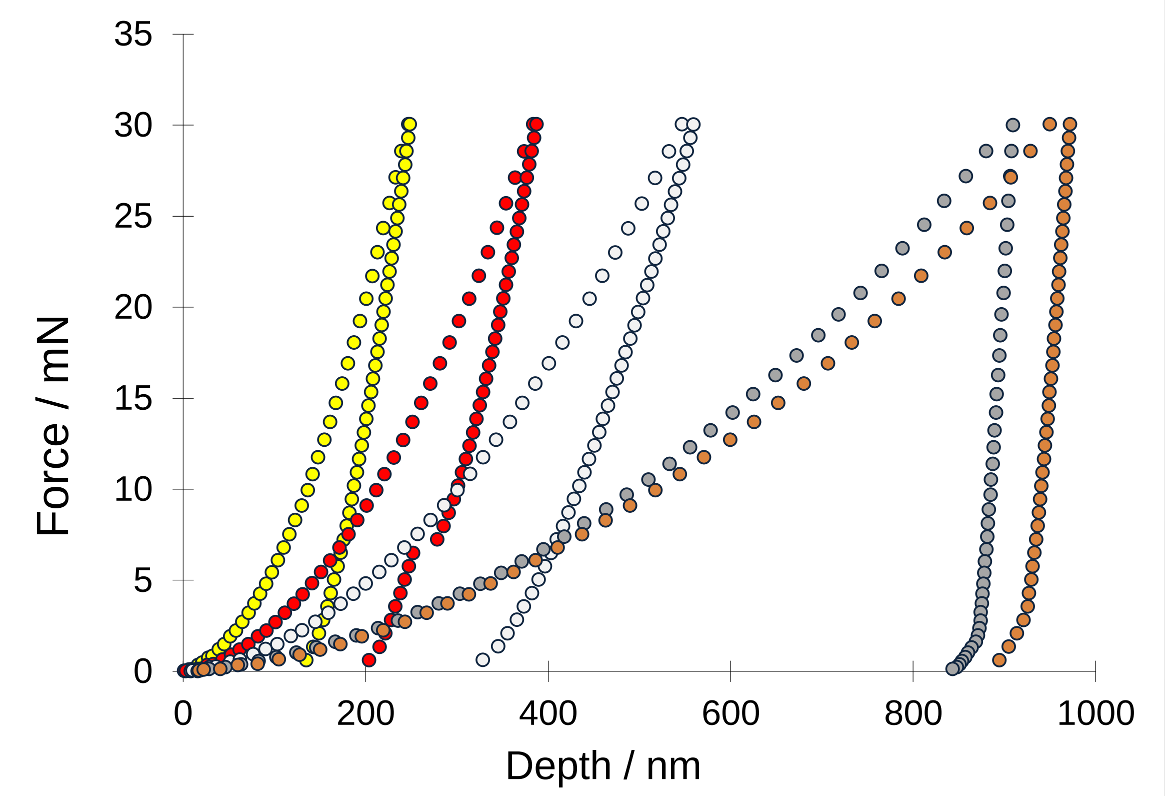
5. Usual and Unusual Behaviours

Andy Bushby

Typical nanoindentation data

Alumina	Silicon	Glass	Nickel	Copper
E = 400 GPa	E = 180 GPa	E = 72.6 GPa	E = 210 GPa	E = 120 GPa
H = 22 GPa	H = 10 GPa	H = 5.4 GPa	H = 1.4 GPa	H = 1.2 GPa
E/H = 18	E/H = 18	E/H = 13.4	E/H = 150	E/H = 100
U_e/U_p = 120%	U_e/U_p = 118%	U_e/U_p = 102%	U_e/U_p = 6.02%	U_e/U_p = 8.34%



Hardness

$$H = \frac{F}{A}$$

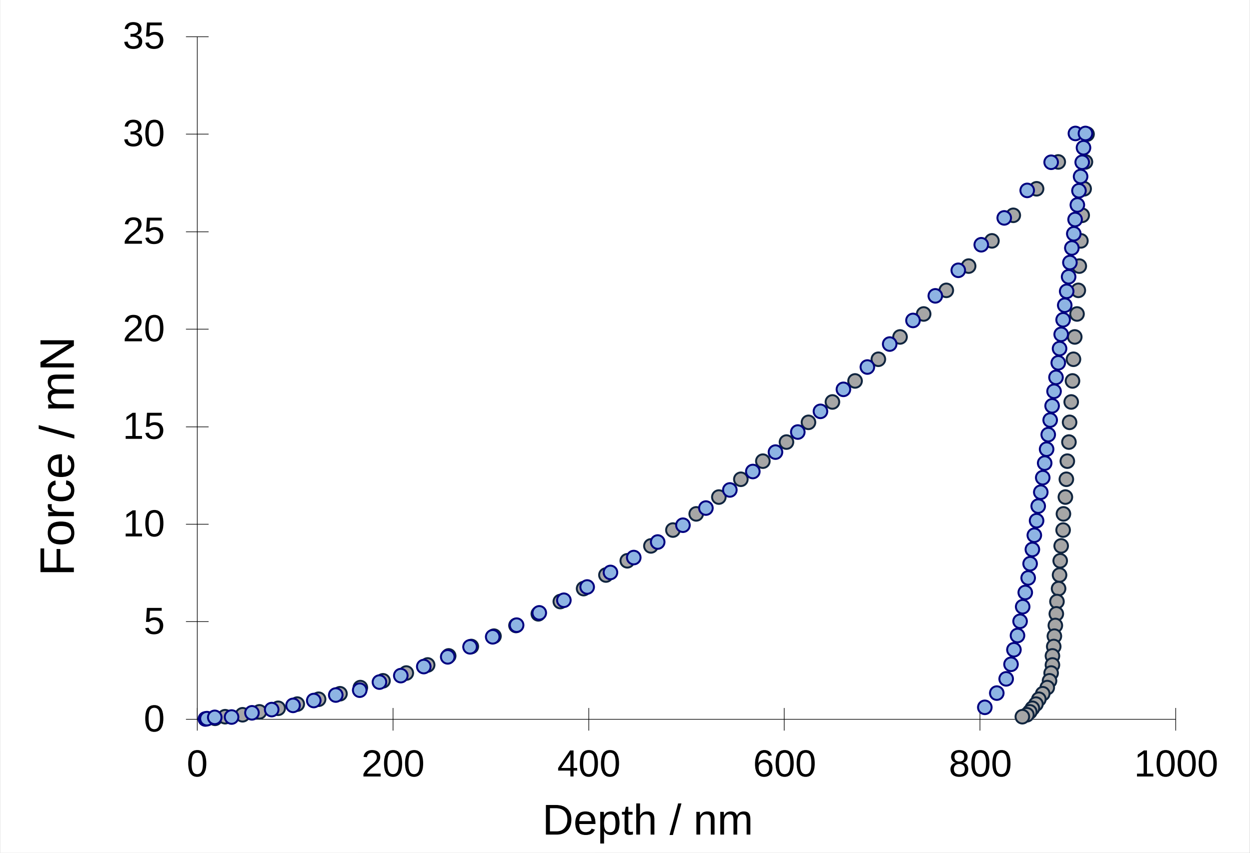
Elastic modulus

$$E^* = \beta \frac{\sqrt{\pi}}{2} \frac{\left(\frac{dF}{dh}\right)}{\sqrt{A}}$$

Typical nanoindentation data

Same hardness
Different modulus

Nickel	Aluminium
E = 210 GPa	E = 70 GPa
H = 1.4 GPa	H = 1.4 GPa

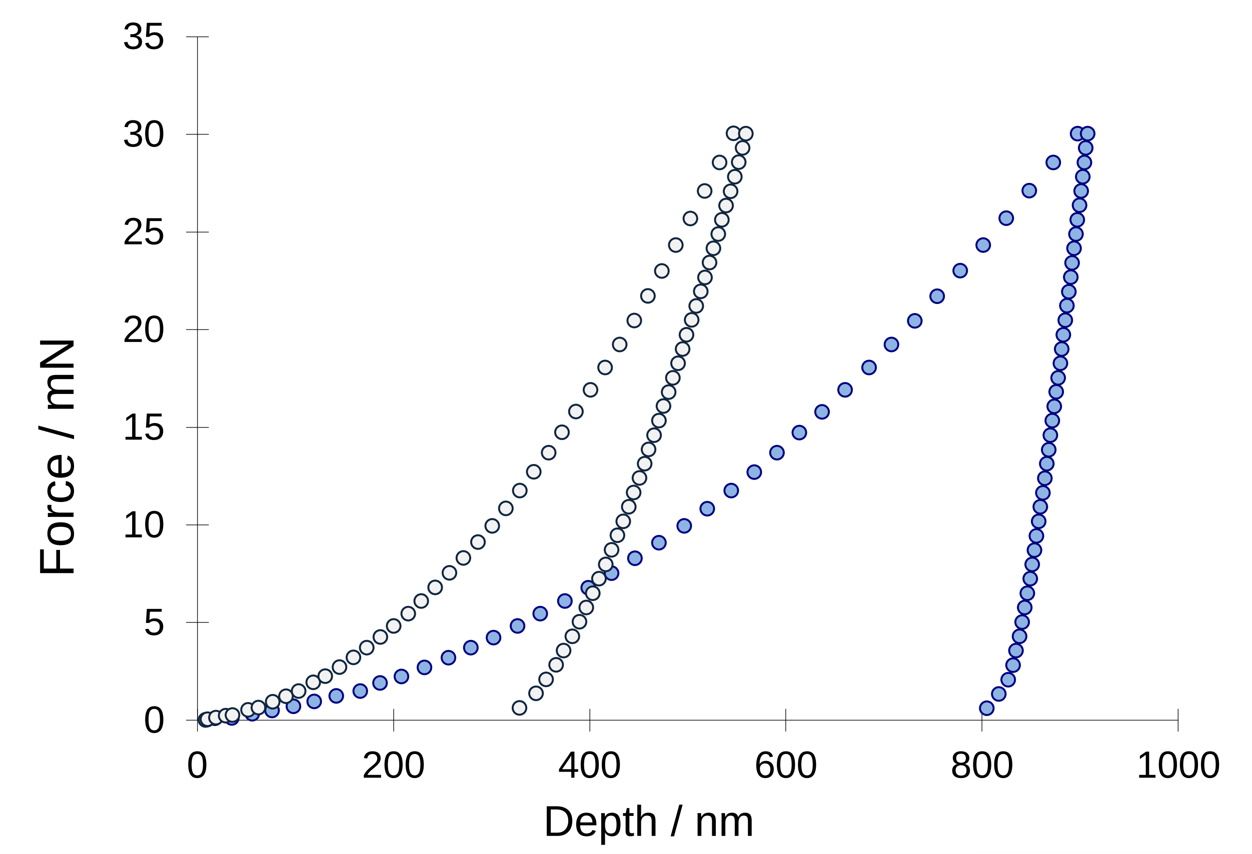


Typical nanoindentation data

Same modulus
Different hardness

Glass
E = 72.6 GPa
H = 5.4 GPa

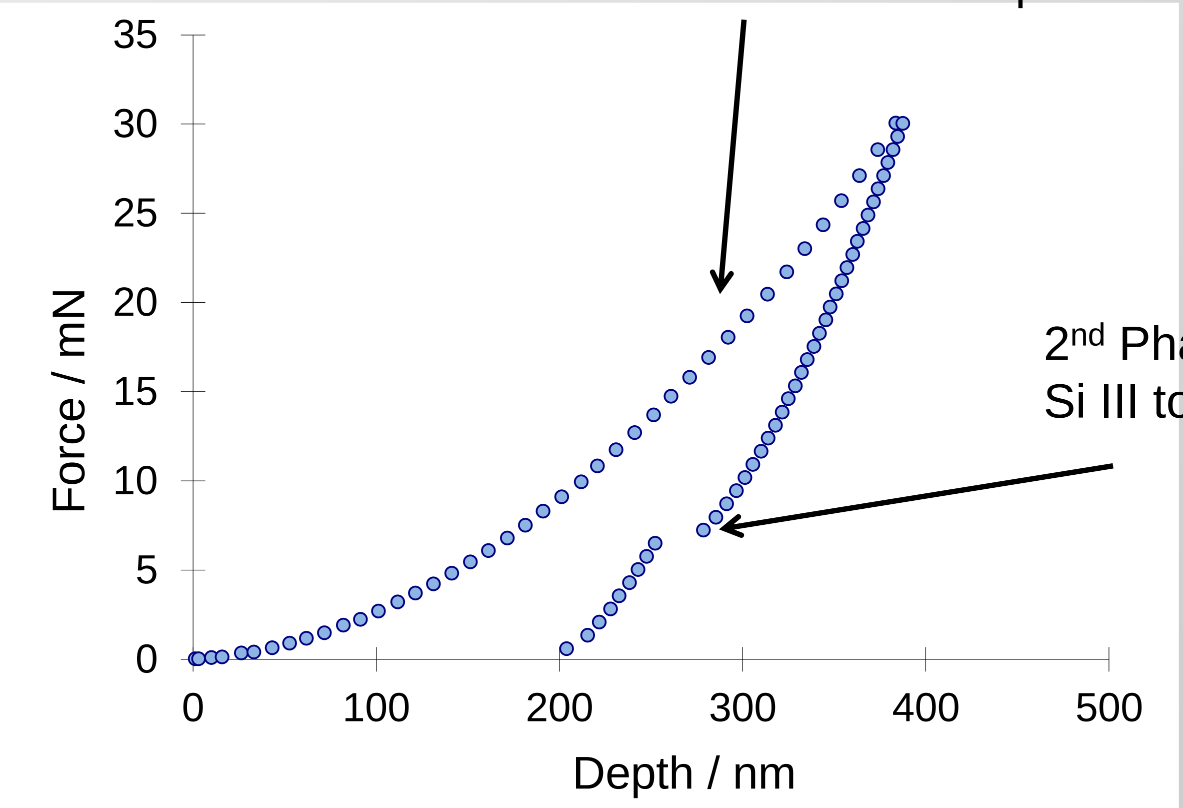
Aluminium
E = 70 GPa
H = 1.4 GPa



Phase transformation

Silicon
E = 180 GPa
H = 10 GPa

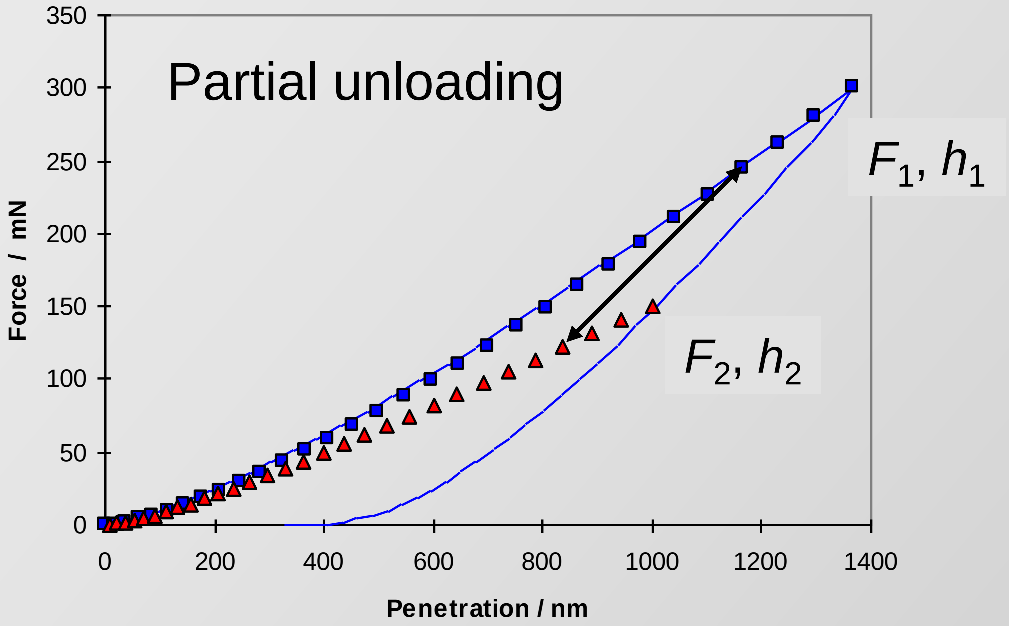
Pressure induced phase transformation
From Si I to amorphous Si at ~12GPa

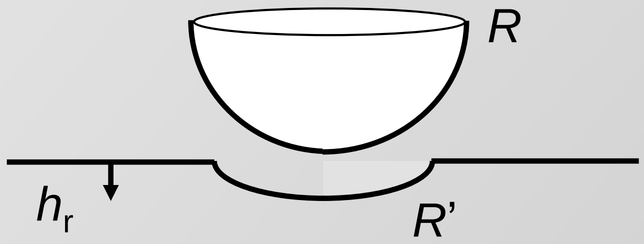


2nd Phase transformation from
Si III to Si XII on unloading
(crystallisation)

Multi-cycle tests: load – partial unload

Determine contact radius, *a*, as a function of depth (spherical indenters)





$$h_c = \frac{h_{max} + h_r}{2}$$

$$h_e = 2(h_{max} - h_c)$$

$$a^2 = (2Rh_c - h_c^2)$$

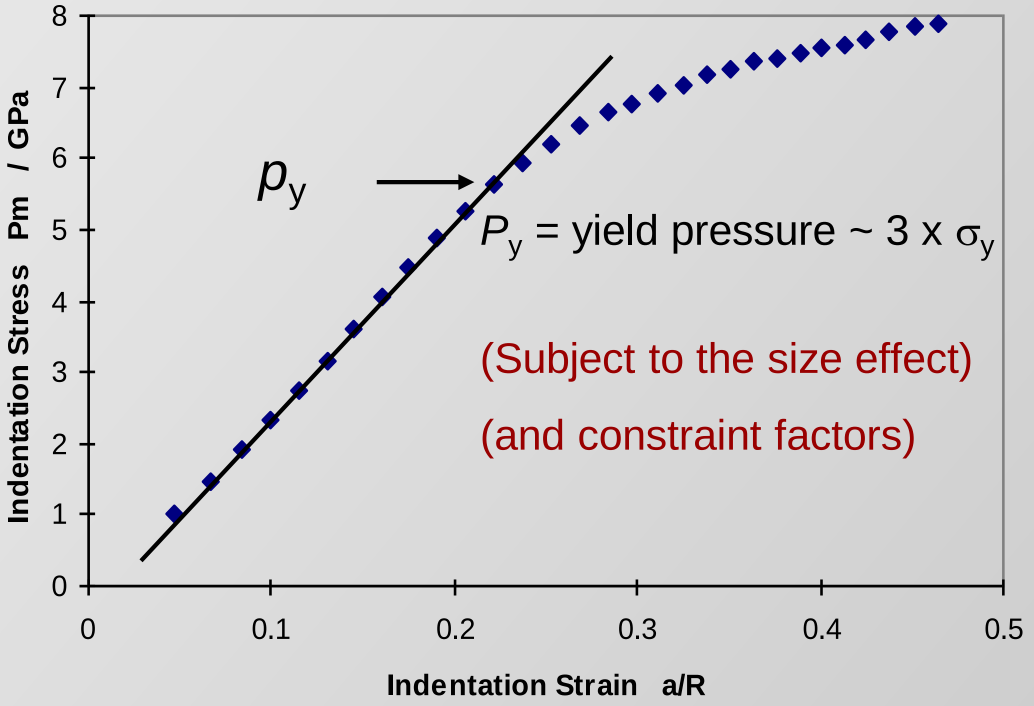
$$h_e = \left(\frac{9}{16}\frac{F^2}{E^{*2}}\frac{1}{R^*}\right)^{1/3}$$

$$E^* = \left(\frac{3}{4}\frac{F}{h_e^{3/2}}\frac{1}{R^{*1/2}}\right)$$

Indentation stress-strain curves

Indentation stress = $P_m = F / \pi a^2$

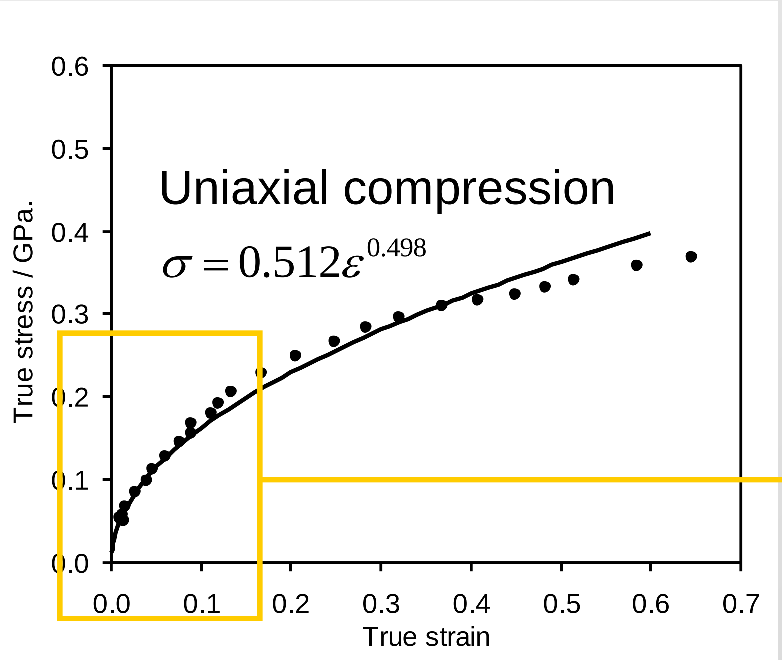
Indentation strain = a / R

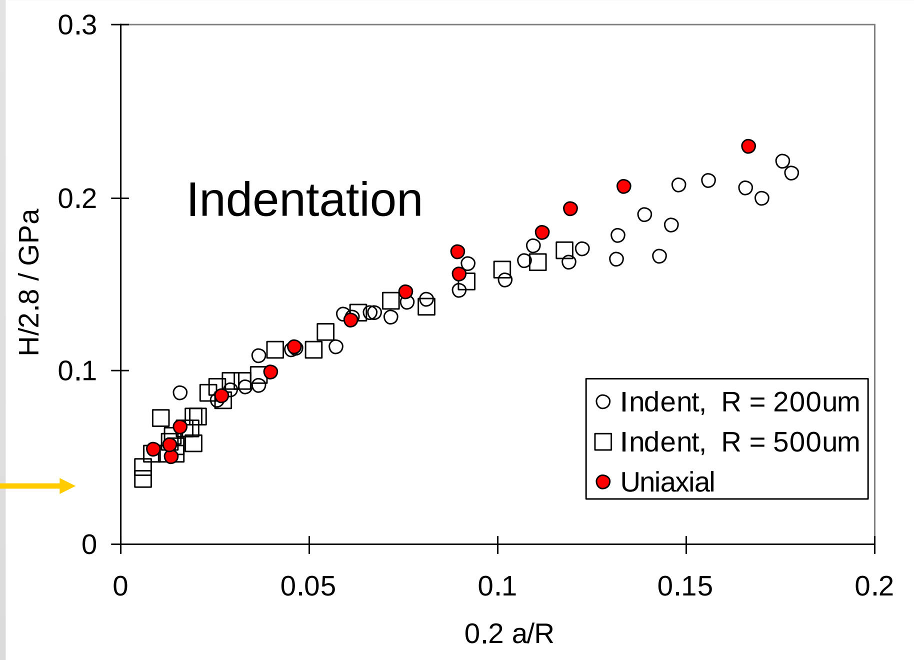


Meyer's Law $$P_m = k\left(\frac{a}{R}\right)^m$$ m = hardening rate

Indentation stress-strain curves

Example for annealed copper



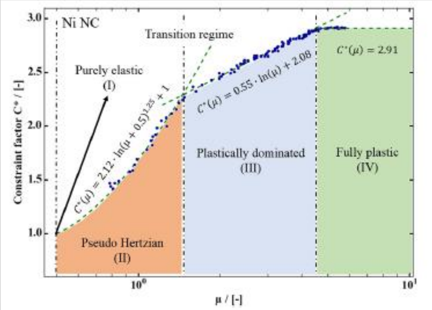


The Tabor relations

$$\begin{cases} \text{Representative stress} = P_m / 2.8 \\ \text{Representative strain} = 0.2\, a/R \end{cases}$$

Constraint factor, C*(μ)

$$\mu = \frac{E_r}{H}\frac{\cot\theta}{\pi}$$



Tabor – The Hardness of Metals, 1951

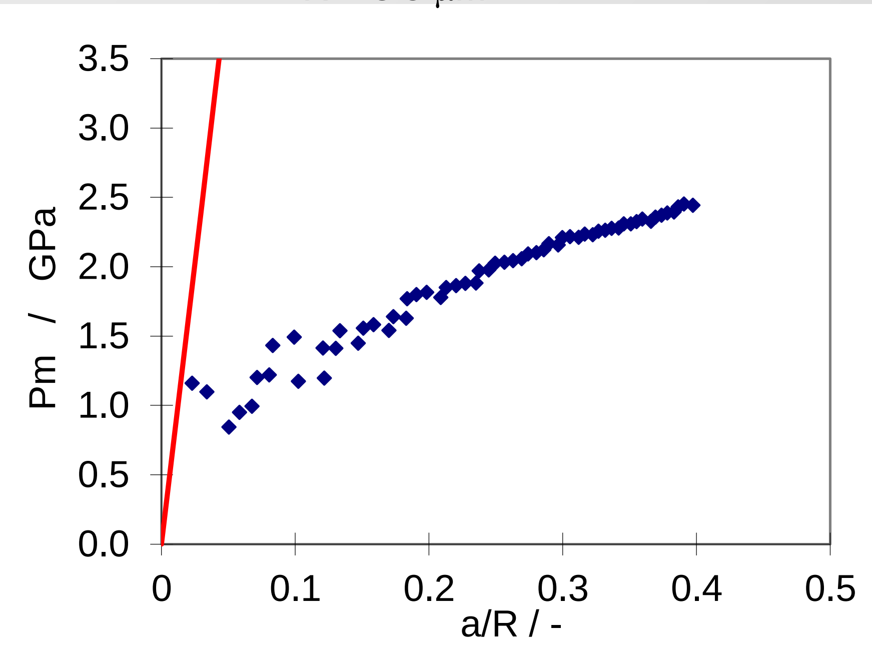
Spary, Bushby and Jennett, Phil. Mag., **A86**, 5581 (2006)

Leitner et al. / Materials and Design **146** 69 (2018)

Strain hardening and softening

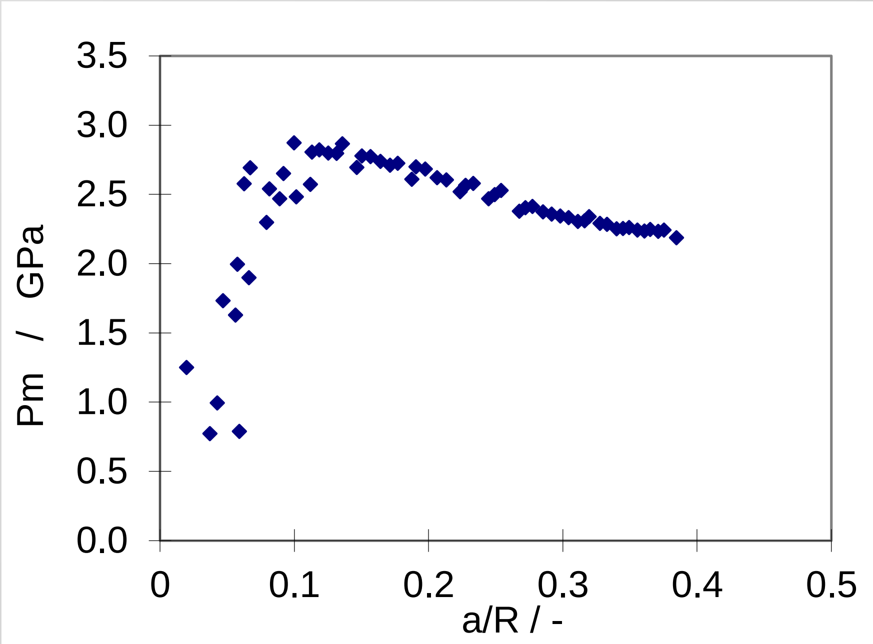
Strain hardening

Example for annealed Ni
R = 3.5 μm



Strain softening

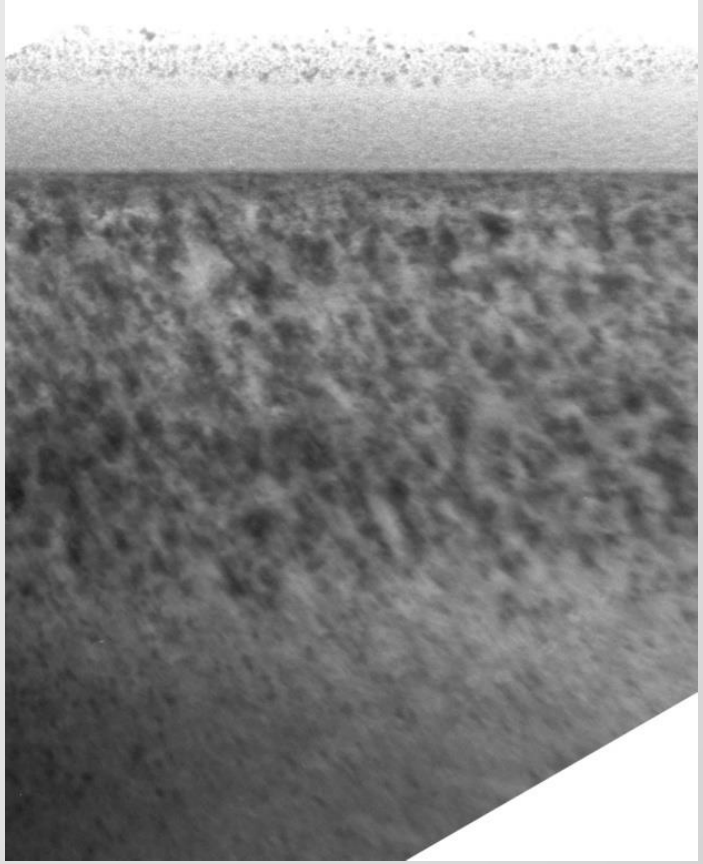
Example for irradiated Fe 12% Cr
R = 3.5 μm



Bushby Roberts and Hardie J. Mater. Res. **27**, (2012), 85-90

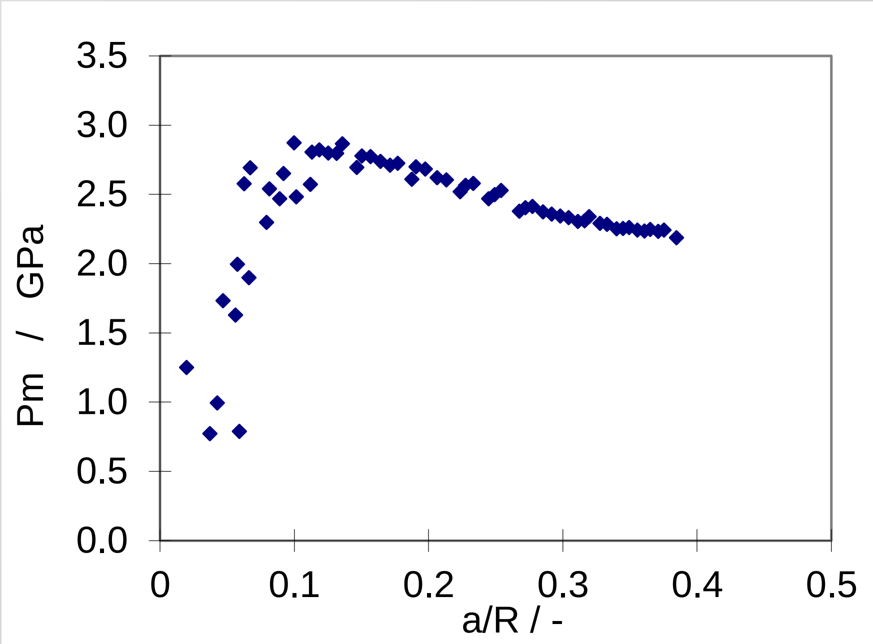
Strain hardening and softening

Irradiation damage
Dislocation loops and Frenkel defects
Swept out by dislocations from indentation



Strain softening

Example for irradiated Fe 12% Cr
R = 3.5 μm



Bushby Roberts and Hardie J. Mater. Res. **27**, (2012), 85-90

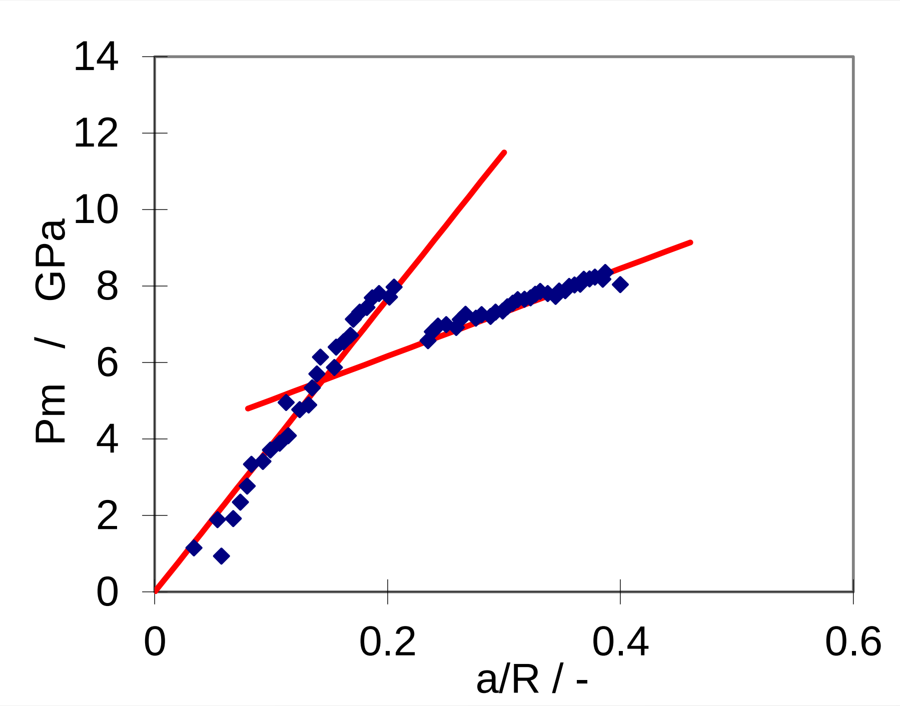
Pop-in: sudden transition to plasticity

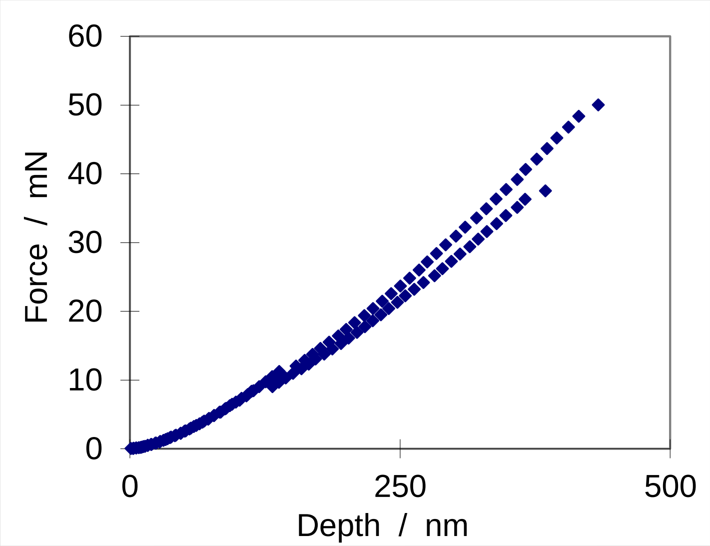
alemnis

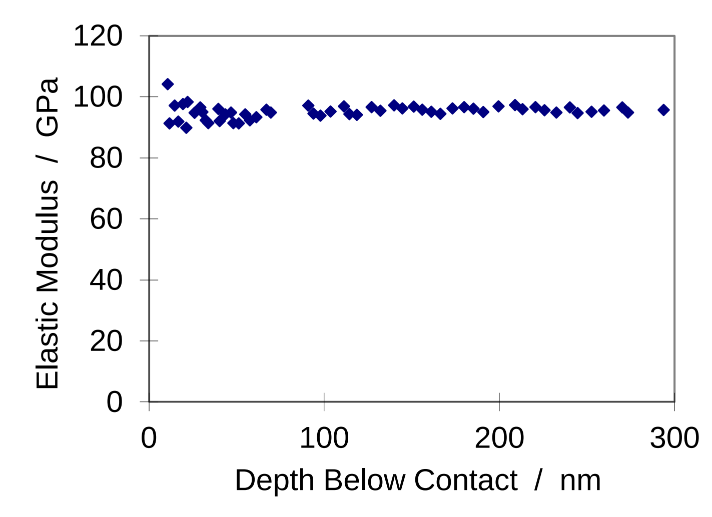
Queen Mary
University of London

Nucleation of dislocation source
- no change in sample stiffness

Example for annealed InGaAs
R = 3.5 µm

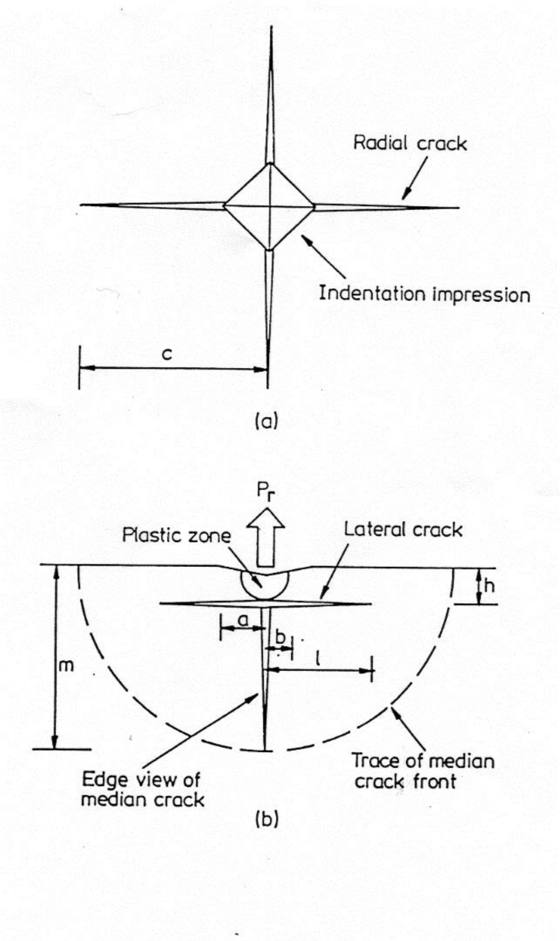




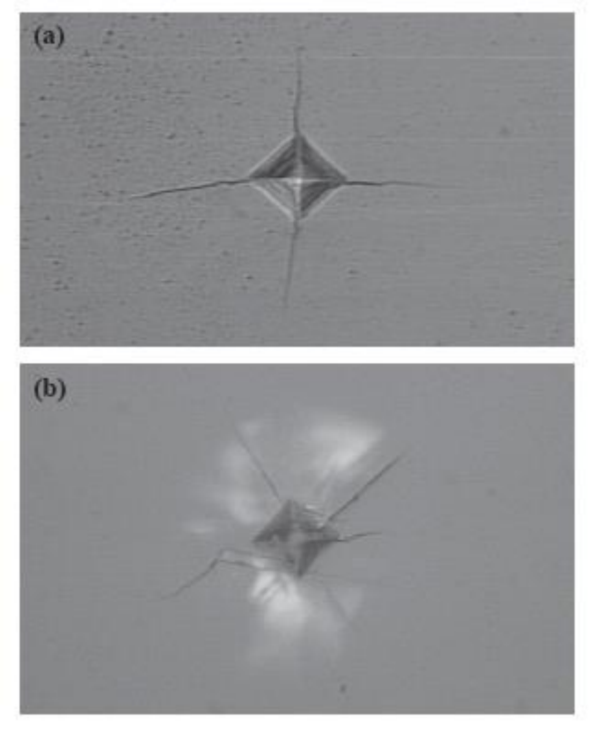


Brittle fracture

Cracking beneath sharp indenters:

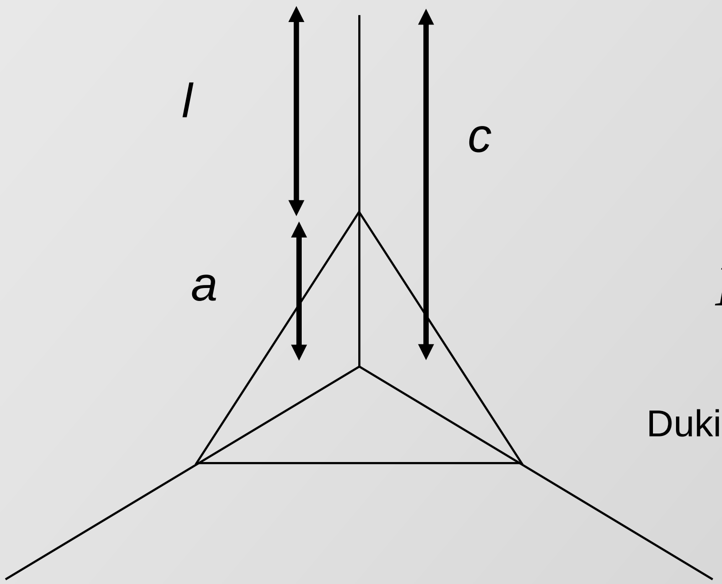


Two systems of cracks
Radial (on loading)
Lateral (on unloading)



Fracture toughness measurement

K_{IC} from indentation crack length measurements:



$$K_c = 0.0161\left(\frac{a}{l}\right)^{1/2}\left(\frac{E}{H}\right)^{2/3}\frac{F}{c^{3/2}}$$

Dukino and Swain, J. Amer. Ceram. Soc. 75, 3299 (1992)

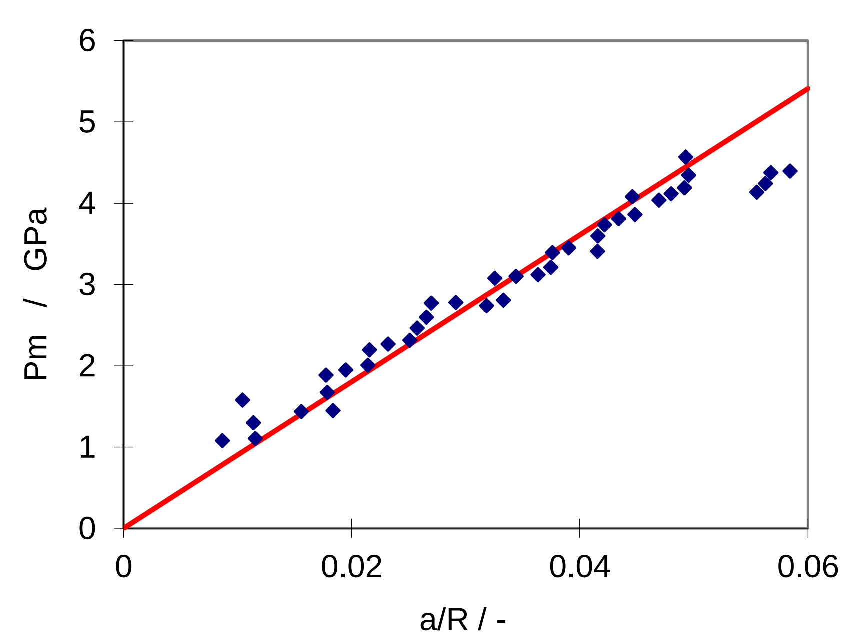
Requires sharp indenter (Berkovich or Corner cube)
Make a series of measurements at different forces
SEM to measure crack lengths
Plot F vs $c^{3/2}$, find K_{IC} from slope
Only works on very brittle materials (ceramics), $K_{IC} < 10$ MPa√m

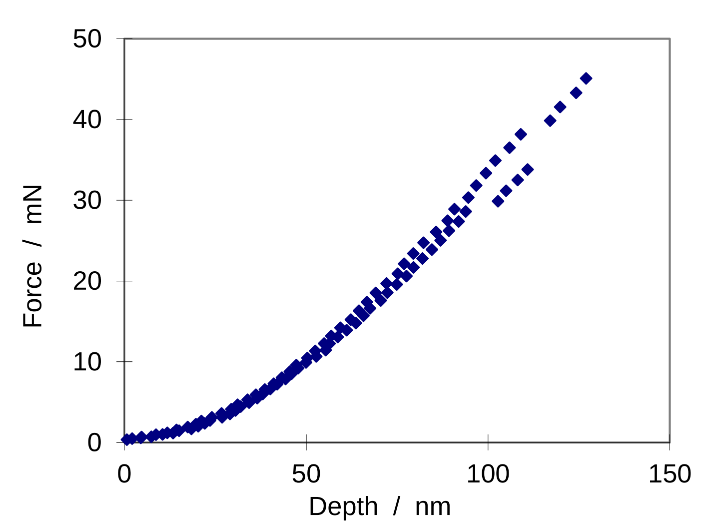
Toughness measurements from very small amount of material

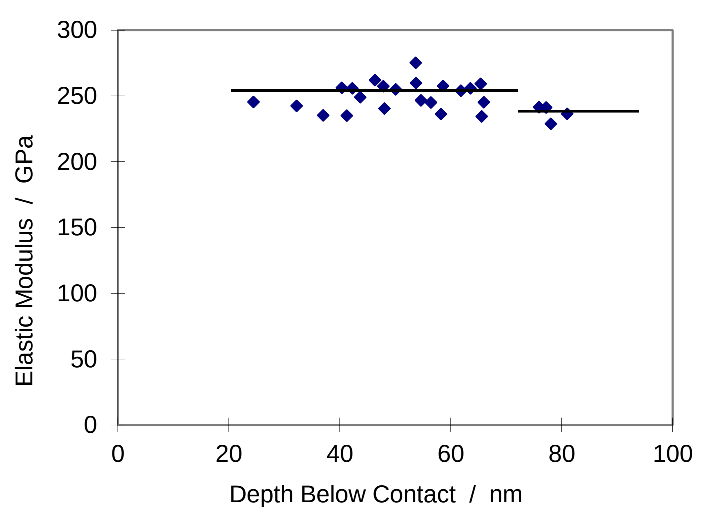
Cracking: sudden increase in depth

Nucleation of cracks
- drop in sample stiffness

Example for NbN / TiN multilayer
R = 20 μm



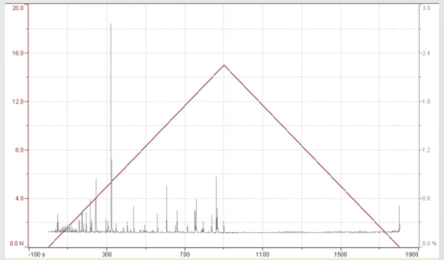


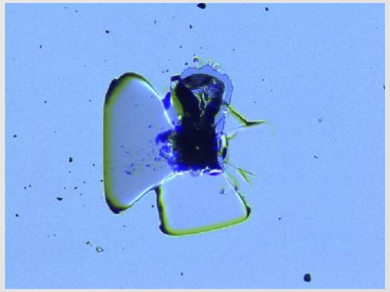


Microindentation with Acoustic Emission (AE)

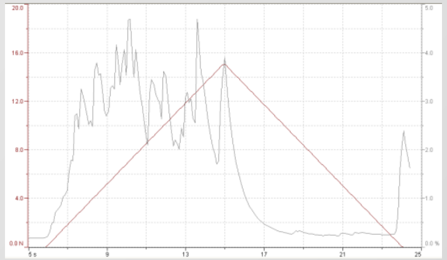
Queen Mary University of London

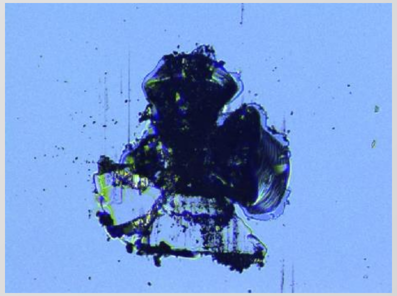
1 N/m



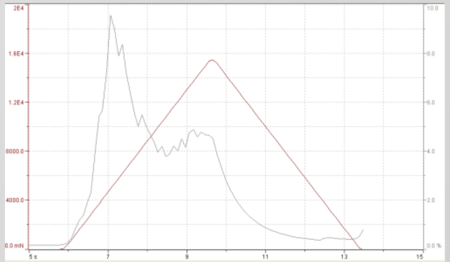


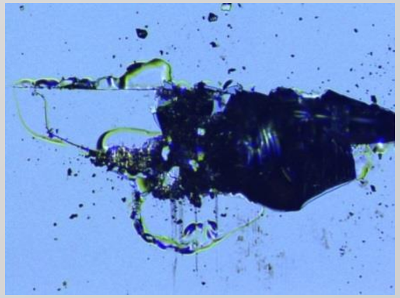
100 N/m





250 N/m





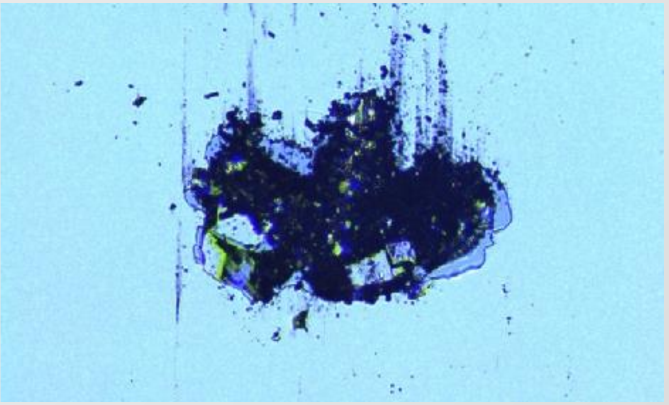
Sample: Glass

Indenter: Vickers

Max load: 15 N

Instrument: MCT

Microindentation with Acoustic Emission (AE)



Sample: Si wafer

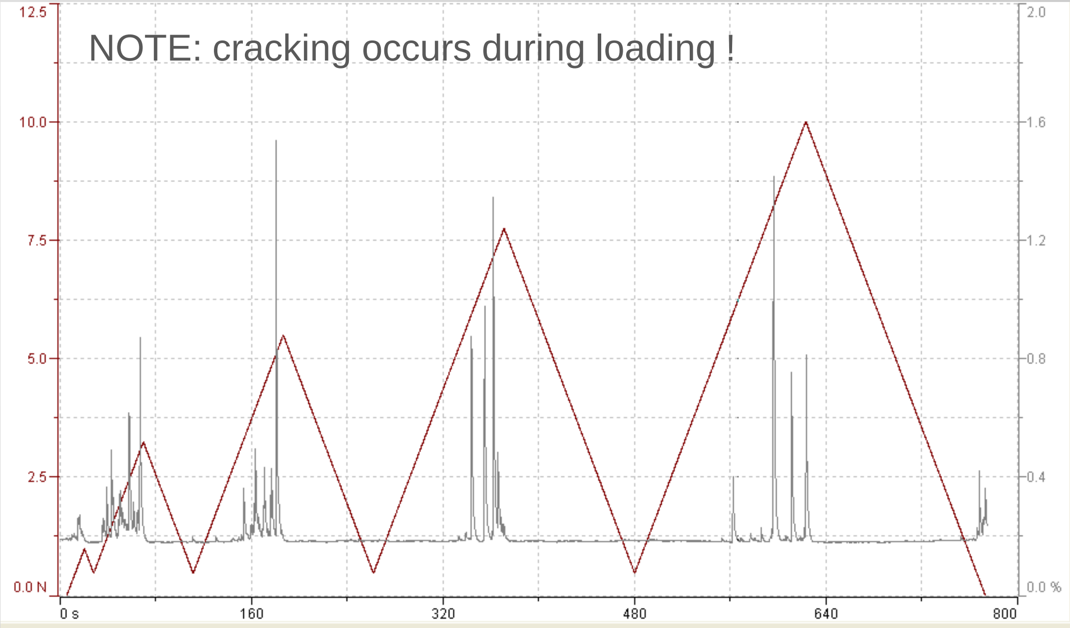
Indenter: Vickers

Load: 1 - 10 N

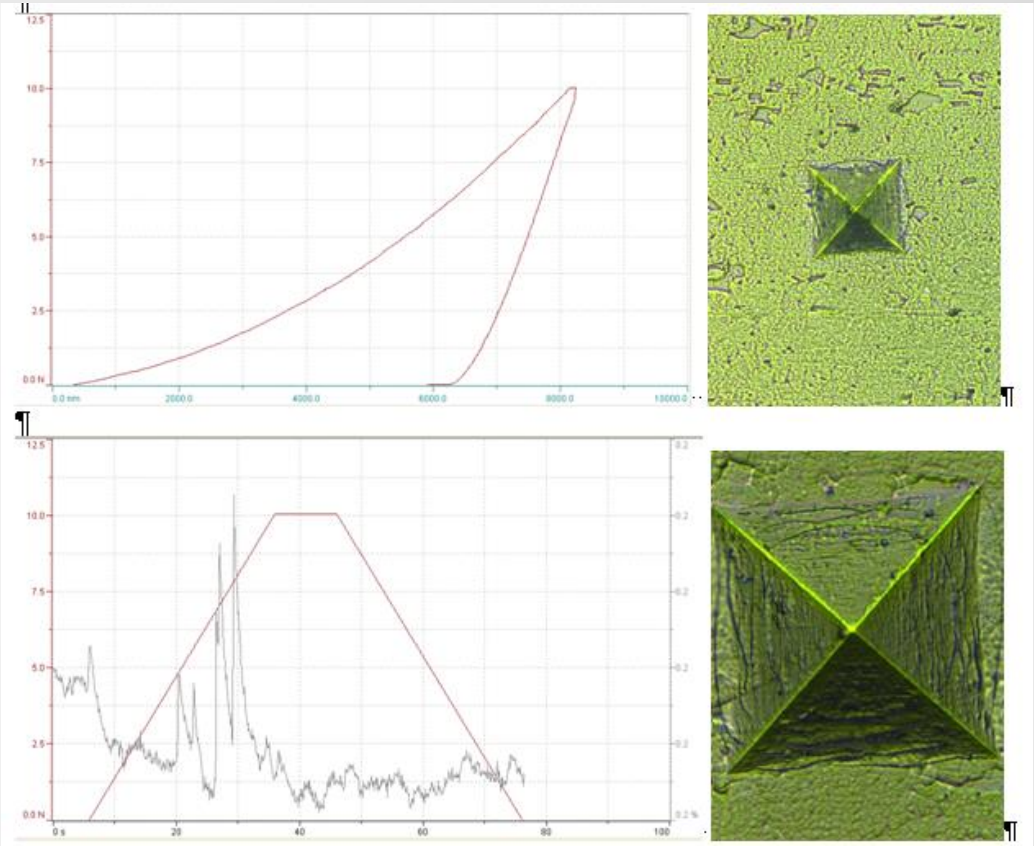
Progressive multicycle

(4 cycles)

Instrument: MCT



Microindentation with Acoustic Emission (AE)



Sample: TiN-on-steel

Indenter: Vickers

Max load: 10 N

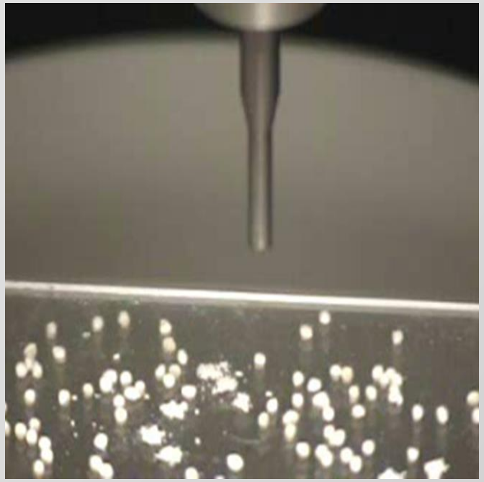
Loading rate: 20 N/min.

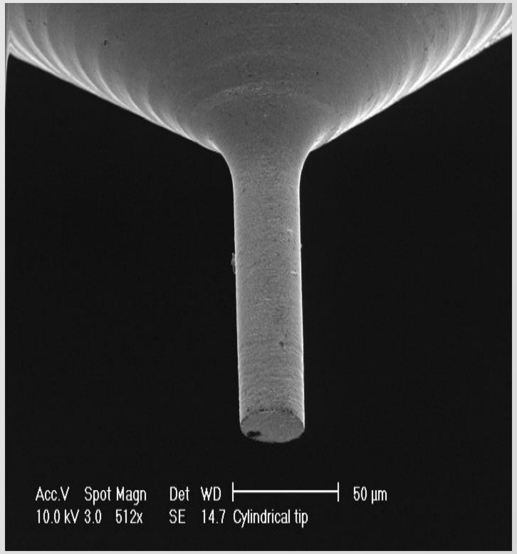
Pause at mx load: 10s

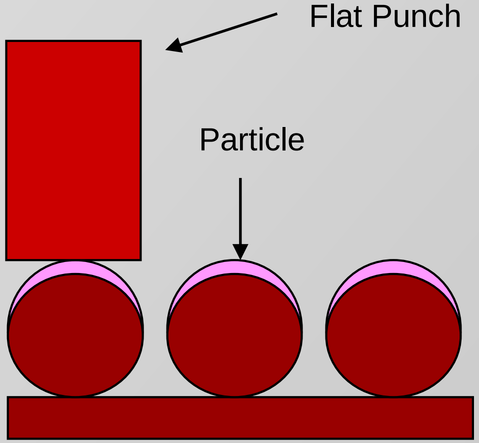
Instrument: MCT

Crush testing of powder particles

- Particles are scattered randomly across glass slide
- Flat punch (e.g. diameter 1.5 mm) used to crush
- Optical microscope used to predefine particle position
- Many particles can be tested with full automation
- Maximum load required to crush particle is recorded

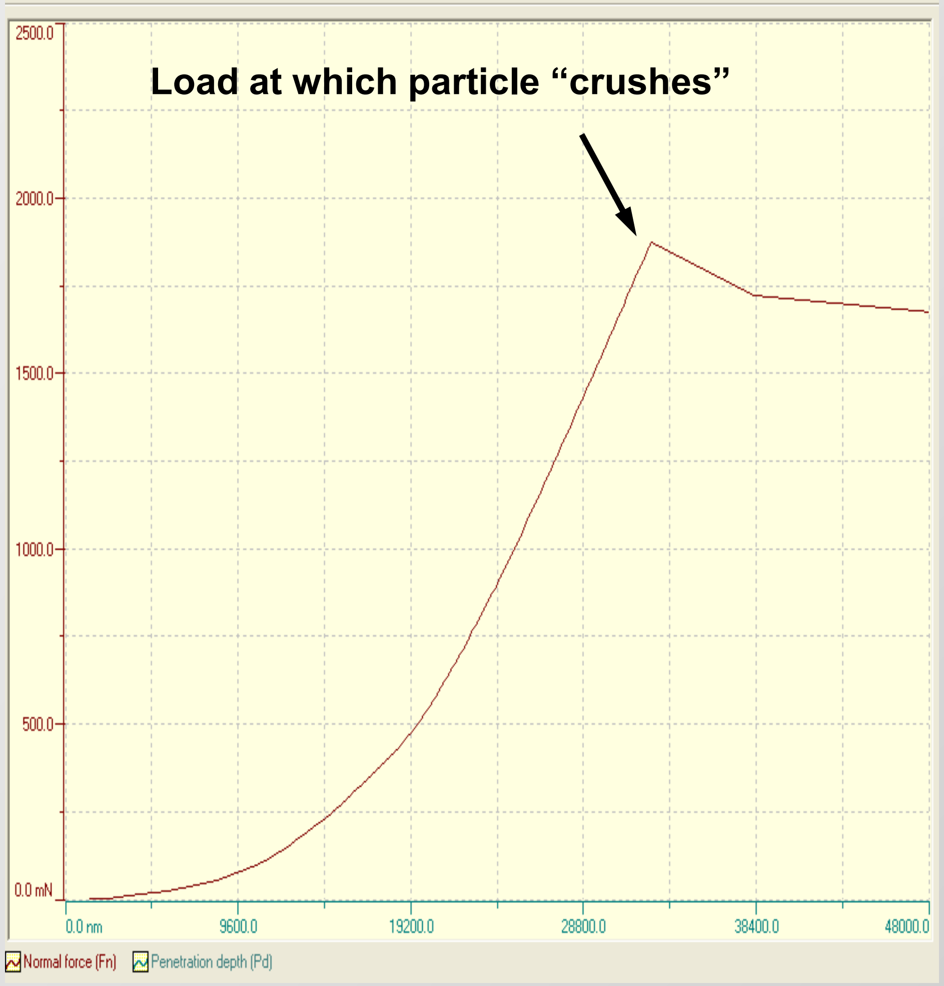






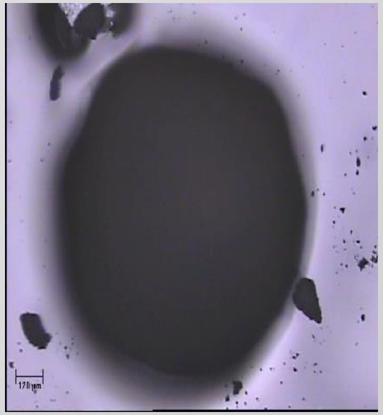
Crush testing of powder particles

Applied Load



Displacement

Before

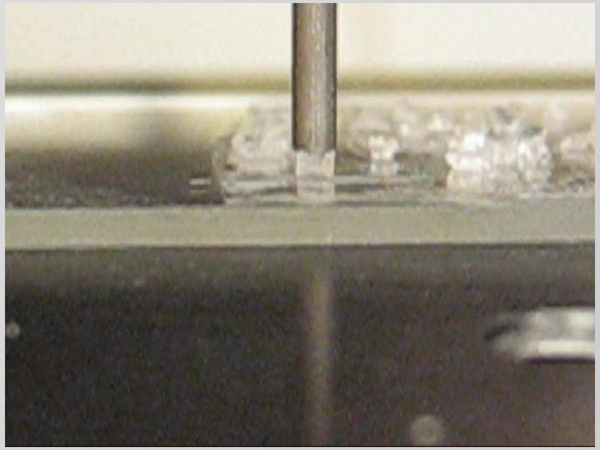


After

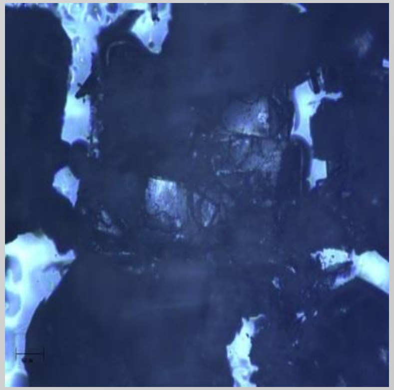
Crush testing of powder particles (movie)

Microindentation of sucrose particles

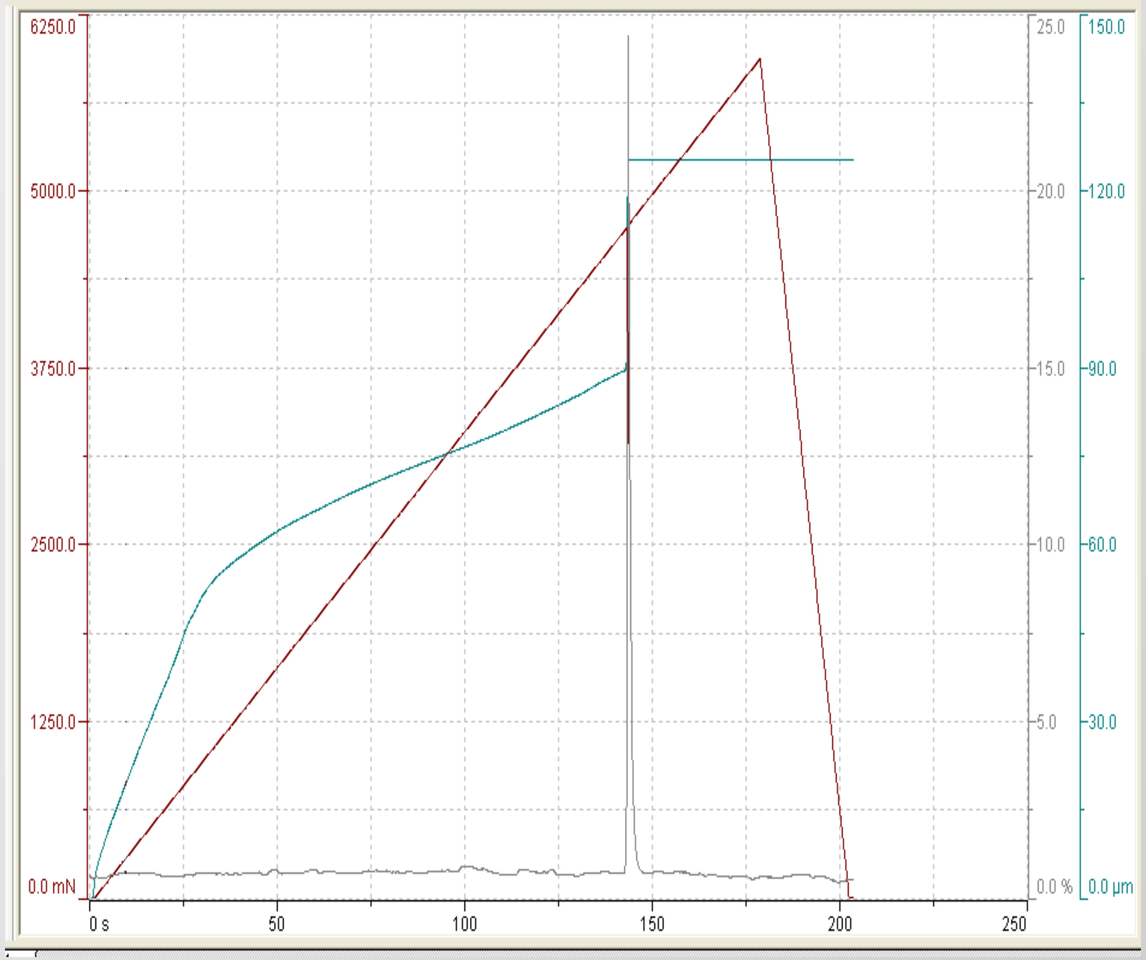
Before



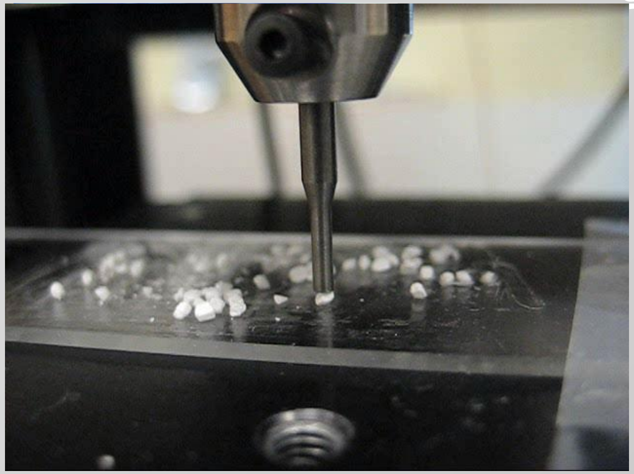
After



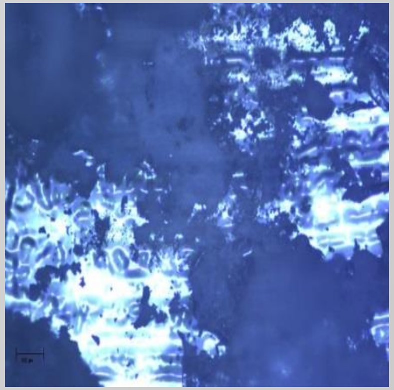
Microindentation of citric acid particles



Before



After



Summary

- Response of material related to E/H
- Elastic recovery vs plasticity, work of indentation
- Spherical indenters for indentation stress-strain behaviour
- Plastic deformation - strain hardening or softening, pop-in events
- Fracture toughness and fracture events (acoustic emissions)
- Mechanical testing – compression testing