Nanoindentation

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Mechanical Testing:
How does it respond when you poke it, squish it, or stretch it?
Choose the Right Technique for Your Sample

• Sensitive enough to measure the sample
  – Appropriate force resolution

• Spatial resolution on an interesting scale
  – Lateral resolution: 10s-100s of nm, 10s of μm, mm
  – Displacement: nm, μm, mm

nanoindentation
Small-scale Mechanical Testing

- **Nanoscale**
  - AFM
  - Nanoindenter

- **Microscale**
  - Nanoindenter
  - Microindenter

- **Milliscale**
  - Rheometer (twisting)
  - DMA (stretching, compressing)
Why Measure Nano- or Micromechanical Properties?

• Mechanical properties help define materials
  – Optimize applications
  – Flexibility, biomechanical compatibility
  – Crack formation, wear resistance, delamination
Why Measure Nano- or Micromechanical Properties?

• Mechanical properties help define materials
  – **Nanoindentation**
  – Forces: $\mu$N to mN, compatibility
  – Distances: nm to $\mu$m, distance, delamination

• Samples may be inherently small
  – Thin films, MEMS devices, nanopillars

• Local composition variations in samples
  – Spatially-resolved mechanical testing
What Mechanical Properties Do People Measure?

• **Quasistatic**
  - Elastic modulus (related to stiffness)
  - Hardness

• **Dynamic**
  - Time-dependent (viscoelastic) properties
  - Storage modulus, loss modulus, tan delta
What Mechanical Properties Do People Measure?

- **Quasistatic**
  - Stress vs. strain curves
  - Load (force) vs. displacement curves

- **Dynamic**
  - Properties as a function of time or frequency
  - Creep or stress relaxation
How do People Measure Mechanical Properties?

• Quasistatic
  – AFM force curves
  – Nanoindentation, microindentation
  – Stress vs. strain curves

• Dynamic
  – AFM dynamic measurements
  – nanoDMA, Modulus Mapping
  – Dynamic Mechanical Analysis
How do People Measure Mechanical Properties?

• Quasistatic
  – AFM force curves
  – Nanoindentation, microindentation
  – Stress vs. strain curves

• Dynamic
  – AFM dynamic measurements
  – nanoDMA, Modulus Mapping
  – Dynamic Mechanical Analysis

Remember yesterday’s talks from Scott MacLaren (AFM and force curves) and Marta Kocun (AM-FM)

More detailed talk by Marta Kocun on AM-FM tomorrow at 9am in room 280 MRL
How do People Measure Mechanical Properties?

• Quasistatic
  – AFM force curves
  – Nanoindentation, microindentation
  – Stress vs. strain curves

But this talk is about nanoindentation, more local measurements
  – nanoDMA, Modulus Mapping
  – Dynamic Mechanical Analysis

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How do People Measure Mechanical Properties?

• Quasistatic
  – AFM force curves
  – Nanoindentation, microindentation
  – Stress vs. strain curves

• Dynamic
  – AFM dynamic measurements
  – nanoDMA, Modulus Mapping
  – Dynamic Mechanical Analysis
(Instrumented) microindentation is sometimes more useful
- Indents to greater depths
- Cares less about
  - Surface roughness
  - Surface forces (adhesion)
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

Nanopositioning

Transducer

Tip

Stiff frame

Sample

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Why Does the Instrument Frame Stiffness Matter?

Instrument is stiff so sample deforms instead

Stiff frame

Tip

Sample

Sample is the most compliant part

Measure Sample Not Apparatus

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It’s Basically About the Tip and Sample

Nanopositioning

Transducer

Tip

Stiff frame

Sample
Nanoindentation 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 (pyramids)
  – Flat punch, conospherical (bending, soft materials)
Nanoindenter Tips

• Nanoindentation
  – Up to a few µm deep
  – Up to several mN

• Most popular tip shape for nanoindentation:
  – Berkovich 3-sided pyramid

Nanoindentation residual imprint
Berkovich tip on aluminum foil

atomic force microscopy image
Microindenter Tips

• Microindentation
  – Many μm deep
  – Up to several N

• Most popular tip shape for microindentation:
  – Vickers 4-sided pyramid
  – Soft materials: sphere

Microindentation residual imprints
Vickers tip on steel

bright field and dark field optical microscopy images
Contact Area Between Tip and Sample

- Crucial for getting the correct answers
- To ensure well-defined contact area between tip and sample...
- make your sample as smooth as possible
  - Polishing
  - But beware of surface damage, work hardening

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Contact Area

• Contact area between tip and sample
  – Very, very important (crucial calibration)
  – How much of your tip is applying force on how much of your sample?
  – Depends on depth indented into sample
  – Depends on roughness
Contact Area

- Contact area between tip and sample
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  - How much of your tip is applying force on how much of your sample?
  - Depends on depth indented into sample
  - Depends on roughness

Berkovich indent on aluminum foil (AFM image)
The 5% Rule

- Contact area between tip and sample
- Sample roughness should be $\leq 5\%$ of indent depth...
- ... *indent 20x deeper than surface roughness*
- Can get tricky for thin samples because of the 10% rule

This indent is not deep enough for good results (especially for hardness)

Nanoindenters indent a few microns deep, so try to keep sample roughness < 100nm (microindenter samples can be rougher)
The 10% Rule

- The substrate effect
- *Indent depth should be ≤ 10% of sample thickness*
- If indent too deep, start measuring substrate properties

thin films require care, so it’s easiest to study films ≥ 1µm thick

feels like foam

feels stiffer

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The 10% Rule

- The substrate effect
- *Indent depth should be $\leq 10\%$ of sample thickness*
- If indent too deep, start measuring substrate properties

indent here

feels like foam

feels stiffer

sample

substrate

5% + 10% rules: thin samples can’t be rough

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The 5% and 10% Rules

• 5% rule and 10% rule are just rules of thumb... the actual values are sample-dependent
  – Compliant sample on stiff substrate: can probably go deeper than 10%
  – Stiff sample on compliant substrate: probably see substrate effect at depths shallower than 10%
Performing an Indent

This is actually a non-instrumented microhardness tester.
Performing an Indent

this is when an instrumented indentation would happen

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Performing an Indent

This is actually a non-instrumented microhardness tester.
Example Nanoindentation Data (Quartz)

“Load—displacement curve”
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)

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Example Nanoindentation Data (Quartz)

“Load—displacement curve”

- Loading curve
- Unloading curve

(optional) hold segment to measure creep
Example Nanoindentation Data (Quartz)

“Load—displacement curve”

unloading curve fit for analysis
Getting “the Answer”

Reduced modulus

\[ E_{\text{reduced}} \propto \frac{S}{\sqrt{A}} \]

Hardness

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

(Actually, \( E_{\text{reduced}} = \frac{1}{\beta} \frac{S}{2 \sqrt{A(h_c)}} \), but just look up “fundamental equation of nanoindentation.”)
Why Does Contact Area Matter, Again?

Reduced modulus

\[ E_{\text{reduced}} \propto \frac{S}{\sqrt{A}} \]

Hardness

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

Contact area: projected area of tip in contact with sample at a given depth
Elastic from Reduced Modulus

Elastic (Young’s) modulus

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

- Poisson’s ratio of the sample
- Already known (diamond tips)
- Measure using nanoindentation
- Many people just quote this value

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• Many materials are somewhat viscoelastic
  – Time-dependent mechanical behavior
• Creep or stress relaxation
  – Hold a constant load or displacement for a long time
  – Beware of drift
• Dynamic testing
Nanomechanical Properties as a Function of Depth

properties can differ with depth
Nanomechanical Properties as a Function of Depth

- 5% rule
- Surface effects and calibrations matter more for shallower indents
- Hardness and (reduced) modulus
- Indentation depth (tens of nm to few µm typical)

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Nanomechanical Properties as a Function of Depth

- Hardness
- (Reduced) modulus

Indentation depth (tens of nm to few µm typical)

May start to measure substrate properties

10% rule

Indent here

Sample

Substrate

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Nanoindentation gives nanomechanical properties as a function of depth and location.

These indents were done at different places on the sample as well as at different depths.

- Hardness
- Reduced modulus
Nanoindenters are usually designed for the “engineering materials” community
- Metals, composites, non-porous materials

Using a traditional nanoindenter to study soft, compliant, porous, or sticky materials
- Usually doesn’t go deep enough
- Usually doesn’t have awesome enough force resolution

comfortable modulus range: ~MPa to many GPa
(try cantilever-based techniques for more compliant samples)
Nanoindenters are usually made for the “engineering materials” community

- Cantilever-based techniques:
  - AFM (force curves and so forth)
  - Cantilever-based nanoindenters

comfortable modulus range: ~MPa to many GPa
(try cantilever-based techniques for more compliant samples)
• Fixative changes mechanical properties!
  – Fixative makes things stiffer
  – If that’s the only way you can study your sample...
    • Do a comparative study (no absolute numbers)
    • Make control samples with just the fixative

• May need a heating cell/stage to stay at biorelevant temperatures

• May need to work in fluid
Keeping the “Hydro” in Hydrogel

• Keep wet samples wet...
  – Drying and rewetting can change properties
  – Samples may dry out during measurements

• ... and the instrument dry
  – Don’t get fluid (or vapors) into the instrument
  – Petri dishes, special tips
Sample Preparation

• Know what your surface looks like first
  – Look at it under an optical microscope
  – Check sample roughness

• Mounting the sample
  – Can’t measure mechanical properties of something that’s floating
  – Compliant glue affects results

Measure Sample Not Apparatus
Many Options for Sample Mounting

- **Glue (usually superglue)**
  - Thin layer
  - Porous samples may get partially filled with glue
- **Vacuum chuck**
- **Clamp or wrap samples**
  - Don’t stress the area you want to measure
- **Cast gels directly onto substrate**
  - Glass slide (frosted is great), Petri dish
1. Approach surface
2. Drift correction
3. Indent
4. Withdraw
5. Analyze data

The trickiest part
How to Approach Your Data

- Oliver—Pharr model
  - Elastoplastic materials

Note that contact area may change due to material behavior and contact area is important.

\[ E_{reduced} \propto \frac{S}{\sqrt{A}}, \quad H = \frac{P_{max}}{A} \]

pile-up

sink-in
How to Approach Your Data

- Oliver—Pharr model
  - Elastoplastic materials
- But your samples may be...
  - Sticky, compliant
  - Poroelastic
  - Viscoelastic
  - Poroviscoelastic
  - Thin films

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  – Poroviscoelastic
  – Thin films \(\text{sharp tips, continuous stiffness measurements}\)
Nano/micromechanical Testing Facilities at MRL

Nanoindentation and Friends (nano/microscale)

Leitz Wetzlar Miniload II Microhardness Tester

Hysitron TI-950 TriboIndenter transducers: standard, nanoDMA, high load (2.8N), AE, nanoECR

Optics11 Piuma Soft Material Nanoindenter

Coming later this summer

Image courtesy Optics11

TA Instruments Q800 DMA clamps: dual cantilever, tension

Asylum MFP-3D-SA (x2) 15µm z range 90µm x 90µm scan size

Asylum Cypher 5µm z range 30µm x 30µm scan size

DMA (milliscale)

AFM (nanoscale)

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Useful Books about Nanoindentation

- **Nanoindentation (3rd ed., 2011)**
  - Anthony C. Fischer-Cripps
  - Classic text on nanoindentation

- **Handbook of Nanoindentation with Biological Applications**
  - Michelle L. Oyen
  - Soft materials people: read this one

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

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