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: 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)

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 $L_{\text{contour}}$ (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 intact, 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)

- e.g. 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**

- **Side and back view**
- **Front view**
- **Bottom view**

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

**Instrumented Indentation**

- Berkovich

**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

Elastic
\[ h_i = 0 \]

Plastic
\[ h_i = h_{\text{max}} \]

Elastoplastic or Inelastic
\[ P = a_i h^m \]
\[ P = a_2 (h-h_i)^n \]

Analytical solution
\[ m = 1 \text{ for flat cylinders} \]
\[ m = 2 \text{ for cones} \]
\[ m = 1.5 \text{ for spheres} \]

\[ h_f = h_{f_i} = \text{residual / final depth} \]
\[ U_e = \text{elastic energy} \]
\[ U_r = \text{energy dissipated (elastoplastic / inelastic)} \]
\[ U_{\text{total}} = \text{total work of deformation} = U_e + U_r \]

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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 : \( h = h_c + h_s \)
\[ A(h_c) = \text{contact (projected) area at } h_c \]

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

(i.e. two springs in series)

\( E = \text{modulus} \)
\( \nu = \text{Poisson's ratio} \)
\( h_f = \text{residual final depth (indicates inelasticity; e.g. viscoelasticity, plasticity)} \)
\( S = \text{contact (initial unloading) stiffness} = \left( \frac{dP}{dh} \right)_{\text{max}} \)

(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 \( P_{\text{max}} \))

\[ h_c = h_{\text{max}} - \frac{\varepsilon P_{\text{max}}}{S} \tag{2} \]

**Tip Geometry**

<table>
<thead>
<tr>
<th>Tip 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);

\[ A(h_c) = \frac{\pi}{4} \left( \frac{S}{E_r} \right)^2 \tag{3} \]

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

\[ A(h_c) = C_0 h_c^2 + C_1 h_c + C_2 h_c^{0.5} + C_3 h_c^{0.25} + C_4 h_c^{1/8} + C_5 h_c^{1/16} \]

Gives \( A(h_c) \) for every indentation depth, \( h_c \)

\( C_0 = 24.5; A(h_c) = 24.5 h_c^2 \) (Ideal Berkovich Geometry) \( \tag{4} \)

(see Appendix for Derivation), coefficients reflect indenter geometry

Schematic courtesy of B. Bruet

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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.