NANO-INDENTATION
Basics, theory, principles and techniques

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OUTLINE

• Why Nanoindentation?
• Principle of Nanoindentation
• Applications
• Instrumentation
• Theoretical analysis (indentation with conical and non-conical indenter, Oliver-Pharr method)
• Practical indentation
Hardness test on the nanoscale

The idea of nanoindentation arose from the realization that an indentation test is an excellent way to measure very small volumes of materials. The only problem is determining the indentation area. For the indents below 1 μm in size the resolution of optical microscope is insufficient. To solve this problem depth sensing indentation method was developed. In this method the load and displacement of the indenter are recorded during indentation. Mechanical properties of the indented volume can be determined without seeing the indent.

SEM micrograph of the indent made in nc-Cu by Berkovich indenter under the load of 3mN.
Why nanoindentation?

Definition: Mechanical probing of a material surface to nm-scale depths, while simultaneously monitoring LOAD and DEPTH.

1. Materials engineering: Optimizing material composition, structure, processing for particular applications (bulk and thin films)
2. Mechanics of small volumes: Understanding whether thin films, lines, dots have different mechanical properties than bulk counterparts
3. Material physics: Measuring deformation processes such as dislocation nucleation, crack growth, etc.
What is nanoindentation?

Show variables in response:
- C = loading curvature
- $\frac{dP}{dh}$ = unloading stiffness
- $P_{\text{max}}$, $h_{\text{max}}$
- $W_p$ = plastic work of indentation
- $W_t$ = total work of indentation
Hysitron’s Triboindenter
Current Nanoindentation application areas include...

- Automotive
- Bearings
- Biomedical Devices
- Ceramics
- Composites
- Contact Lenses
- Cutting Tools
- Hard Coatings
- Laminates
- Magnetic Disks
- Microelectronics
- Optical Coatings
- Optical Disks
- Packaging Materials
- Paints
- Paper Coatings
- Pharmaceuticals
- Photographic Film
- Polymers
- Powders
- Printing Plates
- Semiconductors
- Thin Film Adhesion
- Turbine Blades

...future application areas will be in?
## Where does depth-sensing indentation fit in?

<table>
<thead>
<tr>
<th>SPM</th>
<th>Nanoindentation</th>
<th>Macro-hardness</th>
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</thead>
<tbody>
<tr>
<td>•  <em>Surface</em></td>
<td>•  <em>Surface/near-surface</em></td>
<td>•  <em>Bulk</em></td>
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<tr>
<td>•  Friction</td>
<td>•  Mechanical properties of thin films and coatings</td>
<td>•  Hardness of thick coatings and bulk materials</td>
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<tr>
<td>•  Adhesion (local)</td>
<td>•  Hardness</td>
<td>•  Fracture toughness of bulk materials</td>
</tr>
<tr>
<td>•  (Wear)</td>
<td>•  Modulus</td>
<td></td>
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<tr>
<td>•  Topography</td>
<td>•  Creep</td>
<td></td>
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<tr>
<td>•  Role of specific chemical interactions?</td>
<td>•  Depth-profiling</td>
<td></td>
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<td></td>
<td>•  Wear resistance</td>
<td></td>
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<tr>
<td></td>
<td>•  Interfacial adhesion</td>
<td></td>
</tr>
<tr>
<td></td>
<td>•  Fracture toughness</td>
<td></td>
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</tbody>
</table>

MD simulations of nanoindentation of gold nanoparticles (D. Mordehai)
The advantage of Hysitron is a combination of nanoindenting head with the SPM positioning stage. This allows a precise selection of the object to be tested. The SPM is operating in the STM mode and the Control Unit is “cheated” by providing the voltage from capacitance sensor instead of tunnel current.
What is Measured?

Techniques
• Nanoindentation
• Microindentation
• Load-partial-unload
• High temperature testing
• Continuous compliance

Properties
• Hardness
• Elastic modulus
• Fracture toughness
• Adhesion
• Creep
• Elastic and plastic work
• Elastic recovery parameter
• Stress-strain
What is measured?

For calculation of nanohardness the knowledge of $h_c$ (contact depth at maximal load) is necessary.

According to Oliver and Pharr (1992)

$$H = \frac{P_{\text{max}}}{A_c(h_c)}$$

where $\varepsilon=0.72$ for conical indenter and $\varepsilon=0.75$ for paraboloid of revolution. $S$ is a contact stiffness.

$$A_c = 3\sqrt{3}h_p^2 \tan^2 65.3 = 24.5h_p^2$$

for Berkovich indenter
Young’s Modulus, $E$

- $E$ is calculated based on the Sneddon equation:

$$E_r = \frac{\pi}{2\beta} \frac{dp}{dh} \frac{1}{\sqrt{A_c}}$$

Later, Pharr, Oliver and Brotzen were able to show that the equation is a robust equation which applies to tips with a wide range of shapes.

- where $E_r$ is the composite (Reduced)Young’s modulus,

- $\frac{dp}{dh}$ is the contact stiffness and

- $\beta$ is a correction coefficient near to 1

- $\beta=1.034$ for a Berkovich tip

- $E$ can be calculated as:

$$\frac{1}{E_r} = \frac{1 - \nu^2}{E} + \frac{1 - \nu_i^2}{E_i}$$

Er is the reduced modulus of indentation contact, $E_i$ and $\nu_i$ are the elastic modulus and Poisson’s ratio of the indenter respectively.

$E$ and $\nu$ are the elastic modulus and Poisson’s ratio of the test sample.
Determination of Plasticity index ($\psi$)

- It is usually used to characterize
  - the elastic-plastic response of a material under external stresses and strains.
- The plasticity index can be calculated as follows:
  \[
  \psi = \frac{A_1 - A_2}{A_1}
  \]
  where $A_1$ is the area under the loading curve and $A_2$ is the area under the unloading curve.
- The plasticity index is in the range of $0 < \psi < 1$.
- $\Psi = 0$ represent the fully-elastic and
- $\Psi = 1$ denotes fully-plastic behavior of materials.

Fig. 1. The schematic load-displacement curve in nanoindentation test.
Nanoindentation-induced adhesion failure

- indentation + residual stress = coating failure
Combining of Laugier proposed toughness model and Ouchterlony’s radial cracking modification factors, fracture toughness can be determined.

Fracture toughness expression

\[ K_c = 1.073 \times v (a/l)^{1/2} (E/H)^{2/3} \frac{P}{c^{3/2}} \]
Creep measurement

- Plastic deformation in all materials is time and temperature dependent.

- Important parameter to determine is the strain rate sensitivity.

- The average strain rate can be given by

\[ \varepsilon_{ind} = \frac{1}{h_c} \frac{dh_c}{dt} \]

- It can be done by experiments at different loading rate or by studying the holding segment of a nanoindentation.
How does nanoindentation work?

Procedure:

1. Prepare sample surface:
   Flat, Parallel faces, Smooth
2. Find region of interest on surface
3. Program load profile
4. Program spatial matrix
5. Execute experiment
6. Analyze data to obtain properties
Indenter Tips

### Sharp 3-sided indenters

- **Berkovich** 
  - Angle: $a = 65.03^\circ$ 
  - Mod. Berkovich: $a = 65.27^\circ$ 
  - Available as Traceable Standard

- **Cube corner** 
  - Angle: $a = 35.26^\circ$ 
  - Available as Traceable Standard

- **3-sided Custom** 
  - Sides: $a > 20^\circ$

- **Sharpness** 
  - TEM micrograph

### 4-sided indenters

- **Vickers** 
  - Standard Vickers indenter: $a = 68.00^\circ$

- **Knoop** 
  - Standard Knoop indenter defined by 2 angles: $d = 172.50^\circ, g = 130.00^\circ$

- **4-sided Custom** 
  - Custom 4-sided indenters: $80^\circ > a > 20^\circ$

- **End Line** 
  - TEM micrograph

### Cone Indenters

<table>
<thead>
<tr>
<th>Indenter</th>
<th>Contact Area</th>
<th>Eq. cone angle</th>
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</thead>
<tbody>
<tr>
<td>Berkovich</td>
<td>$3\sqrt{3}h^2 \tan^2 65.27 = 24.5h^2$</td>
<td>70.3°</td>
</tr>
<tr>
<td>Vickers</td>
<td>$4h^2 \tan^2 68 = 24.5h^2$</td>
<td>70.3°</td>
</tr>
<tr>
<td>Knoop</td>
<td>$2h^2 \tan 86.25 \tan 65 = 65.43h^2$</td>
<td>77.64°</td>
</tr>
<tr>
<td>Cube corner</td>
<td>$3\sqrt{3}h^2 \tan^2 35.26 = 2.6h^2$</td>
<td>42.278°</td>
</tr>
</tbody>
</table>

Figure 6  CONE INDENTERS
Issues

• Initial penetration is an unavoidable part of the depth that is lost before the contact is detected. It can be neglected if the contact force is set small or estimated using extrapolation to the zero force level.

• Surface roughness. If the surface roughness is too large relative to the penetration depth than the contact equations become invalid.
Other Issues

- The **layer thickness** must be larger (approx. 10) than the indent's depth.
- Otherwise, the substrate influences the measurement and evaluation of material constants.

- **Material creep** can influence the evaluation of even the elastic properties.
- Using long dwell periods can minimize these effects.

- If the dwell time is absent, the sample will continue to deform viscoplastically, while the load is being removed.
- This will significantly distort the shape of the unloading curve leading to inaccurate modulus measurement.
TIME-DEPENDENT MATERIALS

• Many materials (plastics, metals, wood, concrete, etc.) exhibit time-dependent behavior under steady load conditions called creep.
• During indentation test, creep acts during the whole loading history.
• It manifests itself mainly during the dwell phase and may be observed also at the unloading portion of the P-h curve.
• Standard elastic parameters such as hardness and modulus are affected by creep because the slope of the P-h curve computed from the upper part of the unloading curve contains not only elastic but also creep deformations.
Type of Sample

• Materials such as metals, semiconductors, ceramics, and polymers, as well as biomedical and biological samples.
• Samples may be in various formats, such as bulk materials, thin films, and composites.

Figure 1: 20 μm scan of wood cells after nanoindentation tests around cell wall.
Sample Preparation

• **Sample surface must be:**
  
  ▪ Flat (with plan-parallel top and bottom surfaces).
  ▪ Clean (free of dust and adhesive particles that can contaminate (i) the measurement and (ii) the tip).
  ▪ Smooth (surface roughness must be checked and kept much less than indentation depth).

• **Ways of preparation:**
  
  ▪ Parallel cutting of both top and bottom surfaces (e.g. by precise diamond saw).
Sample Preparation

• Cleaning with pressurized air or washing in alcohol and in an ultrasonic bath.
• Polishing (using emery papers, diamond pastes and solutions, polishing on cloth)

• Ways of checking:
  ▪ Optical or electron microscopy
  ▪ AFM, 3-D image, exact roughness computation over the scanned area
  ▪ Typical indentation depth 100 - 500nm requires roughness 10 - 50nm.
In-Situ Testing Techniques Used:

**In-situ SPM imaging:** for qualitative surface analysis before and after testing and quantifying surface roughness

- **Nanoindentation:** for quantitative material properties including hardness, modulus, and stiffness; depth profiling

- **Nanoscratch:** ramping force nanoscratch for quantification of film delamination/breakthrough

- **Nanoscratch:** reciprocating nanoscratch for tribology, friction, and wear failure

- **ScanningWear:** for wear resistance and wear volume quantification
NanoScratch Testing:

• Nanoscratch Resistance
• Interfacial Adhesion of Thin Films
• Delamination
• Lateral Force Measurement
• Mar Resistance

Post-test *in-situ* SPM image of an indent, scratch, and ScanningWear test within a 5 μm test area.
Principle of Scratching
Nano Wear

Figure 2: In situ SPM image of wear track made on DLC film to determine wear resistance of coating.
ScanningWear Features

• Programmable forces for precise load control within a wear box
• Adjustable wear track size from <1 µm to 100 µm
• Single or multiple pass wear testing capabilities
• Wear volume easily measured using *in-situ* SPM imaging
• Roughness measurement utility as standard
• Sensitive enough to measure <1 nm of material removal
• Ability to test in fluid medium such as lubricants and oils.
Ultra-Thin Film Applications:

Optimizing thin film properties requires a well-understood relationship between processing parameters, modeling, and model confirmation through novel testing techniques.

- Microelectronics
- Optics and optical coatings
- MEMS devices
- Hard and corrosion resistant coatings
- Photovoltaics
- Shape memory alloys
Effect of Surface Roughness

Fig. 12. Cross-sectional schematic view of the wear mechanism model for the polished (a), dimpled (b) and over-coated dimpled specimens.
Image showing a residual high-load indent impression with low-load indentation tests placed along the pile-up.
In-situ SPM Imaging

• The *in-situ* images are obtained by raster scanning the indenter probe over the sample surface to allow for pre- and post-test observation of the material surface.

• The **TI 950** offers quantitative topographical imaging with an extremely low imaging contact force (≤70 nN).

![Figure 1: SPM image of polished nitride hard coating. (left: before test, right: after test, image scan size: 20μm)](image-url)
**In-situ SPM imaging**

- *In-situ* SPM imaging allows observation and quantification of material deformation, such as:
  - pile-up,
  - wear volume,
  - crack length and
  - scratch morphology, incurred during testing
Hardness Mapping: Example

- A 10x10 automated grid of indents was performed on a defect-free region of the sample identified by SPM imaging and indents were spaced 1 μm apart.

- Indentation results were analyzed and mapped using the Hysitron TriboAnalysis™ software package.

Figure 1: Hardness map showing distinct ferrite and martensite phases.  
Figure 2: Reduced modulus map of a martensitic/ferritic dual-phase steel.