LECTURE 22: THEORETICAL ASPECTS OF NANOINDENTATION

Outline :

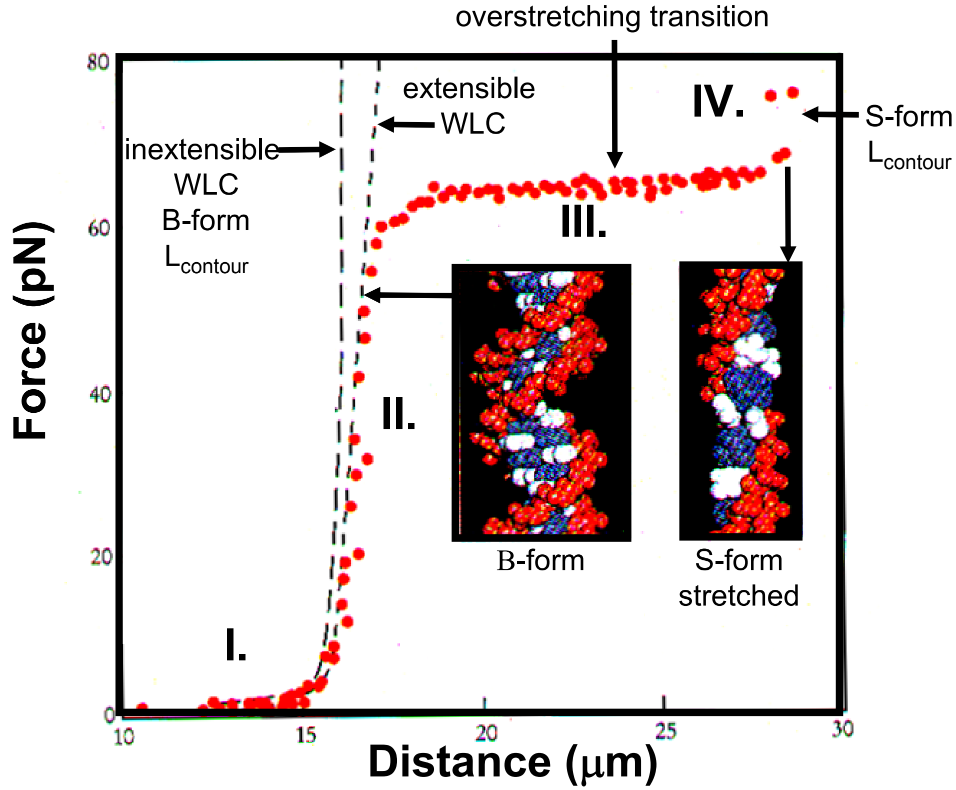
Objectives: To understand general theoretical formulations for reducing material properties from nanoindentation experiments

Readings: Course Reader Documents 45 (one of the most cited papers in Materials Science)-46, Additional Historical Ref (posted on stellar's Supplementary Materials) : Sneddon **1965** *Int. J. Engng.* 3, 47-57.

SINGLE MOLECULE ELASTICITY OF TITIN (AFM) & DNA (OPTICAL TWEEZERS)

- Structure and physiological role of Titin *(Rief, et al. CHEMPHYSCHEM 2002, 3, 255-261)→sawtooth force profiles*

(Bustamante, et al. Science 1999, 271, 795)



Biological Relevance of Overstretching Transition? Ability to switch between different structures is critical to the processes of transcription, replication, condensaton, e.g. the base pairs are much more exposed in S-DNA than normal DNA, the transition may be biologically significant for accessing information contained in the DNA code

I. low stretched behaves like WLC (p ≈ 50 nm under physiological conditions, much larger than most polymers ~ 1nm, hence much smaller forces, need optical tweezers)

II. intermediate stretches -some extensibility as apparent by finite slope beyond $L_{contour}$ (B-form)

III. At 65 pN ~ 0.06 nN, reversible strain-induced conformational transition; chain "yields" and stretches out almost 2× its native B-form contour length at relatively constant force (plateau in force region)
-All of hydrogen bonding and binding between 2 strands is still in tact, tilting of base pairs, tightened helix, reduction in diameter
"overstretching transition"

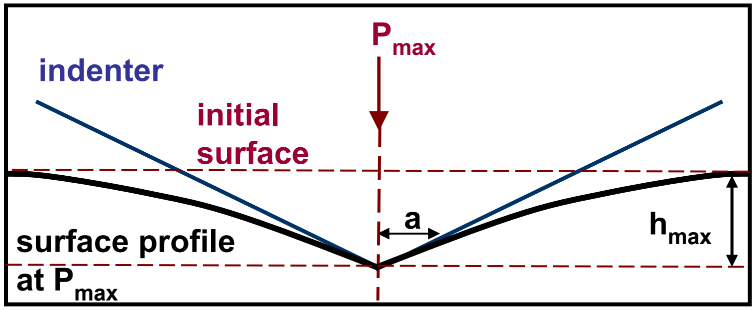
IV. entropic elasticity of S-form

V. can't see here - if you go to high enough stretches, separation between strains (mechanical "melting")

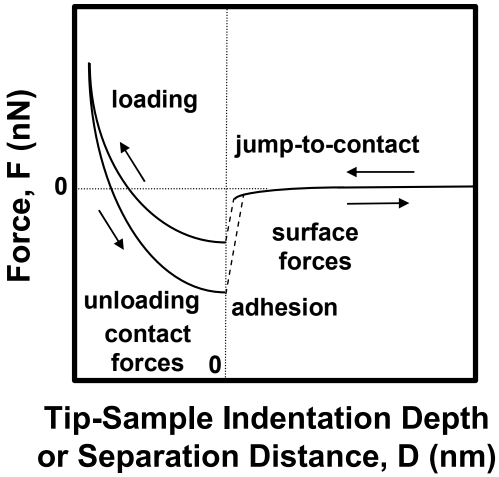
INTRODUCTION TO NANOINDENTATION

Definition : Controlled compression and decompression of a probe tip into a sample surface while measuring force (load, *P*) versus indentation displacement or depth, *h* (nm-scale) continuously

→ probe tip is relatively rigid compared to the sample
→ can measure mechanical properties (e.g. modulus, hardness) on areas nm-μm scale; e.g. thin films and small volume structures
→ called "nano" since the indentation depth is of nanometer scale, however lateral contact areas and forces can be > nanoscale
-multiaxial deformation

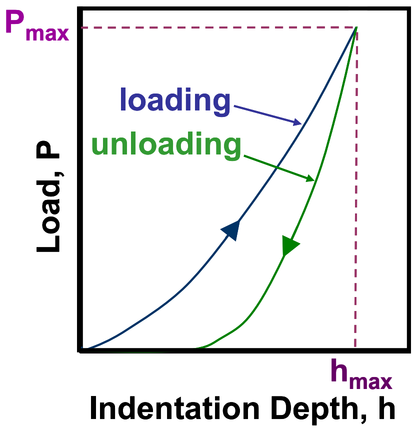


AFM-based Indentation



-e.g. silicon or silicon nitride indenter probe on a cantilever force transducer
-cantilever oriented at an angle to the surface (~11°)
-indenter geometries, e.g. pyramidal (less well defined)
-load range ~ nN-mN, smaller contact radii ~ 10s of nm

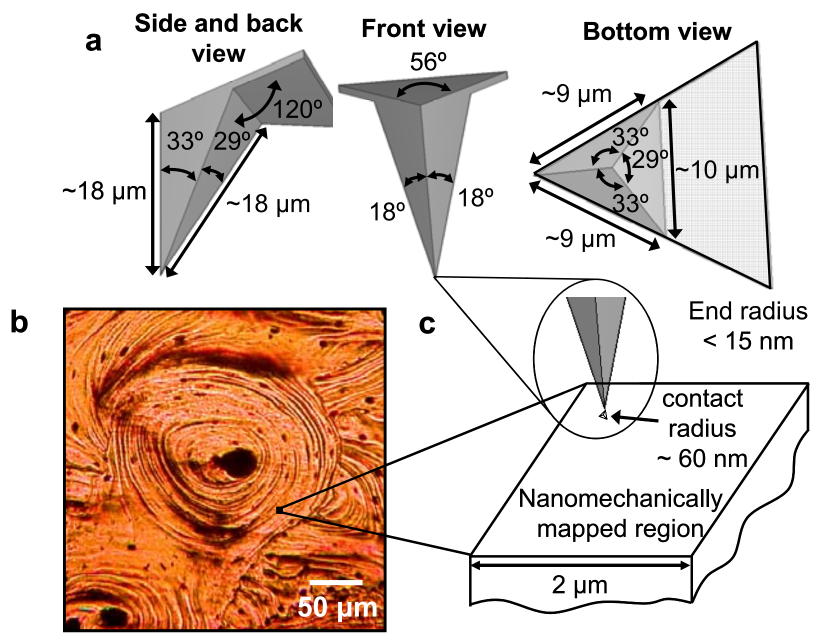
Instrumented or Depth-Sensing Indentation (DSI)



(Hysitron, Micromaterials, Appendix→extension of conventional hardness testing to smaller length scale)
- diamond indenter
- indenter oriented perpendicular to the surface
- variable indenter geometries; Berkovich, cube corner, etc. - load range ~ μN-mN, larger contact radii ~ μm

NANOINDENTATION : INDENTER GEOMETRIES

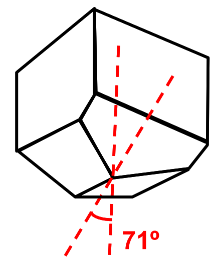
AFM-Based Indentation

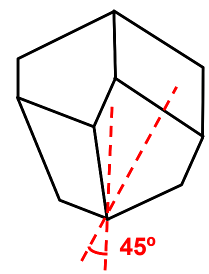


Silicon tetrahedral probe tip indenter (k~ 56 N/m)

Instrumented Indentation

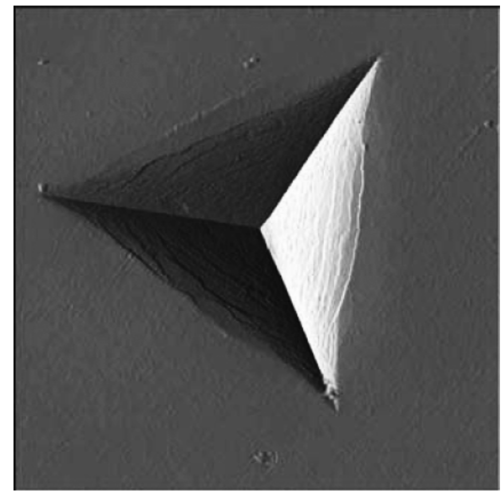
Berkovich



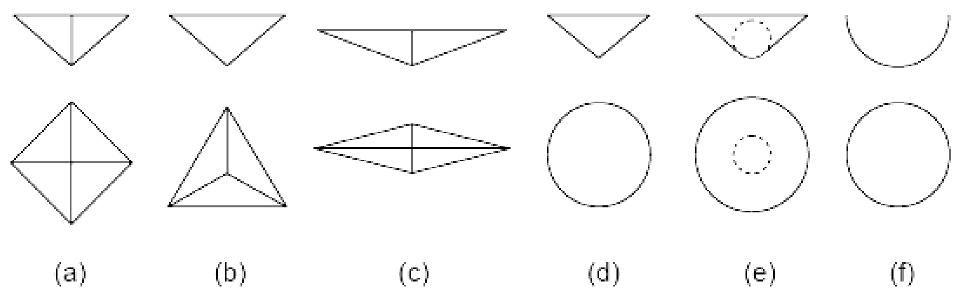


Cube Corner

Residual Berkovich Indent Impression

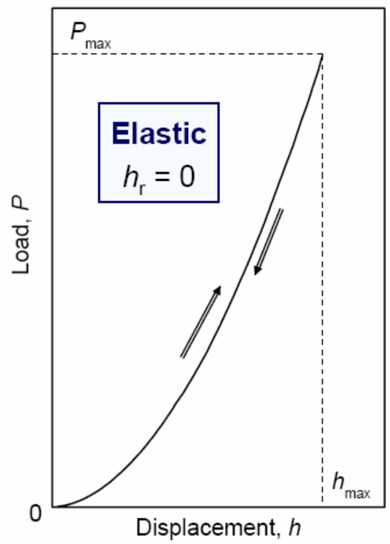


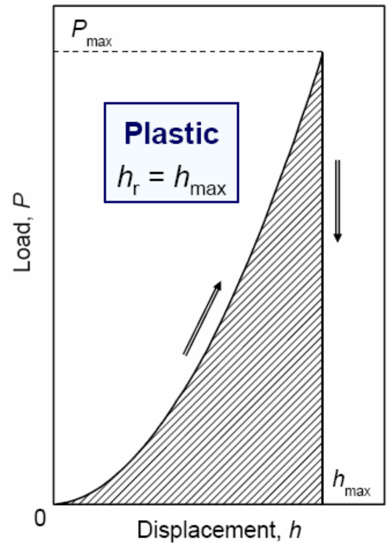
See Appendix for full geometric details



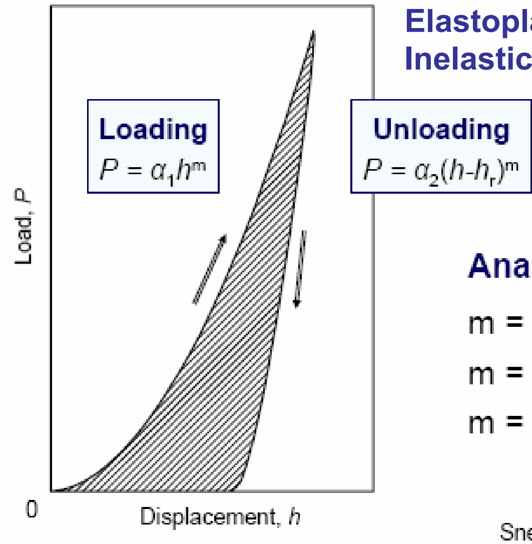
a=Vickers, b= Berkovich, c= Knoop, d = conical, e=Rockwell, f=spherical

NANOINDENTATION : TYPES OF DEFORMATION





Elastoplastic or Inelastic



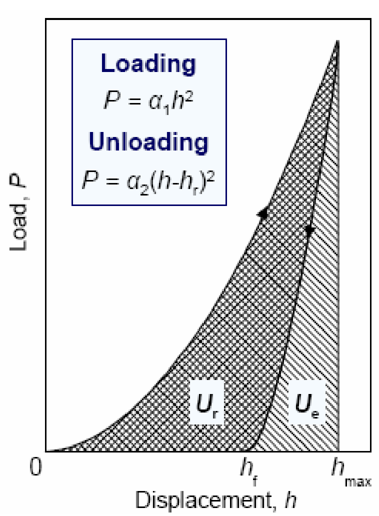
Analytical solution

m = 1 for flat cylinders

m = 2 for cones

m = 1.5 for spheres

Sneddon, *Int. J. Engng. Sci.* 1965

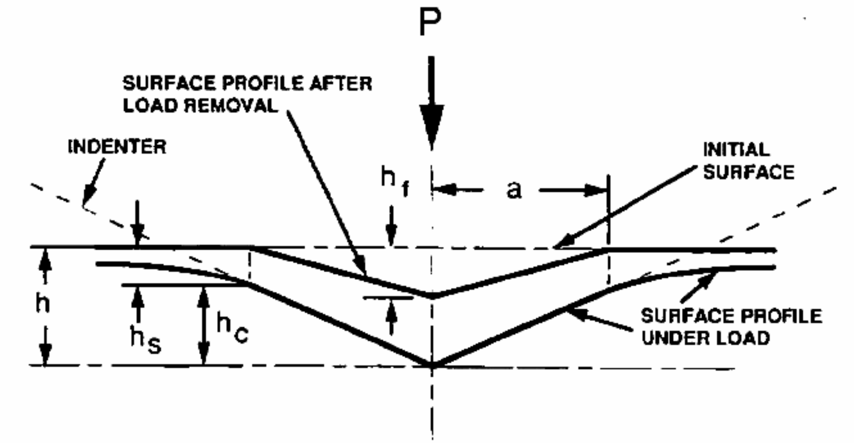


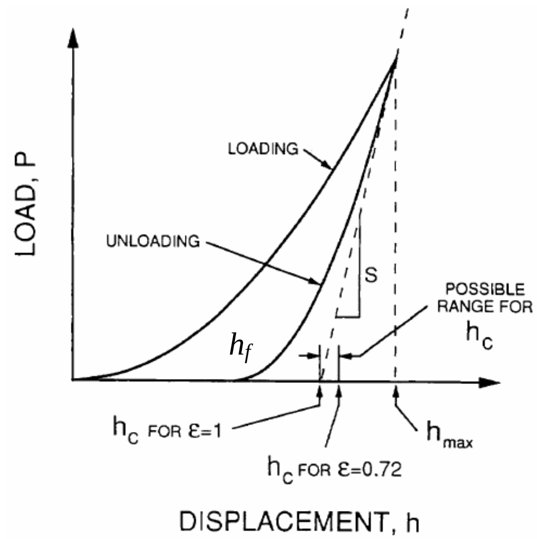
$h_r = h_f$ = residual / final depth
U_e = elastic energy
U_r = energy dissipated (elastoplastic / inelastic)
U_{total} = total work of deformation= $U_e + U_r$

in.materials.drexel.edu/blogs/280_advanced_materials_lab/attachment/469.ashx

OLIVER-PHARR ANALYSIS: GEOMETRIC SET-UP

Linear Elastic, Isotropic, Continuum Contact Mechanics Theory (*Oliver and Pharr, 1992 JMR, 7(6) 1564*) : Geometry set-up and definitions of geometric parameters : assumes "sink-in"





P = applied load, ***P****max* = peak applied load
h = indentation depth (at P_{max}; $h = h_{max}$ maximum depth)
a = radius of contact circle
h_c = contact depth, vertical distance along which contact is made between sample and tip
h_s = displacement of the surface at the perimeter of contact
From geometry : $h = h_c + h_s$
$A(h_c)$ = contact (projected) area at h_c

$$E_r^{-1} = \text{reduced modulus} = \left(\frac{1-\nu^2}{E}\right)_{\text{sample}} + \left(\frac{1-\nu_i^2}{E_i}\right)_{\text{indenter}}$$

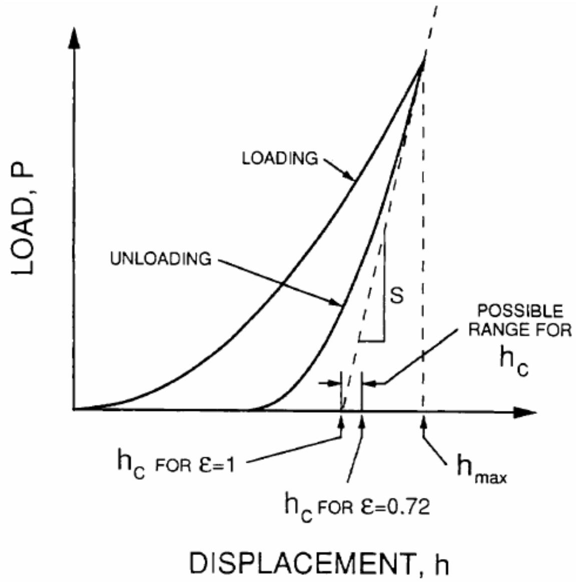
(i.e. two springs in series)
E = modulus
ν = Poisson's ratio
h_f = residual final depth (indicates inelasticity; e.g. viscoelasticity, plasticity)

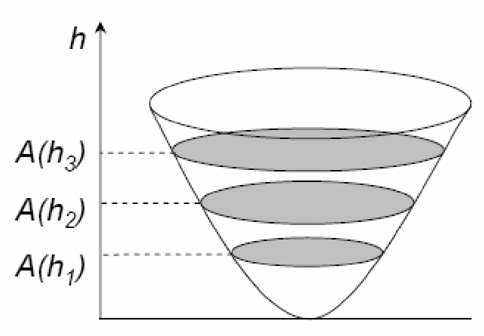
$$S = \text{contact (initial unloading) stiffness} = \left(\frac{dP}{dh}\right)_{P_{max}}$$

(typically evalulated between 95% and 20% of P_{max})

OLIVER-PHARR ANALYSIS : MATHEMATICAL FORMULATION

(*Oliver and Pharr, 1992 JMR, 7(6) 1564*)





Schematic courtesy of B. Bruet

$$E_r = \frac{\sqrt{\pi}}{2\sqrt{A(h_c)}} S \rightarrow \textit{Sneddon Equation holds for any indenter geometry (1)}$$

S is measured directly from the data (typically evalulated between 95% and 20% of P_{max})

$$h_c = h_{max} - \frac{\varepsilon P_{max}}{S} \; (2)$$

Tip Geometry	ε
flat-ended cylindrical punch	1
paraboloid of revolution	0.75
Cone	2(π-2)/π

Indenter (Probe Tip) Area Function Calibration :

$A(h_c)$ = tip area function; representative of tip geometry, can be calibrated on sample of known modulus (e.g. fused quartz) by inverting Sneddon equation *(1)*;

$$A(h_c) = \frac{\pi}{4}\left(\frac{S}{E_r}\right)^2 \; (3)$$

Carry out indentations at successively higher loads; at each P_{max} calculate h_c from *(2)* and $A(h_c)$ from (3), these data are fit to a polynomial:

$$A(h_c) = C_o h_c^2 + C_1 h_c + C_2 h_c^{0.5} + C_3 h_c^{0.25} + C_4 h_c^{1/8} + C_5 h_c^{1/16}$$

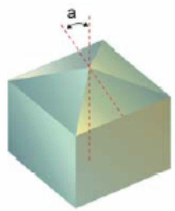
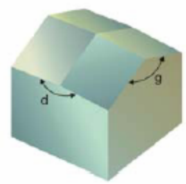
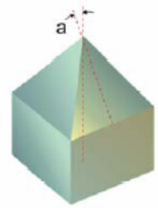
Gives $A(h_c)$ *for every indentation depth*, h_c

$C_o = 24.5; A(h_c) = 24.5 h_c^2$ (Ideal Berkovich Geometry) *(4)*

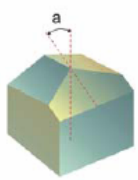
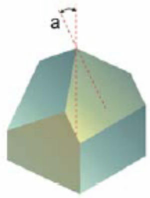
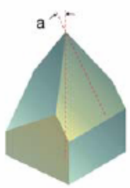
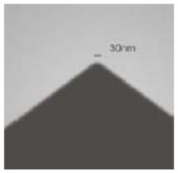
(see Appendix for Derivation), coefficients reflect indenter geometry

APPENDIX : DETAILED GEOMETRY OF INDENTERS 1

4-sided:

VICKERS **FV**  	**KNOOP INDENTER** **FK**  	**4-SIDED CUSTOM** **FD**  	**END LINE** TEM micrograph 
Standard Vickers indenter: a = 68.00° Available as **Traceable Standard**	Standard Knoop indenter defined by 2 angles: d = 172.50°, g = 130.00°	Custom 4-sided indenters: 80° > a > 20°	Micro Star indenters maximum line of conjunction: 400nm.

3-sided:

BERKOVICH **TB**  	**CUBE CORNER** **TC**  	**3-SIDED CUSTOM** **TD**  	**SHARPNESS** TEM micrograph 
Berkovich: a = 65.03° Mod. Berkovich: a = 65.27° Available as **Traceable Standard**	Cube corner: a = 35.26° Available as **Traceable Standard**	Custom 3-sided indenters: 80° > a > 20°	Micro Star 3-sided sharp indenters tip radius < 50nm.

APPENDIX : DETAILED GEOMETRY OF INDENTERS 2

Cones:

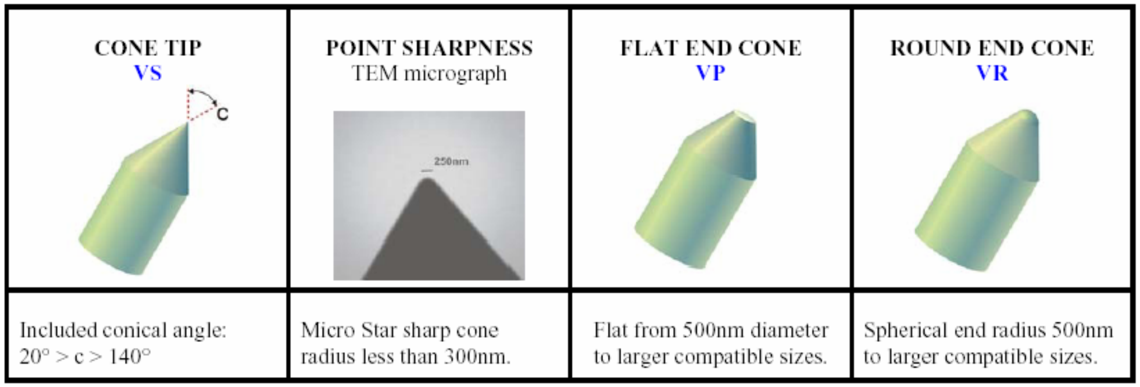
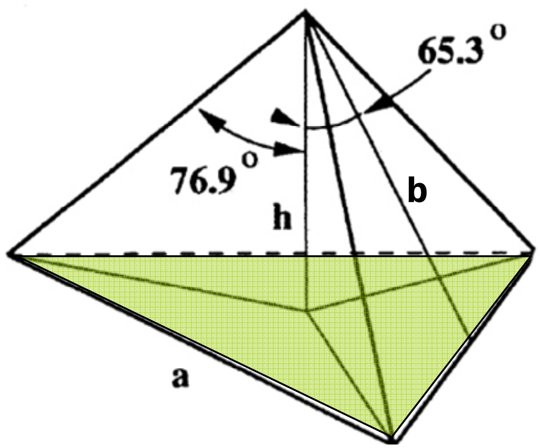


Figure 6 CONE INDENTERS

APPENDIX : DETAILED GEOMETRY OF INDENTERS 3 *(Do Kyung Kim, KAIST)*

Indenter type	Projected area	Semi angle (θ)	Effective cone angle (α)	Intercept factor (ε)	Geometry correction factor (β)
Sphere	$A \approx \pi 2Rh$	N/A	N/A	0.75	1

APPENDIX : BERKOVICH GEOMETRY CALCULATION OF CONTACT AREA



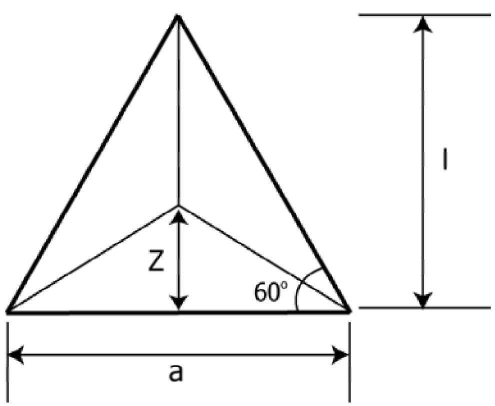
$$\tan 60^o = \frac{l}{a/2}$$

$$l = \frac{\sqrt{3}}{2}a$$

$$A_{proj} = \frac{al}{2} = \frac{\sqrt{3}}{4}a^2$$

$$\cos 65.27^o = \frac{h}{b}$$

Projected area



$$h = \frac{a\cos 65.3^o}{2\sqrt{3}\sin 65.3^o} = \frac{a}{2\sqrt{3}\tan 65.3^o}$$

$$a = 2\sqrt{3}h\tan 65.3^o$$

$$A_{proj} = 3\sqrt{3}h^2\tan^2 65.3^o = 24.56h^2$$

(Do Kyung Kim, KAIST)

APPENDIX : OLIVER-PHARR CITATIONS

One of the most cited paper in Materials Science

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Full Record

Record 5 of 16 (Set #2) SUMMARY

Title: AN IMPROVED TECHNIQUE FOR DETERMINING HARDNESS AND ELASTIC-MODULUS USING LOAD AND DISPLACEMENT SENSING INDENTATION EXPERIMENTS

Author(s): OLIVER WC, PHARR GM

Source: JOURNAL OF MATERIALS RESEARCH 7 (6): 1564-1583 JUN 1992

Document Type: Article

Language: English

Cited References: 37 **Times Cited:** 2975 FIND RELATED RECORDS

Abstract: The indentation load-displacement behavior of six materials tested with a Berkovich indenter has been carefully documented to establish an improved method for determining hardness and elastic modulus from indentation load-displacement data. The materials included fused silica, soda-lime glass, and single crystals of aluminum, tungsten, quartz, and sapphire. It is shown that the load-displacement curves during unloading in these materials are not linear, even in the initial stages, thereby suggesting that the flat punch approximation used so often in the analysis of unloading data is not entirely adequate. An analysis technique is presented that accounts for the curvature in the unloading data and provides a physically justifiable procedure for determining the depth which should be used in conjunction with the indenter shape function to establish the contact area at peak load. The hardnesses and elastic moduli of the six materials are computed using the analysis procedure and compared with values determined by independent means to assess the accuracy of the method. The results show that with good technique, moduli can be measured to within 5%.

KeyWords Plus: SILICON; BEHAVIOR

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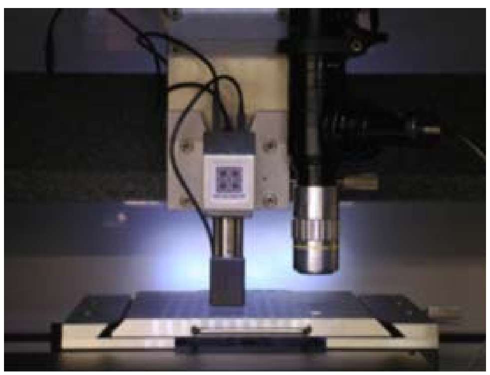
APPENDIX : NANOINDENTATION INSTRUMENTATION

- MTS_Nano-Indenter XP

- CSIRO_UMIS
- (Ultra-Micro-Indentation System)

- Hysitron_Triboscope



(Do Kyung Kim, KAIST)

- CSM_NHT
- (Nano-Hardness Tester)