LECTURE 22: THEORETICAL ASPECTS OF NANOINDENTATION

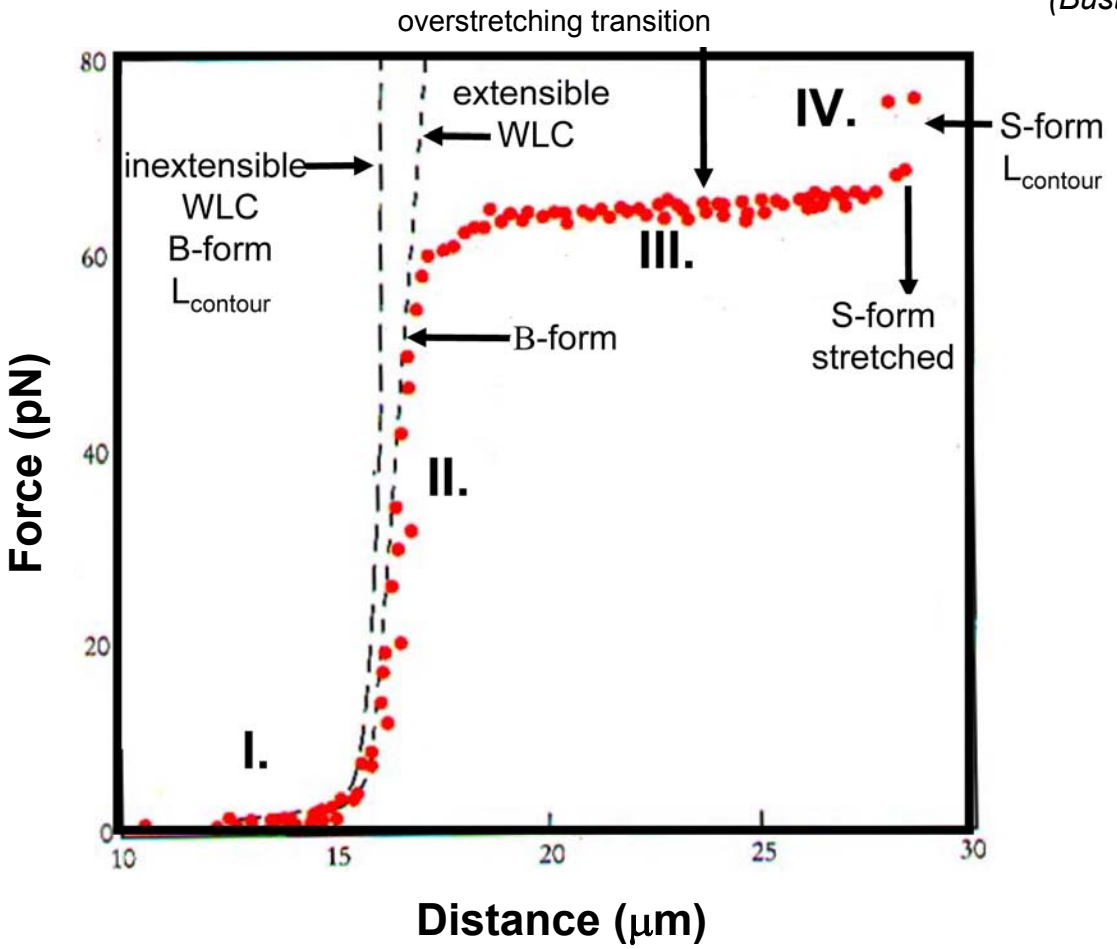
Outline :

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: 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"

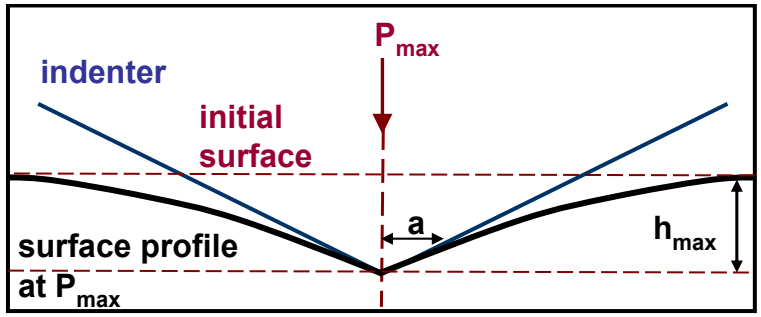
IV. entropic elasticity of S-form

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

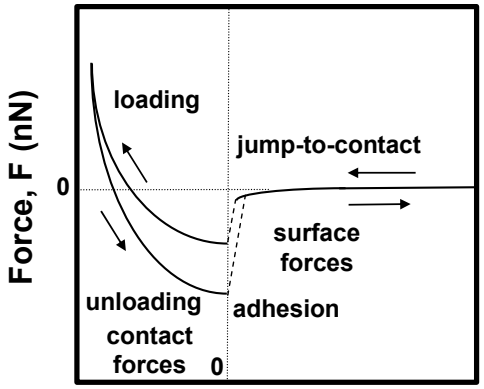
INTRODUCTION TO NANOINDENTATION

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



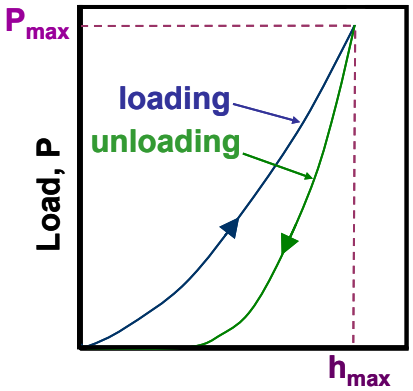
AFM-based Indentation



Tip-Sample Indentation Depth or Separation Distance, D (nm)

-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)

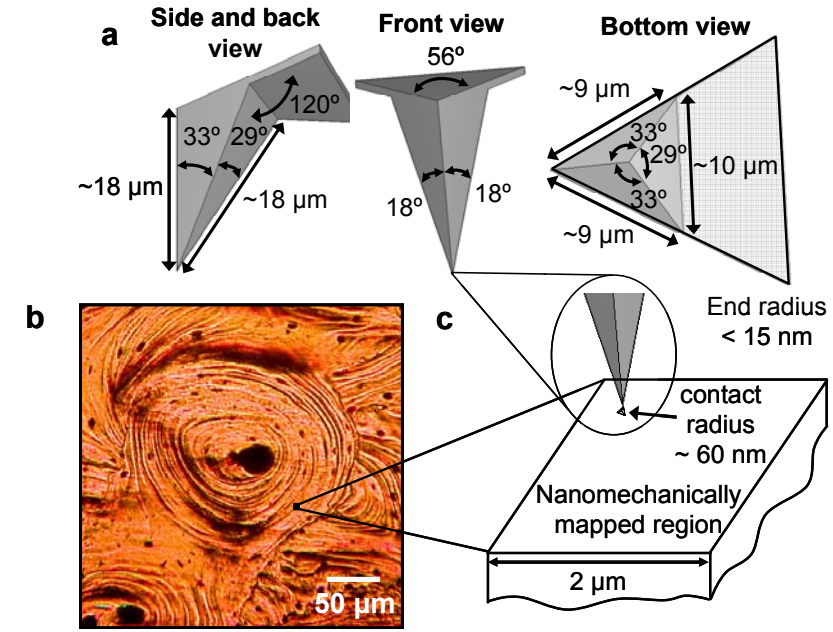


Indentation Depth, h

(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

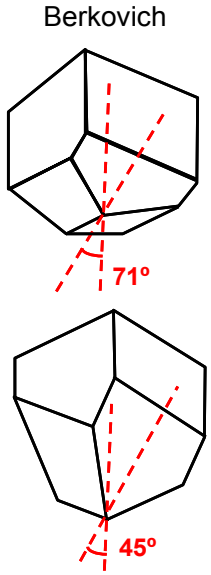
NANOINDENTATION : INDENTER GEOMETRIES

AFM-Based Indentation

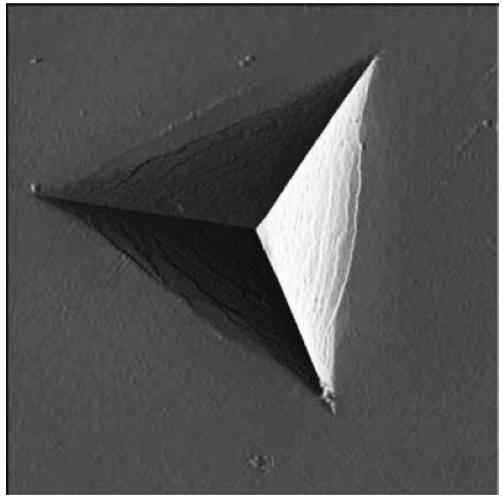


Silicon tetrahedral probe tip indenter (k~ 56 N/m)

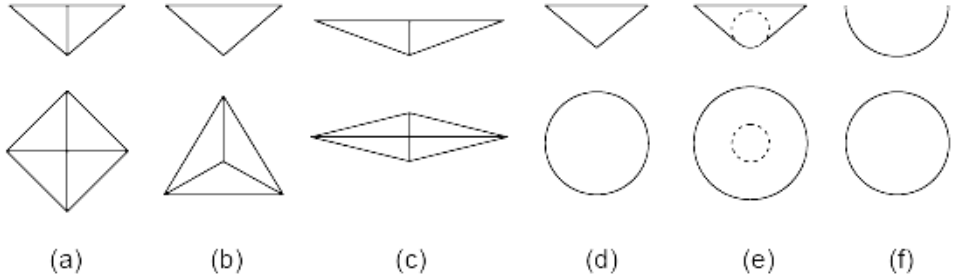
Instrumented Indentation



Residual Berkovich Indent Impression

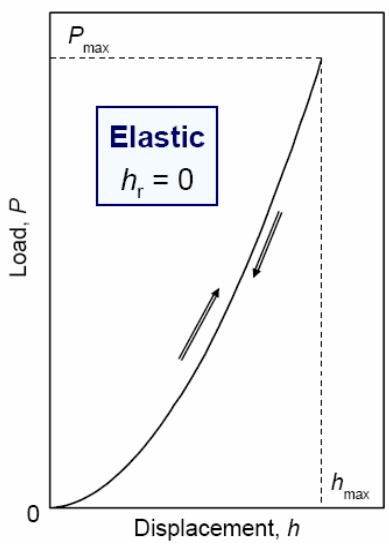


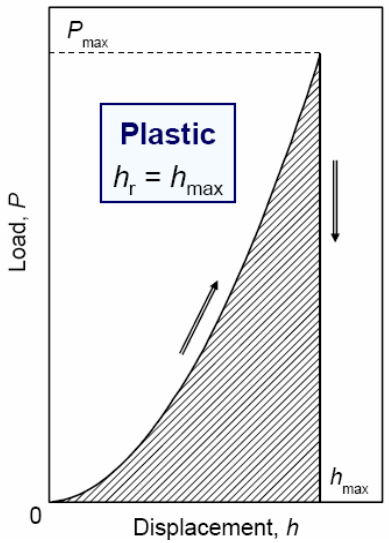
See Appendix for full geometric details

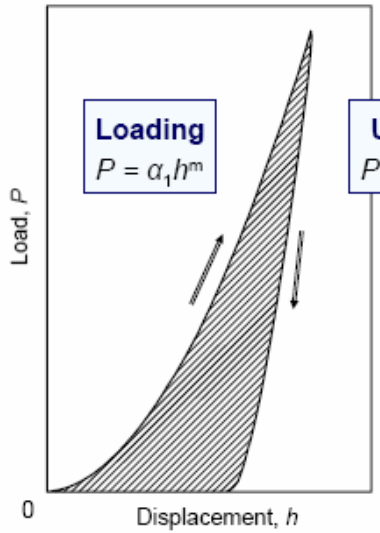


a=Vickers, b= Berkovich, c= Knoop, d = conical, e=Rockwell, f=spherical

NANOINDENTATION : TYPES OF DEFORMATION







Elastoplastic or Inelastic

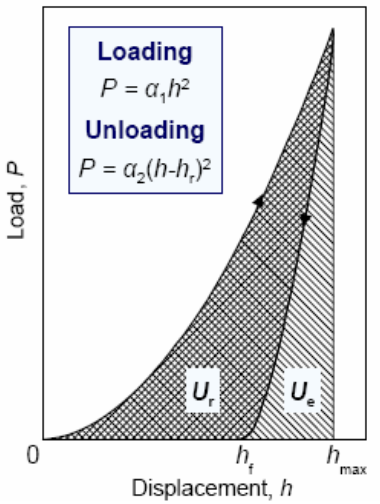
Analytical solution

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)

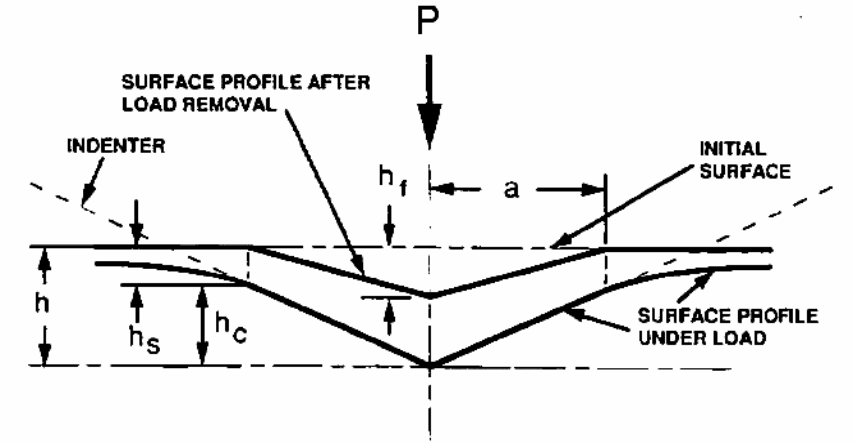
U_{total} = total work of deformation= $U_e + U_r$

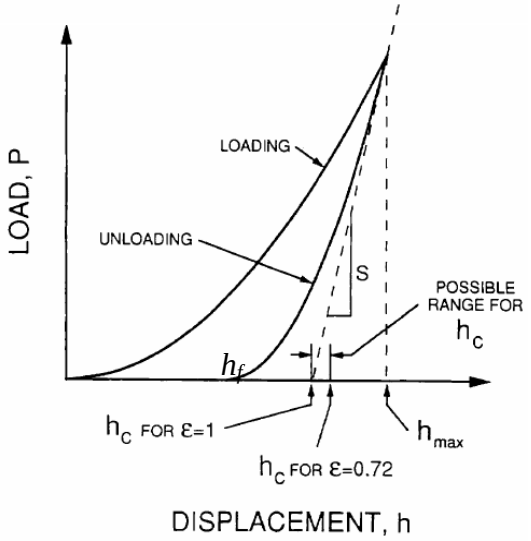
in.materials.drexel.edu/blogs/280_advanced_materials_lab/attachment/469.ashx

Courtesy of Prof. Yury Gogotsi. Used with permission.

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"





Courtesy of George M. Pharr and Journal of Materials Research. Used with permission.

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

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

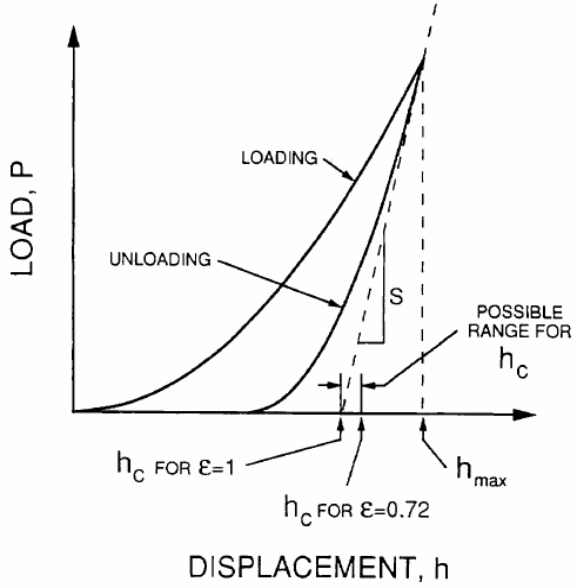
(i.e. two springs in series)
E = modulus
ν = Poisson's ratio
h_f = residual final depth (indicates inelasticity; e.g. viscoelasticity, plasticity)

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

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

OLIVER-PHARR ANALYSIS : MATHEMATICAL FORMULATION

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



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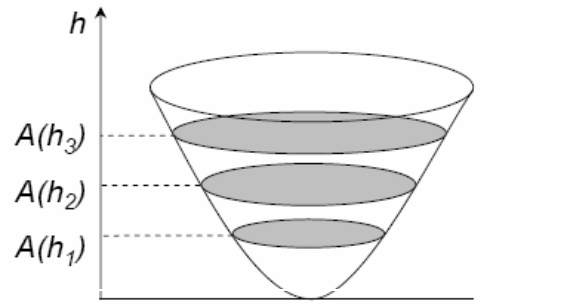

Schematic courtesy of B. Bruet

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

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	$2(\pi-2)/\pi$

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

Four appendices removed due to copyright restrictions.

- Detailed geometry of indenters
- Berkovich geometry calculation of contact area
- Web screenshot: Oliver and Pharr JMR 1992 article is one of the most cited papers in Materials Science, has been cited >2975 times
- Photos of nanoindentation instruments.