Nano- and Micromechanics

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How does stuff respond when you poke it, squeeze it, or stretch it?

• (Quasi)static
  – Elastic (Young’s) modulus
  – Hardness

• Dynamic
  – Viscoelastic properties for time-dependent materials, creep or stress relaxation
  – Storage modulus, loss modulus, tan delta
Why Measure Nano- or Micromechanical Properties?

• Mechanical properties help define materials
  – Useful for optimizing applications
  – Flexibility, biomechanical compatibility
  – Crack formation, wear resistance

• Local composition variations in samples
  – Spatially-resolved mechanical testing

• Samples may be inherently small
  – Thin films, MEMS device cantilevers, nanopillars
What Mechanical Properties Do People Measure?

• Quasistatic (elastic modulus, hardness)
  – Stress vs. strain curves
  – Load (force) vs. displacement curves

• Dynamic (viscoelastic properties)
  – Properties as a function of time or frequency
  – Creep or stress relaxation
  – Storage modulus, loss modulus, tan delta
How do People Measure Mechanical Properties?

• Quasistatic (elastic modulus, hardness)
  – AFM force curves
  – Nanoindentation, microindentation
  – DMA static measurements (stress vs. strain)

• Dynamic (time-dependent properties)
  – AFM dynamic measurements
  – nanoDMA, Modulus Mapping
  – Dynamic Mechanical Analysis
How do People Measure Mechanical Properties?

- **Quasistatic (elastic modulus, hardness)**
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  - Nanoindentation, microindentation
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How do People Measure Mechanical Properties?

• Quasistatic (elastic modulus, hardness)
  – Nanoindentation, microindentation

• Elastic (Young’s) modulus
  – Related to stiffness

• Hardness
  – Related to amount of plastic deformation
Instrumented Indentation

• Different names, same technique
  – Nanoindentation
    • Indentation depths shallower than a few µm
    • Microindentation if deeper (some instruments)
  – Instrumented Indentation
  – Depth-Sensing Indentation

Poke a sample and record its response
Nanoindenter Basic Parts

- **Transducer**
- **Tip**
- **Sample**

Apply/measure force and displacement
Why Does the Instrument Frame Stiffness Matter?

Instrument is stiff so sample deforms instead

Sample

Sample is the most compliant part

Tip

Stiff frame

Measure Sample Not Apparatus
Nanoindenter Basic Parts

Nanopositioning

Transducer

Tip

Stiff frame

Sample
Nanoindenter Tips

• Tips are made of diamond or sapphire
  – Tip characteristics are well-known
  – Tip compliance is negligible

• Variety of shapes for different applications
  – Induce different deformation mechanisms
  – Berkovich, Vickers, cube corner
  – Flat punch, conospherical (bending, soft materials)

Measure Sample Not Apparatus
Microindenter Tips

- Microindentation
  - Many μm deep
  - Up to several N
- Most popular tip shape for microindentation:
  - Vickers 4-sided pyramid

Sample courtesy of James Zhu, UIUC
Nanoindentation Tips

- Nanoindentation
  - Up to a few \( \mu \text{m} \) deep
  - Up to several mN
- Most popular tip shape for nanoindentation:
  - Berkovich 3-sided pyramid

Nanoindentation residual imprint
Berkovich tip on aluminum foil
Sample Preparation

- Mount it securely
  - Don’t want to measure adhesive
  - Preferably use a substrate stiffer/harder than sample
- Make it smooth enough
  - Roughness causes uncertainties in contact area
  - Polishing can add complications
- Make it thick enough
  - Preferably ≥ several hundred nm
What Do You Get from Nanoindentation Data?

Popular Nanomechanical Properties to Measure

- Elastic modulus
- Hardness

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• Young’s/elastic modulus
  – Units: Pascal (kPa, MPa, GPa)
  – Slope of stress vs. strain curve during elastic deformation (still following Hooke’s law)
  – Proportional to slope of load vs. displacement curve in nanoindentation (stiffness)
Popular Mechanical Properties: Hardness

• Hardness
  – Units: Pascal (kPa, MPa, GPa) (sorry)
  – Here, technically “nanoindentation hardness”
  – Proportional to maximum force during a nanoindentation indent
How to Extract Values from the Data

Three Equations for Nanoindentation

#1 Elastic (Young’s) modulus
#2 Reduced modulus
#3 Hardness
Three Equations for Nanoindentation #1

Elastic (Young’s) modulus

\[ E_{\text{sample}} = \frac{1 - \nu^2_{\text{sample}}}{\frac{1}{E_{\text{reduced}}} - \frac{1 - \nu^2_{\text{tip}}}{E_{\text{tip}}}} \]

- Elastic (Young’s) modulus
- Guess the Poisson’s ratio of the sample
- Already known (diamond tips)
- Obtain using nanoindentation
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Three Equations for Nanoindentation #2

Reduced modulus

\[
E_{\text{sample}} = \frac{1}{E_{\text{reduced}}} - \frac{1 - \nu_{\text{sample}}^2}{E_{\text{tip}}} - \frac{1 - \nu_{\text{tip}}^2}{E_{\text{tip}}}
\]

\[
E_{\text{reduced}}^{(h_c)} = \frac{1}{\beta} \frac{\sqrt{\pi}}{2} \frac{S}{\sqrt{A(h_c)}}
\]

- Contact stiffness (from load—displacement curve)
- Contact depth (depth of indent)
- Area of tip in contact with sample
- Tip shape type
Thinking about the Results

Measuring the modulus

The Result

\[ E_{sample}^{(h_c)} = \frac{1 - \nu^2_{sample}}{\frac{1}{E_{reduced}} - \frac{1-\nu^2_{tip}}{E_{tip}}} \]

\[ E_{reduced}^{(h_c)} = \frac{1}{\beta \frac{\sqrt{\pi}}{2}} \frac{S}{\sqrt{A(h_c)}} \]

Given

Measured

Calibrated

Fit

Estimated
Three Equations for Nanoindentation #3

Hardness

$$H_{(h_c)} = \frac{P_{\text{max}}}{A(h_c)}$$

- Maximum load (force) during the indent
- Contact depth (depth of indent)
- Area of tip in contact with sample
Thinking about the Results

Measuring the hardness

\[ H_{(h_c)} = \frac{P_{\text{max}}}{A(h_c)} \]

Calibrated

Measured
Thinking about the Results

Dependence of results on contact area calibration

Hardness has a stronger dependence on the tip area function

Both hardness and modulus are functions of contact depth

\[ H_{(h_c)} = \frac{P_{\text{max}}}{A(h_c)} \]

\[ E_{\text{reduced}}^{(h_c)} = \frac{1}{\beta} \frac{\sqrt{\pi}}{2} \frac{S}{\sqrt{A(h_c)}} \]
Contact Area and the Tip Area Function

• Contact area between tip and sample
  – Very, very important (crucial calibration)
  – Affects hardness more than modulus
  – Depends on depth indented into sample
  – Depends on roughness

Sample courtesy of James Zhu, UIUC
How to Determine the Tip Area Function

• Indent a sample with known modulus
  – Quartz (fused silica) is standard
  – Low-modulus calibrations are complicated (usually still use quartz)

• Correct measured values to match “real” values for quartz
  – Correction defines the “tip area function”
  – Apply correction to future measurements
Example Nanoindentation Data (Quartz)

"Load—displacement curve"

Load $P$ (few $\mu$N to few mN)

Displacement $h$ (few tens of nm to few $\mu$m)
Example Nanoindentation Data (Quartz)

“Load—displacement curve”

- Loading curve
- Unloading curve

(optional) hold segment to measure creep
Popular and Trending Analysis Models

- **Oliver—Pharr**  
  Most people start here

- **JKR, DMT**  
  - When surface interactions matter  
  Sticky samples

- **Hay—Crawford**  
  - Attempt to correct for thin film complexities

Jennifer Hay (Agilent) has produced great "Nanoindentation University" webinars
Example Nanoindentation Data (Quartz)

Oliver—Pharr model:

\[ H = \frac{P_{\text{max}}}{A(h_c)} \]

Hardness = 10 GPa
Reduced modulus = 69 GPa

within uncertainty of correct values
Nanomechanical Properties as a Function of Depth

- Hardness
- (reduced) modulus

Indentation depth (tens/hundreds of nm typical)
Nanomechanical Properties as a Function of Depth

Hardness = 8.6 ± 0.2 GPa

Reduced Modulus = 65 ± 1 GPa

Just standard deviation... the real uncertainty is greater
Nanoindentation gives nanomechanical properties as a function of depth and location.

Hardness: 8.6 ± 0.2 GPa

Reduced modulus: 65 ± 1 GPa

Load [µN] vs. Displacement [nm] graph shows the relationship between load and displacement for different samples.
Useful Books about Nanoindentation

• General information
  – Anthony C. Fischer-Cripps

• Soft material applications
  – *Handbook of Nanoindentation with Biological Applications*
  – Michelle L. Oyen

• Both books are available for free online through the U of I library
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