EPFL

Nanoindentation

2023 Characterization of Materials Summer School
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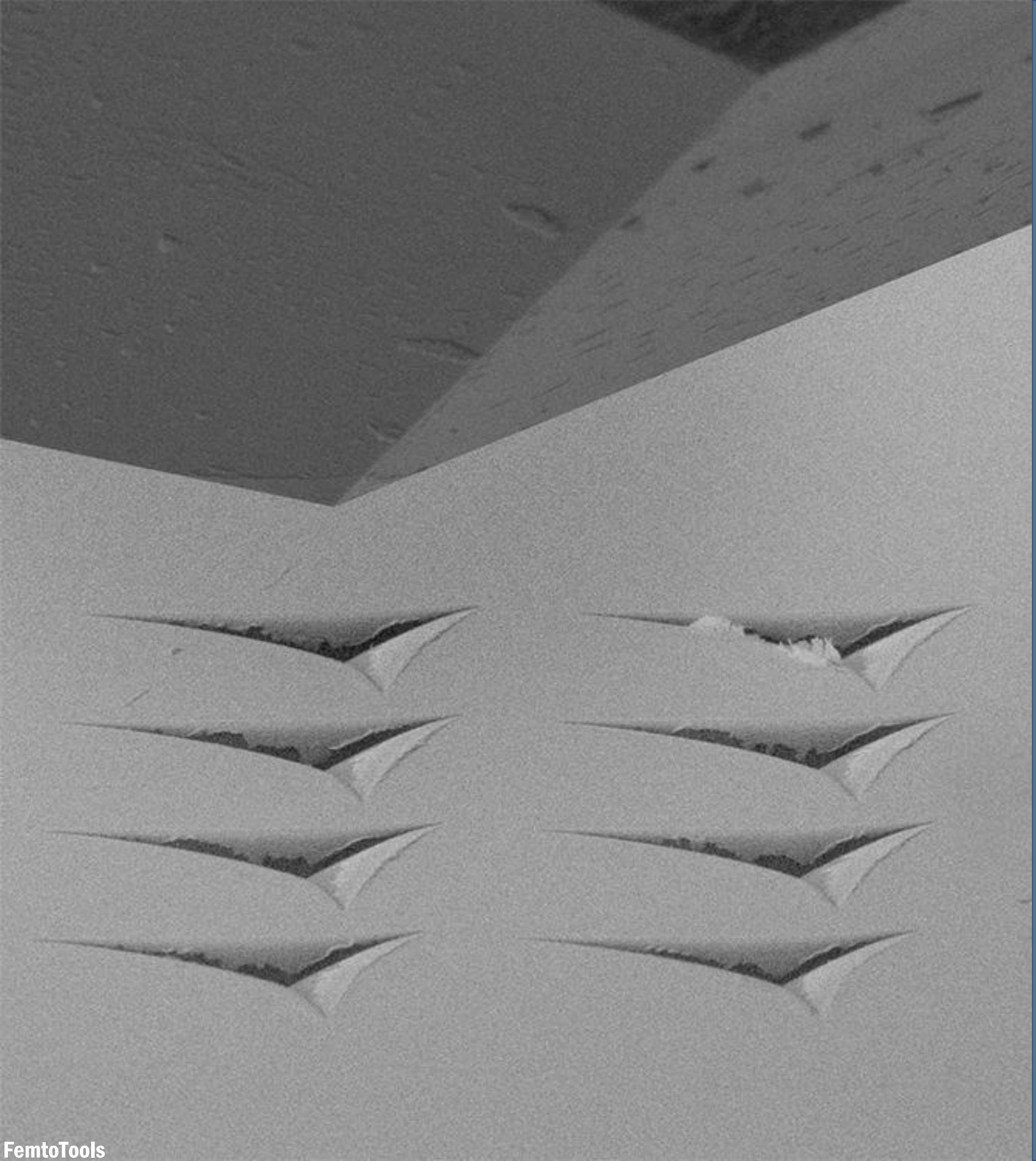
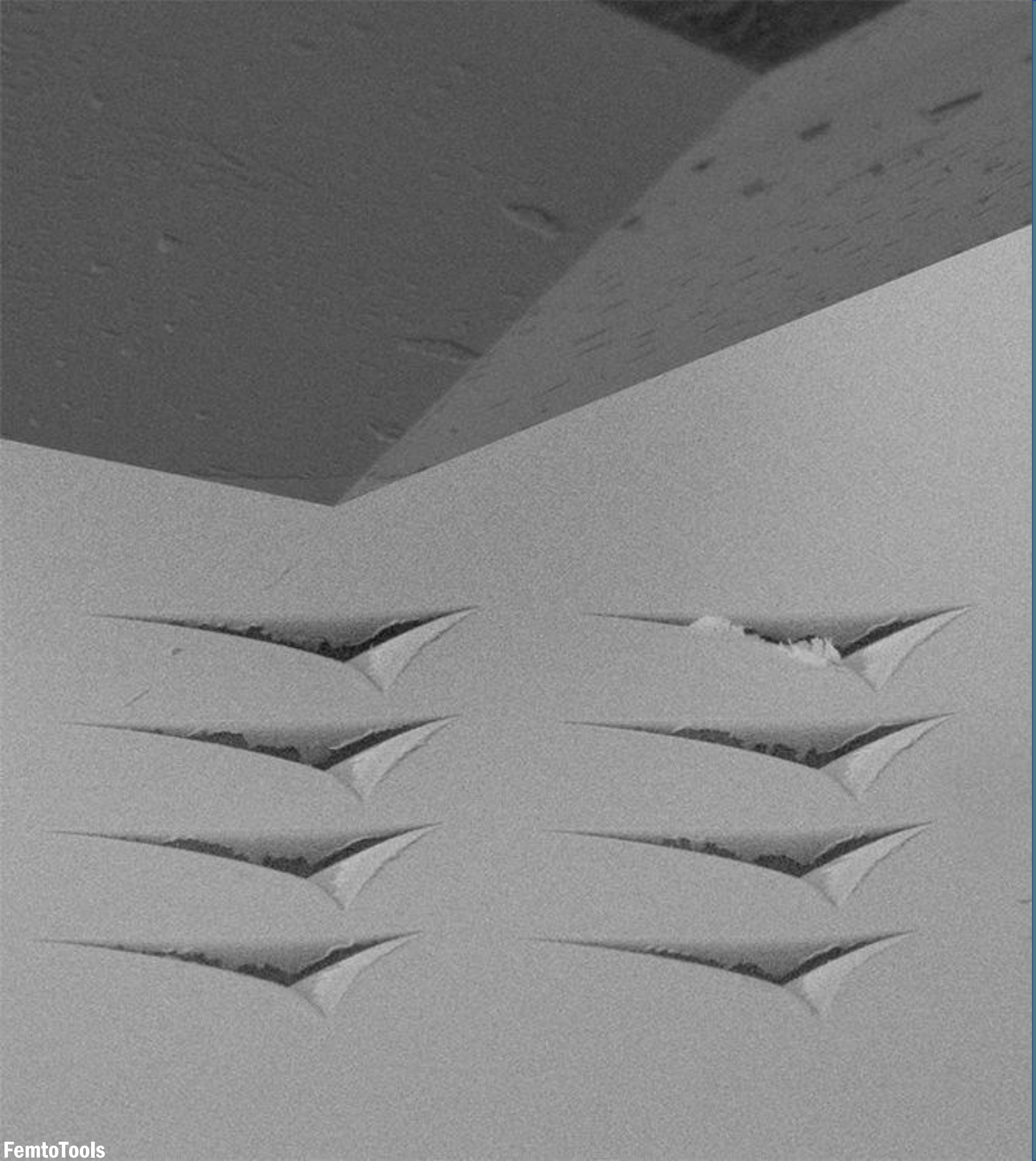


OUTLINE

- Why nanoindentation?
- Contact mechanics
- Instrumented Indentation
- Case studies



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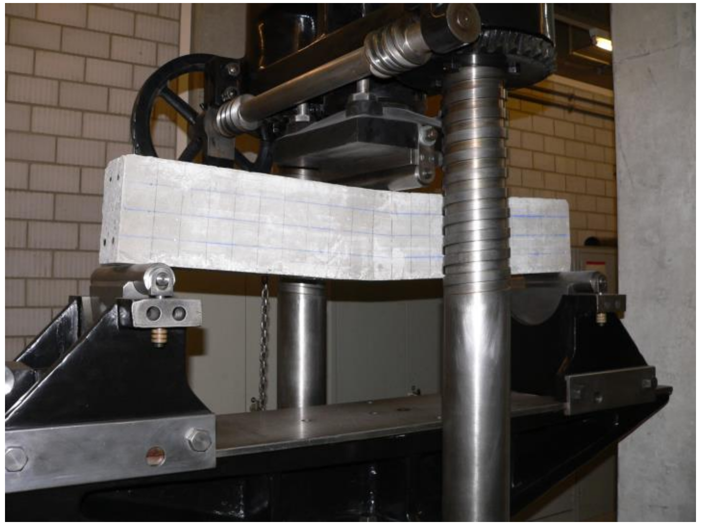
Measurement of local mechanical properties at nano-/micro- scale

- Elastic Modulus (E)
- Hardness (H)

MACRO-scale testing
(three point bending)

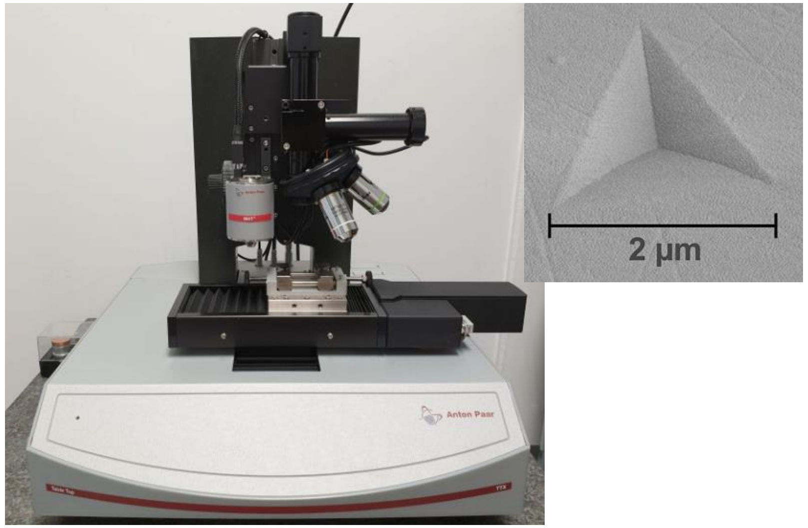


- Samples in the cm – m scale range
- Whole sample is tested. Then, it is replaced to test more

MICRO-scale testing
(Vickers hardness)

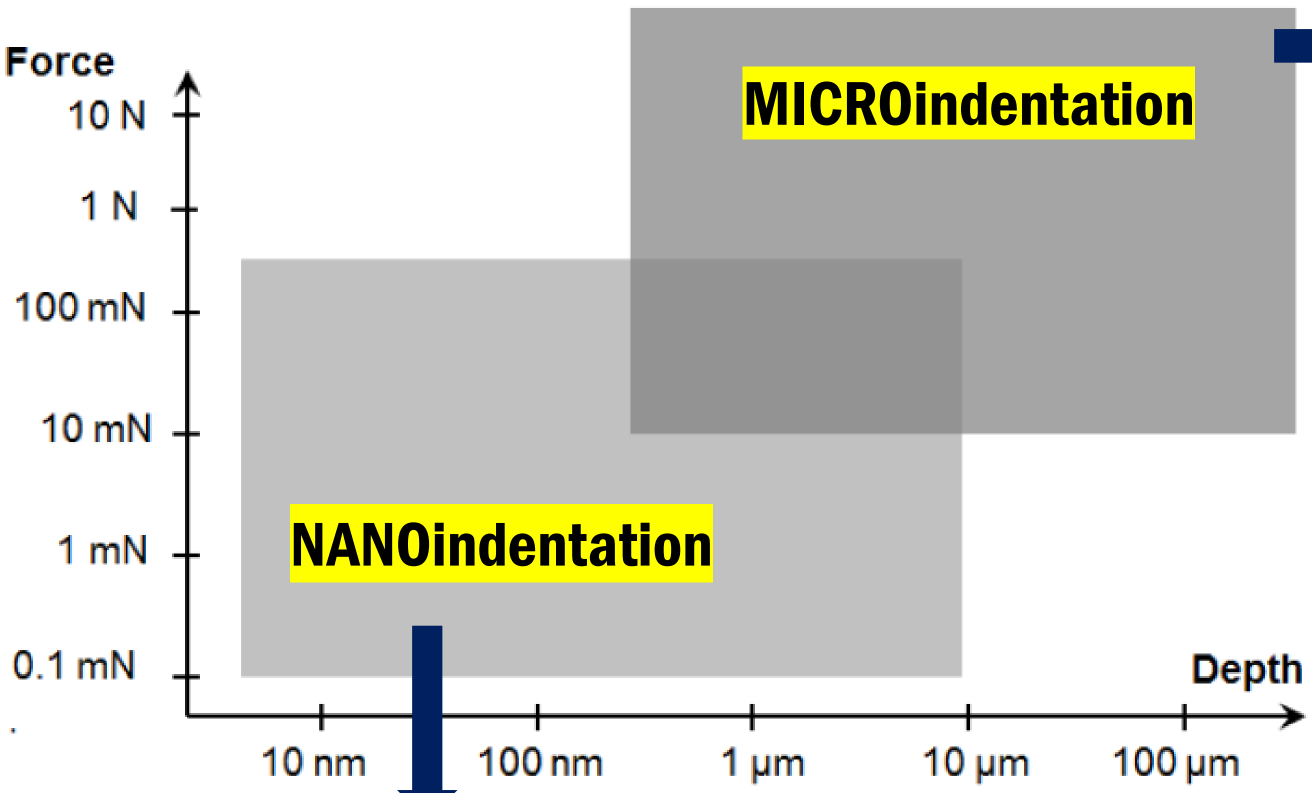
- Surface penetration ≈ 3-100 µm
- Load applied range ≈ 1-50 N
- Indentation area assessed visually

NANO-scale testing
(Berkovich nanoindentation)



- Surface penetration: nm to few µm
- Load applied range: nN to µN
- Indentation area computed from recorded displacement

Overlap between conventional microindentation & nanoindentation



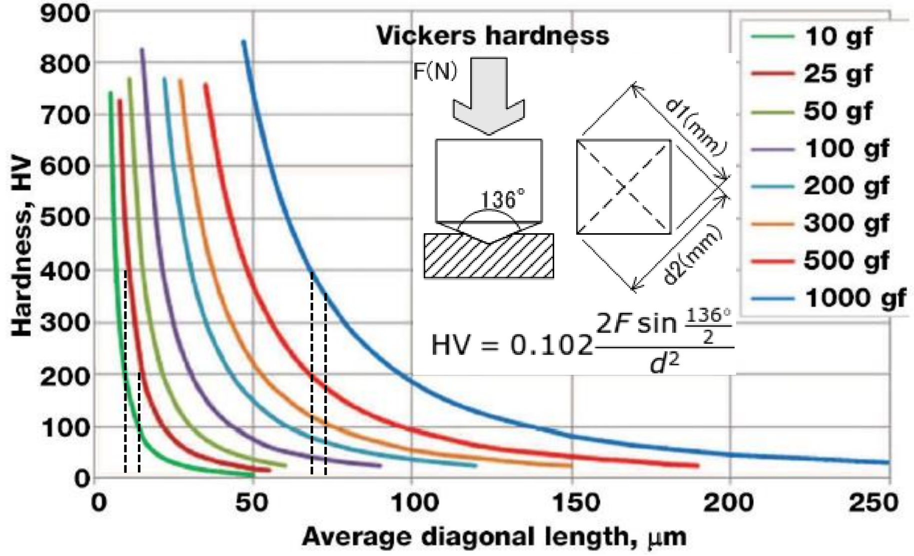
Contact area directly measured from residual imprint diagonal

Sources of measurement uncertainty:

- **Low image resolution of shallow indents**
- **Microstructural inhomogeneities**
- **Surface roughness, oxides, contaminants**
- **Geometrical imperfections of the tip apex**

Contact area computed indirectly from load-displacement curve

Indentation size effects < 1000 mN (100 gf)



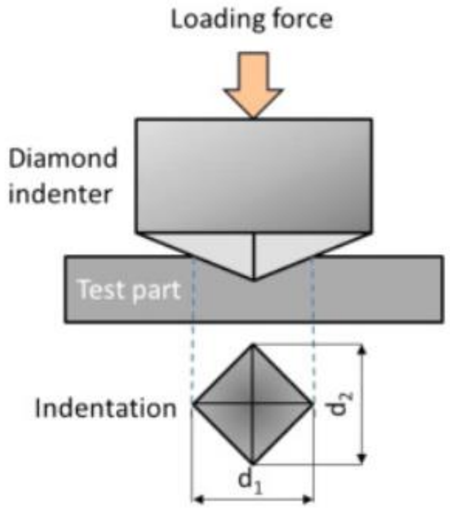
<u>ASTM E384</u> microindentation standard:
Indents should have a minimum diagonal of 20 µm, which corresponds to an indentation depth of approximately 3 µm

Any <u>indentation depth less than 3 µm</u> should use an <u>instrumented nanoindentation</u> rather than conventional microindentation.

Conventional hardness (micro- and macro-)

Test	Indenter	Shape of indentation: Side view	Shape of indentation: Top view	Load, P	Hardness number
Brinell	10-mm steel or tungsten-carbide ball	D, d	d	500 kg 1500 kg 3000 kg	$HB = \frac{2P}{(\pi D)(D - \sqrt{D^2 - d^2})}$
Vickers	Diamond pyramid	136°	L	1–120 kg	$HV = \frac{1.854P}{L^2}$
Knoop	Diamond pyramid	$L/b = 7.11$ $b/t = 4.00$ t	b, L	25 g–5 kg	$HK = \frac{14.2P}{L^2}$
Rockwell A C D	Diamond cone	120° $t =$ mm		60 kg 150 kg 100 kg	HRA HRC HRD $\}= 100 - 500t$
B F G E	$\frac{1}{16}$-in. diameter steel ball $\frac{1}{8}$-in. diameter steel ball	$t =$ mm		100 kg 60 kg 150 kg 100 kg	HRB HRF HRG HRE $\}= 130 - 500t$

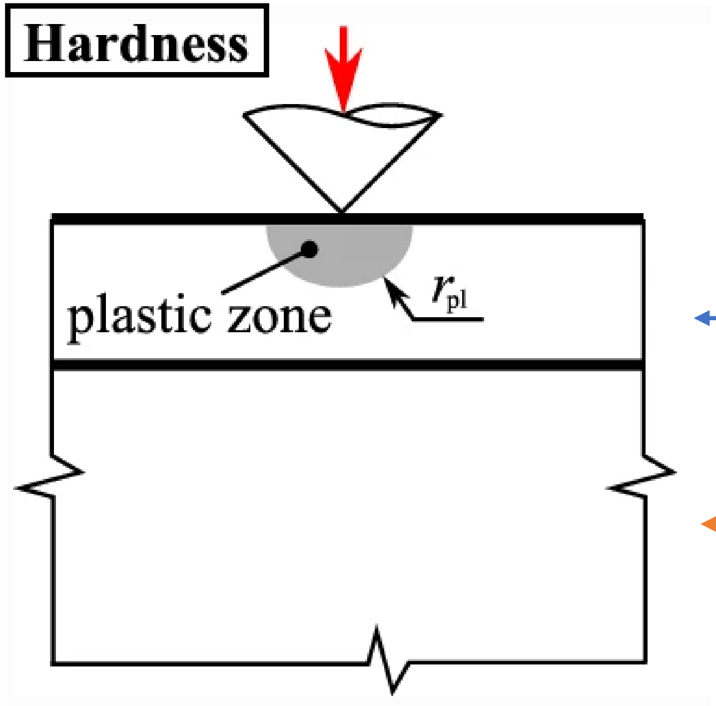
Uncertainty in a measurement of a 5 μm diagonal* of a residual impression made by a Vickers indenter is on the order of 20% when using an optical method and increases with decreasing size of indentation and can be as high as 100% for a 1 μm indent

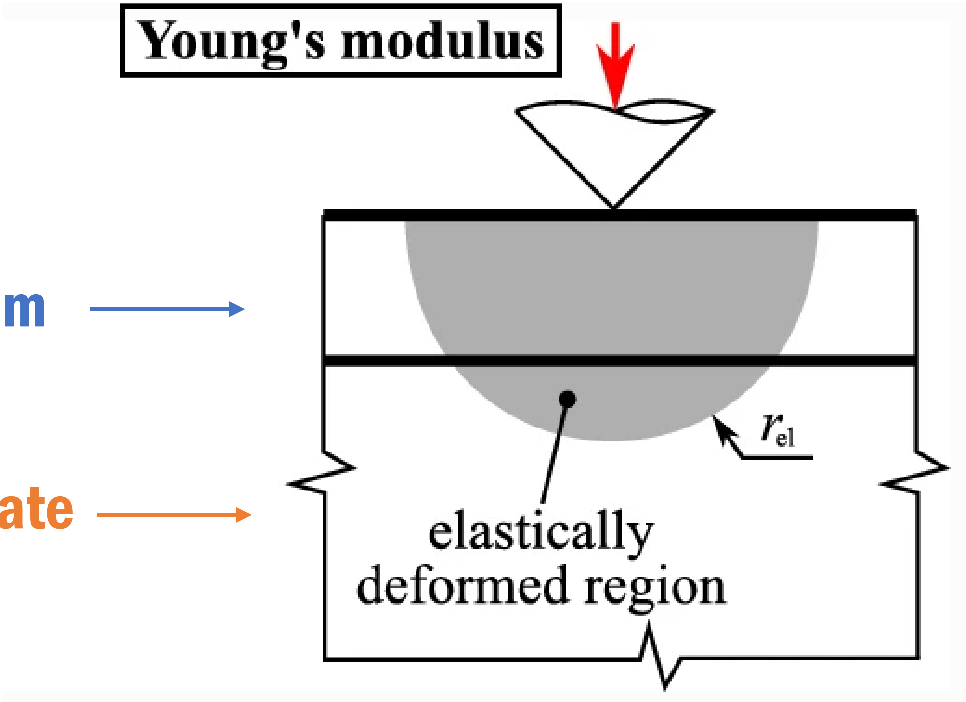


***Vickers hardness: indent diagonal (d) and penetration depth (t) are related as d = 7t**

Macro- and micro- indentation testing are not suitable for evaluating the hardness (H) of microscale features, and do not allow to measure the Elastic Modulus (E).

Thin film mechanical properties through nanoindentation





thin film

substrate

- Plastic zone (short-range field) can be "easily" confined inside the thin film
- H can be measured from indents made to 10% or less of the film thickness (Bückle, 1953)
- 10% rule originally deduced for much thicker (i.e., 8 µm) metallic films on steel substrates

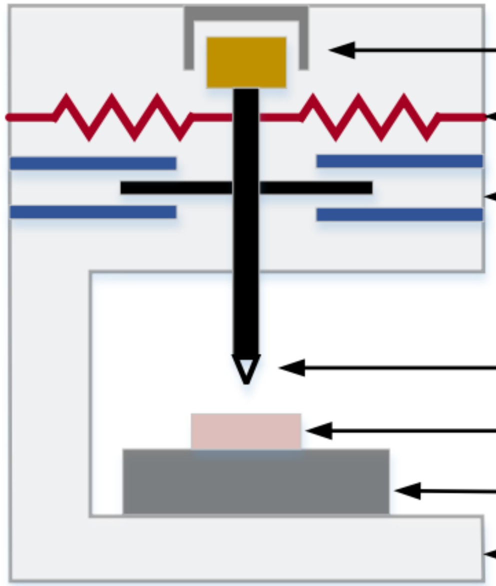
- Elastic zone (long-range field) can be at least three times larger than the plastic zone.
- E can be measured from indents made to 1% or less of the film thickness (Bull, 2019)
- Equations to correct for substrate elasticity contributions when measuring E on thin films have been proposed (King, 1987 and Hay & Crawford, 2010)

Accurate measurement of thin film mechanical properties using nanoindentation, JMR 2022 (Zak et al.)

Nanoindentation experimental set up

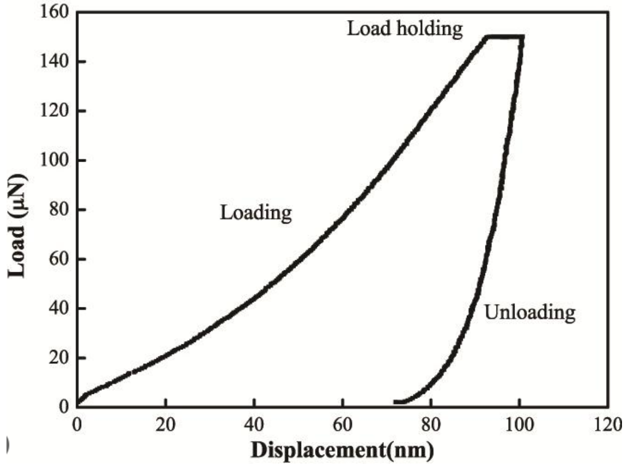
Application of a controlled load/displacement to sample surface to induce local surface deformation

Components of the nanoindenter:



- **Loading unit:** force applied by electromagnetic/electrostatic actuation
- **Detecting unit:** displacement of center plate measured by capacitive sensor
- **Leaf springs:** couple load-displacement raw data
- **Indenter tip**: different geometries- dull (spherical, flat ended), sharp (Berkovich, cube corner)
- **Sample stage:** x-y or x-y-z coordinated motorized platform to move sample
- **Microscope:** visualize and select test location
- **Control system:** dedicated software to operate instrument and analysis

Continuous record of load and displacement during the nanoindentation test



GOAL: to extract the elastic modulus (E) and hardness (H) of the specimen

Why nanoindentation?

Nanoindentation of Soft Biological Materials – Micromachines, 2018 (Qian & Zhao)

Nanoindentation test data

Conventional microindentation (i.e. Vickers, Rockwell, etc) relies on the direct measurement of the residual imprint after unloading (by optical microscopy) to calculate the residual contact area

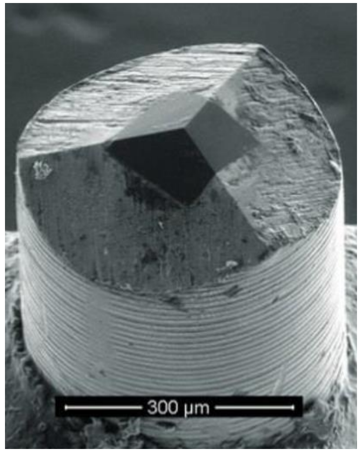
VS.

Nanoindentation tests record the displacement of the indenter beneath the specimen surface as a function of the applied indenter load

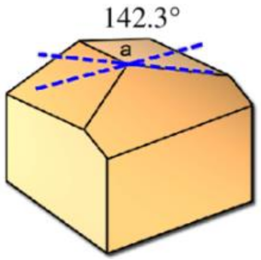
Nanoindentation tips

- Made of diamond (E=1070GPa; ν=0.07): very stiff, very hard, low friction, smooth surface
- Nanoindentation tests: generally performed with either pyramidal or cono-spherical indenters

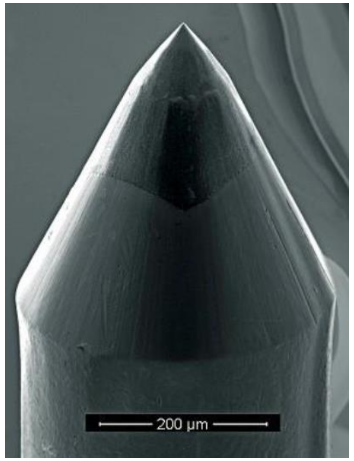
Berkovich (pyramidal)



- Most frequently used tip
- Induces sample plasticity at very small loads → H, E
- Not easily damaged, readily manufactured
- Large included angle (142.3°) minimizes friction
- Ideal for most testing purposes
  - Bulk materials
  - Thin films
  - Scratch testing
  - Wear testing

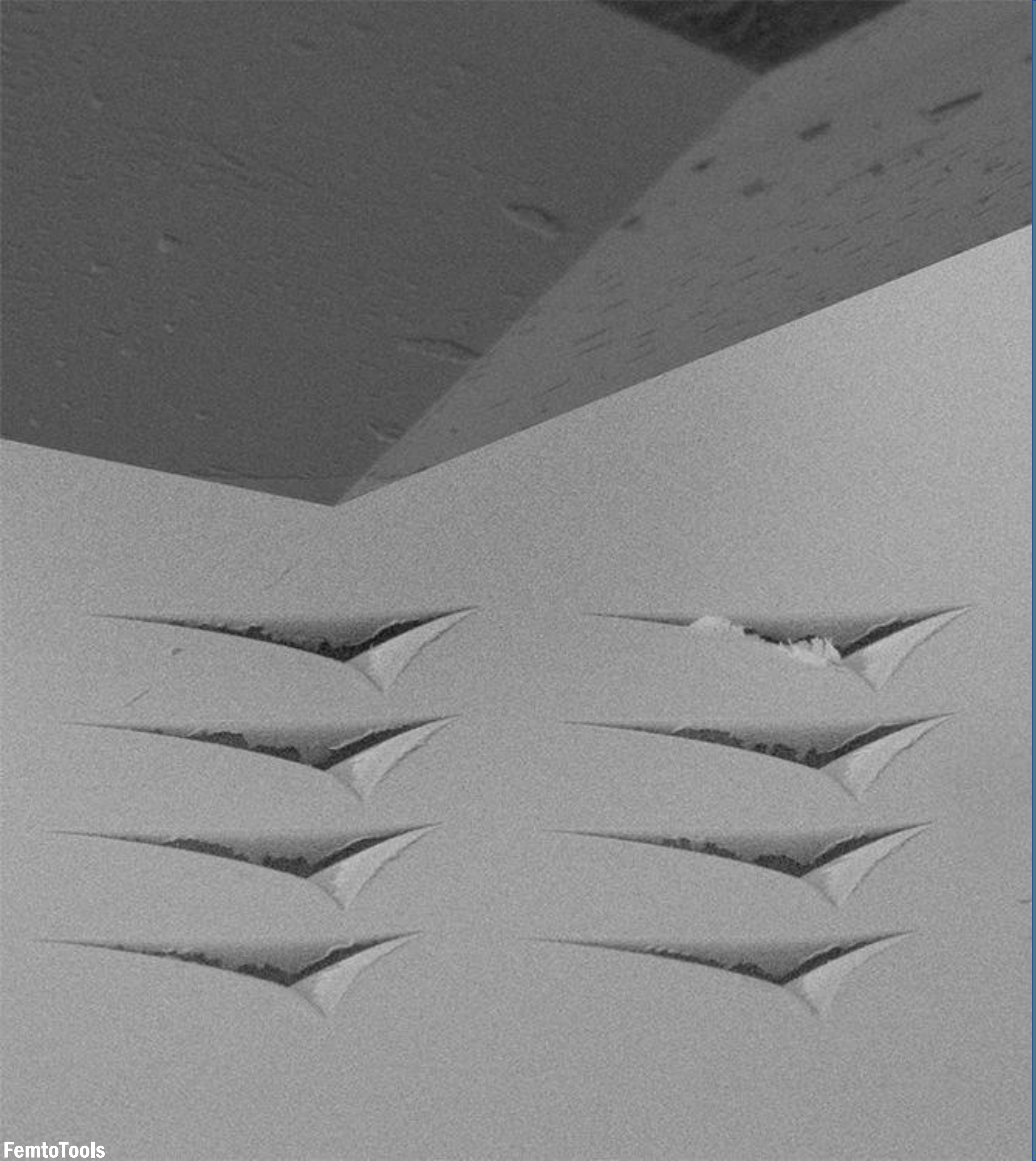


Cono-spherical



- Cylindrical symmetry → modeling
- No sharp edges, stress concentration
- Sharp tip, difficult manufacturing
- Slow transition from elastic to plastic regime (yielding, work hardening)
- Applications:
  - Scratch testing
  - Wear testing

How to select the correct indenter tip – Support note, Agilent Technologies



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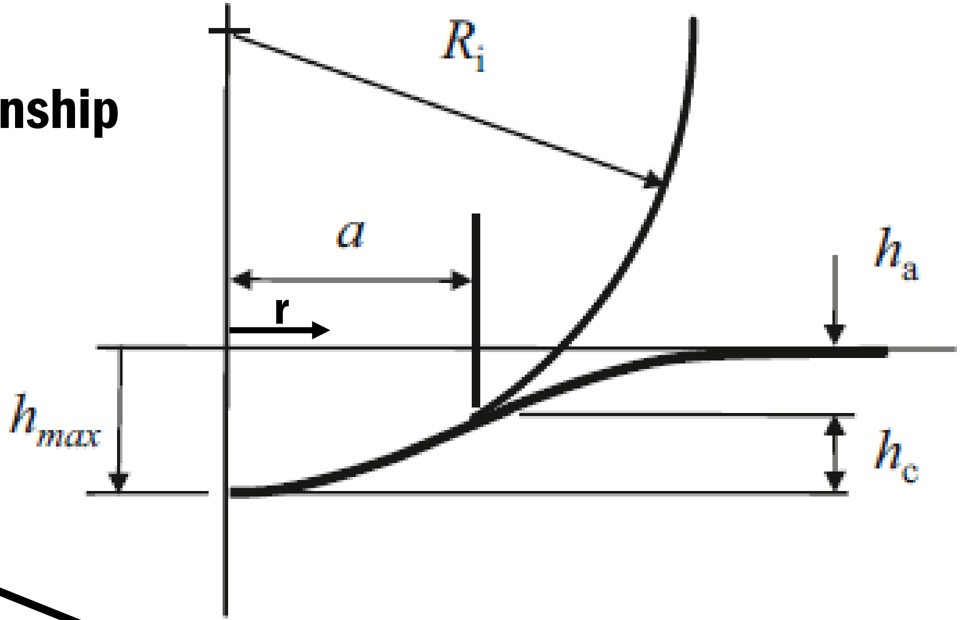
Indentation brings two bodies into contact: (1) stiff probe and (2) flat specimen

ELASTIC CONTACT:
- Spherical indenter
- Flat surface

Hertzian contact (1882): P-a relationship

$$a^3 = \frac{3}{4}\frac{PR}{E^*}$$

a: radius of contact
P: indenter load
R: indenter radius



E*: reduced modulus

$$\frac{1}{E^*} = \frac{(1-\nu^2)}{E} + \frac{(1-\nu'^2)}{E'}$$

$$p_m = \left(\frac{4E^*}{3\pi}\right)\frac{a}{R}$$

Indentation stress

Indentation strain

h: displacement of contact surface

$$h = \frac{1}{E^*}\frac{3}{2}\frac{P}{4a}\left(2 - \frac{r^2}{a^2}\right) \qquad r \le a$$

r: radial distance from the axis of symmetry

P_m: mean contact pressure

$$p_m = \frac{P}{\pi a^2}$$

πa^2: contact area

Nanoindentation – Book, A.C. Fischer-Cripps

Indenter geometry has a significant influence on the pressure distribution during indentation

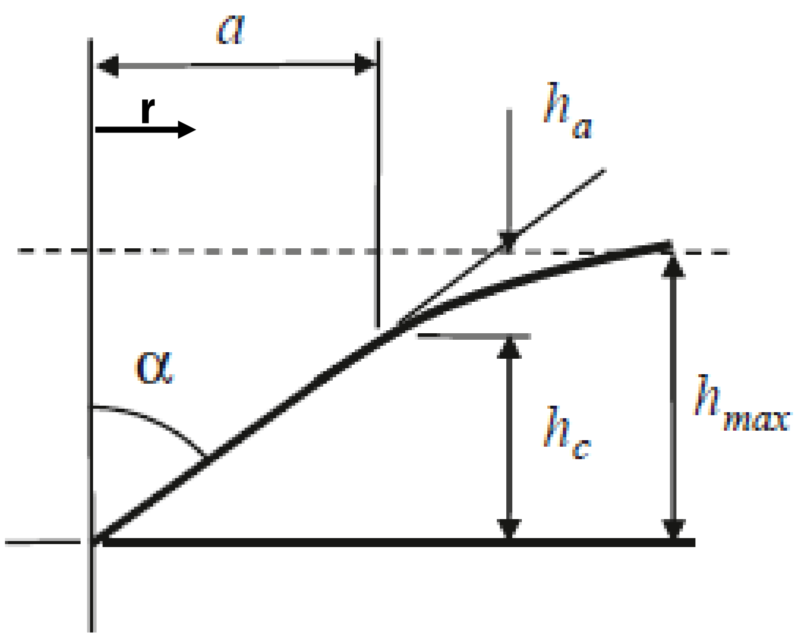
ELASTIC CONTACT:
- Conical* indenter
- Flat surface

P-a relationship

* Pyramidal indenters are treated as conical indenters

$$P = \frac{\pi}{2} a^2 E^* \cot\alpha$$

a: radius of contact (h_c*tanα)
P: indenter load
~~R: indenter radius~~
α: cone semi-angle



E*: reduced modulus

$$\frac{1}{E^*} = \frac{(1-\nu^2)}{E} + \frac{(1-\nu'^2)}{E'}$$

$$P = \frac{2}{\pi} E^* \tan\alpha \, h_{max}^2$$

h: displacement of contact surface

$$h = \left(\frac{\pi}{2} - \frac{r}{a}\right) a\cot\alpha \quad r \le a$$

r = 0

r = 0

r: radial distance from the axis of symmetry

h_c: depth of penetration at circle of contact

h_{max}: depth of penetration of the indenter apex

A: projected area

$$E^* = \frac{1}{2}\frac{dP}{dh}\frac{\sqrt{\pi}}{\sqrt{A}}$$

Elastic contact with any axisymmetric indenter

Contact stiffness

$$\frac{dP}{dh} = 2\left[\frac{2}{\pi} E^* \tan\alpha\right] h$$

Nanoindentation – Book, A.C. Fischer-Cripps

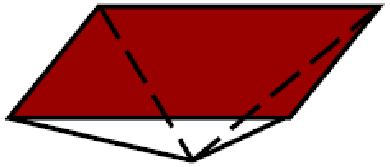
Projected area (A_p) vs. Developed area (A_d)

The difference between instrumented hardness (i.e. nanoindentation) and conventional hardness (i.e. Vickers) is the definition of the contact area between the indenter and the tested material.

Instrumented indentation

$$H_{IT} = \frac{F[N]}{A_p}$$

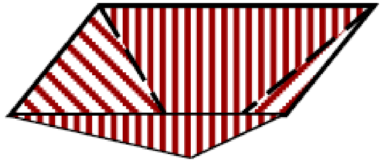
Projected area (A_p)



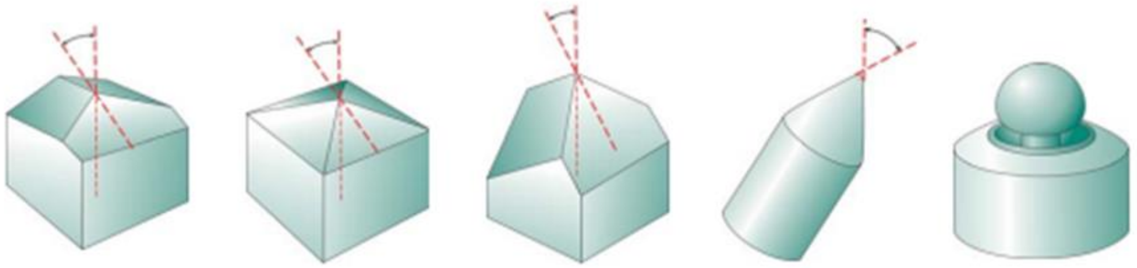
Conventional indentation

$$H_V = \frac{F[Kgf]}{A_d}$$

Developed area (A_d)



Indenting Tips Summary



	Berkovich	Vickers	Cube-Corner	Cone (angle ψ)	Sphere (radius R)
Features					
Shape	3-sided pyramid	4-sided pyramid	3-sided pyramid w/ perpendicular faces	Conical	Spherical
Parameter					
Centerline-to-face angle, α	65.3°	68°	35.2644°	—	—
Area (projected), A(d)	$24.56d^2$	$24.504d^2$	$2.5981d^2$	πa^2	πa^2
Volume-depth relation, V(d)	$8.1873d^3$	$8.1681d^3$	$0.8657d^3$	—	—
Projected area/face area, A/A_f	0.908	0.927	0.5774	—	—
Equivalent cone angle, ψ	70.32°	70.2996°	42.28°	ψ	—
Contact radius, a	—	—	—	d tan ψ	$(2Rd-d^2)^{1/2}$

How to select the correct indenter tip – Support note, Agilent Technologies

Elastic-Plastic Contact

- Indentation generally results in both elastic and plastic deformation of the specimen
- Three regions of the elastic-plastic response:

1.Full elastic deformation	2. Onset of plastic deformation	3. Continued plastic deformation

$0 \xleftrightarrow{p_m}$ (spherical) 1.1*Y, (conical) 0.5*Y $\xleftrightarrow{p_m}$ C*Y $\xleftrightarrow{p_m}$ ≈ C*Y

p_m: mean contact pressure
Y: uniaxial compressive yield stress
C: constraint factor (material and indenter geometry)

plastic deformation exists beneath the surface but is constrained by the surrounding elastic material

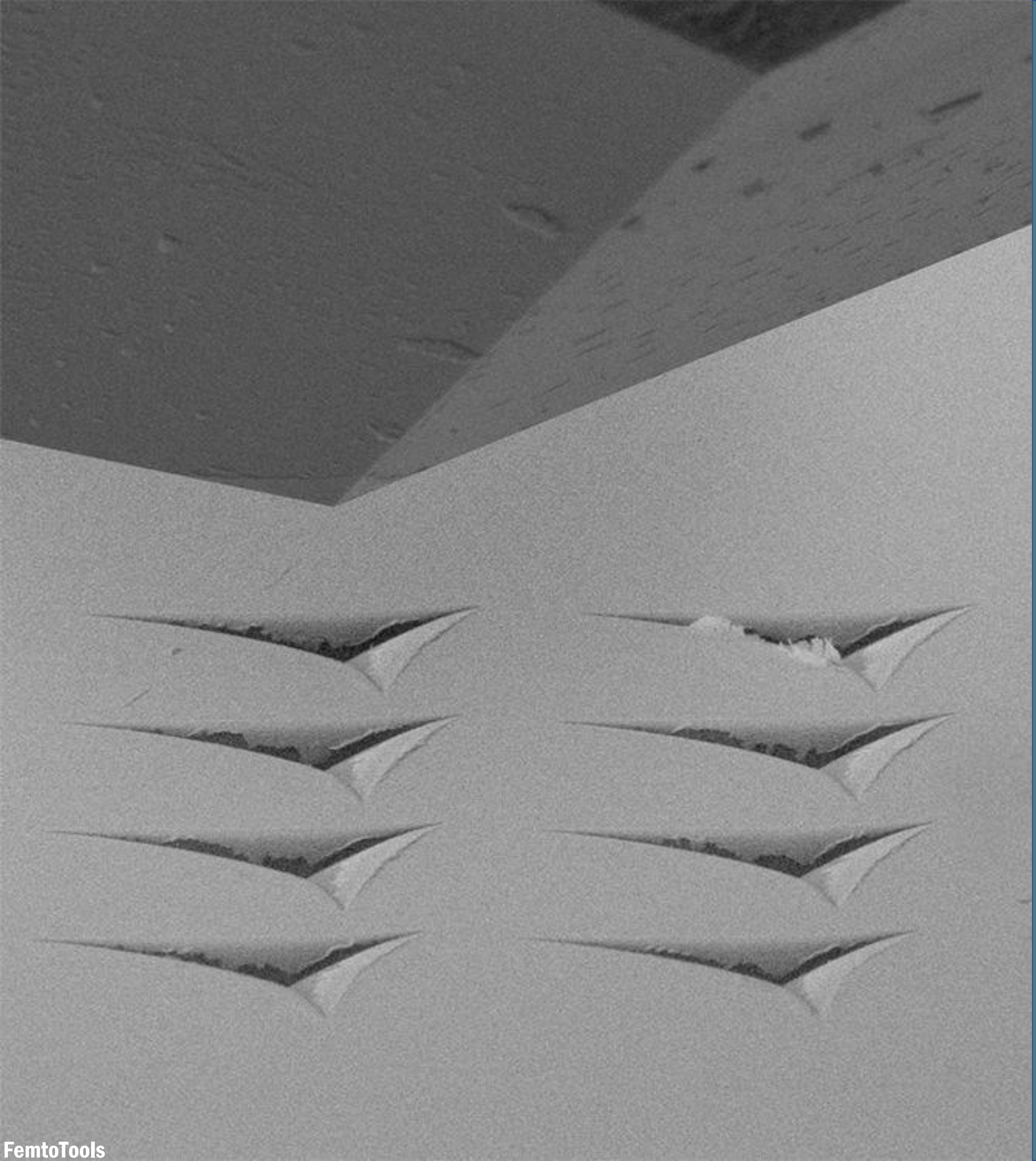
p_m remains constant* (load independent) and depends only on the materials response   *assuming no strain hardening

The constraint factor (C) and hardness (H)

There is a point at which p_m does not change despite increasing the load (p_m = C*Y).
Beyond this limiting condition: p_m = H

$$\boxed{H \approx CY} \quad \begin{cases} C \approx 3 & \text{For materials with \underline{large} E/Y ratio (metals)} \\ C \approx 1.5 & \text{For materials with \underline{low} E/Y ratio (glasses)} \end{cases}$$

Nanoindentation – Book, A.C. Fischer-Cripps



OUTLINE

- Why nanoindentation?
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- **Instrumented Indentation**
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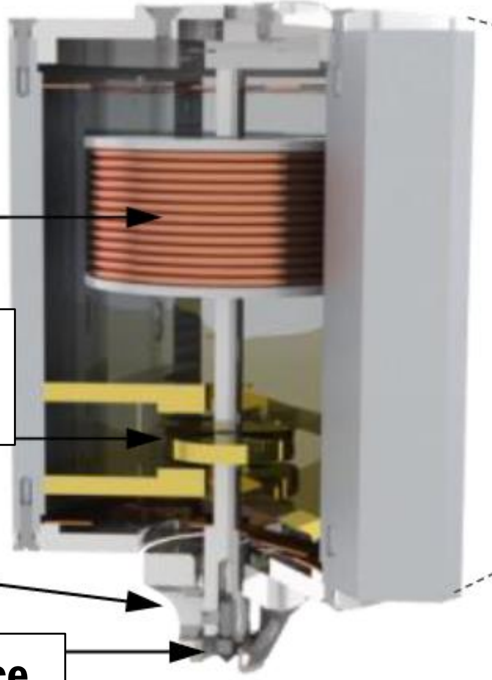
How is load-displacement measured?

Measuring head

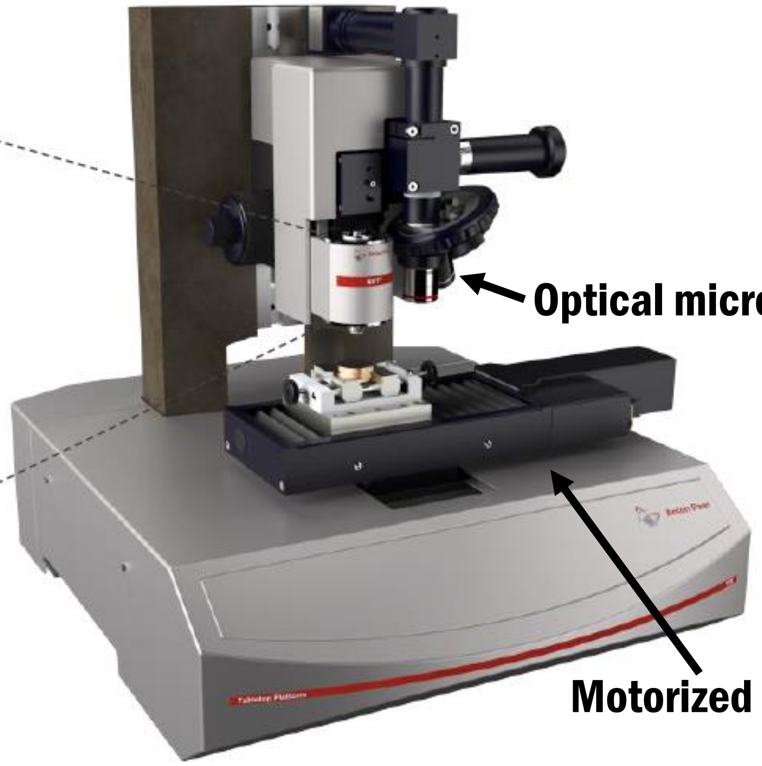


1. Electromagnet applies load on the indenter shaft

2. Displacement (e.g. capacitive) sensor measures the indenter displacement

3. Indenter tip moves differentially to the reference ring

4. Reference ring sits on the sample surface, compensating for frame deformation and minimizing thermal drift



Benefits of such a top-referencing technique are:

1. Fast indenter approach time (because the relative position of the indenter tip in relation to the surface is always known)
2. Protection of the indenter from air currents due to the indenter being encapsulated by the reference ring.

Nanoindentation test chronology

1. Approach: The indenter approaches the test surface until contact is sensed.

2. Loading: The indenter is pressed into contact with the test material until the set load (or displacement) is reached, at a specified loading rate

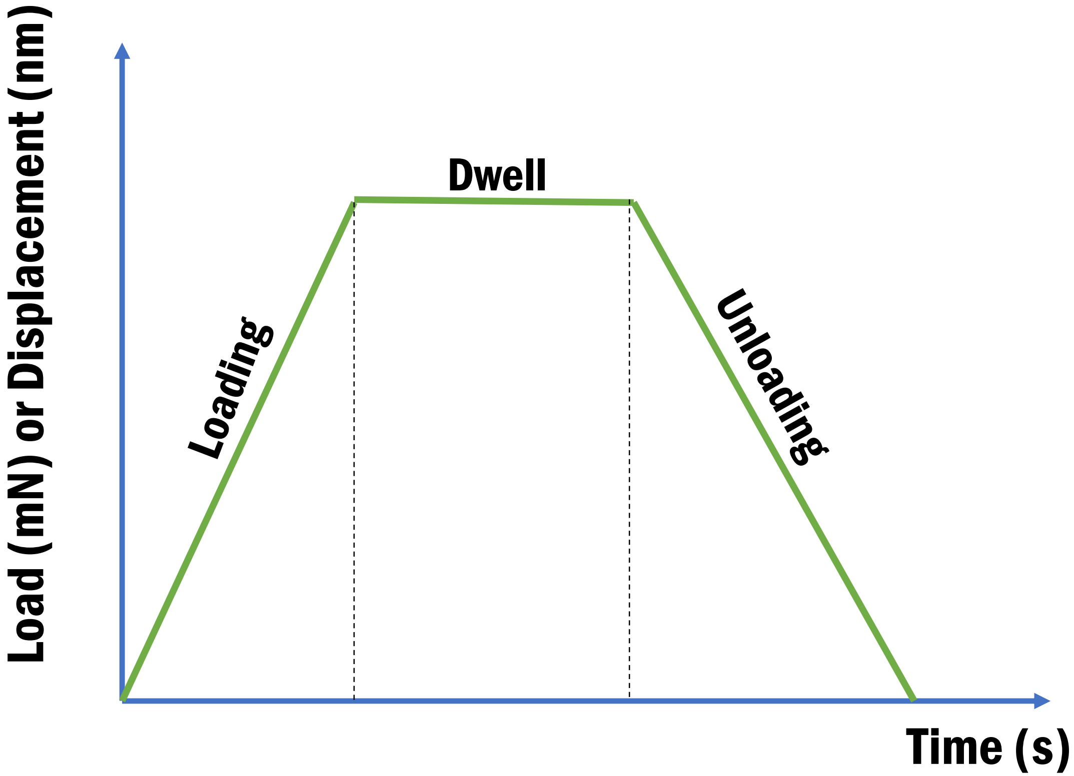
3. Dwell: The force on the indenter is held constant for a dwell time at the peak force.

4. Unloading: The indenter is withdrawn from the sample at a given rate (typically similar to loading rate)

   *Thermal-drift (expansion and contraction of the equipment + test material) is often evaluated here.

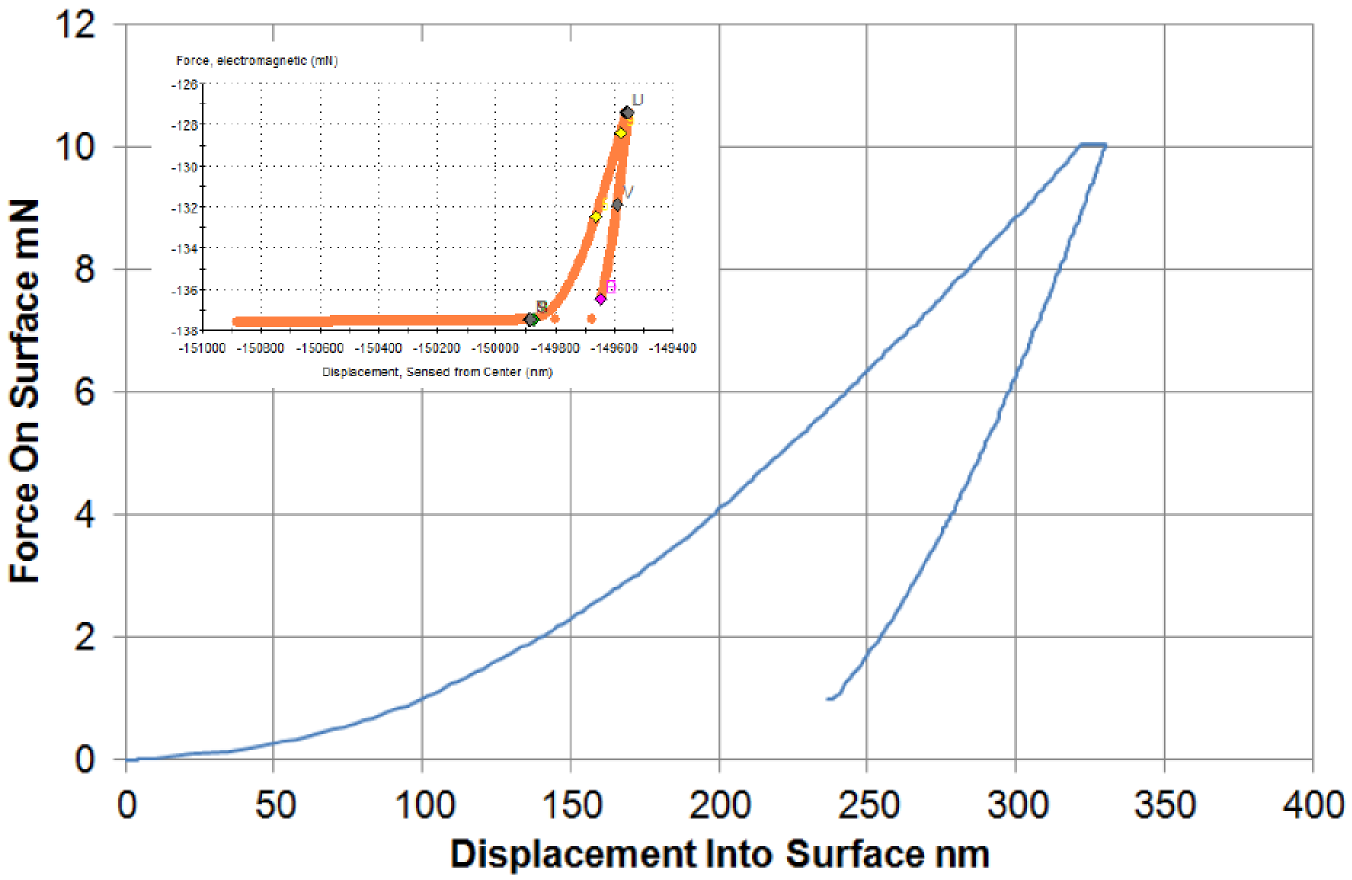
5. Final unloading: The indenter is withdrawn from the sample

Typical trapezoidal loading profile



From raw measurements to analysis of the load-displacement curve

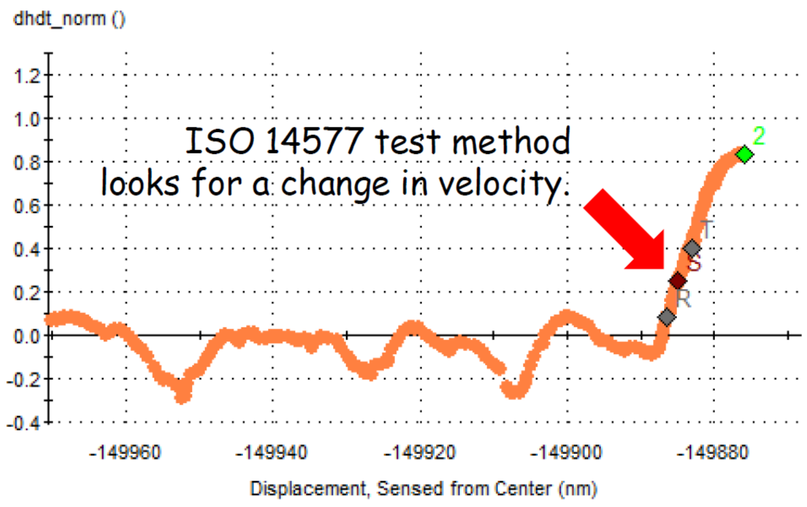
1. Determine the point of contact (not necessarily the same point at which approach terminated and loading began).
2. Reference all measurements to values at contact.
3. Correct displacement for frame compliance.
4. Correct displacement for thermal drift.
5. Correct force for influence of supporting mechanism



From raw measurements to analysis of the load-displacement curve

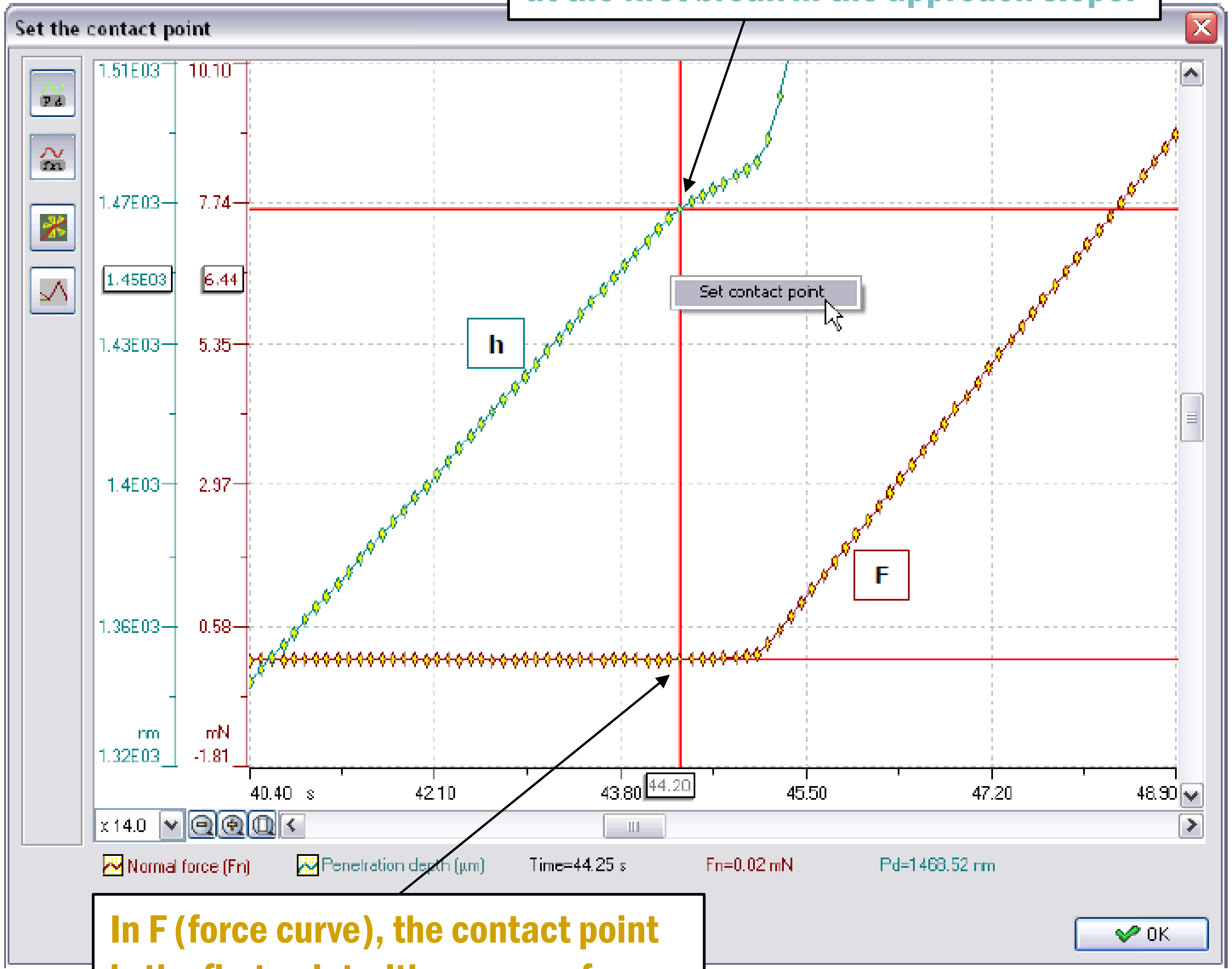
1. **Determine the point of contact (not necessarily the same point at which approach terminated and loading began).**

Different test methods use different algorithms



Sometimes, it is difficult to see the change in the F curve, therefore it is <u>better to use the curve h to determine the contact point</u>.

In h (depth curve), the contact point is at the first break in the approach slope.



In F (force curve), the contact point is the first point with a no zero force.

Anton Paar: Indentation software manual

From raw measurements to analysis of the load-displacement curve

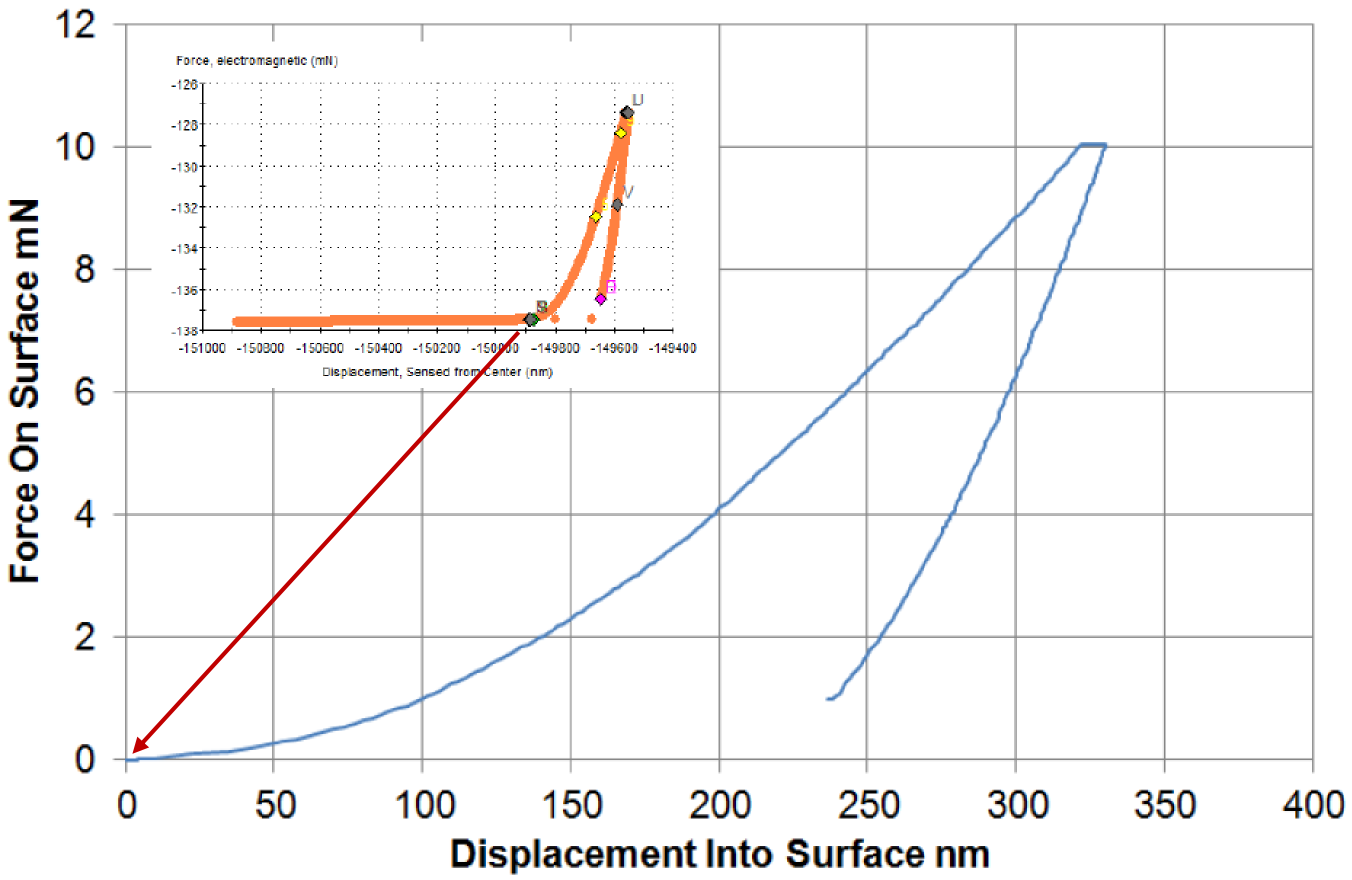
1. Determine the point of contact (not necessarily the same point at which approach terminated and loading began).

2. **Reference all measurements to values at contact.**

Taring channels

Displacement: $h = z - z_s$

Force: $P = F - F_s$



From raw measurements to analysis of the load-displacement curve

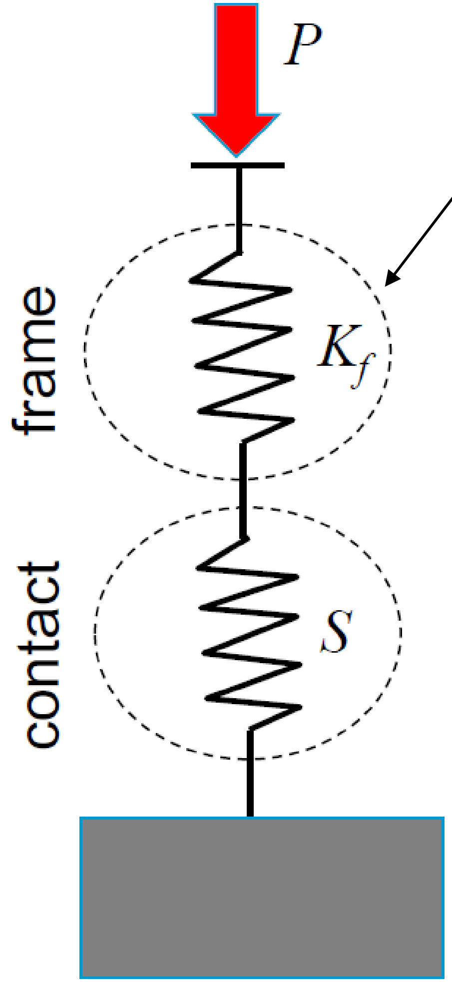
1. Determine the point of contact (not necessarily the same point at which approach terminated and loading began).

2. Reference all measurements to values at contact.

3. **Correct displacement for frame compliance**

- The displacement measured by the sensor includes deformation of the sample AND deflection of the equipment.
- Thus, we must subtract the displacement which occurs in the equipment (frame compliance), calculated as P/K_f
- The value for K_f is determined by a calibration procedure in which a material of homogeneous (and known) properties is indented at multiple loads

K_f: frame stiffness
P/K_f: frame compliance



Compensating for Frame Stiffness

Displacement: $h = z - z_s - (F - F_s)/K_f$

Force: $P = F - F_s$

From raw measurements to analysis of the load-displacement curve

1. Determine the point of contact (not necessarily the same point at which approach terminated and loading began).

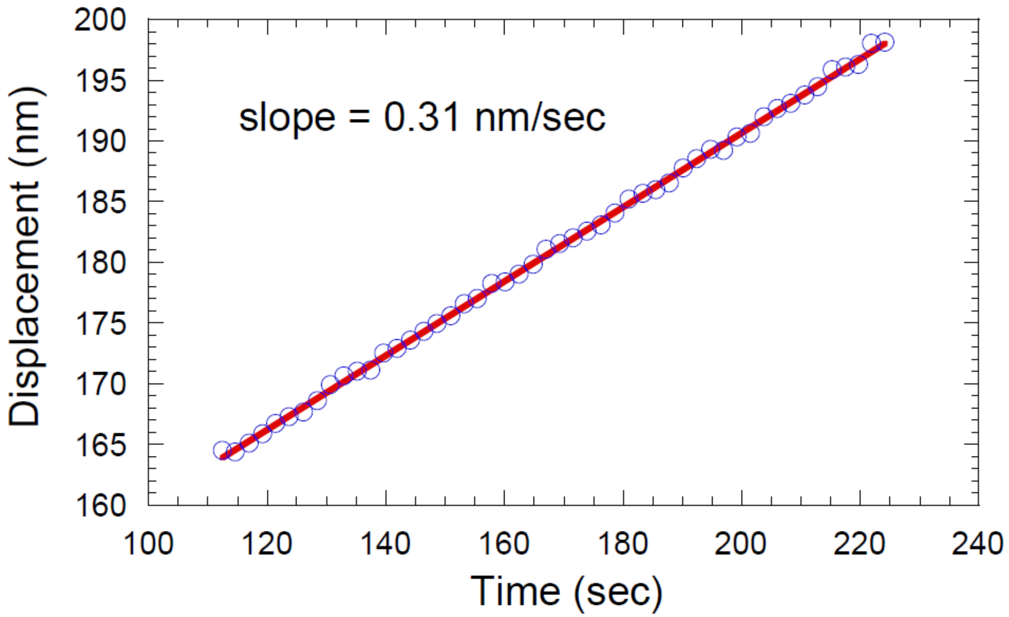
2. Reference all measurements to values at contact.

3. Correct displacement for frame compliance.

4. **Correct displacement for thermal drift.**

- **Just before final unloading, the force on the indenter is held constant at a relatively low value for a dwell time.**
- **Measured displacements are attributed to thermal drift**
- **Calculating the thermal drift rate, displacements can be compensated relative to the time at which they were acquired**

Determining thermal drift rate



Compensating for Thermal Drift

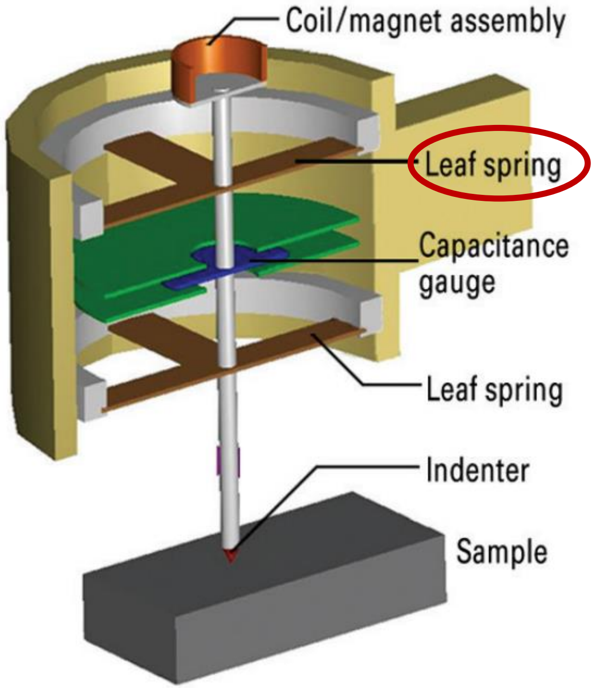
Displacement: $h = z - z_s - (F - F_s)/K_f - \boldsymbol{DR\,(t - t_s)}$

Force: $P = F - F_s$

From raw measurements to analysis of the load-displacement curve

1. Determine the point of contact (not necessarily the same point at which approach terminated and loading began).
2. Reference all measurements to values at contact.
3. Correct displacement for frame compliance.
4. Correct displacement for thermal drift.
5. **Correct force for influence of supporting mechanism**
   - **The force applied includes that from the coil/magnet assembly AND supporting mechanism.**

Load application	Coil/magnet assembly
Displacement measurement	Capacitance gauge
Typical leaf spring stiffness	~100 N/m



K_f: leaf spring stiffness

Figure 1. Schematic diagram of the actuating and sensing mechanisms of the Nano Indenter G200.

Compensating for Support-Spring Deflection

Displacement: $h = z - z_s - (F - F_s)/K_f - DR\,(t - t_s)$

Force: $P = F - F_s - \boldsymbol{K_s}(\boldsymbol{z} - \boldsymbol{z_s})$

Keysight Technologies Nano Indenter G200 – official brochure
Agilent seminar (Instrumented indentation)– Jennifer Hay

100 years of nanoindentation…from From Hertz to Oliver & Pharr

Hertz (1882) → **Sneddon (1965)** → **Oliver & Pharr (1992)**

Hertz (1882)

- Classical elastic contact problem
- Rigid spherical indenter
- Indentation depth significantly smaller than tip radius

$$p_m = \left(\frac{4E^*}{3\pi}\right)\frac{a}{R}$$

p_m: mean contact pressure
a: radius of contact
R: indenter radius
E*: reduced modulus

Sneddon (1965)

- General relationship between load and displacement for any axisymmetric indenter

$$P = \alpha h^m$$

P: indenter load
h: elastic indenter displacement
α : material constant
m: indenter geometry constant

Oliver & Pharr (1992)

- E and H calculation from contact stiffness (S) measured during unloading and projected area function (A)
- Valid for any indenter geometry defined as a solid of revolution

$$E_r = \frac{\sqrt{\pi}}{2\beta}\frac{S}{\sqrt{A}}$$

S: unloading stiffness
A: projected contact area
β : correction factor

An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments - Oliver & Pharr (1992)

Oliver & Pharr (1992): MOST CITED article in the entire field of Material Science

An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments

W. C. Oliver
Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee 37831-6116

G. M. Pharr
Department of Materials Science, Rice University, P. O. Box 1982, Houston, Texas 77251

(Received 23 December 1991; accepted 22 January 1992)

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An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments
WC Oliver, GM Pharr - Journal of materials research, 1992 - cambridge.org
... where Pmax is the peak **indentation load** and A is the projected area of the **hardness** ... During the past several years, we have conducted **load** and **displacement sensing indentation tests** ...
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Measurement of hardness and elastic modulus by instrumented indentation: Advances in understanding and refinements to methodology

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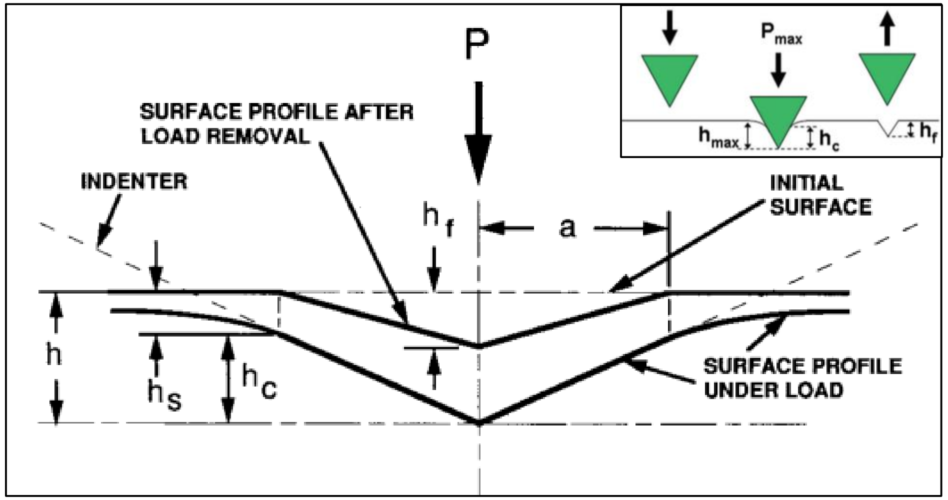
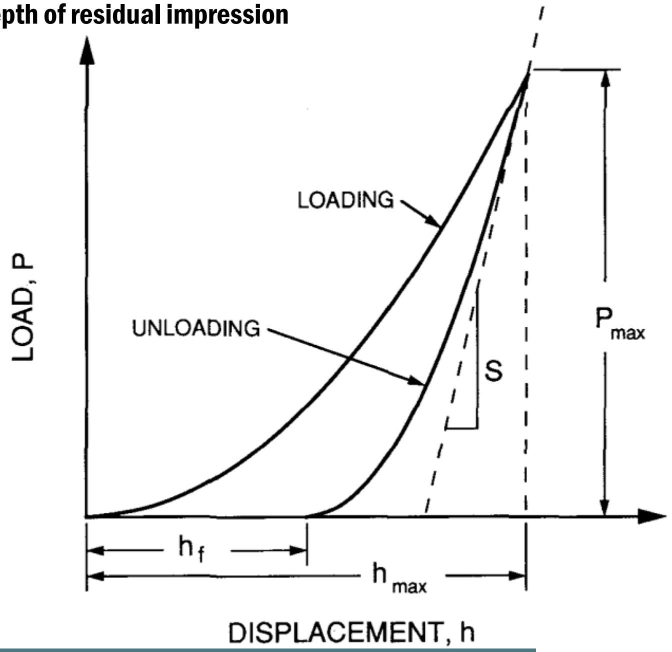
(Received 15 June 2003; accepted 23 September 2003)

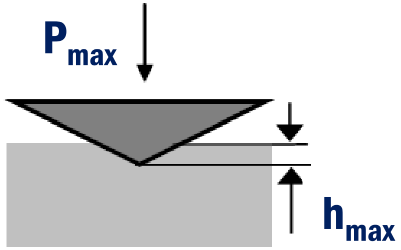
From load-displacement to Young's Modulus and Hardness (Oliver & Pharr)



h: total displacement
h_c: contact depth
h_s: surface displacement at radius of contact
h_f: final depth of residual impression



1. Measure P_{max} and h_{max}



2. Calculate unloading stiffness (S)

$$S = \frac{dP}{dh} = \frac{2}{\sqrt{\pi}} E_r \sqrt{A}$$

3. Calculate contact depth (h_c)

$$h_c = h_{\max} - h_s$$

$$h_s = \epsilon \frac{P_{\max}}{S}$$

ϵ: indenter geometry constant
(ϵ= 0.75 for Berkovich)

4. Calculate projected contact area A(h_c)

$$A(h_c) = 24.5 h_c^2 + C_1 h_c^1 + C_2 h_c^{1/2} + C_3 h_c^{1/4}$$

Hardness (H)

$$H = \frac{P_{\max}}{A}$$

Reduced Modulus (E_r)

$$E_r = \frac{\sqrt{\pi}}{2\beta} \frac{S}{\sqrt{A}}$$

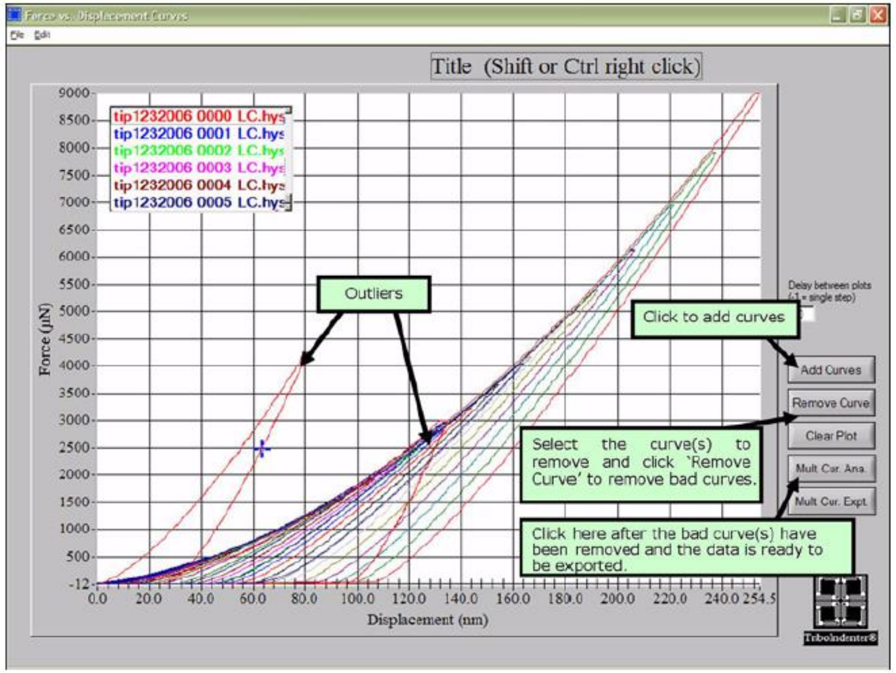
β: correction factor for non-axisymmetric indenter and large strains (β =1.0226 for Berkovich)

Young Modulus (E)

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

Tip calibration to determine Area function

- An array of indents (minimum 25) at various contact depths (decreasing loads) are performed in a reference sample (typically fused quartz)

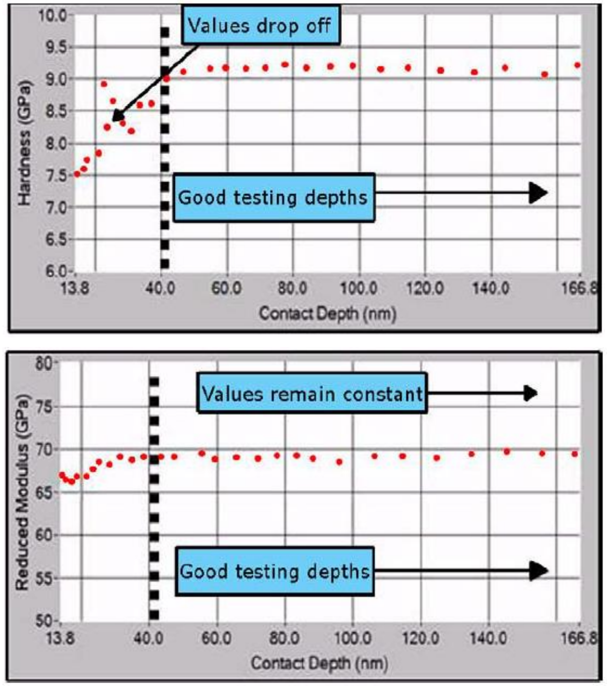


- The calculated area (A) is plotted as a function of the contact depth (hc), and the polynomial fit determines the C coefficients (C_1, C_2, ..., C_5)

$$E_r = \frac{S\sqrt{\pi}}{2\sqrt{A}} \longrightarrow A = \frac{\pi}{4}\left[\frac{S}{E_r}\right]^2$$

Berkovich (C_0=24.5)
Cube corner (C_0=2.598)
Conical (C_0=-π)

$$A = C_0 h_c^2 + C_1 h_c + C_2 h_c^{1/2} + C_3 h_c^{1/4} + C_4 h_c^{1/8} + C_5 h_c^{1/16}$$



H values become sporadic when contact depth < one-third the radius of curvature of the probe

Er stays constant because that value was defined as 69.6 GPa when the area function was fit

TI 950 Triboindenter User Manual - Hysitron

Machine compliance (1/Stiffness)

Displacement associated with the measuring instrument (to be subtracted from the measured displacement)

Factors that determine the machine compliance:
- Transducer (lateral vs. normal force)
- Indenter probe (epoxy, shank length)
- Sample mounting (no soft material)
- Transducer mounting (leveled + fixed)

C_c: Contact compliance

C_m: Machine compliance

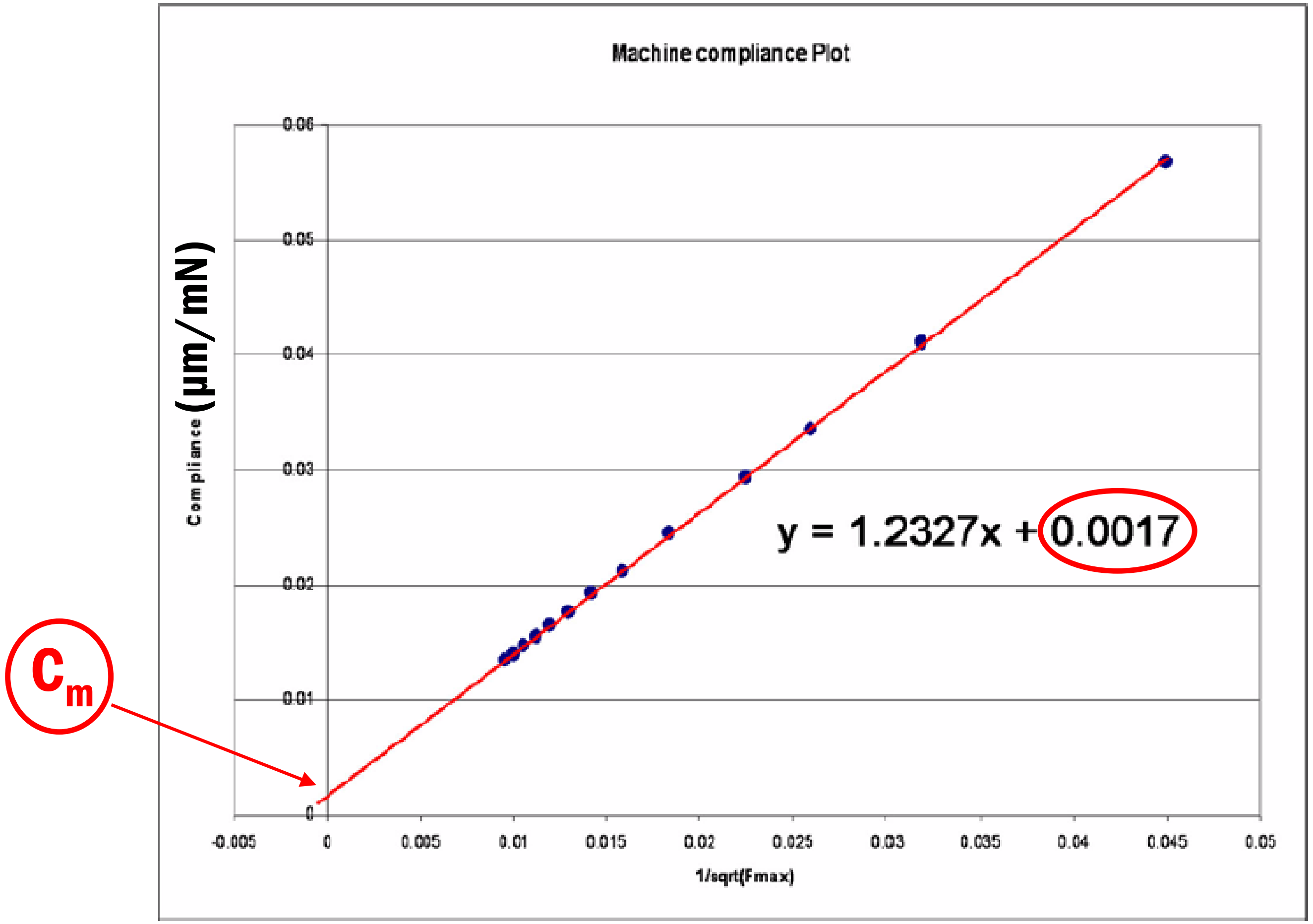
$$\frac{1}{E_r} = \frac{(1-v_i^2)}{E_i} + \frac{(1-v_s^2)}{E_s}$$

$$C_c = \frac{dh}{dP} = \frac{\sqrt{\pi}}{2} \times \frac{1}{\sqrt{A}} \times \frac{1}{E_r}$$

$$C_{total} = C_m + C_c = C_m + \frac{\sqrt{\pi}}{2} \times \frac{1}{E_r} \times \frac{1}{\sqrt{A}}$$

$$H = \frac{P_{max}}{A}$$

$$\boxed{C_{total} = C_m + \frac{\sqrt{\pi}}{2} \times \frac{\sqrt{H}}{E_r} \times \frac{1}{\sqrt{P_{max}}}}$$



C_m = 0.0017*1000 = 1.7(nm/mN)

Into system calibration

TI 950 Triboindenter User Manual - Hysitron

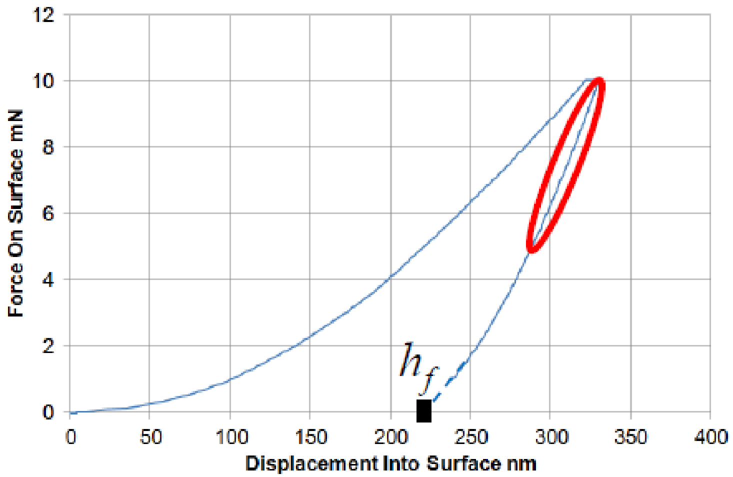
"When all you have is a hammer, everything looks like a nail" – Abraham Maslow

We interpreted the load-displacement curves obtained with Berkovich indenters with elastic contact models derived for axisymmetric conical indenters.

But…WHY??

- Originally, this is what the Oliver & Pharr did.

Elastic models apply to the unloading curve



Later, 3D finite-element simulations comparing Berkovich and conical elasto-plastic indentation have justified this tactic. Although the stress fields are different, the load-displacement curves are the same.

$$E_r = \frac{\sqrt{\pi}}{2\beta}\frac{S}{\sqrt{A}}$$

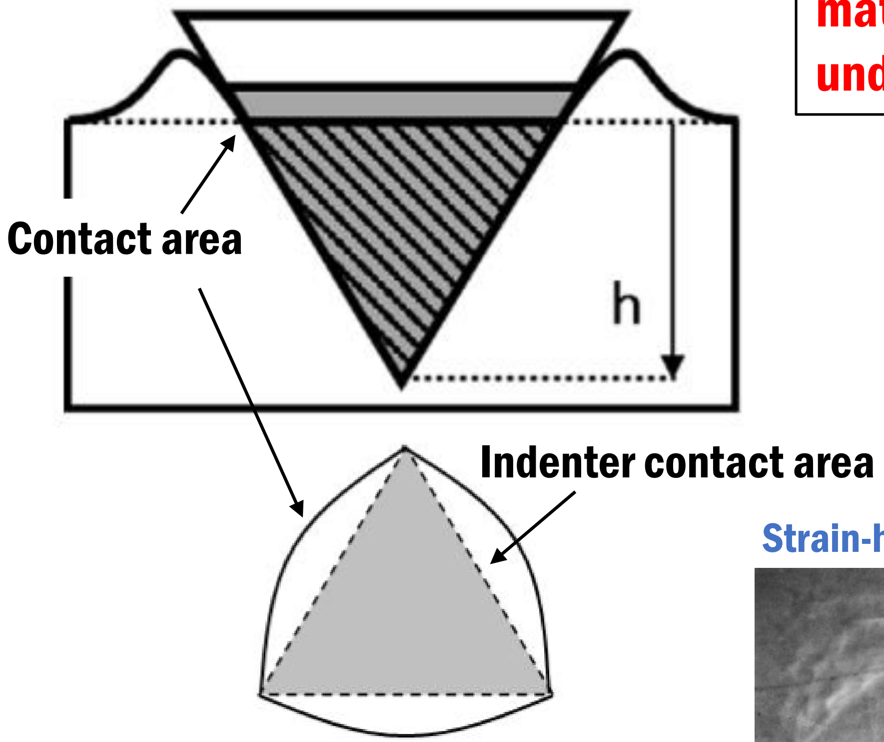
β = 1; for the case of small deformation of an elastic material by a rigid axisymmetric indenter of smooth profile

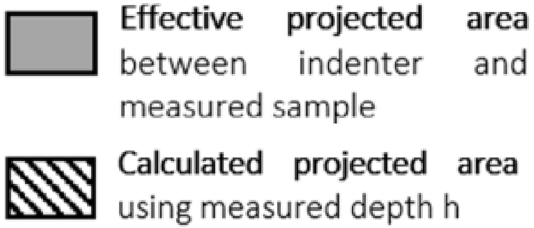
β =1.0226; for Berkovich

Agilent seminar (Instrumented indentation)– Jennifer Hay
Measurement of hardness and elastic modulus by instrumented indentation: Advances in understanding and refinements to methodology – Oliver & Pharr (2003)

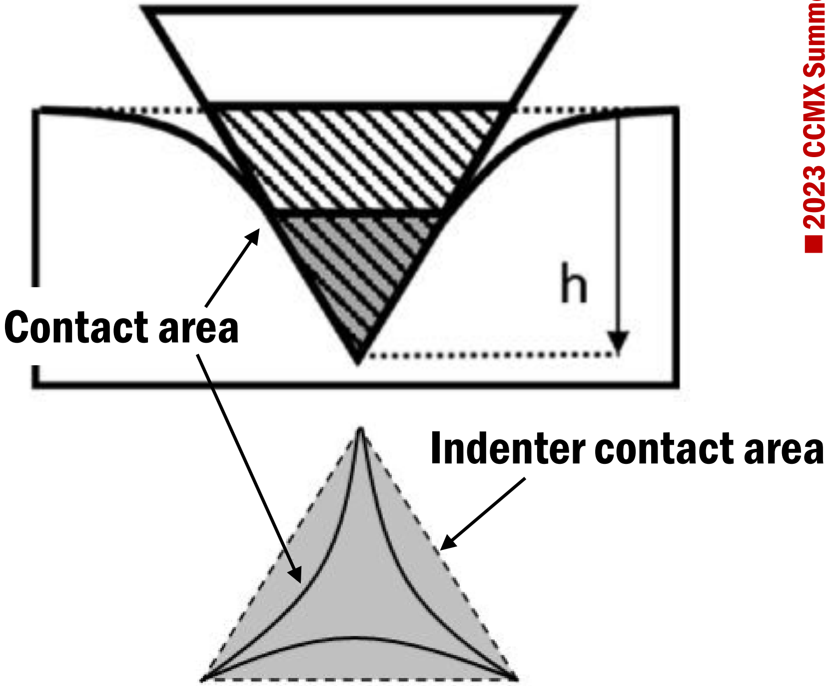
The strain-hardening properties of a material can affect its behavior underneath the indenter tip

Pile-up

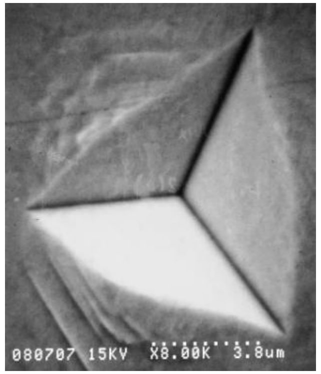




Sink-in



Strain-hardened Cu



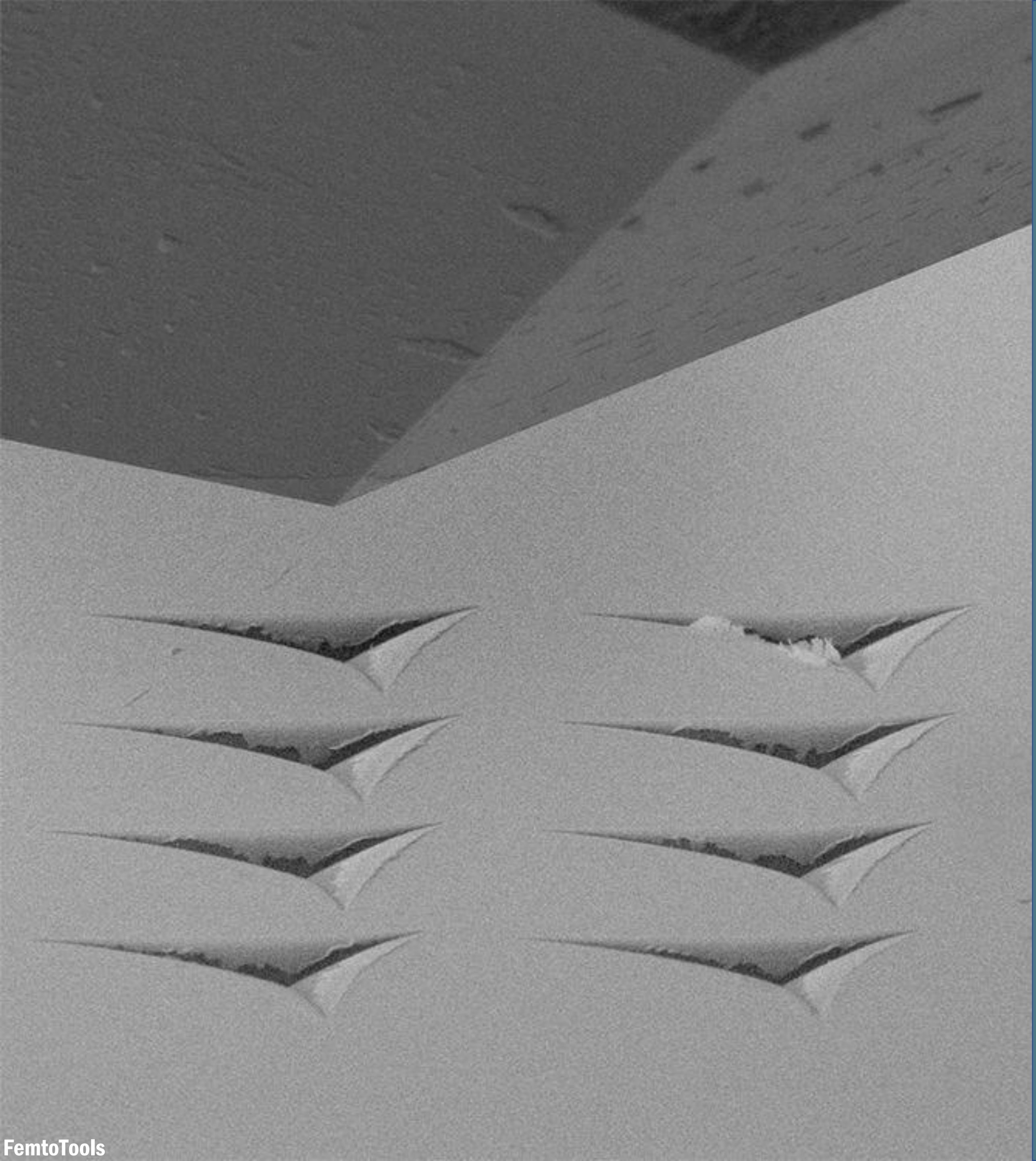
Annealed Cu

- Displaced volume is pushed out to the sides of the indenter
- Projected contact area larger than the cross-sectional area of the indenter
- Strain-hardened materials (low strain-hardening rate) deform very locally

A parameter considering the ratio of the indentation contact to triangular area is introduced to account for such effects in the calculated projected area

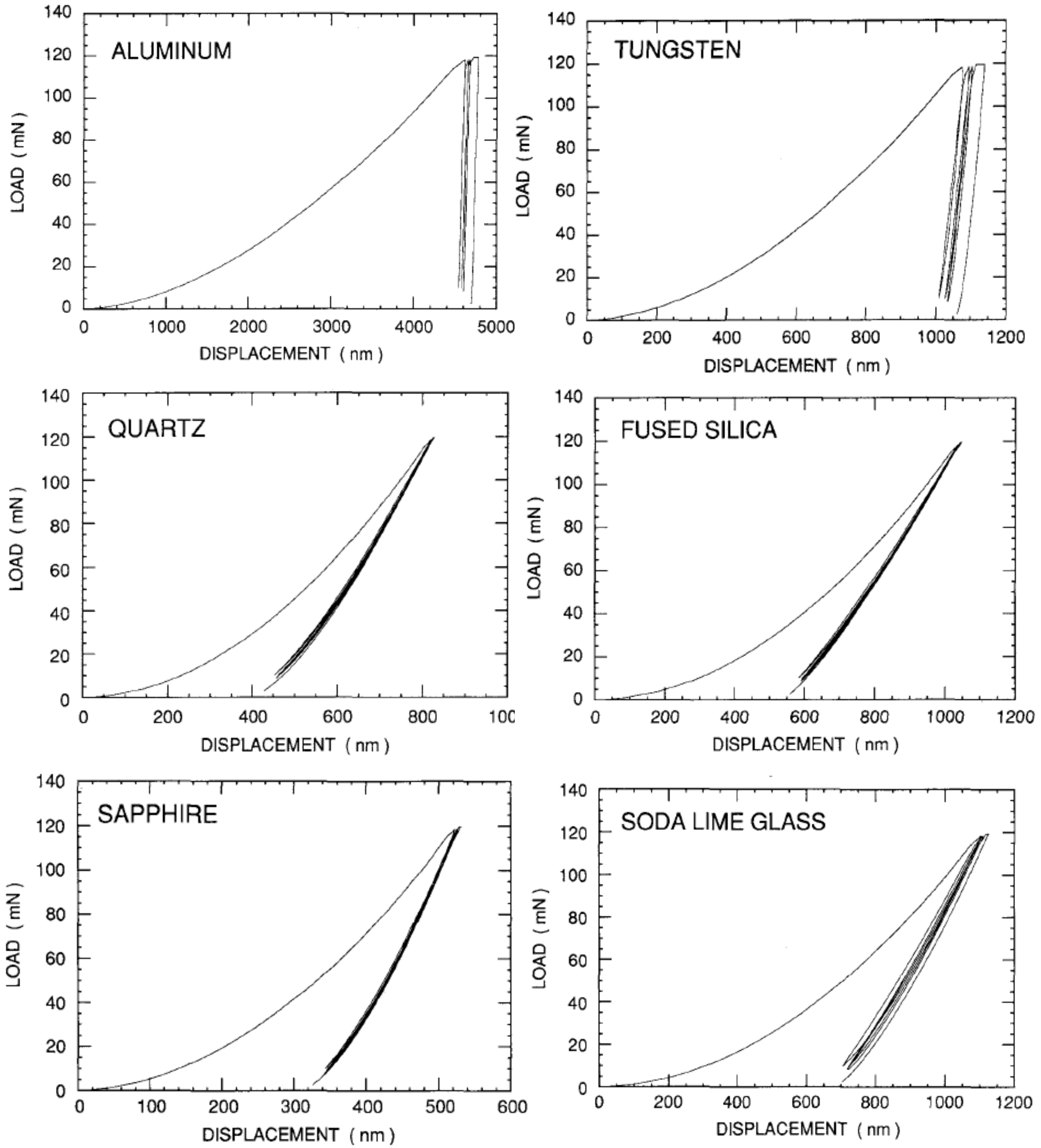
- Displaced volume is accommodated mainly by far-field elastic displacements
- Projected contact area smaller than the cross-sectional area of the indenter
- Annealed materials (high strain-hardening rate), exhibit plastic deformation further away from the contact

Determination of indenter tip geometry and indentation contact area for depth-sensing indentation experiments -K. W. McElhaney et al. (1997)



OUTLINE

- Why nanoindentation?
- Contact mechanics
- Instrumented Indentation
- Case studies



An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments

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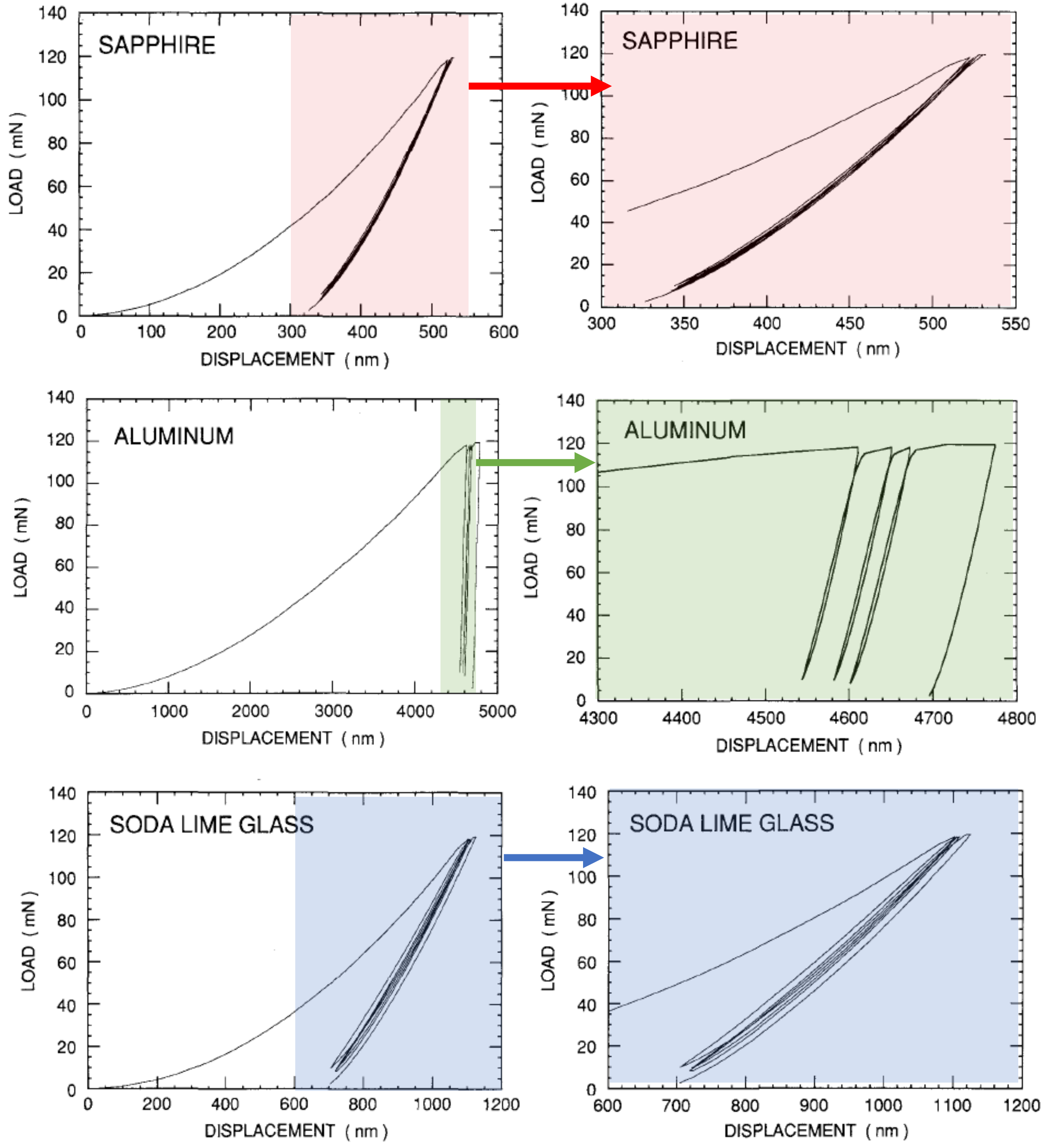
6 materials studied
Indentation to peak loads of 120 mN

Apparent differences in hardness, from the large differences in depth attained (softest: Al, hardest: sapphire)

- **<u>Metals (Al, W):</u> relatively small H compared to E, since most of the deformation is accommodated plastically, and only a small portion recovered on unloading**

- **<u>Ceramics</u>: varying degrees of elastic recovery during unloading**



Load-disp behavior upon unloading/reloading :

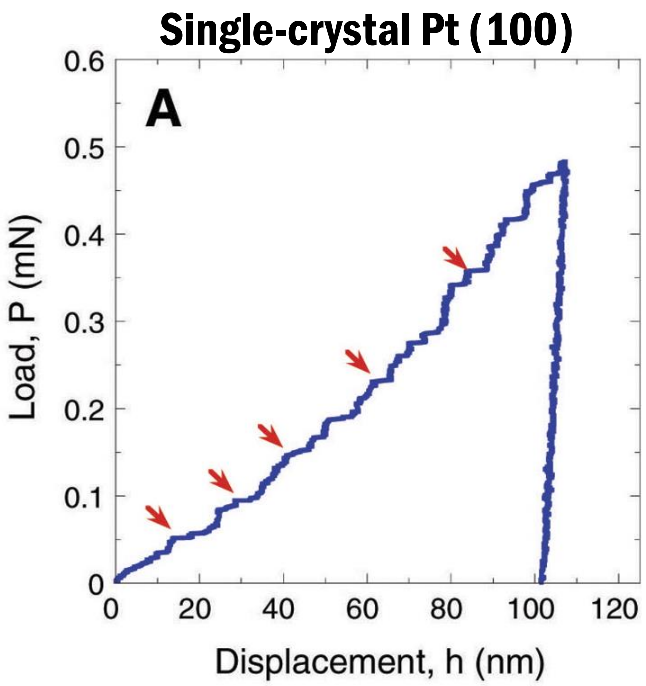
- **<u>Saphire, Quartz, fused silica</u>: near perfect reversibility suggests that deformation after the initial loading is almost entirely elastic.**

- **<u>Al, W</u>: peak load displacements shift to higher values in consecutive cycles: continued plastic deformation. Creep during hold period at peak load before final unloading.**

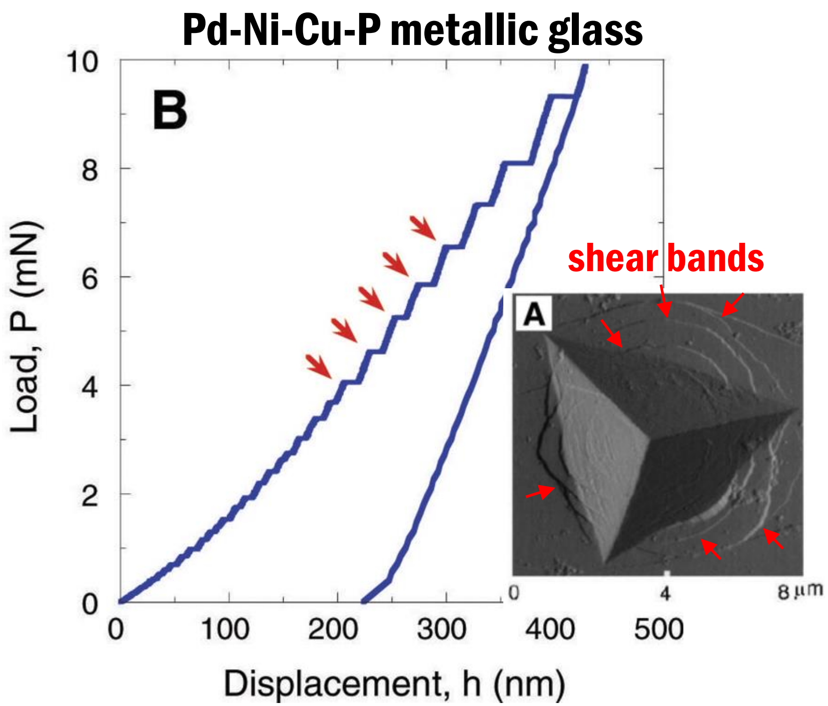
- **<u>Soda lime glass</u>: hysteresis loops (small amount of reverse plasticity upon unloading). Looping degenerates with cycling, largely elastic behavior after 3-4 cycles.**

Displacements recovered during first unloading may not be entirely elastic → use of first unloading curve may be inaccurate (introduce hold period at peak load to minimize time dependent plastic effects)

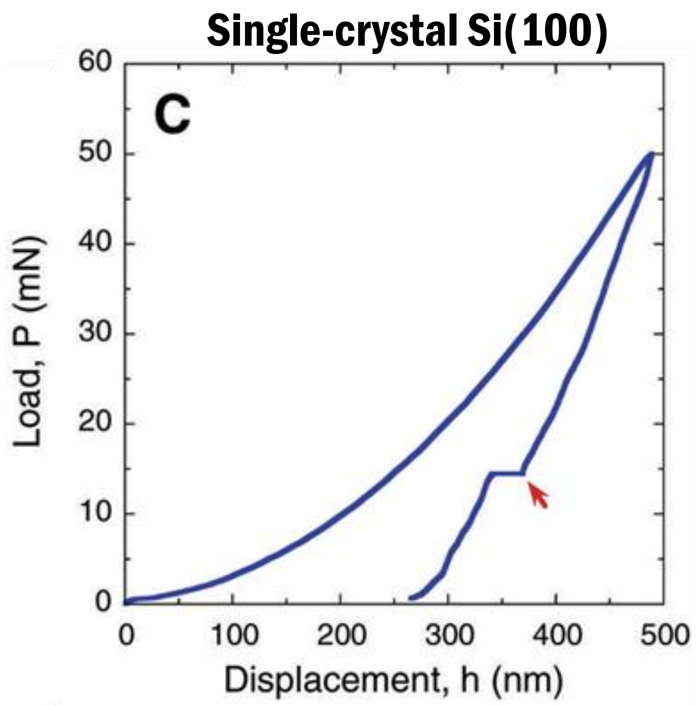
Operation of discrete physical phenomena beneath the indenter tip



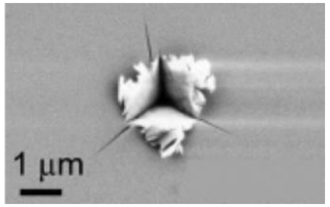
- **<u>Dislocation nucleation</u> is detected during a shallow indentation**
- **<u>Incipient plasticity</u>: pop-in event at 10-20nm marks the onset of plastic deformation**
- **Additional pop-in are due to <u>dislocation motion</u>, multiplication and interaction**



- **<u>Shear localization</u> into 'shear bands' is measured in a amorphous alloy**
- **Unstable plastic deformation (no dislocations), occurring in bursts of <u>highly localized strain</u>**
- **Number and size of pop-in events decreases with faster indentation rates**



- **A <u>phase transformation</u> with a significant volume increase is detected during unloading**
- **Mean contact pressure (≈ GPa) can trigger structural transformations (i.e. Si, Ge)**



Case studies

Nanoindentation studies of materials – C. Schuh (2006)

Main takeaways

- Over 100 years of evolution from Hertz (1882) to Oliver-Pharr (1992) have made nanoindentation a robust and versatile technique to characterize micron scaled volumes of material
- During nanoindentation, we obtain a load-displacement curve, from which we can derive the E and H of the material by analyzing the unloading elastic response
- Calibration of the indenter tip is essential to determine a correct contact area function
- Correction of the machine compliance and thermal drift are fundamental steps for interpreting the raw load-displacement curve and retrieve E and H accurately
- The Oliver-Pharr model provides a means for determining contact area, H, and E without imaging for any indenter shape
- Pile-up and sink-in events require extra careful determination of contact area for correct E and H
- Physical phenomena like incipient plasticity, phase transformations and shear banding can be identified during nanoindentation

Thank you