Session 6: Basic Instrumented Indentation to Measure Hardness and Young's Modulus

Jennifer Hay
Factory Application Engineer
Nano-Scale Sciences Division
Agilent Technologies

jenny.hay@agilent.com

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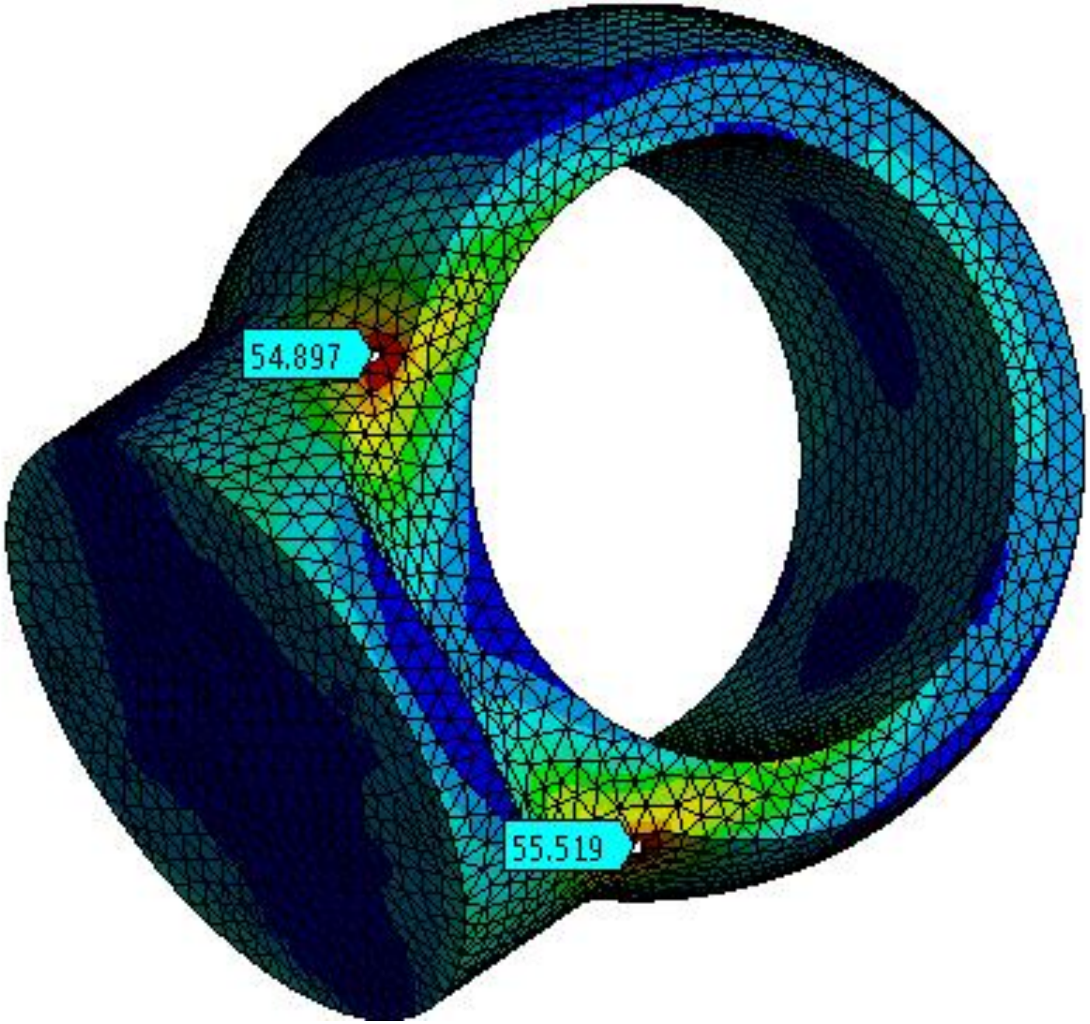
Young’s modulus (E) and yield stress (σ)



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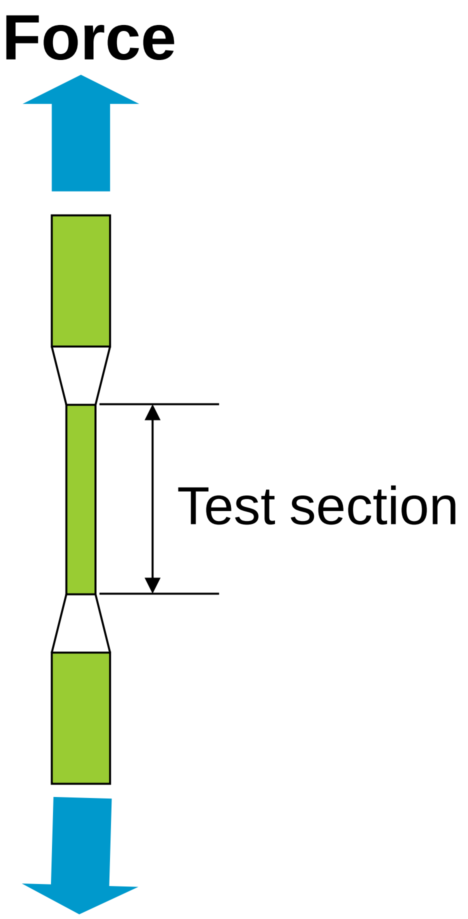
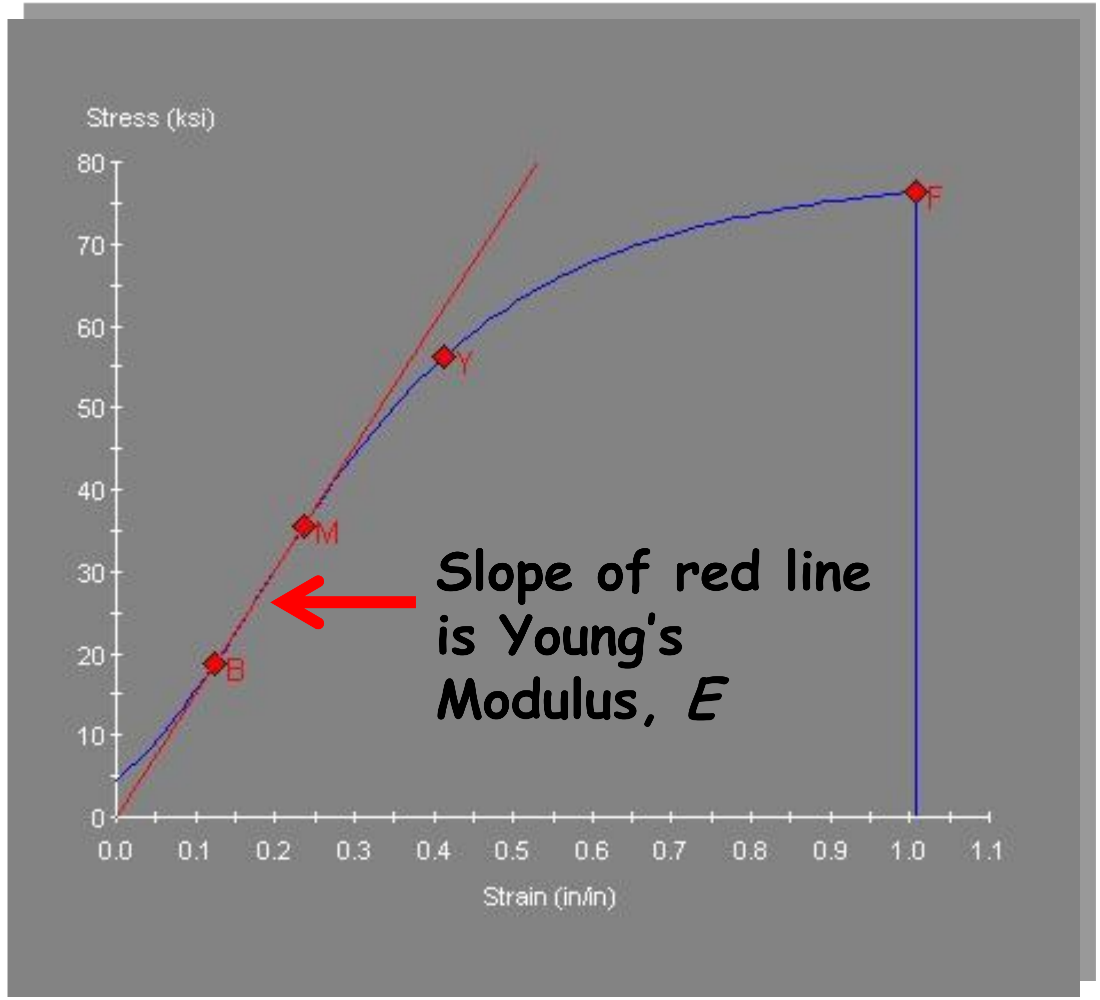
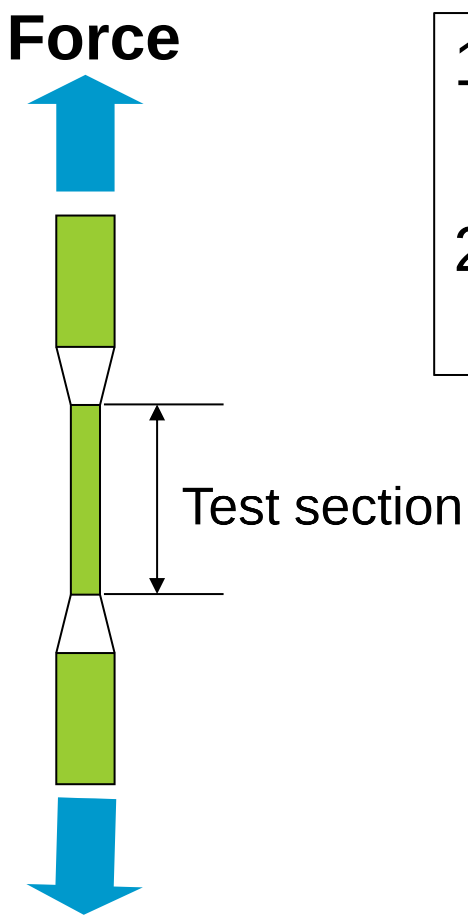
Young's modulus and yield stress

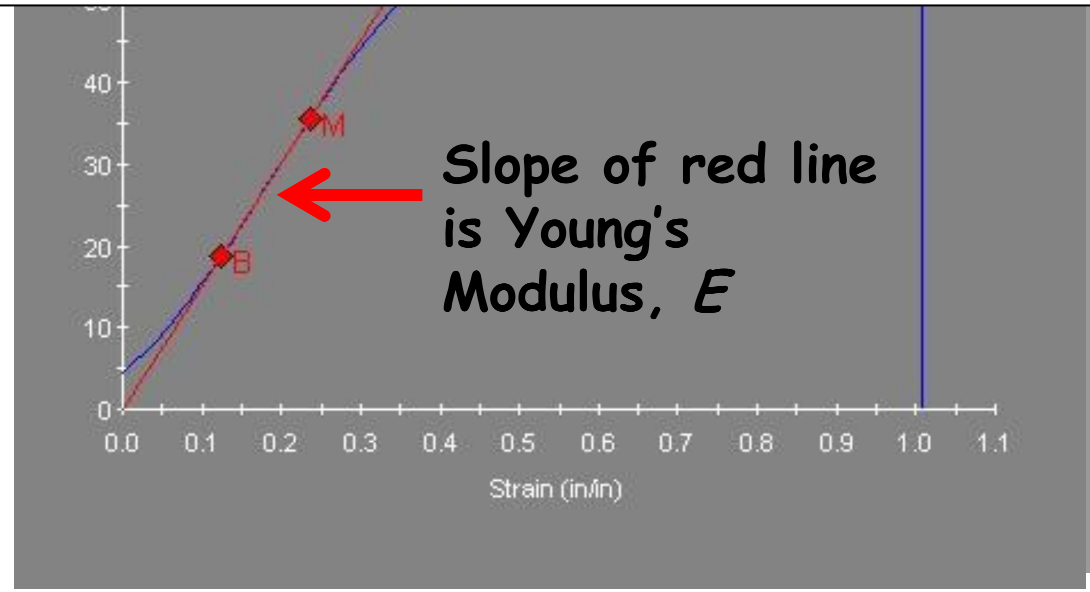




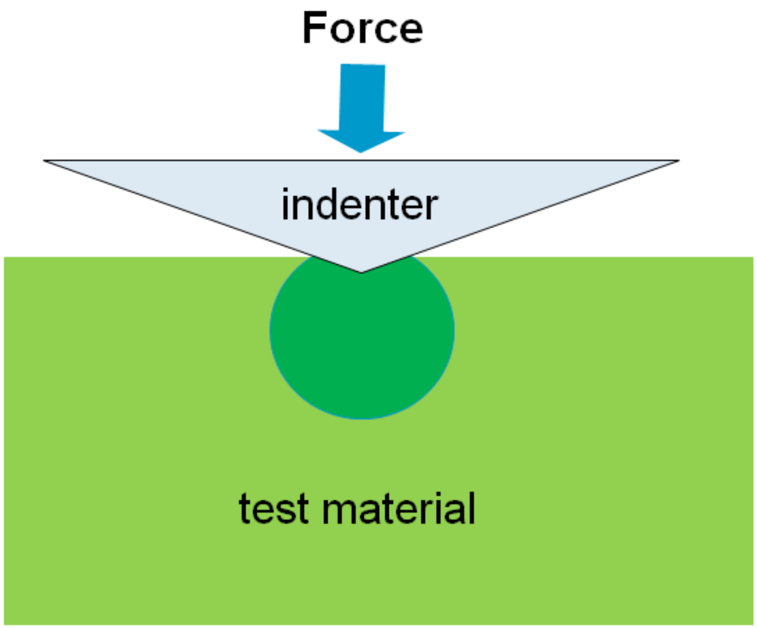
Young's modulus and yield stress

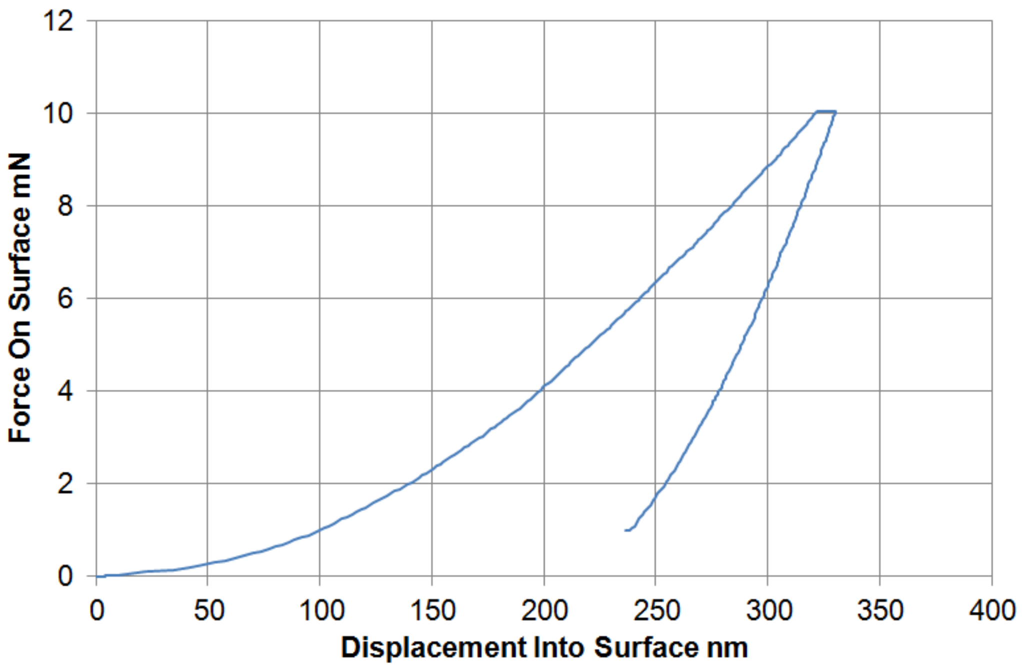


1. Material doesn't exist in volume or form suitable for fashioning a test piece.
2. Material behaves differently at small length scales.



Young's modulus and yield stress <u>by indentation</u>





The most cited article in the entire field of material science (over 10,000 according to Google Scholar): Oliver, W.C. and Pharr, G.M., "An Improved Technique for Determining Hardness and Elastic-Modulus Using Load and Displacement Sensing Indentation Experiments," *Journal of Materials Research* **7**(6), 1564-1583, 1992.

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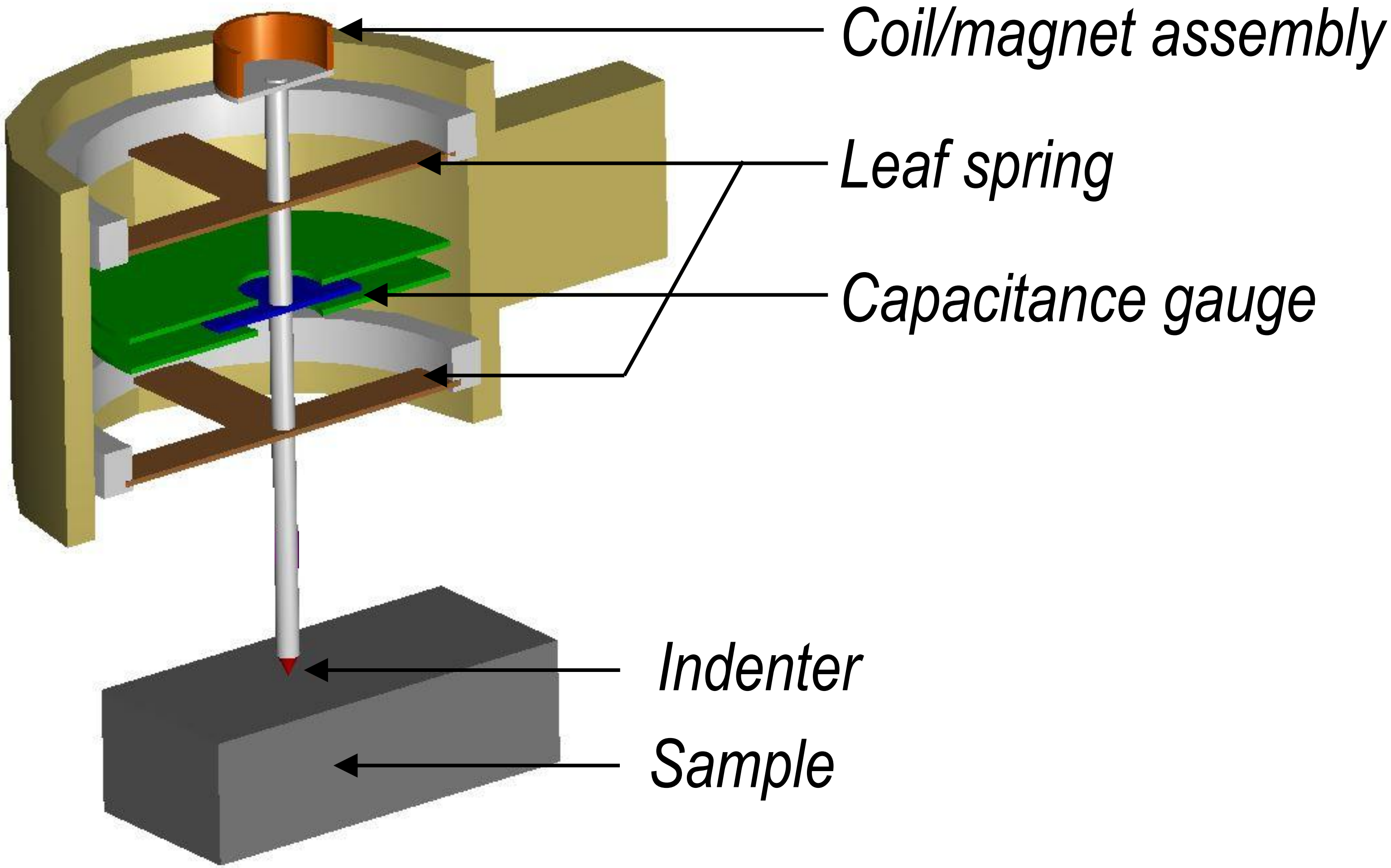
NanoIndenter ® G200 (XP or DCM) schematic



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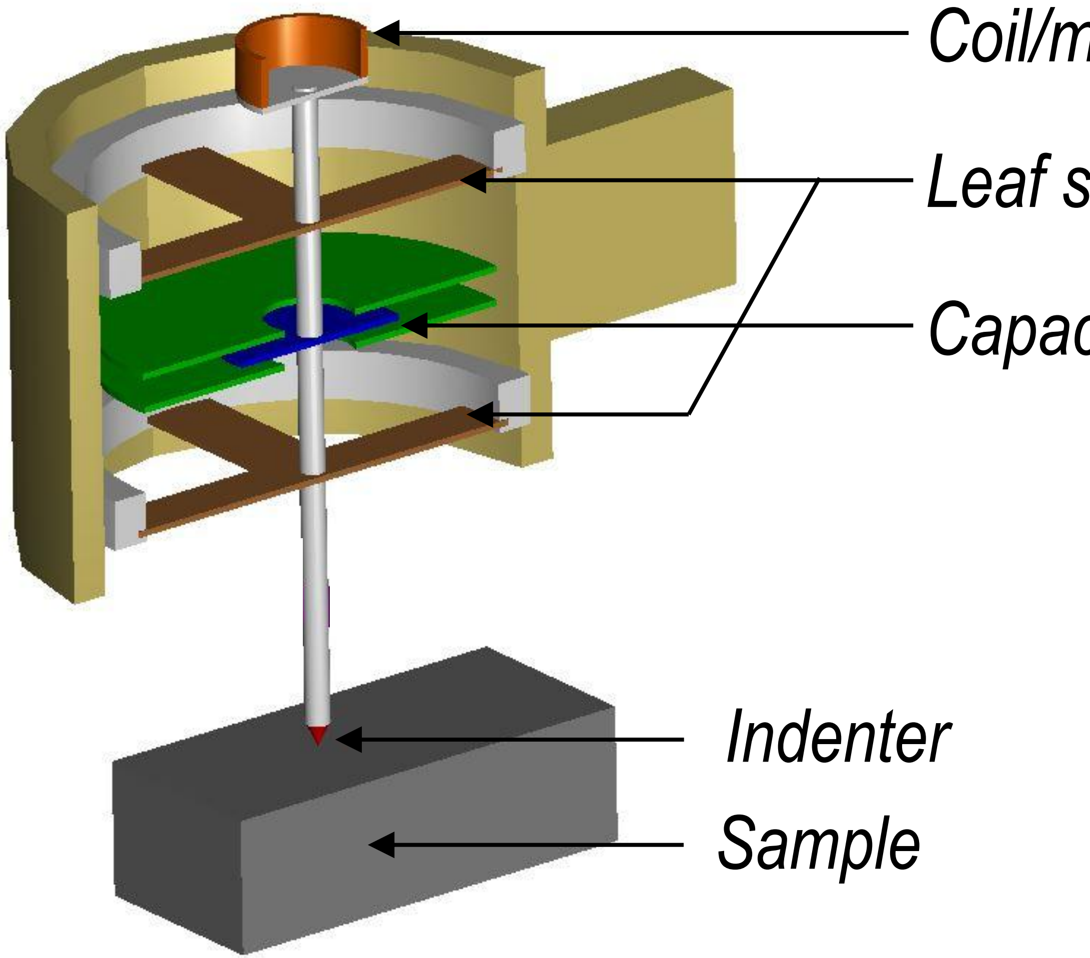
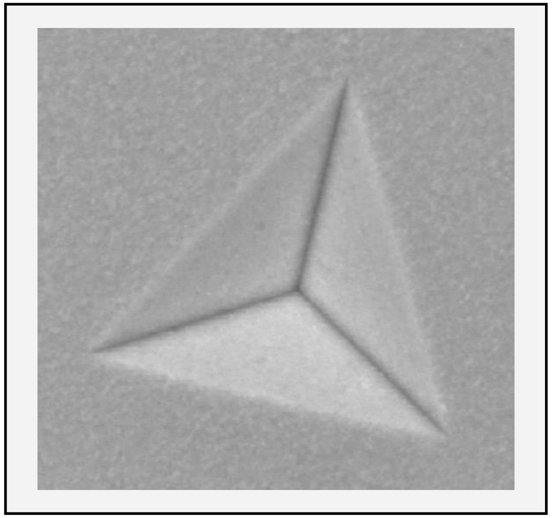


Nickel



Typical test chronology

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

Loading: The indenter is pressed into contact with the test material until the maximum force or penetration is achieved.

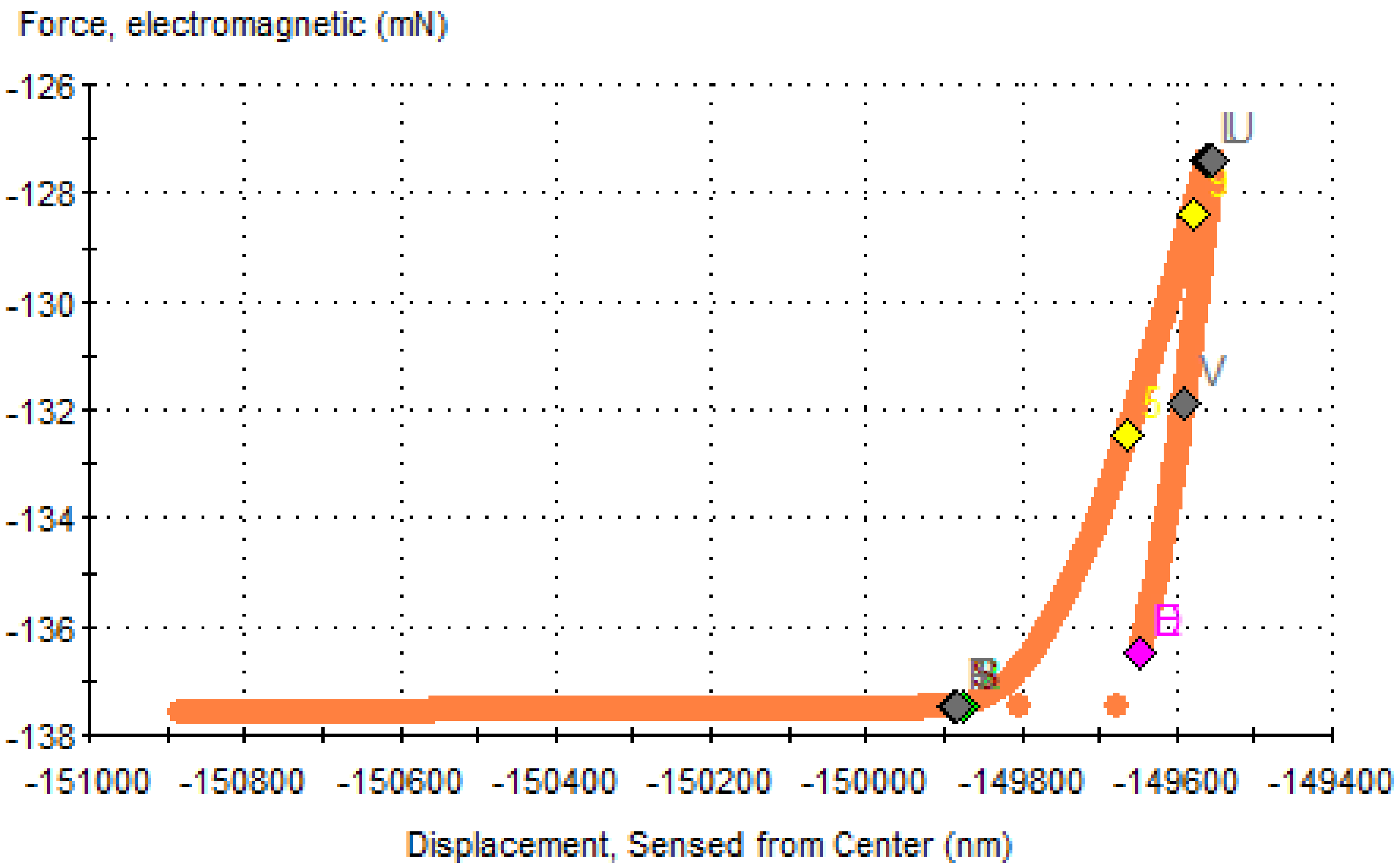
Dwell: The force on the indenter is held constant for a dwell time at the peak force.

Unloading: The indenter is withdrawn from the sample at a rate that is comparable to the pressing rate until the force becomes a small percentage of the peak force, usually 10%.

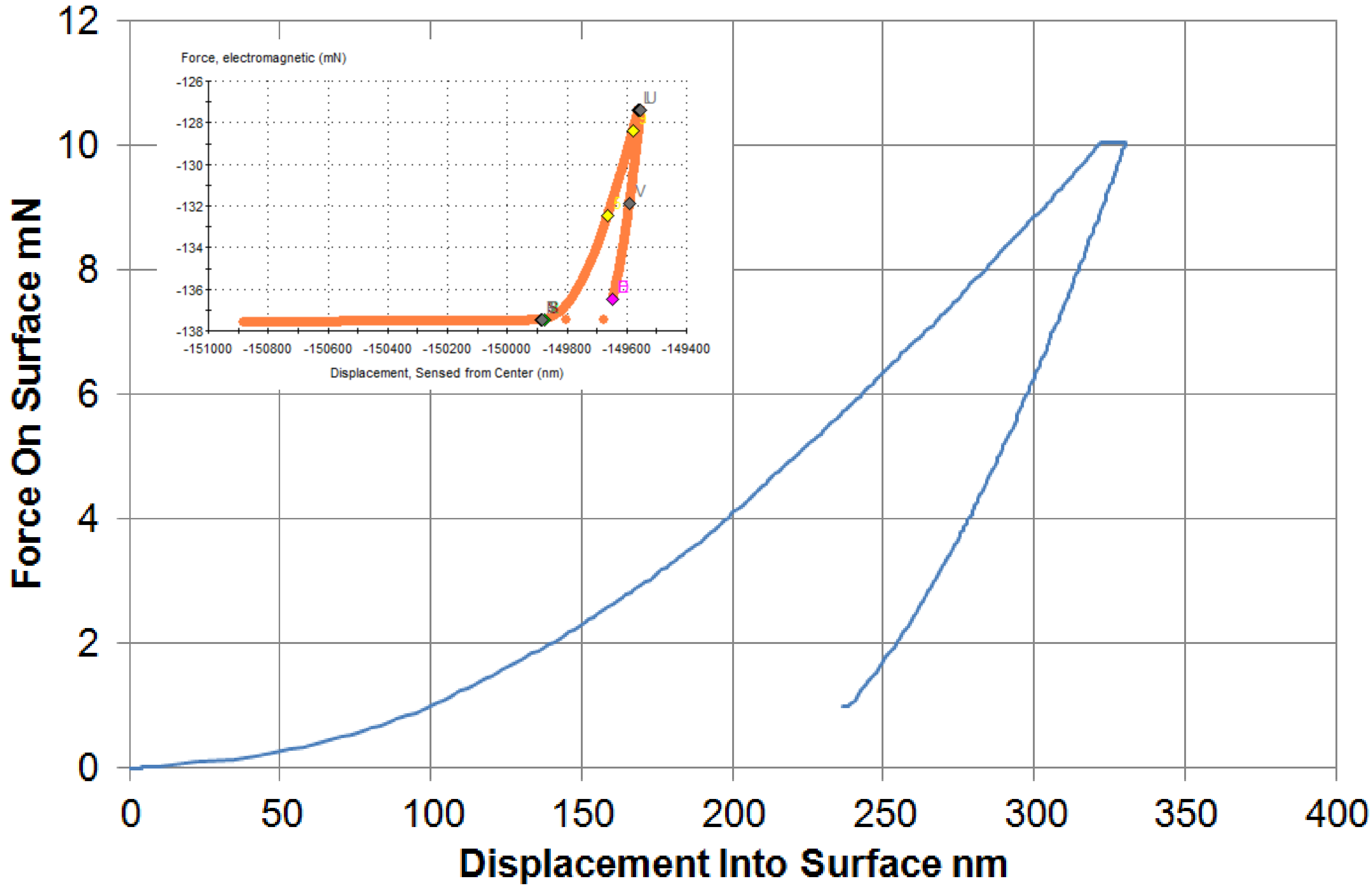
Dwell for thermal-drift evaluation: The force on the indenter is held constant for a dwell time. Measured displacements are attributed to *thermal drift* (expansion and contraction of the equipment and/or test material). If thermal drift is expected to be small relative to the overall penetration of the test, this segment may be omitted.

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

From raw measurements….



To a usable load-displacement curve



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All instruments do these things, but differently

- Determine the point of contact (not necessarily the same point at which approach terminated and loading began).
- Reference all measurements to values at contact.
- Compensate displacement for instrument deflection (frame compliance).
- Compensate displacement for thermal drift.
- Compensate force for influence of supporting mechanism.

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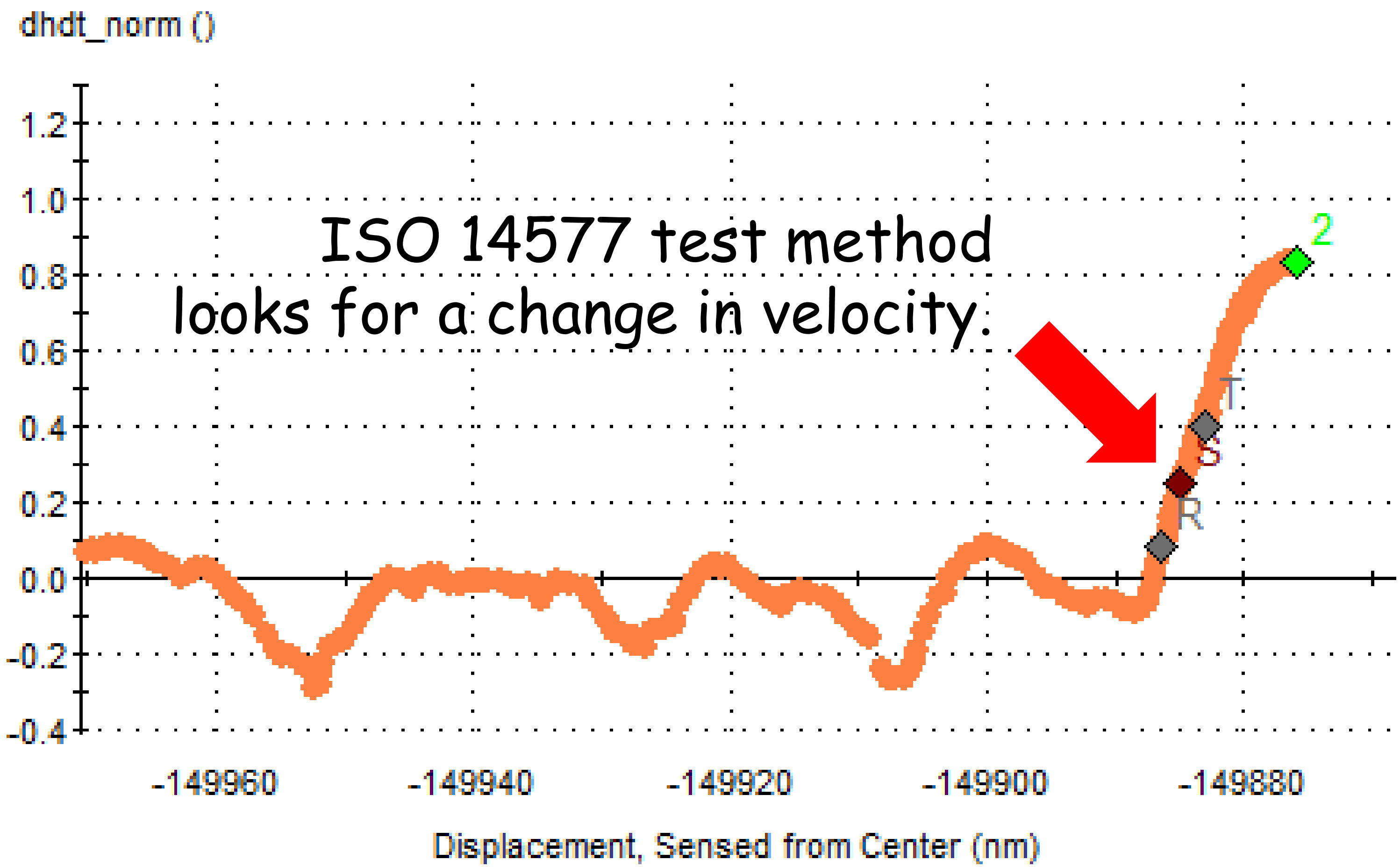
Determining contact point



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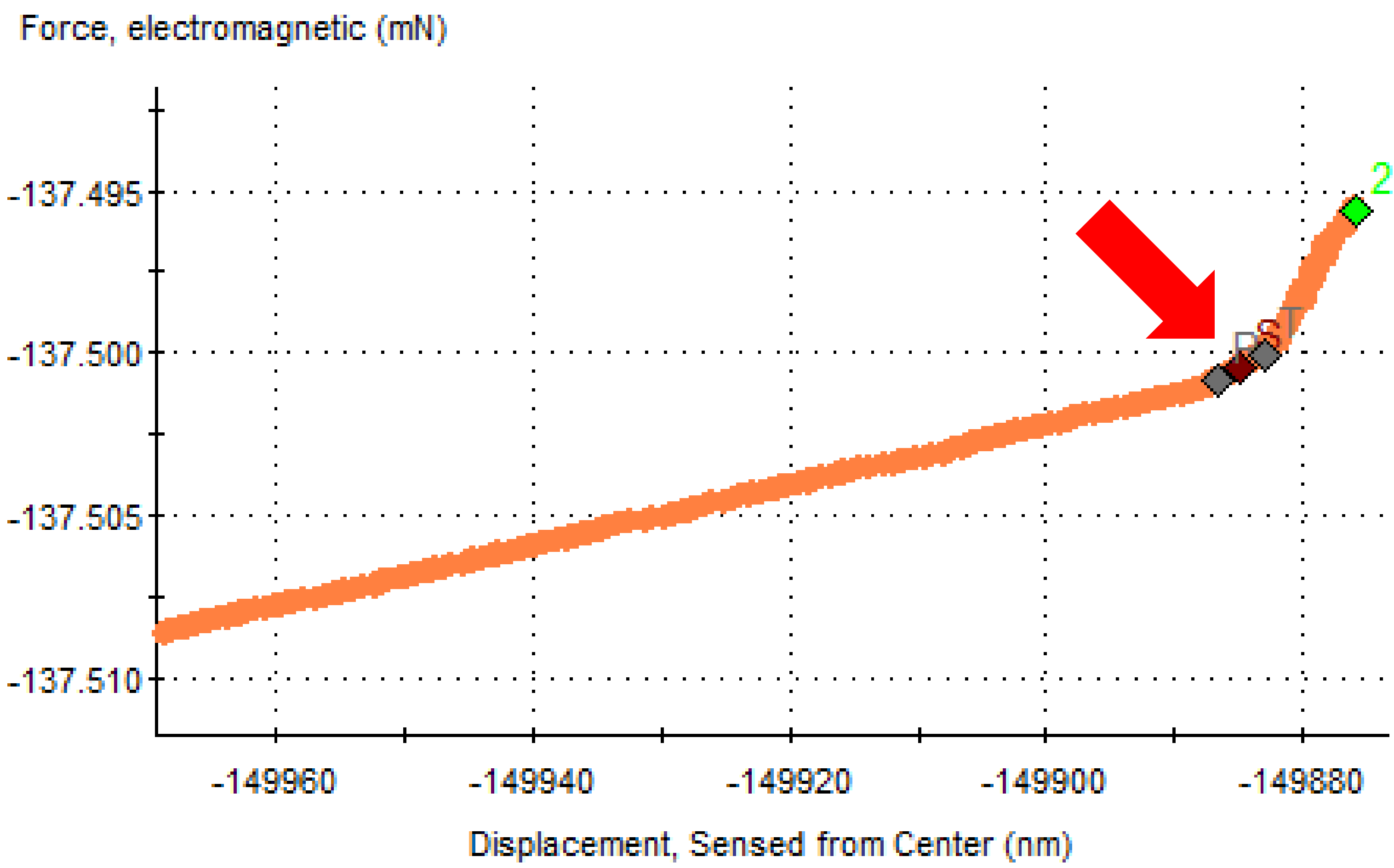

Determining contact point

- Different test methods use different algorithms
- Algorithm of choice depends on available hardware and target materials for the method.
- In most methods (but not ISO 14577), user may override automatically determined contact point by positioning the crosshairs over the desired point and pressing ‘s’ on the keyboard.

Getting to the load-displacement curve

- Determine the point of contact (not necessarily the same point at which approach terminated and loading began).
- **Reference all measurements to values at contact.**
- Compensate displacement for instrument deflection (frame compliance).
- Compensate displacement for thermal drift.
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Taring channels

Displacement: $h = z - \boldsymbol{z_s}$

Force: $P = F - \boldsymbol{F_s}$

Getting to the load-displacement curve

- Determine the point of contact (not necessarily the same point at which approach terminated and loading began).
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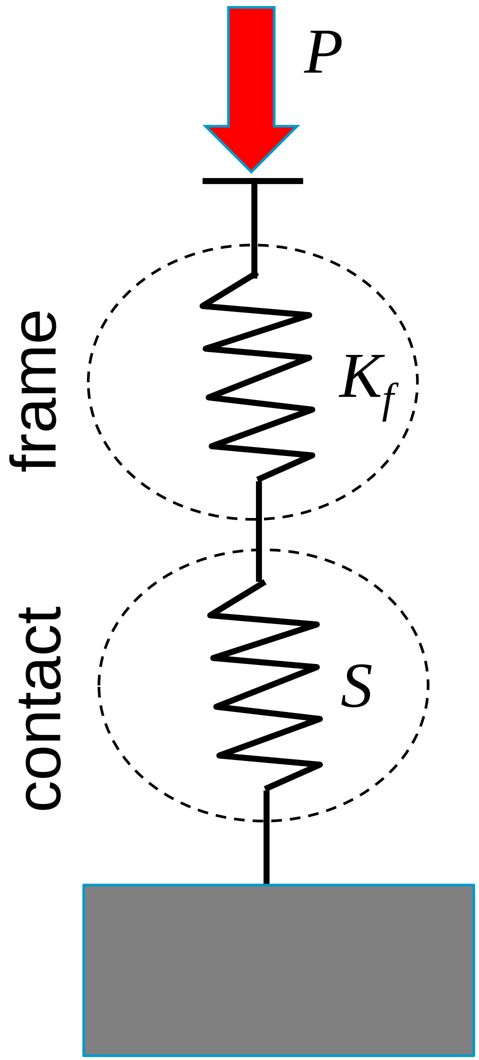
Instrument Frame Stiffness in Common Analysis



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

Determining Frame Stiffness

Determining Frame Stiffness (K_f) and Area Function (Part 1-Theory)
https://agilenteseminar.webex.com/agilenteseminar/lsr.php?AT=pb&SP=EC&rID=5138702&rKey=1be38082f71e07ff

Determining Frame Stiffness (K_f) and Area Function (Part 2-Practice)
https://agilenteseminar.webex.com/agilenteseminar/lsr.php?AT=pb&SP=EC&rID=5142917&rKey=057c3649a13f54e6

Compensating for Frame Stiffness

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

Force: $P = F - F_s$

Getting to the load-displacement curve

- Determine the point of contact (not necessarily the same point at which approach terminated and loading began).
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- **Compensate displacement for thermal drift.**
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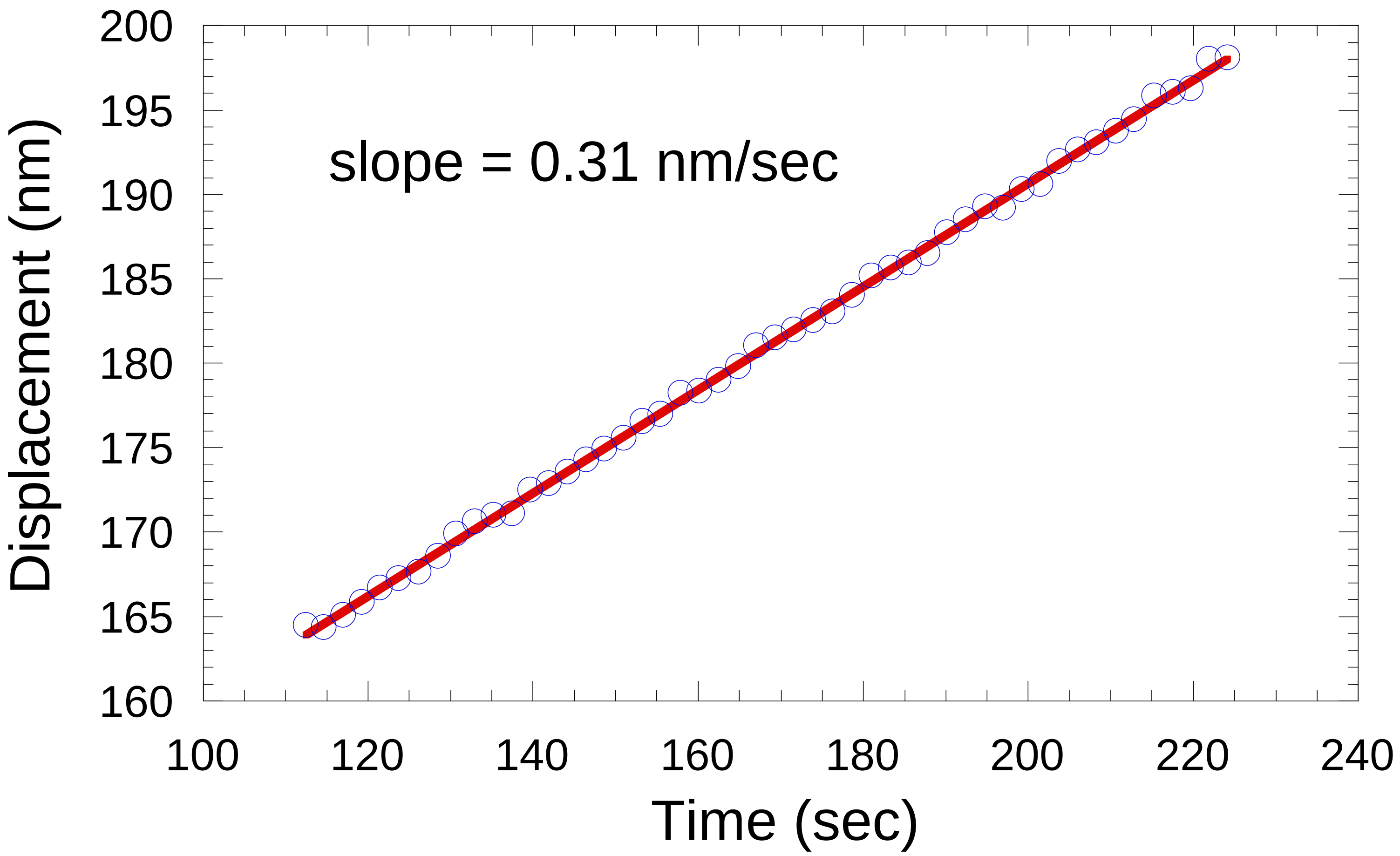
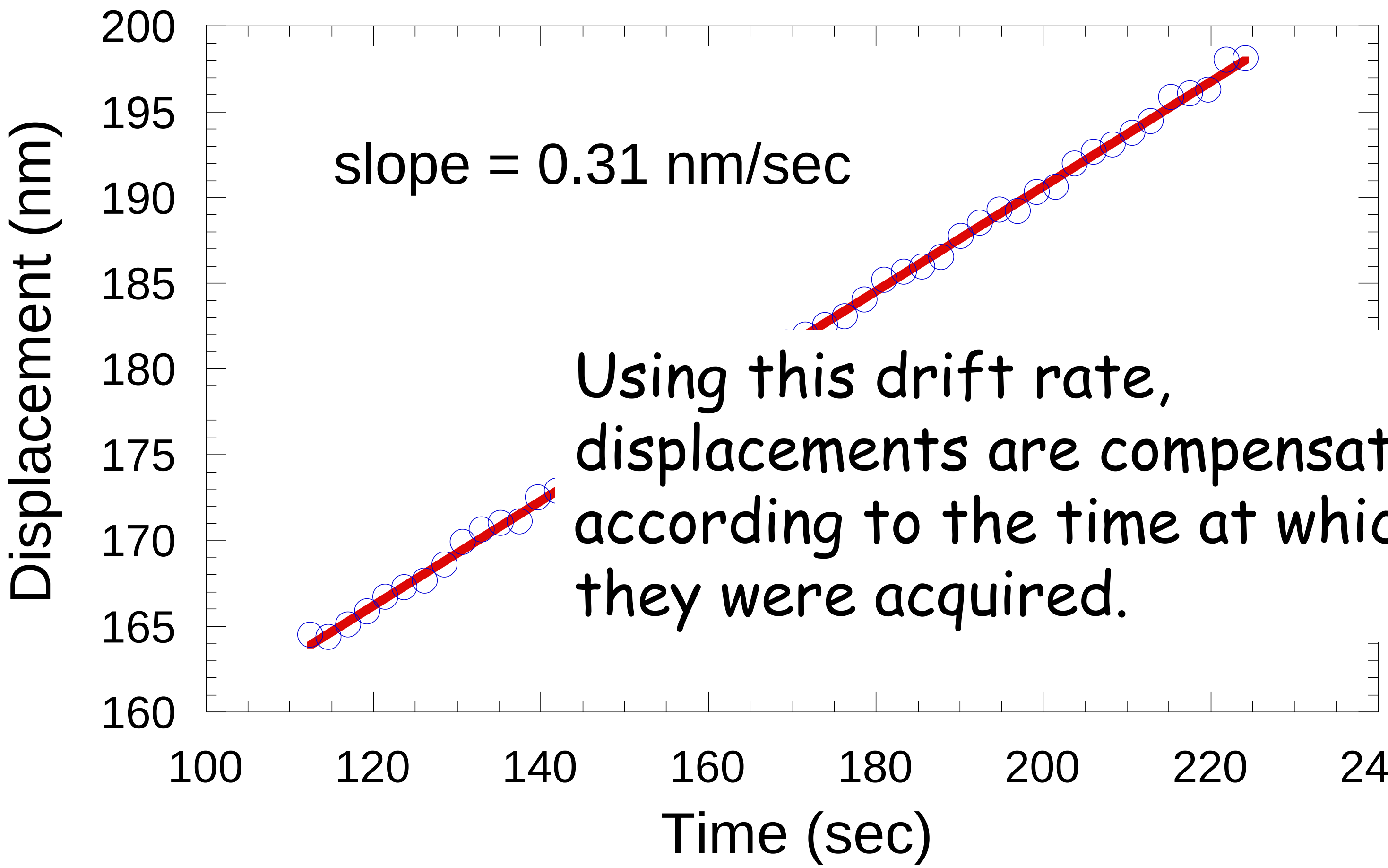
Determining Thermal Drift



Determining Thermal Drift



Using this drift rate, displacements are compensated according to the time at which they were acquired.

Compensating for Thermal Drift

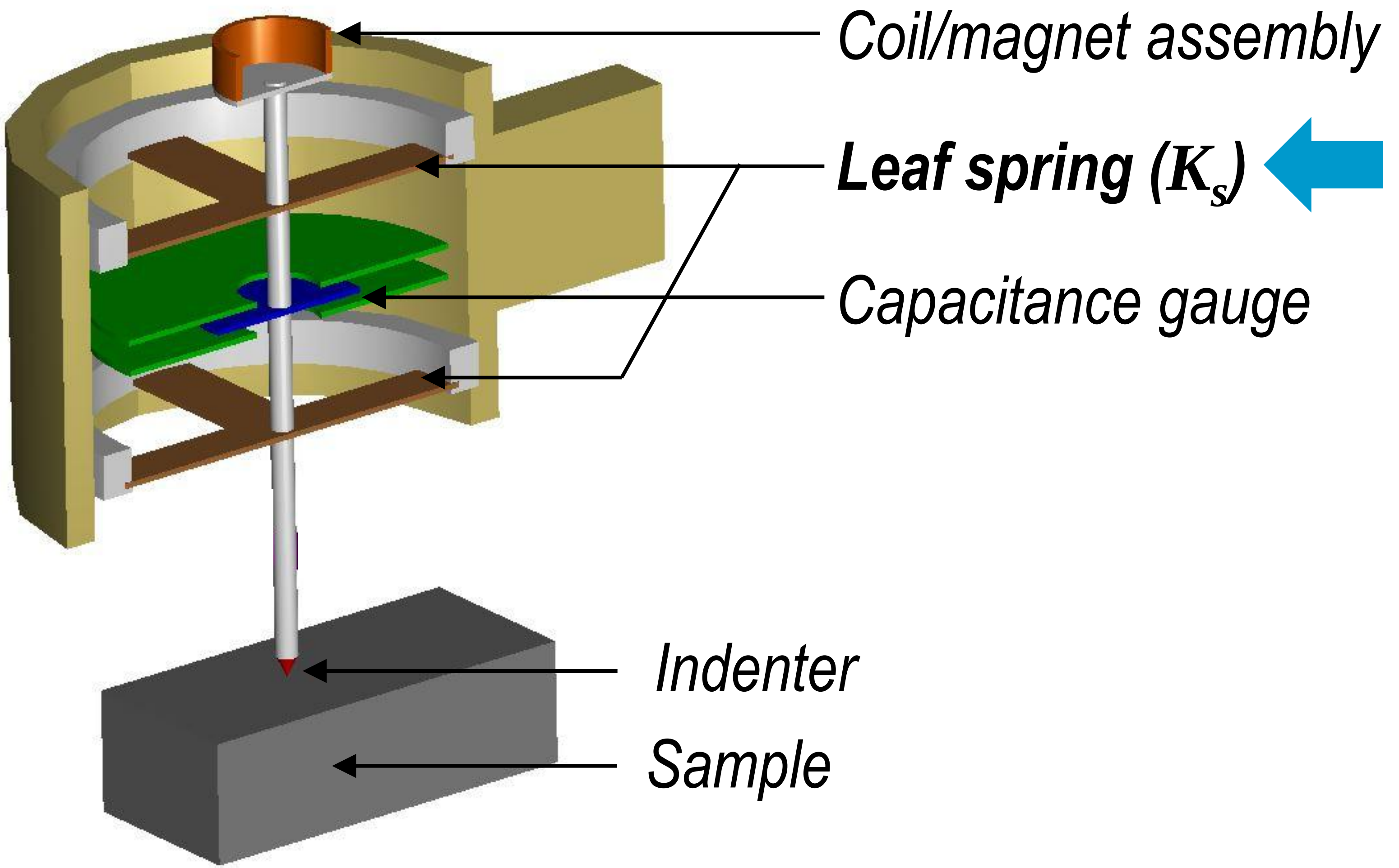
Displacement: $h = z - z_s - (F - F_s)/K_f - \boldsymbol{DR\,(t - t_s)}$

Force: $P = F - F_s$

Getting to the load-displacement curve

- Determine the point of contact (not necessarily the same point at which approach terminated and loading began).
- Reference all measurements of force, displacement, and time to contact point.
- Compensate displacement for instrument deflection (frame compliance).
- Compensate displacement for thermal drift.
- **Compensate force for influence of supporting mechanism.**

NanoIndenter ® G200 (XP or DCM) schematic



Compensating for Support-Spring Deflection

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

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

NanoSuite Channel: Displacement Into Surface

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

DisplacementIntoSurface =
_Displacement - _Displacement[SurfaceMarker]
- (1.0*(_Load-_Load[SurfaceMarker])/(_Frame +
FrameStiffnessCorrection))
-DriftCorrection*(_Time - _Time[SurfaceMarker])

NanoSuite Channel: Displacement Into Surface

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

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NanoSuite Formula: Drift Correction

DriftCorrection =
PerformDriftCorrection*
HoldForDriftDone*
SlopeValue
(_Displacement,_Time,
DriftDeterminationMarker,
EndDriftMarker)

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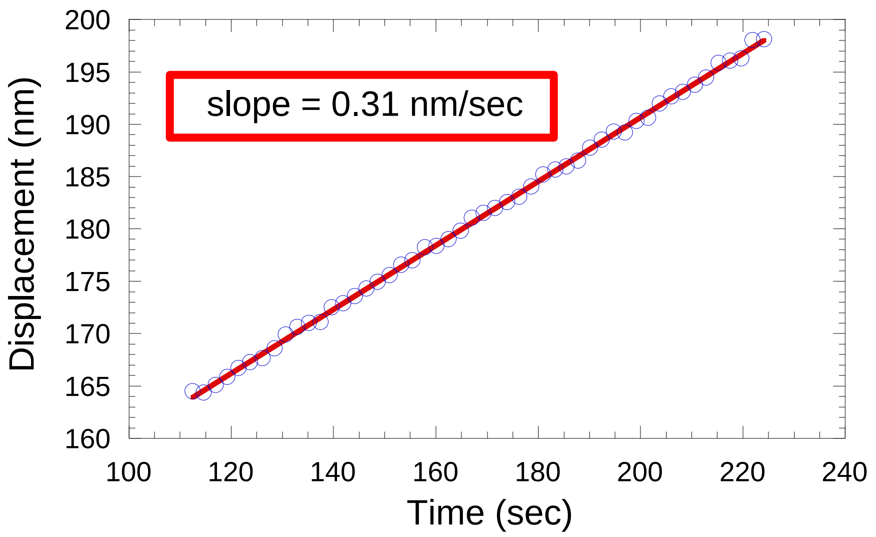

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NanoSuite Formula: Drift Correction

Editable Inputs	Value	Units
Percent Unload In Stiffness Calculation	50.000	%
Perform Drift Correction	1	(Integer)
Beta	1.000	(None)
Poissons Ratio	0.180	(None)
Poisson Ratio Unc.	0.000	(None)
Frame Stiffness Correction	0.000	N/m

DriftCorrection = **PerformDriftCorrection*** HoldForDriftDone* SlopeValue (_Displacement,_Time, DriftDeterminationMarker, EndDriftMarker)

NanoSuite Formula: Drift Correction

DriftCorrection =
PerformDriftCorrection*
HoldForDriftDone*
SlopeValue
(_Displacement,_Time,
DriftDeterminationMarker,
EndDriftMarker)

Display Name	Value	Units
Do Drift Hold When Max Force Below	100.000	mN
Peak Hold Time	10.000	s
Percent To Unload	90.000	%
Force Limit First Test	500.000	mN
Force Decrement Factor	1.000	
Time To Load	30.000	s
Poissons Ratio	0.180	

NanoSuite Channel: Force On Surface (or in some methods 'Load On Sample')

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

Force On Surface =
_Load-_Load[SurfaceMarker] -(_Displacement-_Displacement[SurfaceMarker])*_Column

NanoSuite Channel: Force On Surface (or in some methods ‘Load On Sample’)

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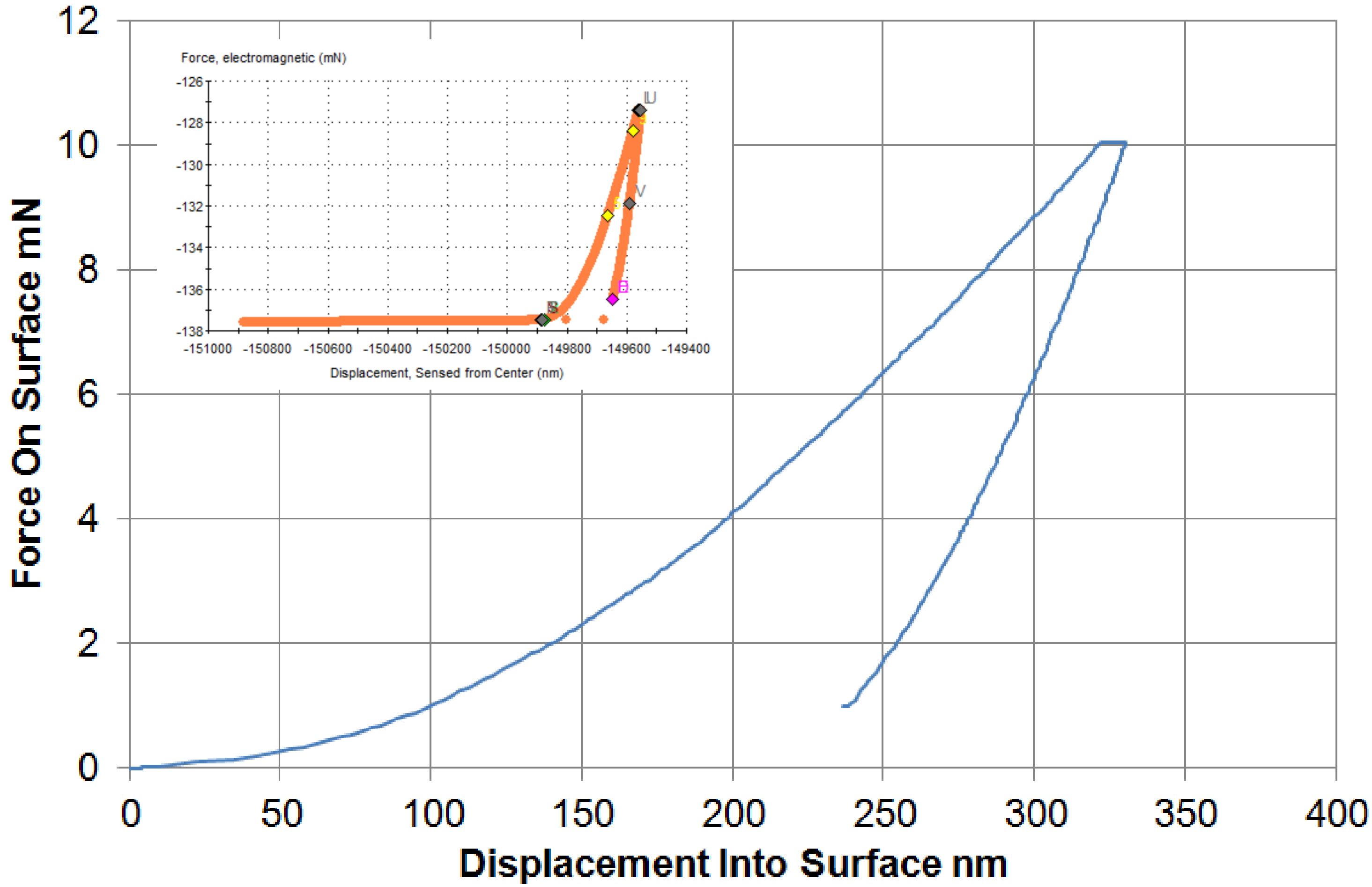
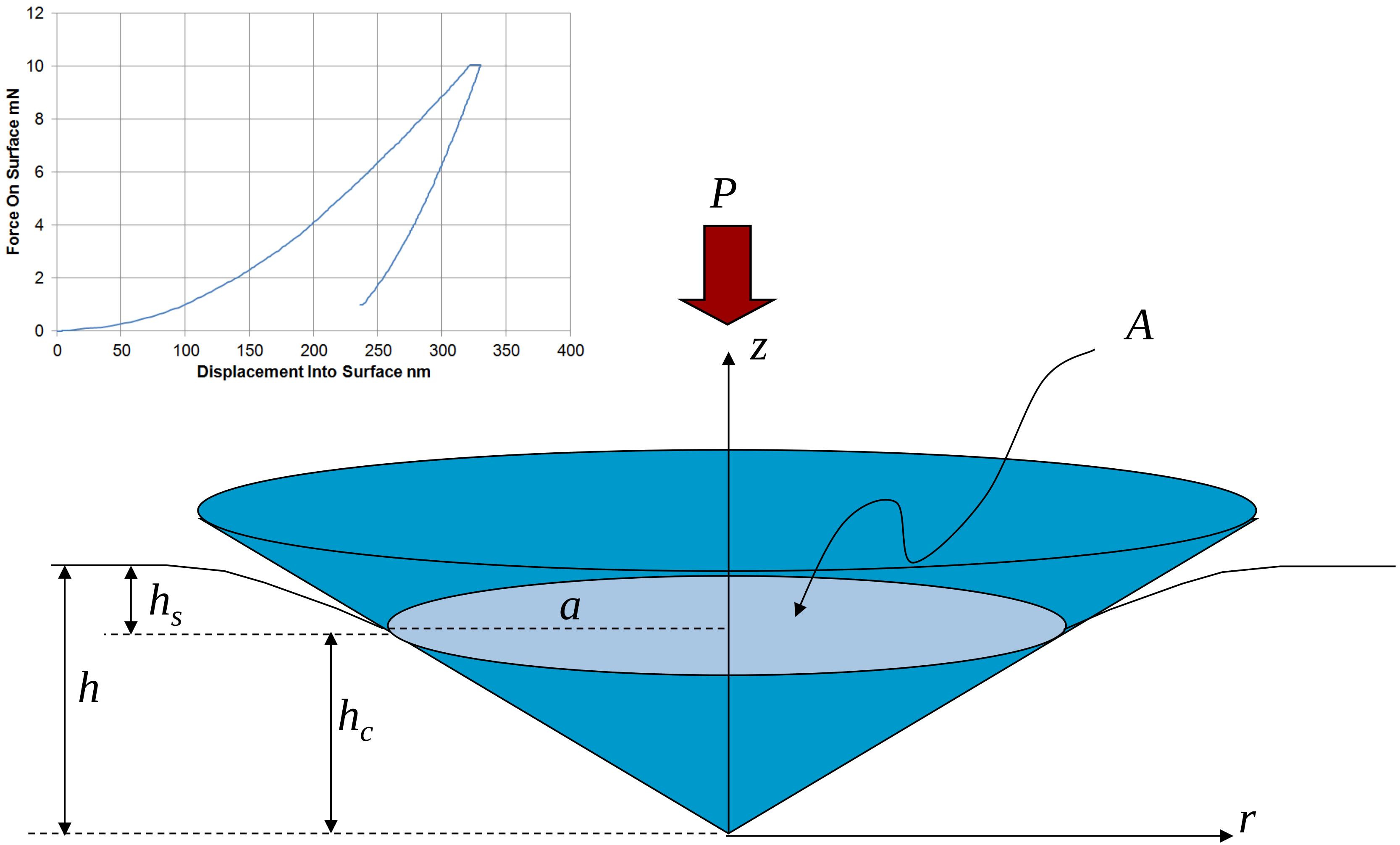
A usable load-displacement curve



Schematic for nomenclature



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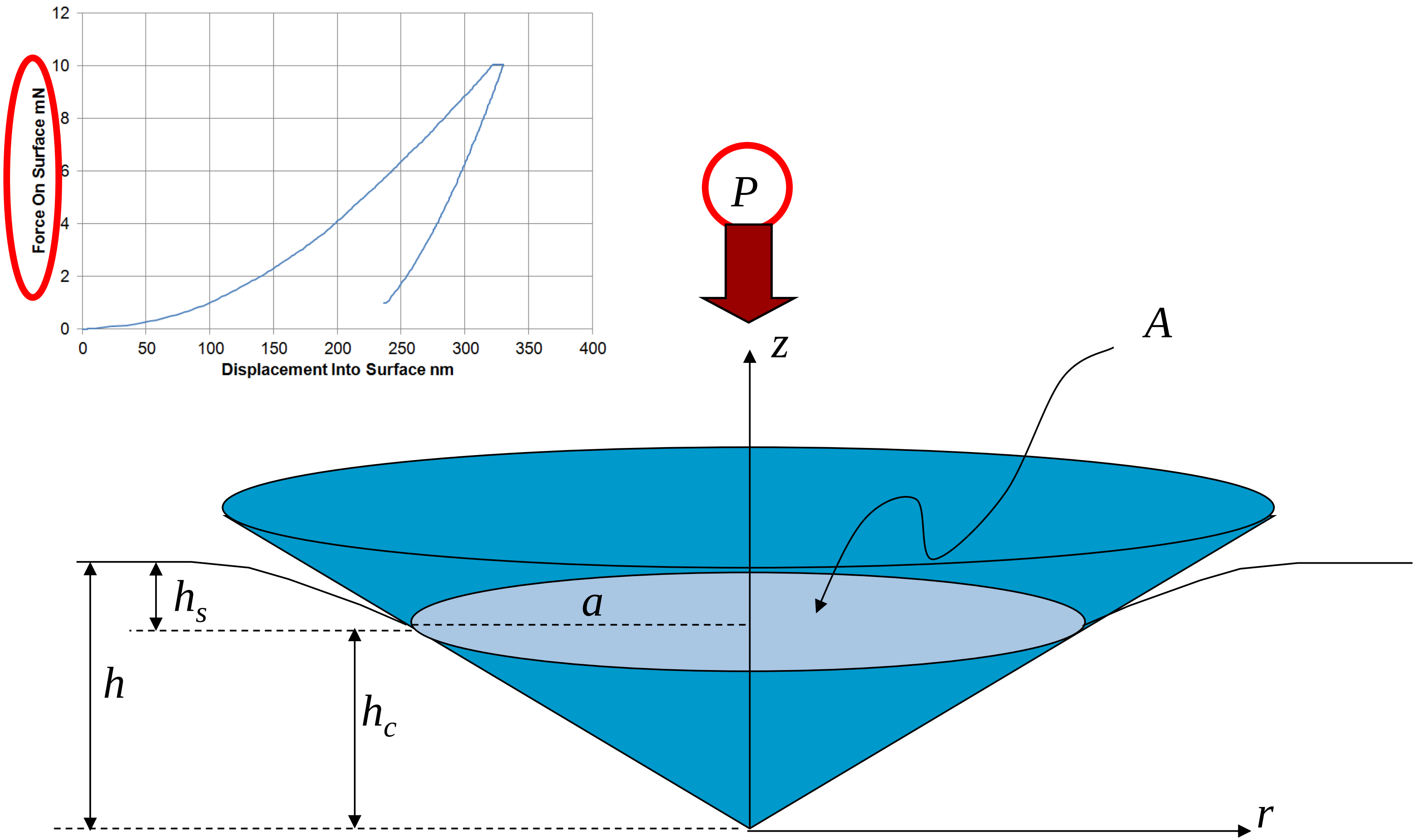

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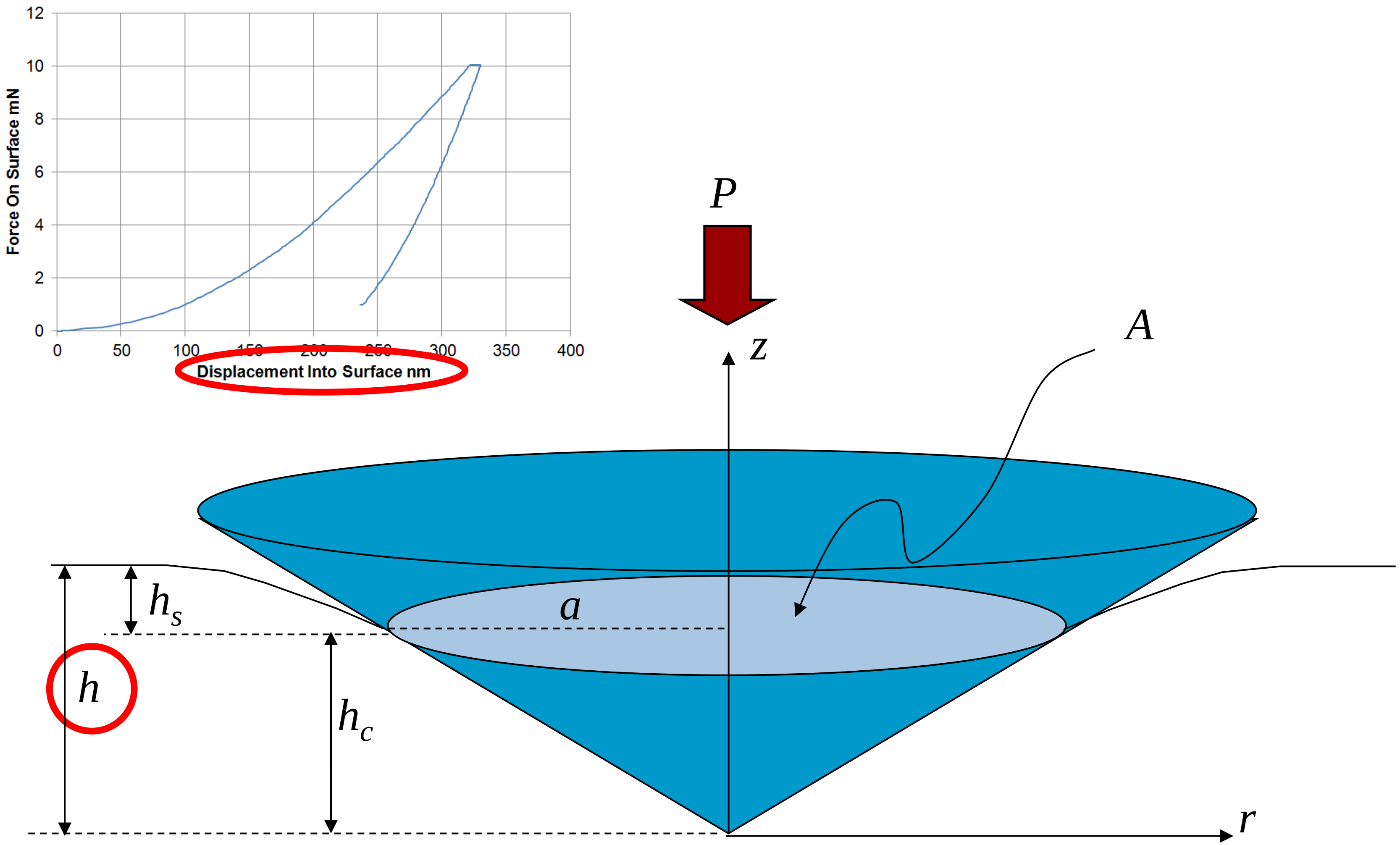
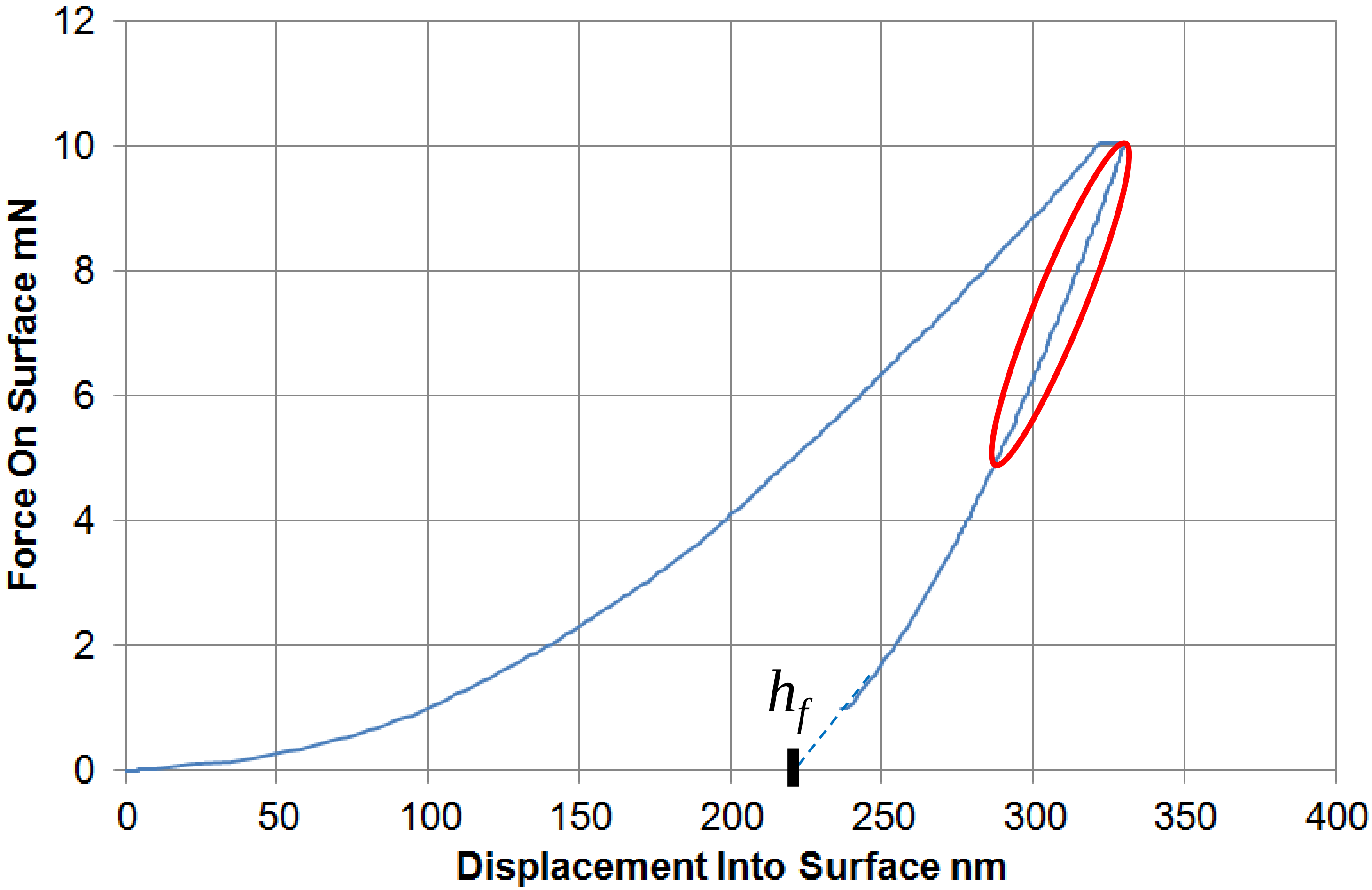


Axisymmetric geometry models for Berkovich indentation?

- We unabashedly interpret the load-displacement curves obtained with Berkovich indenters with elastic contact models derived for axisymmetric conical indenters.
- Why?
- Historically, that is what the founders did.
- “When all you have is a hammer, everything looks like a nail.”
- 3D finite-element simulations comparing Berkovich and conical elasto-plastic indentation have since justified this tactic. Although the stress fields are different, the load-displacement curves are the same.

Elastic models apply to the unloading curve



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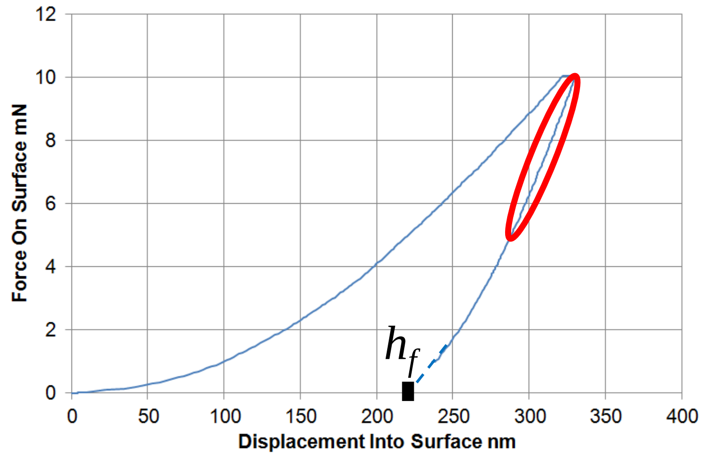
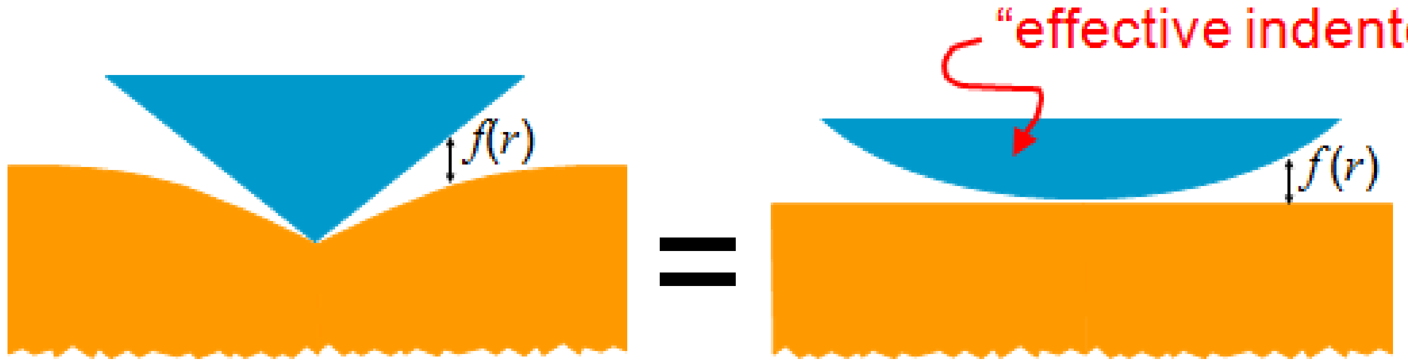


1. Relevant **elastic** displacement is $(h - h_f)$.
2. Effective indenter shape is best modeled as paraboloid, due to prior elastic deformation.



Summary of IIT (nanoindentation) analysis

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

$$\sigma_y \approx H/2.8$$

$$H = P/A$$

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

$$A = f(h_c)$$

$$h_c = h - 0.75P/S$$

$$S = \left.\frac{dP}{dh}\right|_{h_{max}}$$

Nomenclature:

E	Young's modulus
H	hardness
σ_y	Yield stress
E_r	reduced modulus
ν	Poisson's ratio
i	(as subscript) indenter
S	contact stiffness
A	projected contact area
h_c	contact depth
h	displacement
P	applied force (load)

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Determining contact stiffness, *S*

Editable Inputs	Value	Units
Percent Unload In Stiffness Calculation	50.000	%
Perform Drift Correction	1	(Integer)

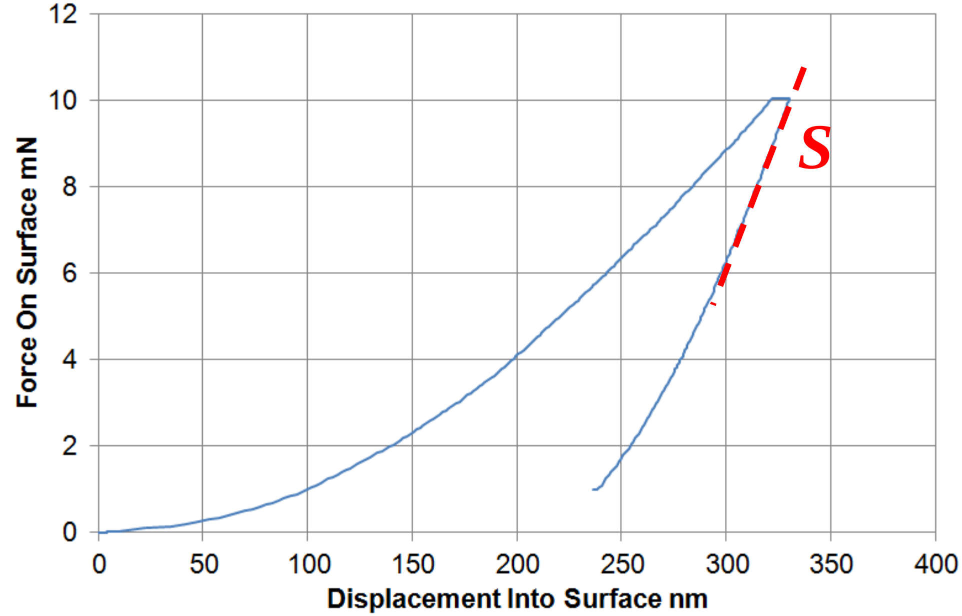
- Fit all unloading data for which $P > 0.5P_{max}$ (i.e. top half of unloading curve)

- Fit to the functional form

$$P = B(h-h_f)^m$$

- Analytically differentiate and evaluate at $h = h_{max}$:

$$S = \mathrm{d}P/\mathrm{d}h = Bm(h_{max} - h_f)^{m-1}$$



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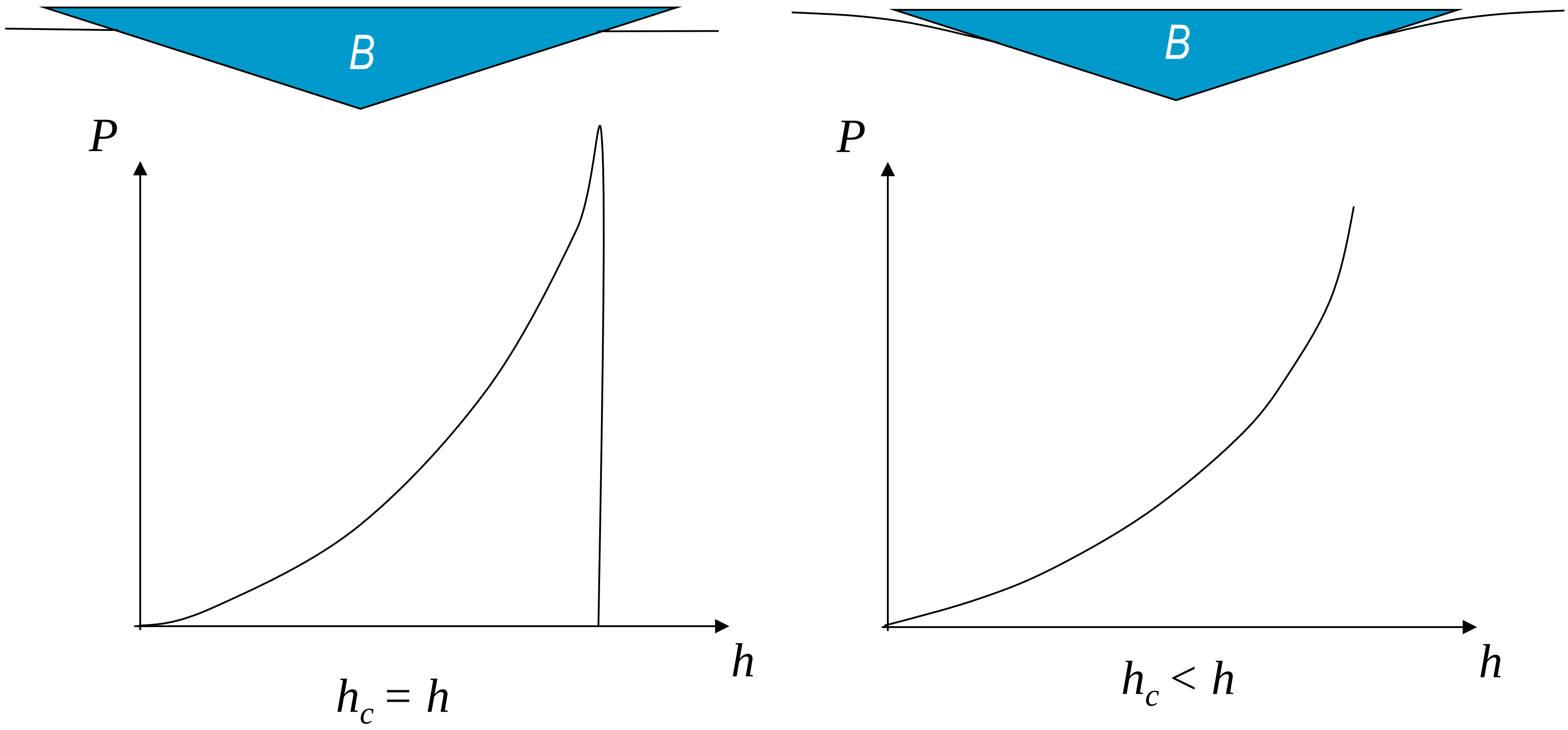
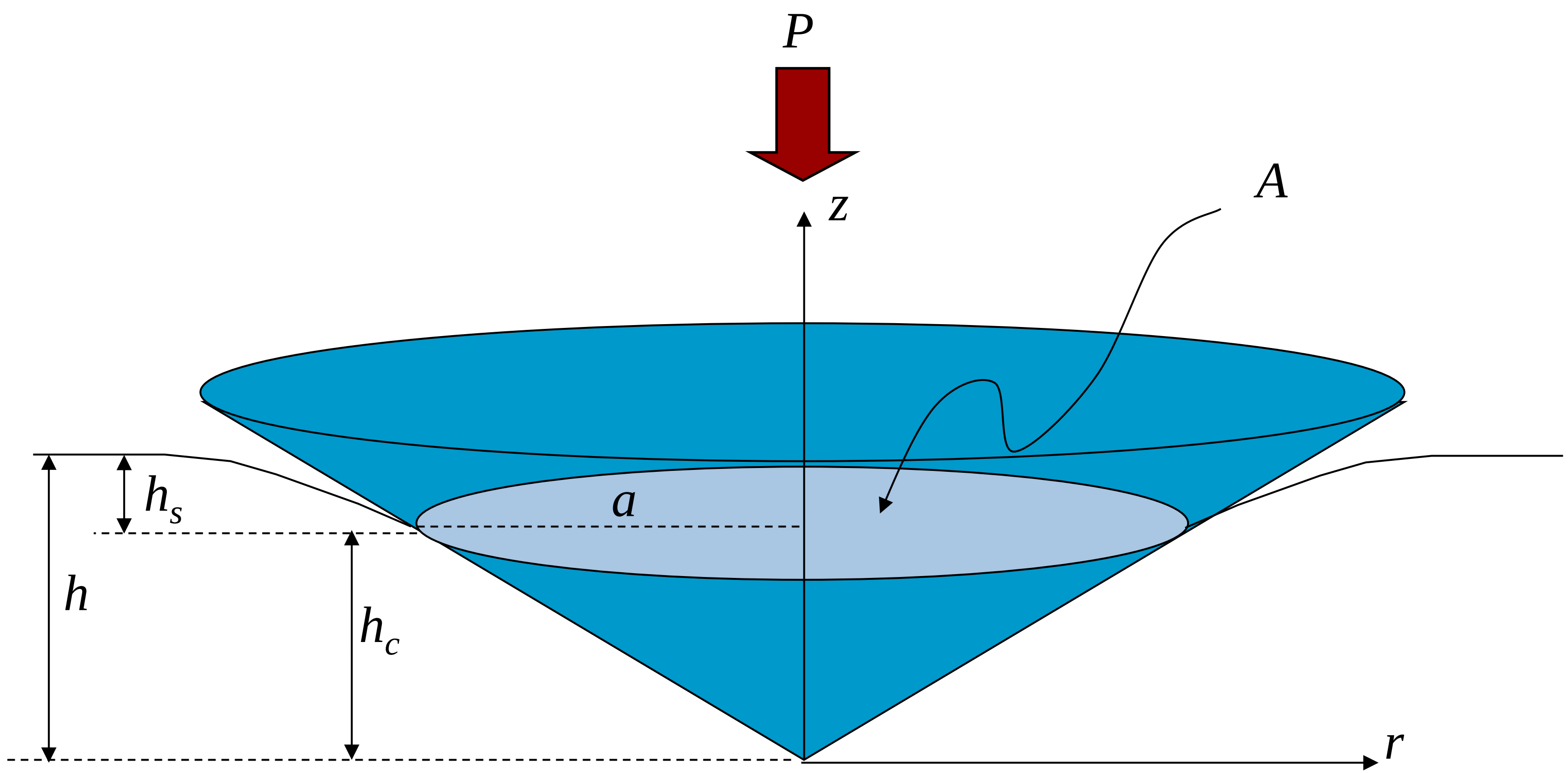
Intuitive "rightness" of $h_c = h - 0.75P/S$



Calculating contact depth, h_c

$h_c = h - h_s$ (always true, for any indenter, even with pileup)



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Calculating contact depth, h_c

$$h_c = h - h_s$$

Always true, for any indenter, even with pileup

Strategy: Assume that the material outside the contact area deforms elastically; use elastic contact mechanics to derive an expression for h_s (the elastic deflection of the surface) in terms of P and S.

Calculating contact depth, h_c

$h_c = h - h_s$ Always true, for any indenter, even with pileup

$h_c = (h - h_f) - h_s$ Version of the previous expression, applied to the elastic unloading curve for which the relevant displacement is $h - h_f$

Calculating contact depth, h_c

$h_c = h - h_s$

Always true, for any indenter, even with pileup

$h_c = (h - h_f) - h_s$

Version of the previous expression, applied to the elastic unloading curve for which the relevant displacement is $h - h_f$

$h_c = (h - h_f)/2$

Sneddon's expression for contact depth for paraboloid on flat (Hertizan contact) using relevant displacement $h - h_f$

Calculating contact depth, h_c

$h_c = h - h_s$ Always true, for any indenter, even with pileup

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$h_c = (h - h_f)/2$ Sneddon's expression for contact depth for paraboloid on flat (Hertizan contact) using relevant displacement $h - h_f$

$h_s = (h - h_f)/2$ Algebra

Calculating contact depth, h_c

$h_c = h - h_s$ Always true, for any indenter, even with pileup

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Calculating contact depth, h_c

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$$P = \frac{4E_r a^3}{3R}$$

Sneddon's expression for force for paraboloid on flat (Hertzian contact)

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Algebra

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$$h - h_f = \frac{a^2}{R}$$

Sneddon's expression for displacement for paraboloid on flat (Hertzian contact); remember that relevant elastic displacement is $h - h_f$

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$$h_c = h - 0.75P/S$$ Algebra

Note: We derived this value assuming that the effective indenter is paraboloid. If you assume the effective indenter is conical, this value comes out to 0.72 (this analysis is in O&P, 1992).

Summary of IIT (nanoindentation) analysis

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

$$\sigma_y \approx H/2.8$$

$$H = P/A$$

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

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Nomenclature:

E	Young's modulus
H	hardness
σ_y	Yield stress
E_r	reduced modulus
ν	Poisson's ratio
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A	projected contact area
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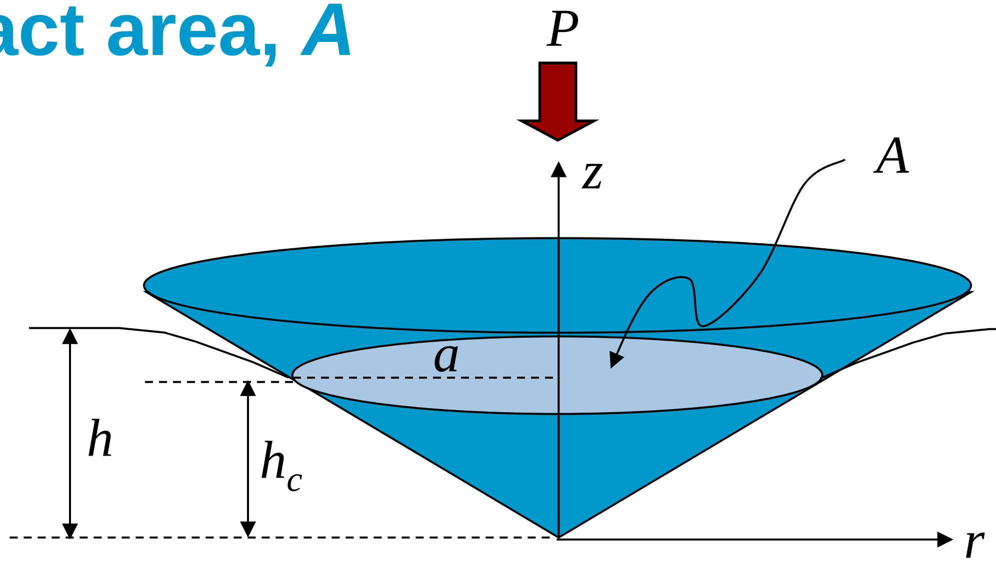
Determining contact area, *A*



Generally:

$A = \mathbf{f}(h_c)$

Specifically:

Tip type	Area function	Comments
Perfect Berkovich	$A = 24.56h_c^2$	Used when $h_c > 2$ microns
Real Berkovich	$A = 24.56h_c^2 + Ch_c$	C is determined by indenting a known material and is about 150nm.
Sphere	$A = 2\pi Rh_c^2$	R is tip radius; value may be determined by indenting a known material.
Real cube-corner	$A = 2.60h_c^2 + Ch_c$	C is determined by indenting a known material and is about 150nm.
Flat-ended cylinder	$A = \pi a^2$	a is the punch radius; A is constant (independent of indentation depth)

Determining area function, A = f(h_c)

Determining Frame Stiffness (K_f) and Area Function (Part 1-Theory)
https://agilenteseminar.webex.com/agilenteseminar/lsr.php?AT=pb&SP=EC&rID=5138702&rKey=1be38082f71e07ff

Determining Frame Stiffness (K_f) and Area Function (Part 2-Practice)
https://agilenteseminar.webex.com/agilenteseminar/lsr.php?AT=pb&SP=EC&rID=5142917&rKey=057c3649a13f54e6

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Sneddon's stiffness equation

$$E_r = \frac{S}{2\gamma a}$$

True for any contact governed by Sneddon's analysis (Oliver, Pharr, and Brotzon, 1992)

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Geometry

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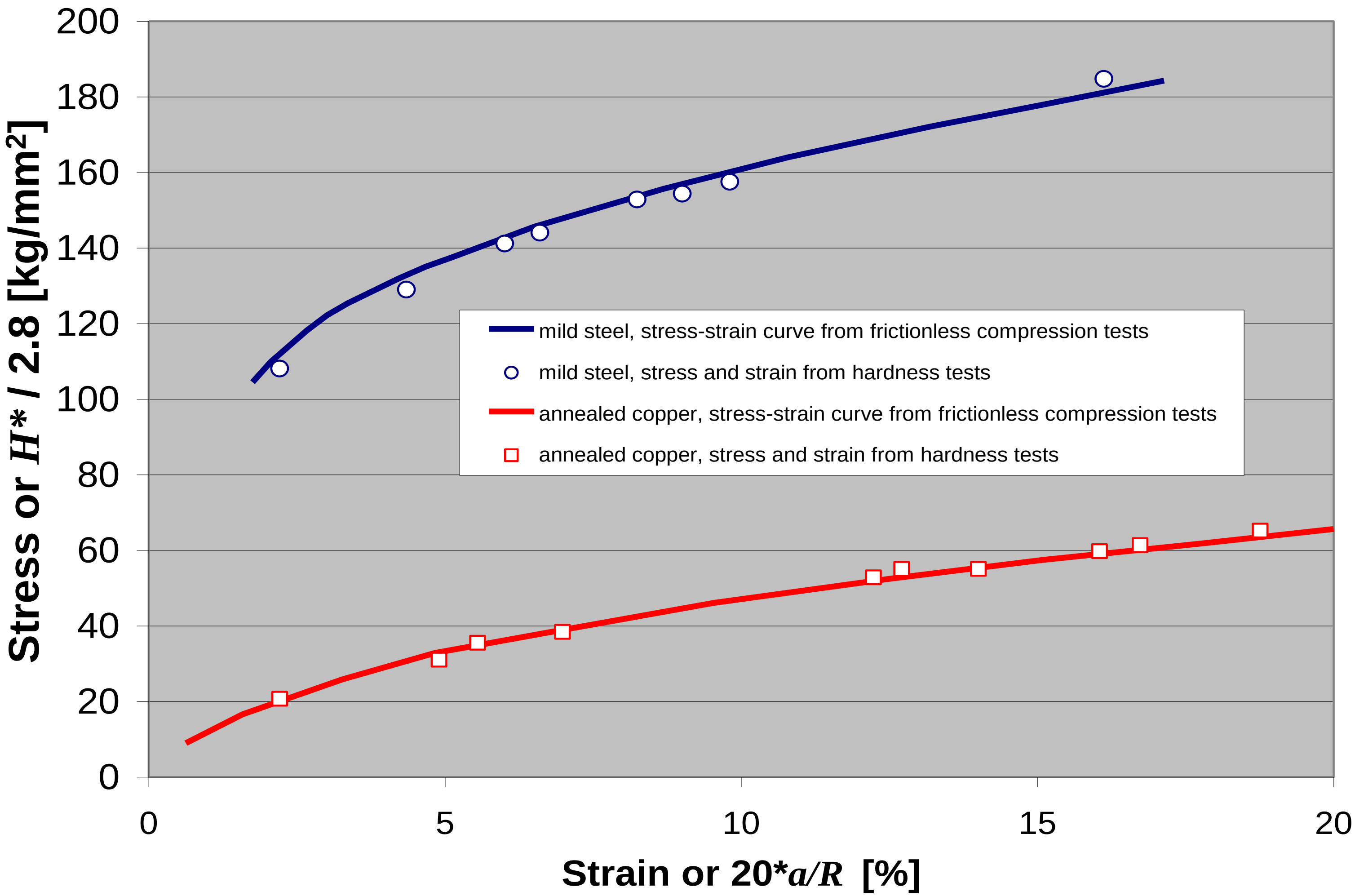
Calculating yield stress from hardness*



From *Hardness of Metals* (Tabor, 1951)

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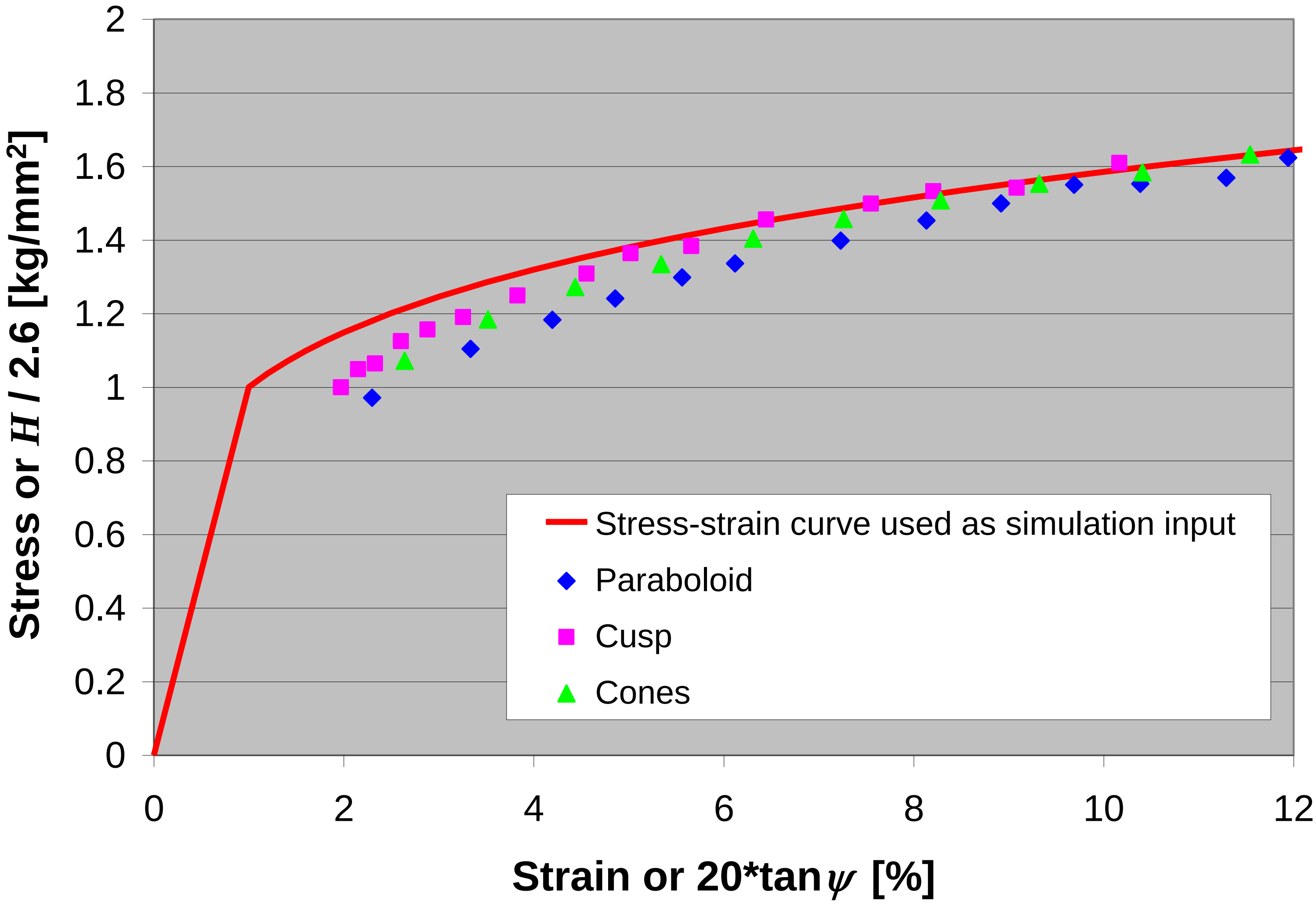
Calculating yield stress from hardness (IIT)



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Determining Young's modulus

$$\frac{1}{E_r} = \frac{\left(1-\nu_s^2\right)}{E_s} + \frac{\left(1-\nu_i^2\right)}{E_i}$$

(For diamond tip, ν_i = 0.07, E_i = 1141 GPa)

What? I have to know one elastic property (ν_s) in order to get the other (E_s)? Actually, this is not as bad as it sounds.

$$E_s = E_r(1-\nu_s^{\ 2})$$

$$\delta E_s = \frac{\mathrm{d}\,E_s}{\mathrm{d}\,v}\delta v = 2E_r v \delta v$$

$$\frac{\delta E_s}{E_s} = \frac{2v}{\left(1-v^2\right)}\delta v$$

So, for a generous uncertainty of ν = 0.25±0.1

$$\frac{\delta E}{E} = \frac{2(0.25)}{\left(1-0.25^2\right)}(0.1) = 5.3\%$$

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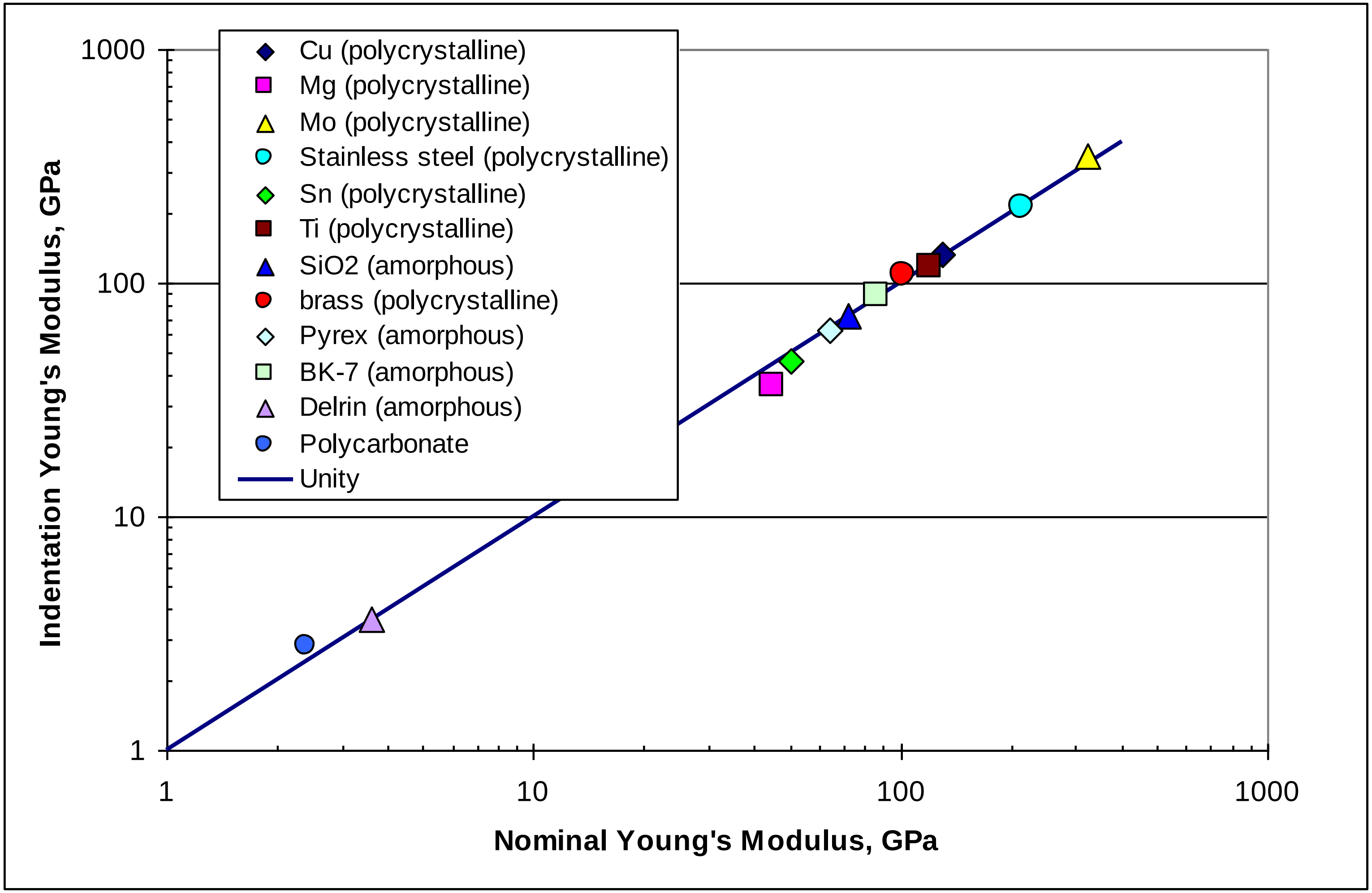
“Oliver-Pharr doesn’t work”

$$h_c \neq h - 0.75P/S$$

Choices are limited:

1. Decide that the error is acceptable.
2. Modify the expression for h_c using analytic or computational modeling.
3. Measure the contact area directly using some form of microscopy, and use this directly measured area for A in the calculation of reduced modulus and hardness.

But when it works, it works



Young's modulus measured by IIT vs. nominal Young's modulus.
Nominal values were determined by tensile test, ultrasound, or DMA.

Summary

- Process for deriving the P-h curve from raw data is instrument specific, but involves (at least) determining the contact point and compensating for frame compliance, thermal drift, and the changing influence of the supporting mechanism.
- Once the P-h curve is obtained, analyses for deriving Young's modulus, hardness, and yield stress are common.
- The primary benefit of the Oliver-Pharr model is that it provides a means for determining contact area without imaging.
- For many materials, the Oliver-Pharr model returns the same value for Young's modulus as a tensile test.

Session 7: Dynamic Instrumented Indentation
Wednesday, April 10, 2013, 11:00 (New York)

Abstract
During basic instrumented indentation (IIT), contact force and penetration are measured continuously as a hard indenter is pressed into contact with, and then withdrawn from, a test material. Dynamic instrumented indentation also involves pressing an indenter into contact with a test material while continuously measuring contact force and penetration. But in addition, a small oscillation is superimposed on the semi-static force, and a frequency-specific amplifier is used to measure the response of the indenter. From the amplitude quotient and phase shift, one derives both the stiffness and damping of the contact. This additional dynamic information affords a number of advantages over basic IIT, the most common of which is the ability to measure hardness and elastic modulus as a continuous function of surface penetration.

To register:
https://agilenteseminar.webex.com/agilenteseminar/onstage/g.php?p=117&t=m

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Suggested reading for Session 6

(Appendix) Oliver, W.C. and Pharr, G.M., "An Improved Technique for Determining Hardness and Elastic-Modulus Using Load and Displacement Sensing Indentation Experiments," *Journal of Materials Research* **7**(6), 1564-1583, 1992.

Hay, J.L., Agee, P., and Herbert, E.G., "Continuous Stiffness Measurement during Instrumented Indentation Testing," *Experimental Techniques* **34**(3), 86-94, 2010.

Upcoming workshops

- **June 2**: Theory & Practice of Instrumented Indentation Testing Course, Sunday, 1:00-5:00p.m., The Westin Lombard Yorktown Center, Lombard, Illinois. A short course offered in conjunction with the SEM 2013 Annual Conference & Exposition on Experimental and Applied Mechanics (http://sem.org/CONF-AC-TOP.asp)
- **June 25-26**: Nano Measure 2013—A symposium for sensing and understanding nano-scale phenomena, University of Warsaw, Poland, Abstract submission deadline: March 18, 2013 (www.nano-measure.com).

Thank you!