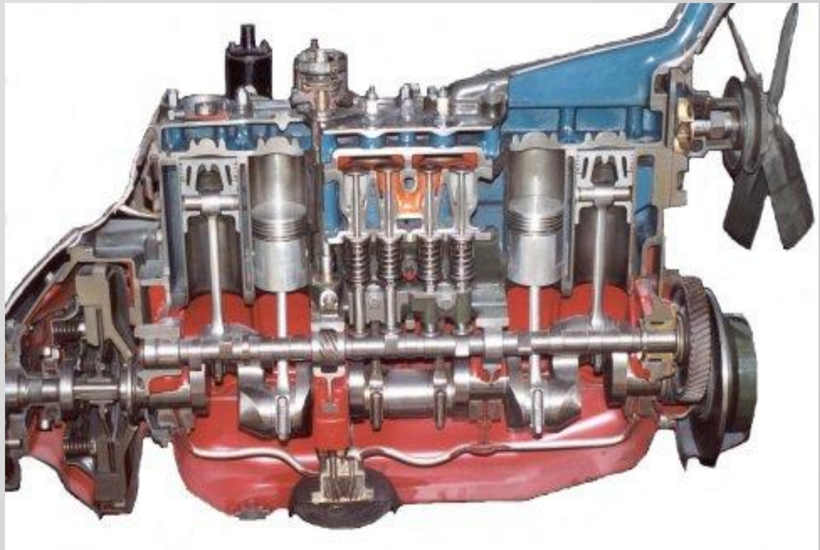
10. Testing of Coatings

Andy Bushby

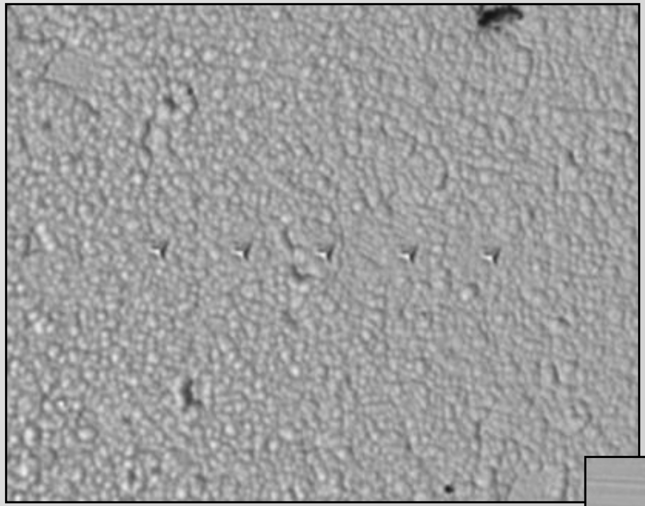
Why do we need to test coatings….?

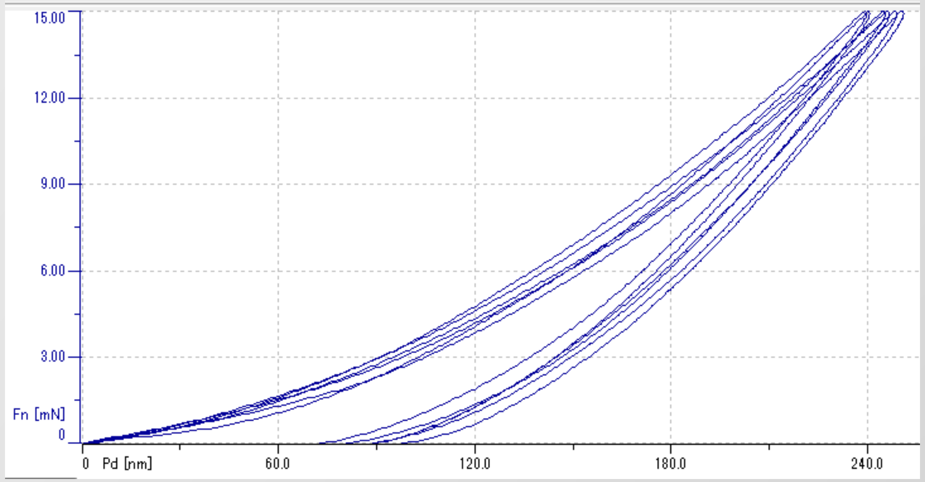
… because they are everywhere !

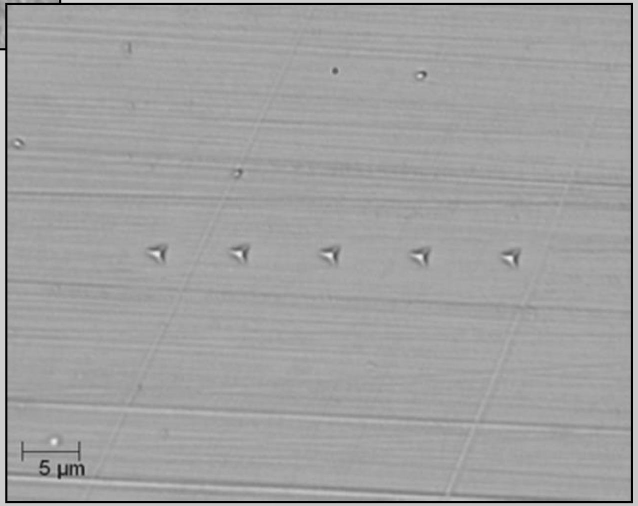


Industrial QC testing of hard coatings

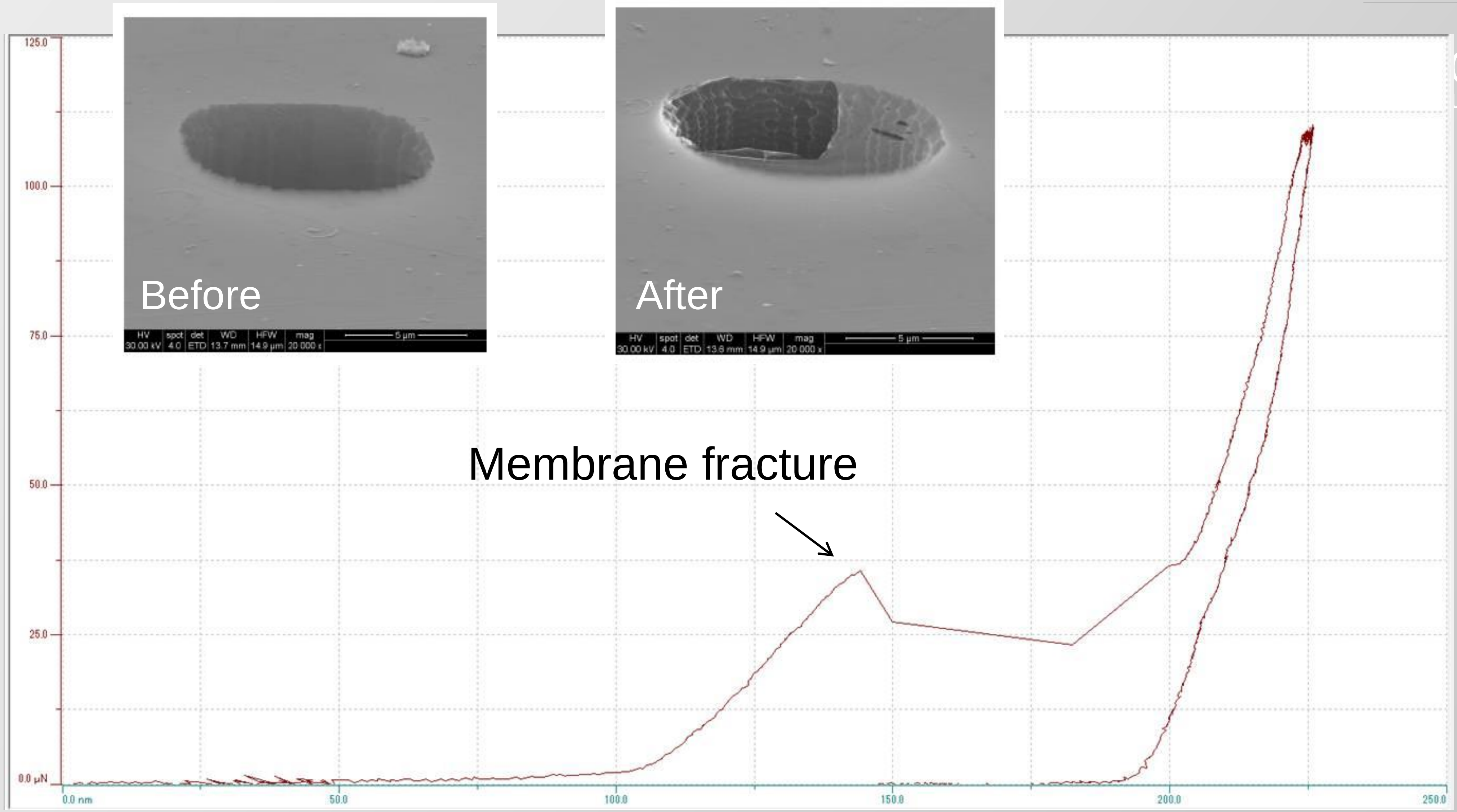
Nanoindentation of DLC-coated piston rings







1 nm thick freestanding graphene membrane

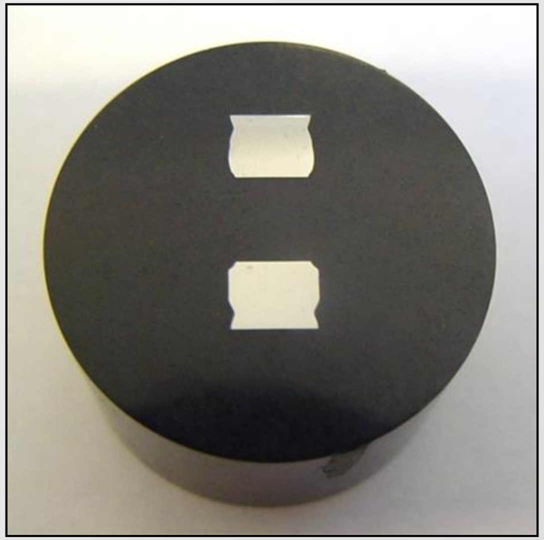


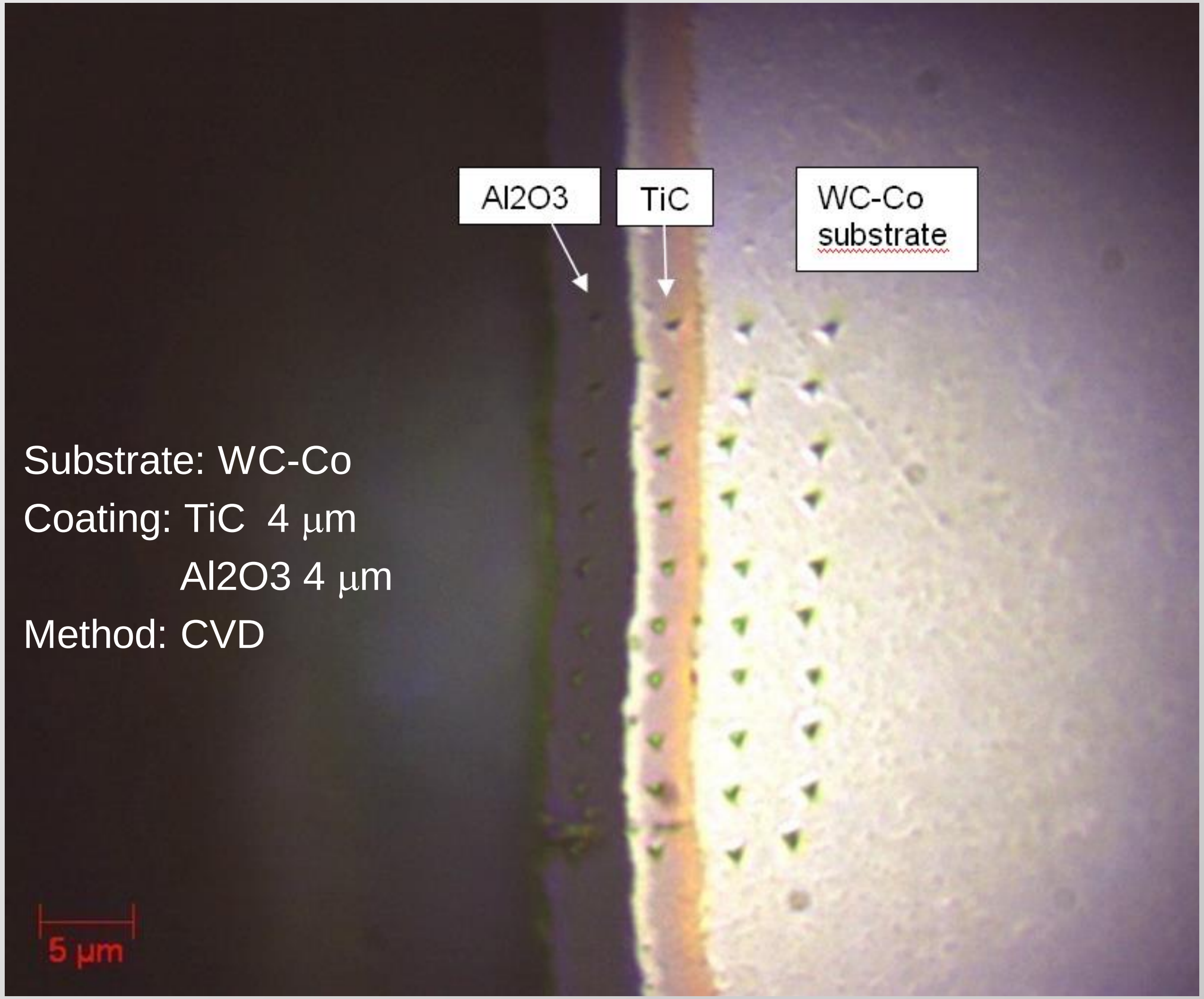
Queen Mary
University of London

Nanoindentation of coating sections

alemnis

Queen Mary
University of London





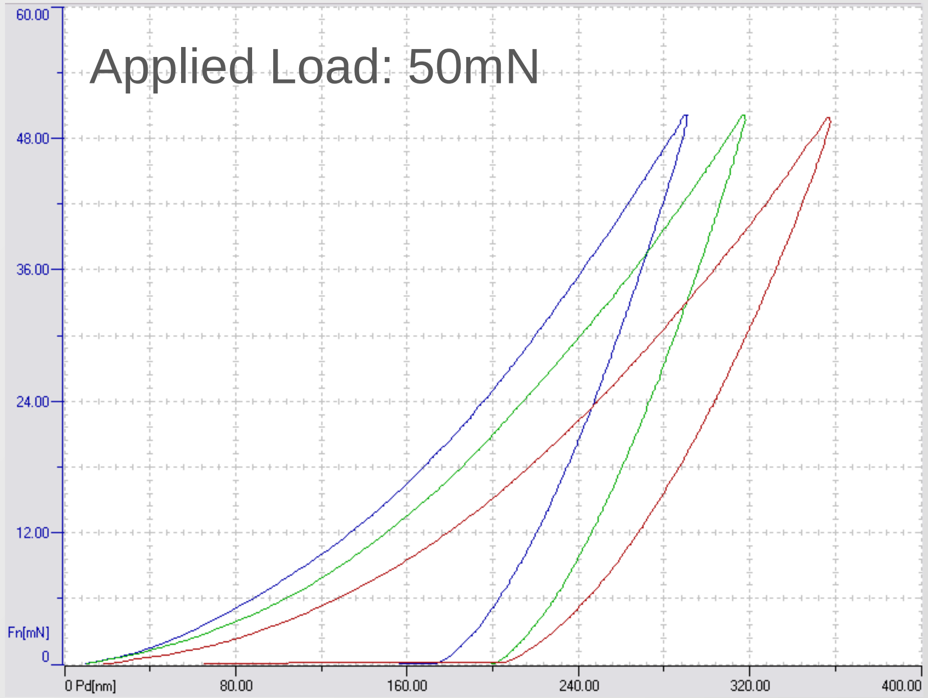
Substrate: WC-Co

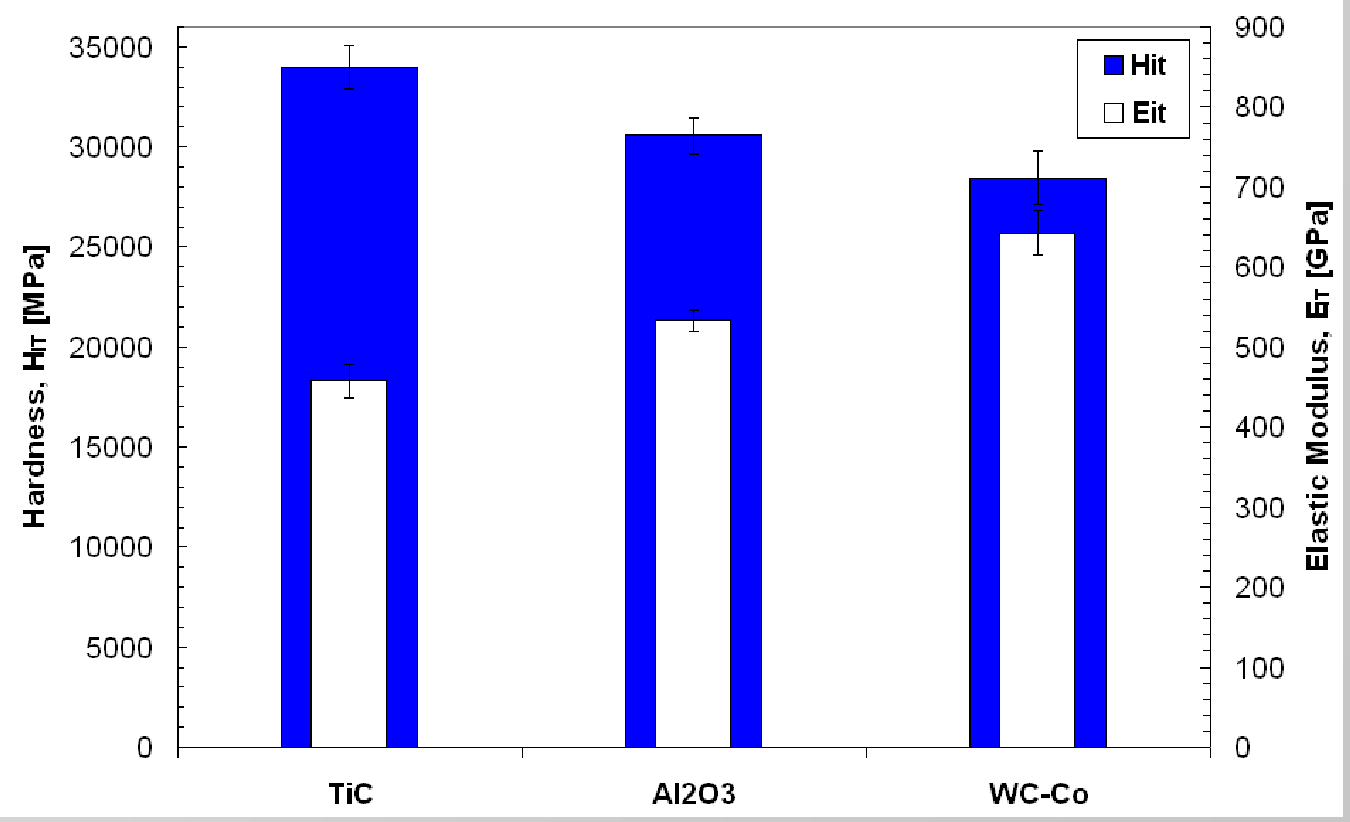
Coating: TiC 4 μm

Al2O3 4 μm

Method: CVD

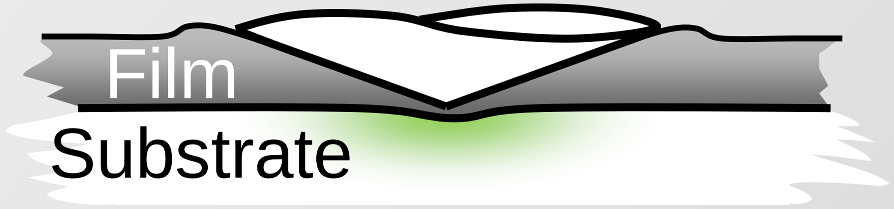
Nanoindentation of coating sections



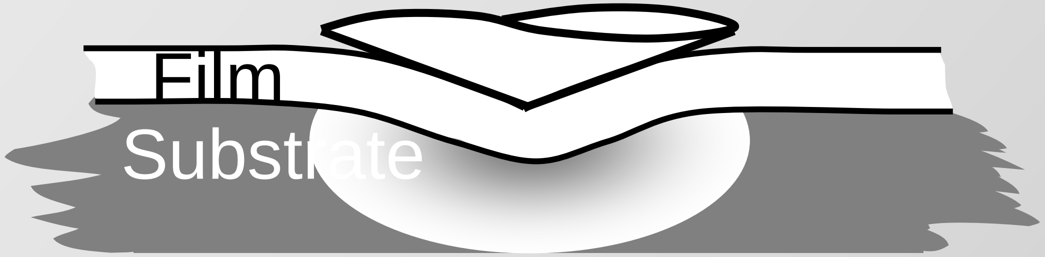


The two extremes of coating-substrate mismatch

Soft film on hard substrate



Hard film on soft substrate

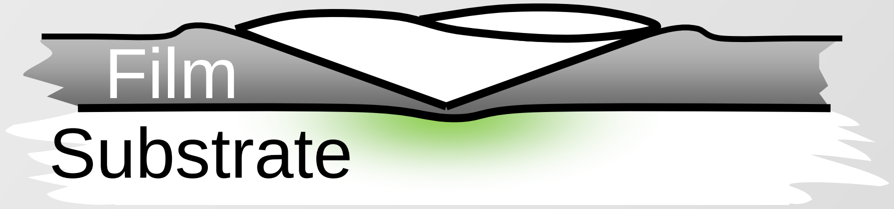


Both the film and substrate respond to the indentation loading

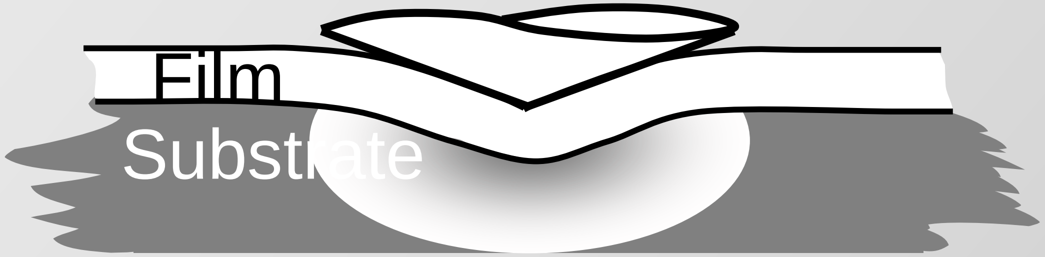
How to we distinguish the film only properties from the combined response?

The two extremes of coating-substrate mismatch

Soft film on hard substrate



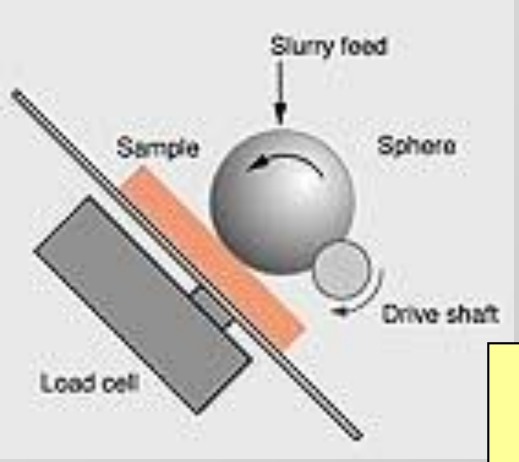
Hard film on soft substrate



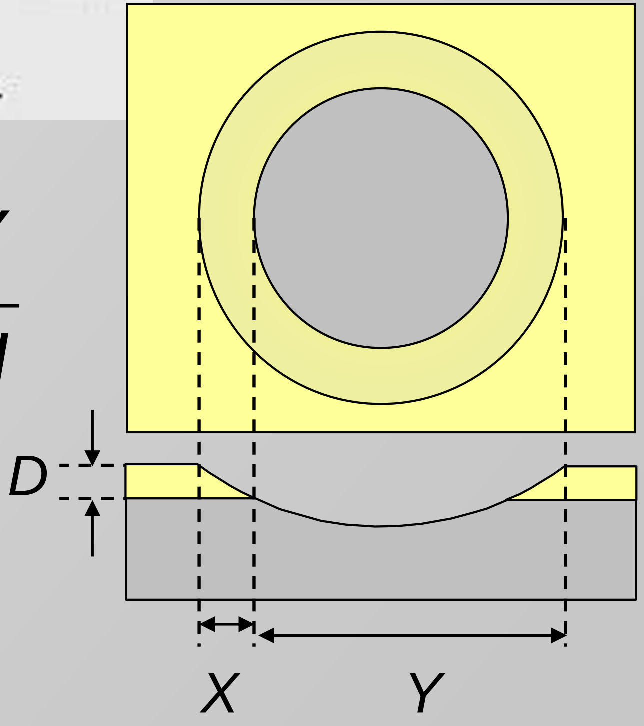
We need to know the film thickness
Often difficult to determine:

- **Cross-sectional microscopy**
- **AFM step-height**
- **XRF**
- **Elipsometry**
- **Surface acoustic wave**
- **Calotest**

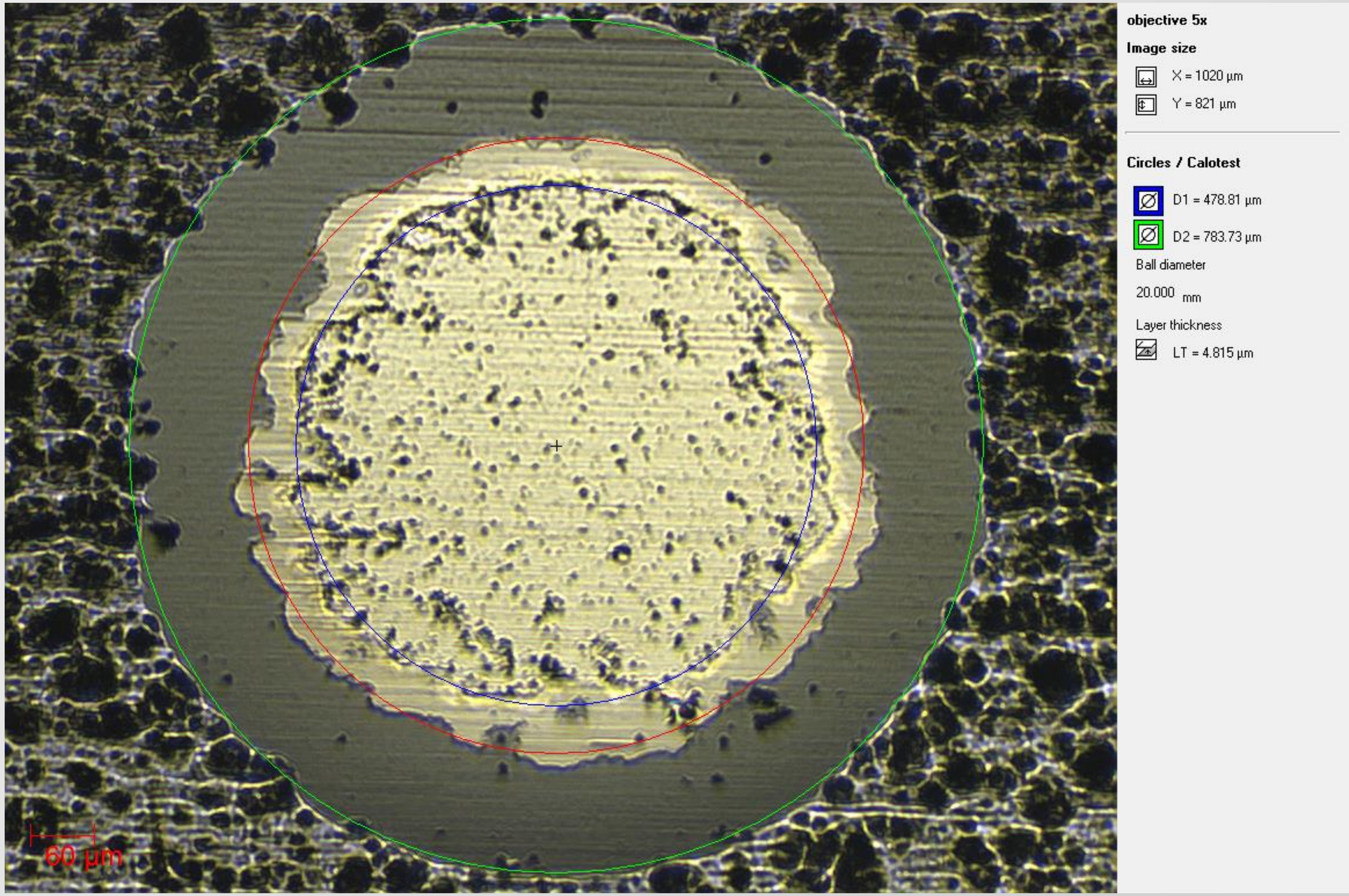
Calotest to measure coating thickness



$$D = \frac{X \times Y}{\phi\, ball}$$



Calotest to measure coating thickness

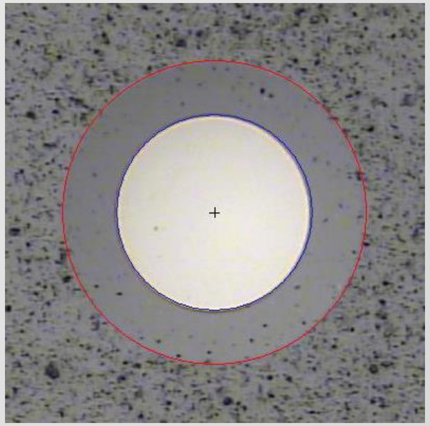


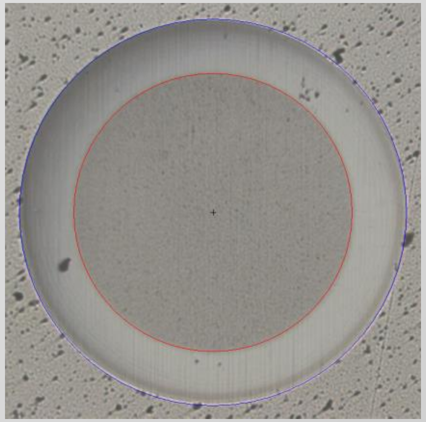
DLC coating of thickness 4.8 µm on piston ring

20 mm ball, 1800s with fine slurry, 900s with super fine slurry

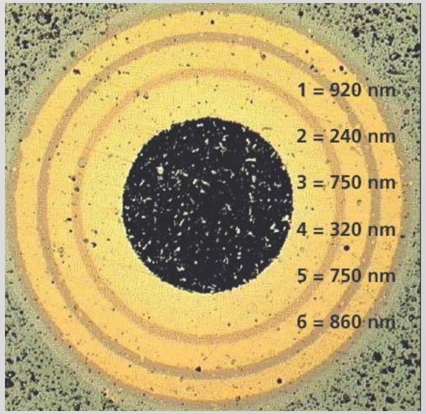
Calotest to measure coating thickness

DLC-on-steel:





TiN-TiCN multi-layers on steel:



alemnis

Queen Mary
University of London

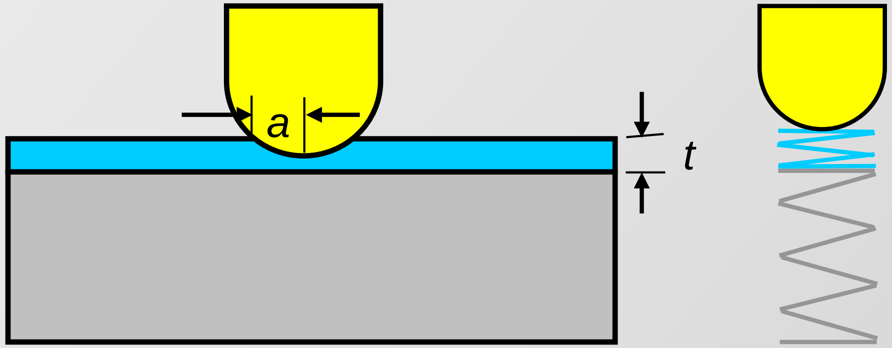
Consider elastic and plastic properties separately

- Require different approaches to measure *E* and *H*
- Unlikely to measure both *E* and *H* in the same test
- Cannot measure coating properties from a single test
- What do you actually want to know about the coating-substrate system?

How can you know that you are separating film only properties?

- Changing the indentation geometry
- Have you actually measured the coating hardness?
- How do you know if the film has cracked?

Coatings on a substrate



$$E^*_{\mathrm{IT}} = E'_{sub} + \left(E'_{film} - E'_{sub}\right)\ f\left(\frac{a}{t}\right)$$

Menčík *et al*. JMR, **12**, 2475 (1997)

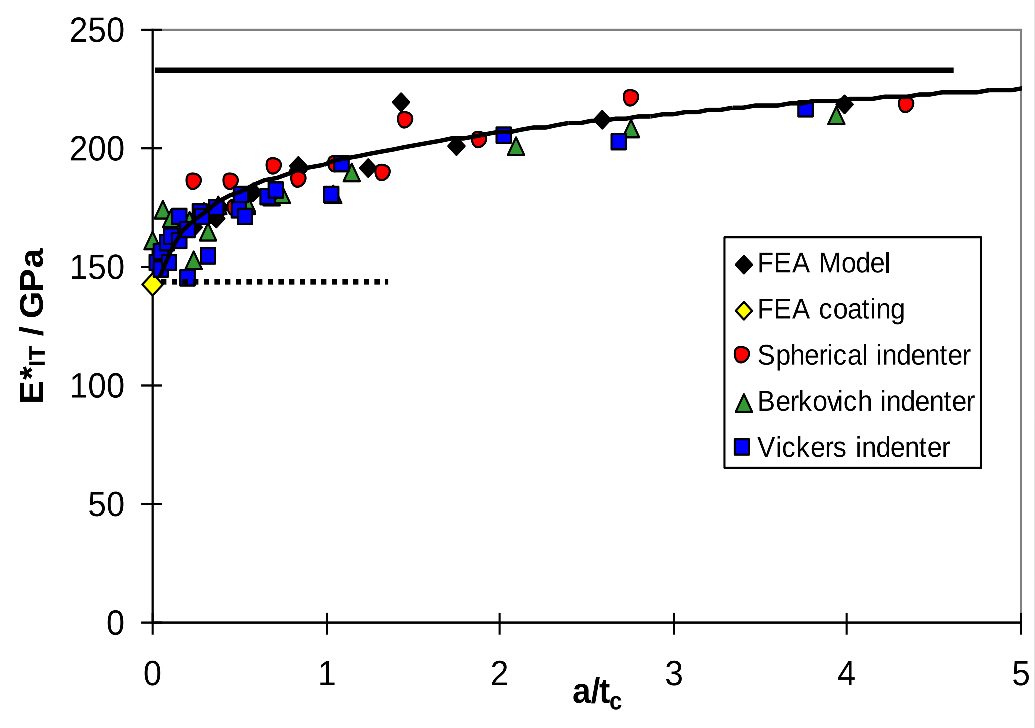
Elastic modulus

The elastic response is always a combination
of the film and substrate

Collect data over a range of relative depths (*a/t* or *h/t*)
to understand the combined response
and project data to zero depth to obtain ‘film only’ properties

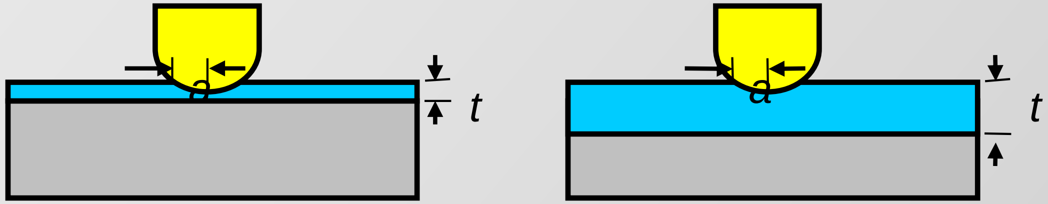
Elastic response – dlc on tool steel

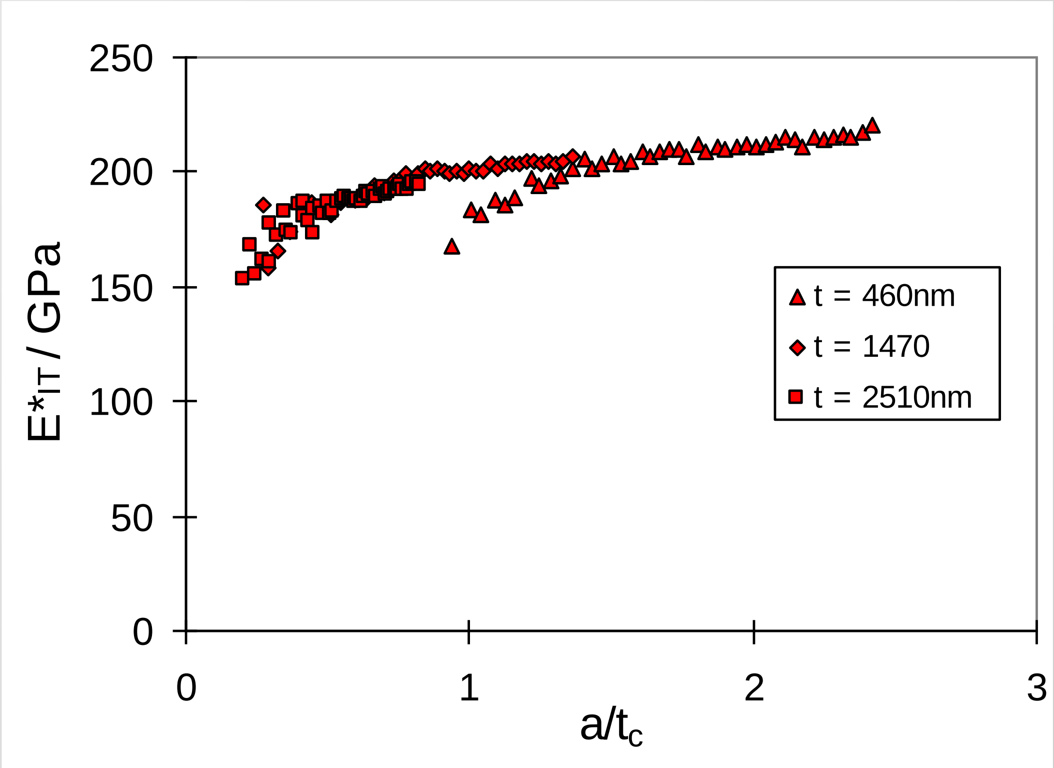
t_c = 460nm, 1470nm and 2510nm, 3 indenter geometries



Extrapolate for *a/t* < 1 to get estimate of coating only properties
Requires data to be collected in this range
Need to know coating thickness, *t*

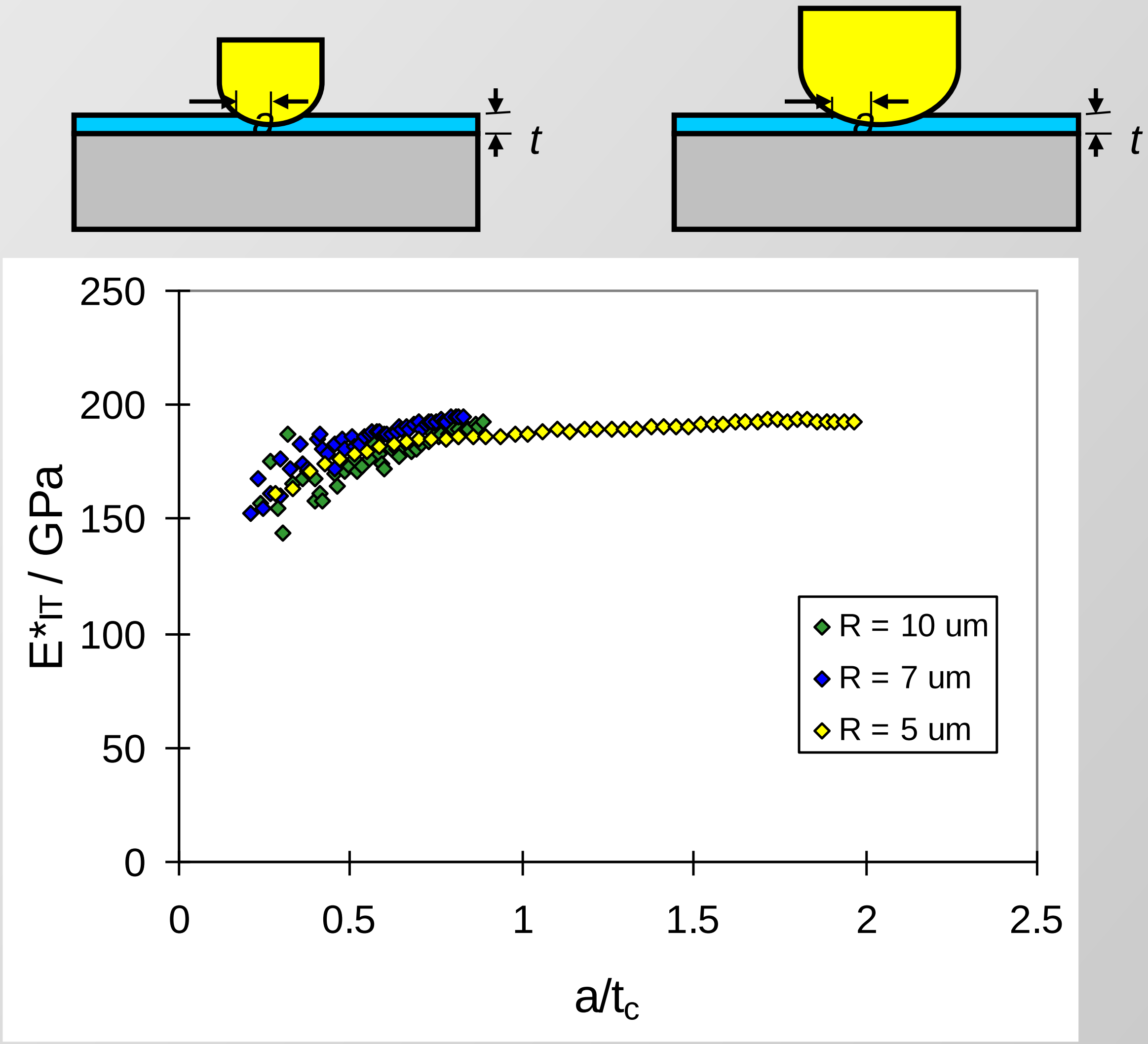
Vary a/t by film thickness





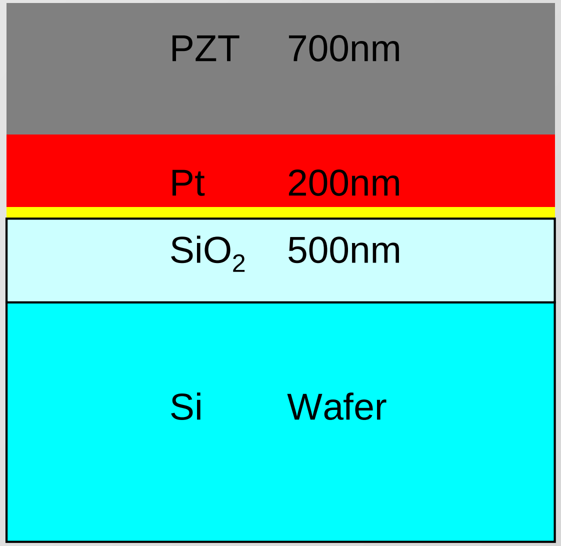
Spherical indenter R = 7 μm, partial unloading

Vary a/t by indenter radius



t = 1470nm,   partial unloading

Multi-coating response



E = 120 GPa

E = 190 GPa

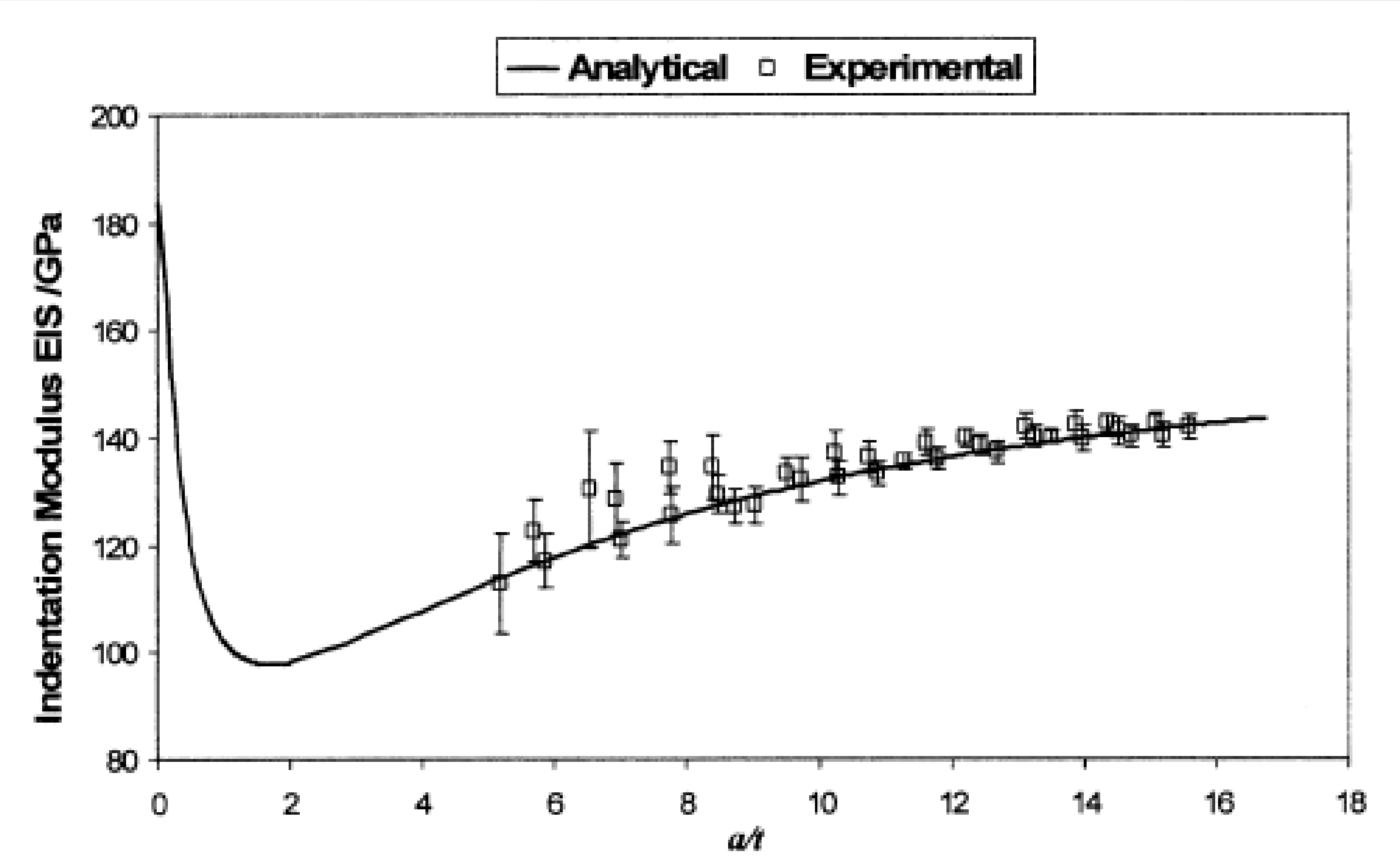
E = 65 GPa

E = 180 GPa

Analytical model of Hertzian indentation displacement

$$h_{Herztain} = \frac{3}{8}F\left(\frac{2(-1+v_1^2)}{aE_1} - \frac{\alpha}{\left(a^2E_1\pi t_1\sqrt{a^2+t_1^2}\right)} + \frac{\gamma}{\left(a^2E_2\pi t_1\sqrt{a^2+t_1^2}\right)} - \frac{\varphi}{\left(a^2E_2\pi t_2\sqrt{a^2+t_2^2}\right)} + \frac{\lambda}{\left(a^2E_3\pi t_2\sqrt{a^2+t_2^2}\right)} - \frac{\mu}{\left(a^2E_3\pi t_3\sqrt{a^2+t_3^2}\right)} + \frac{\psi}{\left(a^2E_4\pi t_3\sqrt{a^2+t_3^2}\right)}\right) \quad ...(9)$$

Multi-coating response

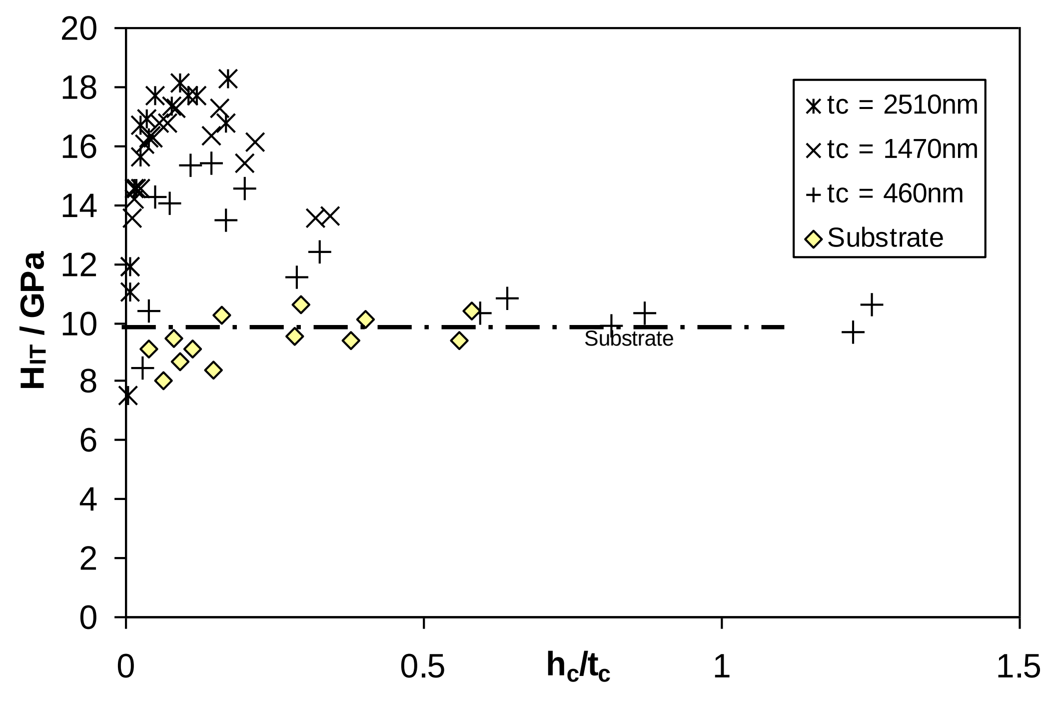


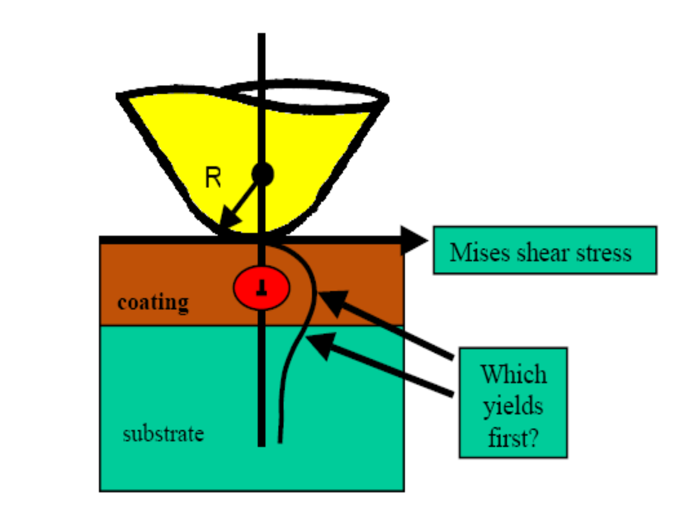
Complicated elastic response of multilayer films
Complex combination of moduli and layer thicknesses
- not simple to get the properties of the top layer

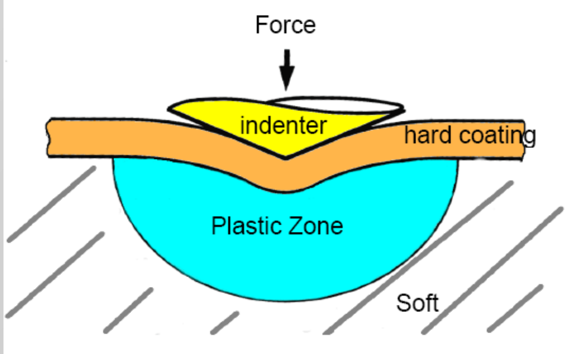
Chibisi Chima-Okereke *et al*. 2006

Plastic response – dlc on tool steel

Example for dlc coatings on steel substrate

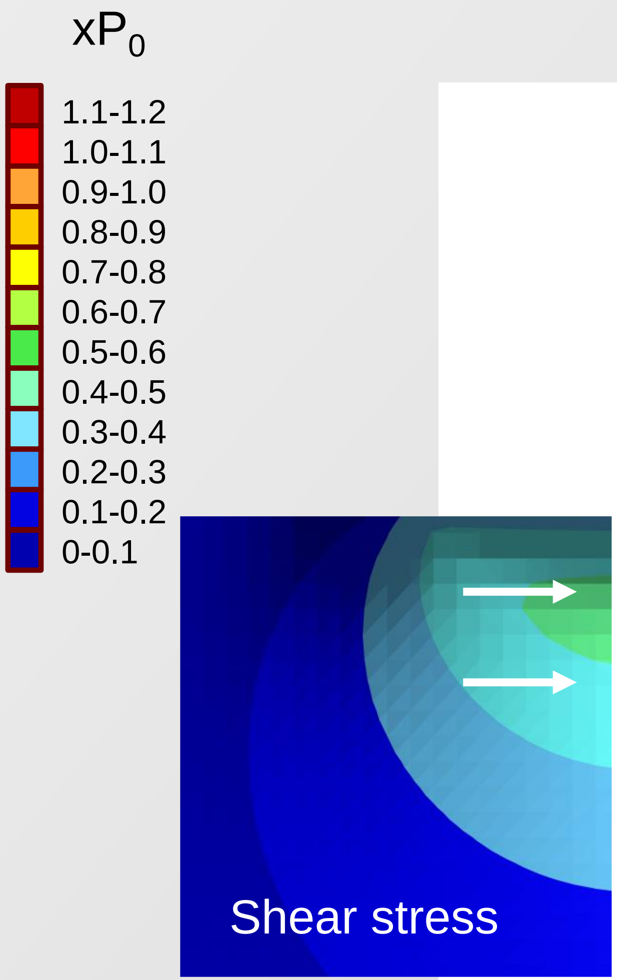


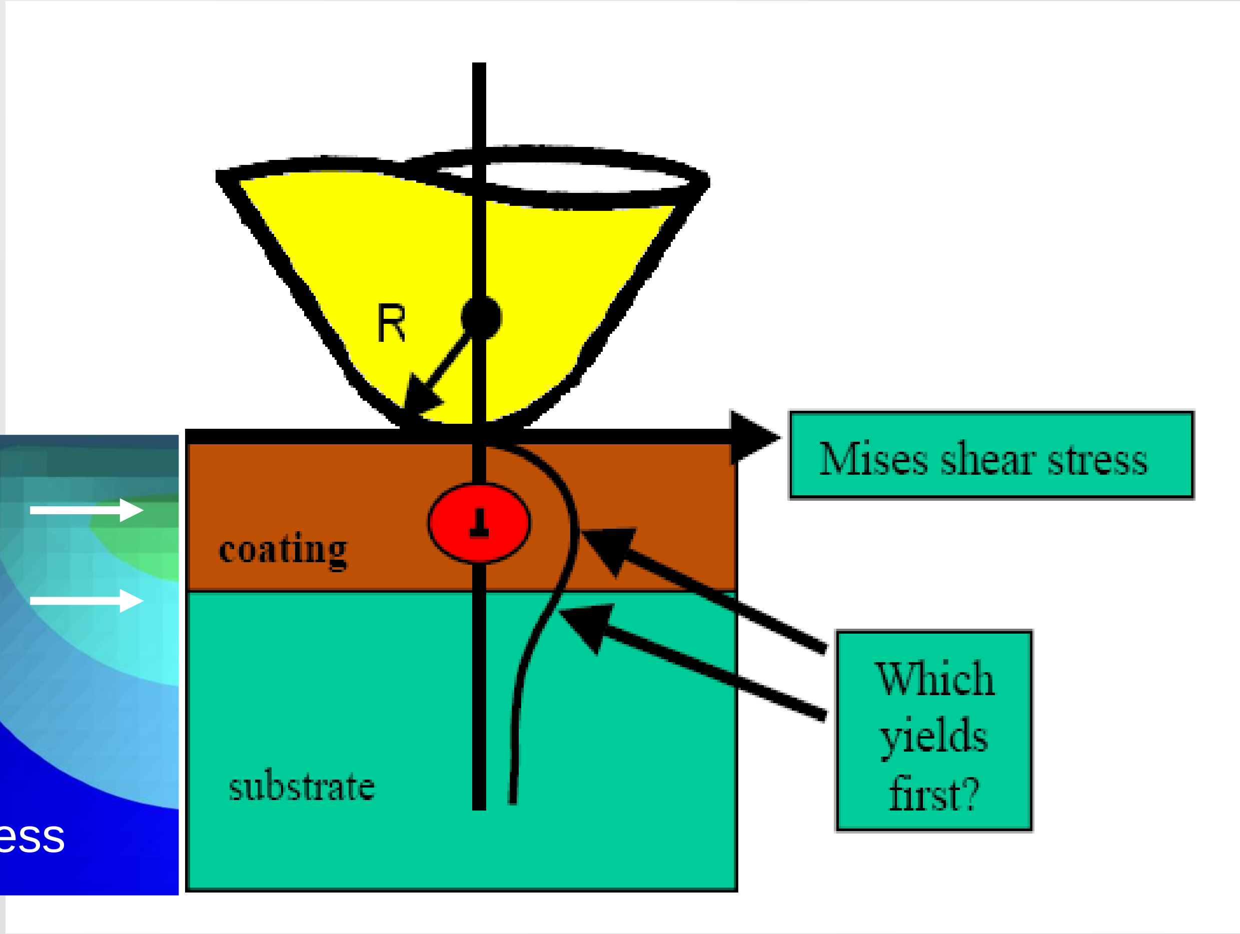


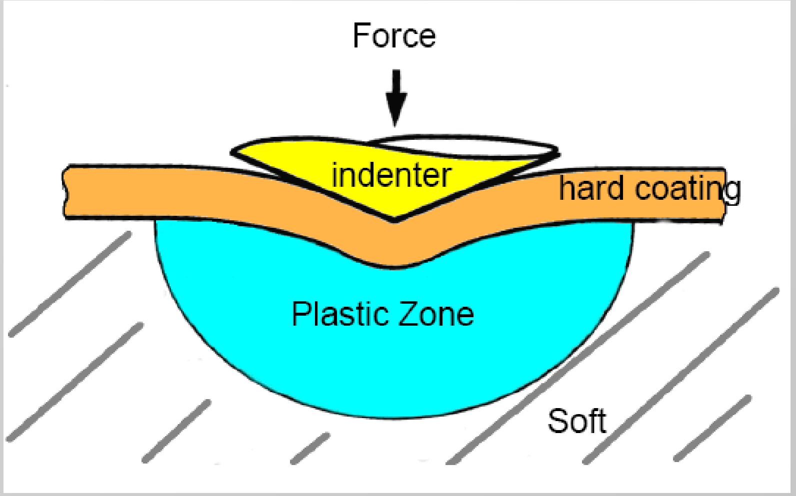


Requires a sharp indenter, i.e. small tip radius, *R.*

Plastic response – substrate yield





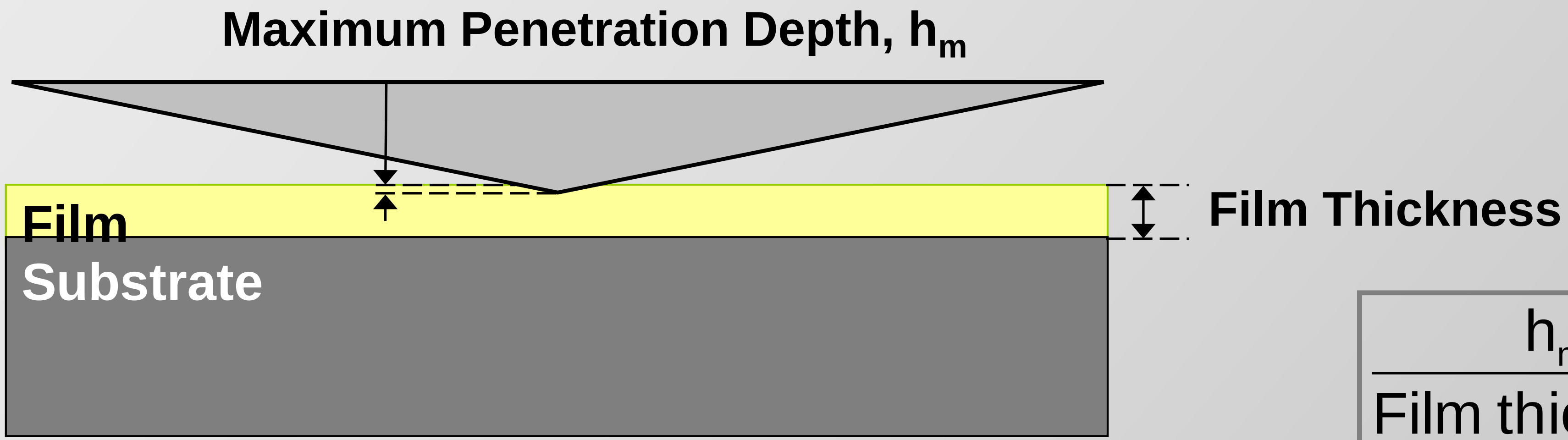


Requires a sharp indenter, i.e. small tip radius, *R*.

The "10% rule"

Developed for hardness of ductile electroplated metal coatings

– that are tens of micrometres in thickness

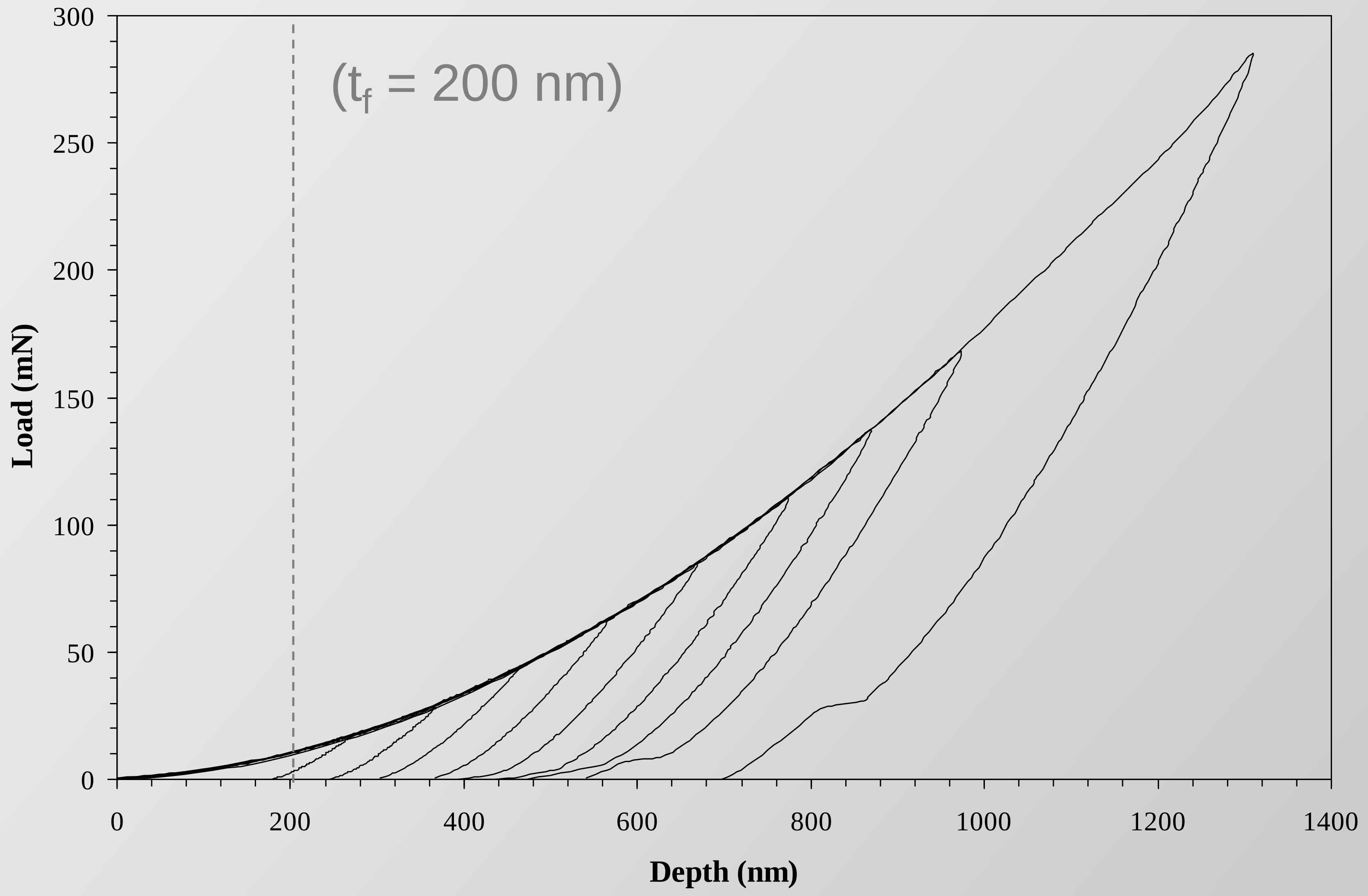


$$\frac{h_m}{\text{Film thickness}} \leq 10\%$$

NOT a general rule for indentation measurement!

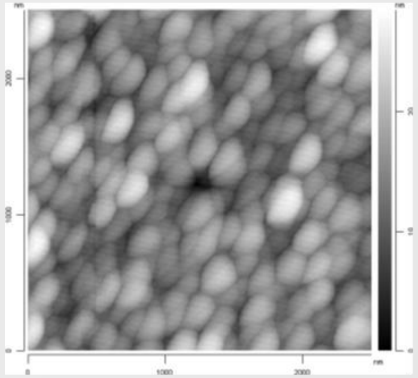
NOT ever applicable to elastic modulus!

Ti-on-Si (Penetration depths 25 - 1225 nm)

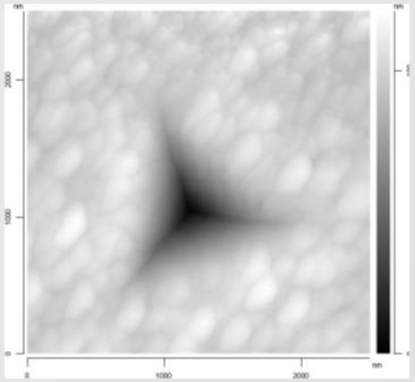


N. X. Randall and C. Julia-Schmutz, *Evolution of contact area and pile-up during the nanoindentation of soft coatings on hard substrates*, MRS Symposium Proceedings, 522 (1998) 21-26

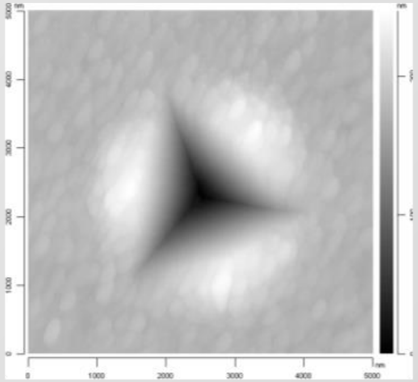
Ti-on-Si



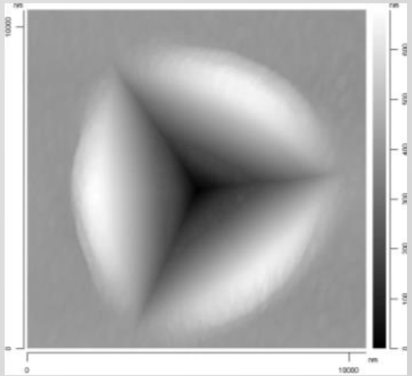
25 nm



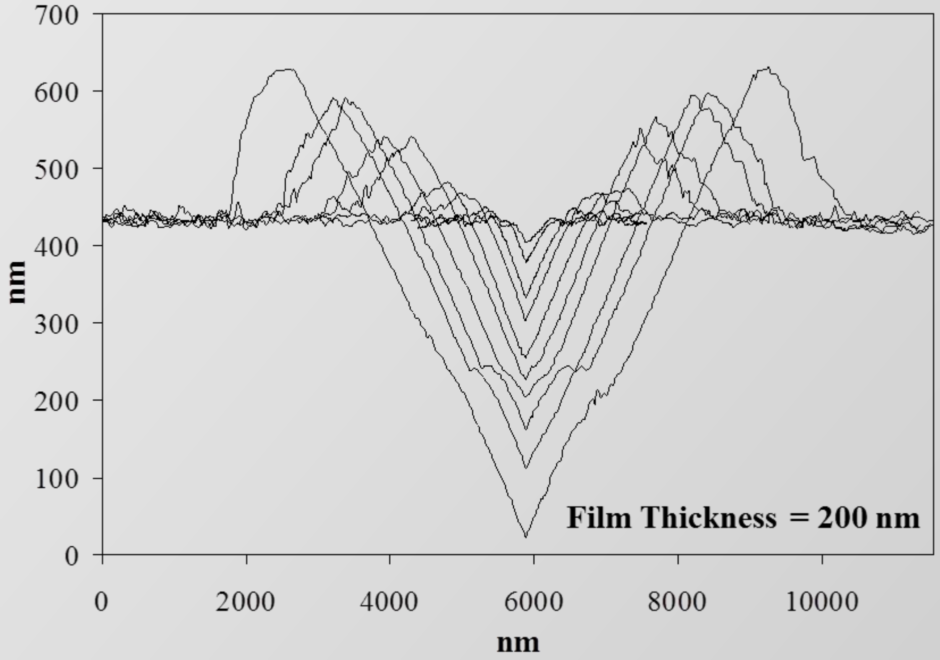
200 nm



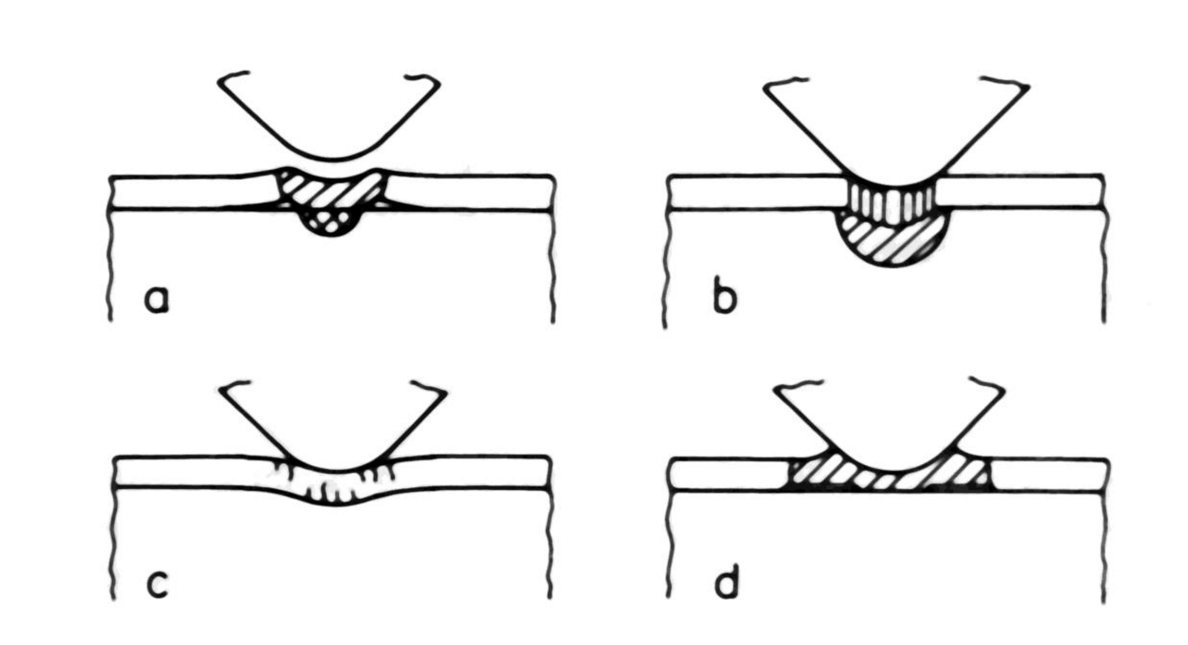
400 nm



1225 nm



Fracture and delamination



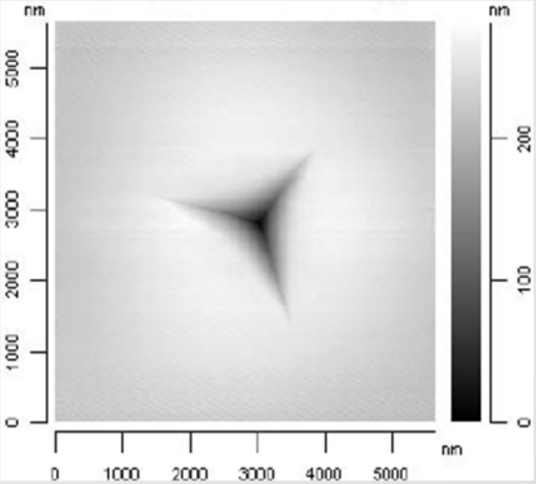
Different types of crack can be identified
from the indentation response (change in stiffness)

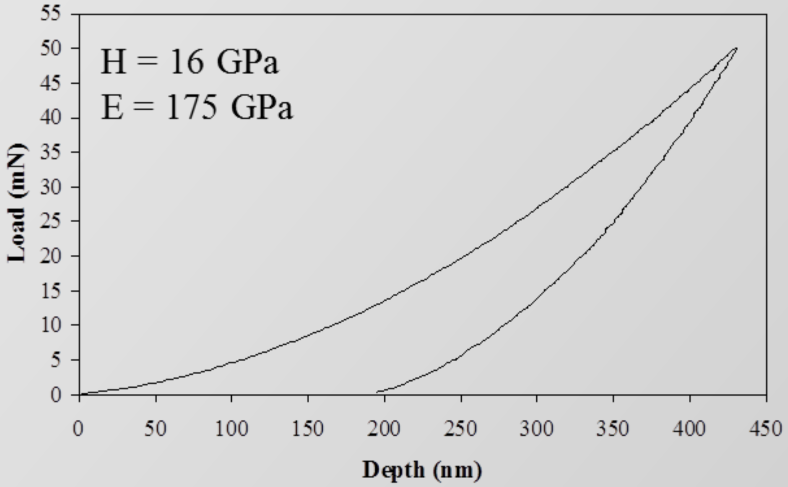
Nanoindentation of Cr_2O_3 thin films

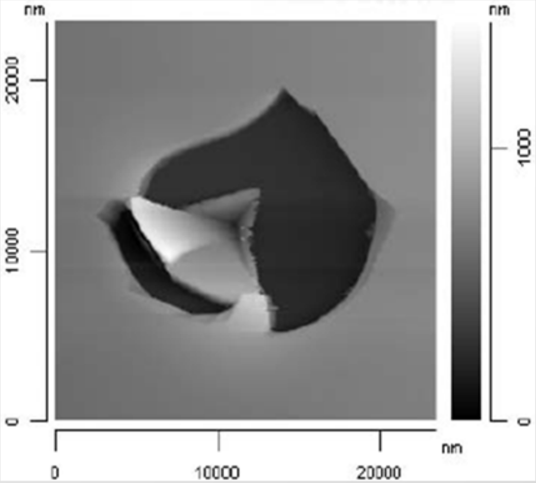
alemnis

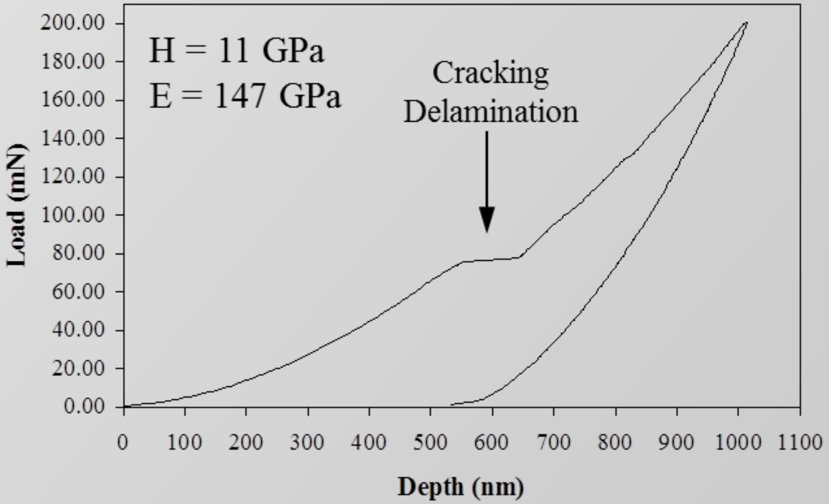
Queen Mary
University of London

Using indentation to characterize delamination:









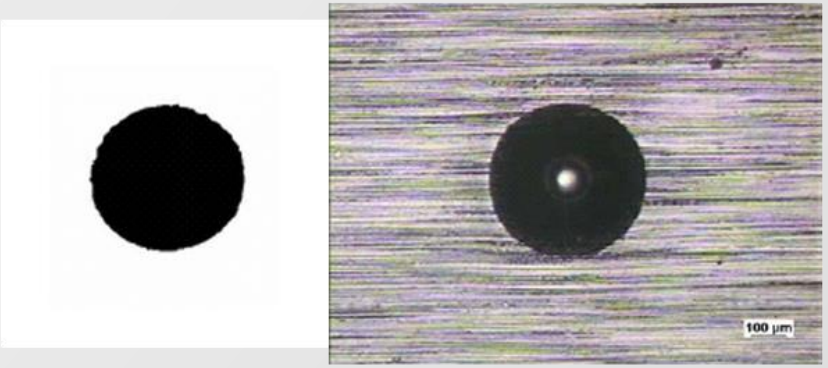
P. Hones, F. Levy and N. X. Randall, *Influence of deposition parameters on mechanical properties of sputter-deposited Cr_2O_3 thin films*, J. Mater. Res. , Vol. 14, No. 9 (1999) 3623-3629

Rockwell indentation for testing hard coatings

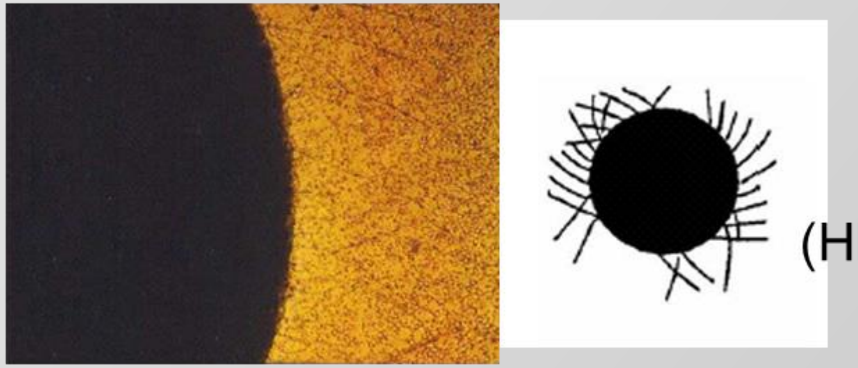
alemnis

Queen Mary
University of London

Class 0
Entirely plastic indent without any visible brittle damage

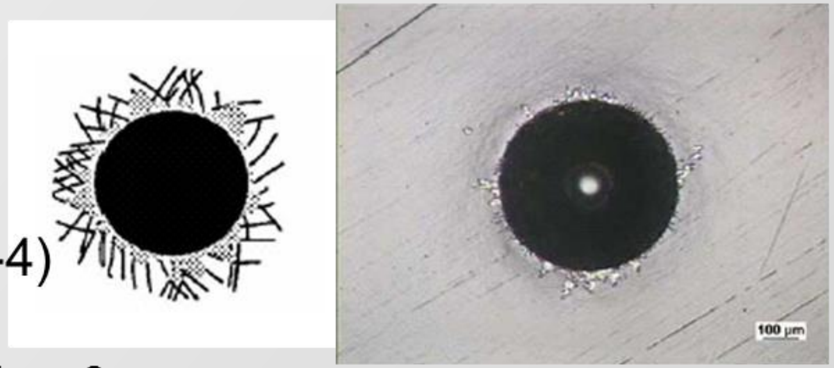


Class 1
First cracking damage (radial or tangential cracks) on indent edge

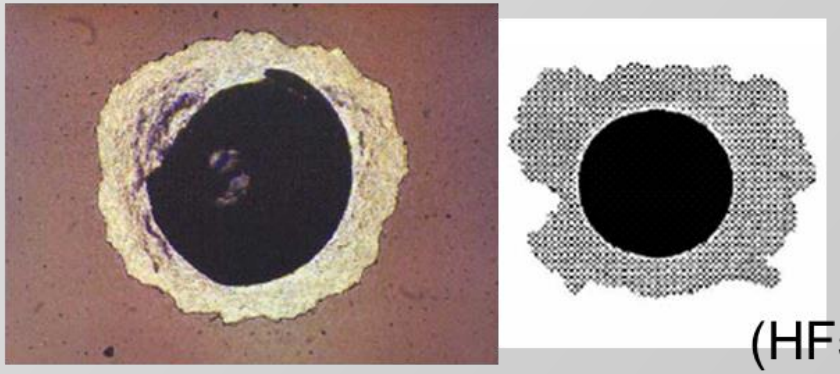


(HF1-2)

(HF 3-4)



Class 2
First minor chipping, initial delamination around indent



(HF5-6)

Class 3
Large area spallation, mostly outside of the indent

Summary

Testing of coatings

- Coatings are used everywhere in modern engineering!
- Consider elastic and plastic responses separately
  Need to make a series of measurements at different loads / depths
- Indentation **elastic** response is ALWAYS a combination of film and substrate
- To measure the **hardness** of the coating – it must yield (before the substrate does)
  Use a sharp tip to ensure yield in the coating
  Sometimes impossible to measure coating hardness
- Remember all of the indentation responses can occur
  e.g. surface roughness, pile-up, cracking, etc