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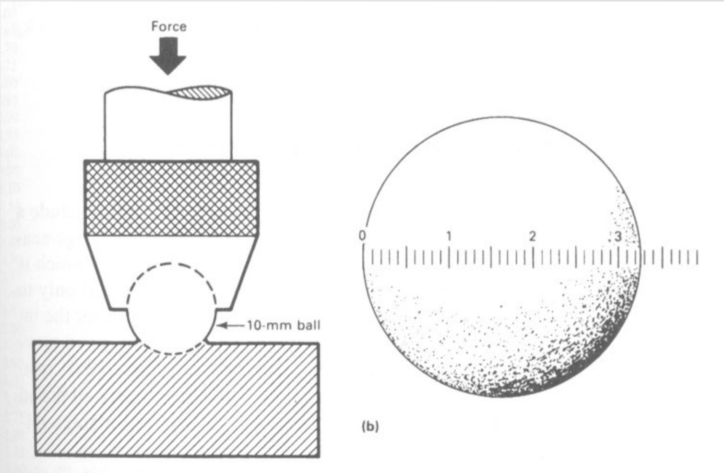
3. Instrumentation: How does it work?

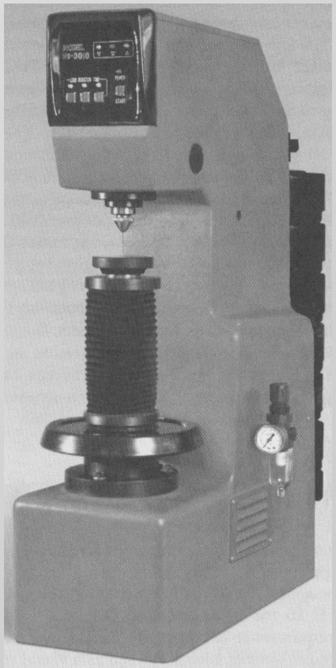
Nick Randall

Conventional hardness

Brinell Test

- Fixed loads: 3000, 1500 or 500 kgf
- Optical measurement of the residual print area
- Hardened steel or diamond ball, 10 mm diameter





[Developed by Johann Brinell in 1900]

Conventional hardness

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Brinell Hardness, H_B

$$HB = \frac{0.102 \times F}{S}$$

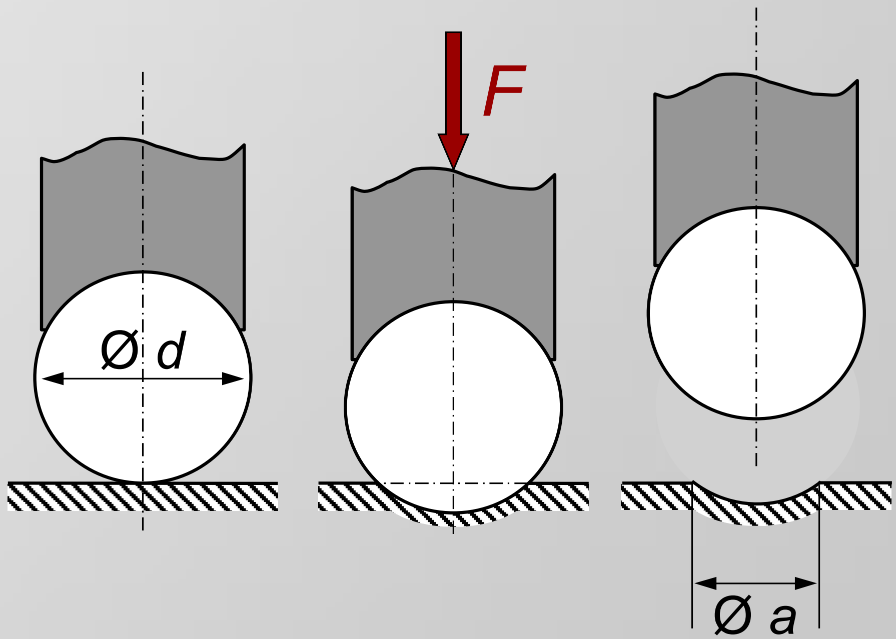
where S = developed residual print area (A_d)

$$S = \frac{\pi d}{2}\left(d - \sqrt{d^2 - a^2}\right)$$

F in N

a and d in mm

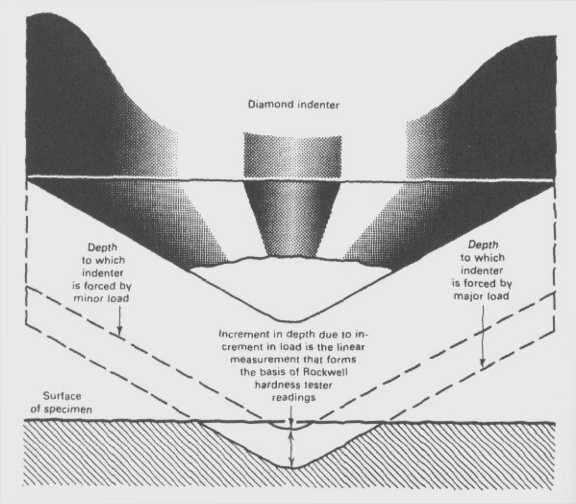
$$HB = \frac{\text{Force}}{\text{Developed residual print area}}$$

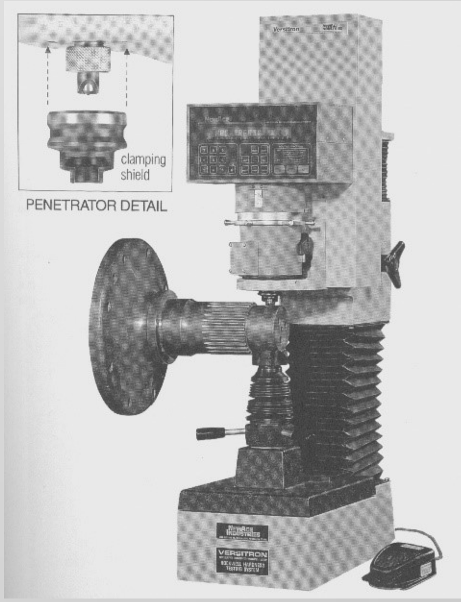


Conventional hardness

Rockwell Test

- Apply fixed load
- Measure penetration depth difference
- Diamond indenter, 200 µm radius

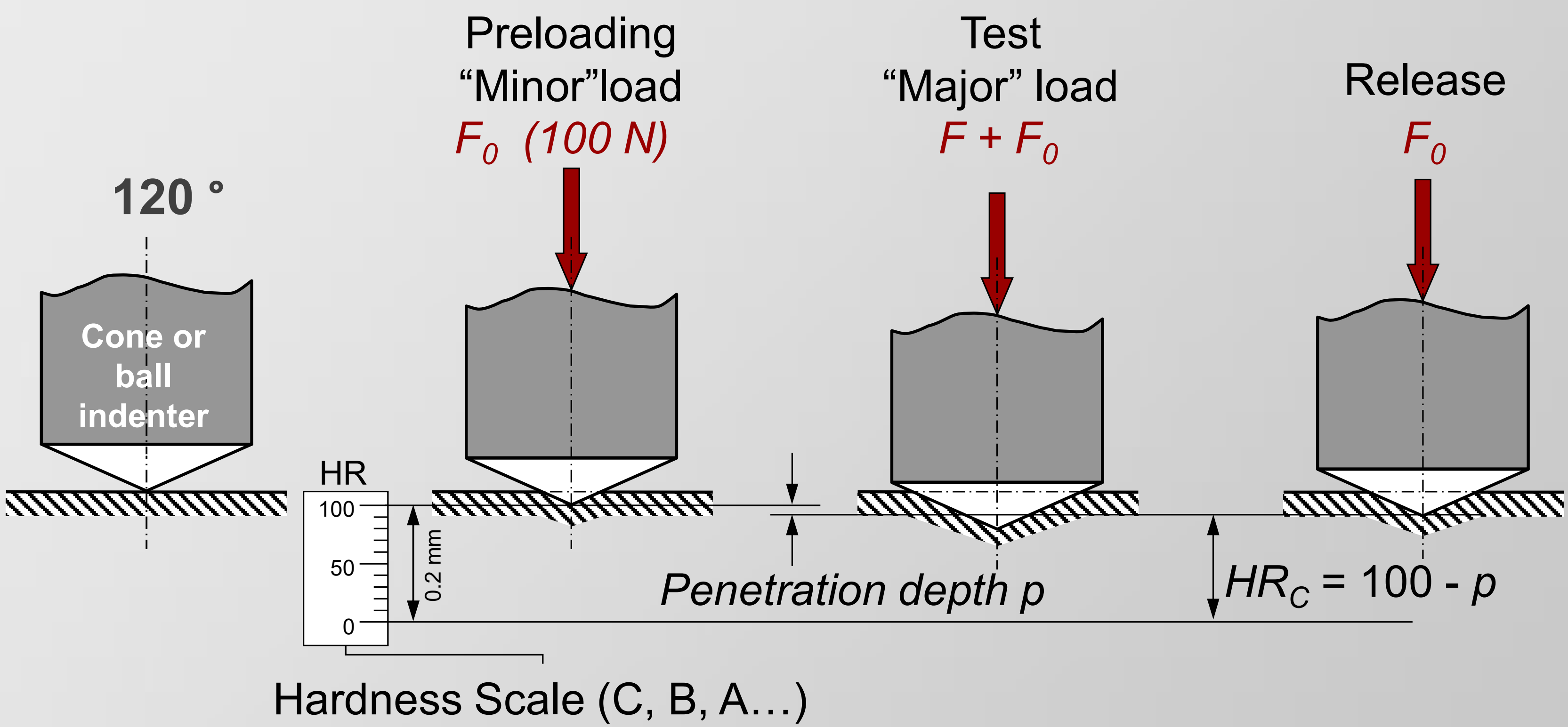




[Developed by Stanley Rockwell in 1919]

Conventional hardness

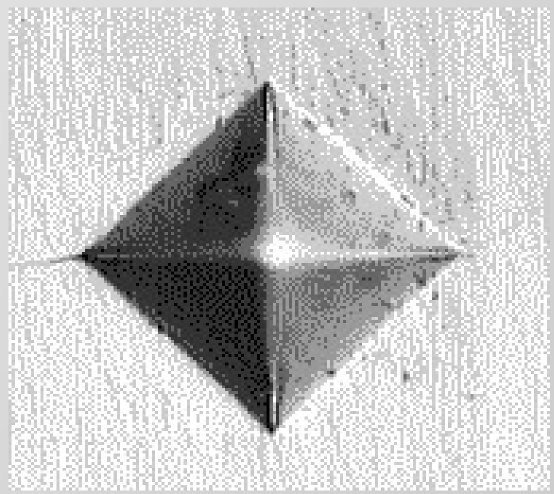
Rockwell Hardness, *HR* (HR_C, HR_B, …)

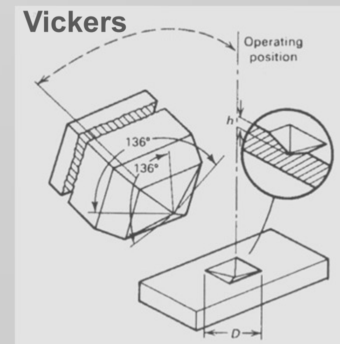


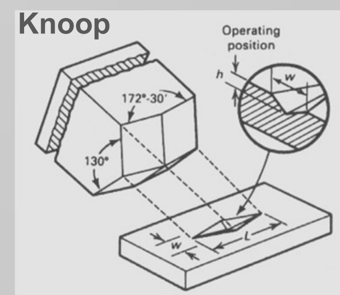
Conventional indentation

- Static load applied via an indenter of known geometry
- Residual contact area determined by measuring the residual imprint diagonal with an optical microscope

$$\text{Hardness} = \frac{\text{Applied Load}}{\text{Residual Contact Area}}$$







Conventional indentation

Vickers Hardness, *HV*

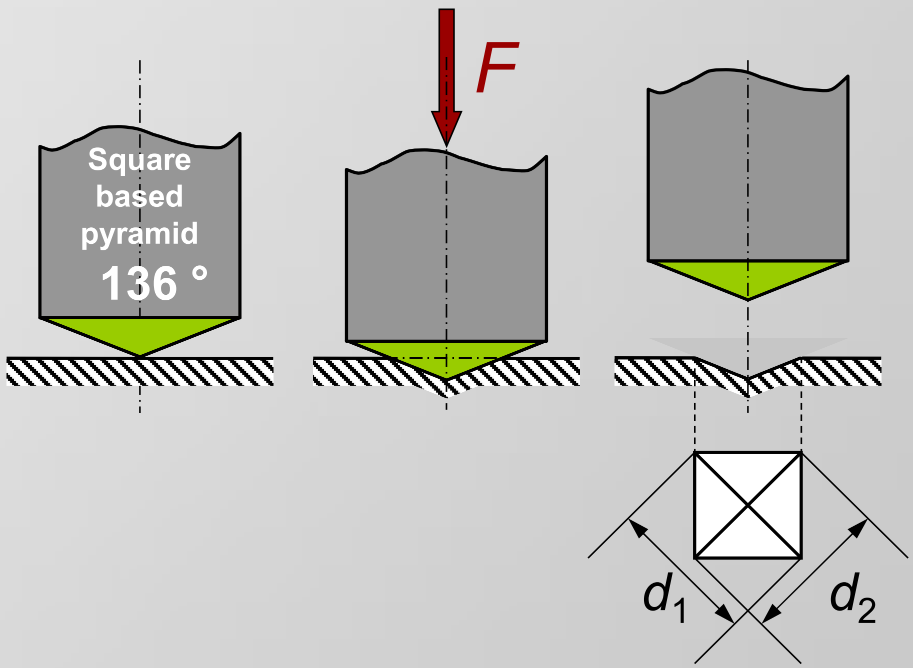
$$HV = \frac{\text{Force}}{\text{Developed residual print area}}$$

$$HV = \frac{0.189 \times F}{d^2}$$

where $d = \frac{d_1 + d_2}{2}$

F in N

d_1 and d_2 in mm



Instrumented Indentation Testing (IIT)

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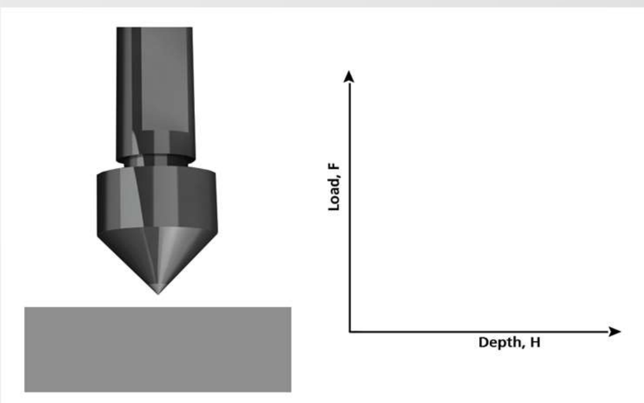
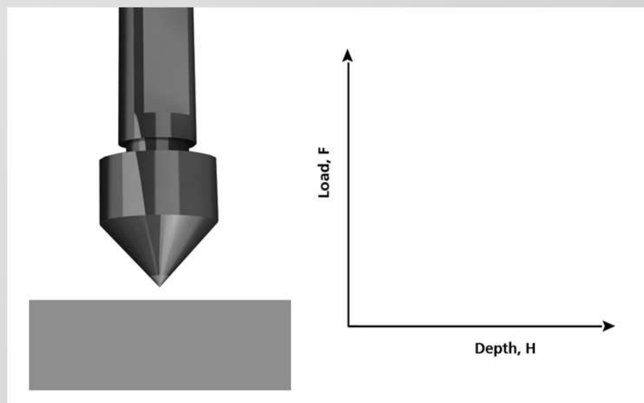
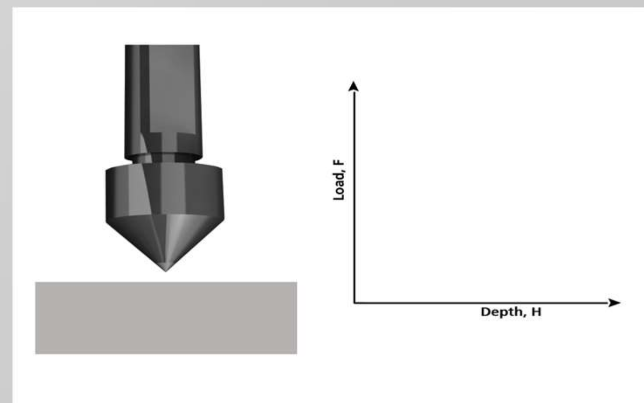
- Instrument which provides the ability to measure the indenter penetration depth, *h*, under the applied force, *F*, throughout the testing cycle.
- Applied force and depth are measured dynamically during a load-unload cycle
- Hardness and Elastic Modulus are calculated directly from the resultant force-displacement curve



Elastic



Elastoplastic



Plastic

Conventional nanoindenter design

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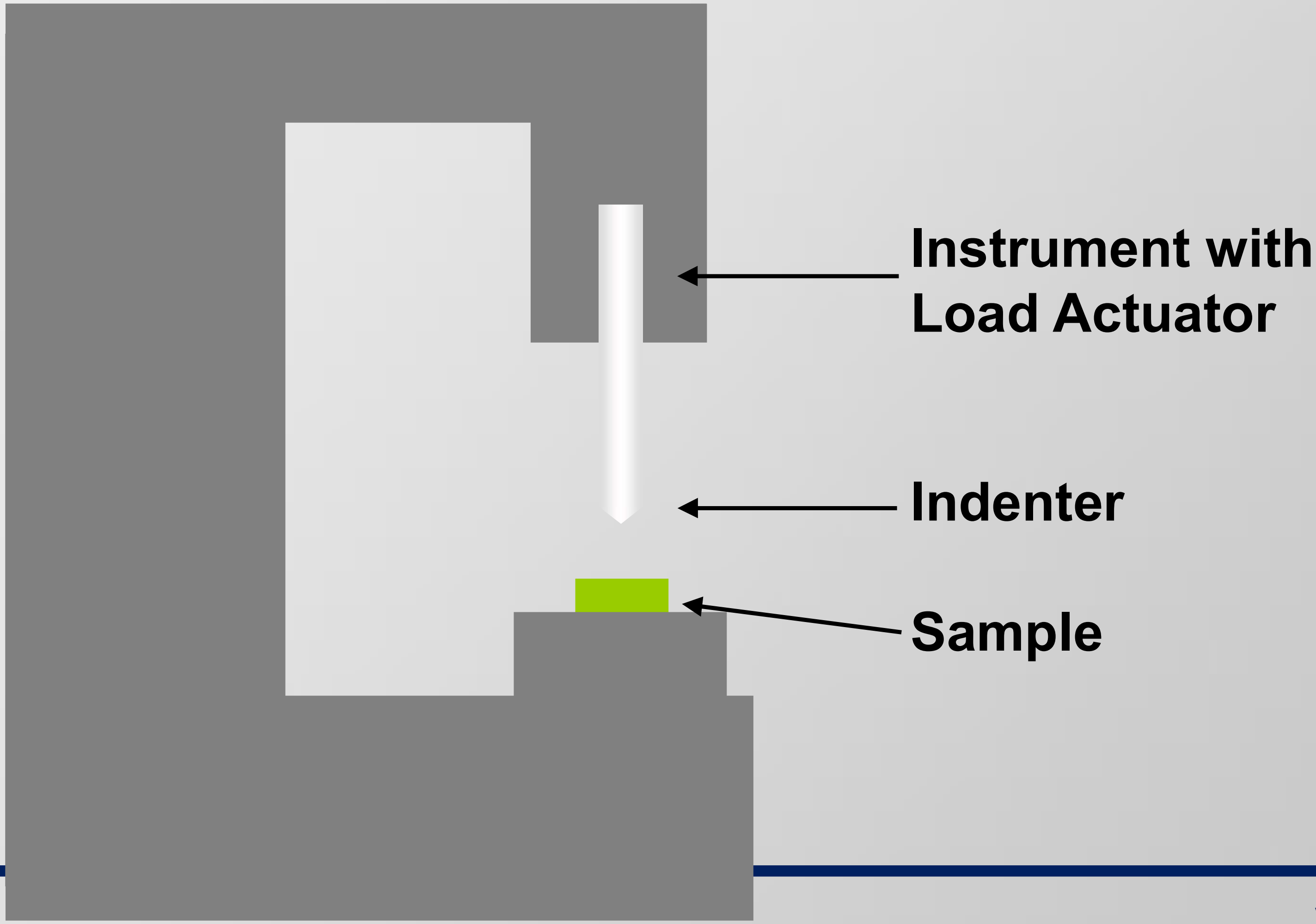

Conventional nanoindenter design

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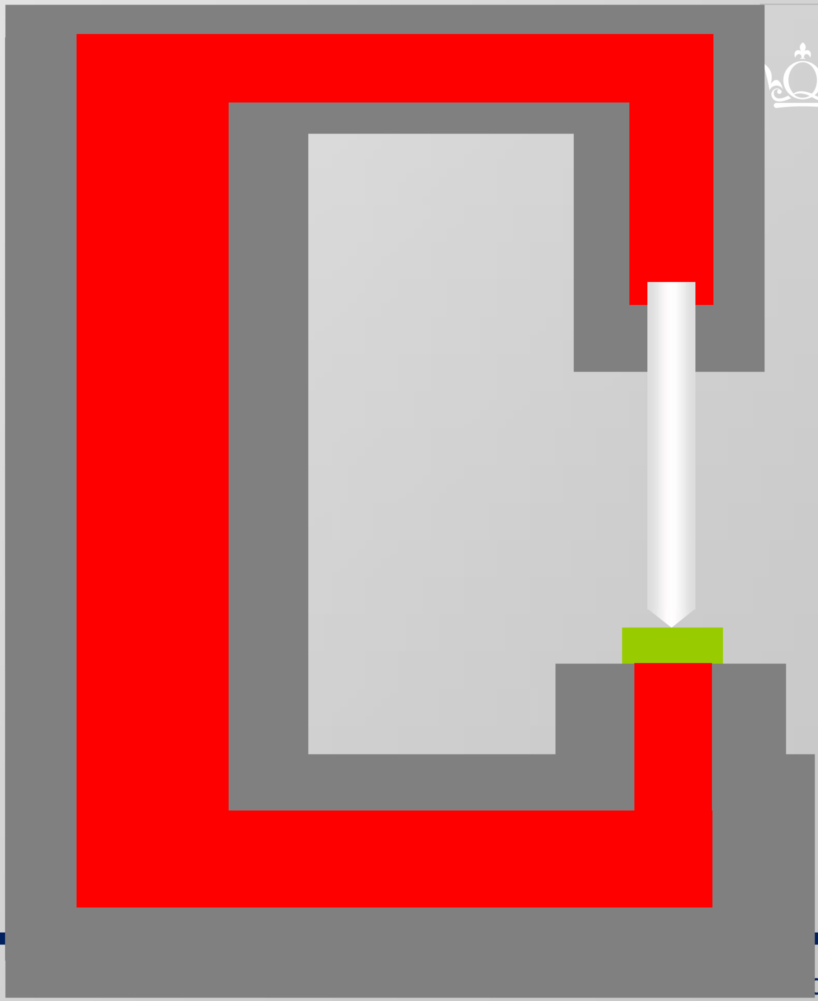
Most instruments use a load-application device coupled with a displacement sensor. Potential issues are:

- Calibration of sensors
- Thermal drift
- Frame compliance
- Contact point detection
- Indenter area function calibration
- Cleanliness of indenter
- Laboratory conditions (T, RH, dust, etc.)
- Sample mounting



Thermal drift

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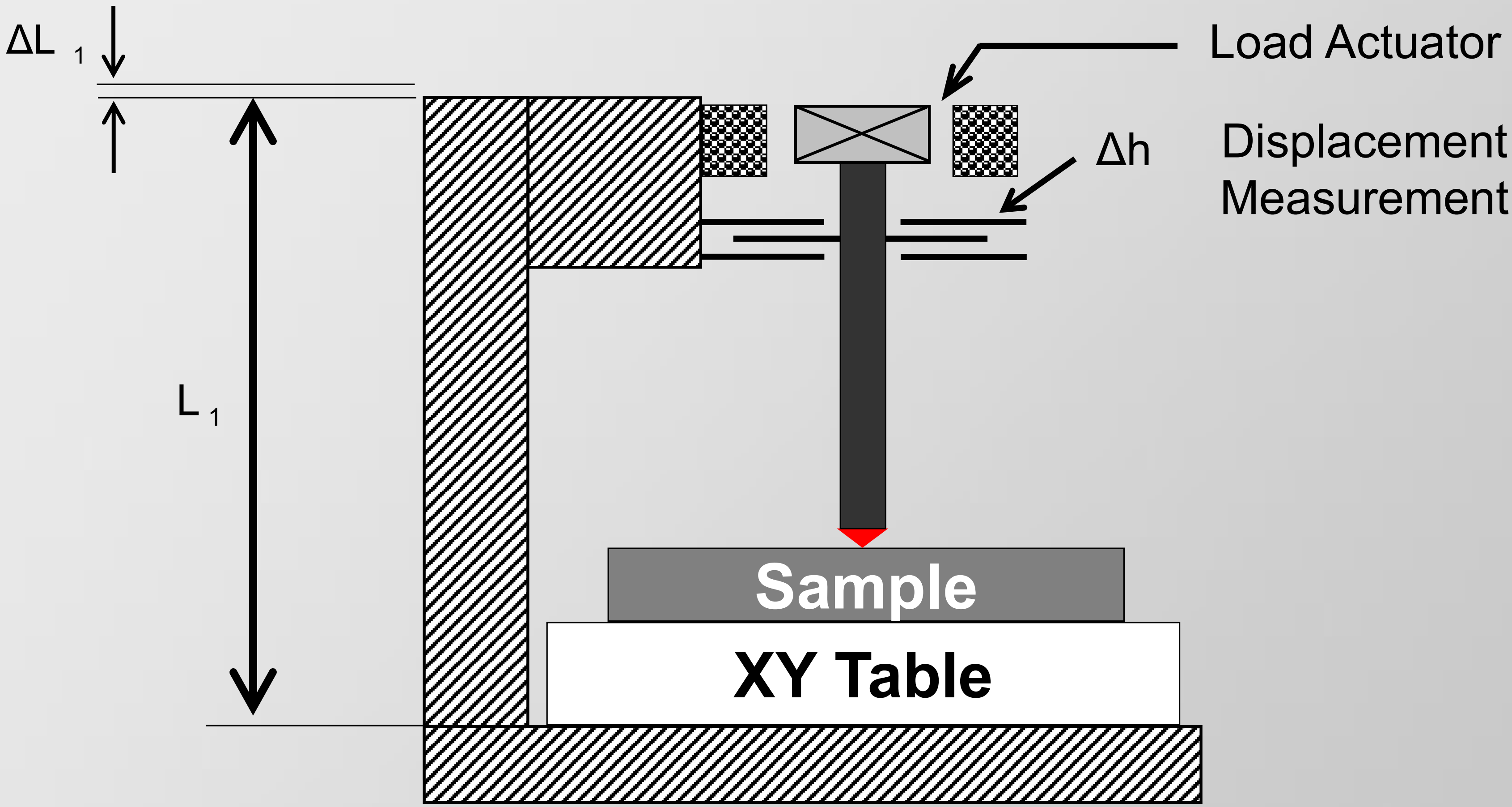
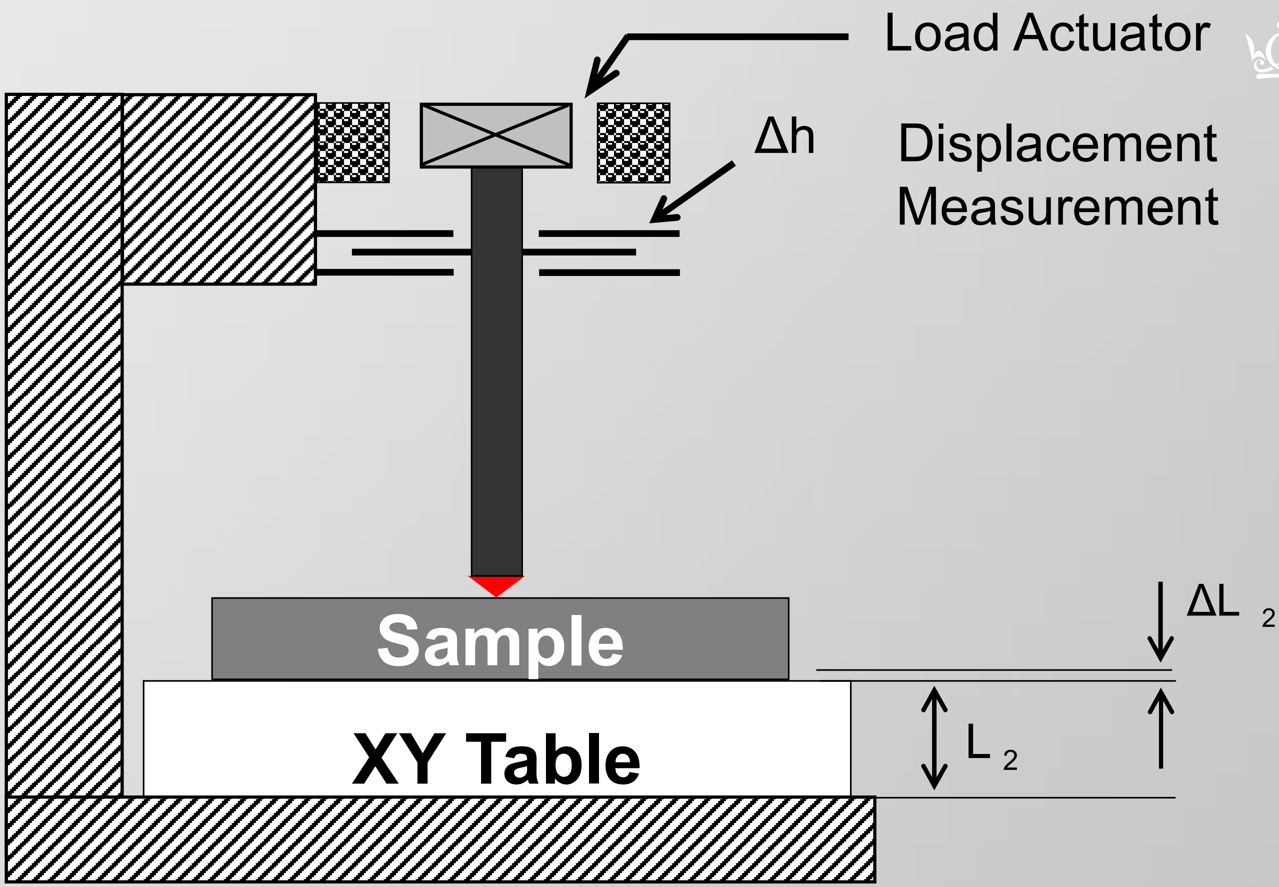
- The thermal drift refers to the variations in the depth measurement signal resulting from thermal expansion or contraction of the sample or the indentation apparatus during an indentation test.

- These variations, unless minimized or corrected for, will be interpreted as real displacements of the indenter into the specimen and so have an adverse effect on the accuracy of the results.

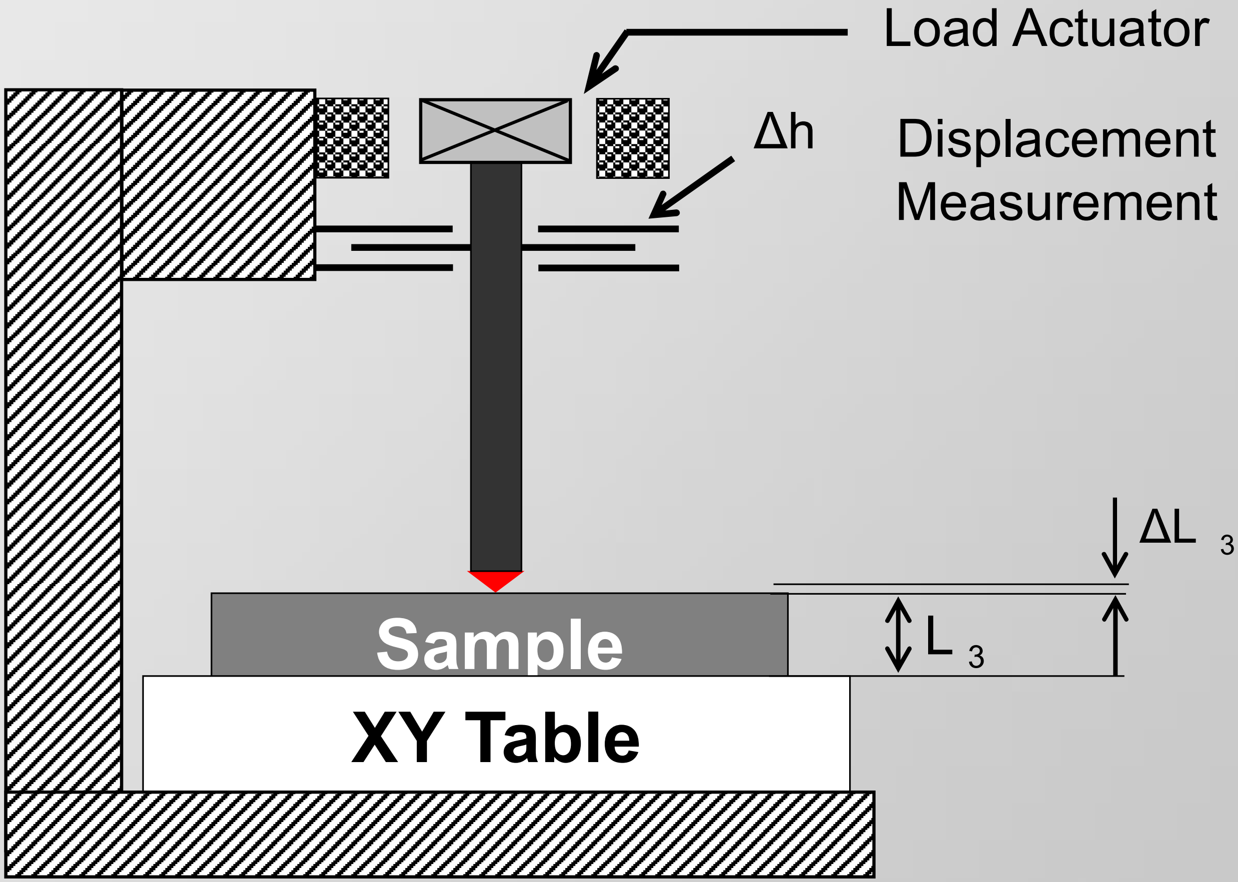
Thermal drift



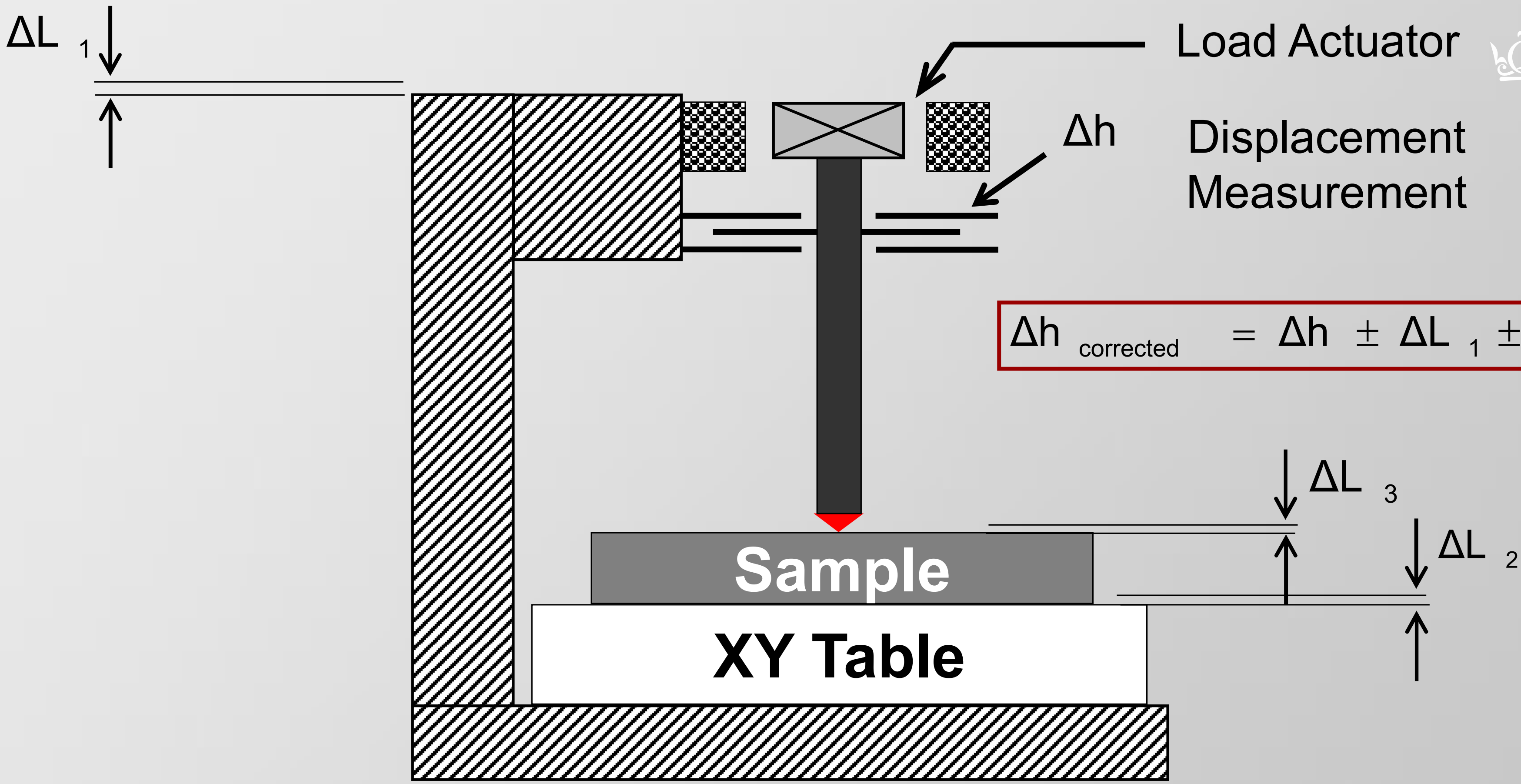
Thermal drift



Thermal drift



Thermal drift



$$\Delta h_{corrected} = \Delta h \pm \Delta L_1 \pm \Delta L_2 \pm \Delta L_3$$

Thermal drift

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- ΔL is the change in length due to the change in temperature, L_p is the length of material considered, C' the coefficient of linear thermal expansion and ΔT the change in temperature.

$$\Delta L = L_P . C' . \Delta T$$

- The larger L_p, the larger ΔL

- Example:
  - Frame length = 300mm
  - Coefficient of thermal expansion of aluminum = 23.1×10^{-6} / °C
  - Variation of temperature = 0.01°C per minute
  - Variation of length = 69nm per minute

Thermal drift

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Three potential solutions:

1. Make the assumption that the thermal drift is constant all along the test, measure the thermal drift and correct the data (does not work for materials with time dependent properties as soft metals, polymers and elastomers).

2. Make the test very fast (2 sec) to minimize the effect of thermal drift on the data (Modulus and Hardness results can be influenced by high loading rate and creep as for soft metals, polymers and elastomers)

3. Design an instrument with extremely low thermal drift (e.g., surface referencing indentation tester philosophy).

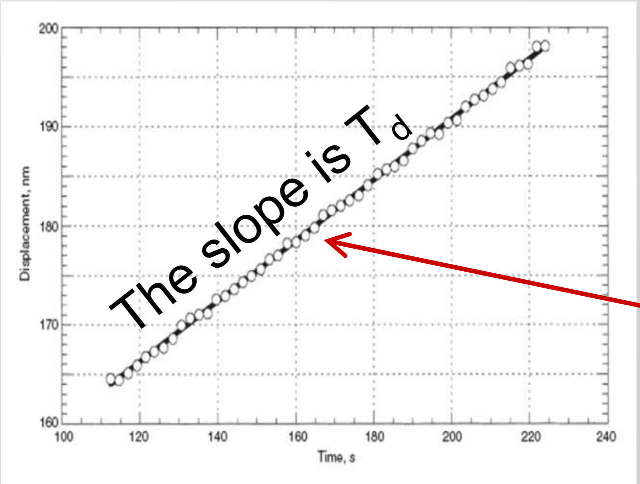
1. Thermal drift correction

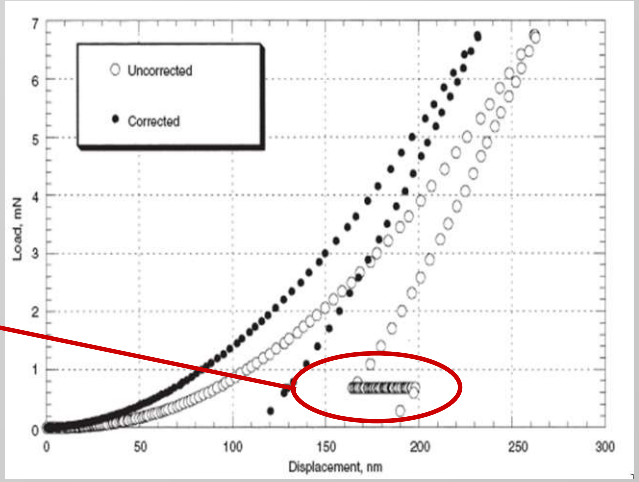
- Requires an environmental enclosure.
- Measure the thermal drift and wait until stabilization (can take several hours).
- Make the assumption that the thermal drift is constant all along the test : not always true, the thermal drift is usually oscillating and exponential in character.
- Make the indentation test and measure the thermal drift (variation of depth at a constant load).
- Correct the data from the thermal drift.

1. Thermal drift correction

Correction made on the depth measurement
(T_d , Thermal drift coefficient in nm/s)

$$h_{corrected} = h_{measured} - T_d \cdot t$$





Thermal drift = 0.3 nm/s

1. Thermal drift correction

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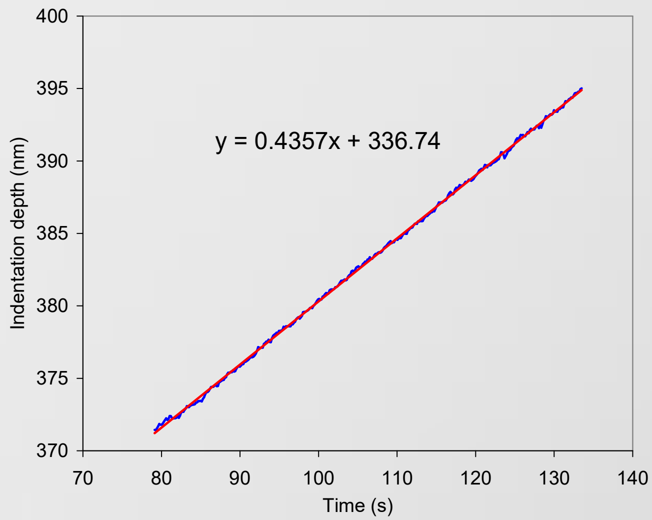
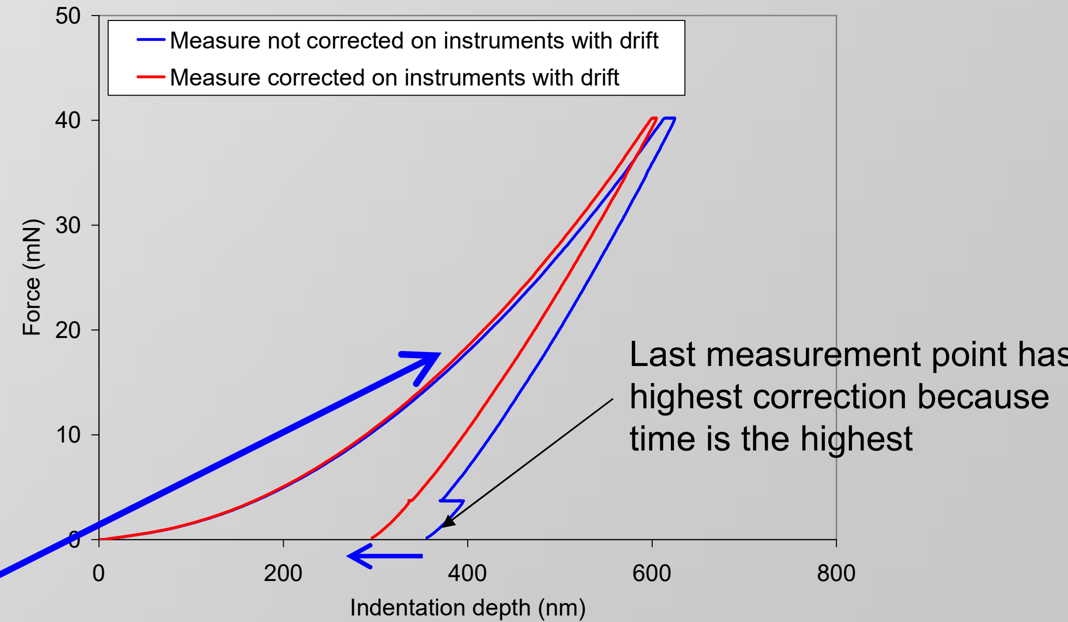


Thermal drift, T_d = 0.436 nm/s

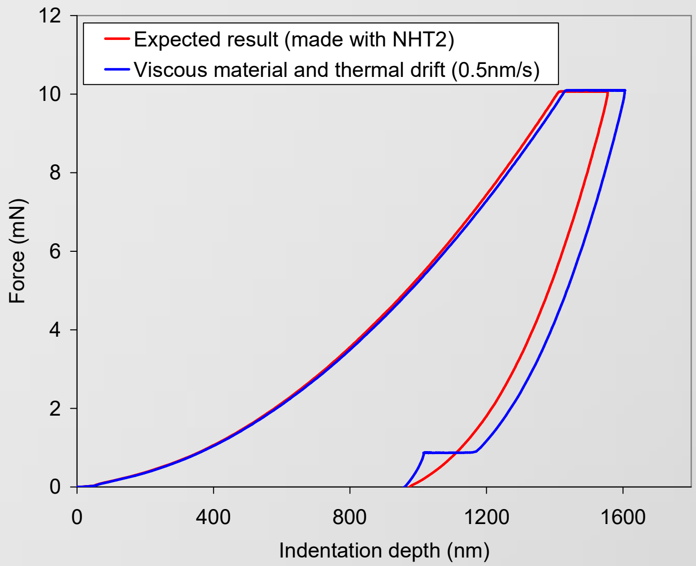
All indentation depth points are corrected using the following equation

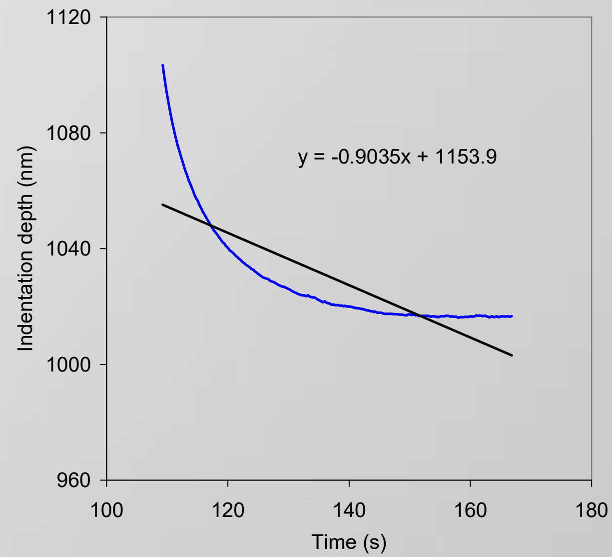
$$h_{corrected} = h_{measured} - T_d . t$$



1. Thermal drift correction

1. Thermal drift correction on Polymer (e.g., PMMA)





The thermal drift measurement on a polymer is not linear due to viscoelasticity also occurring during that period of time

1. Thermal drift correction

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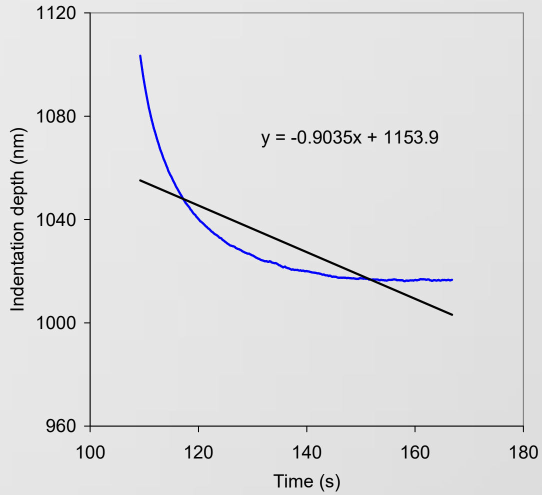
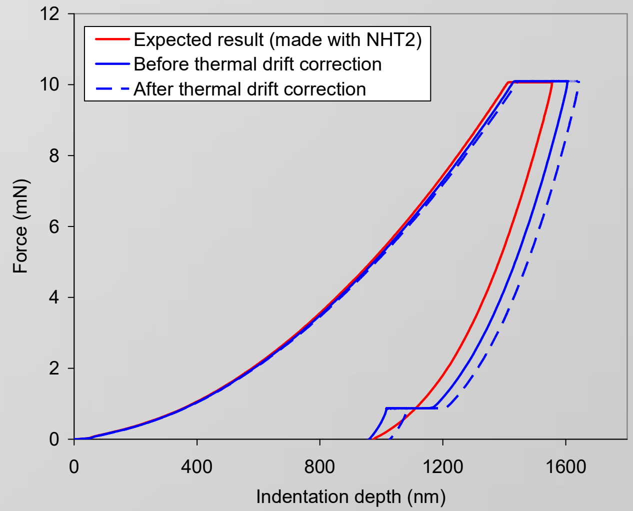
1. Thermal drift correction on Polymer (e.g., PMMA)

Thermal drift measurement is not linear due to viscoelasticity



Thermal drift correction is even worse than not doing it.



Viscoelasticity of polymers massively influences the pause for thermal drift measurement.

Sometimes, it is not recommended to correct for thermal drift on soft materials !

1. Thermal drift correction: Conclusions

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- Waiting time for thermal drift stabilization can take several hours
- The assumption that the thermal drift is constant all along the test is not always true introducing errors
- Thermal drift correction on indentation depth can be more than 50% of the measured value which may introduce large error on calculations
- Measuring thermal drift rate in air (no contact) will not be necessarily the same as when indenter contacts the sample surface.
- Takes extra time to measure the drift rate (60s per test)
- Impossible to distinguish creep or viscoelasticity from thermal drift:
  - Does not work for soft metals with creeping behavior
  - Does not work for polymeric materials due to viscoelastic properties

1. Thermal drift correction

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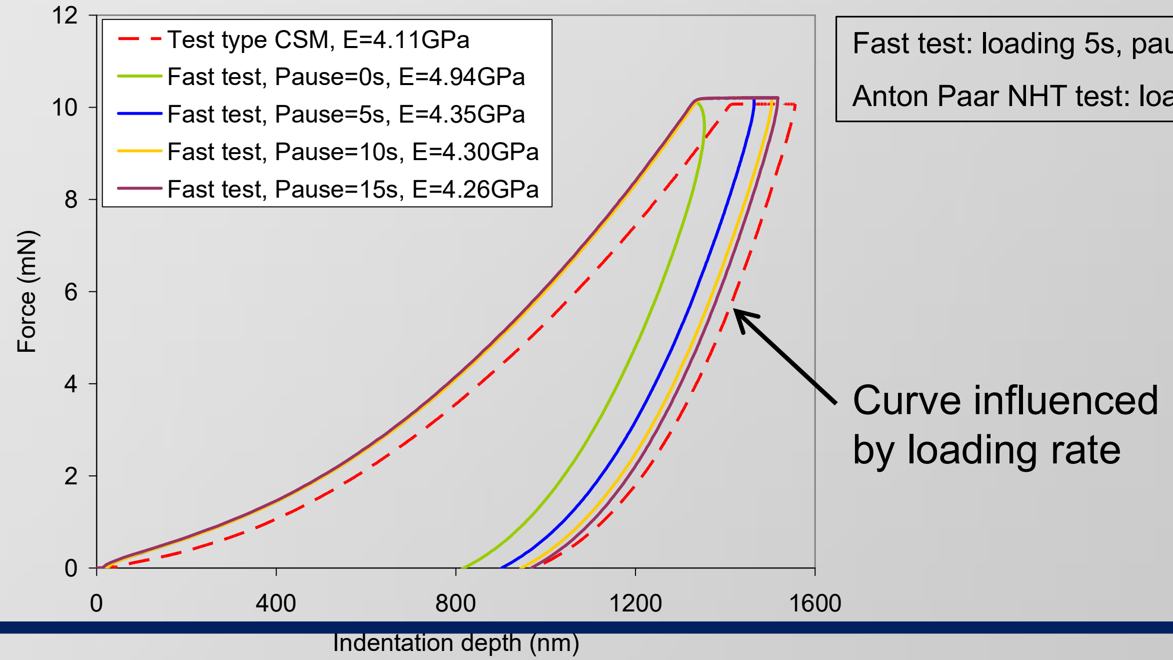
2. Fast test on Polymer (e.g., PMMA)

- Viscoelastic materials are affected by loading/unloading rate
- Long pause is necessary to achieve good result
- Fast test is not recommended for soft metals and polymers



Fast test: loading 5s, pause variable, unloading 5s

Anton Paar NHT test: loading 30s, pause 60s, unloading 15s

1. Thermal drift correction

2. Fast test on Polymer (e.g., PMMA)

- Waiting time for thermal drift stabilization can take several hours
- Perform the test very fast (high loading/unloading rate) to minimize the effect of thermal drift on the data
- Modulus and Hardness results are influenced by loading rate for soft metals, polymers and elastomers.
- Important to have a long pause time at max load which is incompatible with fast test!

1. Thermal drift correction

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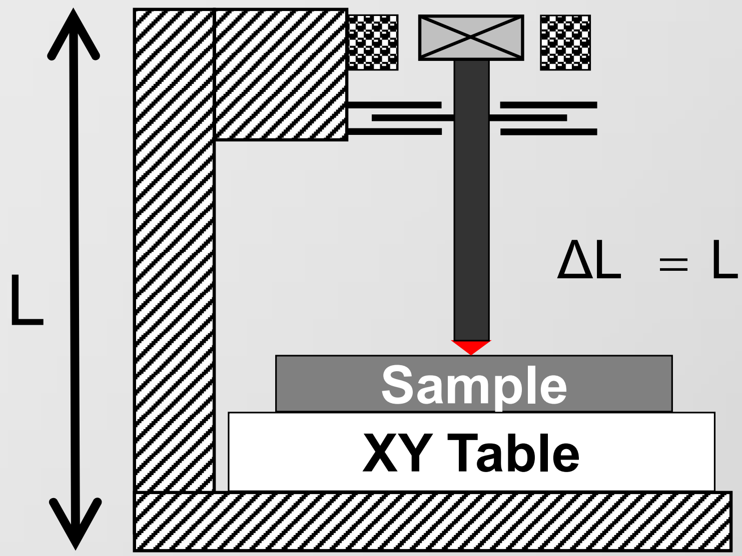
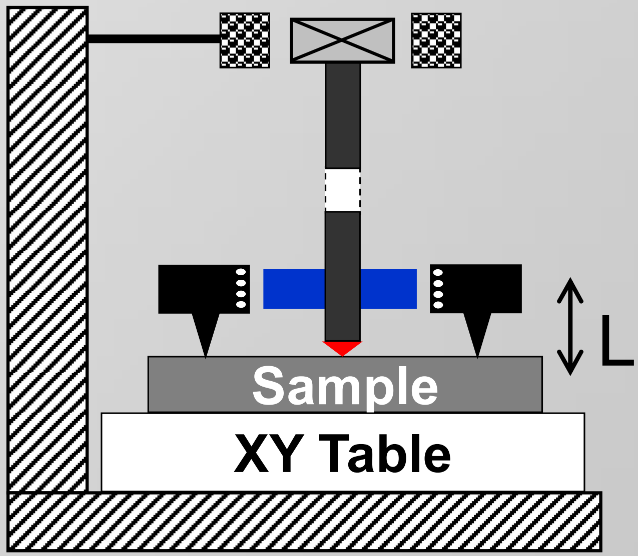
3. Instrument with extremely low thermal drift

Conventional indentation tester
Frame length, L = 300 mm

Indentation tester with top referencing
Frame length, L = 5 to 10 mm



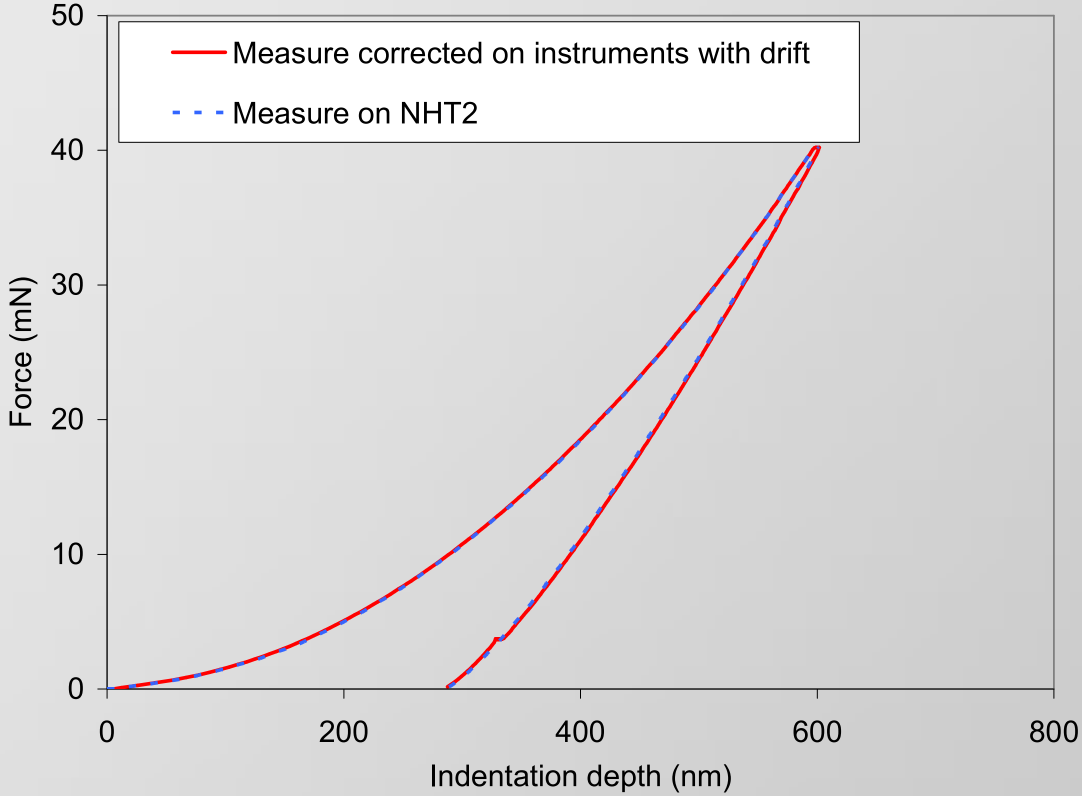
$$\Delta L = L_P . C' . \Delta T$$



Due to smaller frame length, instruments with top referencing may have smaller influence of thermal drift on measurements

1. Thermal drift correction

Top referencing indentation testers have low thermal drift and may not need any thermal drift correction



2. Frame compliance correction

- At any application of the load to the indenter, the reaction force is taken up by deflection of the load frame and may be registered by the depth sensor. If not accounted for, an error proportional to the force will be introduced into the displacement readings.

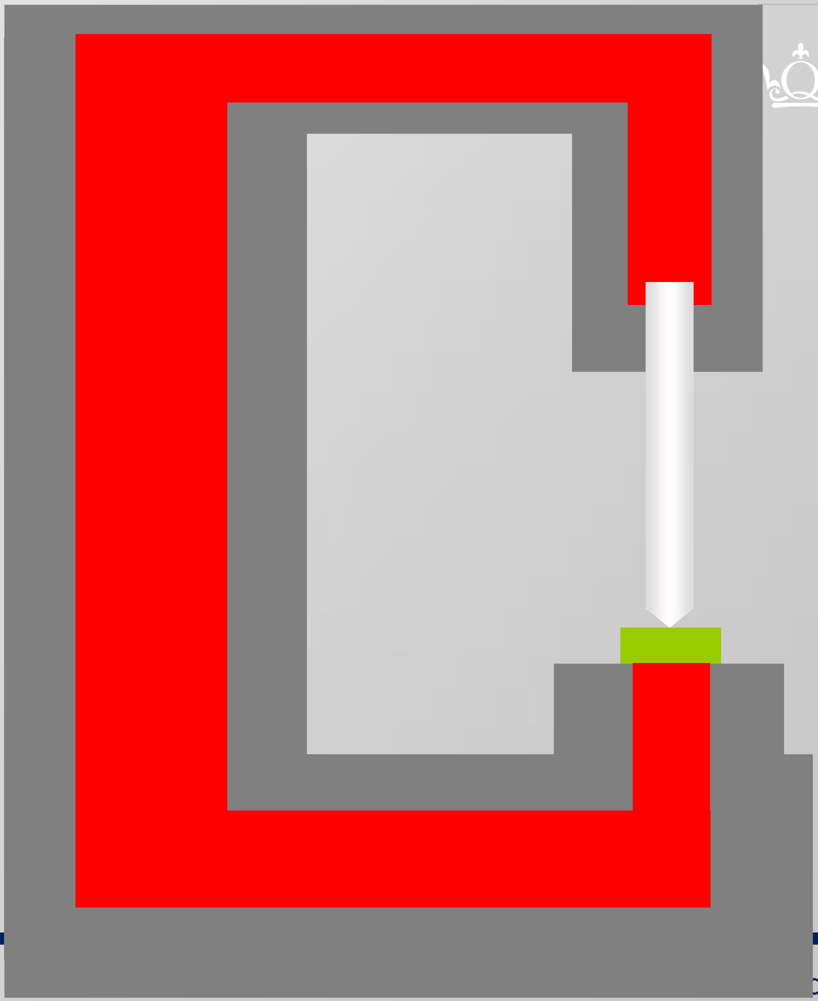
- The frame compliance correction seeks to minimize this error by correcting the displacement readings by the deflection of the load frame.

2. Frame compliance correction

▸ **Frame Compliance includes:**

1. Indenter
2. Instrument frame
3. XY Tables
4. Sample holder

NOTE: The amount of material that constitutes the frame easily reaches a distance of 300 mm



2. Frame compliance correction

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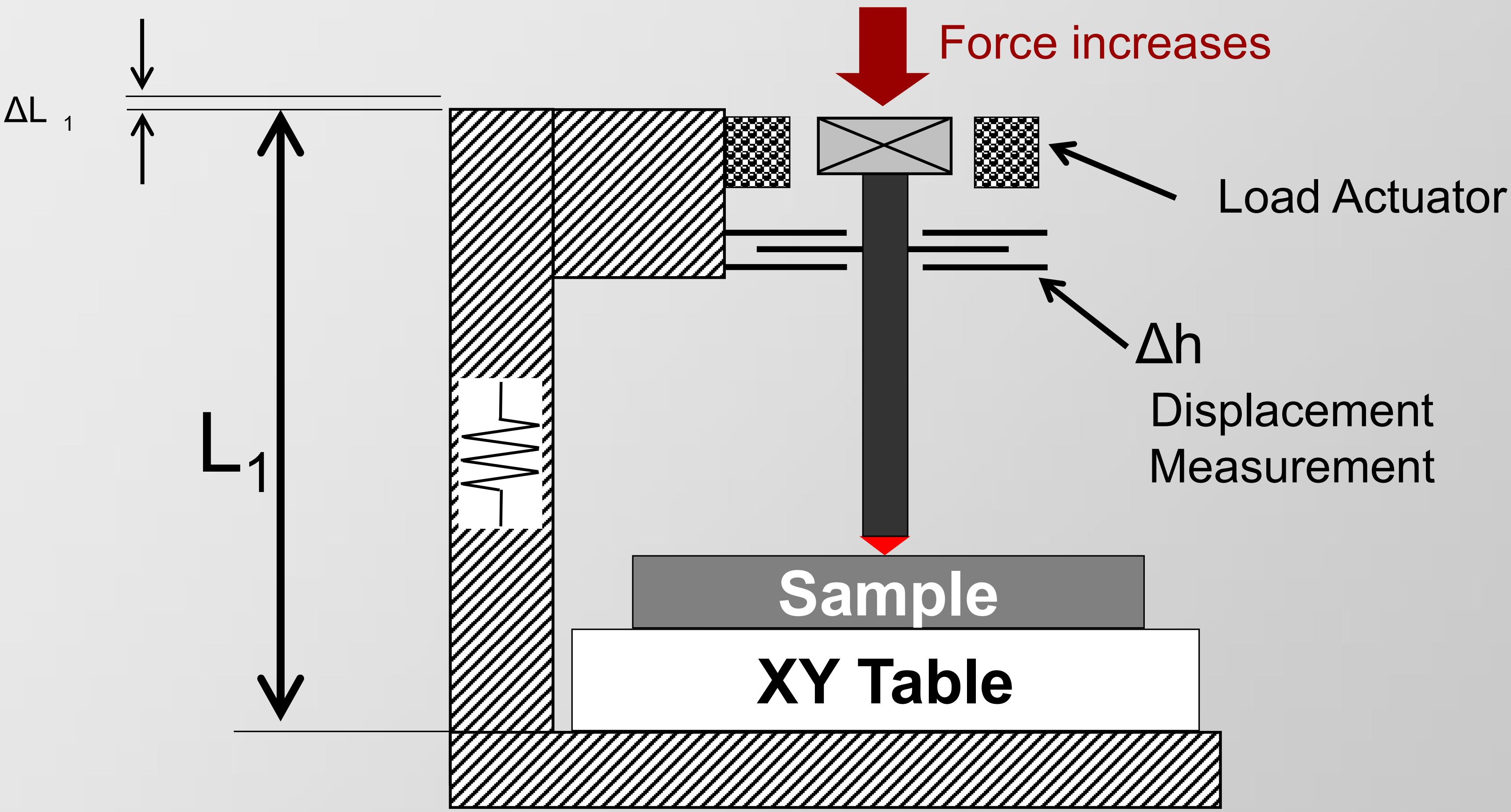
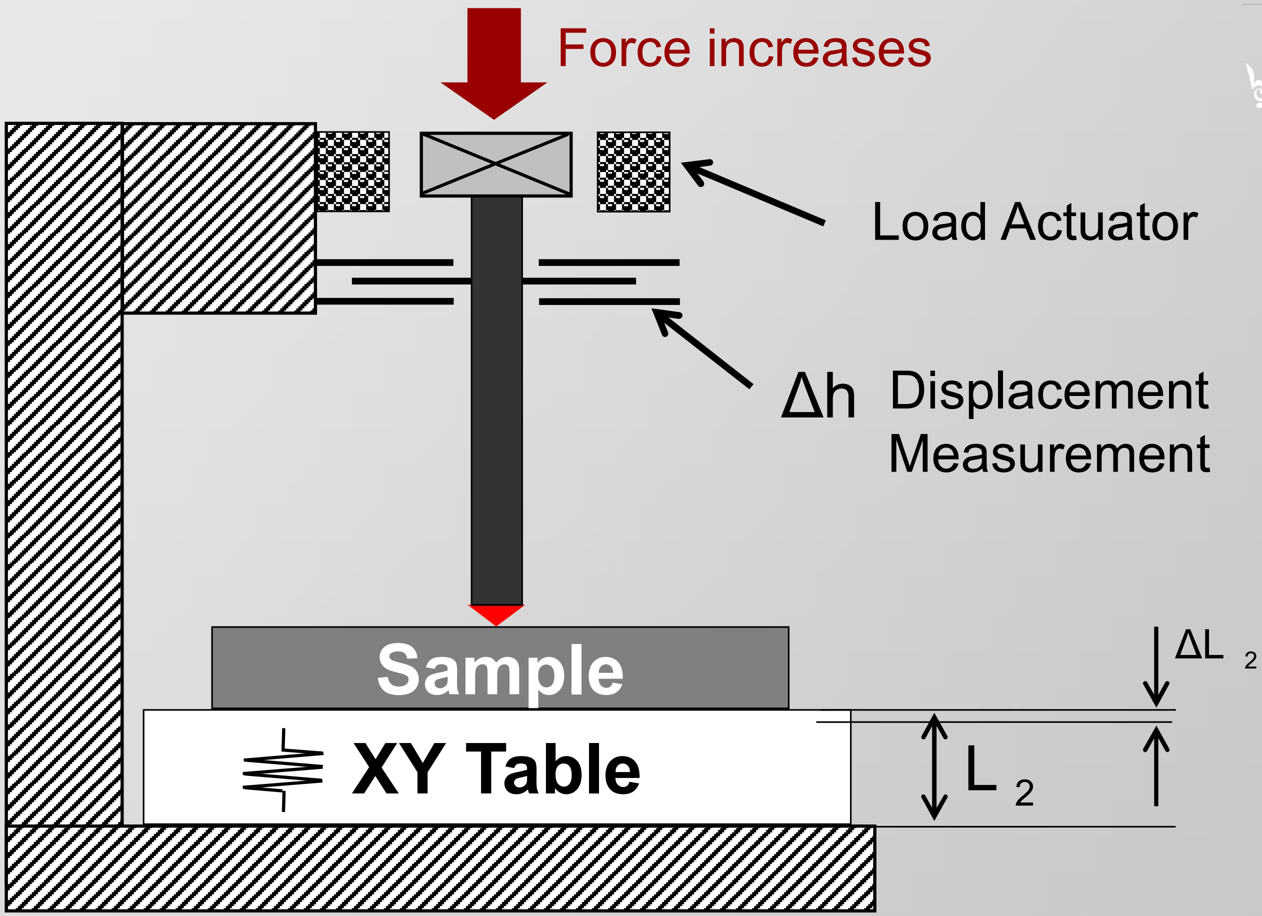
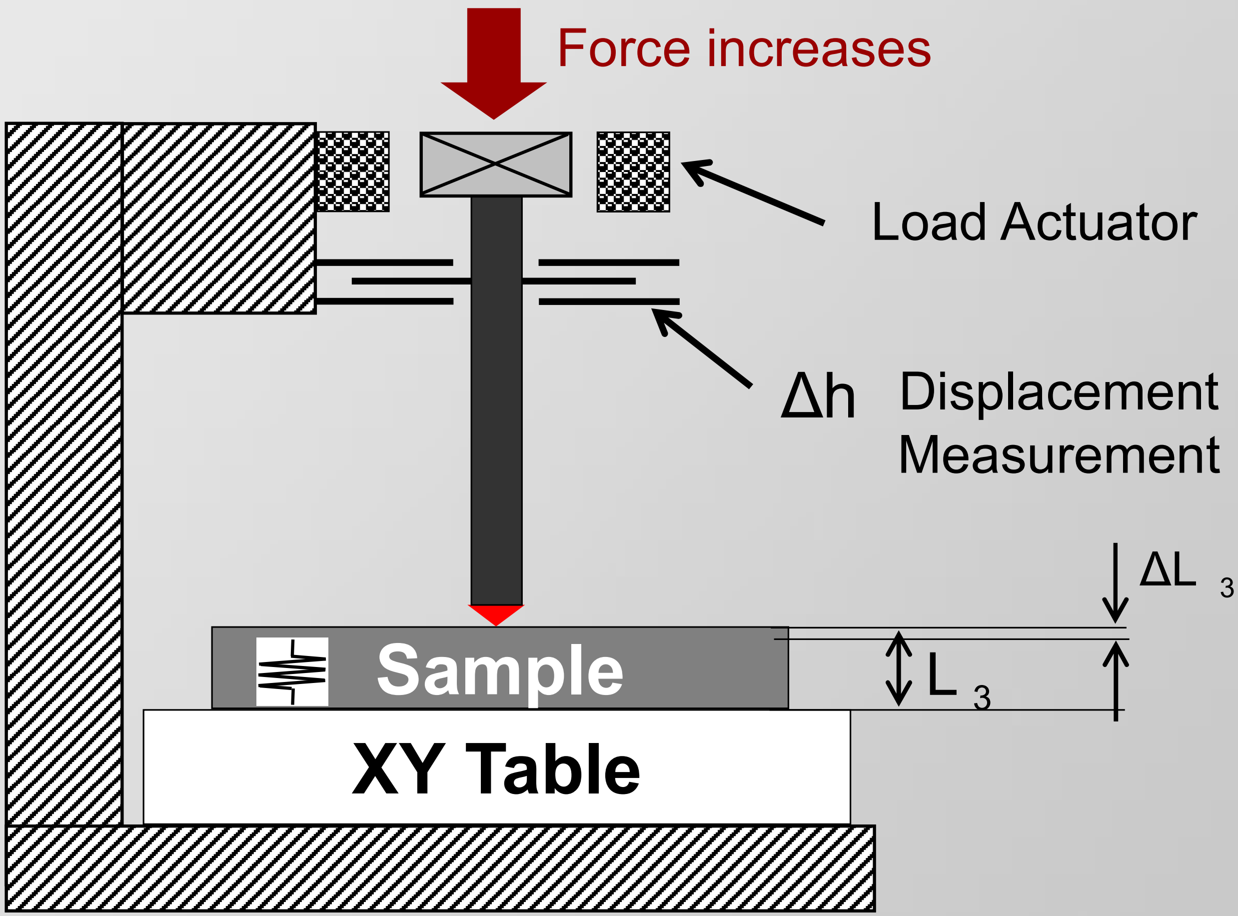


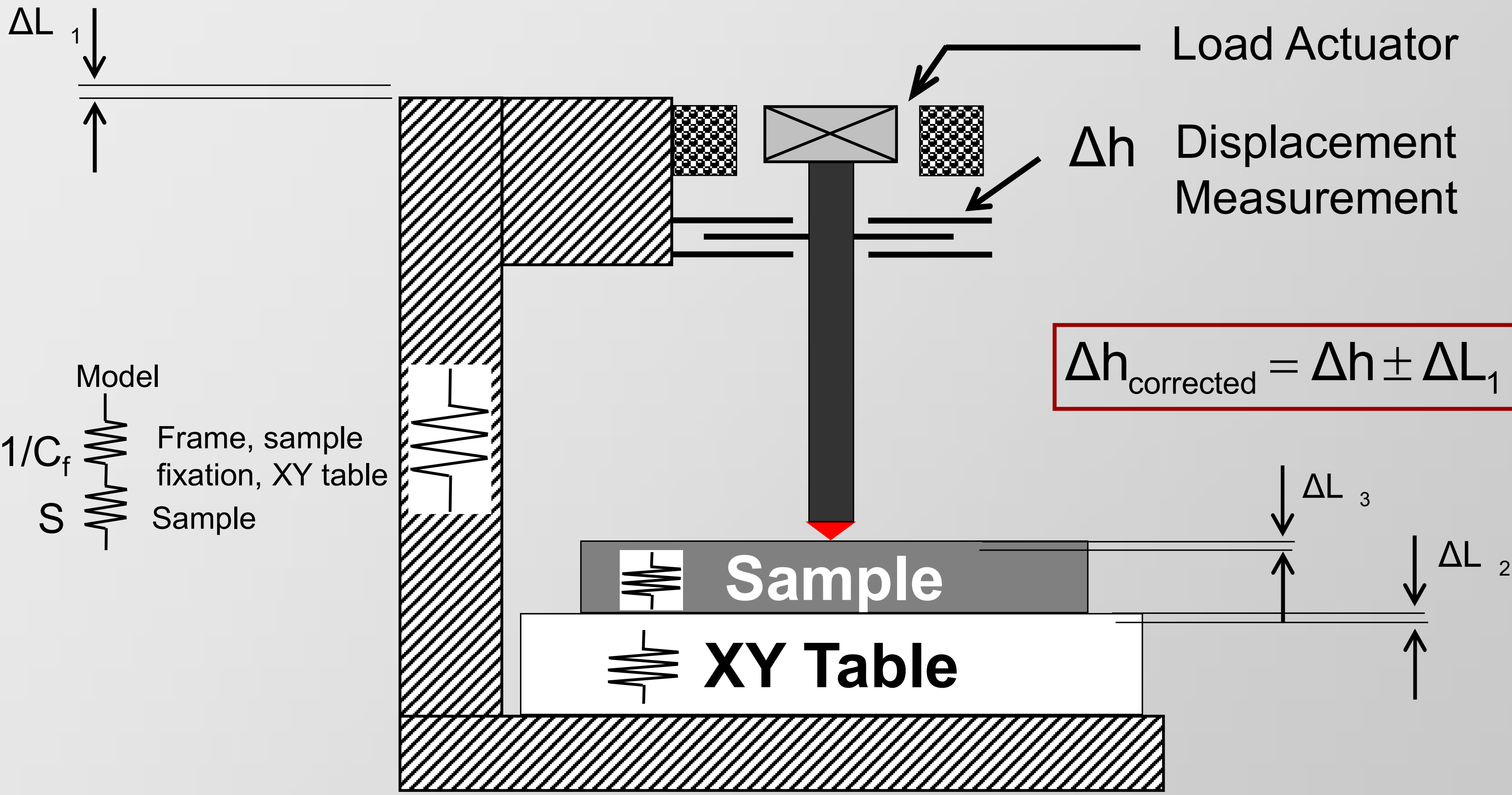
2. Frame compliance correction



2. Frame compliance correction



2. Frame compliance correction



$$\Delta h_{corrected} = \Delta h \pm \Delta L_1 \pm \Delta L_2 \pm \Delta L_3$$

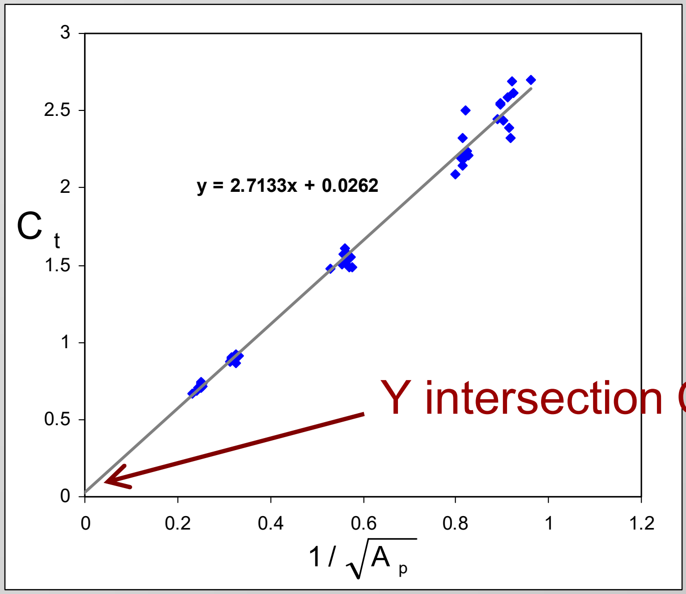
2. Frame compliance correction

Total compliance is the sum of the frame and contact compliances

$$C_t = C_f + C_s$$

$$C_t = C_f + \frac{\sqrt{\pi}}{2E_r} \cdot \frac{1}{\sqrt{A_p}}$$

Calibration of the frame compliance



Y intersection C_f = 0.026 nm/mN

Finally the correction on the indentation data is:

$$h_{corrected} = h_{measured} - C_f \cdot F$$

2. Frame compliance correction

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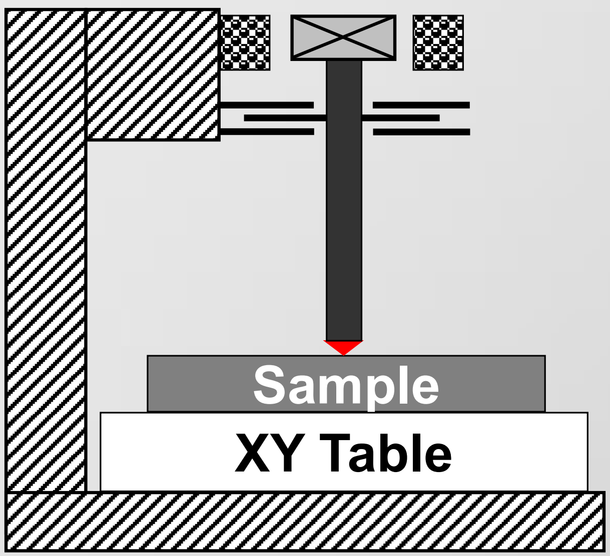
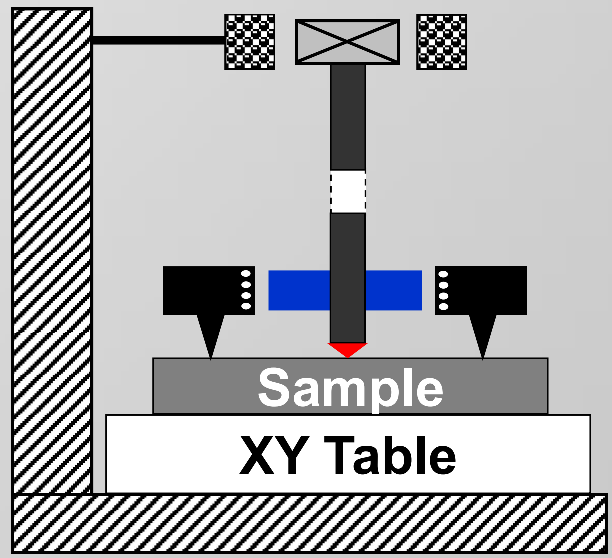
Instrument with extremely low frame compliance

Conventional indentation tester
Frame compliance > 0.5-1 μm/N



Indentation tester with top referencing
Frame compliance < 0.1 μm/N



Due to smaller frame length and more compact design, instruments with top referencing have 10 times smaller influence of frame compliance on measurements

3. Contact point detection

- One of the most important events in a nanoindentation test is the point of initial contact of the indenter and the sample.
- It is this position of the indenter that is the reference for all the depth measurements.

3. Contact point detection

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- There are a variety of methods used to determine the point of contact.
- For example, the indenter approaches until:
  - A very low force is recorded.
  - A deflection of the displacement versus time is recorded.
  - A very low contact stiffness is recorded.

3. Contact point detection

FORCE CURVES

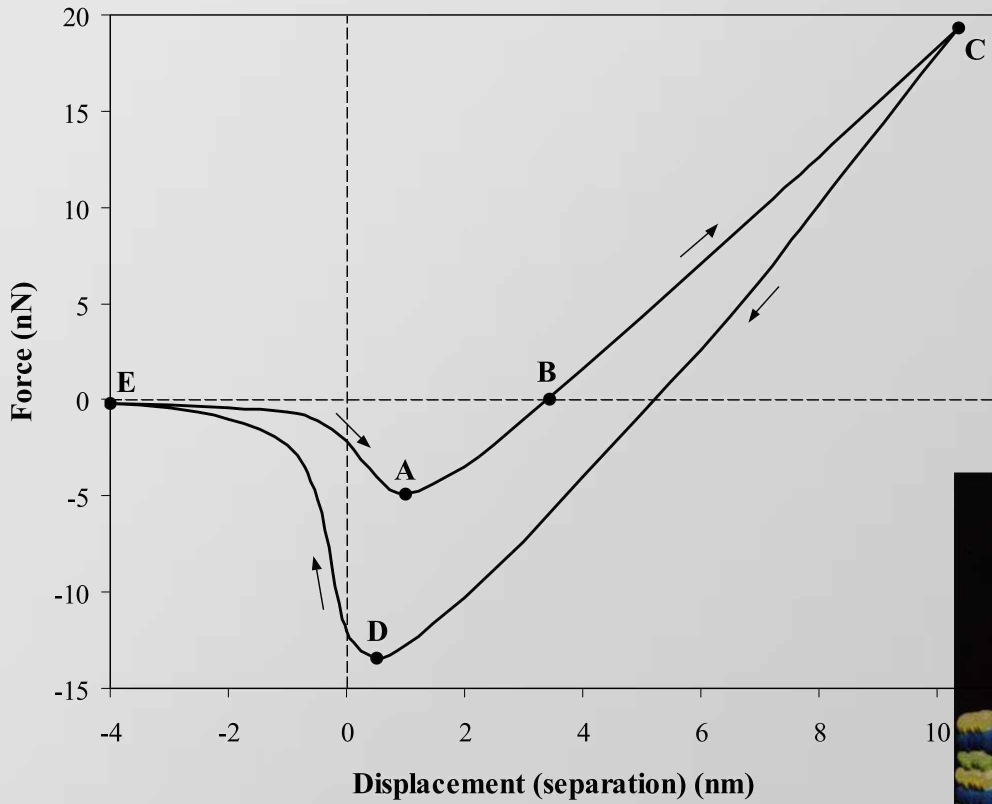
A: Maximum pull-on force

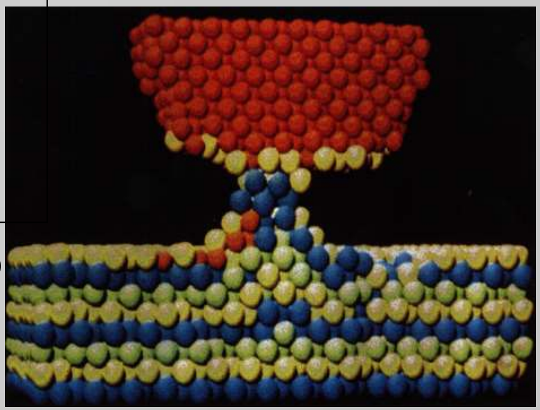
B: Zero applied load

C: Largest applied load

D: Maximum pull-off force

E: Separation





3. Contact point influence

The Modulus and Hardness results are directly affected by the contact point detection.

The influence of the contact point is higher for lower indentation depth.

Error of 5 nm made on contact point depth on Fused Silica		
F_m	Error on E_{IT}	Error on H_{IT}
100 mN	0.8%	1.5%
1 mN	8.5%	14.7%

It is then very important to verify, and correct it if necessary, the contact point in order to achieve correct E and H results.

Instruments used in this course

Anton Paar TTX-NHT[2]
(Load-controlled)

Load range: 0.1 - 500 mN
Reference ring around indenter
Robust and reliable
High Frame Stiffness
High throughput

Alemnis Standard Assembly (ASA)
(Displacement-controlled)

Load ranges: 0.5, 1.5, 2.5, 4.0 N
Can be used in-situ or ex-situ
Robust and reliable
High Frame Stiffness
High throughput

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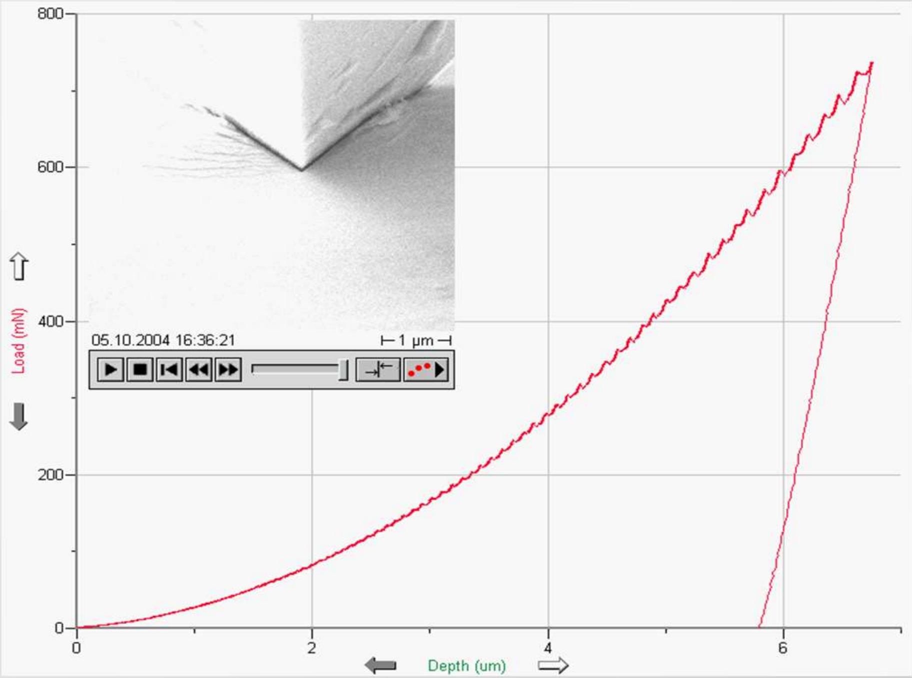
Load vs. Displacement Control

Both control modes are acceptable for shallow indents in hard materials



Load vs. Displacement Control

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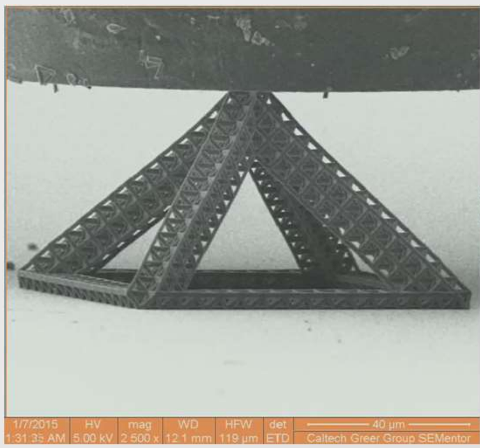
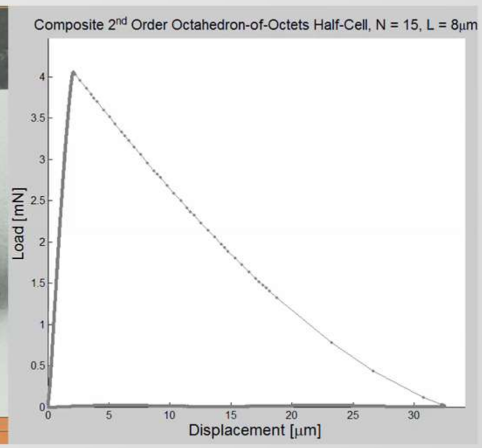
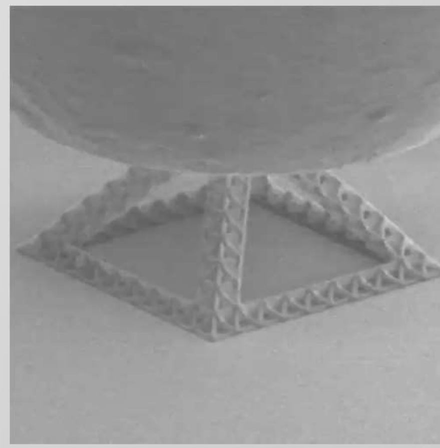
But what happens with softer structures and materials..?

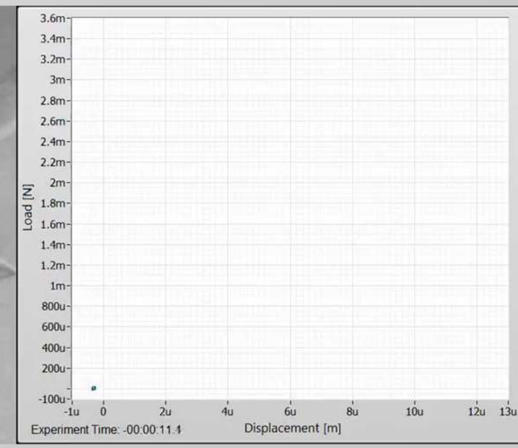
LOAD CONTROL

DISPLACEMENT CONTROL









Anton Paar NHT² head design

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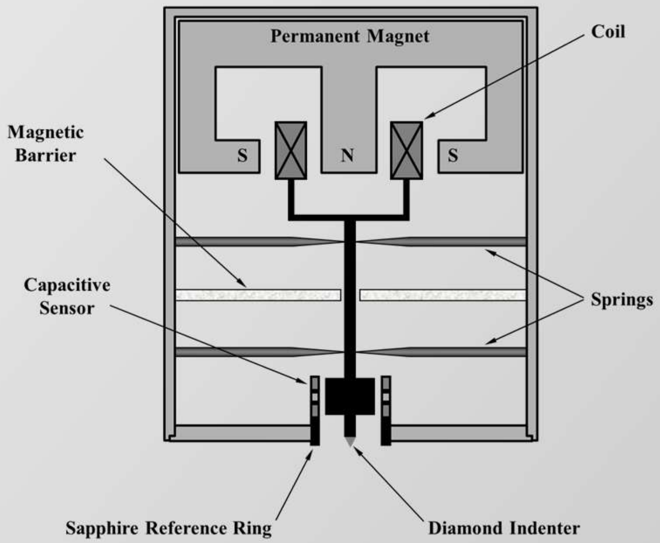

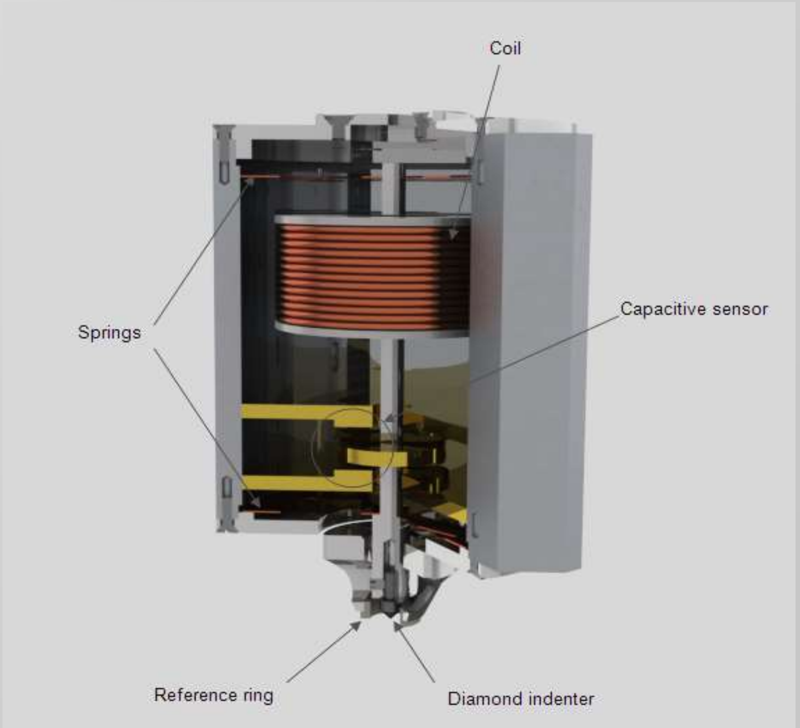

Nanoindentation Tester NHT^2

Alemnis Standard Assembly (ASA) design

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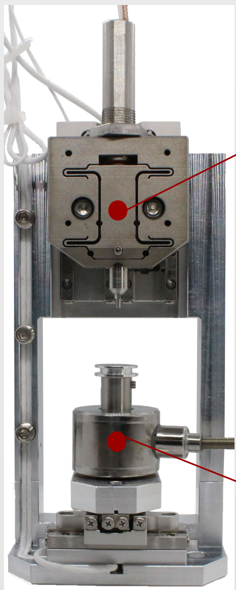


DISPLACEMENT HEADS

Displacement Head (DH)

DH-20 Displacement range up to 20 µm (rec. for high strain rate)

DH-40 Displacement range up to 40 µm (standard with ASA)

DH-100 Displacement range up to 100 µm

SmarTip

The SmarTip is a piezoelectric transducer which can be used either as an actuator or as a load sensor. It can be mounted either on the indenter side of the ASA or on the sample side, depending on use.

LOAD CELLS

Standard Load Cell (SLC)

SLC-0.5 Load range up to 0.5 N (typ. 4 µN RMS noise) µN RMS noise)

SLC-1.5 Load range up to 1.5 N (typ. 8 µN RMS noise) µN RMS noise)

SLC-2.5 Load range up to 2.5 N (typ. 15 µN RMS noise) noise)

SLC-4.0 Load range up to 4.0 N (typ. 30 µN RMS noise)

Mini Load Cell (MLC)

MLC-0.5 Load range up to 0.5 N (typ. 4

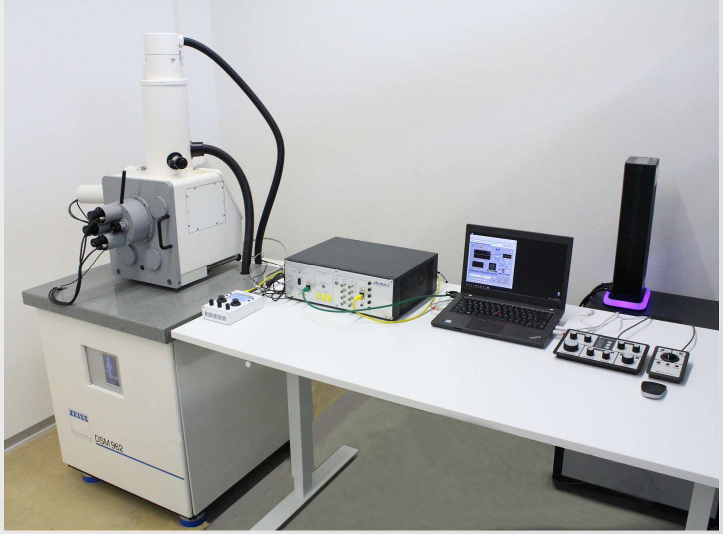
MLC-1.5 Load range up to 1.5 N (typ. 8

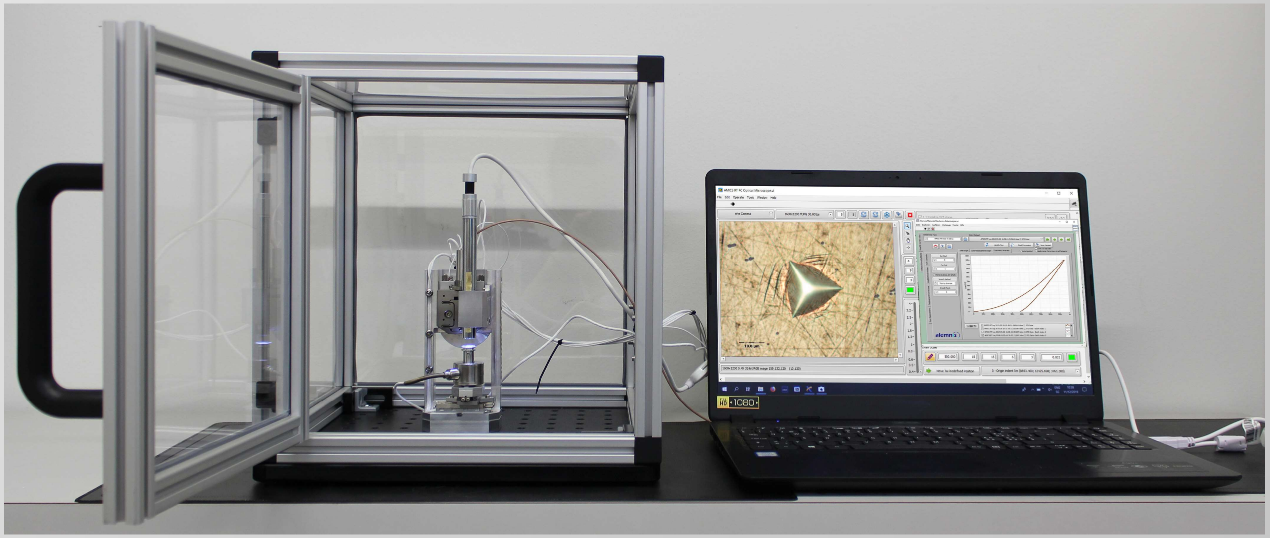
MLC-2.5 Load range up to 2.5 N (typ. 15 µN RMS

MLC-4.0 Load range up to 4.0 N (typ. 30 µN RMS noise)

Alemnis Standard Assembly (ASA) design

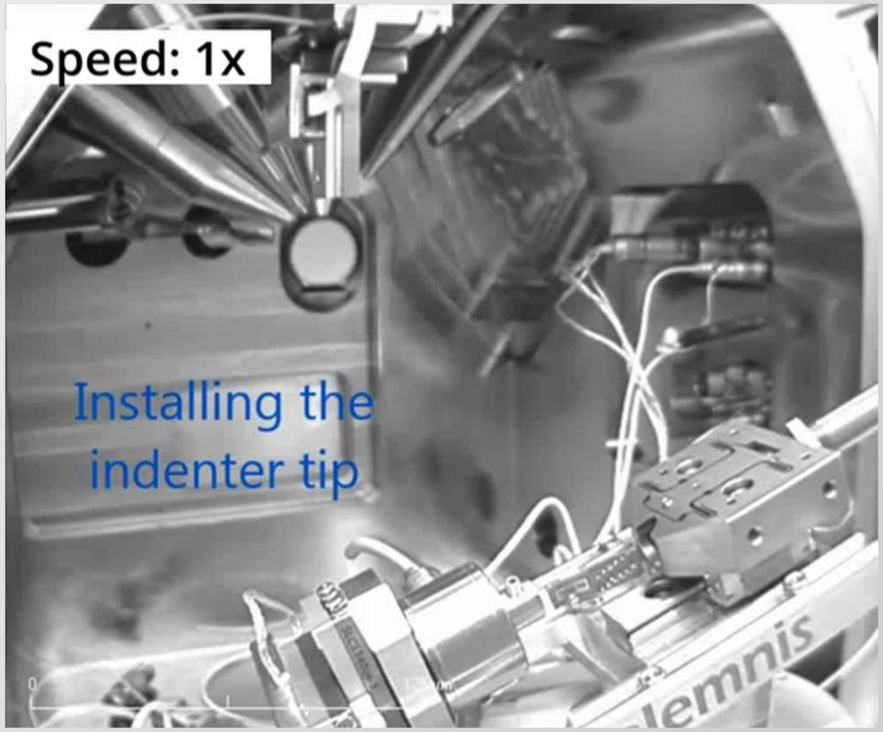
ASA can be used either in-situ or ex-situ:





Alemnis Standard Assembly (ASA) design

Typical in-situ operation:



Alemnis Standard Assembly (ASA) design

alemnis

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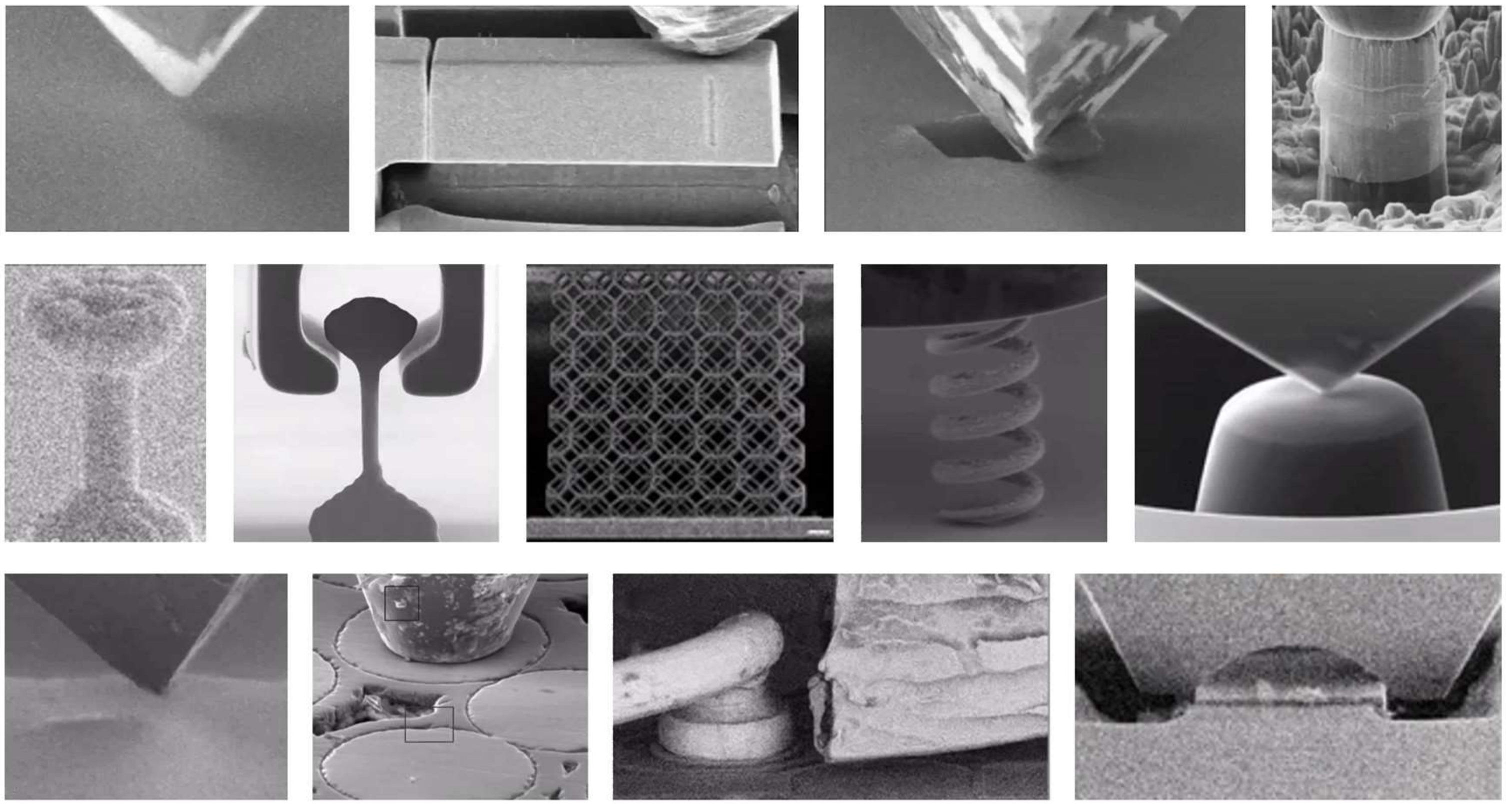

Summary

- Conventional hardness uses optical measurement of contact
- Instrumented indentation instruments designed with sensitive sensors
- Instrument design should minimise thermal drift and optimise frame stiffness
- Sensor design to maximise measurement sensitivity
- Limited ranges of displacement and force
- Load or displacement control depending on sample