TSOM Webinar on Instrumented Indentation

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11-19-2015

Atomic Force Microscopy
3D Optical Microscopy
 Fluorescence Microscopy
 Tribology
 Stylus Profilometry
 Nanoindentation

Innovation with Integrity
Outline

• Motivation
• Theory of instrumented-indentation
• Experimental aspects
• NanoForce
• Application examples
Motivation

Why do we need mechanical properties?

Understand and prevent irreversible deformation
Motivation

Fundamentals of mechanical properties

Stress and Strain:

- **Normal Stress**
  \[ \sigma = \frac{F}{A} \]

- **Normal Strain**
  \[ \varepsilon = \frac{\Delta l}{l} \]

- **Shear Stress**
  \[ \tau = \frac{F}{A} \]

- **Shear Strain**
  \[ \tan \gamma = \frac{\Delta x}{h} \]

Constitutive Equation:

\[ \sigma_{ij} = S_{ijkl} \varepsilon_{kl} + S_{ijklmn} \varepsilon_{kl} \varepsilon_{mn} + \ldots \]
Motivation

Measuring mechanical properties

Hooke: \( \sigma_{ij} = S_{ijkl} \varepsilon_{kl} \)

homogenous, isotropic:

\[
\nu = -\frac{\varepsilon_{12}}{\varepsilon_{11}}, \quad E = \frac{\sigma_{11}}{\varepsilon_{11}}
\]

Test & specimen:
- Uniaxial tension/compression
- Bending
- Acoustic wave

Properties:
- \( E, \nu, K, G, \)
- \( Y_0, K_I \)

\( \Rightarrow \) Mechanical properties on macroscopic scale
Motivation

Hardness test

Concept: 1. Apply load.
2. Analysis of residual imprint after load removal

VICKERS-Hardness number

\[ HV = 1.8544 \frac{F}{d^2} \]

[Applied load] = kgf
[Diagonal] = mm

Fracture-Toughness

\[ K_I = 0.015 \left( \frac{a}{l} \right)^{\frac{1}{2}} \left( \frac{E}{H} \right)^{\frac{2}{3}} \frac{F}{c^{\frac{3}{2}}} \]

F…Applied load (N)
H…Hardness (Gpa)
E…Young’s Modulus (Gpa)
c,a,l…distances from img (m)

Pros:
• Simple application
• Small specimen
• Easy to scale

Cons:
• HV difficult co-relate to physical properties
• Scaling limited by imaging resolution
• No elastic properties
Motivation

Instrumented indentation

Concept: 1. Accurate measurement of compliance curve
2. Elastic analysis of tri-axial stress-state in contact

Nano-indentation

Typical load: ~10 mN
Typical depth: ~100 nm

Property access: $E$, $H$, $Y$, $K_V$, $\sigma_{\text{Rep}}(e_{\text{Rep}})$

$\Rightarrow$ Mechanical properties on mesoscopic scale
Theory of Nano-indentation

Fundamentals of contact mechanics

Obtaining contact stress field and surface deformation

$$u_z = \frac{1 - \nu^2}{\pi E} \int \int p(r, \phi) dr d\phi$$

Superposition

Point-contact solution

$$\sigma, \varepsilon \neq ?$$
### Theory of Nano-indentation

#### Elastic surface deformation

<table>
<thead>
<tr>
<th>Contact Geometry</th>
<th>Pressure distribution</th>
<th>Mean pressure</th>
<th>Surface Deflection (r=0)</th>
<th>Surface Deflection (r=a)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Flat Punch</strong></td>
<td>$\frac{\sigma_z}{p_m} = -\frac{1}{2} \left( 1 - \frac{r^2}{a^2} \right)^{\frac{1}{2}}$</td>
<td>$p_m = \frac{1 - \nu^2}{2E\alpha} F$</td>
<td>$h = \frac{1 - \nu^2}{2E\alpha} F$</td>
<td></td>
</tr>
<tr>
<td><strong>Sphere (Hertz)</strong></td>
<td>$\frac{\sigma_z}{p_m} = -\frac{3}{2} \left( 1 - \frac{r^2}{a^2} \right)^{\frac{1}{2}}$</td>
<td>$p_m = HB = \left( \frac{4E\alpha}{3\pi} \right) \frac{a}{R}$</td>
<td>$h = \left( \frac{3F}{4E\sqrt{R}} \right)^{\frac{3}{2}}$</td>
<td>Sneddon: $f(r) = Br^n$</td>
</tr>
<tr>
<td><strong>Cone (Sneddon)</strong></td>
<td>$\frac{\sigma_z}{p_m} = -\cosh^{-1} \frac{a}{r}$</td>
<td></td>
<td>$h = \left( \frac{\pi(1 - \nu^2)F}{2E\tan \alpha} \right)^{\frac{1}{2}}$</td>
<td>$h = \frac{F}{S}$ with $\epsilon = \frac{m}{\Gamma \left( \frac{m}{2m-1} \right)}$</td>
</tr>
</tbody>
</table>
Theory of Nano-indentation

Hertzian contact stress

Tensile stress

Von Mises stress

\[ \sigma_{\text{von Mises}} = \sqrt{\frac{1}{2} \left( (\sigma_{xx} - \sigma_{yy})^2 + (\sigma_{yy} - \sigma_{zz})^2 + (\sigma_{zz} - \sigma_{xx})^2 + 6(\tau_{xy}^2 + \tau_{yz}^2 + \tau_{zx}^2) \right)} \]

Related material failure modes

Hertzian cone fracture
Residual imprint by sub-surface plasticity
Theory of Nano-indentation

Elastic-plastic indentation

Ideal cone/Pyramid: elastic-plastic transition occurs instantly

Stress field during unloading altered by elastic constraint plastic zone

\[ \sigma = \sigma_i + \sigma_R \]
Theory of instrumented-indentation

Oliver & Pharr method

Fitting unloading curve: \( F = a(h - h_0)^m \)

Contact stiffness: \( S = \frac{dF}{dh}|_{F_{\text{max}}} = ma(h-h_0)^{m-1} \)

Contact depth: \( h_c = h_{\text{max}} - \epsilon \frac{F}{S} \)

Tip calibration: \( A_i = A_c(h_c) = c + a_0 h_c^2 + a_1 h_c^4 + a_2 h_c^6 + a_3 h_c^8 + a_4 h_c^{10} \)

Reduced modulus: \( E_r = \frac{\sqrt{\pi}}{\beta 2\sqrt{A_c}} S \)

Indentation modulus: \( E_p = \frac{1 - \nu_i^2}{\frac{1 - \nu_p^2}{E_i} - \frac{1 - \nu_i^2}{E_r}} \)

Indentation hardness: \( H_p = \frac{F}{A_i} \)

- \( A_i, m, a, h_0 \ldots \) fit parameters;
- \( \epsilon, \beta \ldots \) contact model parameters (Sneddon, Oliver&Pharr resp.);
- \( E_i, \nu_i \ldots \) elastic indenter properties;
- \( \nu_p \ldots \) Poisson ratio of specimen;
Theory of instrumented-indentation

Quasi-Static vs. Dynamic indentation

**Oliver&Pharr:**

- \( m \frac{d^2 u_i}{dt^2} - \frac{\partial \sigma}{\partial x} - F_i = 0 \)
- \( \sigma = S \varepsilon \)
- \( S = \left. \frac{dF}{dh} \right|_{h_{max}} \)
- \( h_c = h_{max} - \varepsilon \frac{F}{S} \)
- \( A_c = A_c(h_c) \)
- \( H = \frac{F}{A_c} \)
- \( E_r = \frac{\sqrt{\pi}}{\beta 2 \sqrt{A_c}} S \)

**Dynamic indentation**

- \( m \frac{d^2 h(t)}{dt^2} - D \frac{dh(t)}{dt} - Kh(t) = F_0 e^{i\alpha t} \)
- \( h(t) = h_0 e^{i(\alpha + \phi)} \)

- Oscillator theory

- \( H = \frac{F}{A_c} \)
- \( E_r = \frac{\sqrt{\pi}}{\beta 2 \sqrt{A_c}} \frac{F_0}{h_0} \cos(\phi) \)
- \( E_i = \frac{\sqrt{\pi}}{\beta 2 \sqrt{A_c}} \frac{F_0}{h_0} \sin(\phi) \)

Regular Contact mechanics

Missing Contact mechanics!
Experimental aspects

Typical test setup

Calibrations:
- Tip-shape
- Load frame compliance
- In-line tool offsets

Specimen requirements:
- Roughness
- Flatness
- Mounting stiffness
Experimental aspects

High-level corrections during raw-data post-processing

- **Thermal drift**
  \[ \Delta h_{th} = \frac{\Delta h}{\Delta t_{driftholdtime}} \]
  First order estimate from drift during the unloading hold time. Validity assumes linear-elastic behaviour, stationary drift, and a total drift \( \ll h_{max}, \Delta h_{creep}, \Delta h_{hysteresis} \) etc.

- **Frame Compliance**
  \[ \Delta h_{frame} = \frac{F}{S_i} \]
  Treated as superposition of HOOKEian compliances for load frame deformation small compared to contact
  \[ \frac{1}{S_c} = \frac{1}{S_i} + \frac{1}{S_c} + \ldots \]

- **Zero-Point correction**
  \[ \Delta h_{zero} = h'' \]
  Depth at intersect of loading curve and abscissa. HERTZian contact fit with fit parameter \( A \) and the zero offset \( h''_{zero} \)
  \[ F''(h') = A(h'' - h''_{zero})^3 \]

Metrology chain corrections at a glance:

\[ h = h'' - \Delta h_{zero} - \Delta h_{th} - \Delta h_{frame} = h'' - \left( \frac{\Delta h}{\Delta t_{driftholdtime}} \right) - \left( \frac{F}{S_i} \right) - h''_{zero} \]
NanoForce
NanoForce

System overview

- Electromagnetic actuator (indentation loads between 0.2 uN and 45 mN)
- Three-plate capacitor (indentation depth up to 40 um)
- Load control
- Depth- and strain-rate control by S/W feedback control
- Berkovich tip is included; misc. different indenter geometries optional; easy switch
- In-line microscope with coaxial turret optics and AFM are standard
- Automatic multi-specimen handling (magnetic, vacuum, mech. clamp mounting)

Quantitative property assessment: HIT, EIT, E’, E”, Y, K_{IC}, \sigma_{\text{rep}}(\varepsilon_{\text{rep}}), F_{\text{Crit.}}, \text{topography}
Experimental aspects

Controlled channels:
• Force (H/W)
• Displacement (S/W FBC)
• Strain (S/W FBC)

Profile element types:
• Constant
• Increasing/decreasing
• Calculated (custom)
• Harmonic

Full loading profile:
Sequence of different element types & control modes
### NanoForce

#### System data sheet

<table>
<thead>
<tr>
<th>Indentation head</th>
<th>Load Resolution (uN)</th>
<th>0.003</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Load noise floor PP (uN)</td>
<td>0.05</td>
</tr>
<tr>
<td></td>
<td>max peak load (mN)</td>
<td>45</td>
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<tr>
<td></td>
<td>min peak load (mN)</td>
<td>0.01</td>
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<tr>
<td></td>
<td>min contact force (uN)</td>
<td>0.2</td>
</tr>
<tr>
<td></td>
<td>Displacement Resolution (nm)</td>
<td>0.0003</td>
</tr>
<tr>
<td></td>
<td>noise floor rms (nm)</td>
<td>0.1</td>
</tr>
<tr>
<td></td>
<td>max displacement (um)</td>
<td>40</td>
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<tr>
<td>Dynamics</td>
<td>Minimum Force amplitude (uN)</td>
<td>0.025</td>
</tr>
<tr>
<td></td>
<td>Maximum Force amplitude (uN)</td>
<td>1900</td>
</tr>
<tr>
<td></td>
<td>Bandwidth (Hz)</td>
<td>45-250</td>
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<tr>
<td></td>
<td>Natural frequency oscillator air (Hz)</td>
<td>120</td>
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<tr>
<td>Drift</td>
<td>System in contact (ppm/K)</td>
<td>1.54</td>
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<tr>
<td></td>
<td>Pre-test drift threshold (nm/s)</td>
<td>0.01</td>
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<tr>
<td>Indenter options</td>
<td>misc., including Berkovich, Cube corner, Vickers, Conical, Flat punch</td>
<td></td>
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<tr>
<td>Specimen holder</td>
<td>Automatic ally handled up to</td>
<td>4</td>
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<tr>
<td></td>
<td>max. diameter (mm)</td>
<td>25.4</td>
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<tr>
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<td>max. height (mm)</td>
<td>10</td>
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<tr>
<td></td>
<td>min. lateral dimension (mm)</td>
<td>0.001</td>
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<td></td>
<td>max diameter on Vacuum chuck (mm)</td>
<td>200</td>
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<td>Platform</td>
<td>Max. stage travel X (mm)</td>
<td>154</td>
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<tr>
<td></td>
<td>Max. stage travel Y (mm)</td>
<td>110</td>
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<tr>
<td></td>
<td>Max. stage travel X, useable (mm)</td>
<td>64</td>
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<tr>
<td></td>
<td>Max. stage travel Y, useable (mm)</td>
<td>65</td>
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<tr>
<td></td>
<td>Stage resolution XY (um)</td>
<td>0.1</td>
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<tr>
<td></td>
<td>Max. stage travel Z (mm)</td>
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<td>Max. stage travel Z, useable (mm)</td>
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<td></td>
<td>Stage resolution Z (nm)</td>
<td>34.6</td>
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<tr>
<td></td>
<td>Frame stiffness (10^6 N/m)</td>
<td>17.9</td>
</tr>
<tr>
<td>Imaging</td>
<td>Optical objective magnifications</td>
<td>2.5x, 8x, 20x</td>
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<tr>
<td></td>
<td>max FOV (um)</td>
<td>2241</td>
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<tr>
<td></td>
<td>min FOV (um)</td>
<td>28</td>
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<tr>
<td></td>
<td>Color</td>
<td>Yes</td>
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<tr>
<td></td>
<td>Screen Magnification max (x)</td>
<td>7217</td>
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<tr>
<td></td>
<td>Illumination</td>
<td>Bright-field std.; Dark-filed optional</td>
</tr>
<tr>
<td></td>
<td>NanoLens objective (AFM)</td>
<td>standard, topographical imaging</td>
</tr>
<tr>
<td></td>
<td>Z range (um)</td>
<td>22</td>
</tr>
<tr>
<td></td>
<td>XY range (um)</td>
<td>110</td>
</tr>
<tr>
<td>Environment</td>
<td>Thermal-, acoustic- and seismic insulation hood included</td>
<td></td>
</tr>
</tbody>
</table>
Typical Nanoindentation test

1. Test positioning
2. Applying the test method (running indent-loading profile and analysis)
3. Post inspection of tested surface area
NanoForce

How small the testing object can be (indenter tip not to miss target spot...)?

Even after subsequently adding 9 more indents, all tests remain <0.5 um away from target position

Video: ftp://tmtftp.bruker-nano.com/tmt/outgoing/NanoForce/Nano-Dart.m4v

=> NanoForce can perform NI experiments on specimen under 1 um lateral size
Instrument calibration
NanoForce

Frame Compliance $S_i$

Extraction of Load-Frame stiffness from HERTZian contact modeling

$$h_{\text{Model,Hertz}} = \left(\frac{3F}{4E_r \sqrt{R}}\right)^{\frac{2}{3}} = h_{\text{Experiment}} \frac{F}{S_i}$$

This is what we are after here...

With:
- Tip radius $R$ from AFM (calculations with radius as function of depth ($R(h)$))
- Best Fit for 3 reference materials (Fused Silica, Sapphire (0001) and Si (100))

Best fit: Frame stiffness $\sim 2.5$ N/um

(good agreement with 2.2 N/um from elastic-plastic indentation by load-over-stiffness-square method ($F/S^2$))

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Tip Area-function $A_c$

Example: modified Berkovich tip

The perfect one...

\[ A(h) = 3 \frac{\tan(\alpha)^2}{\tan\left(\frac{\gamma}{2}\right)} h^2 = 24.49h^2 \]

With:
- $\alpha$: included face-angle to axis of symmetry: 65.27°
- $\gamma$: included angle between pyramid base-lines: 60°
- $h$: height of pyramid apex above base

Reality check:

Direct tip characterization by NanoLens AFM

Tip apex radius ~40 nm
(Note: that is comparable with a typical indentation depths and cannot be neglected)

Conclusion:
Direct tip characterization if possible with NF. However, there is a quicker, and more accurate way to quantify $A_c$ ....
NanoForce

PennForce

Tip Area-function $A_c$

Indirect method:

Indentation into material with well-known properties, then reverse contact contact modeling

Typical results – dynamic indentation

- Modulus
- F/S$^2$
- Hardness
- Force-Displacement curve (corrected raw-data)

$$A_c(h_c) = \left( \frac{\sqrt{\pi}}{\beta 2E_r} S(h_c) \right)^2$$

This is corrected for $S$, already…
Typical results - quasi-static indentation:

- **Modulus**
- **Hardness**
- **Force-Displacement curve** (corrected raw-data)

**NanoForce**

Typical results - quasi-static indentation:

Typical results - quasi-static indentation:

**Tip Area-function** $A_c$

<table>
<thead>
<tr>
<th>Test 1</th>
<th>Test 2</th>
<th>Test 3</th>
<th>Test 4</th>
<th>Test 5</th>
<th>Test 6</th>
<th>Test 7</th>
<th>Test 8</th>
<th>Test 9</th>
</tr>
</thead>
<tbody>
<tr>
<td>Load (mN)</td>
<td>0.032</td>
<td>0.032</td>
<td>0.035</td>
<td>0.035</td>
<td>0.080</td>
<td>0.080</td>
<td>0.080</td>
<td>0.080</td>
</tr>
<tr>
<td>Load (mN)</td>
<td>45.10</td>
<td>45.10</td>
<td>45.15</td>
<td>45.15</td>
<td>45.15</td>
<td>45.15</td>
<td>45.15</td>
<td>45.15</td>
</tr>
</tbody>
</table>

**E(h_c) if were calculated with ideal $A_c$**
NanoForce

Results on other typical reference materials

Hardness- and Modulus-measurements on for instrument verification and calibration.

...Not any material makes a suitable reference material.

Some key requirements:

...homogenous, isotropic, linear-elastic (with well-known elastic constants),
...surface w/o contamination; low roughness
NanoForce

Surface Roughness measurement

Topographical surface pre-inspection with AFM

HPFS (Fused Silica), Contact, 256pts/ln

Result:  RMS ~0.9 nm

Impact on experimental design:
=> Indentations must be deeper than ~20 nm (>20 x h) to neglect surface micro-structure in contact model
Thin film applications
Traditional approaches to measure thin-film Hardness

Example: Hardness by Indentation into 100 nm DLC

Oliver&Pharr:

\[
S = \left. \frac{dF}{dh} \right|_{F_{\text{max}}} \\
h_c = h_{\text{max}} - \varepsilon F S \\
A_c = A_c (h_c) \\
H = \frac{F}{A}
\]

10 % rule [1]
Film hardness if data within: \( h_c < 0.1d \)

Film hardness from Interpolation [2]:

\[
H_f' = H_s \left( \frac{H_f}{H_s} \right)^{1.59}
\]


November 19, 2015
Cyclic (quasi-static) measurements of thermal-SiO$_2$ on Silicon

Strain-rate controlled indentation with Berkovich tip and 30 partial unloading cycles, applied to SiO$_2$ of different thickness on Si (100) substrate:

- **Note:**
  - Noisy data; property-depth-profile recognize-/quantify- able.
  - Modulus shows strong gradient even for the thickest film
  - Effective modulus @ ~50 nm depth: 137 Gpa, 110 Gpa, 83 Gpa, 71.8 Gpa (thin=>Bulk)

- **50 nm**
- **100 nm**
- **300 nm**

**Modulus [GPa]**:
- Bulk Si: ~164 GPa
- Bulk SiO$_2$: ~72 GPa

**Hardness [GPa]**:
- Depth [nm]

*November 19, 2015*
Dynamic measurements of thermal-SiO$_2$ film on Silicon

Strain-rate controlled indentation with Berkovich tip and superimposed 2 nm harmonic excitation amplitude @ 120 Hz on various thickness SiO$_2$ on Si (100) on substrate:

- 50 nm
- 100 nm
- 300 nm

Bulk Si: ~164 GPa
Bulk SiO$_2$: ~72 GPa

Note:
- Dynamic test data are much less noisy!
- Modulus- and hardness- gradients exhibit clear trend
- Effective modulus @ 50 nm depth is still disclosing significant substrate-effect
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Cube Corner

\[ A = \sqrt{3} \frac{\tan^2(\alpha)}{\tan^2\left(\frac{\gamma}{2}\right)} h^2 \]

\[ \alpha = a \sin\left(\tan\left(\frac{\beta}{2}\right) \cdot \tan\left(\frac{\gamma}{2}\right)\right) \]

With:
- \(\alpha\): included face-angle to axis of symmetry: 35.26°
- \(\beta\): included angle between ribs: 90°
- \(\gamma\): included angle between pyramid base-lines: 60°
- \(h\): height of pyramid apex above base
Dynamic measurements of thermal-SiO2 film on Silicon

Strain-rate controlled indentation with Cube corner tip and superimposed 2 nm harmonic excitation amplitude @ 120 Hz on various thickness on Si (100) on substrate:

Note:
- With dynamics and sharper (Cube corner) tip the Young’s modulus of SiO2 films can be measured practically independent on thickness down to 50 nm film thickness.
NanoForce

Quasi-static measurements with Cube Corner of thermal-SiO2 film on Silicon

Strain-rate controlled indentation with Berkovich tip and 30 partial unloading cycles, applied to SiO$_2$ of different thickness on Si (100) substrate:

- Depth-profile converges when substrate-effect becomes negligible

- Hardness-/Modulus coincidence at shallowest depths with sharper tip is independent on the loading dynamics (completely different models used)
What happens when we change the tip angle?

Cube corner: sharper tip angle => pile-up, radial fracture due to buildup of stress-intensity

Indentation strain goes up for CC
Static vs. instrumented Hardness on metals

Example: Hardness standard reference block 724HV1

Static:

\[ HV = \left(725.0 \pm 11.4\right) \frac{K_p}{mm^2} \]

Instrumented:

\[ H = (7.11 \pm 0.11) GPa \]

\[ H = (9.82 \pm 0.17) GPa \]

Poor agreement with traceable Hardness standard!

Topographical pile-up analysis

Correction:

\[ A_{c,AFM} = \left(1 + \frac{\Delta A_c}{A_c}\right) A_{c,OP} \]

Accurate traditional instrumented tests require correction of pile-up!
Pile-up volume scales approx. inversely-proportional with film thickness. While quasi-static measurements would become erroneous, dynamic measurements help to minimize this impact from measuring thin-film properties.
Accuracy improvement for measurements on a sub-100 nm coatings possible with:

- Noise suppression by superimposed dynamics and low-pass filtering
- Dynamics to take into account for pile-up
- Cube corner tip to reduce the apex radius
Brittle material characterization
Nano-indentation results

Fracture toughness

Step 1: Measure Hardness and Young’s modulus of the specimen
Step 2: Produce a series of indentations at different loads and surface locations
Step 3: Image indents to find critical load for fully developed set of lead corner radial cracks

Step 4: Calculate Fracture toughness:

Cube Corner [1]:
\[ K_1 = 0.036 \left( \frac{E}{H} \right)^{1/2} \frac{P}{c^{3/2}} \]

Berkovich [2]:
\[ K_1 = 1.073(0.015)(a/l)^{1/2} \left( \frac{E}{H} \right)^{2/3} \frac{P}{c^{3/2}} \]

Method inputs:
- E...Young’s modulus
- H...hardness
- P...test load
- a, c, l,...distances from image

Nano-indentation results

Fracture toughness

BK7

HPFS

Cube Corner:

\[ K_1 = 0.036 \left( \frac{E}{H} \right)^{1/2} \frac{P}{c^{3/2}} \]

Method Inputs:
- E...Young’s modulus
- H...hardness
- P...test load
- c....distance from image

BK7 glass is ~ twice as tough as synthetic Silica

<table>
<thead>
<tr>
<th>Specimen ID</th>
<th>HIT (*) (Gpa)</th>
<th>EIT (*) (Gpa)</th>
<th>EIT/HIT</th>
<th>K1C (**) (Mpa m^{1/2})</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>HPFS</td>
<td>9.20 ± 0.10</td>
<td>71.6 ± 0.5</td>
<td>7.8</td>
<td>0.440 ± 0.070</td>
<td>1 measure</td>
</tr>
<tr>
<td>BK7</td>
<td>8.30 ± 0.10</td>
<td>89.0 ± 0.6</td>
<td>10.7</td>
<td>0.997 ± 0.070</td>
<td>1 measure</td>
</tr>
</tbody>
</table>

* Average of measurements on at least 9 different surface locations at depths between (50-300) nm
** uncertainty is (Max-Min)/2
All uncertainty is standard deviation of the mean @ 2-sigma confidence
Characterization of metallic single crystals
NanoForce

Quasi-static indentation with multiple-partial unloading

Modified Berkovich tip on Aluminum (110) single crystal

- Initial elastic-plastic transition can be observed at ~20uN
- Perfect elastic partial unloading cycles (no hysteresis)
- Pop-ins appear associated to the beginning / end of a partial unloading cycle
Dynamic indentation on single crystals:

Cube Corner tip on Copper (110) and (111) orientations

- The (111) orientation appears stiffer than the (110) orientation
- The (111) orientation exhibits pop-in events at approx. 10 μN load
Indentation Creep
NanoForce

Quasi-static indentation with multiple-partial unloading and CREEP

Cube corner on Aluminum (110) single crystal

- Indentation Creep found to be CIT~6%
- Most significant contributor to depth-increase during peak-load-hold time are pop-in events (Plastic flow/ dislocation mobility)
NanoForce

(Soft) Polymers
NanoForce

(soft) Polymers

Quasi-static indentation on PDMS with cube corner tip

(...very sharp on very soft...)

- EIT=(5.4±0.2) Mpa
- HIT=(1.07±0.08) Mpa
- EIT=(2.55±0.53) Mpa
- HIT=(0.36±0.02) Mpa
- CIT=(52±9) %
- CIT=(33.7±2.6) %

- Repeatable raw-data due to precise load-control below 10 uN
- Both PDMS-blends clearly distinguished at hand of raw-curves (F(h), h(t))
- Resultant YOUNG’s modulus is in line with expectations (3.5 Mpa & 2.5 MPa resp. for PeakForce QNM)
- Indentation creep exceeds 30%
NanoForce

Adhesion
NanoForce

Adhesion

Quasi-static indentation on PDMS with 50 um conical tip

Obtain quantitative data:
- DMT modulus, starting @ ~1 Mpa
- Adhesion
- Energy dissipation
- Surface deformation

Nano-Indenter gives insights into new more complex contact physics:

Contact model with stiction (DMT)

\[ P_{\text{indenter}} = \frac{4}{3} E_r \sqrt{Rd^3} + P_{\text{Adhesion}} \]

Tip-stiction forces was measured @ approach (and pull-off) down to 2.5 um radius
Scratch
Mixed loading profile: Dynamic indentation combined with Creep and scratch
=> Conical tip R=2.5 μm on AR glass coating on float glass

- Single test method run tests to measure E’, E”, H, CIT and Scratch resistance.

Note: Coating starts chipping here.

F \_\text{crit} \approx 42 \text{mN}
Other misc. applications
**NanoForce**

**Precision of head mount and Indenter tip mounting**

**NanoForce tip shape options:**
- mod. Berkovich
- Cube Corner
- Vickers
- Conical R=Var
- Flat Punch R=Var
- ...

Requires remove/reinstall tip

and re-calibrate inline offset

**Experiment:** Measure remaining pos. accuracy after tip change

Cube Corner tip => Berkovich tip

Pos error ~7 um

Berkovich Tip => Conical tip

Pos error ~5 um

**Quiz:**
What if re-cal is skipped?:

No big problem: pos accuracy remain better 10 um !

(...and is this is not enough – best accuracy is just s fe clicks away (no new indent required))
NanoForce

Optical Microscope

- FOV All pixels
- Resolution ~500nm opt; ~1.6 nm AFM scanner
- >3000x total magnification + digital zoom
- Bright-field illumination;
- Optional: Extra objectives & Dark-field

Silicon or smooth, reflective surfaces in Bright-field…

Skin or rough, absorbing surfaces: Dark-field…

Standard

Max diag. = 5.7 mm

BF 2.5x

2.5x

2.5x

5x

8x

50x

AFM

Max diag. = 3.4 μm

Vs. dark-field…

BF

2.5x

DF

2.5x
NanoForce

Hardness measurement with Sharp (cone/pyramid) - the traditional way

Static

Concept: 1. Apply load
2. Analysis of residual imprint after load removal

Direct method....
- difficult to co-relate to physical properties
- No elastic properties

Apply static concept with NanoForce

1. Apply loading profile

2. Direct measurement of actual- or projected contact area $A_c$ by AFM...
3. Calculate Hardness
4. Convert to other hardness scales.