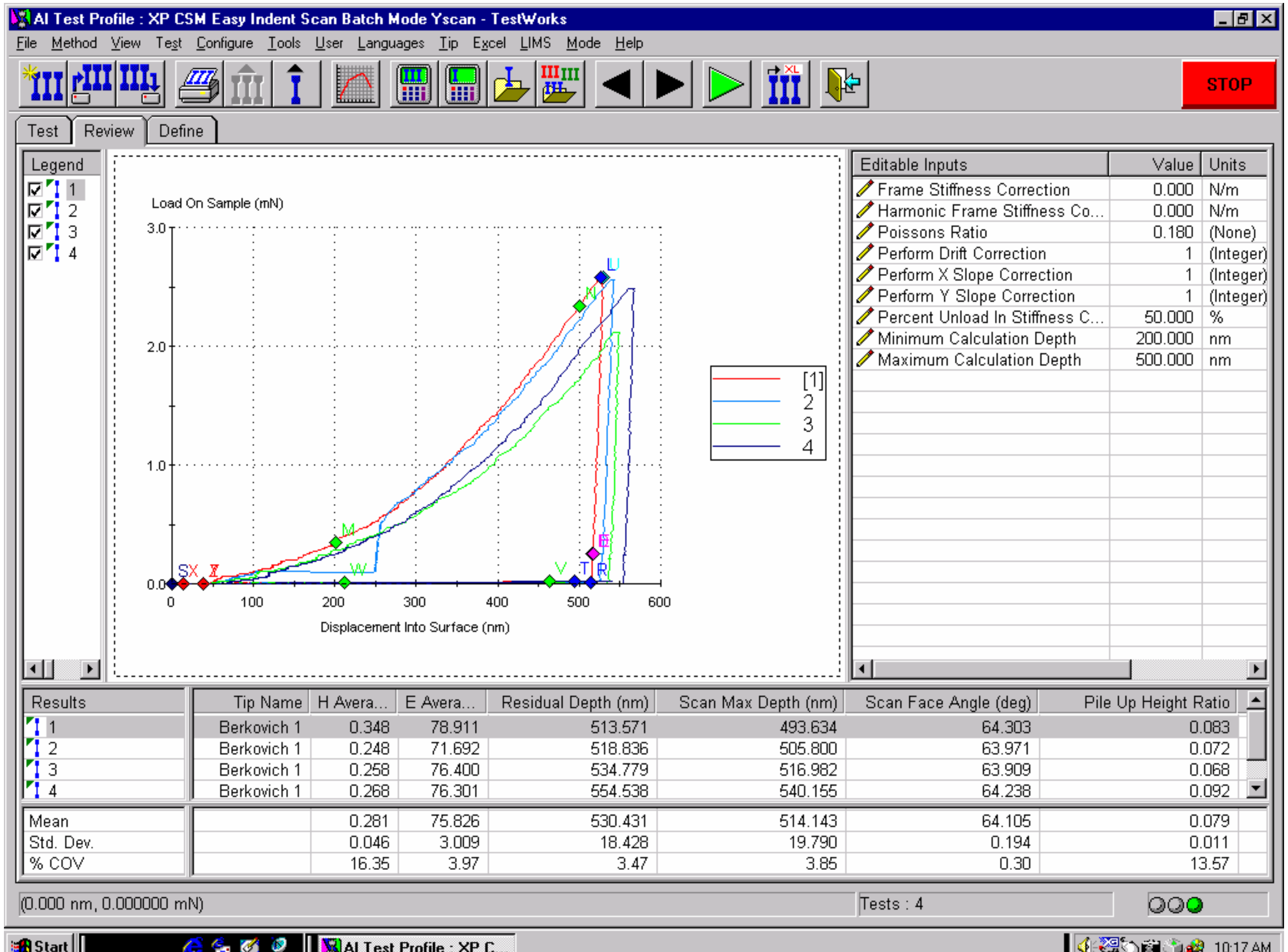
Nano Vision

Nano Vision

Nanomechanical Microscopy Option



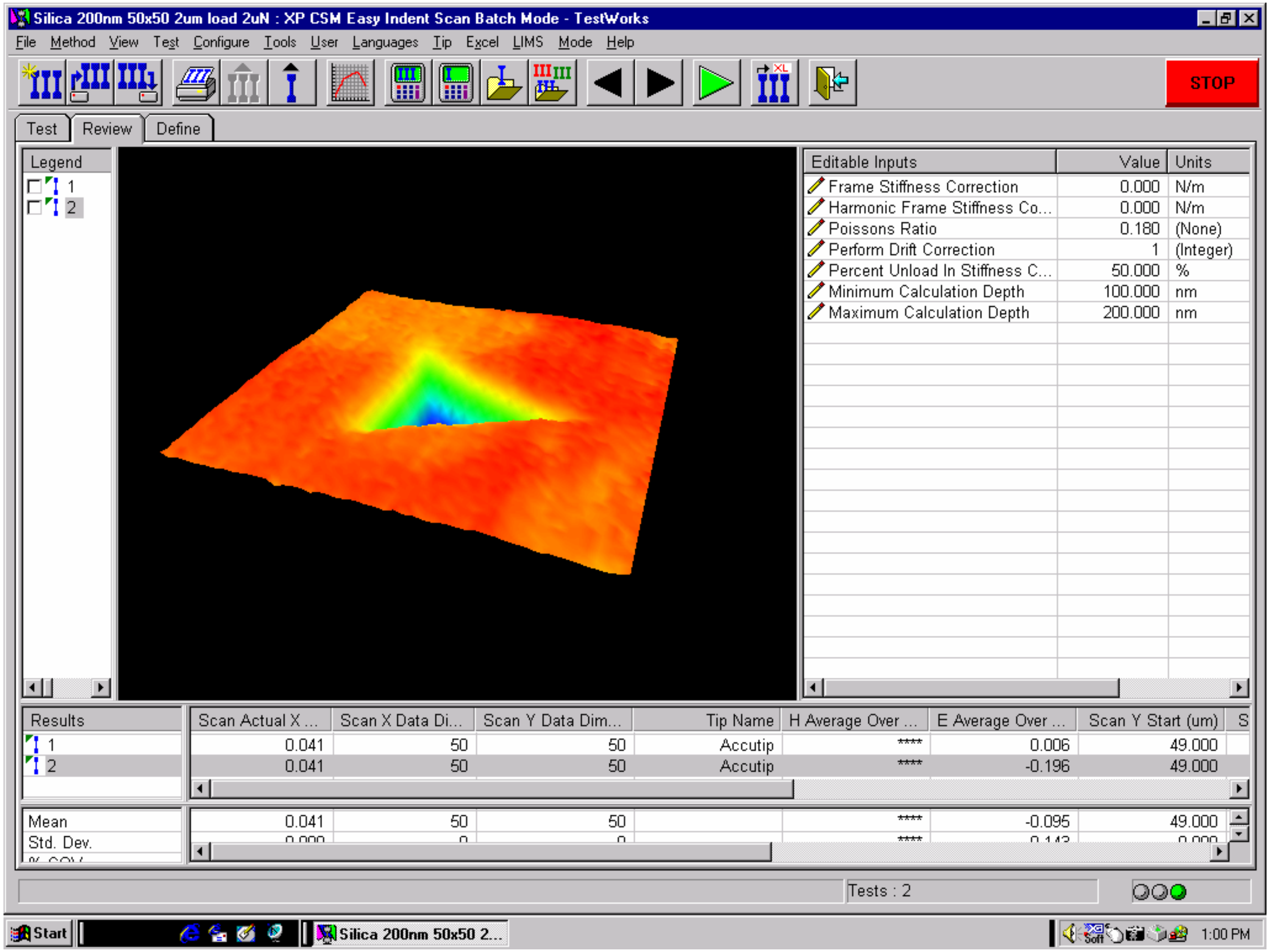
Indent – scan **Indentation**



Load, Displacement, Stiffness….

All the traditional results from regular indentation.

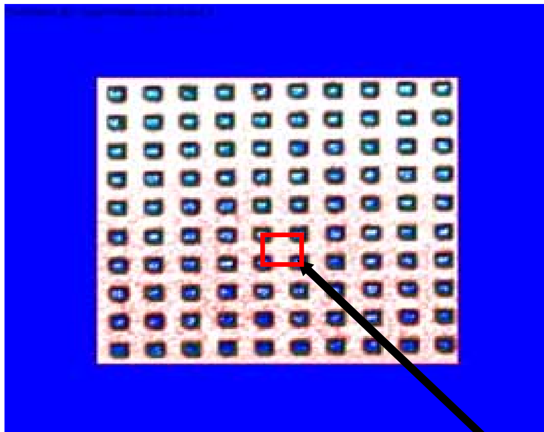
Indent – scan Image

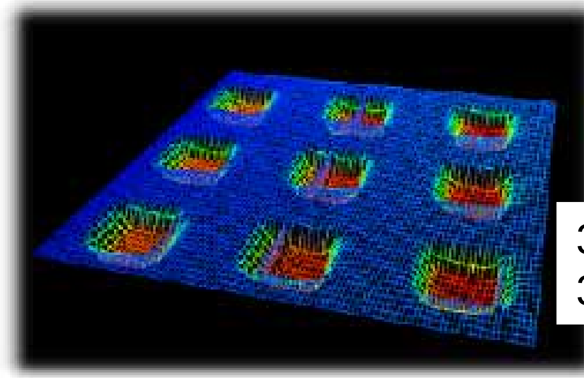


Interactive mode Targeting

Step 1:

Scan



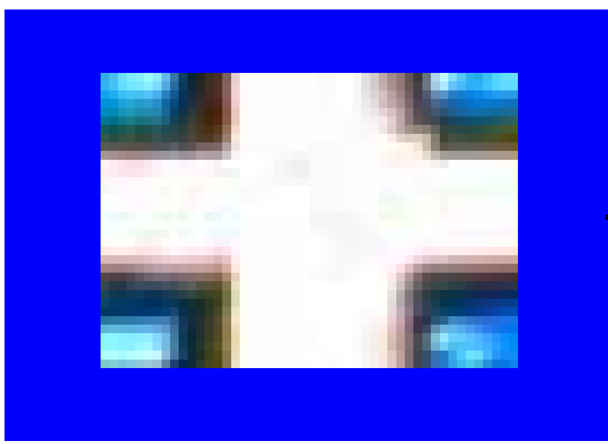


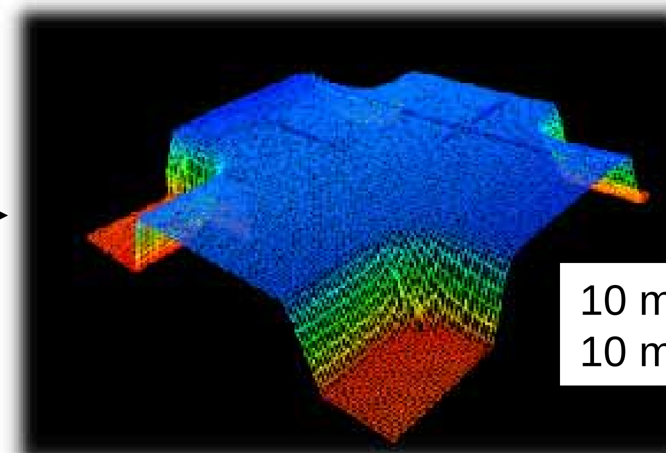
30 microns x 30 microns

Step 2: Select new area of scan

Step 3:

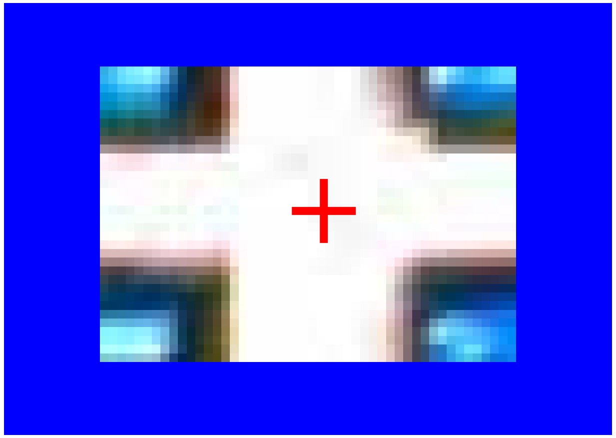
scan





10 microns x 10 microns

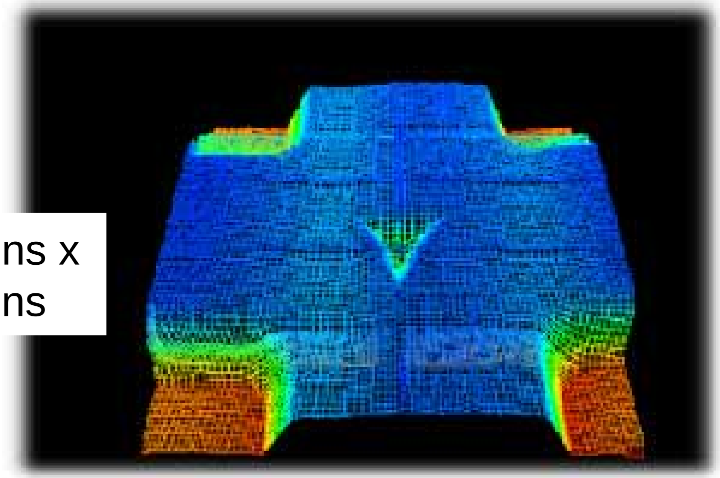
Interactive mode **Indenting**



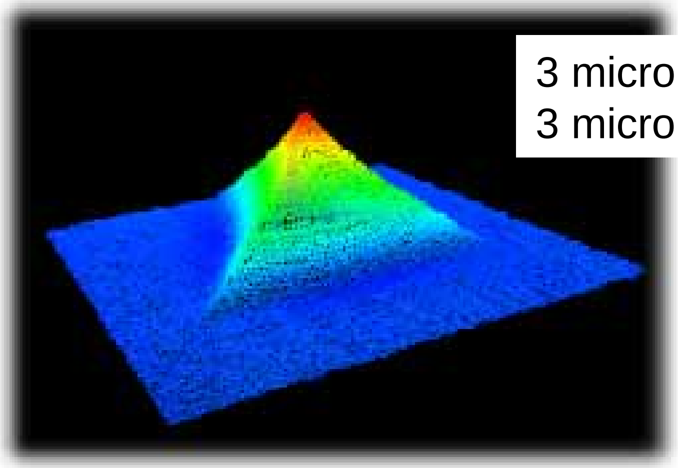
Step 4: Position indent location

Step 5: Indentation

Step 6: Scan the indent



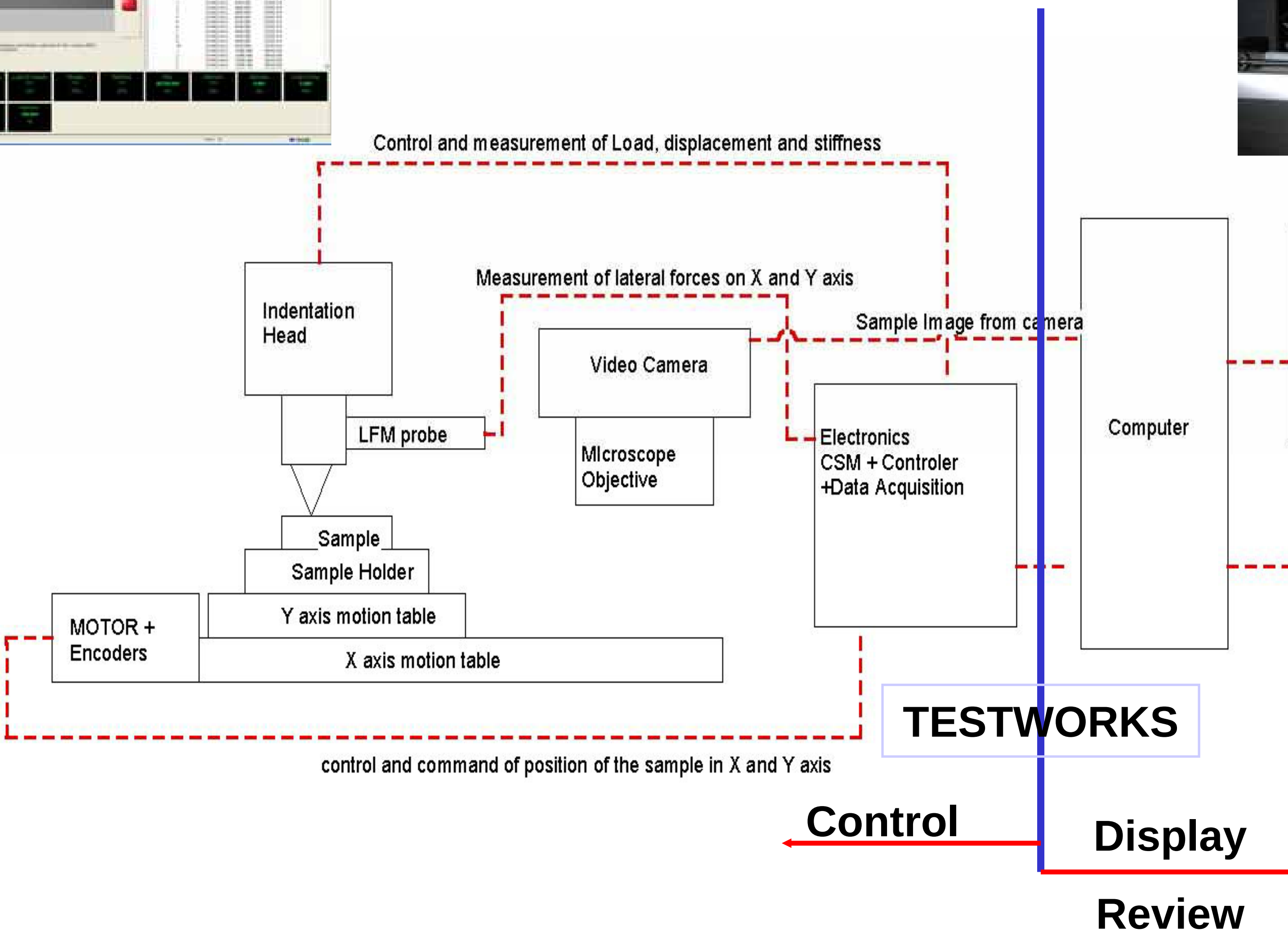
10 microns x
10 microns



3 microns x
3 microns

TestWorks Software

TestWorks' Place in Our Instrument

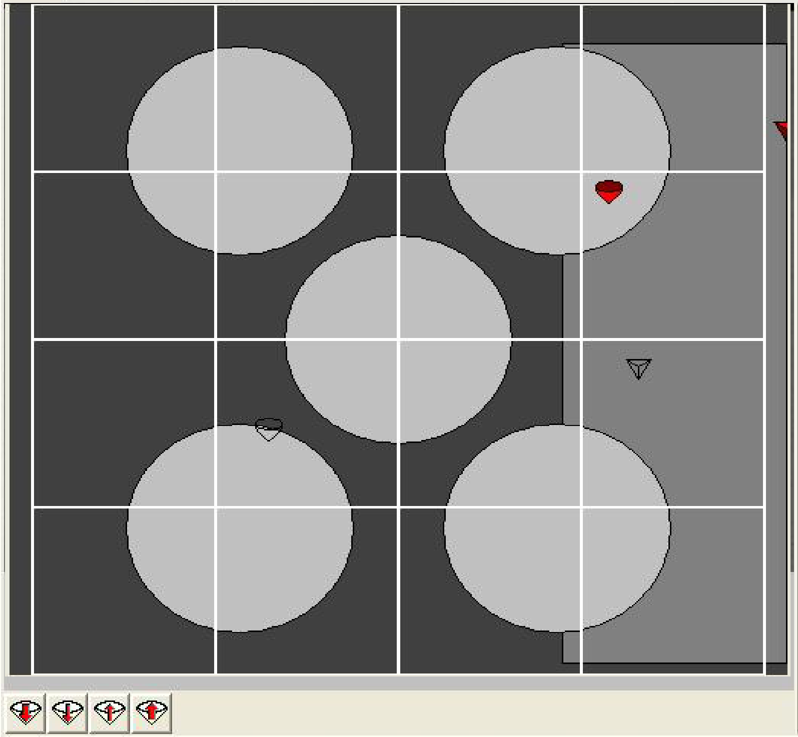


silicabefore : DCM-SA2 CSM Hardness, Modulus for Thin Films (40) - TestWorks

File Method View Test Configure Tools User Tip Excel Mode System Help

STOP

Test | Review | Define



DCM CSM Method for Hardness and Modulus optimized for thin coatings (0031)
Current Tip: DCM TIP B 04-04-03

Sample	Test	Method	X Test Position	Y Test Positi...
silicabefore	1	DCM\DCM-S...	25156.405	32962.189
silicabefore	2	DCM\DCM-S...	25112.405	32962.189
silicabefore	3	DCM\DCM-S...	25068.405	32962.189
silicabefore	4	DCM\DCM-S...	25024.405	32962.189
silicabefore	5	DCM\DCM-S...	24980.405	32962.189
silicabefore	6	DCM\DCM-S...	24980.405	32918.189
silicabefore	7	DCM\DCM-S...	25024.405	32918.189
silicabefore	8	DCM\DCM-S...	25068.405	32918.189
silicabefore	9	DCM\DCM-S...	25112.405	32918.189
silicabefore	10	DCM\DCM-S...	25156.405	32918.189
1	1	DCM\DCM-S...	9021.686	33324.708
1	2	DCM\DCM-S...	9011.686	33324.708
1	3	DCM\DCM-S...	9001.686	33324.708
1	4	DCM\DCM-S...	8991.686	33324.708
1	5	DCM\DCM-S...	8981.686	33324.708
1	6	DCM\DCM-S...	8981.686	33314.708
1	7	DCM\DCM-S...	8991.686	33314.708
1	8	DCM\DCM-S...	9001.686	33314.708
1	9	DCM\DCM-S...	9011.686	33314.708
1	10	DCM\DCM-S...	9021.686	33314.708
2	1	DCM\DCM-S...	4979.856	33335.314
2	2	DCM\DCM-S...	4969.856	33335.314
2	3	DCM\DCM-S...	4959.856	33335.314
2	4	DCM\DCM-S...	4949.856	33335.314
2	5	DCM\DCM-S...	4939.856	33335.314
2	6	DCM\DCM-S...	4939.856	33325.314
2	7	DCM\DCM-S...	4949.856	33325.314
2	8	DCM\DCM-S...	4959.856	33325.314
2	9	DCM\DCM-S...	4969.856	33325.314
2	10	DCM\DCM-S...	4979.856	33325.314
3	1	DCM\DCM-S...	10568.468	39435.639
3	2	DCM\DCM-S...	10558.468	39435.639
3	3	DCM\DCM-S...	10548.468	39435.639
3	4	DCM\DCM-S...	10538.468	39435.639

Displacement Into	Load On Sample	Modulus	Hardness	Raw	Harmonic	Harmonic	Load vs Disp
*****	*****	*****	*****	-20729.924	*****	0.061	0.000
nm	mN	GPa	GPa	nm	N/m	nm	N/m

Time	Harmonic
67880.800	150.000
s	Hz

Tests : 10

DCM

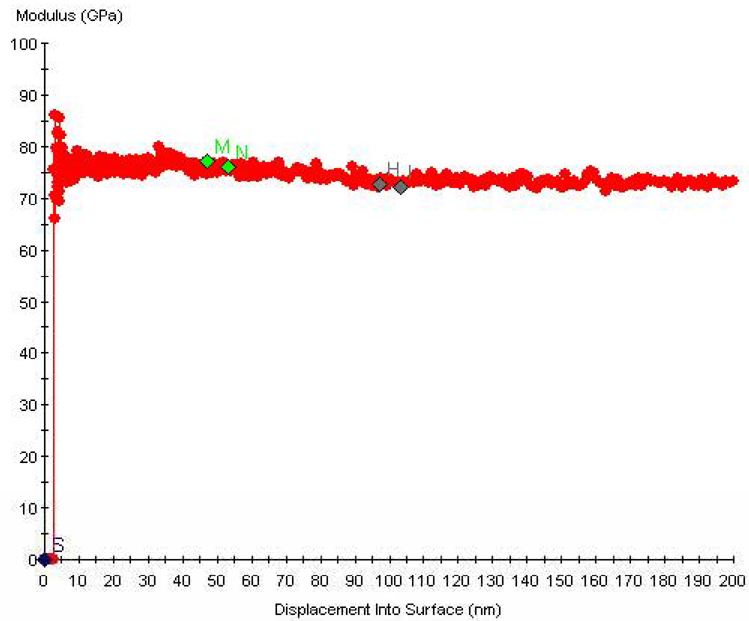
silicabefore : DCM-SA2 CSM Hardness, Modulus for Thin Films (40) - TestWorks

File Method View Test Configure Tools User Tip Excel Mode Help

STOP

Test | Review | Define

Legend	Warning
1	
2	
3	
4	
5	
6	
7	
8	
9	
10	



Editable Inputs	Value	Units	Report Order
Frame Stiffness Correction	0.000	N/m	
Harmonic Frame Stiffness Co...	0.000	N/m	
Poissons Ratio	0.180	(None)	
Perform Drift Correction	1	(Integer)	
Surface Stiffness	40.000	N/m	
Minimum Modulus Depth	47.000	nm	
Percent Unload In Stiffness C...	50.000	%	
Maximum Modulus Depth	53.000	nm	
Minimum Hardness Depth	97.000	nm	
Maximum Hardness Depth	103.000	nm	

Results	E Average Over...	H Average Over...	Modulus From Un...	Hardness From Un...	Drift Correct...	Time At St...	Tip Name	Area Coeff...	Area Coeff...	Area Coefficient 3
1	75.751	10.282	70.737	9.633	0.052	3:32:46 PM	DCM TIP B 0...	2.26426e+...	5.1267520...	2.512513293e+0...
2	71.267	9.760	69.972	9.489	0.096	3:46:48 PM	DCM TIP B 0...	2.26426e+...	5.1267520...	2.512513293e+0...
3	70.479	9.631	71.170	9.217	0.091	3:57:42 PM	DCM TIP B 0...	2.26426e+...	5.1267520...	2.512513293e+0...
4	70.601	9.729	71.653	9.224	0.069	4:08:35 PM	DCM TIP B 0...	2.26426e+...	5.1267520...	2.512513293e+0...
5	69.817	9.507	71.228	9.300	0.050	4:19:11 PM	DCM TIP B 0...	2.26426e+...	5.1267520...	2.512513293e+0...
6	68.755	9.371	69.664	9.305	0.037	4:29:59 PM	DCM TIP B 0...	2.26426e+...	5.1267520...	2.512513293e+0...
7	68.646	9.279	70.898	9.219	0.027	4:40:37 PM	DCM TIP B 0...	2.26426e+...	5.1267520...	2.512513293e+0...
8	70.965	9.846	71.419	9.316	0.017	4:50:58 PM	DCM TIP B 0...	2.26426e+...	5.1267520...	2.512513293e+0...
9	61.740	8.566	70.346	8.882	0.012	5:01:24 PM	DCM TIP B 0...	2.26426e+...	5.1267520...	2.512513293e+0...
10	73.063	9.661	70.733	9.398	0.009	5:12:09 PM	DCM TIP B 0...	2.26426e+...	5.1267520...	2.512513293e+0...
Mean	70.108	9.563	70.782	9.298	0.046			2.26426e+...	5.1267520...	2.512513293e+0...
Std. Dev.	3.607	0.446	0.635	0.197	0.031			0.00000e+...	0.0000000...	0.000000000e+0...
% COV	5.14	4.67	0.90	2.12	68.43			0.00	0.00	0.00

(3.584 nm, 81.610 GPa) Tests : 10

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Thank you !!