6. Potential Pitfalls

Andy Bushby

Difference between theory and practice

Potential pitfalls

- Non-ideal tip shape (not perfect sphere or pyramid)
- Thermal drift or mechanical instability
- Non-ideal surface (not perfectly smooth and flat)
- Non-ideal material response (creep, pile-up, etc.)
- Size effects (changes in material properties with scale)

Difference between theory and practice

Potential pitfalls

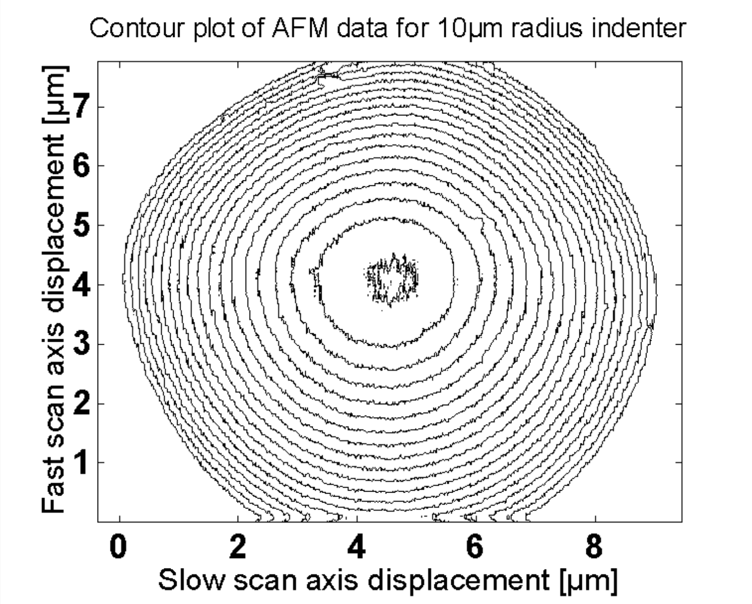
- **Non-ideal tip shape (not perfect sphere or pyramid)**
- Thermal drift or mechanical instability
- Non-ideal surface (not perfectly smooth and flat)
- Non-ideal material response (creep, pile-up, etc.)
- Size effects (changes in material properties with scale)

Non-ideal tip shape:

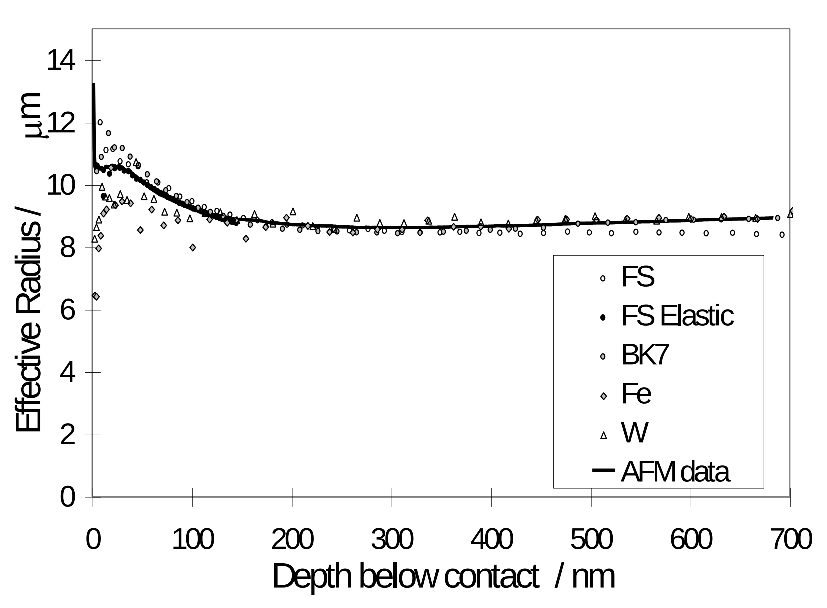
Contact mechanics relies on a knowledge of the indenter tip shape

Indentation Golden Rule #1. – you **MUST** know your tip shape!
Calibrate your indenter tip shape !!!

Direct method – AFM



Indirect method – reference materials

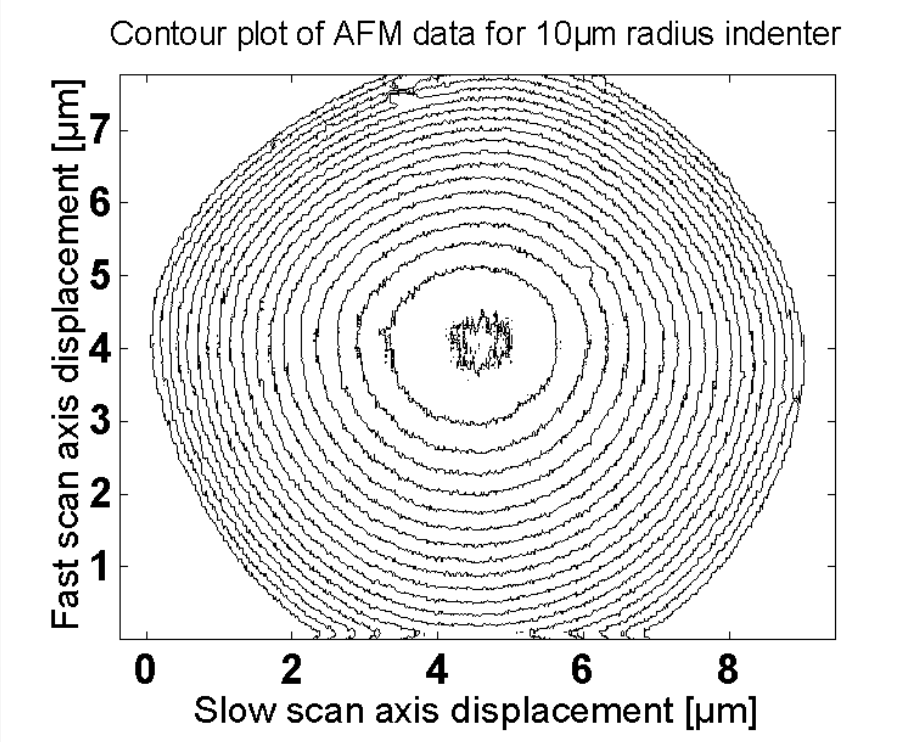


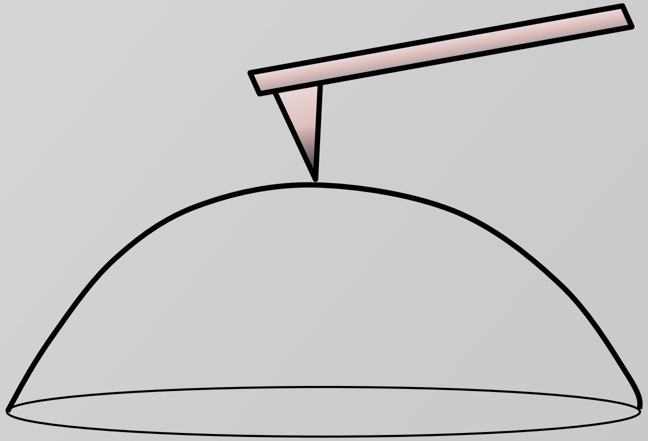
Indenter tip shape calibration

Metrological AFM to measure shape directly

3D image of indenter tip

Area at know distance from the top





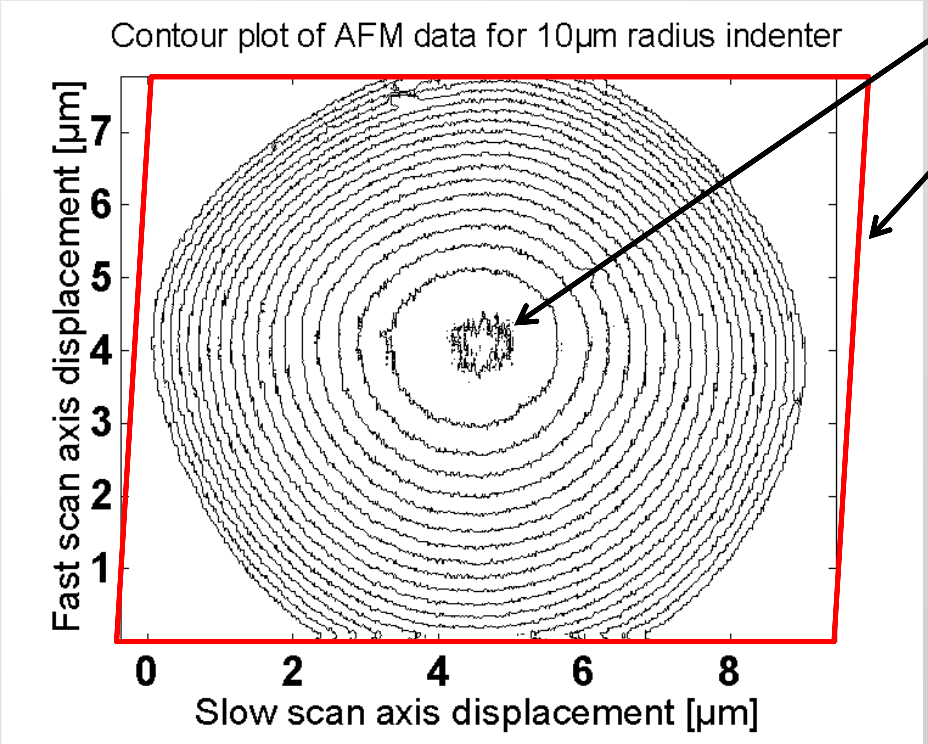
alemnis

Queen Mary University of London

Metrological AFM to measure shape directly

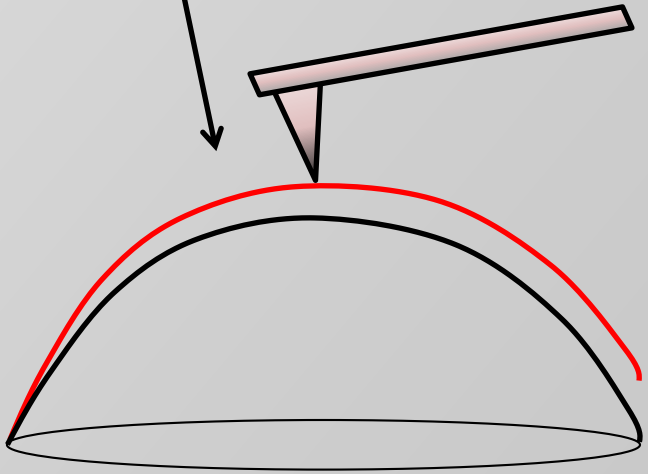
3D image of indenter tip

Area at know distance from the top



Problems:

- Where is the top?
- Slow scan drift
- Z drift



Indenter tip shape calibration

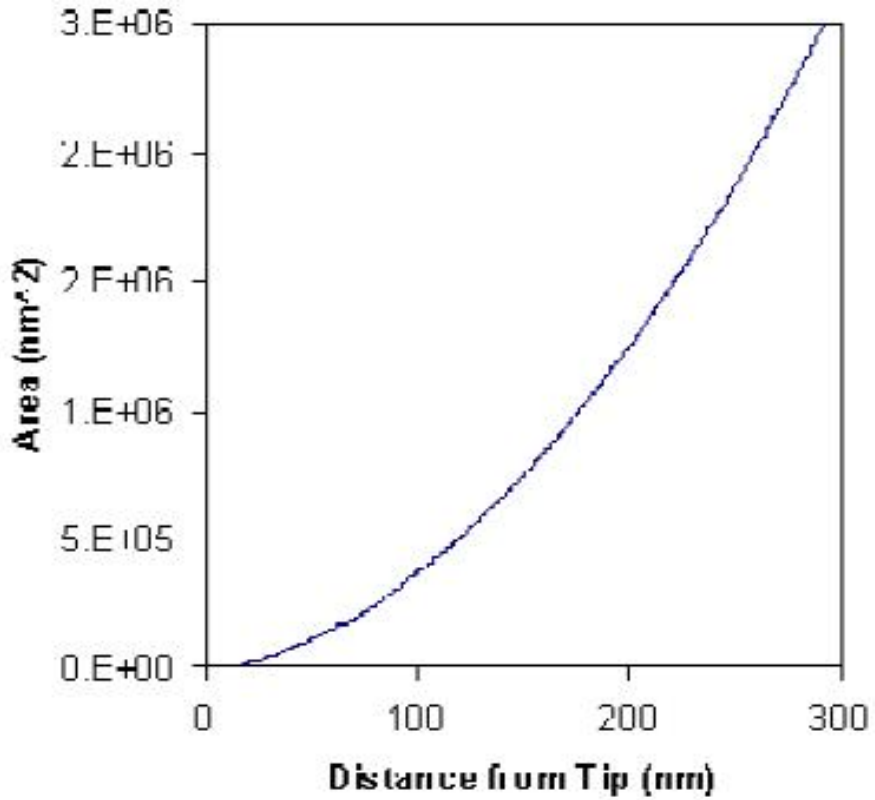
alemnis

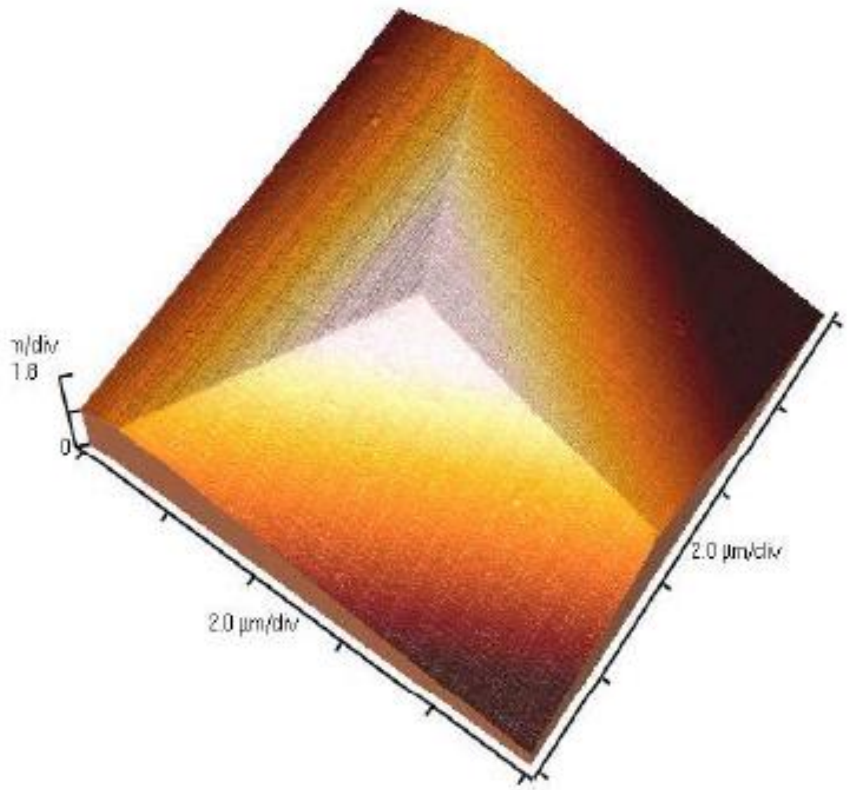
Queen Mary
University of London

Non-ideal tip shape: Calibration: Direct measurement with AFM

Berkovich 3 sided pyramid

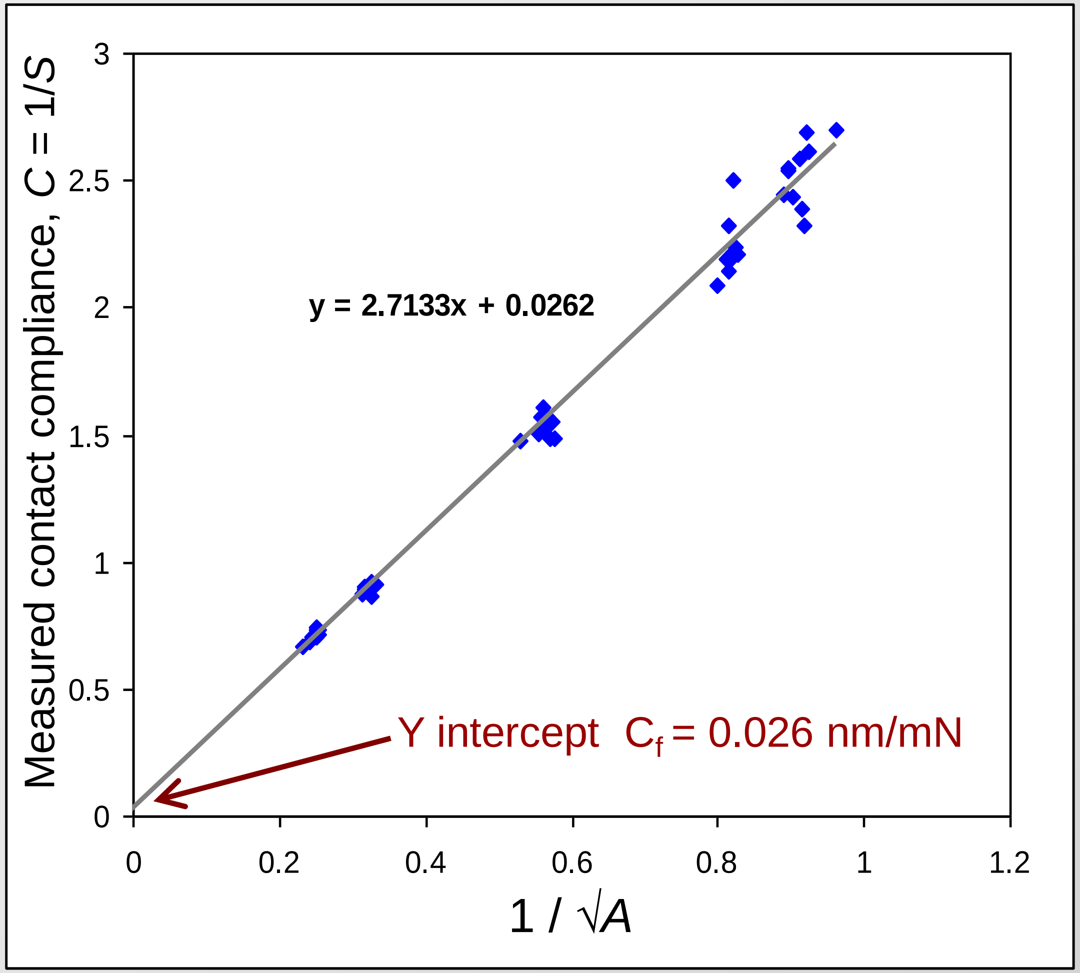
For ideal Berkovich indenter
Area = $A = 24.56\, h_c^2$





Indenter tip shape calibration

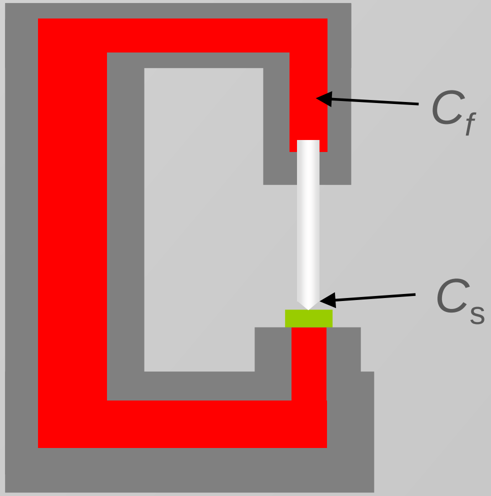
Indirect method – contact compliance



The **measured contact compliance** ($1/S$) is the sum of the contact compliance between indenter and sample, C_s, and the frame compliance, C_f

$$C = C_f + C_s$$

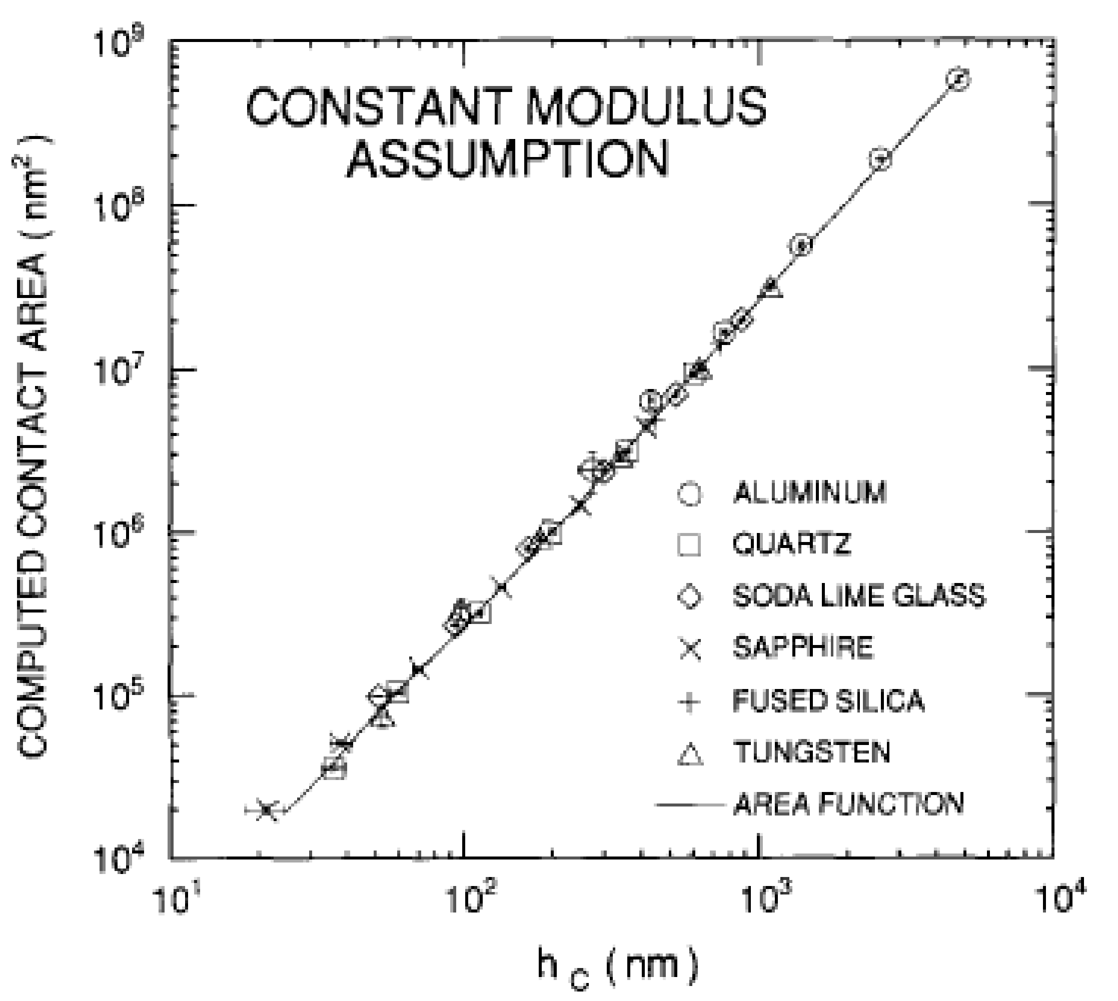
$$C = C_f + \frac{\sqrt{\pi}}{2E_r} \cdot \frac{1}{\sqrt{A}}$$



A plot of C vs $A^{-1/2}$ should be linear (constant E with depth) and has C_f as the intercept on the y-axis

However, this assumes you already know A vs h_c

Indirect method – contact compliance



$$A = 24.5h_c^2$$

$$A = \frac{\pi}{4}\frac{1}{E^{*2}}\frac{1}{\left(C - C_f\right)^2}$$

*E** and C_f are estimates

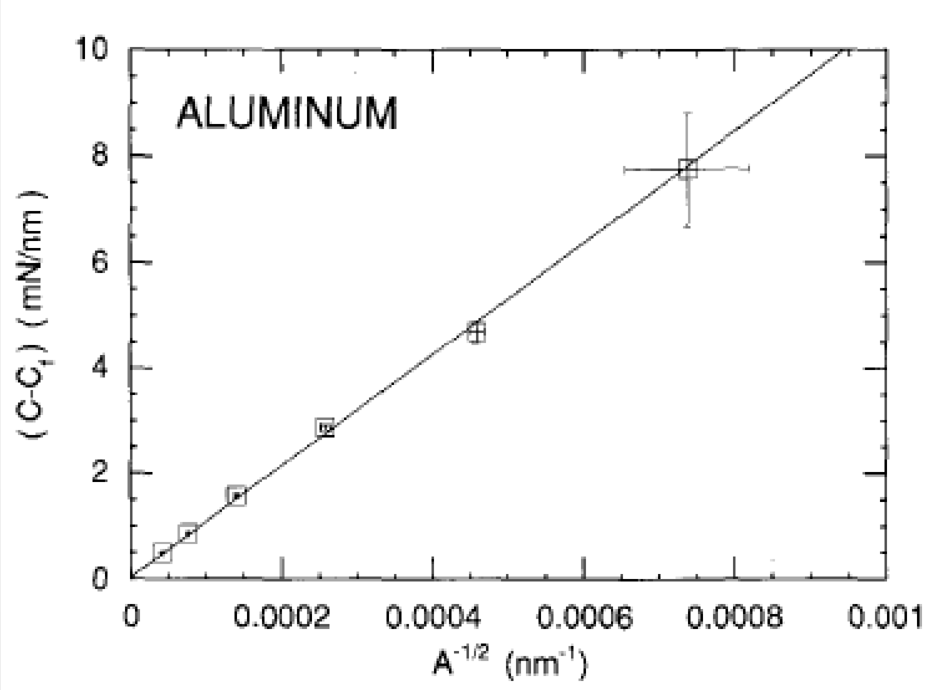
A vs h_c is plotted and fitted with: -

$$A = 24.5h_c^2 + C_1 h_c^1 + C_2 h_c^{1/2} + C_3 h_c^{1/4} + \ldots\ldots + C_8 h_c^{1/128}$$

The process is iterated to obtain better estimates of *E** and C_f

Indenter tip shape calibration

Indirect method – contact compliance



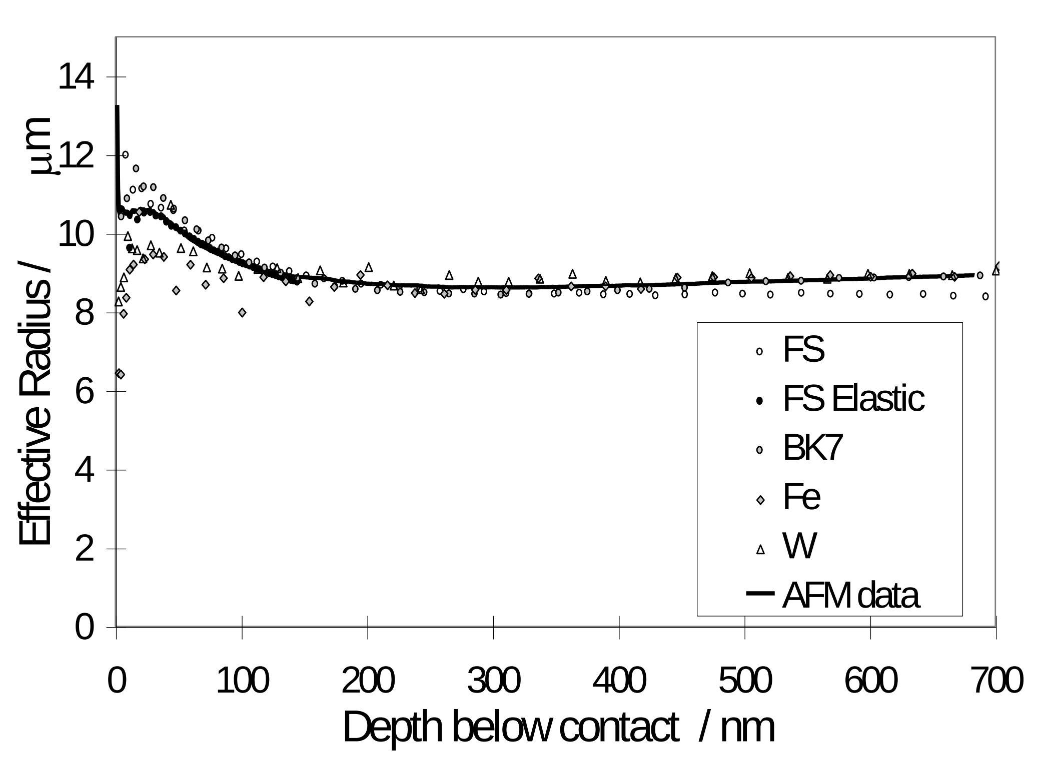
Finally, a plot of C-C_f vs $A^{-1/2}$ should be linear (constant E with depth) with a zero intercept on the y-axis

$$C - C_f = \frac{\sqrt{\pi}}{2E_r} \cdot \frac{1}{\sqrt{A}}$$

This should be done for several reference materials – NOT JUST ONE

Multiple reference material method

Indent into (**several**) materials with known elastic moduli
Using only one reference material is not good enough



Chose materials:

- Wide range of modulus
- High hardness
- Homogeneous and isotropic

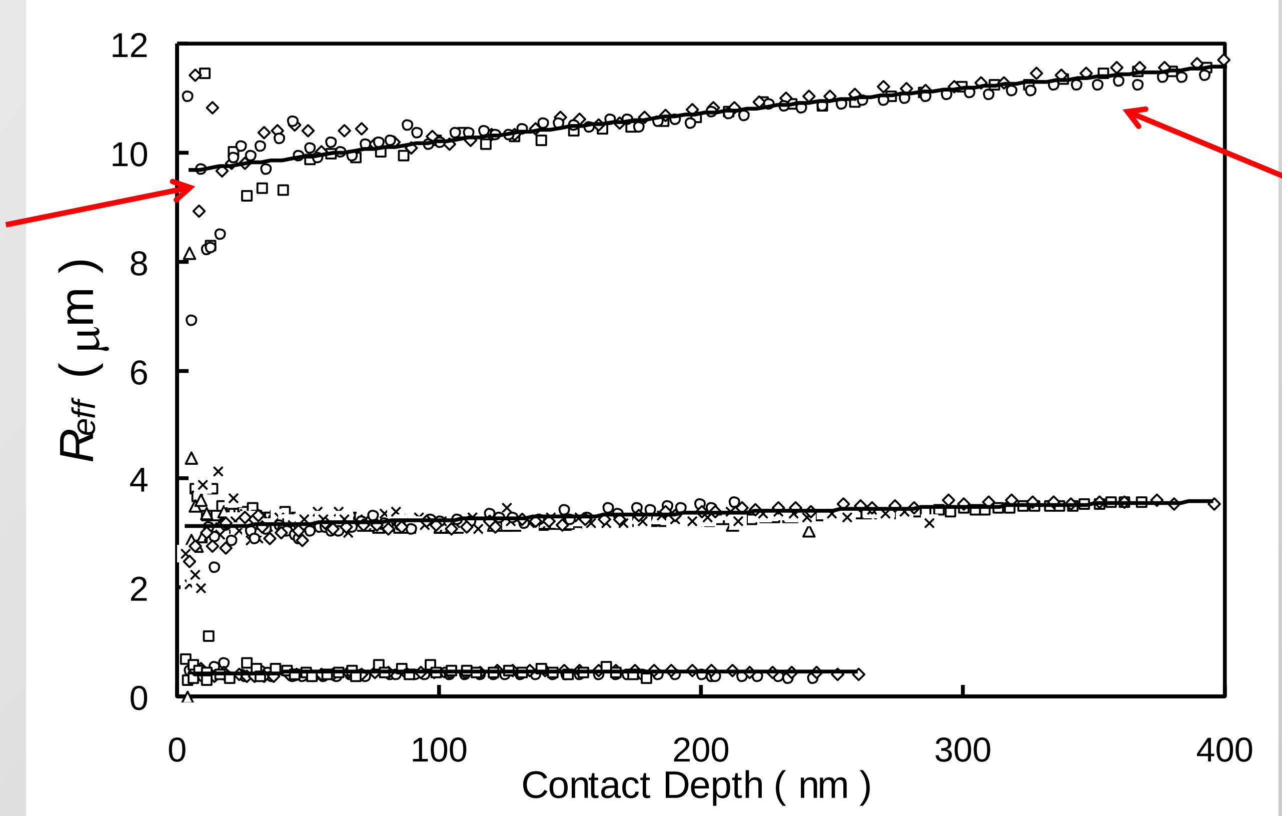
- Hertz equation, solve for *R*

$$R^* = \left(\frac{9}{16} \frac{F^2}{E^{*2}} \frac{1}{h_e^{\,3}} \right)$$

Indenter tip shape calibration

Non-ideal tip shape: Calibration: effective radius vs depth of contact

Low loads:
sensitive to material &
indenter modulus *E**



High loads:
sensitive to
frame stiffness C_f

*E** = 36.8 GPa *E** = 70.5 GPa *E** 150 GPa *E** = 300 GPa

□Glassy Carbon, ◊Fused Silica ○ Si (001) × Tungsten,

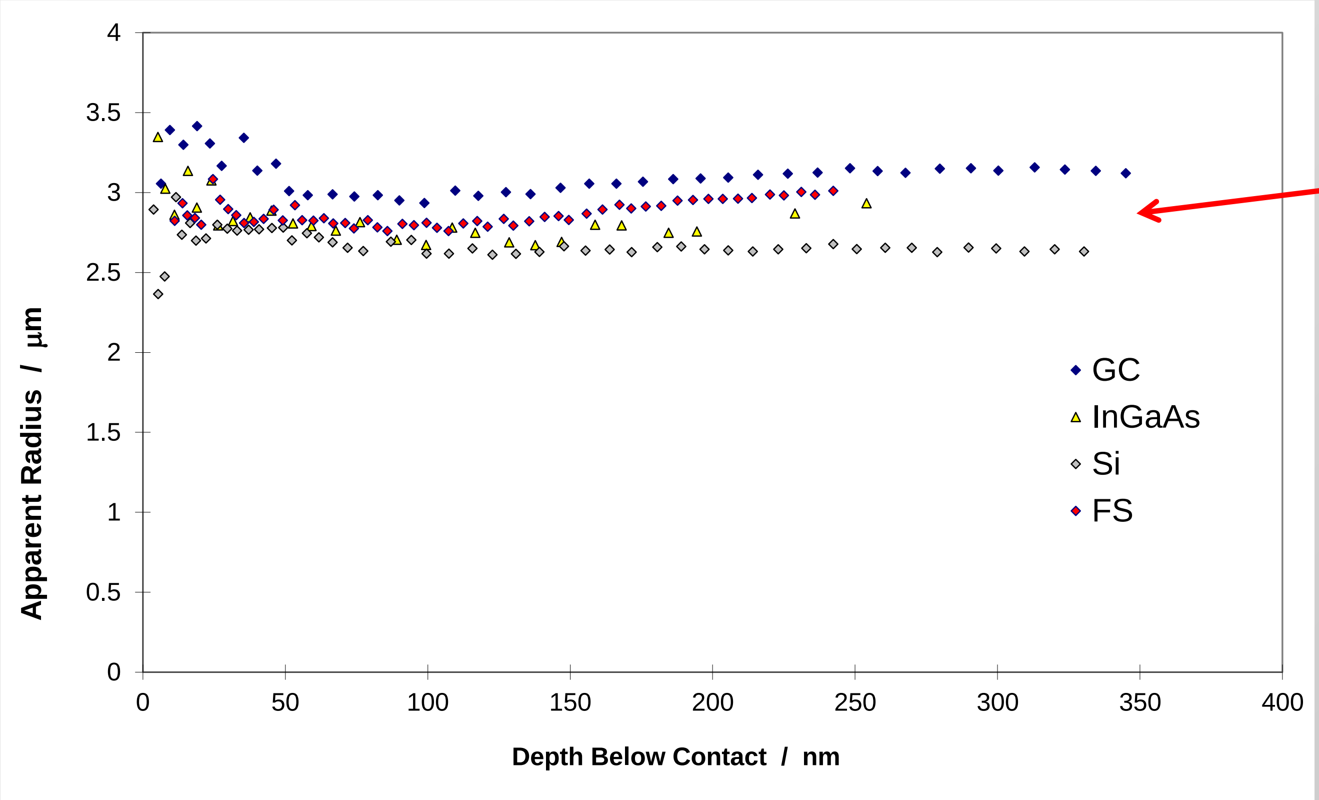
R_{eff} is a function of h_c, and C_f=0.24mm/mN.

Indenter tip shape calibration

alemnis

Queen Mary University of London

Non-ideal tip shape: Calibration: effective radius vs depth of contact



High loads: sensitive to frame stiffness C_f

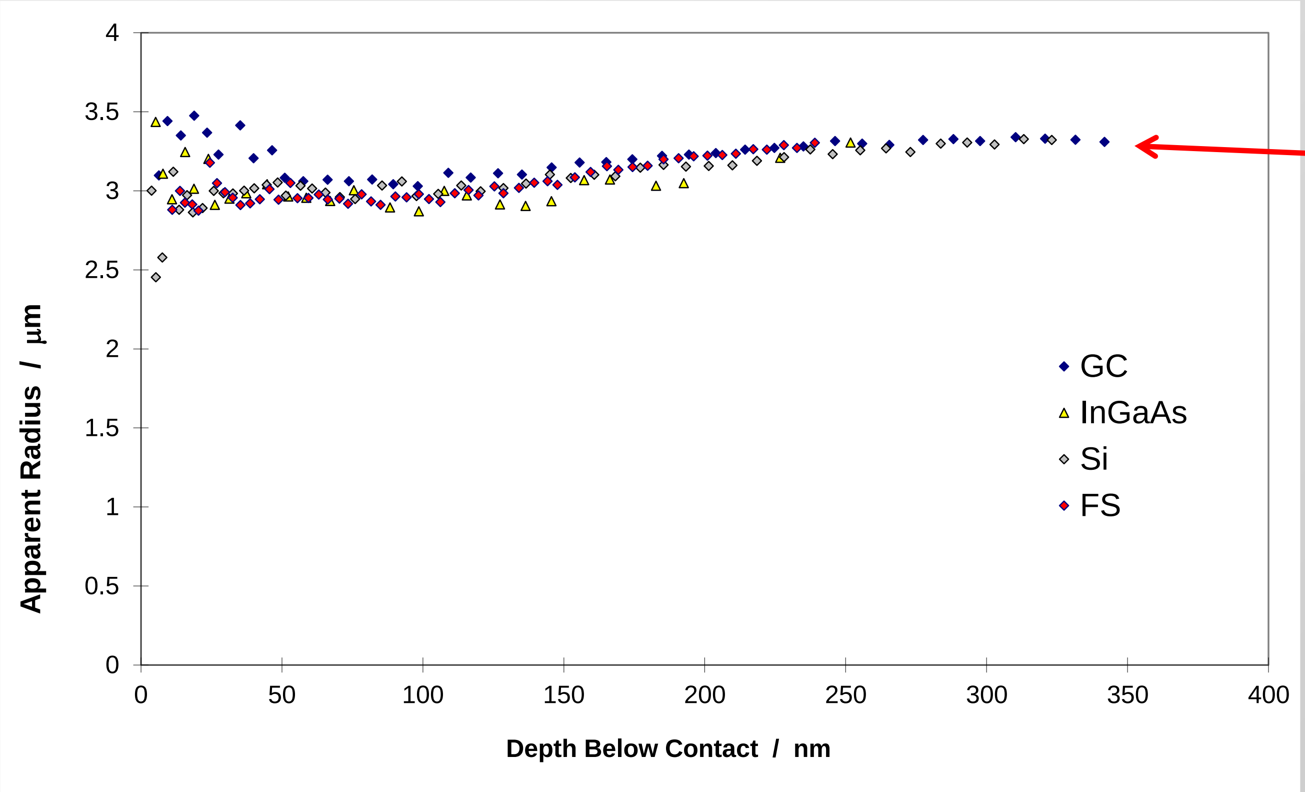
Wrong frame compliance value

Indenter tip shape calibration

alemnis

Queen Mary University of London

Non-ideal tip shape: Calibration: effective radius vs depth of contact



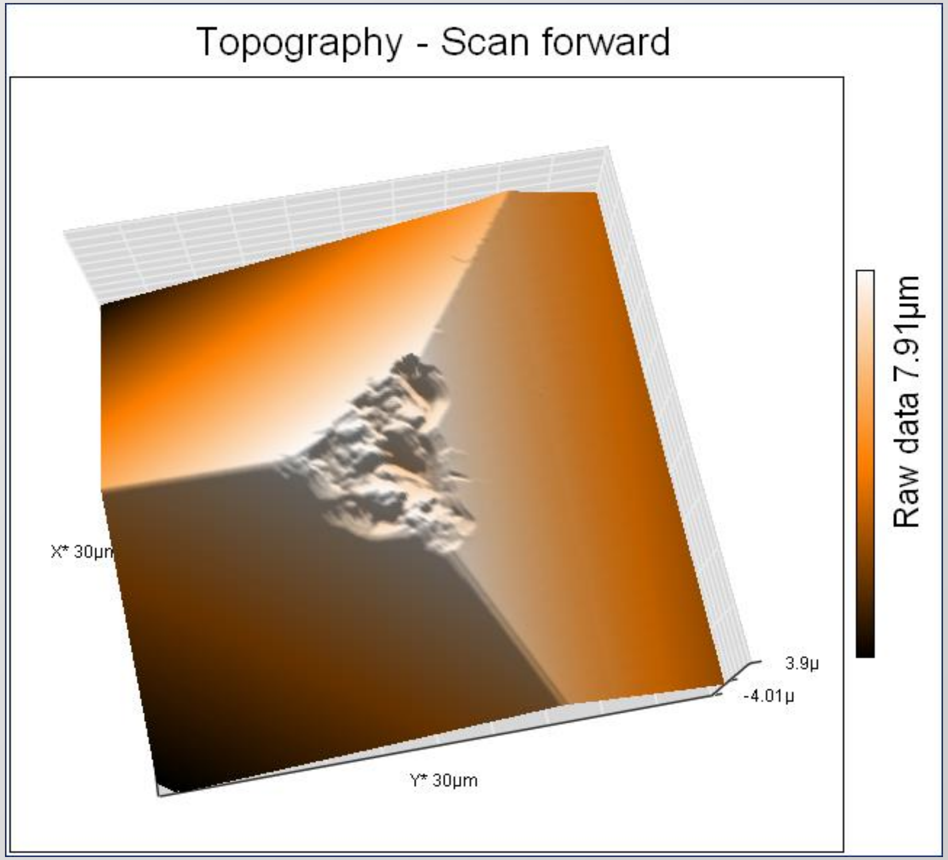
High loads: sensitive to frame stiffness C_f

Correct frame compliance value

Indenter tip shape calibration

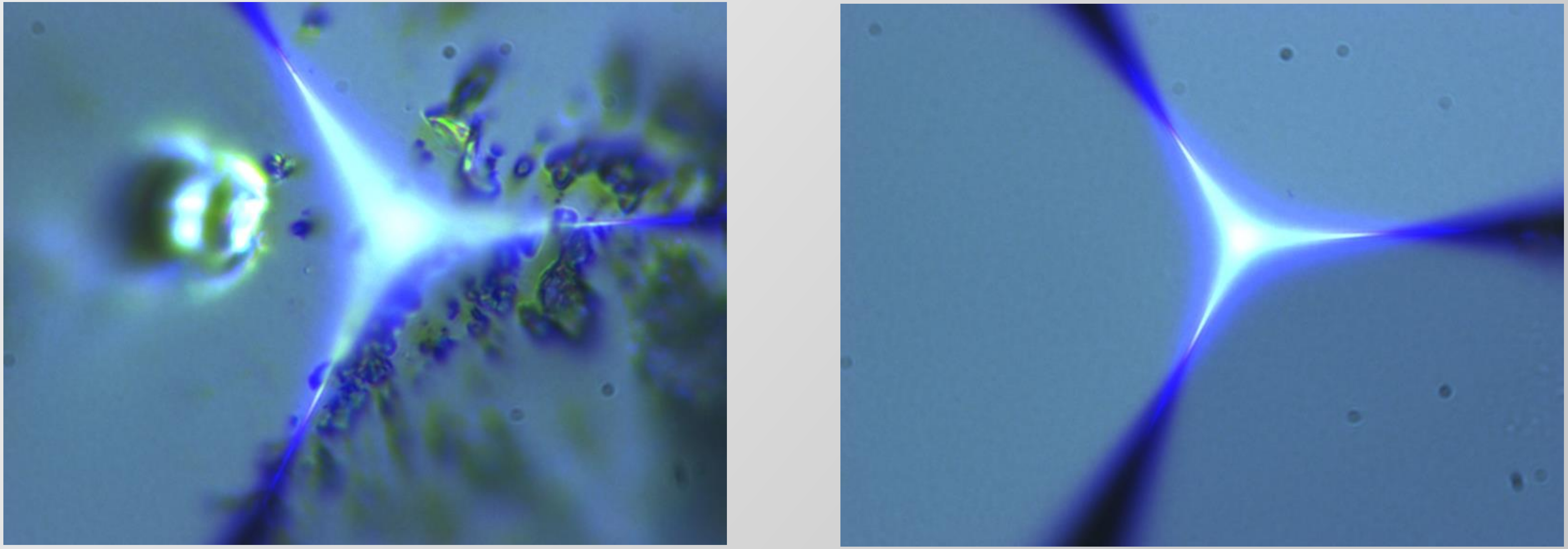
Non-ideal tip shape: Example of damaged tip

Berkovich 3 sided pyramid



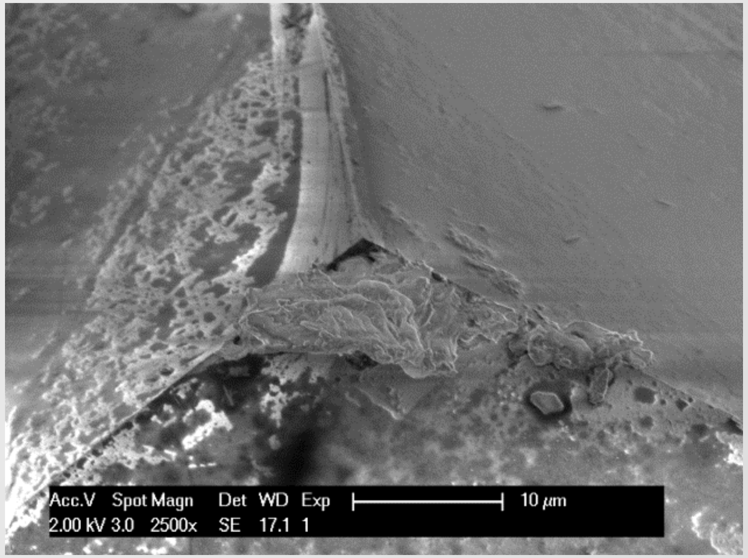
Berkovich indenter before & after cleaning

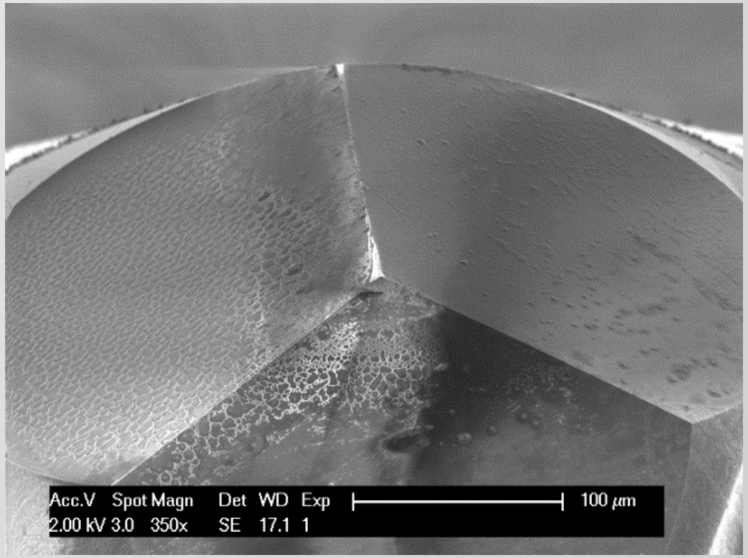
Cleaning with isopropanol, compressed air

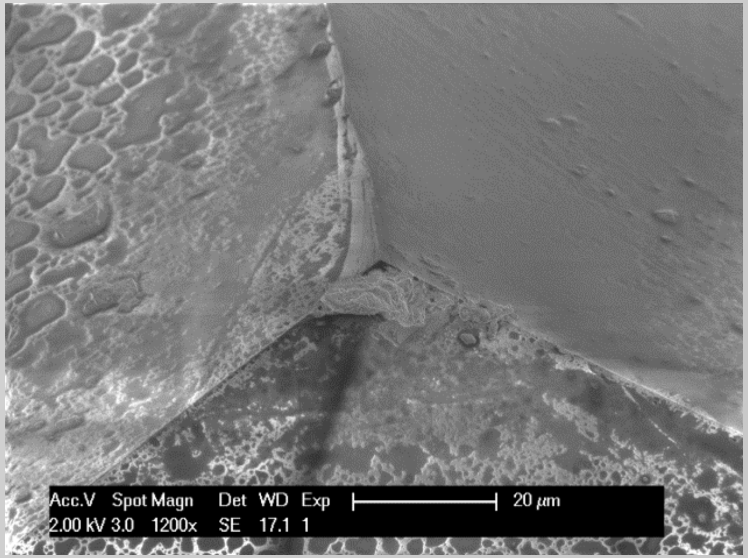


Berkovich indenter before & after cleaning

Cleaning with isopropanol, compressed air



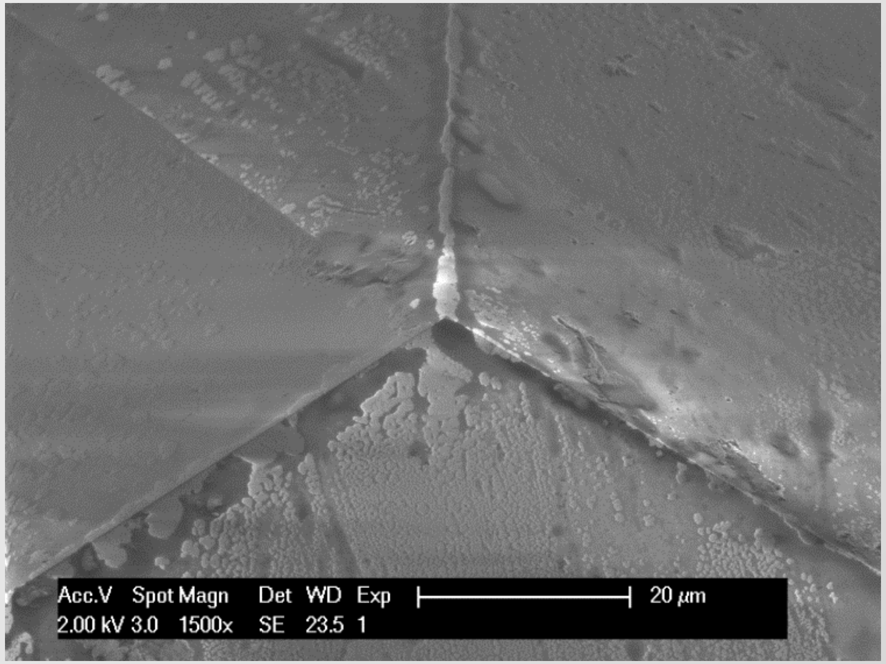


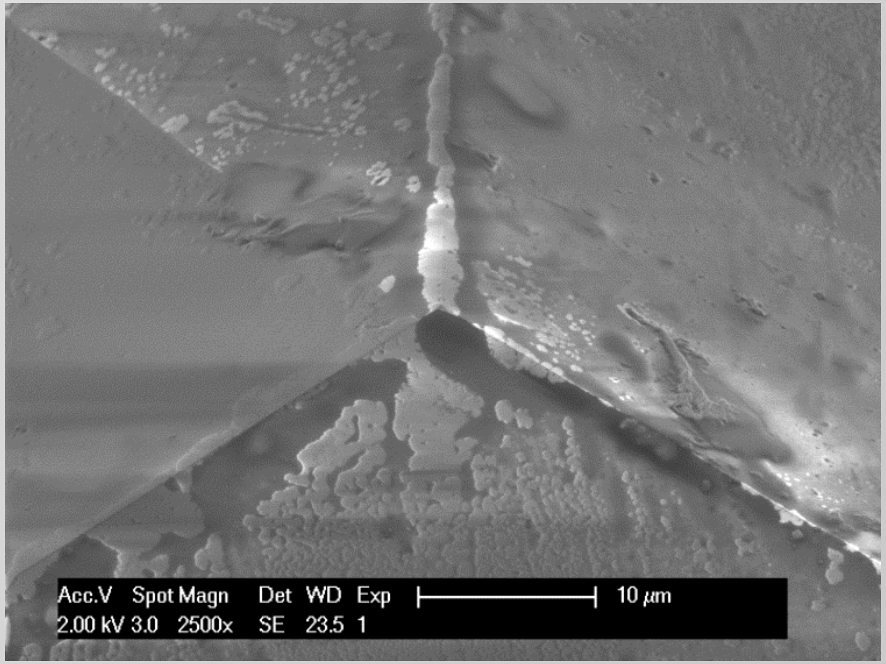


Berkovich indenter before & after cleaning

Cleaning with isopropanol, compressed air…. but still some residue!

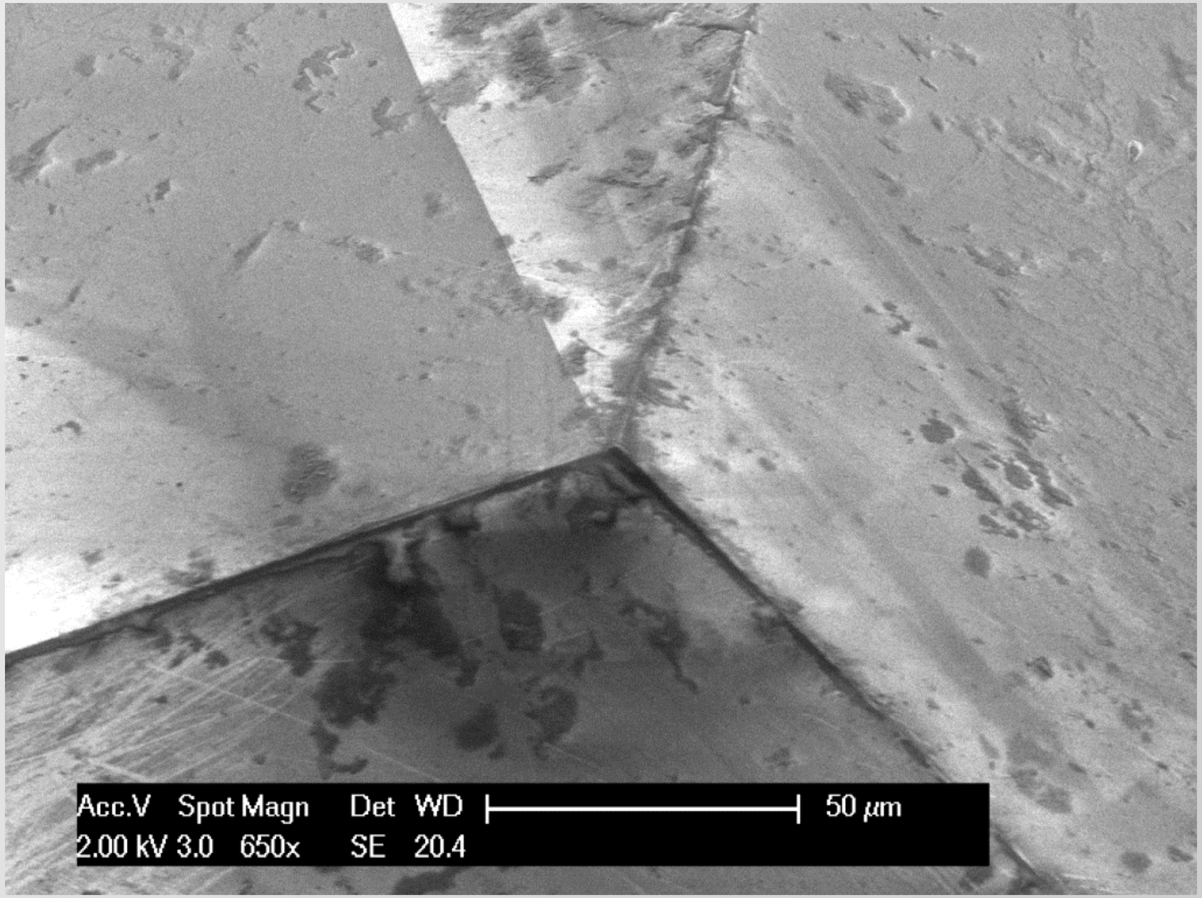
Is the compressed air source clean? i.e., no oil from the compressor?





Berkovich indenter before & after cleaning

More cleaning with isopropanol, compressed air…. now better:



Berkovich indenter cleaning procedure

alemnis

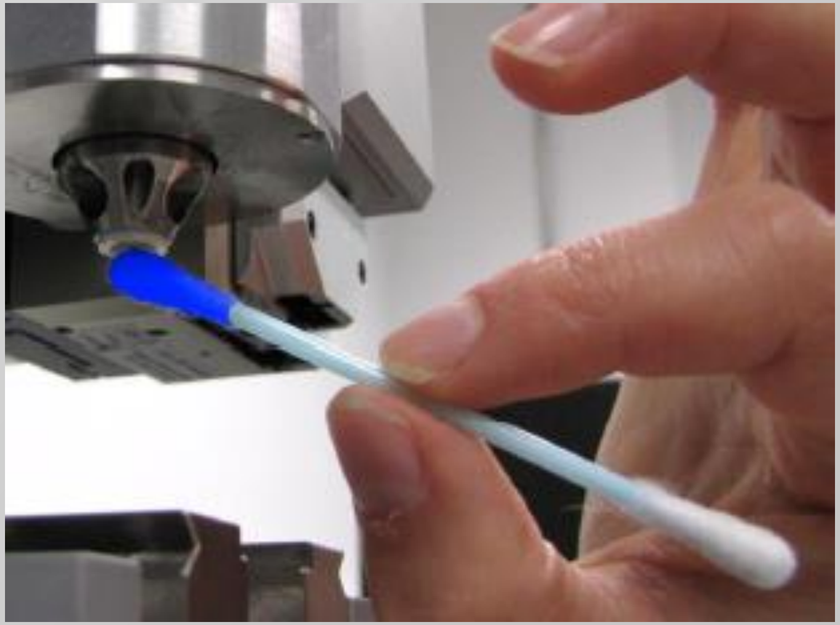
Queen Mary University of London

If indenter seems contaminated, proceed as follows:

1. Looking at the apex through an optical microscope, gently rub the diamond with a cotton bud soaked in isopropanol

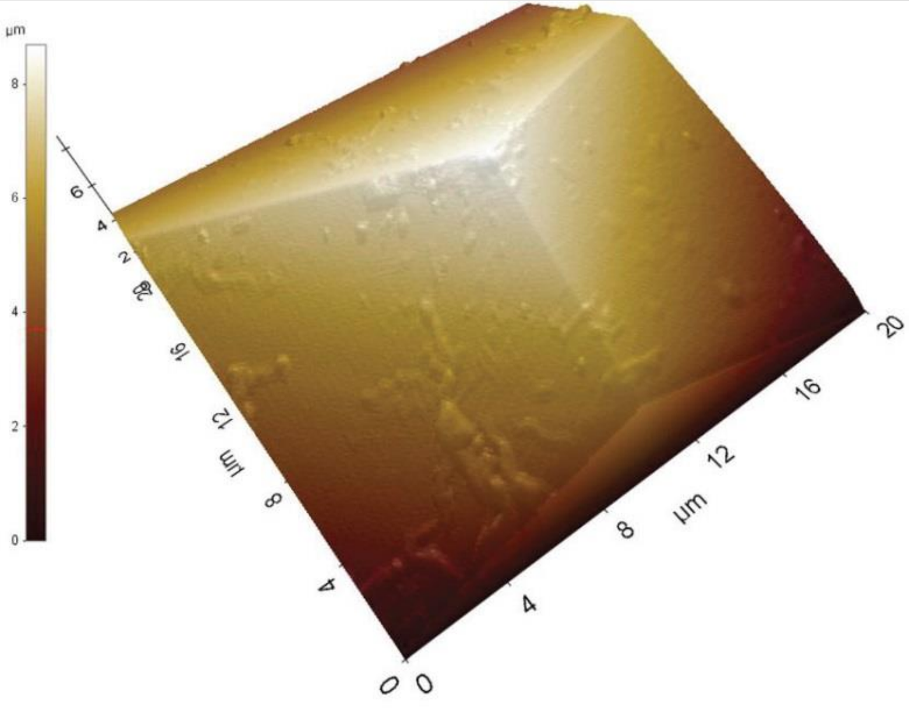
2. Turn the cotton bud around and use the dry end to wipe off any excess solvent. Then blow off immediately with compressed air.

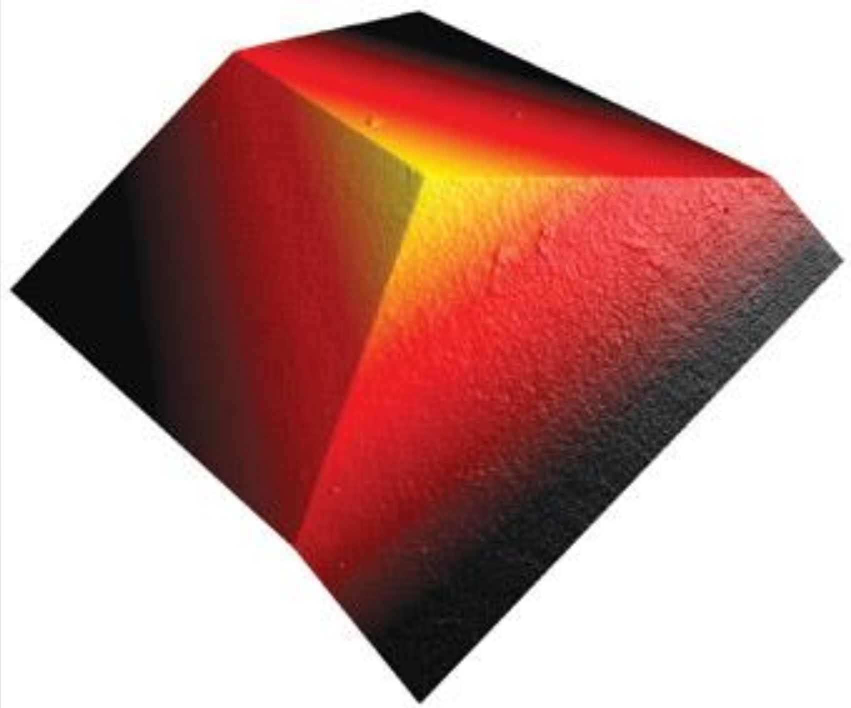
3. IMPORTANT: Do not use ultrasonic cleaning machines as this technique may loosen the braize holding the indenter



Berkovich indenter observation by AFM

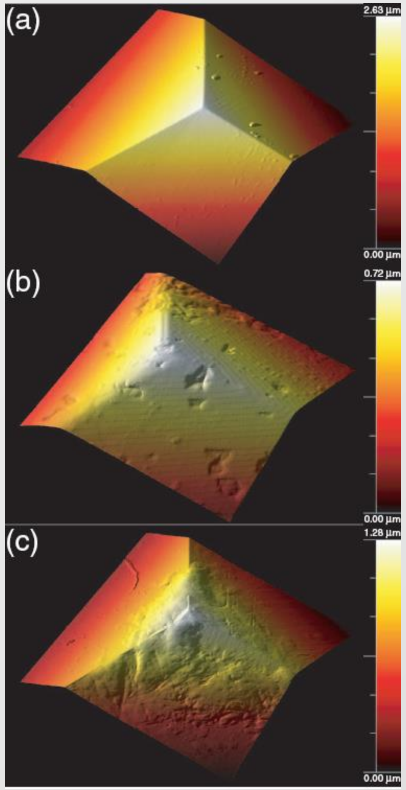
AFM can be used to check cleanliness and geometry (if calibrated)





Diamond indenter oxidation & reactivity

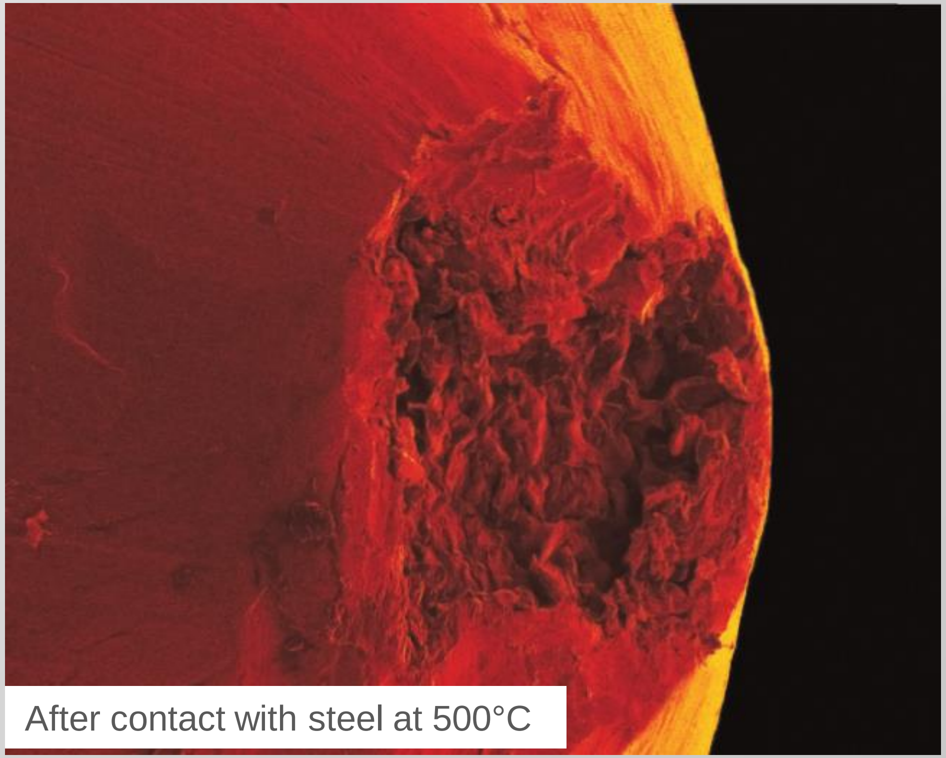
Diamond oxidises > 400°C and can react chemically with C-containing materials



Undamaged after regular use < 400°C

After 90 min. at 900°C in argon

After contact with Al in air at 300°C

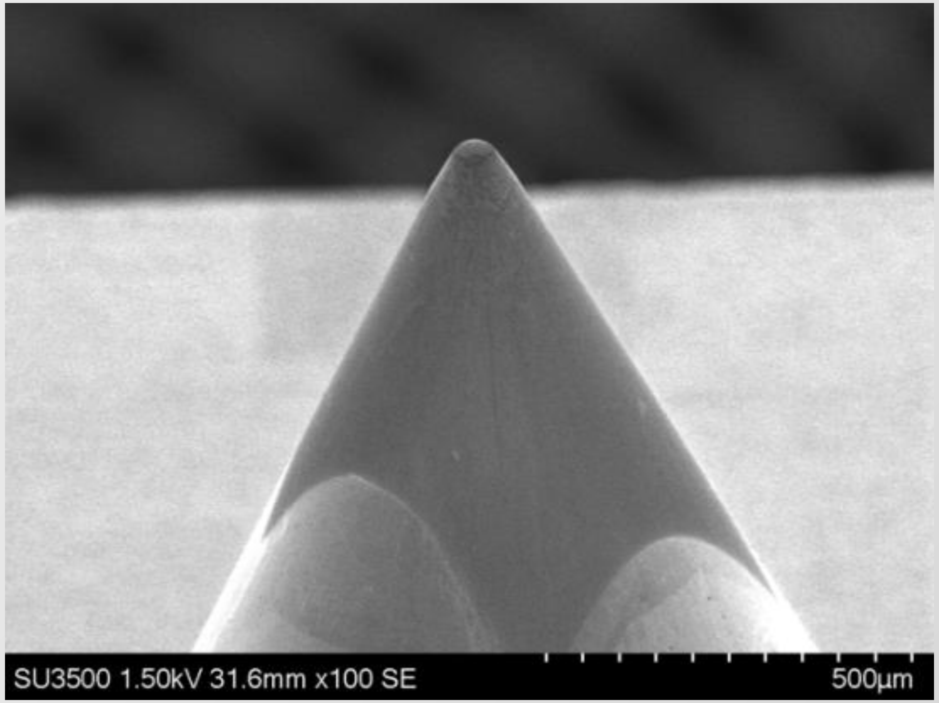


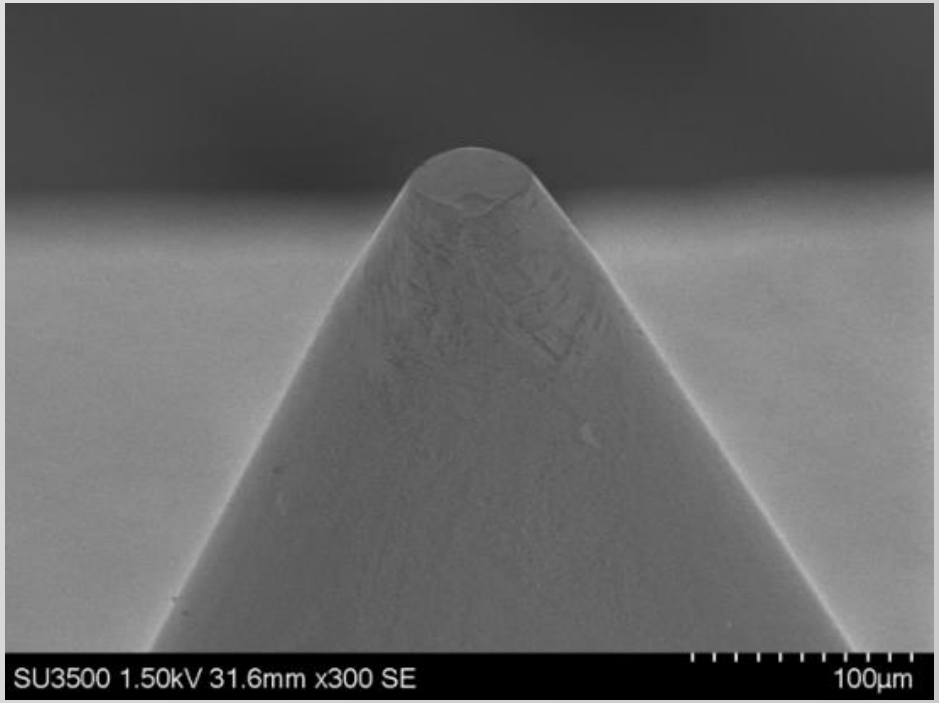
After contact with steel at 500°C

J. M. Wheeler & J. Michler, Indenter materials for high temperature nanoindentation, Rev. Sci. Instr , 84 (2013) 101301

How good is your flat punch indenter..?

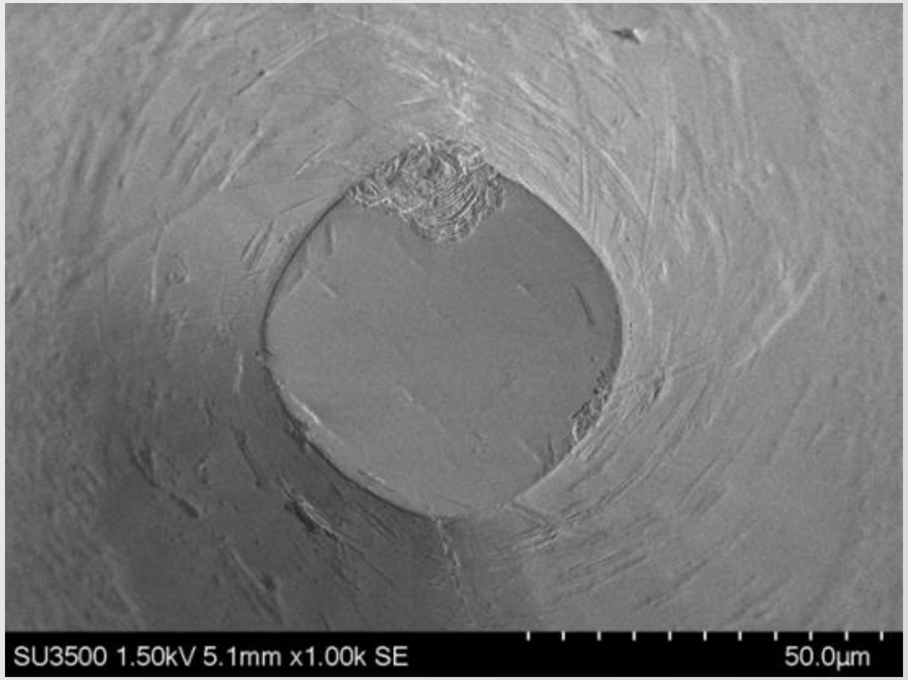
Is the reality what the manufacturer specified?
Taper angle, sphericity, polish, etc…

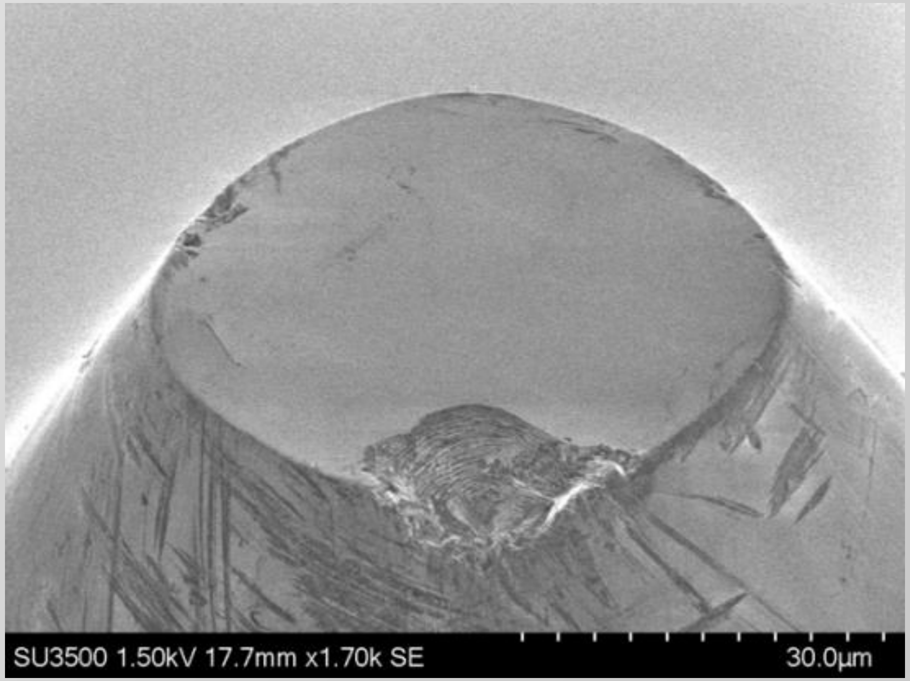




How good is your flat punch indenter..?

This indenter was supposed to be 50 µm diameter but is actually significantly less.
The spherical part is non-perfect and has a bad defect!





Difference between theory and practice

alemnis

Queen Mary
University of London

Potential pitfalls

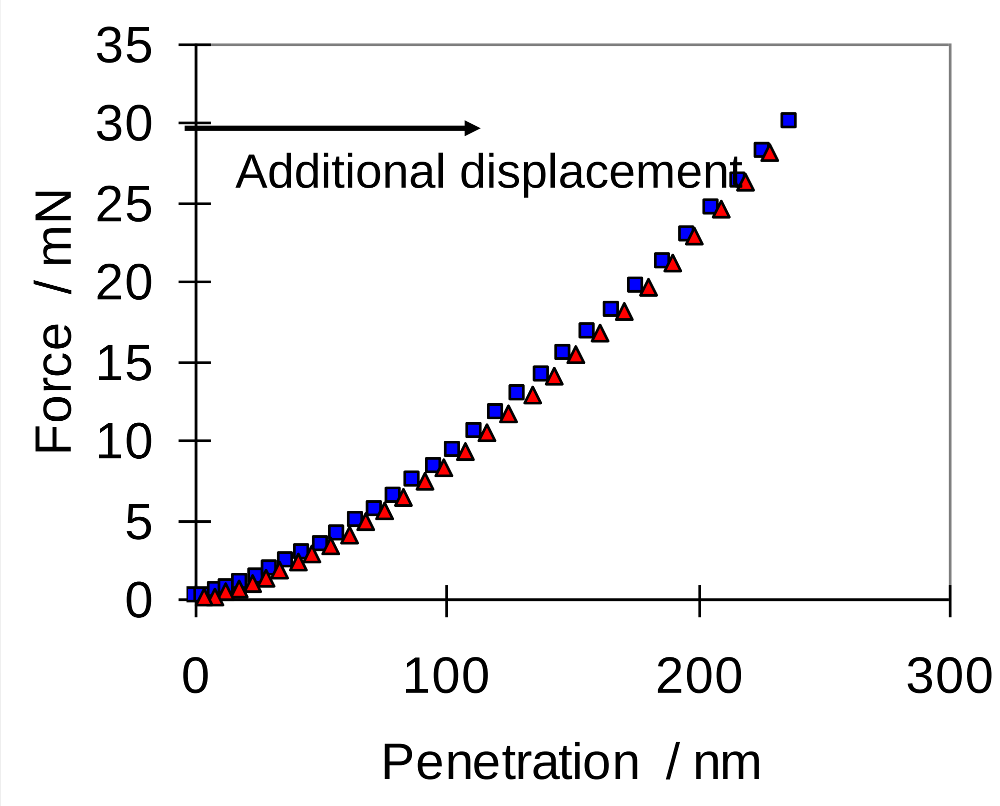
- Non-ideal tip shape (not perfect sphere or pyramid)
- **Thermal drift or mechanical instability**
- Non-ideal surface (not perfectly smooth and flat)
- Non-ideal material response (creep, pile-up, etc.)
- Size effects (changes in material properties with scale)

Instrument frame compliance

Indentation Golden Rule #2 – you **MUST** know your instrument characteristics

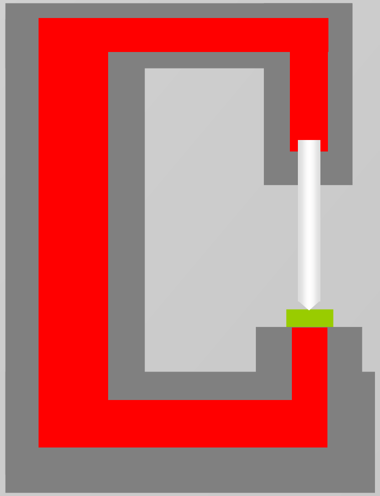
Frame compliance:

When the surface is loaded there is a reaction force in the instrument frame that causes a deflection proportional to load, making the material appear to be more compliant (less stiff)



$$h = C_f F + h_m$$

C_f is a constant ~ 0.2nm/mN



alemnis

Queen Mary
University of London

Instrument stability – thermal drift:

Adds to displacement measurement as a function of time

316 Stainless steel CTE 16μm/m K so 100mm shaft, for 0.01°C change in T = 16nm displacement

Measure a 5mm thick sample of Copper to depth of about 1000nm,
Copper CTE 16.6 μm/m K, 0.01 °C rise in T = 0.17nm change in thickness
Total thermal expansion = 16.2nm = error ~1.6%

Measure a 5mm thick sample of FS to depth of about 100nm,
FS CTE 5.9 μm/m K, 0.01 °C rise in T = 0.3nm change in thickness
Total thermal expansion = 16.3nm = error >16%

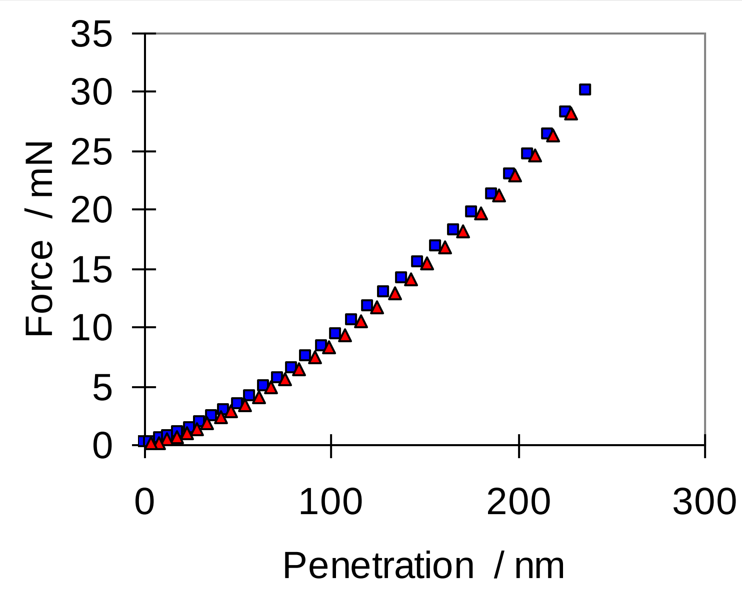
Measure a 10mm thick sample of PMMA to depth of about 100nm,
PMMA CTE 75 μm/m K, 0.01 °C rise in T = 7.5nm change in thickness
Total thermal expansion = 23.5nm = error >23%

(Reduce the measurement path to 1mm, expansion of machine <<< 1%)

Instrument stability – thermal drift:

Adds to displacement measurement as a function of time

$h_t = 220nm$
h_e = 220nm
h_c = 110nm

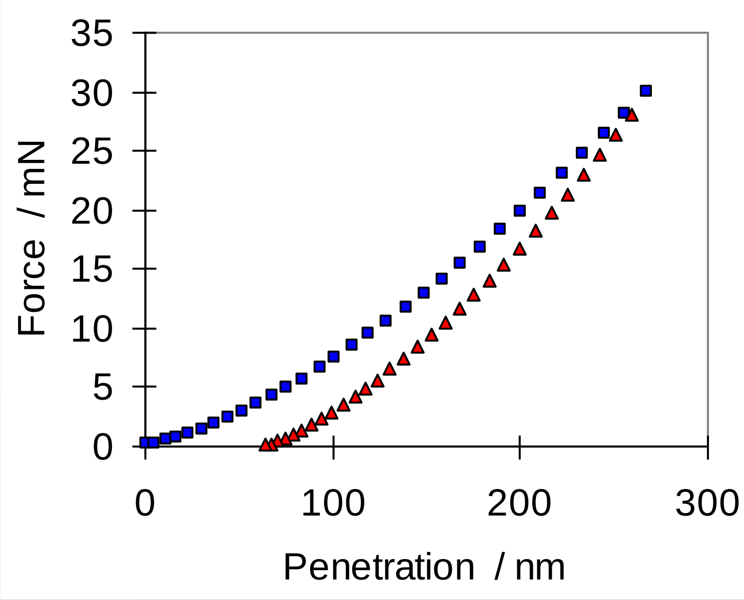


Elastic – no thermal drift

$h_t = 265nm$
h_e = 200nm
h_c = 165nm

E^* ↑
H ↓

Error in contact area $\propto h_c^2$



Elastic with thermal drift
~ 0.3nm / s

Difference between theory and practice

Potential pitfalls

- Non-ideal tip shape (not perfect sphere or pyramid)
- Thermal drift or mechanical instability
- **Non-ideal surface (not perfectly smooth and flat)**
- Non-ideal material response (creep, pile-up, etc.)
- Size effects (changes in material properties with scale)

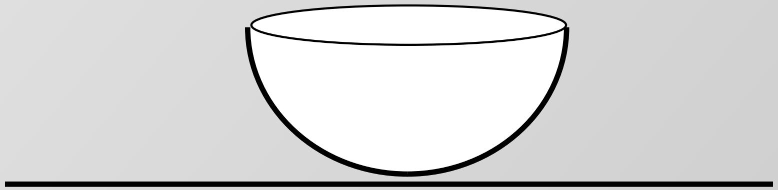
Surface contact

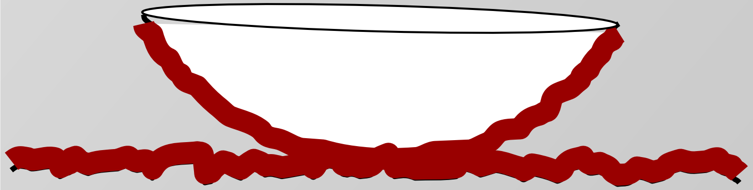
Indentation Golden Rule #3 – you **MUST** know your sample characteristics

Surface roughness:

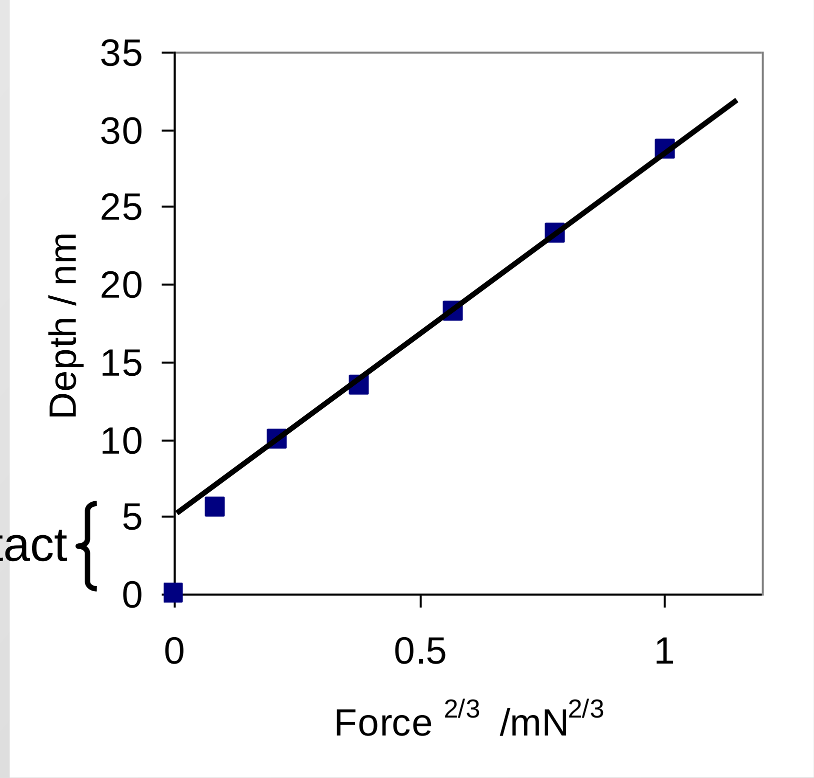
On indenter and test material

Determining surface contact difficult

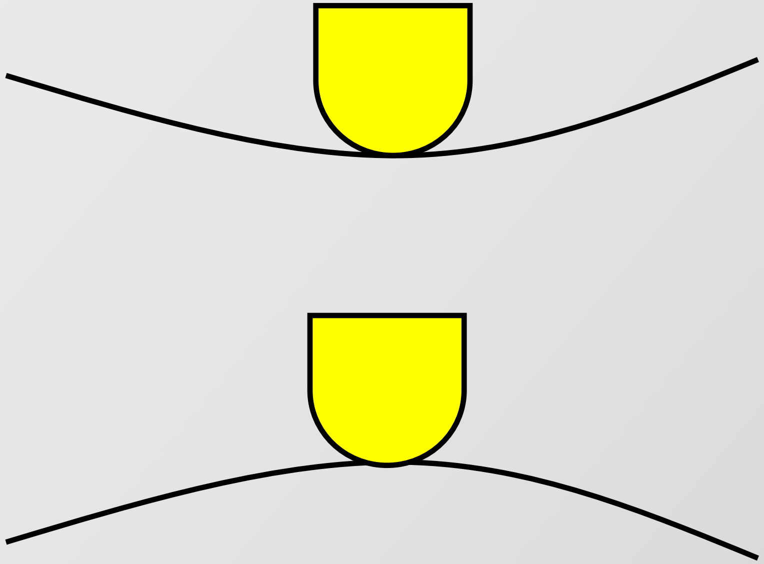


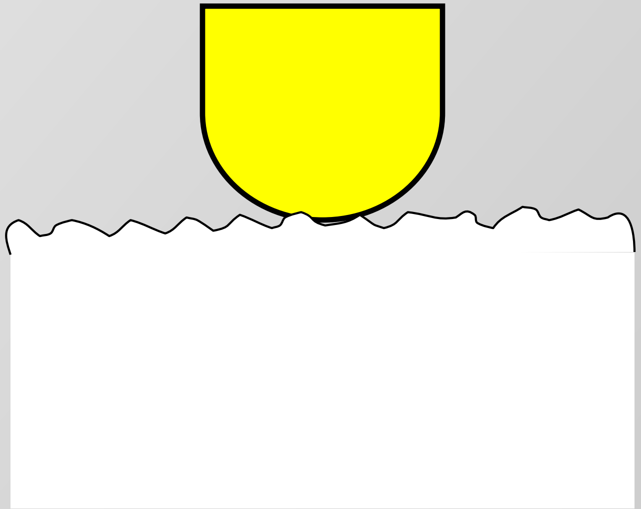


False contact



Roughness and curvature





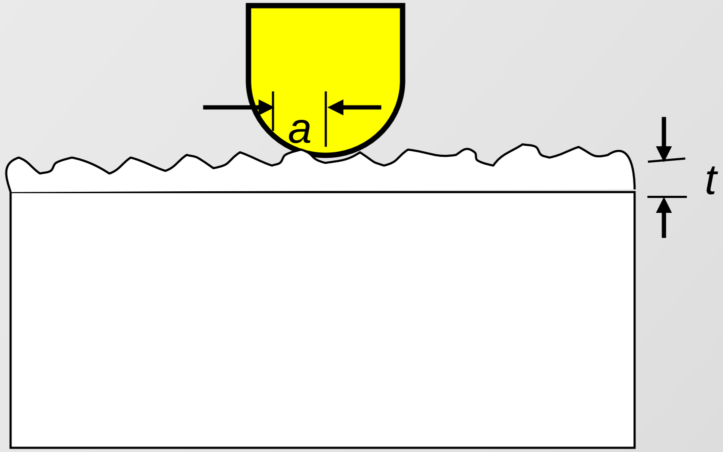
General curvature > 10*R*
< 5% error in *E*

General curvature > 100*R*
< 0.5% error in *E*

Surface roughness much shorter period than indenter tip radius: –

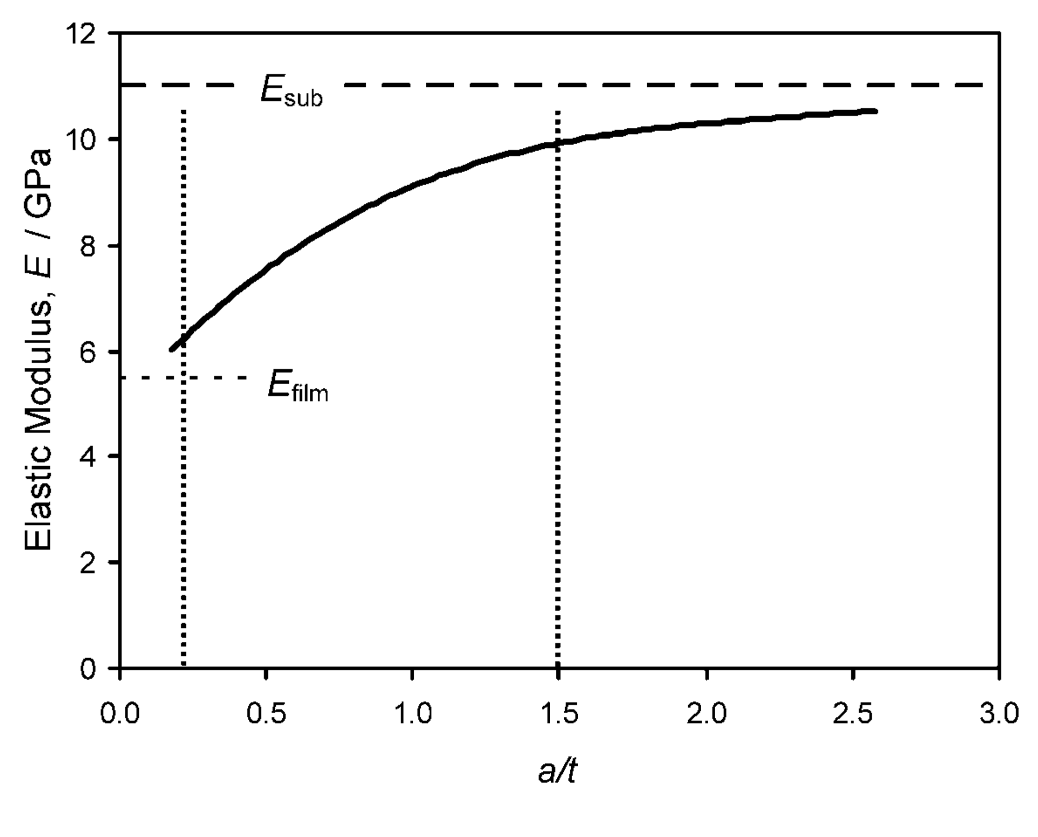
treat as layer on a substrate

Squashy layer model



Soft layer on the bulk

Continuously changing modulus with depth



At *h* < 2 x roughness the influence of roughness is significant

At *h* > 10 x roughness the influence of roughness is insignificant

Difference between theory and practice

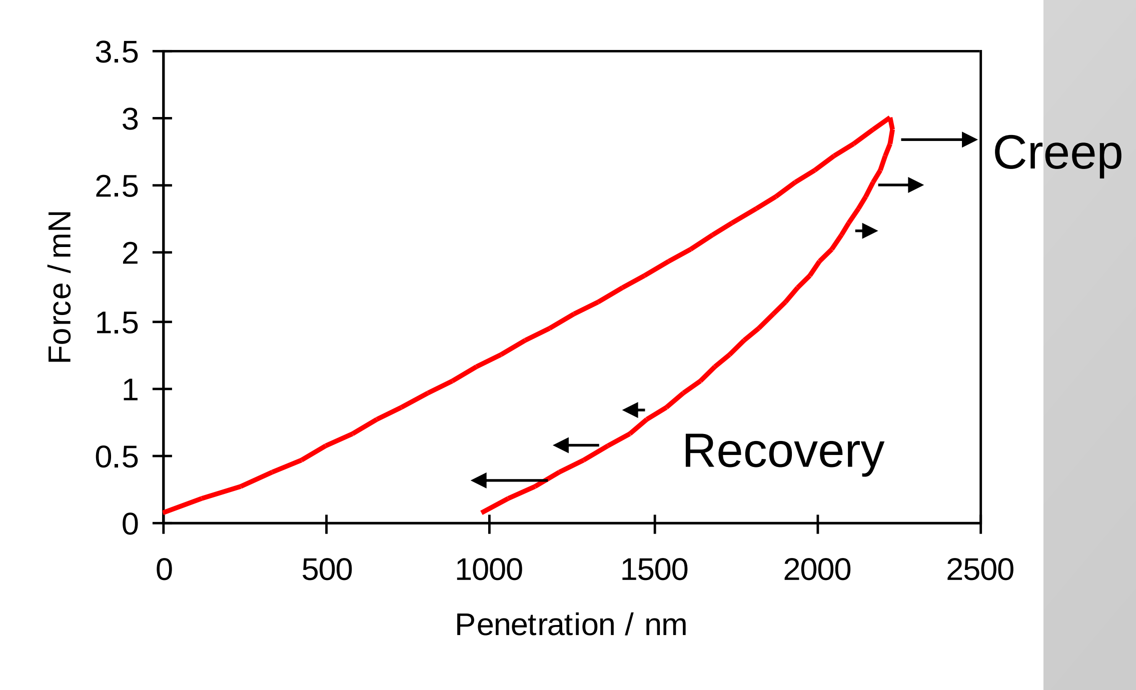
Potential pitfalls

- Non-ideal tip shape (not perfect sphere or pyramid)
- Thermal drift or mechanical instability
- Non-ideal surface (not perfectly smooth and flat)
- **Non-ideal material response (creep, pile-up, etc.)**
- Size effects (changes in material properties with scale)

Creep – the 'nose effect'

Creep:

Can distort unloading slope and the fitting of the unload curve
Over estimating the gradient – overestimating *E* and underestimating *H*

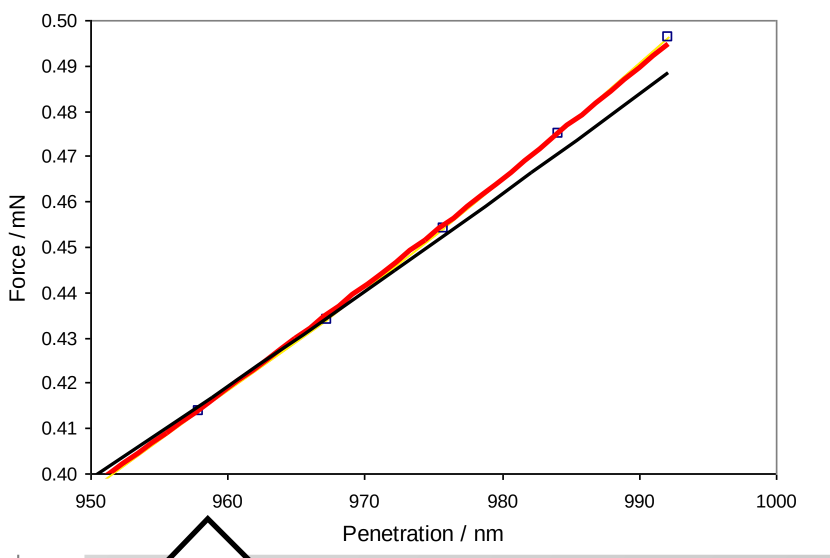


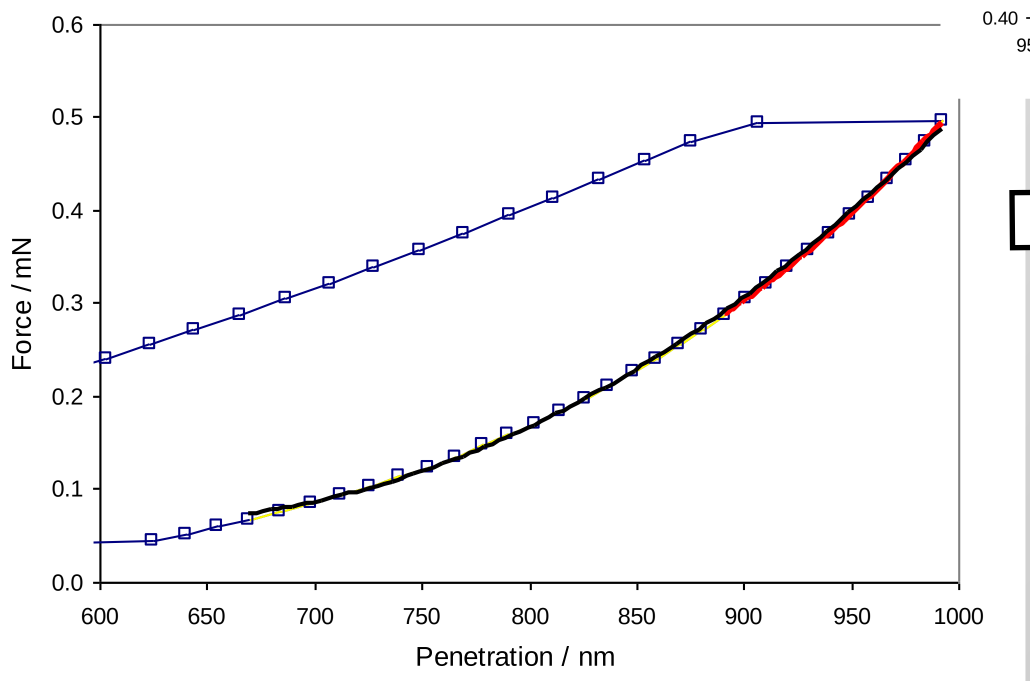
Creep during unloading

Curve fitting errors

Fit to unload curve:
Poor fitting can distort unloading slope

A regression fits best in the middle and worst at the ends



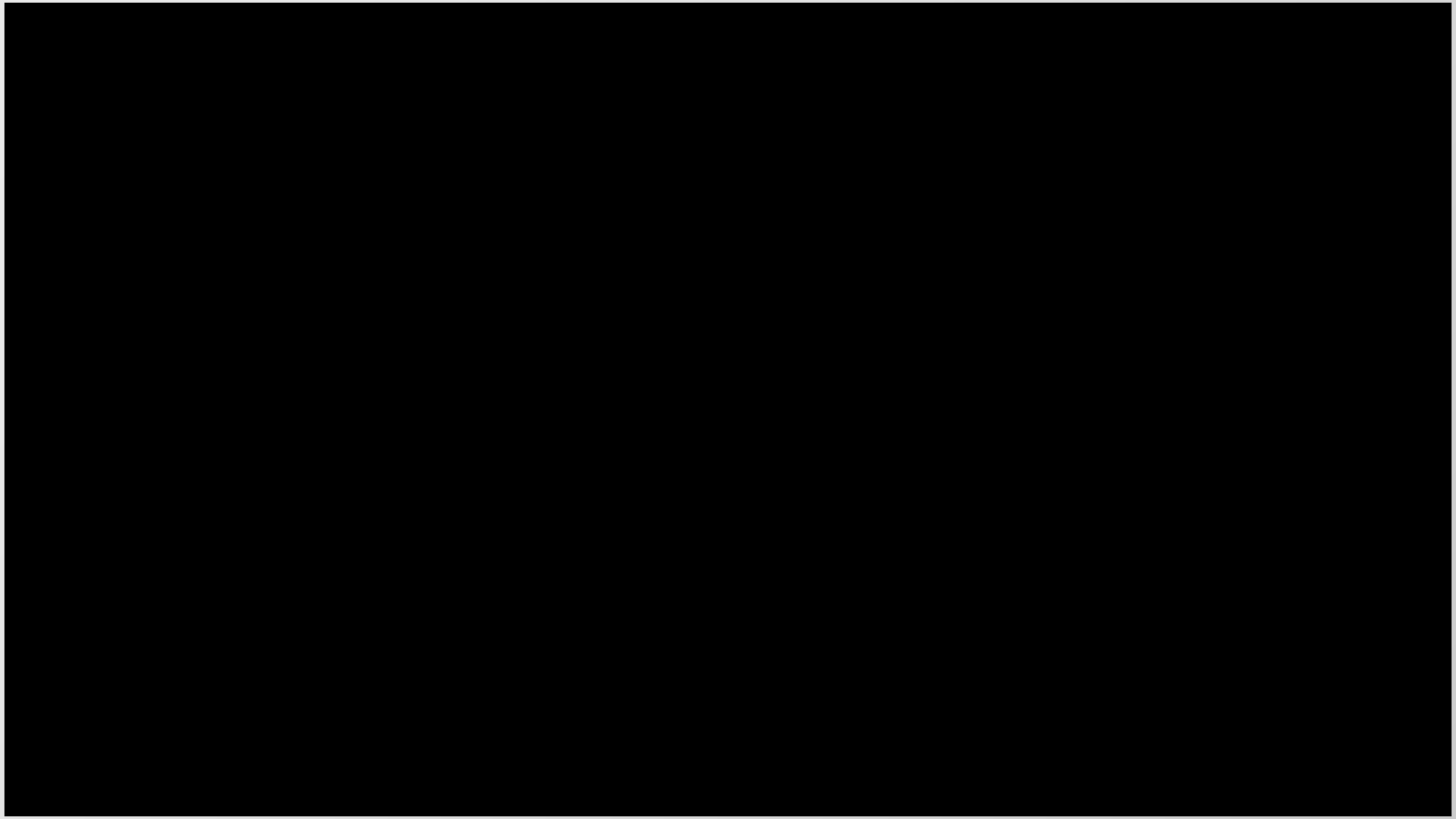


Fitting the unloading data can make a big difference to calculated values

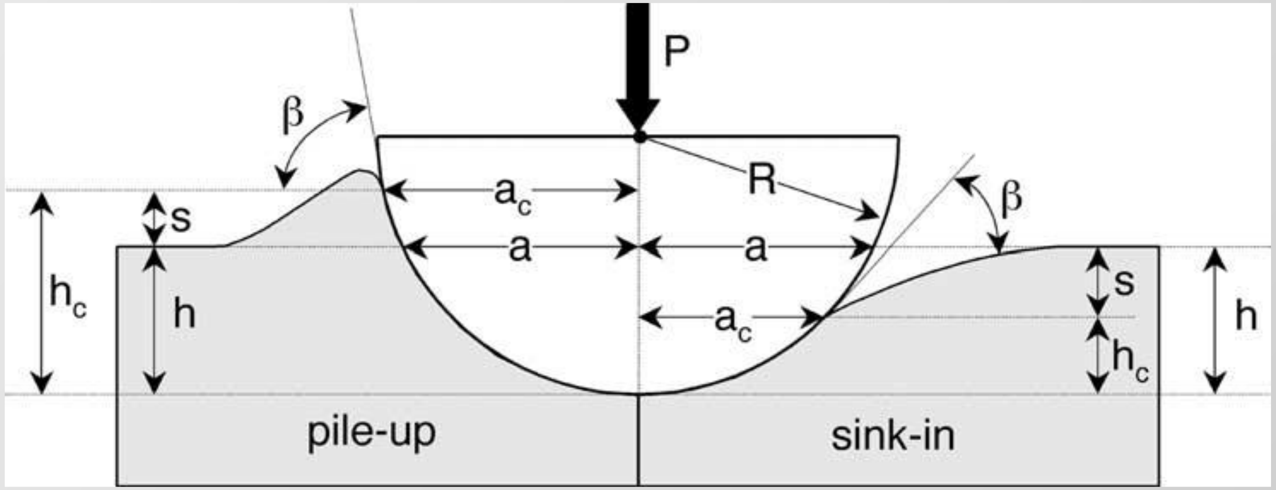
Pile-up and Sink-in

alemnis

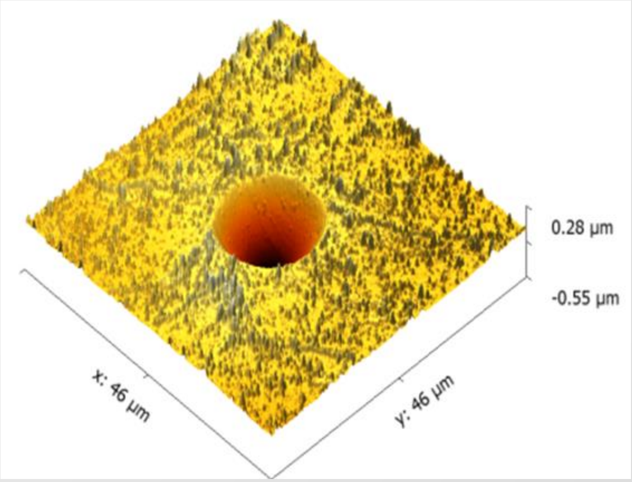
Queen Mary
University of London

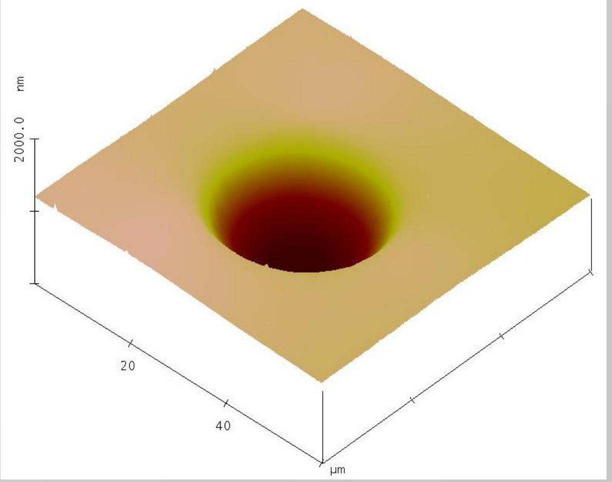


Pile-up and Sink-in



Taljat & Pharr, International Journal of Solids and Structures 41 (2004) 3891–3904





Pile-up and Sink-in

Pile-up:

Push up of plastic material around indent
Real contact area larger than measured

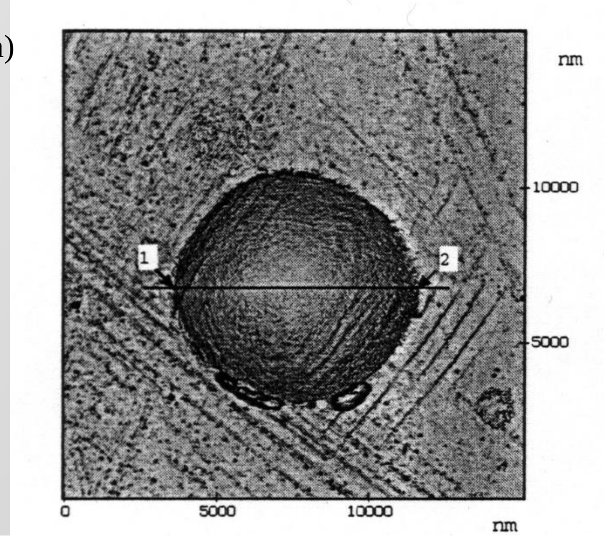
Pile-up: under-estimates contact area

Actual $a = 3.95\ \mu m$
Measured $a = 3.45\ \mu m$, A 24% too small

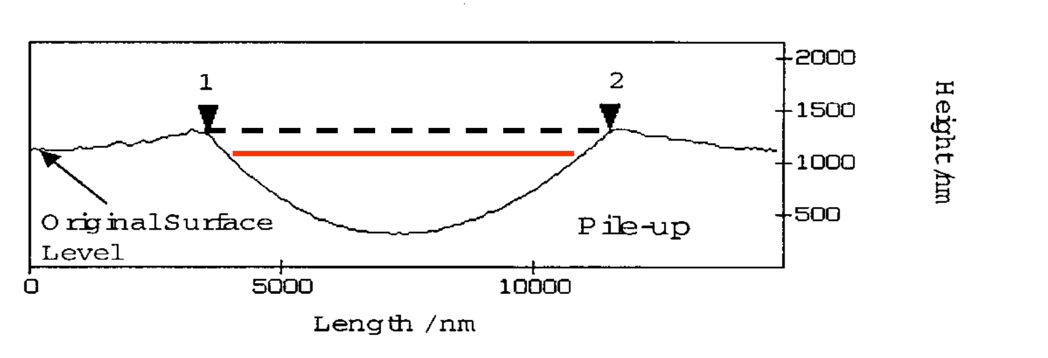
$H \propto 1/A \propto 1/a^2$
H increased by 31% !

$E \propto 1/\sqrt{A} \propto 1/a$
E increased by 14%

(a)



(b)



Example for work hardened copper
(perfectly plastic – material does not flow away)

Pile-up and Sink-in

Sink-in:

Depression of plastic material around indent
Real contact area smaller than measured

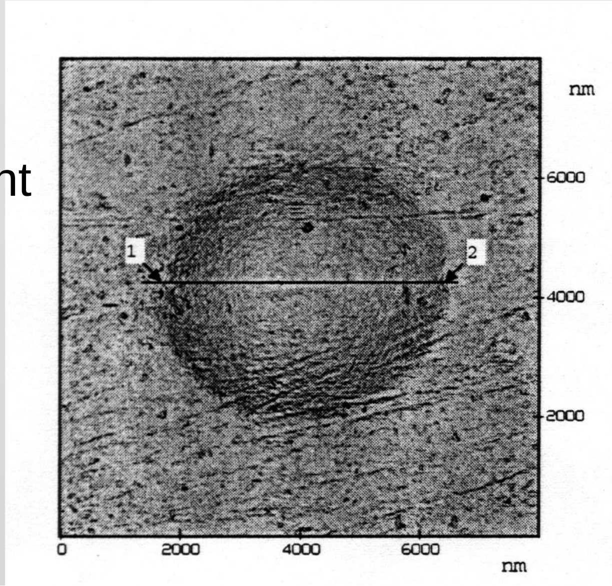
Sink-in: over-estimates contact area

Actual $a = 2.33\ \mu m$
Measured $a = 2.73\ \mu m$, A 37% too big

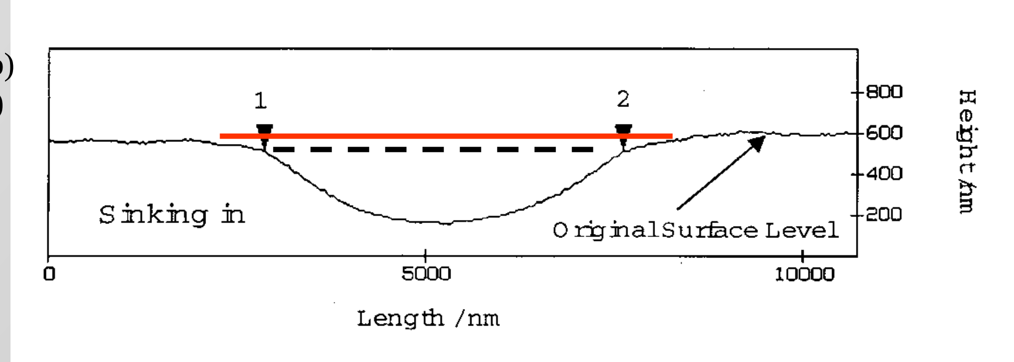
$H \propto 1/A \propto 1/a^2$
H reduced by 27% !

$E \propto 1/\sqrt{A} \propto 1/a$
E reduced by 14%

(a)



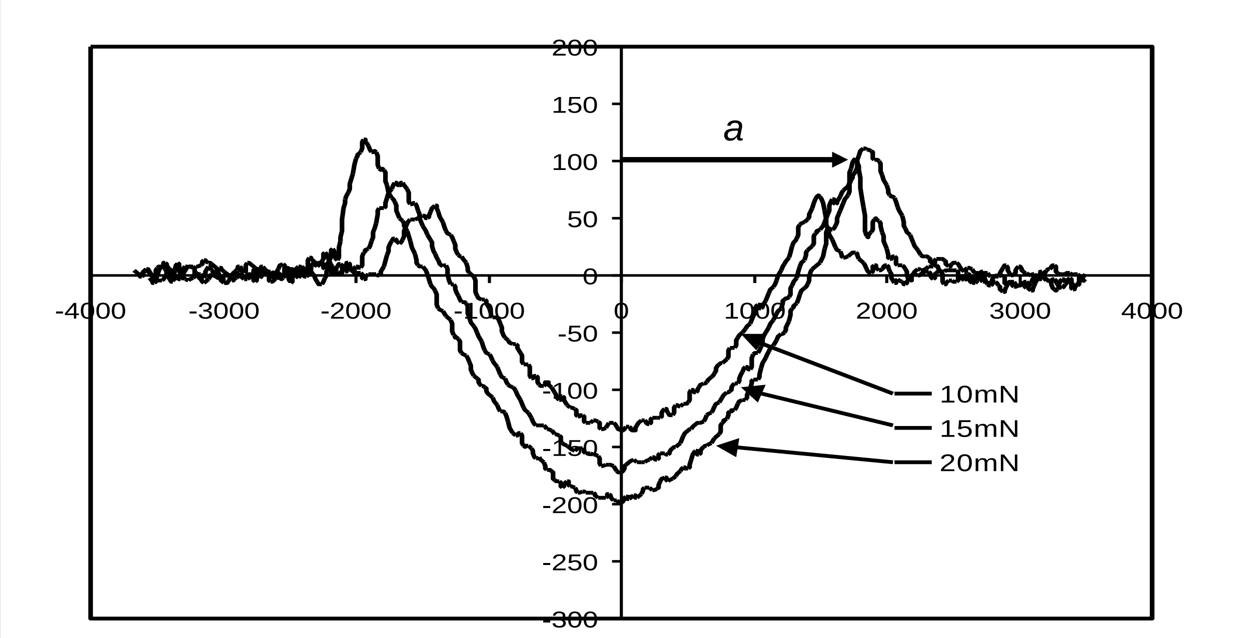
(b)
b)



Example for annealed copper
(work hardens rapidly)

Pile-up and Sink-in

Development of pile-up with increasing load



Measured contact radius, *a*, by metrological AFM

Nominal indenter radius = 5μm
Aluminium

alemnis

Queen Mary University of London

Potential pitfalls

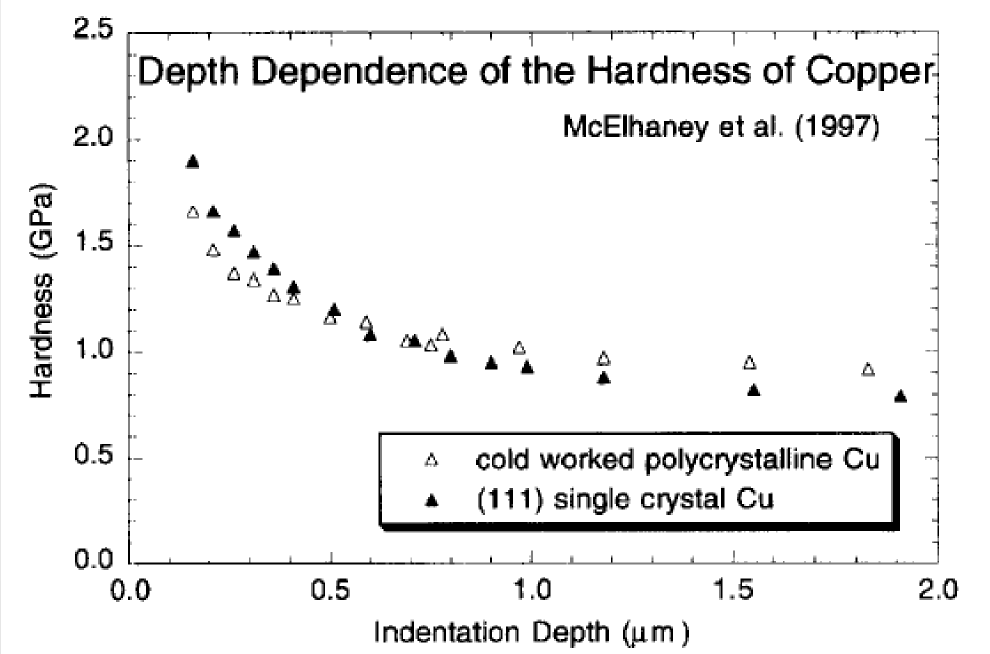
- Non-ideal tip shape (not perfect sphere or pyramid)
- Thermal drift or mechanical instability
- Non-ideal surface (not perfectly smooth and flat)
- Non-ideal material response (creep, pile-up, etc.)
- **Size effects (changes in material properties with scale)**

Hardness size effect

Elastic modulus and hardness are expected to be material constants
i.e. independent of size

Elastic modulus **is** the most constant (and characteristic) materials properties

Hardness shows a ‘size effect’ **smaller = harder**

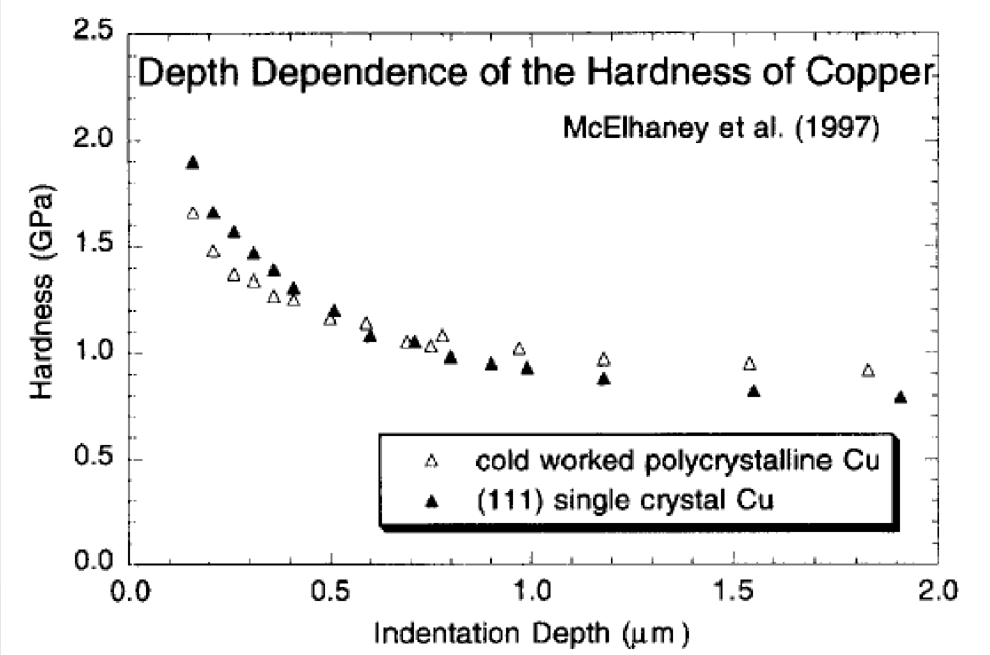


Nix and Gao, *J. Mech. Phys. Sol*., **46**, (1998) 411-425

Hardness size effect

Indentation size effects are not always easy to recognize

Single crystals show the size effect most clearly
For strong materials (e.g. ceramics or fined grain sized metals)
the size effect is only recognizable in very small indentations – near surface

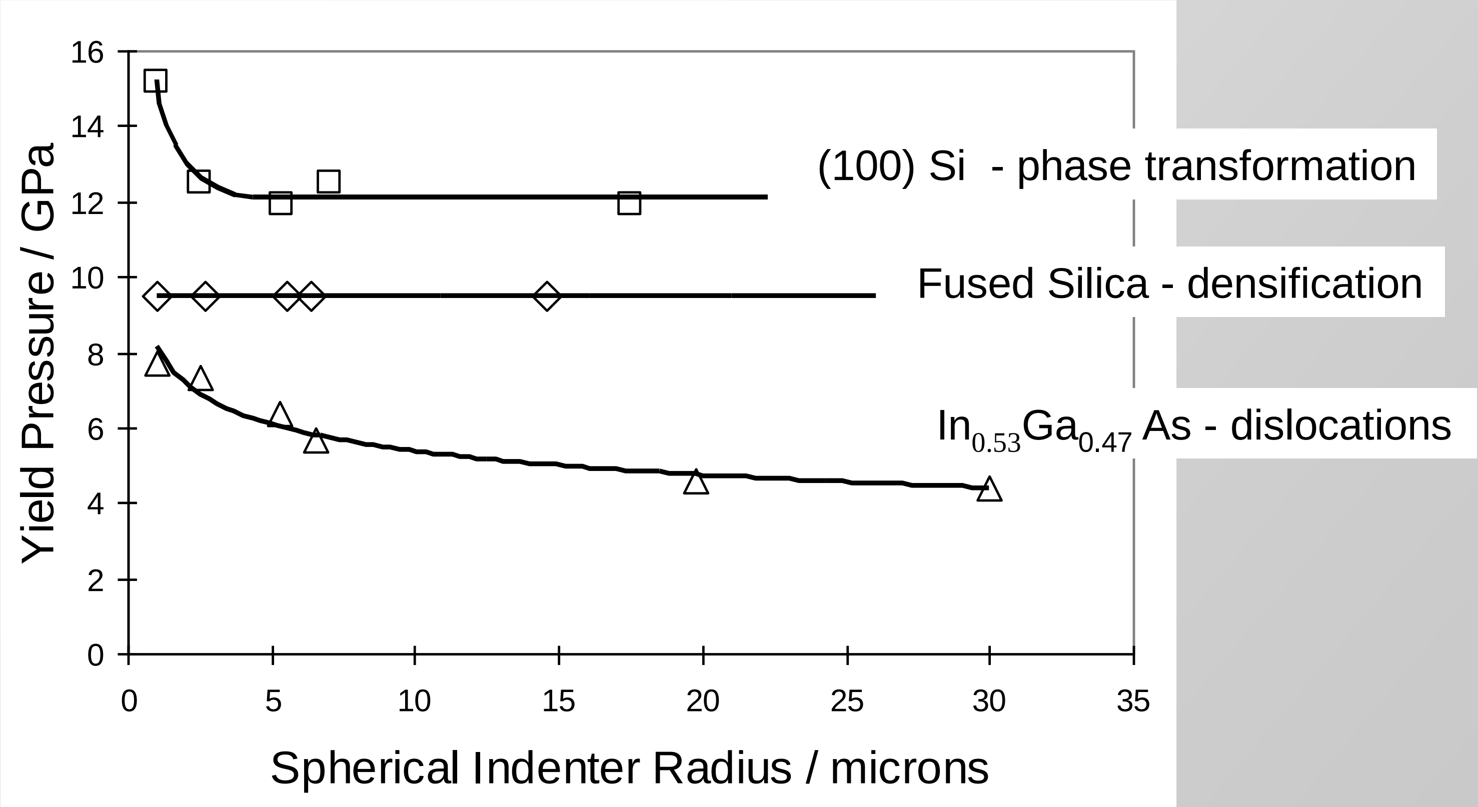


Nix and Gao, *J. Mech. Phys. Sol*., **46**, (1998) 411-425

Mechanism of plastic deformation

Size effect depends on the mechanisms of plastic relaxation

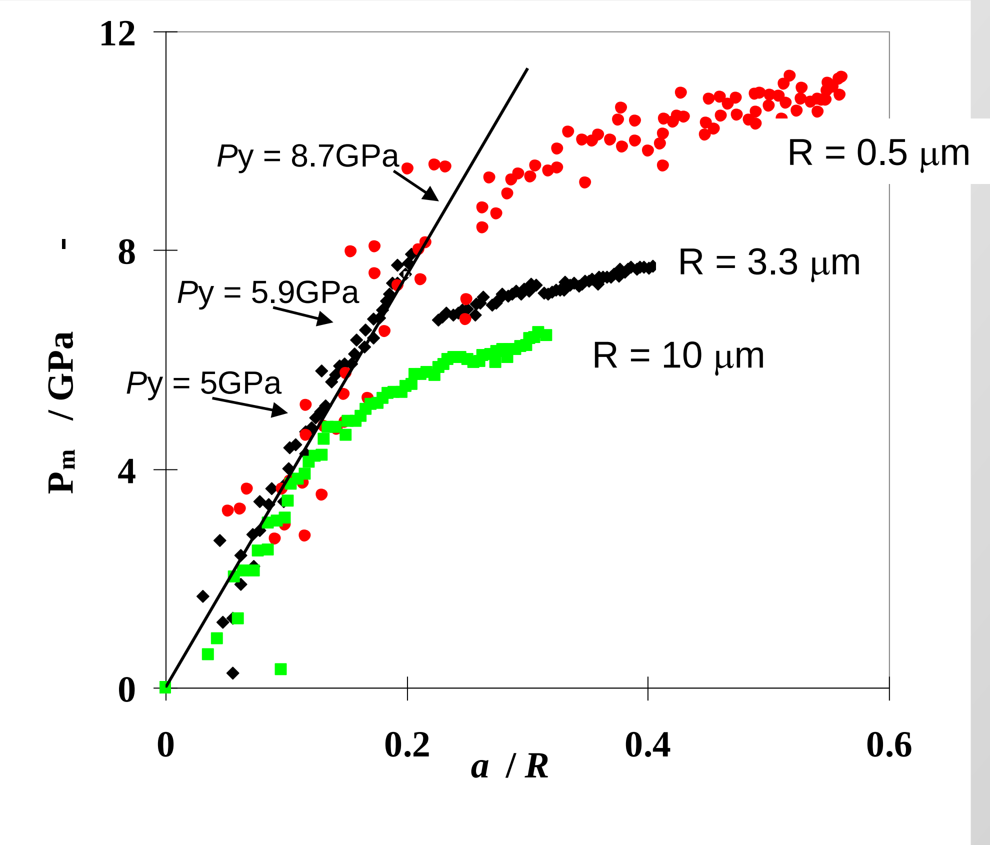
Strongest in crystalline materials that deform by dislocation plasticity



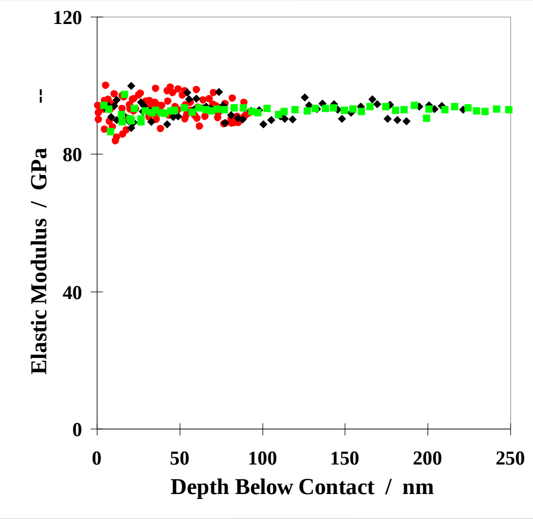
Bushby and Dunstan *J. Mater. Res.* **19**, (2004) 137-142

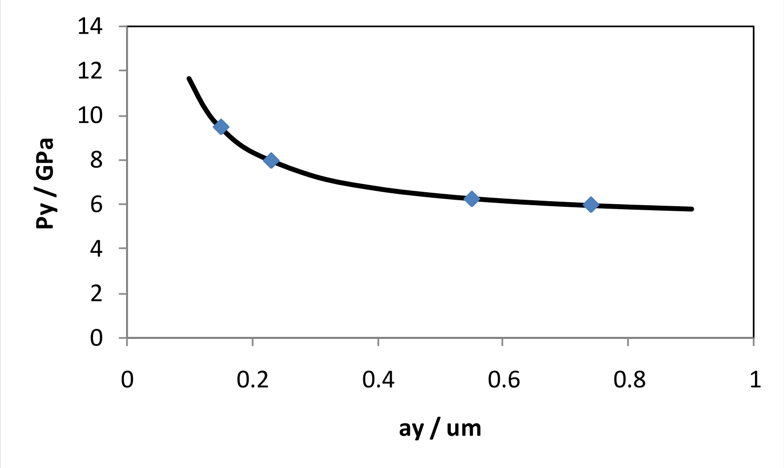
Indentation size effect

Higher yield pressure (stress) for smaller radius indenter but constant *E*



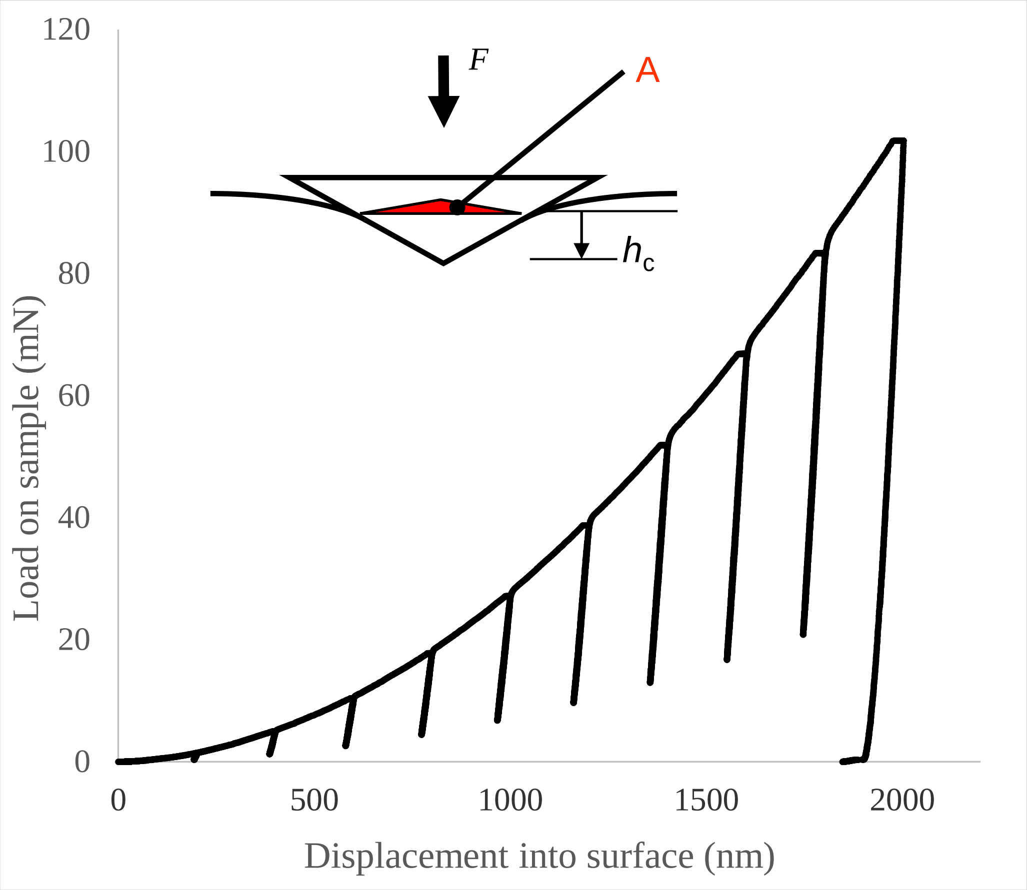
Data for InGaAs





Zhu *et al.*, *J. Mech. Phys. Sol.*, **56**, (2008) 1170-1185

Protocol for assessing indentation size effect



- **Berkovich indenter**
- Multi-cycle test
- 10 depths per test (total displacement)
  up to 2μm depth
- Partially unload to 25% of each max load
- O&P analysis of each partial unload curve

- No pause for creep
- hc ≠ hmax
- Repeatability at different locations

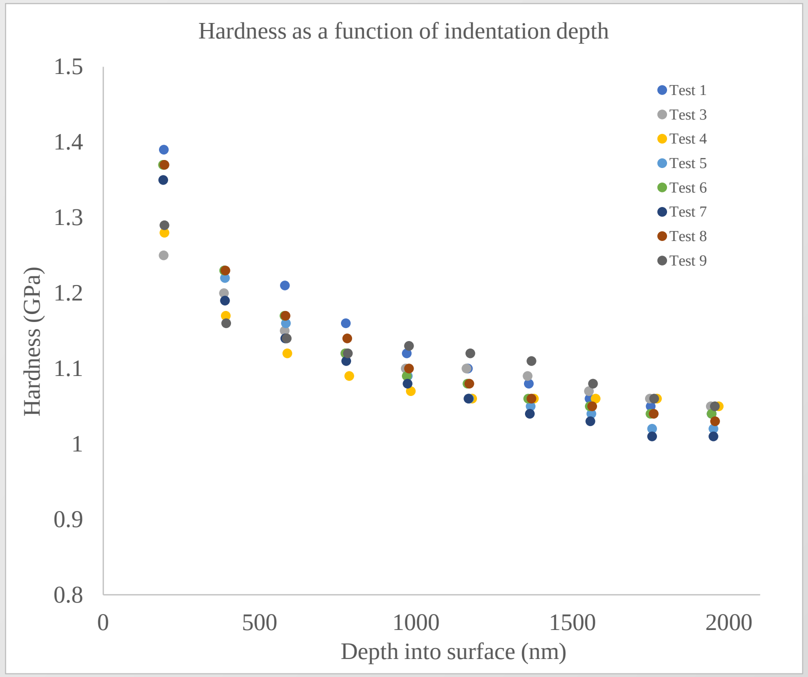
Need calculated contact areas at each depth

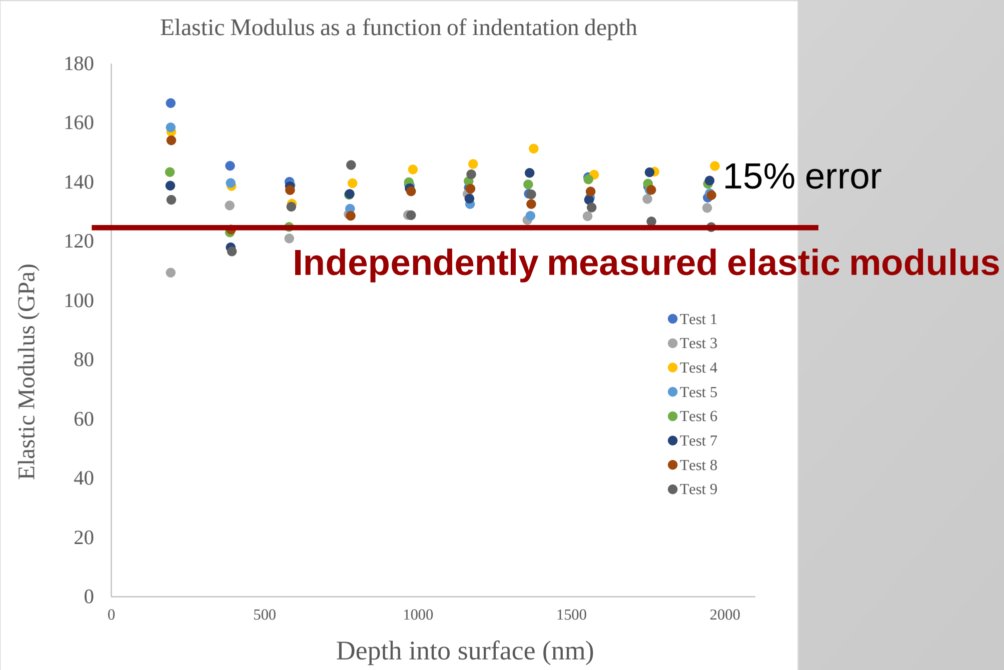
Test results (for CuCrZr alloy) – case study

alemnis

Queen Mary University of London

Berkovich indenter – different depths in a multi-cycle test





Error in *H* due to pile-up

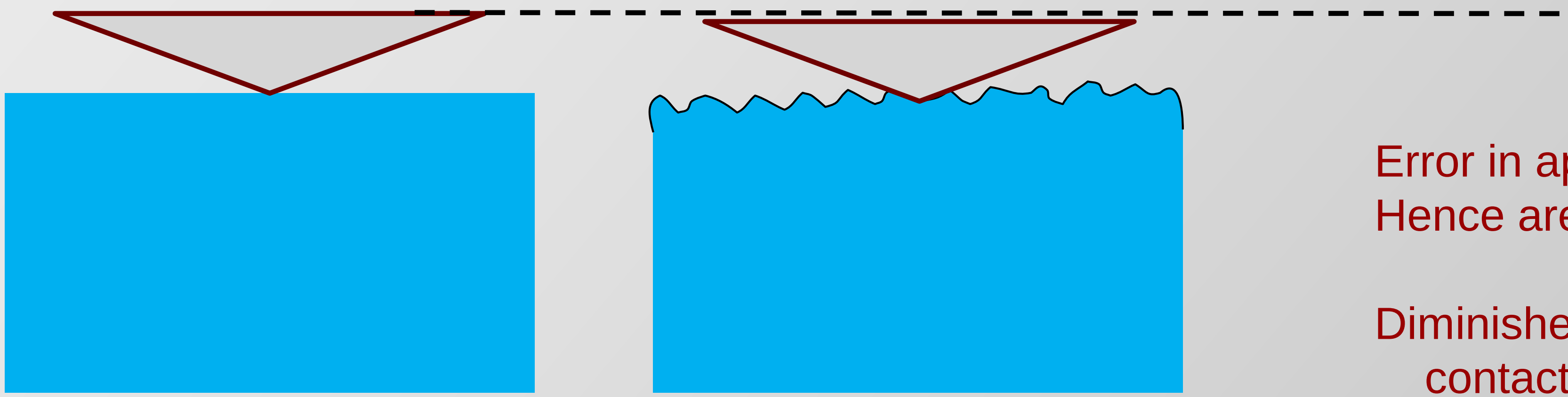
E overestimated by 15% due to pile-up

What can cause the error in elastic modulus?

Modulus not constant with depth:

- Apparent size effect in modulus due to contact detection error caused by surface roughness?

e.g.



Tip calibration – reference sample

Sample measurement

Error in apparent contact depth
Hence area function

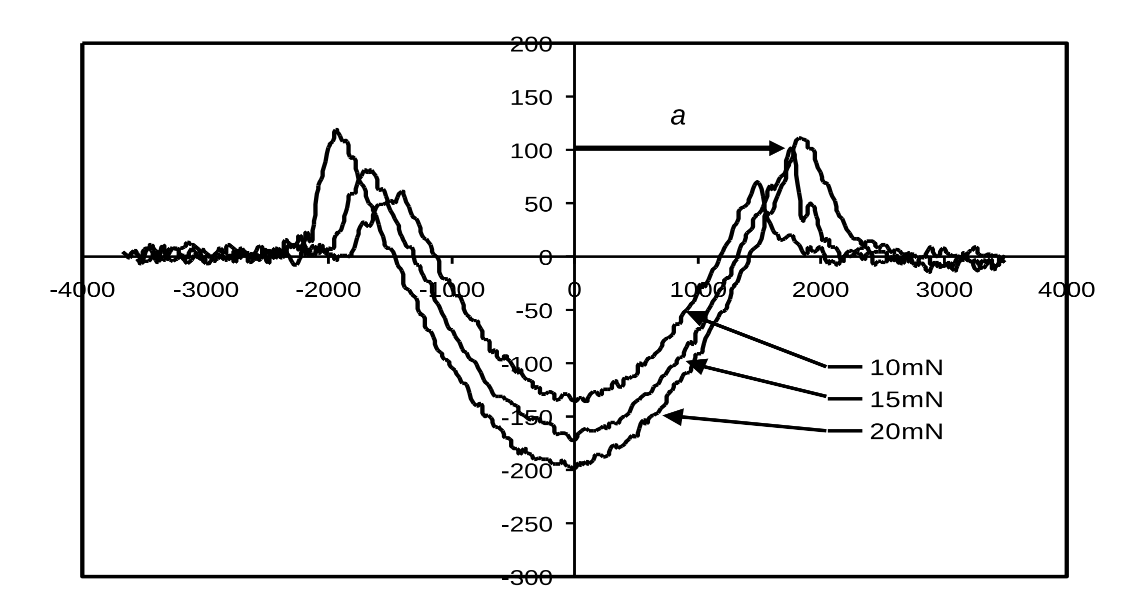
Diminishes with increasing contact depth

What can cause the error in elastic modulus?

Modulus increased compared to expected value:

- Apparent over estimate of elastic modulus due to pile-up caused by plasticity

e.g. Measurement of true contact area by AFM – post-test

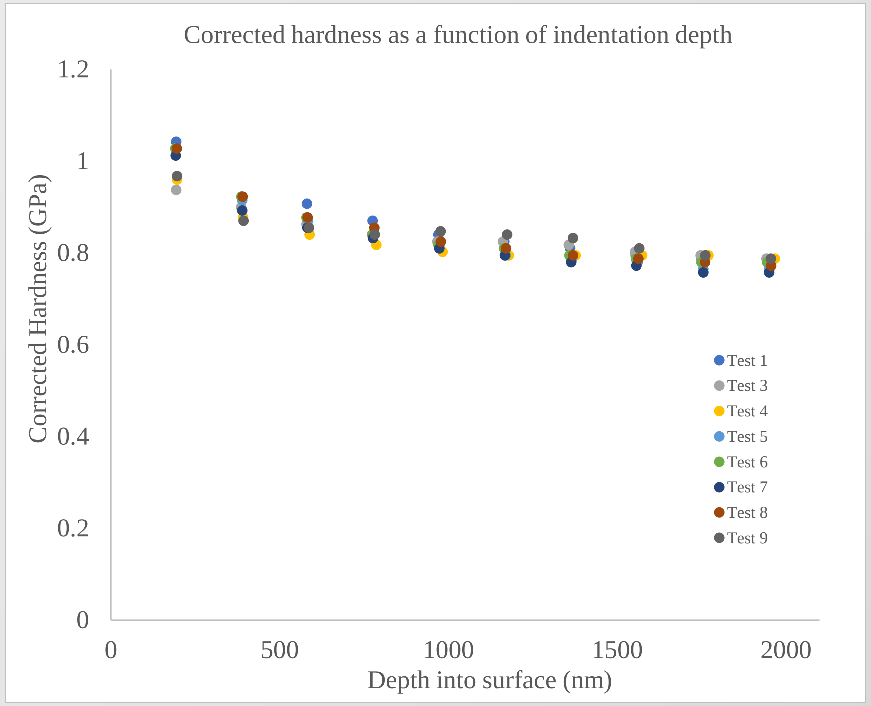


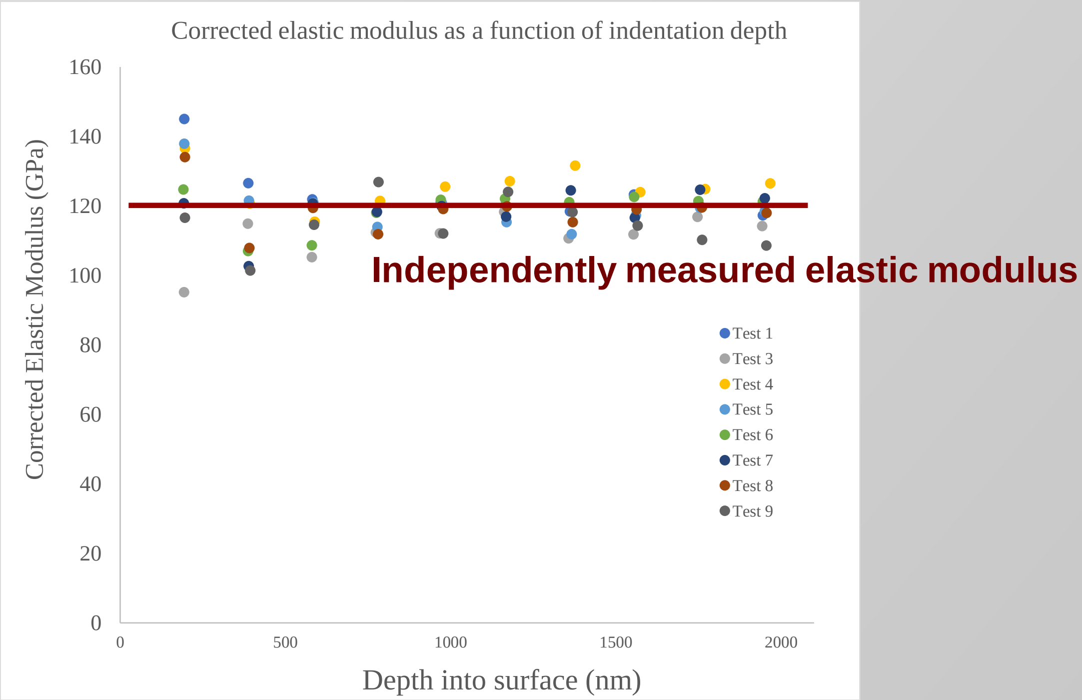
Estimate of true contact area, *a*

If direct measurement not possible – Estimate correction from an independent value for elastic modulus

Test results (for CuCrZr alloy)

Berkovich indenter – different depths in a multi-cycle test
– corrected for contact area (from modulus)





H corrected for estimated contact area error
Project *H* value to macro-scale

Using the known elastic modulus to estimate the error in contact *area A*

Summary

Potential pitfalls

- Non-ideal tip shape (not perfect sphere or pyramid)
- Thermal drift or mechanical instability

} Instrument

- Non-ideal surface (not perfectly smooth and flat)

} Sample

- Non-ideal material response (creep, pile-up, etc.)
- Size effects (changes in material properties with scale)

} Material