4. Selection of test parameters

Andy Bushby

How do we set up a test?

Indentation golden rule # 3

- know your sample

Indentation golden rule # 2

- know your instrument

Understand how the 2 will interact

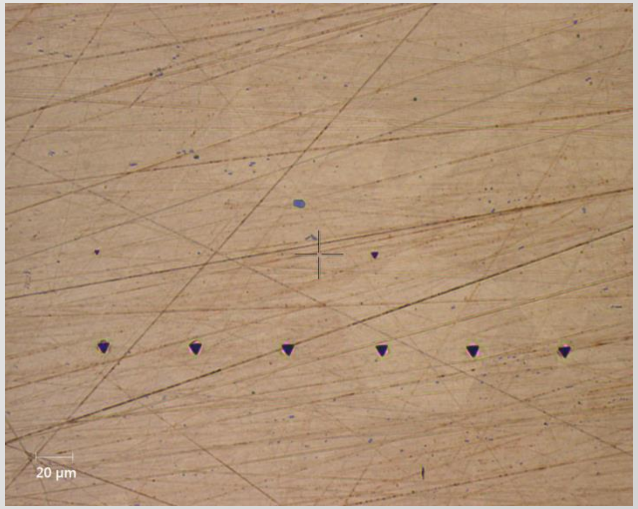
- set machine parameters
- anticipate the response of the 'system'

Take a sample – what is it like?

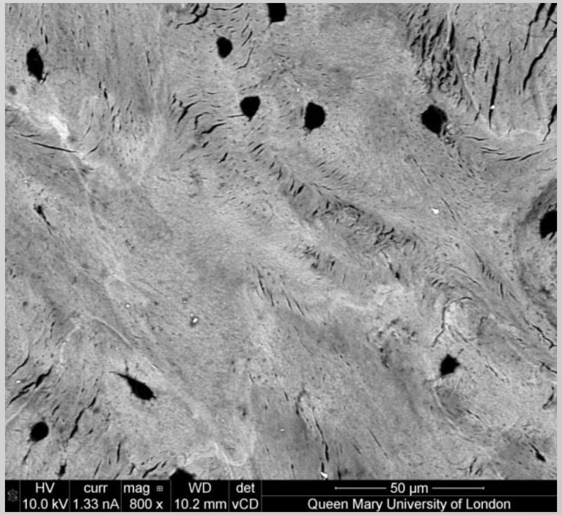
Characteristics

- hard or soft?
- flat or curved?
- polished or rough (on what scale?)
- homogeneous or composite?
- clean surface - how?

Look at it under a microscope



Copper – homogeneous scratched



Bone – inhomogeneous

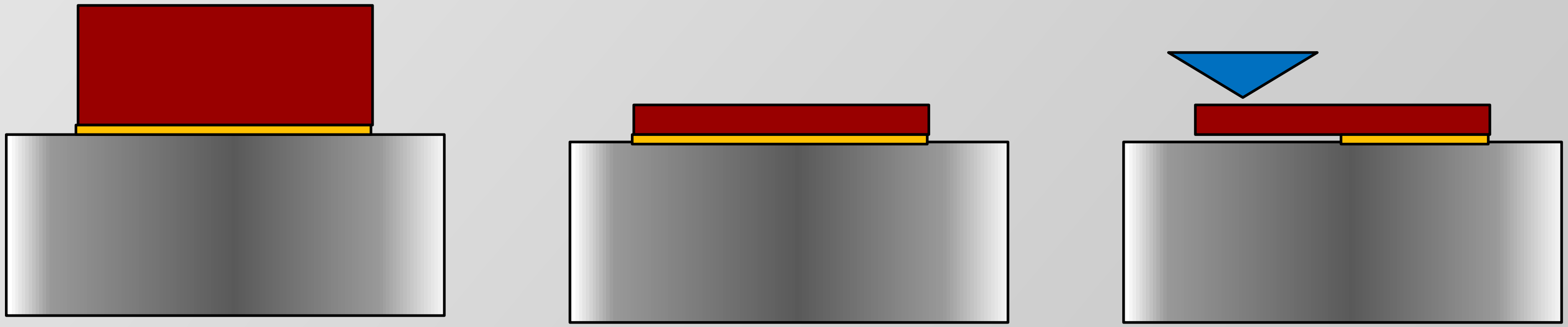
How will it be mounted?

Mounting method

- glue or wax
- embedded in resin
- under liquid
- at high temperature

Considerations

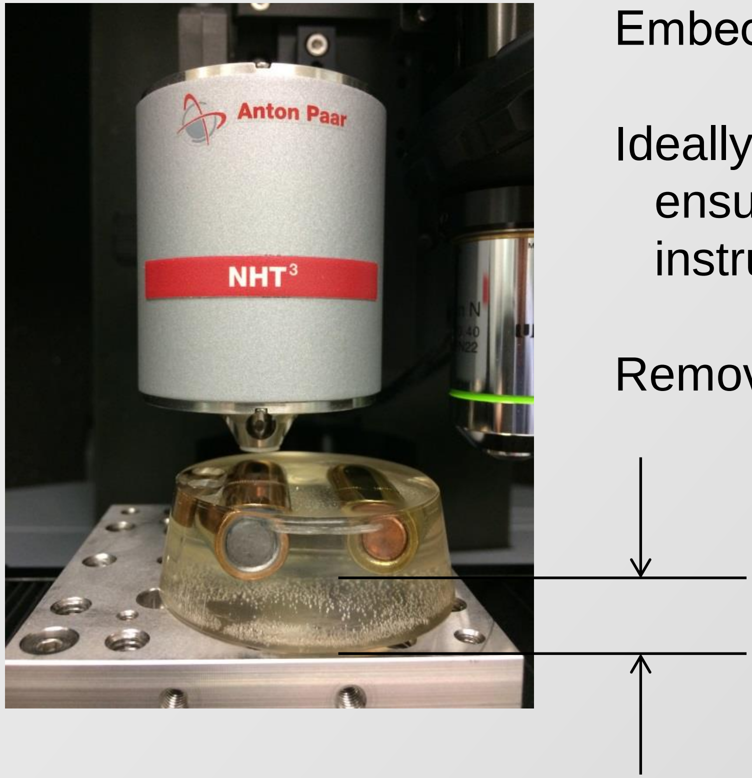
- supported underneath – uniformly?
- thickness of compliant layer to sample thickness < 6%
- changing over time?



Sample mounting issues: compliance

alemnis

Queen Mary University of London

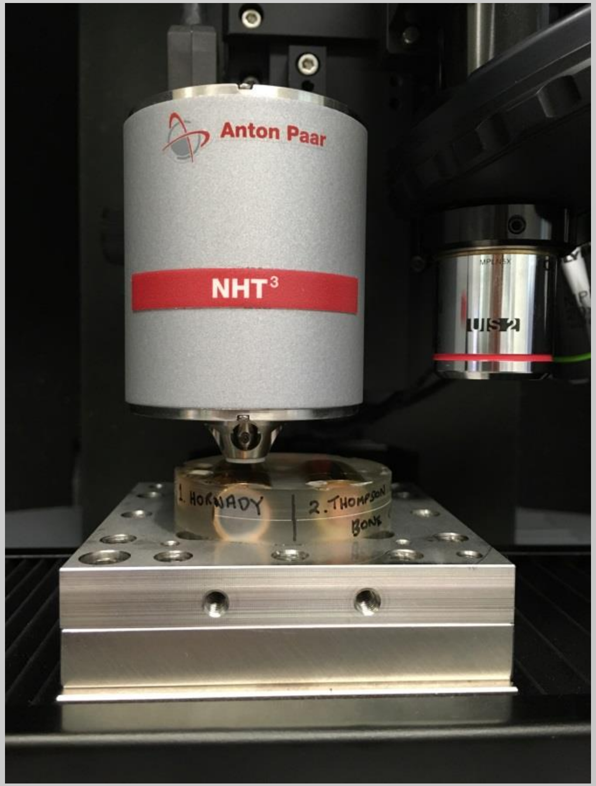


Embedded sample should not be “floating” in epoxy

Ideally, backside should be removed by machining to ensure rigid contact between sample underside and instrument platten

Removal of excess epoxy performed easily in a lathe:

After machining

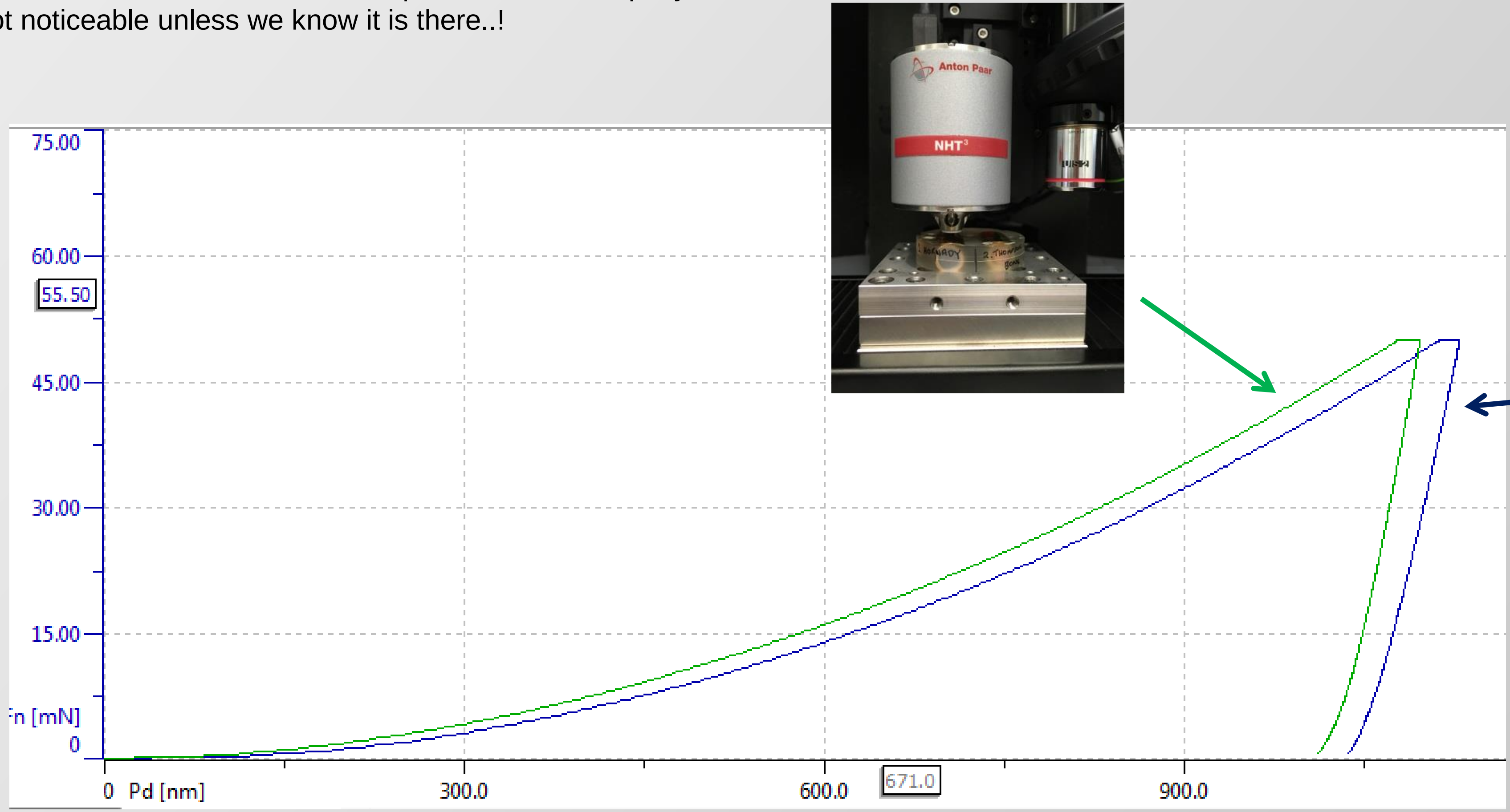


Sample mounting issues: compliance

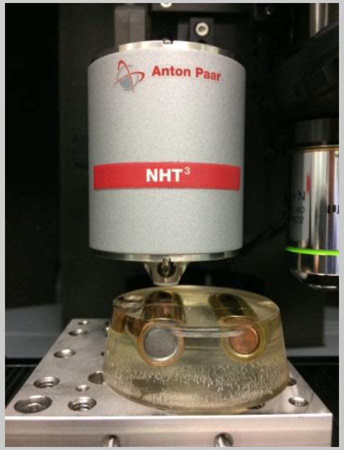
How does this affect the results?

- Small shift in the curve due to compliance from the epoxy
- Not noticeable unless we know it is there..!

After machining



Before machining



Sample mounting issues: compliance

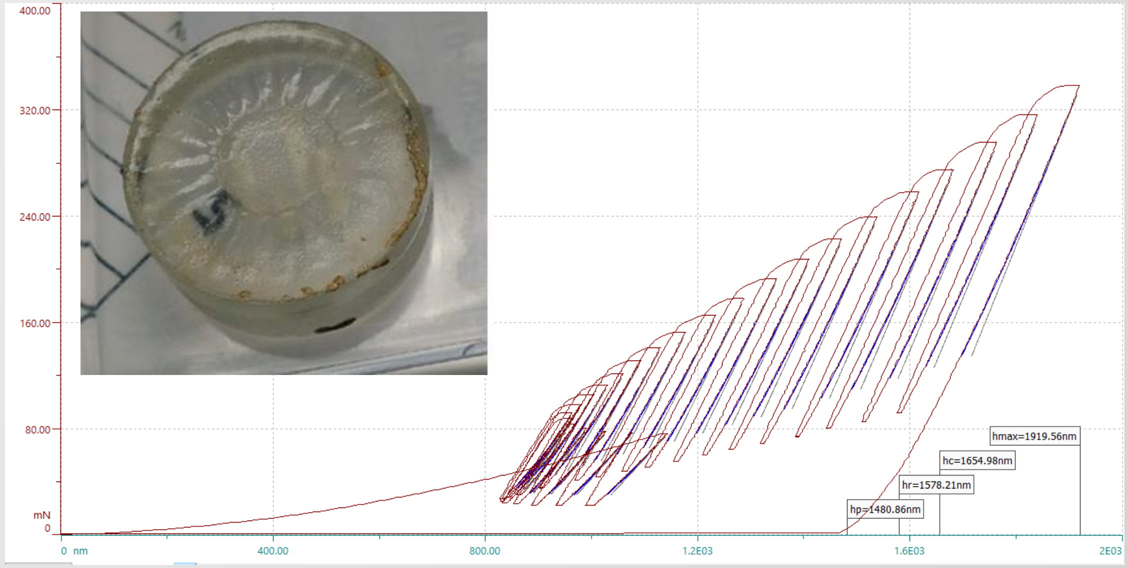
Small components mounted in large volumes of epoxy will be particularly susceptible to compliance due to:

(i) Shrinkage around the components during curing can loosen them and induce lateral compliance as well as vertical…
(ii) Small hard components "floating" on the epoxy become extensions of the indenter…

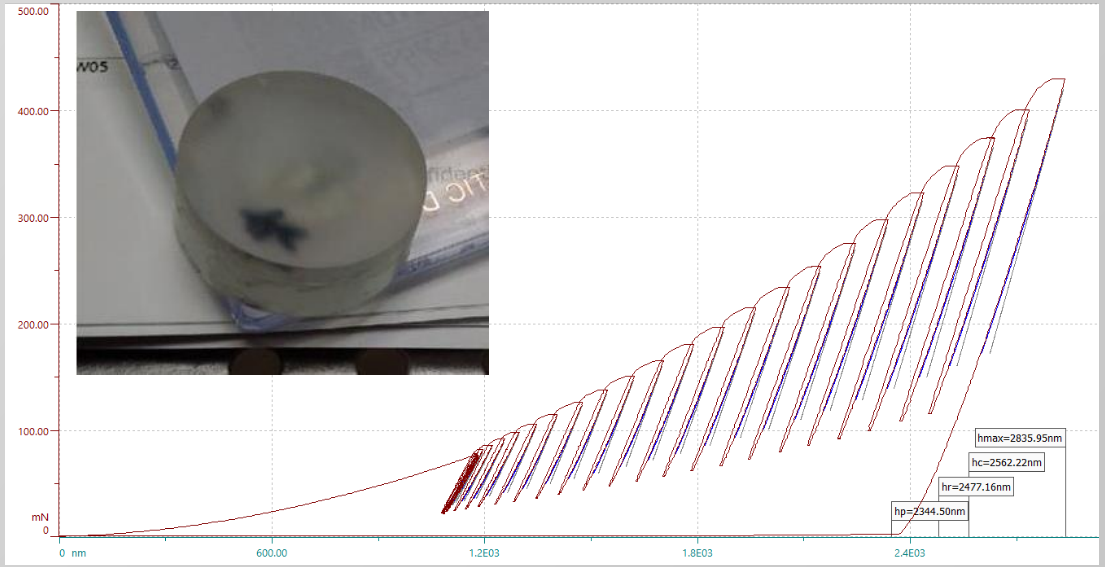
Sample mounting issues: compliance

Backside imperfections must always be removed, as well as the maximum amount of excess epoxy:

Before backside lapping

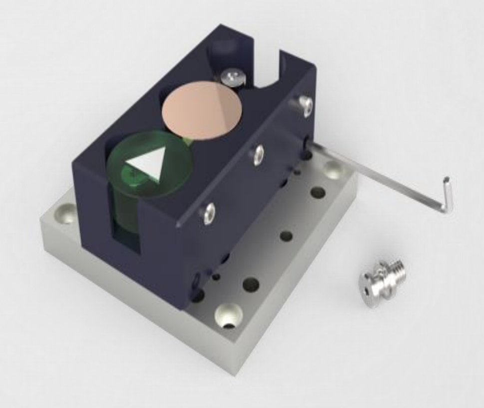


After backside lapping



Multi Sample Holder

- Various versions of multiple sample holders

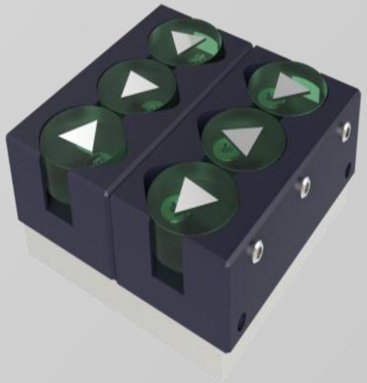


Ref 145076 Multi Sample Holder for 3 samples

Holder for 3 samples: 25-mm diameter per sample
Fixation of sample with a lateral screw
Prepositioning in height with an additional vertical large screw
Pins for installation of sample holder

Ref 145077 Multi Sample Holder for 6 samples

2 blocks of Holder for 3 samples: 25-mm diameter per sample
Fixation of sample with a lateral screw
Prepositioning in height with an additional vertical large screw
Pins for installation of sample holder



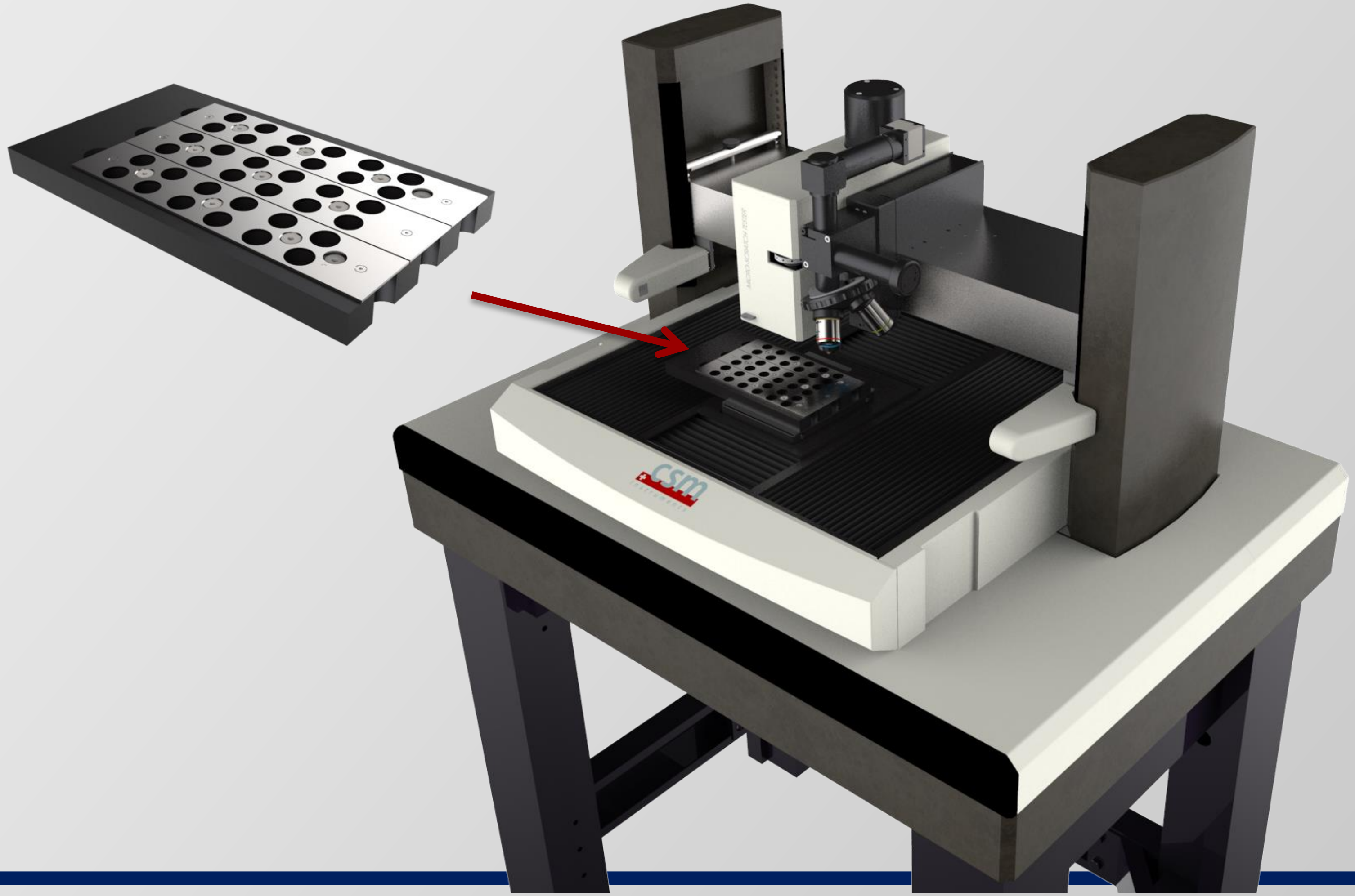
6-sample holder

Multi-Sample adapters: standard modules

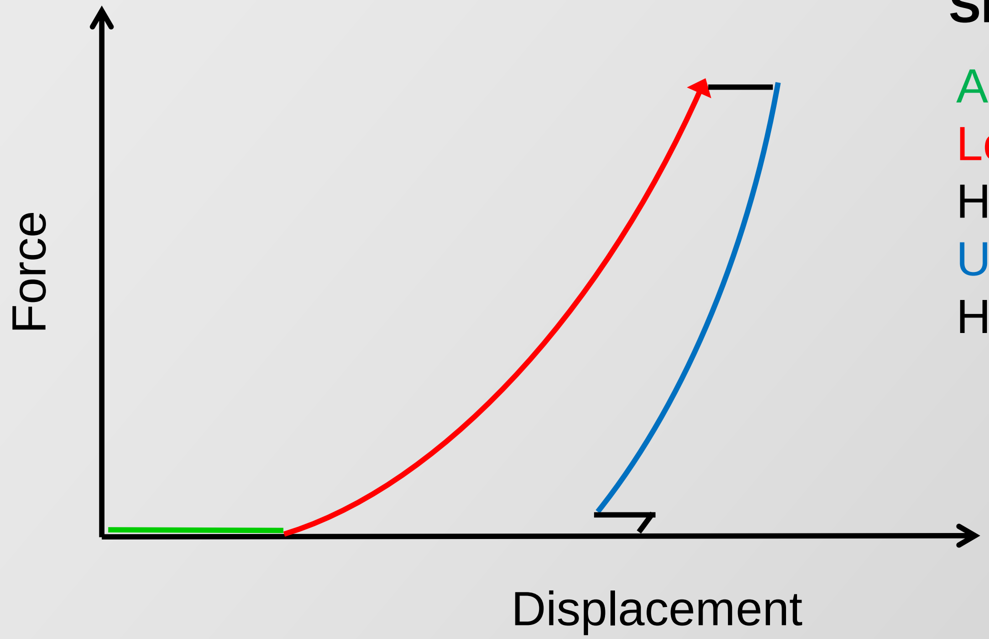
alemnis

Queen Mary
University of London

Measure more than 1 sample automatically!



Simple load cycle



Simple load-unload cycle

Approach and surface detection
Loading
Hold
Unloading
Hold

Parameters to consider

- Approach and detect the surface
- Select load cycle parameters to use
- Assess material and instrument stability
- Inspect data and sample surface
- Data analysis

Simple load cycle

Parameters to consider

Phase 1

Approach to the surface and surface detection

- Approach speed (materials response)
- Detection method
    - force value
    - change in stiffness – over what time scale?

Phase 2

Load cycle parameters

- Maximum load
- Load rate
- Unload rate

Phase 3

Monitor response of material and instrument

- Hold time at max load
- Hold at low load (beginning or end of cycle?)

Limitations on load cycle parameters

Instrument properties:

Depth range – 100nm →1000nm
Force range – 1mN → 500mN
Indenter radius – 1 μm → 30 μm →500 μm

Material properties:

E = 1150GPa (diamond)

E = 400GPa (sapphire)
E = 200GPa (steel)
E = 70GPa (glass)
E = 2.5GPa (polycarbonate)
E = 0.1GPa (elastomers)

Limited by force: ↓R, ↑h

Limited by depth: ↑ R, ↓ h

Select tip radius and force range to reduce depth measurement uncertainty

Examples

For a nanoindentation experiment, estimate the radius of the indenter and the maximum force that would be required for elastic contact on the surface of the following materials:

i) Al_3O_4 (E = 400GPa, ν = 0.2)
ii) Isoprene rubber (E = 100MPa, ν = 0.5)

Assume the indenter to have elastic constants of E = 1150GPa, ν = 0.07

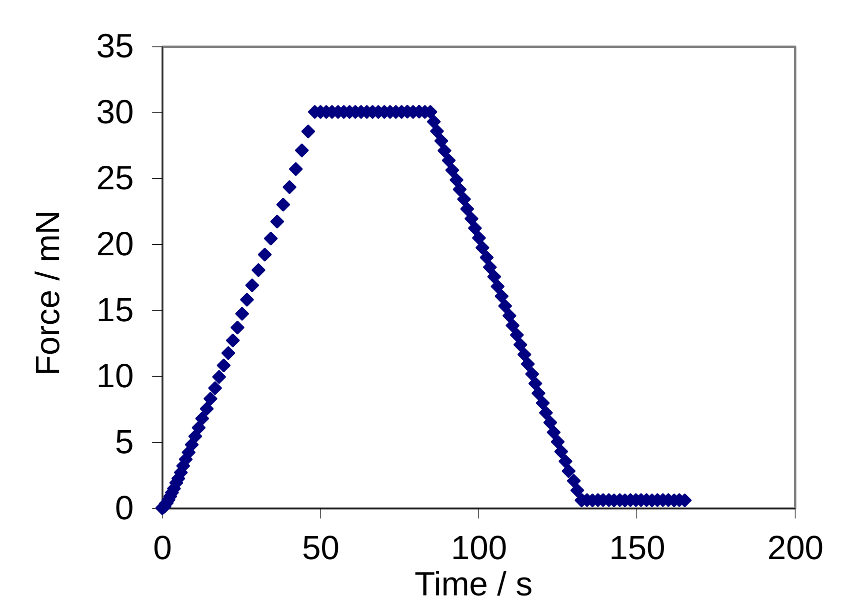
Typical force range available 1mN – 500mN
Typical depth range 100nm – 1000nm

i) E^* = 306GPa,
so for instance if h ~ 1000nm, F ~ 500mN,
$R = 9/16\ F^2/E^{*2}\ 1/h^3$
$R = 1.5\mu m$

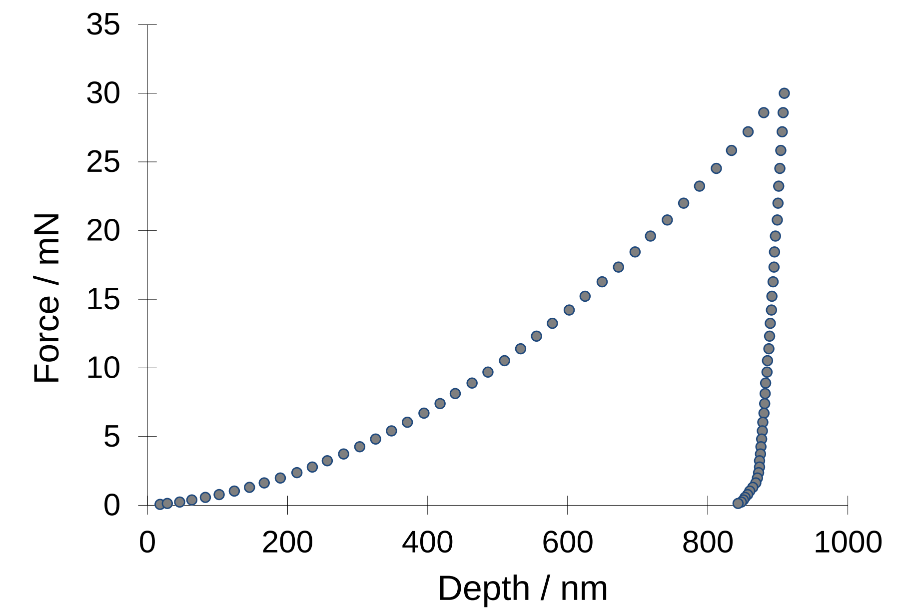
ii) E^* = 133MPa,
so for instance if h ~ 1000nm, F ~ 5mN,
$R = 9/16\ F^2/E^{*2} * 1/h^3$
$R = 800\mu m$

Simple load cycle

- Berkovich indenter
- Elastic-plastic materials
- Oliver and Pharr analysis



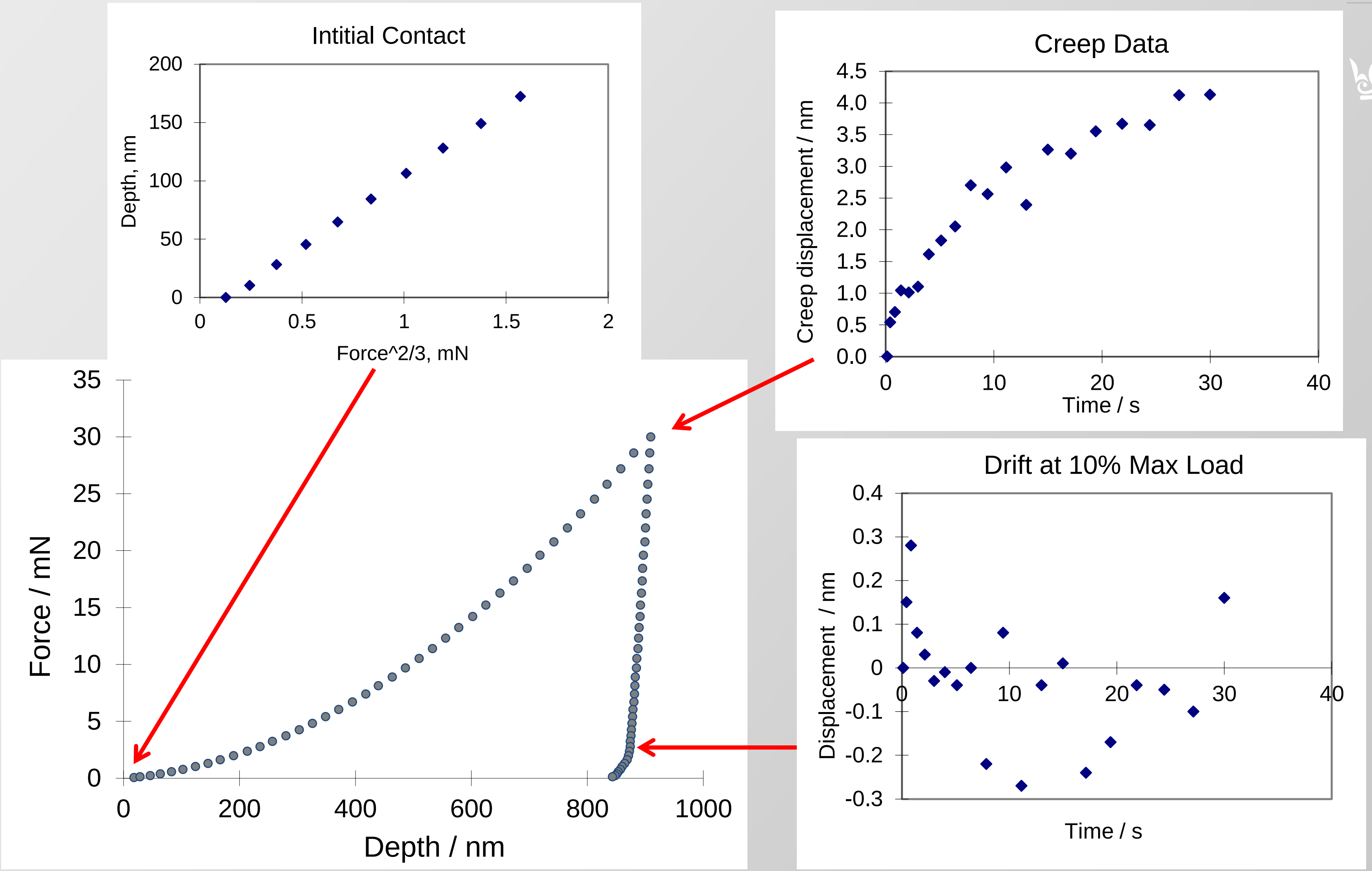
Load cycle



Force displacement curve

Example for Ni single crystal

Simple load cycle



Simple load cycle

Parameters to consider

Phase 4

Inspection of data and sample

- Was the load cycle achieved?
- Are all the load curves the same?
  - what is the scatter like?
  - are there any anomalies?
- Can you see the indents under the microscope?
  - are they where you thought they would be?
  - are there any associated with specific features?

Phase 5

Data analysis

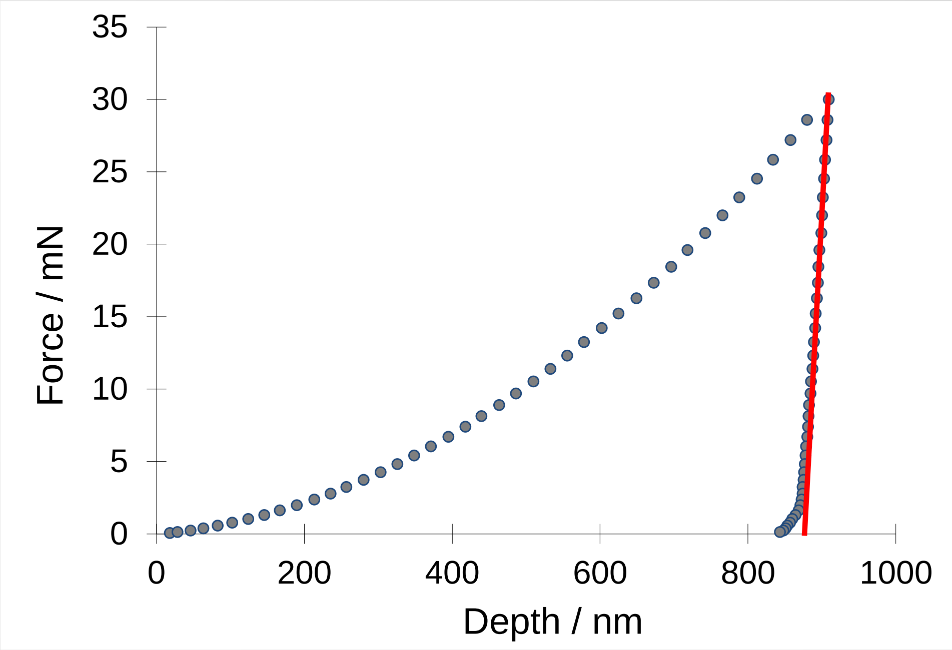
- Which method is appropriate?
- Can the method be applied successfully?
- How good is the fit to the unload slope?
- Are the results as expected?
- If not, what might be the cause?

Simple load cycle

Oliver and Pharr analysis of the data

Calculate contact depth from the unloading slope $$h_c = h_{max} - 0.75\, F \frac{dh}{dF}$$

from h_c and indenter geometry get area of contact, A $$A = 24.56\, h_c^2$$



Hardness

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

Elastic modulus

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

Summary

- Know what your sample is like
- Know the limitation of the measuring system
- Consider the indentation parameters that are appropriate for your material system
- Consider the load cycle and analysis method
- Review the data you get and the observe the indentation sites on the sample surface
- Was the data as expected or did it reveal something unusual?

Now we are ready to try some tests!