LECTURE 22: THEORETICAL ASPECTS OF NANOINDENTATION

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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)


I. low stretched behaves like WLC (p ≈ 50 nm under physiological conditions, much larger than most polymers ~ 1 nm, 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")

Biological Relevance of Overstretching Transition? Ability to switch between different structures is critical to the processes of transcription, replication, condensation, 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.
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)

- diamond indenter
- indenter oriented perpendicular to the surface
- variable indenter geometries; Berkovich, cube corner, etc. - load range ~ μN-mN, larger contact radii ~ μm

(Hysitron, Micromaterials, Appendix → extension of conventional hardness testing to smaller length scale)
NANOINDENTATION : INDENTER GEOMETRIES

AFM-Based Indentation

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

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


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

in.matters.drexel.edu/blogs/280_advanced_materials_fab/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"

\[ P = \text{applied load}, \quad P_{\text{max}} = \text{peak applied load} \]

\[ h = \text{indentation depth (at } P_{\text{max}}, \ h = h_{\text{max}} \text{ maximum depth)} \]

\[ a = \text{radius of contact circle} \]

\[ h_c = \text{contact depth, vertical distance along which contact is made between sample and tip} \]

\[ h_s = \text{displacement of the surface at the perimeter of contact} \]

From geometry: \( h = h_c + h_s \)

\[ A(h_c) = \text{contact (projected) area at } h_c \]

\[ E_r^{-1} = \text{reduced modulus} = \left( \frac{1 - v^2}{E} \right)_{\text{sample}} + \left( \frac{1 - v_i^2}{E_i} \right)_{\text{indenter}} \]

(i.e. two springs in series)

\[ E = \text{modulus} \]

\[ v = \text{Poisson's ratio} \]

\[ h_f = \text{residual final depth (indicates inelasticity; e.g. viscoelasticity, plasticity)} \]

\[ S = \text{contact (initial unloading) stiffness} = \left( \frac{dP}{dh} \right)_{\text{initial unloading}} \]

(typically evaluated between 95% and 20% of \( P_{\text{max}} \))
OLIVER-PHARR ANALYSIS: MATHEMATICAL FORMULATION
(Oliver and Pharr, 1992 JMR, 7(6) 1564)

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

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

\[
h_c = h_{max} - \frac{eP_{max}}{S} \ (2)
\]

### Tip Geometry

<table>
<thead>
<tr>
<th>Geometry</th>
<th>(\varepsilon)</th>
</tr>
</thead>
<tbody>
<tr>
<td>flat-ended cylindrical punch</td>
<td>1</td>
</tr>
<tr>
<td>paraboloid of revolution</td>
<td>0.75</td>
</tr>
<tr>
<td>Cone</td>
<td>(2(\pi-2)/\pi)</td>
</tr>
</tbody>
</table>

**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:

<table>
<thead>
<tr>
<th>Indenter</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>VICKERS (FV)</td>
<td>Standard Vickers indenter: a = 68.00° Available as Traceable Standard</td>
</tr>
<tr>
<td>KNOOP INDENTER (FK)</td>
<td>Standard Knoop indenter defined by 2 angles: d = 172.50°, g = 130.00°</td>
</tr>
<tr>
<td>4-SIDED CUSTOM (FD)</td>
<td>Custom 4-sided indenters: 80° &gt; a &gt; 20°</td>
</tr>
<tr>
<td>END LINE TEM micrograph</td>
<td>Micro Star indenters maximum line of conjunction: 400nm.</td>
</tr>
</tbody>
</table>

3-sided:

<table>
<thead>
<tr>
<th>Indenter</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>BERKOVICH (TB)</td>
<td>Berkovich: a = 65.03° Mod. Berkovich: a = 65.27° Available as Traceable Standard</td>
</tr>
<tr>
<td>CUBE CORNER (TC)</td>
<td>Cube corner: a = 35.26° Available as Traceable Standard</td>
</tr>
<tr>
<td>3-SIDED CUSTOM (TD)</td>
<td>Custom 3-sided indenters: 80° &gt; a &gt; 20°</td>
</tr>
<tr>
<td>SHARPNESS TEM micrograph</td>
<td>Micro Star 3-sided sharp indenters tip radius &lt; 50mm.</td>
</tr>
</tbody>
</table>

APPENDIX : DETAILED GEOMETRY OF INDENTERS 2

Cones:


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

<table>
<thead>
<tr>
<th>Indenter type</th>
<th>Projected area</th>
<th>Semi angle ($\theta$)</th>
<th>Effective cone angle ($\alpha$)</th>
<th>Intercept factor ($\varepsilon$)</th>
<th>Geometry correction factor ($\beta$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sphere</td>
<td>$A \approx \pi 2Rh$</td>
<td>N/A</td>
<td>N/A</td>
<td>0.75</td>
<td>1</td>
</tr>
</tbody>
</table>
APPENDIX: BERKOVICH GEOMETRY CALCULATION OF CONTACT AREA

\[ \tan 60^\circ = \frac{l}{a/2} \]

\[ l = \frac{\sqrt{3}}{2} a \]

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

\[ \cos 65.27^\circ = \frac{h}{b} \]

\[ h = \frac{a \cos 65.3^\circ}{2\sqrt{3} \sin 65.3^\circ} = \frac{a}{2\sqrt{3} \tan 65.3^\circ} \]

\[ a = 2\sqrt{3}h \tan 65.3^\circ \]

\[ A_{proj} = 3\sqrt{3}h^2 \tan^2 65.3^\circ = 24.56h^2 \]

(Do Kyung Kim, KAIST)
APPENDIX : OLIVER-PHARR CITATIONS

One of the most cited paper in Materials Science

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 (5): 1564-1583 JUN 1992
Document Type: Article
Language: English

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%.

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APPENDIX : NANOINDENTATION INSTRUMENTATION

- **MTS_Nano-Indenter XP**
- **CSIRO_UMIS**
  -(Ultra-Micro-Indentation System)
- **Hysitron_Triboscope**
- **CSM_NHT**
  -(Nano-Hardness Tester)

(Do Kyung Kim, KAIST)