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Instrumented Indentation Short Course

Andy Bushby

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Aims and objectives

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- Understand the basics of instrumented indentation testing
- Design and perform indentation experiments
- Recognise unusual materials behaviours
- Identify potential measurement artefacts
- Understand measurement uncertainties
- Interpret indentation data
    - with property variations in depth – coatings
    - with spatially varying property variation – mapping
- Recognise industrial standards
- Interpret relevant case study data

1. Introduction to Indentation

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Why mechanical properties?

- performance indicator – elastic properties
  – deformation behaviour
- sensitive to microstructure

Why nanoindentation?

- reduced scale
- surface properties
- small amount of material available
- components in a composite or a thin film
  (how can you tell if your hard disc drive has a dlc coating?)

A brief history of indentation testing

Hardness testing

1900s – Brinell ball indentation test
1920s – Vickers hardness test
1950s – **David Tabor**
The hardness of metals
1960s – micro-indentation
1970s – **Ken Johnson**
Contact mechanics

Nanoindentation

1984 – first nanoindentation instrument
1990s – instrumentation development
2000s – ISO 14577 adopted

Indentation tests

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Macro Hardness

Vickers'
Rockwell
Brinell
Knoop

Force range:
1500N $\rightarrow$ 10 N

Micro-hardness

Vickers'
Knoop

Force range:
10N $\rightarrow$ 1 N

Nanoindentation

Instrumented

Force range:
1N $\rightarrow$ 0.00000001 N

Visual measurement

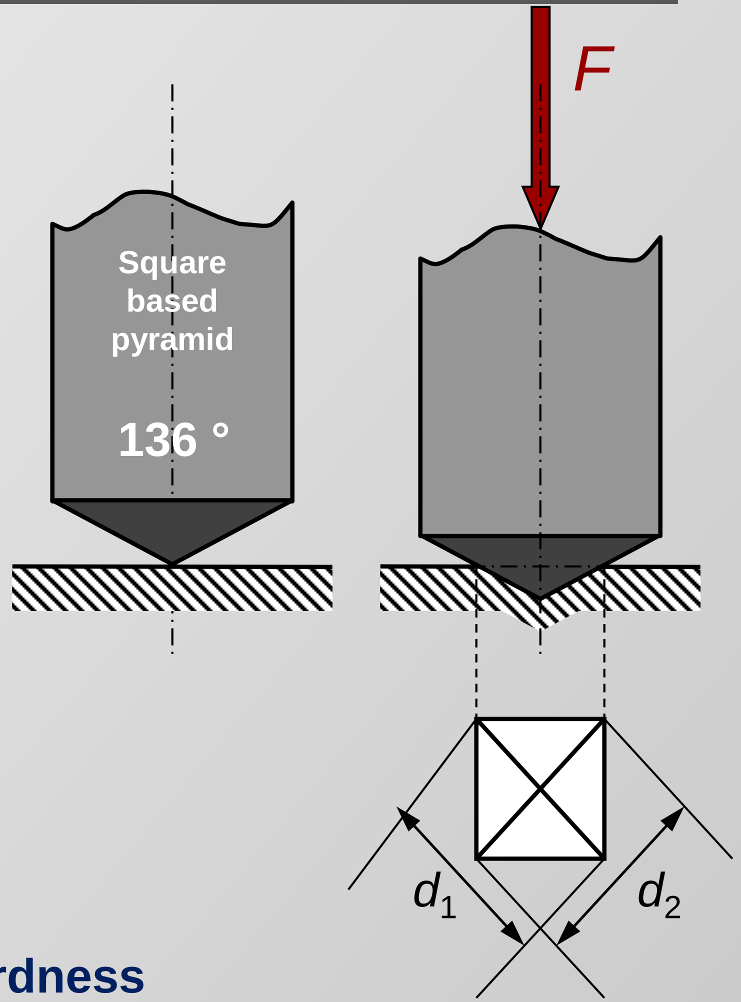
Instrumented measurement

Conventional (macro-scale) hardness testing

> Vickers Hardness, HV

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

F in N
d_1 and d_2 in mm



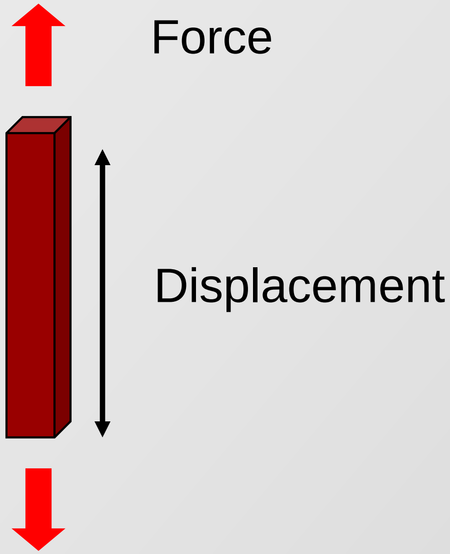
> Limitations of the technique:

- Optical observation required
  => limitation at low load

- No Automatic measurement of hardness

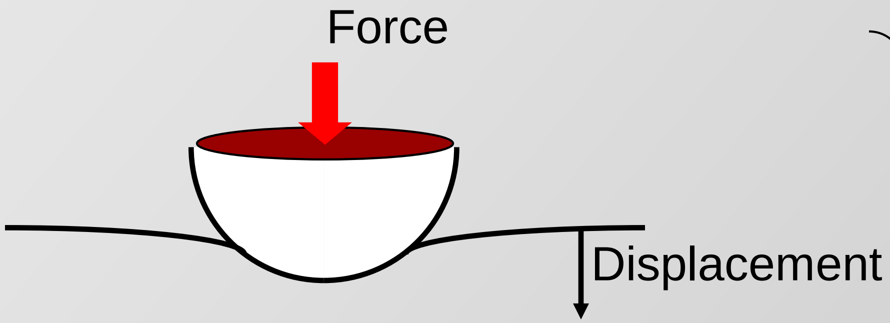
- Highly user dependent

Indentation vs tensile tests



Geometry + Mechanics

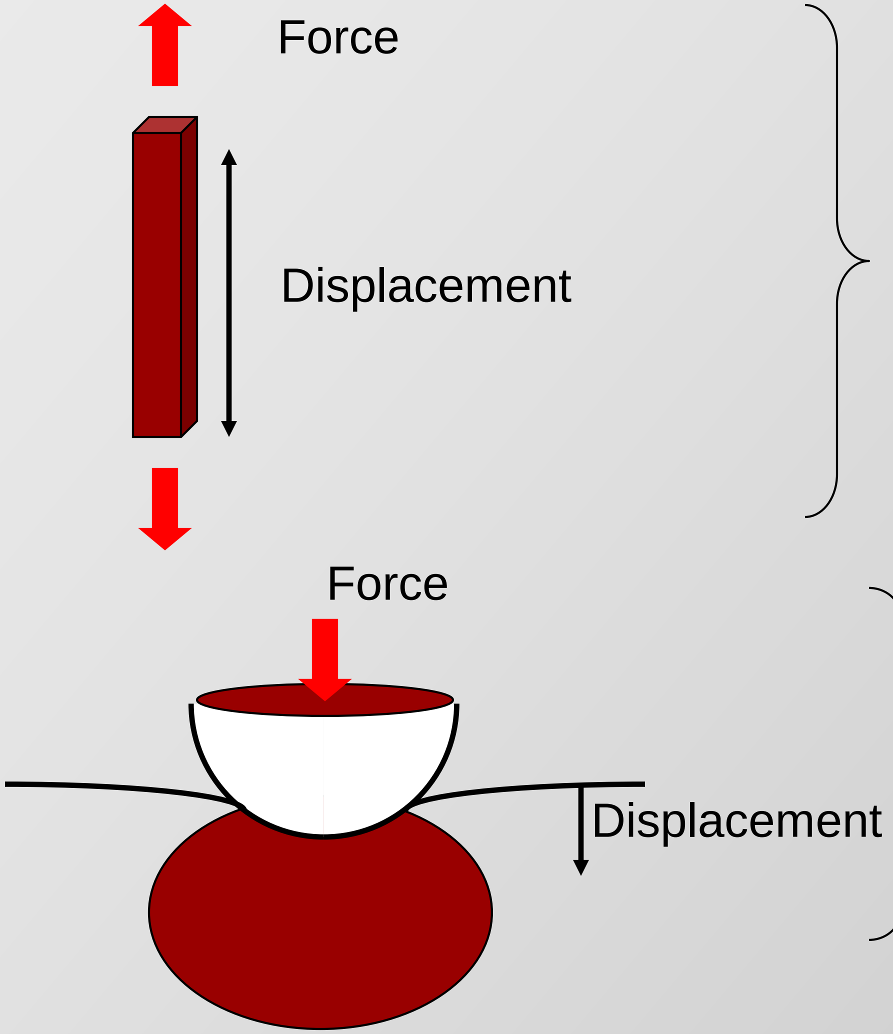
= Material Properties



Geometry + Contact Mechanics

= Material Properties

Indentation vs tensile tests



Test volume:

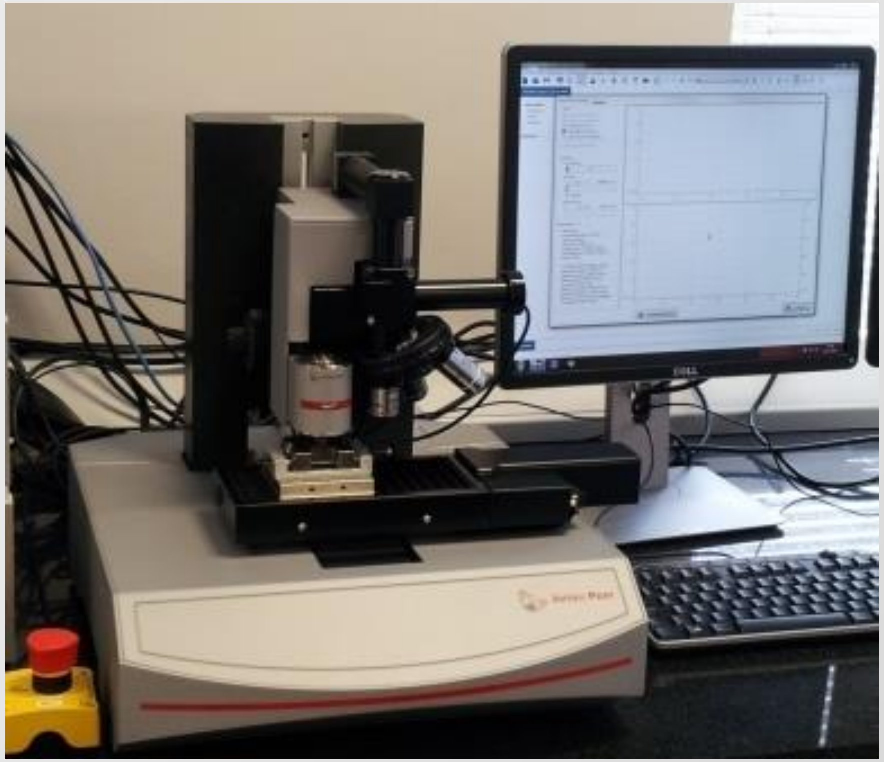
macro ~ 250mm^3

micro ~ 2.5mm^3

10 – 1000 μm^3 (10^{-9} mm^3)

a billion times smaller!

Instrumented indentation testing



Force range 1μN – 500mN

Force sensitivity 0.1μN

Depth range 1nm – 20 μm

Depth sensitivity 0.1nm

nanometre range!

<u>Specimen requirements</u>:

Flat surface

Low surface roughness

Supported underneath

Indenter geometry

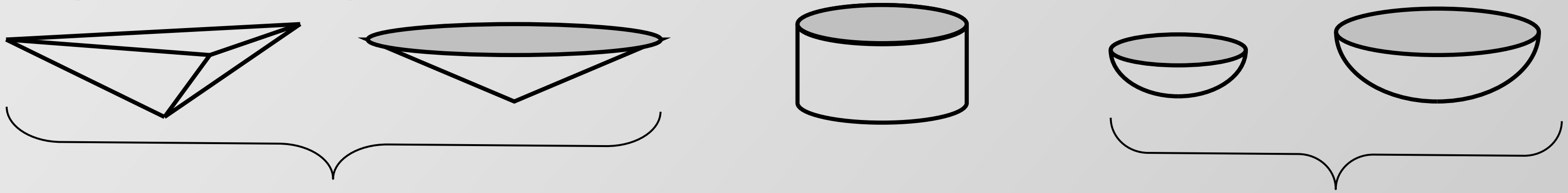
Tip shapes: (usually made from diamond)

Pyramid
(Berkovich)
(Cube corner)

Cone

Punch

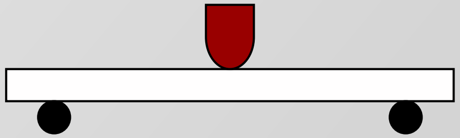
Spheres



Pointed / Sharp
Fixed geometry

Spherical / Blunt
Variable geometry

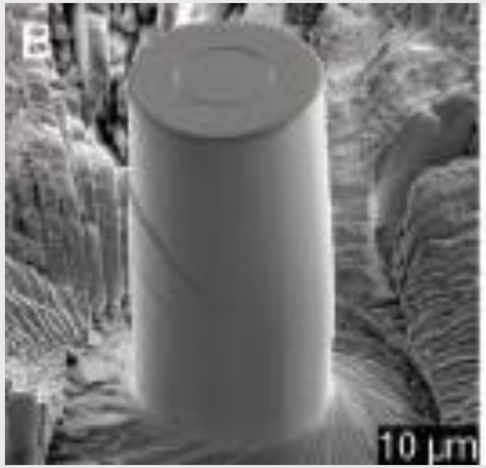
Load frame:



Flexure:
Simple bending
3 point flexure
Bi-axial flexure

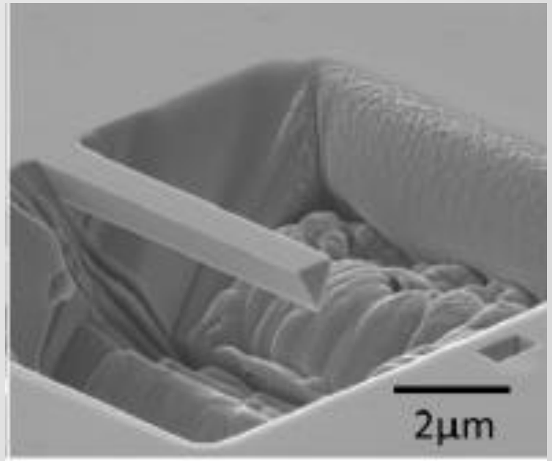
Compression:
Pillars
Spheres

Other mechanical testing geometries



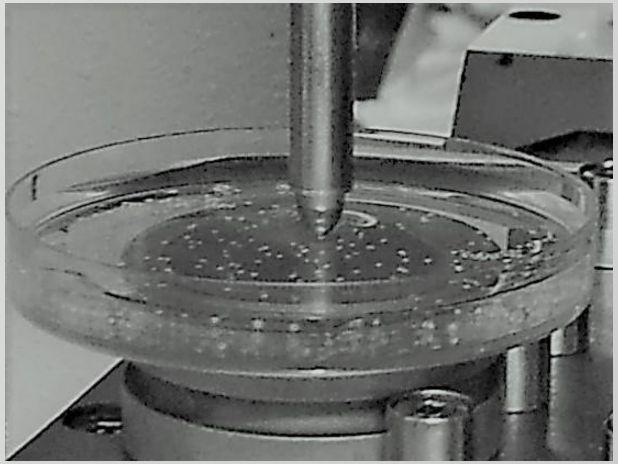
Micro-pillar compression

Compressive strength



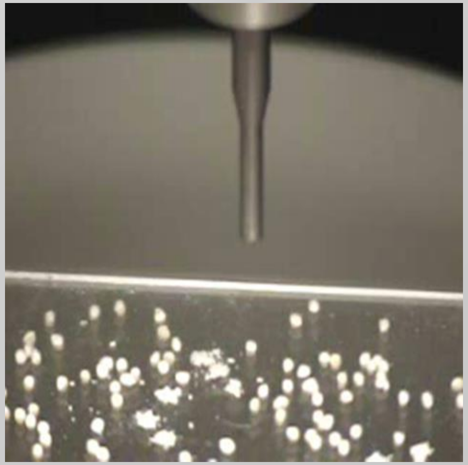
Micro-cantilever flexure

Fracture mechanics



Individual gel bead compression

Elastic properties

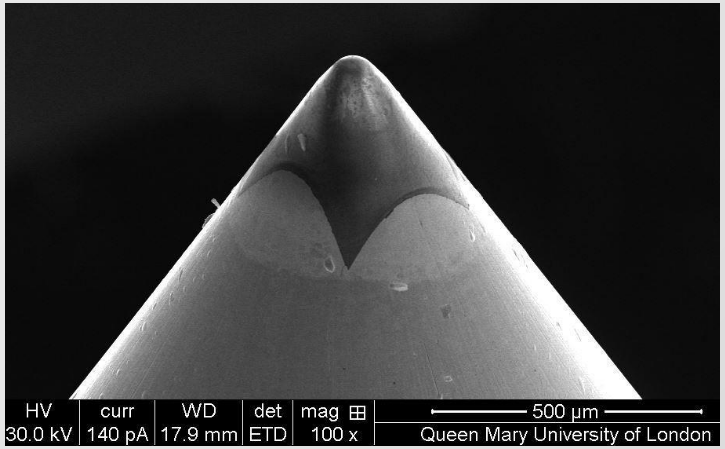


Particle crushing

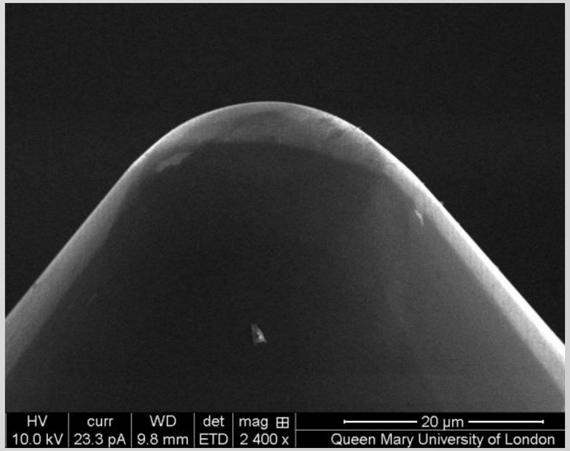
Fracture strength

Indenter geometry

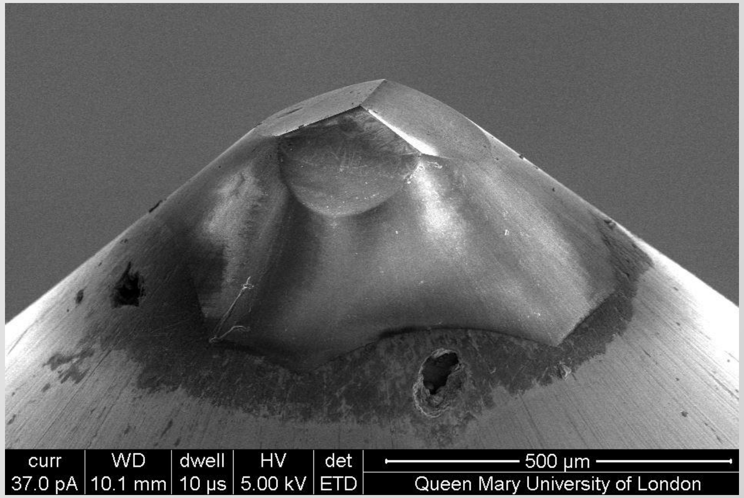
Diamond brazed into steel and polished



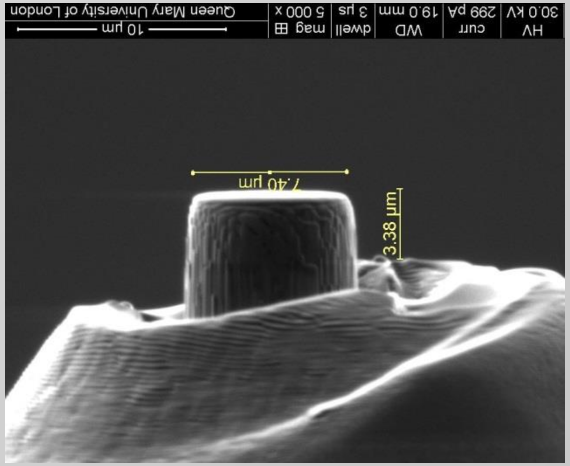
Spherical indenter tip



Berkovich pyramid indenter tip

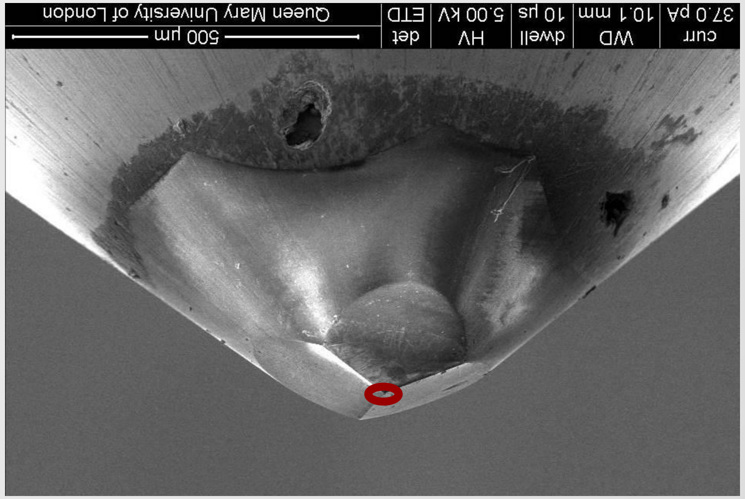


Flat punch indenter tip

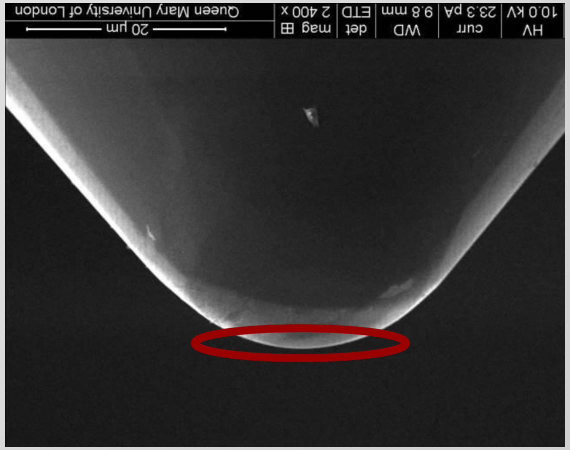


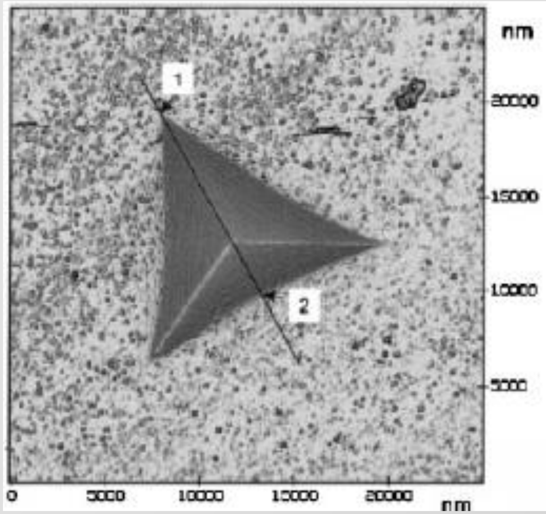
Making an impression

Berkovich pyramid indenter tip

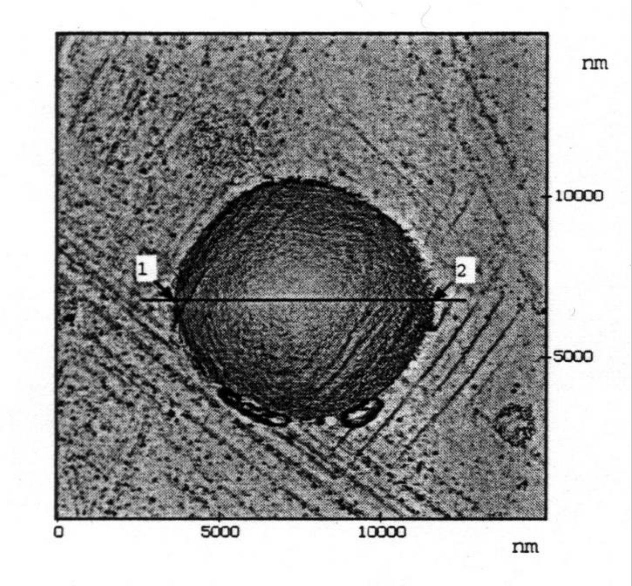


Spherical indenter tip

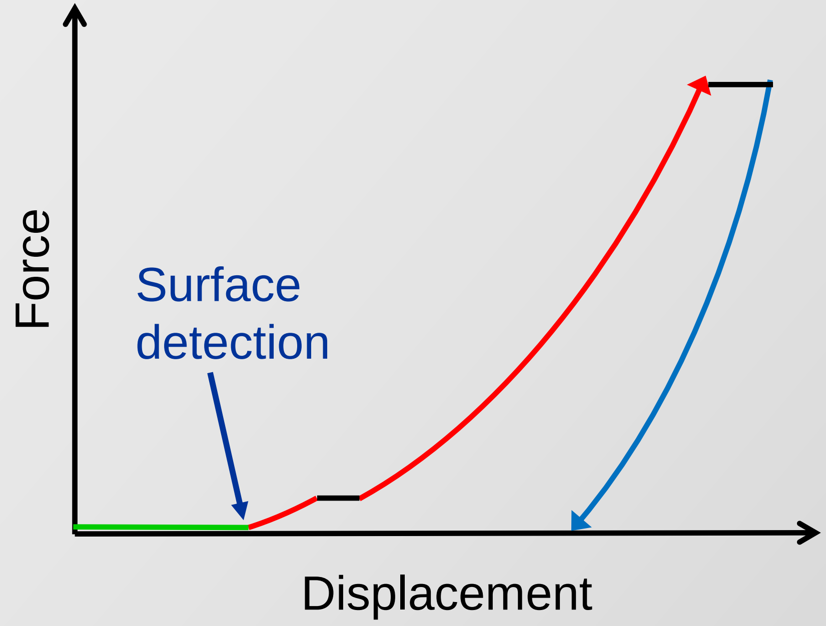




AFM scans
of impressions



Typical nanoindentation load cycle



Approach
Loading
Hold
Unloading

Hold constant force to assess thermal drift or creep
Control load rate to assess strain rate sensitivity

Simple load-unload cycle

Typical load cycle from 3sec - 3mins:

Load includes elastic and plastic responses
Unloading (assumed) only elastic recovery

Standards

Instrumented indentation test standard ISO 14577

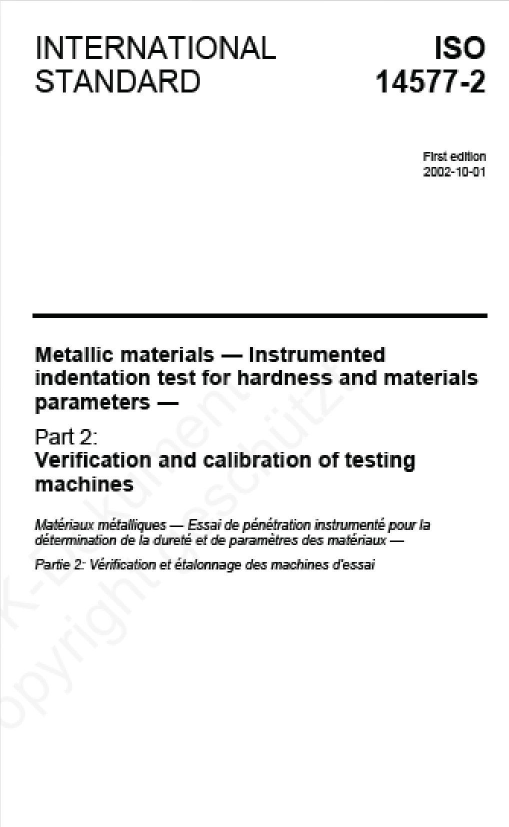
Requirements:

Instrument characteristics:

- Environment
- Thermal and mechanical stability
- Frame compliance
- Tip shape calibration

Sample characteristics:

- Mounting
- Surface roughness
- Cleanliness


INTERNATIONAL STANDARD

ISO 14577-2

First edition
2002-10-01

Metallic materials — Instrumented indentation test for hardness and materials parameters —

Part 2:
Verification and calibration of testing machines

Matériaux métalliques — Essai de pénétration instrumenté pour la détermination de la dureté et de paramètres des matériaux —

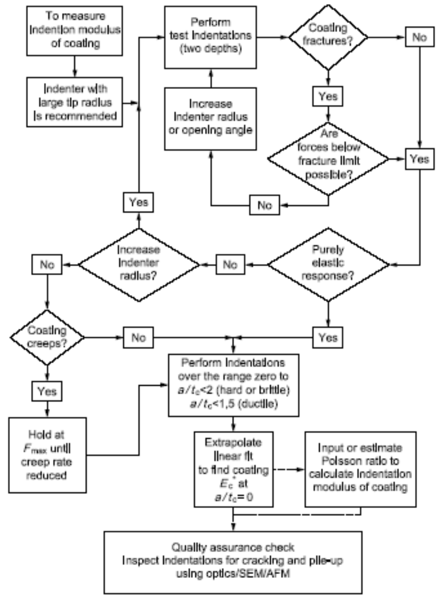
Partie 2: Vérification et étalonnage des machines d'essai

Standards

Instrumented indentation test standard ISO 14577

Selection of test parameters:

- Choice of tip shape
- Load and depth ranges
- Load cycle
- Load rate



b) For indentation modulus of coating

Data treatment:

- Recording of parameters and data
- Analysis methods
- Consideration of uncertainties

ISO 14577-1:2002(E)

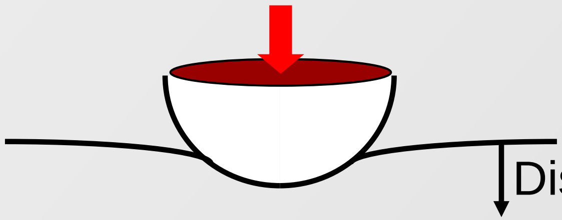
A.4.2 Designation of indentation hardness H_{IT}

EXAMPLE

H_{IT} 0,5 / 10 / 20 / 30 = 11 300 N/mm²

- Hardness value
- Time taken to remove the test force during the fitted portion of the test force removal curve in seconds
- Duration time of test force in seconds
- Application time of test force in seconds
- Test force in newtons

Materials properties



Geometry + Contact Mechanics

= Material Properties

Elastic properties: elastic modulus
important for design, modelling, fracture, residual stress

Plastic properties: yield stress / hardness
important for strength, fatigue, creep, wear

Time dependant properties: creep / viscoelasticity
important for polymers / soft solids / biomaterials

Fracture properties: toughness
important for ceramics / glasses

Electro-mechanical properties: ferroelectric response
important for functional materials and devices