Nanoindentation Technique Introduction

21 Jan 2025

Leo CH Lee
Manager – Materials , Design and Manufacturing Facility

MDMF – MAT Unit

The focal point of MAT unit of MDMF is the research laboratory located in rooms 4228-4230 & 4224 of the Academic Building on the HKUST campus.

Analytical, processing and testing equipment is available.

This Equipment includes instrumentation for surface and interfacial analysis, for characterization of physical properties, and for mechanical properties testing of advanced engineering materials

https://mdmf.hkust.edu.hk/cads
https://mdmf.hkust.edu.hk/mat

Rm 4224 Rm 4228-4230

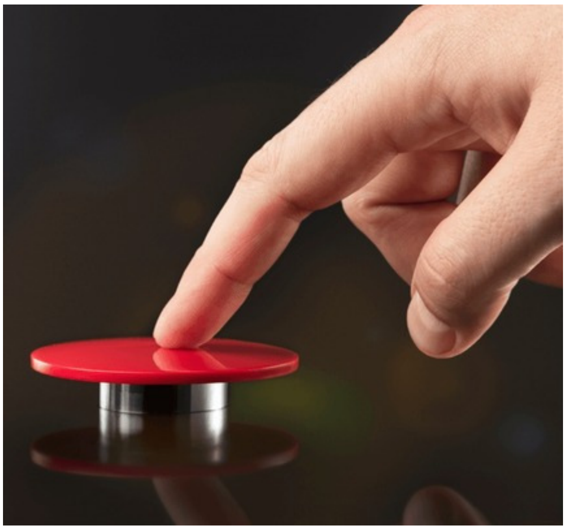
MDMF – MAT Unit

Mechanical Properties Testing

Mechanical Properties Testing Machine	
MTS Universal Testing Machine : 810, 858, 858Torsional	Used to test the tensile strength (pulling) and compressive strength (pushing), flexural strength, bending, shear, and torsion testing.
Nano Indenter : Hysitron, Triboindenter TI 900, TI 980	Used to measure a variety of nanoscale mechanical properties. In addition to hardness measurements, nanoindenter-based techniques enable elastic modulus, creep, viscoelastic properties, interfacial adhesion, and fracture toughness characterization

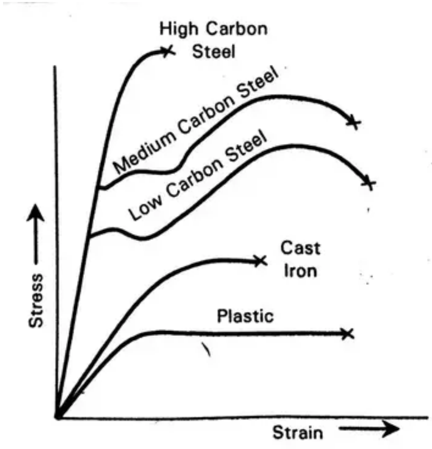
Outline

- Materials Mechanical Properties
- Nanoindentation/Bruker Hysitron System
- Contact Mechanics Model – Elastic Modulus & Hardness Determination
- Factors to consider for nanoindentation
- Variations from Ideality/Other Mechanical Properties Measurement



Materials Mechanical Properties - Tensile Test

Each material has its own unique properties and tensile curve that make it useful for different purposes.
For example : High Carbon steel is Strong & Stiff and Plastic is often Lightweight and Flexible



Background Review

What is Modulus , Hardness , Elastic Deformation, Plastic Deformation ?

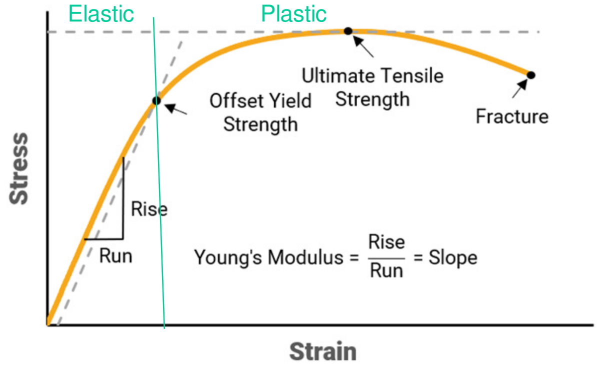
Materials Mechanical Properties - Tensile Test

In General, tensile testing provides material mechanical properties such as

•Young's Modulus

•Yield Strength/Ultimate Tensile Strength

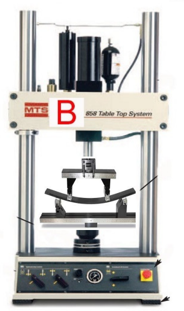
•Ductility



Materials Mechanical Properties - Tensile Test

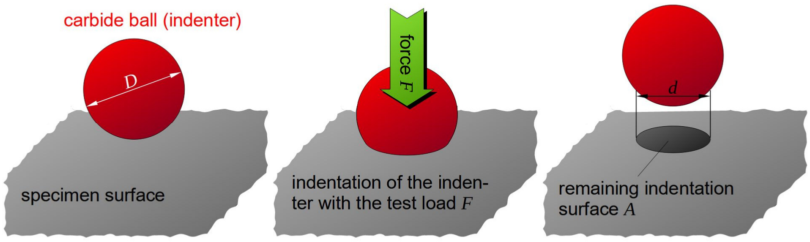
MAT is equipped with four Universal Testing Machines of capacities:

- Tension/Compression/Dynamic : 100 KN
- Tension/Compression/Dynamic : 15 KN
- Flexural/Compression/Dynamic : 5 KN
- Tension/ Torsion/ Compression/Dynamic : 25 KN / ± 200Nm



Hardness Testing

In materials science, hardness (antonym: softness) is a measure of the resistance to localized plastic deformation, such as an indentation (over an area) or a scratch (linear), induced mechanically either by pressing or abrasion.



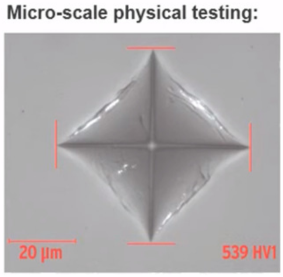
Why Nanoindetation

Primarily used for measuring mechanical Properties of thin films or small volumes of material

- ✓ Elastic Modulus
- ✓ Hardness
- ✓ Viscoelastic Properties/Creep
- ✓ Cracking/Fracture Toughness

Macro-scale Physical Testing

Micro-scale physical testing:



Source: ASTM, Standardization News May/June 2010 Microindentation Hardness

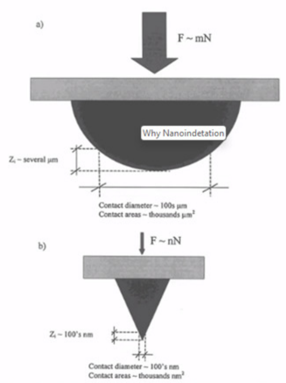


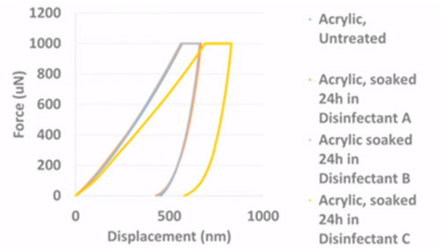
Figure 2. Schematic comparison of the surface adhesion techniques: (a) microindentation; (b) nanoindentation. The penetration depth characteristic of each is noted as Z_i.

Source: Paiva et al., Macromolecules 2001, 34, 2269-2276

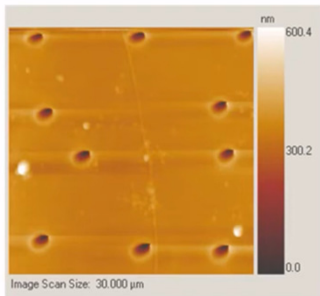
MATERIALS, DESIGN AND MANUFACTURING FACILITY(CWB)

Practical Application : Effect of disinfectant chemistry on polymer

Nanoindentation used in this study to Determine effect of disinfectant chemistry on polymer materials found in healthcare facilities.



Source:
Strader et al.. **Nano Life;** Vol. 9, Iss. 4, (Dec 2019). DOI:10.1142/S1793984419500028



Scanned Probe Microscopy (SPM) Image of acrylic sample post-indentation

	Reduced Modulus (GPa)	Hardness (MPa)
Acrylic Untreated	5.20 GPa ± 0.03	356.5 MPa ± 6.09
Acrylic Disinfectant A	4.97 GPa ± 0.03	335.1 MPa ± 1.53
Acrylic Disinfectant B	5.28 GPa ± 0.05	355.3 MPa ± 3.26
Acrylic Disinfectant C	4.50 GPa ± 0.04	256.0 MPa ± 3.26

Average modulus and hardness measured for each sample

MATERIALS, DESIGN AND MANUFACTURING FACILITY(CWB)

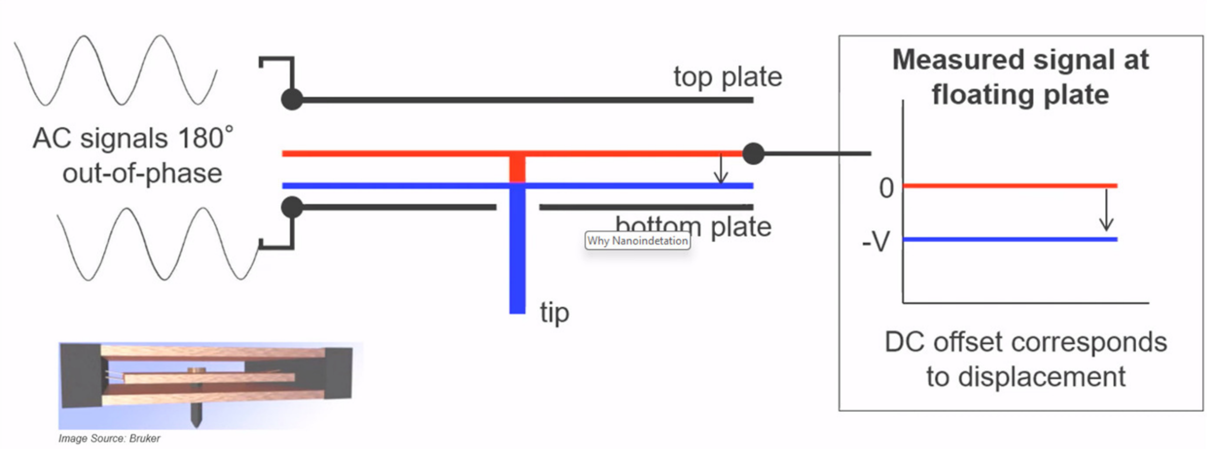
Nanoindentation

How is Nano Displacement Measured ?

- High displacement sensitivity
- High load sensitivity
- Low thermal drift

How is Displacement Measured ? Electrostatic Transducer

Three-plate capacitor with floating center plate attached to tip :



Nanoindentation

What is the available instrument and accessories in MAT?

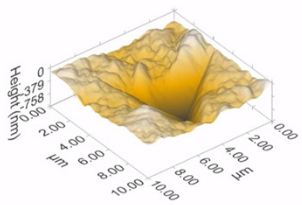
MATERIALS, DESIGN AND MANUFACTURING FACILITY(CWB)

Bruker Hysitron TI980 Triboindenter in MAT

MAT also have a larger transducer 50mN and High Load modulus10N for different application

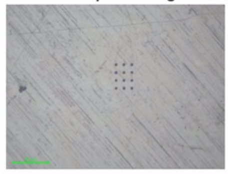
	Indentation Axis	Lateral Axis
Maximum Force	10 mN	2 mN
Maximum Indentation Displacement	5 µm	500 nm
Maximum Lateral Displacement	N/A	15 µm
Thermal Drift	<0.05 nm/sec	<0.05 nm/sec
Load Noise Floor	20 nN	3.5 µN
Load Resolution	1 nN	50 nN
Displacement Resolution	0.006 nm	0.02 nm
Displacement Noise Floor	0.1 nm	2 nm
Maximum Range of Piezoelectric Scanner	60 µm x 60 µm	60 µm x 60 µm

In situ SPM Imaging



Indented surface of bovine dentin

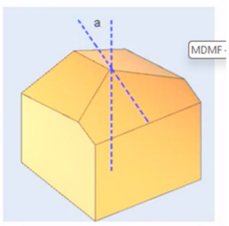
Optical Microscope to navigate sample



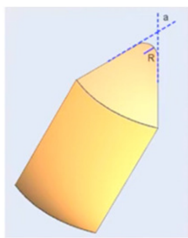
Array of indents in Polycarbonate

MATERIALS, DESIGN AND MANUFACTURING FACILITY(CWB)

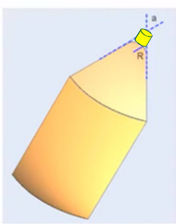
Indentation Tip Selection



Berkovich Tip
- 3-side pyramidal tip
- A= 65.35° (half angle)
- Multi-purpose tip
- Hardness and modulus testing for thin films > 100 nm



Cube Corner Tip
- 3-side pyramidal tip
- A= 35.26° (half angle)
- Fine tip
- Multi-purpose tip
- Hardness and modulus testing for thin films < 100 nm

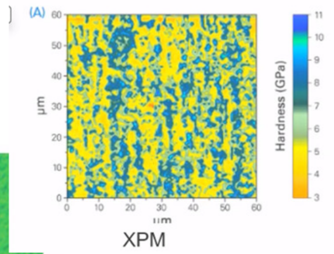


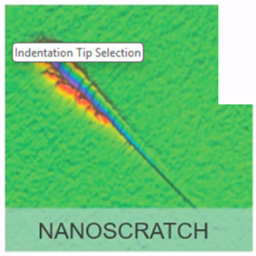
Flat End Tip
- 10 um in diameter
- Compression Testing
- Application: micro-pillar and bending test

MATERIALS, DESIGN AND MANUFACTURING FACILITY(CWB)

All Capabilities of Bruker TI980

- Quasistatic Nanoindentation
- Dynamic Nanoindentation (nanoDMA)
- Accelerated Property Mapping (XPM)
- Scanning Probe Microscopy (SPM+)
- Nanoscratch
- Nanowear





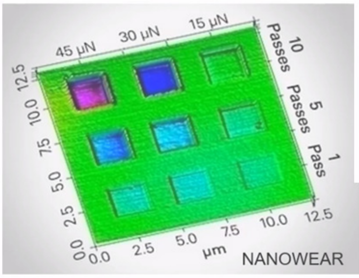


Image Sources: Bruker

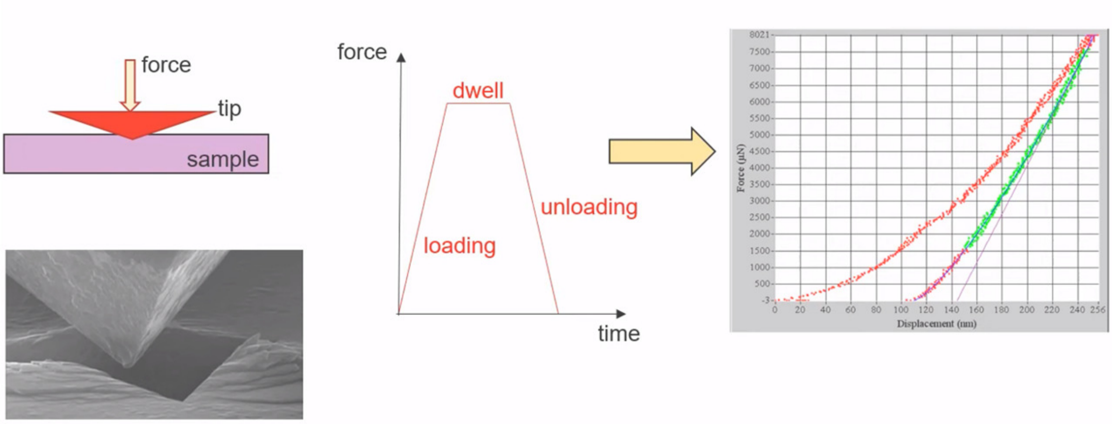
MATERIALS, DESIGN AND MANUFACTURING FACILITY(CWB)

Nanoindentation

How to measure the Hardness & Modulus using nanoindentation?

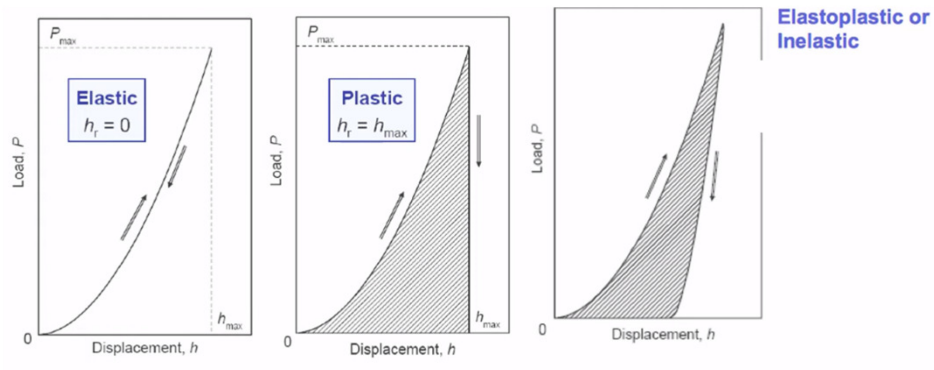
Deformation During Indentation

A rigid probe nanoindenter (like the Bruker TI980) records the vertical displacement (h) of the probe During controlled loading and unloading.



Source: Bruker

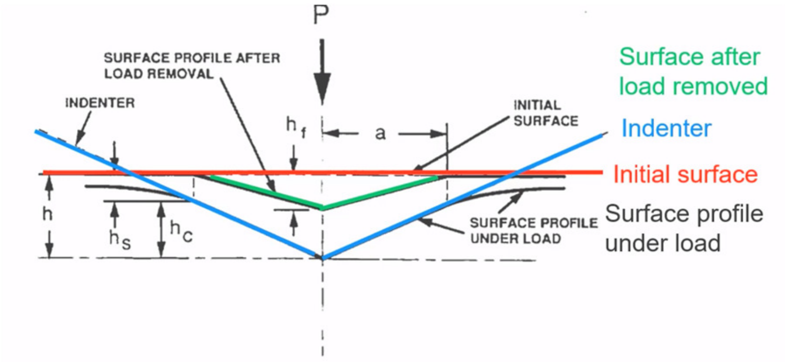
Deformation During Indentation



$h_r = h_f$ = residual / final depth

- Can treat the response of an elastoplastic material like a completely elastic material with a surface perturbation from plastic deformation . Therefore, similar contact mechanics models can be used.
- Elastic moduli and hardness can be calculated from the mechanical response of material during unloading (by **Oliver-Pharr** Method.)

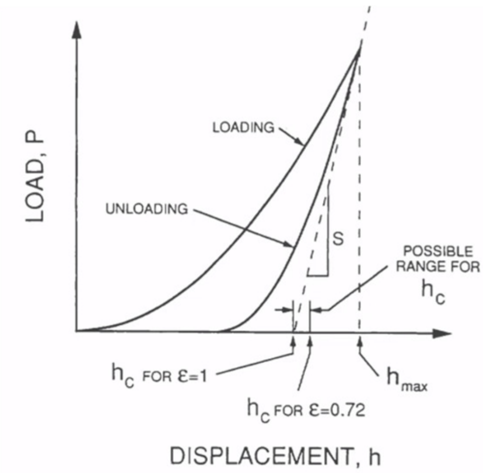
Surface Profile & Contact Depth



h = ***penetration depth***
h_c = contact depth = vertical distance along which contact is made
h_s = surface displacement at perimeter of contact (sink-in)

W.C. Oliver, G. M. Pharr. J. Mater. Res. 7[6] (1992).

Sink-in Correction (Oliver-Pharr Method)



h_{max} = *penetration depth*
h_c = contact depth = vertical distance along which contact is made
h_s = surface displacement at perimeter of contact (sink-in)

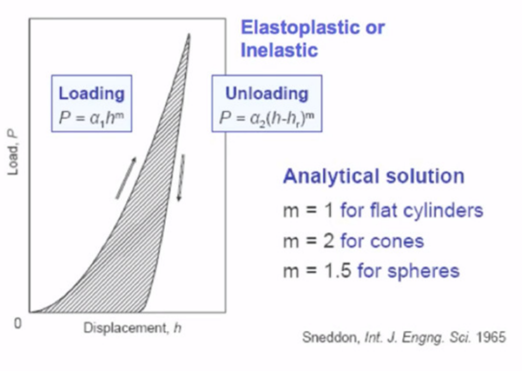
Correction for sink-in:
S = dP/dh @ P_{max} = stiffness
$h_c = h_{max} - \varepsilon P_{max}/S$
ε = geometric constant = 0.75 (paraboloid)
= 0.72 (conical)
= 1 (flat punch)

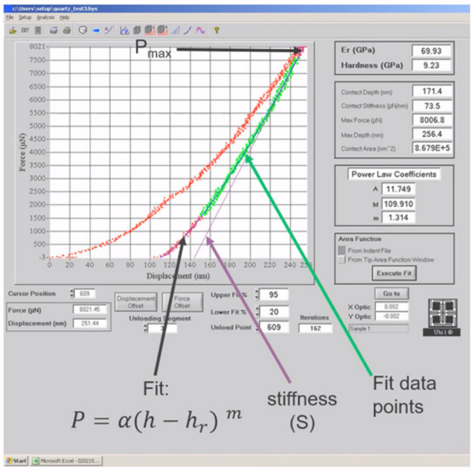
W.C. Oliver, G. M. Pharr. J. Mater. Res. 7[6] (1992).

MATERIALS, DESIGN AND MANUFACTURING FACILITY(CWB)

Unloading Curve Fit

Stifness determined from slope of function fitted to unloading curve at Pmax



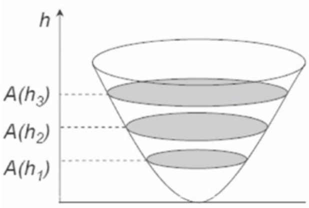


MATERIALS, DESIGN AND MANUFACTURING FACILITY(CWB)

Tip Area Function / Contact Area Determination

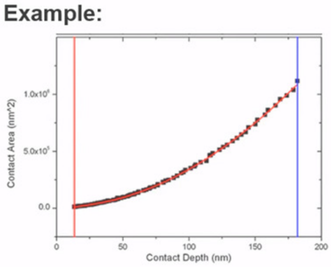
Determine tip area function by indenting a sample of known modulus (e.g., fused quartz with E_r = 69.6 GPa):

$$A(h_c) = C_0 h_c^2 + C_1 h_c + C_2 h_c^{1/2} + C_3 h_c^{1/4} + C_4 h_c^{1/8} + C_5 h_c^{1/16}$$



10 µm x 10 µm SPM Image of indented silica surface

Example:



Source: Bruker

The calculated projected areas as a function of contact depth are applied to unknown samples to determine modulus and hardness

Elastic Modulus & Hardness

MATERIALS, DESIGN AND MANUFACTURING FACILITY(CWB)

$$\frac{1}{E_r} = \frac{(1-v)^2}{E} + \frac{(1-v_i)^2}{E_i}$$

E_r = reduced elastic modulus
E_i = indenter elastic modulus = 1141 GPa (diamond)
v_i = indenter Poisson's ratio = 0.07 (diamond)

Based on stiffness and **contact area (*$A(h_c)$*)** at h_{max}, can calculate modulus and hardness (H):

$$E_r = \frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A(h_c)}} \qquad H = \frac{P_{\max}}{A(h_c)}$$

W.C. Oliver, G. M. Pharr. J. Mater. Res. 7[6] (1992).

Factors to consider for Nanoindentation

MATERIALS, DESIGN AND MANUFACTURING FACILITY(CWB)

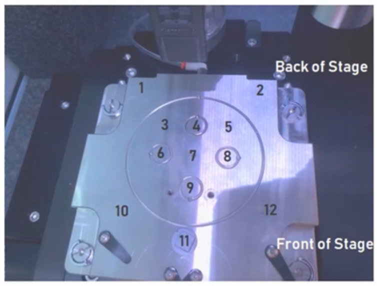
- Sample Prep
- Surface Roughness
- Film Thickness / Substrate Effect
- Indentation Size Effect / Indentation Depth
- Initial Penetration Depth
- Pile-up
- Tip Rounding
- Creep Effects

Sample Preparation

Loose objects/samples are generally not good enough to put in the nanoindenter

Recommended:

- Super glue the sample to a magnetic specimen disk (shown right)
- Super glue to a glass slide
- Smooth samples (Wafers, glass slide with coating) can be adhered via air suction without gluing



Nanoindentation

Roughness Effect

roughness <5% of depth

MATERIALS, DESIGN AND MANUFACTURING FACILITY(CWB)

Surface Roughness

Roughness can affect the measured values of modulus and hardness:

Potential solutions:

- Indent to a depth at which roughness has little effect (roughness <5% of depth)
- Correct for roughness using analytical contact model

$$\frac{H}{H_{flat}} = 1 - \frac{K}{\left(\frac{h}{d_r}\right)^n}$$

d_r = roughness parameter
K, n = constants

M. S. Bobji and S. K. Biswas. J. Mater. Res. 13[11] 3227 (1998).

MATERIALS, DESIGN AND MANUFACTURING FACILITY(CWB)

Nanoindentation

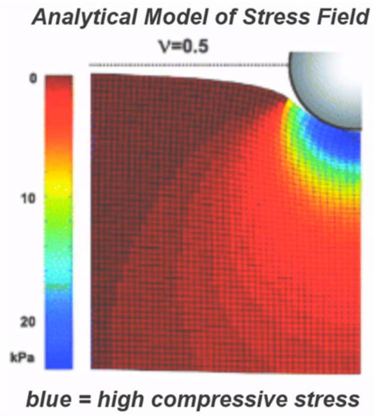
Rule of thumb

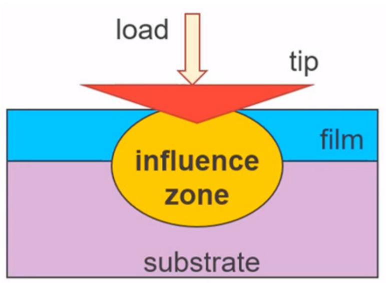
Indent to depths <10% of film thickness

Film Thickness

MATERIALS, DESIGN AND MANUFACTURING FACILITY(CWB)

Stress fields much larger than indentation depth. Substrate effects can begin to dominate measured properties for thin films.



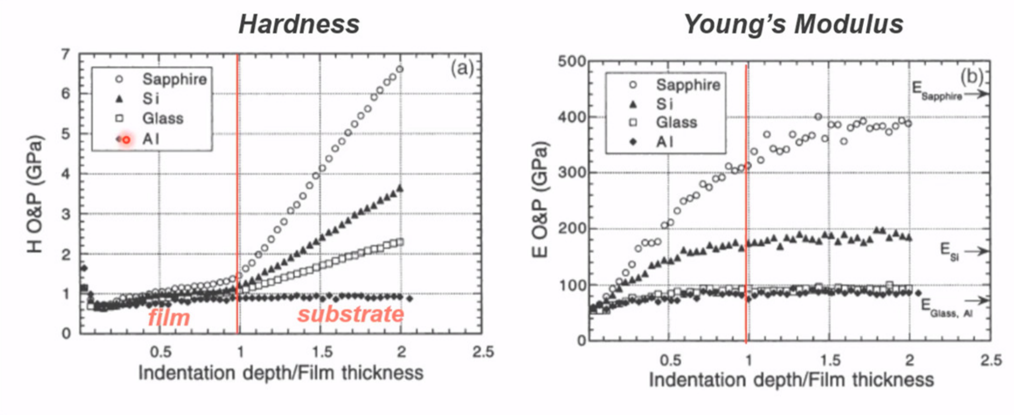


- Indent to depths <10% of film thickness (or smaller for soft materials).
- Use analytical models separate substrate effects from measured values.

Film Thickness & Substrate Effect

MATERIALS, DESIGN AND MANUFACTURING FACILITY(CWB)

0.5um aluminum film deposited on different substrates:



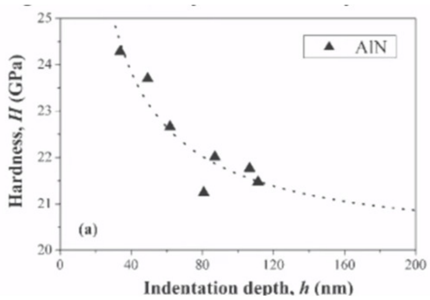
R. Saha and W. Nix. Acta Mater. 50 23 (2002).

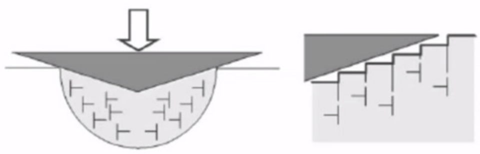
Nanoindentation

Size is matter

Indentation Size Effect

For very shallow indents, hardness may increase due to geometrically necessary dislocations loops.





A certain concentration of dislocations is required to maintain the shape of the surface below the indenter.

- For deeper and deeper indents less and less dislocations are required until that concentration reaches the bulk sample concentration
- Dislocation concentration ↑, yield strength↑,hardness ↑
- Always good practice to indent at multiple depths to confirm consistency

Gao and Huang. Scripta Mater. 48 113 (2003).
Lu, et al. Key Engineering Materials 312 363 (2006).

MATERIALS, DESIGN AND MANUFACTURING FACILITY(CWB)

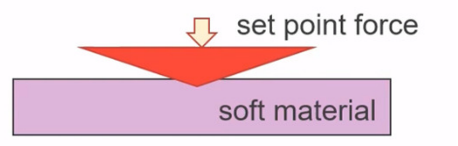
Nanoindentation

Problem of testing very Soft Materials

MATERIALS, DESIGN AND MANUFACTURING FACILITY(CWB)

Initial Penetration Depth / Set Point

In order to locate and hold the tip in contact with a sample surface, the indenter applies a minimum force (the set point force) before initiating indentation.
For soft materials, even a small force can lead to significant sink-in and/or deformation



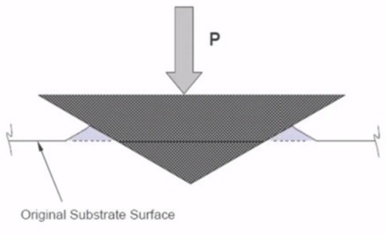
Potential solutions:
- Decrease set point (typically 2uN, decrease to ~1 uN), but be careful not to lose contact with surface
- Lift up tip immediately prior to indenting (function available on Hysitron)

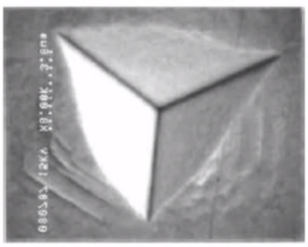
Nanoindentation

Testing Error

Pile-up

Pile-up = deformation around tip(as opposed to exclusively below tip)



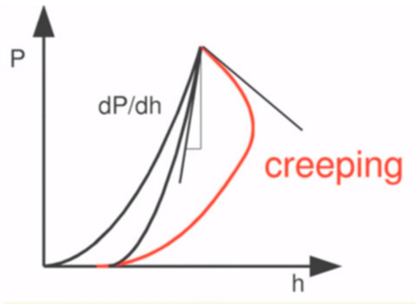


- Oliver-Pharr interpretation does not account for pile-up
- Interpretation of nanoindentation data frequently gives values somewhat higher than those measured by other methods.

MATERIALS, DESIGN AND MANUFACTURING FACILITY(CWB)

Creep & Viscoelastic Effects

Time-dependent deformation can lead to:
- Rate-dependent measured E and H
- Convolution of unloading with continued deformation



Potential solutions:
- Set unload rate such that it is ideally >10x creep/relaxation rate
- Increase dwell time to wait for creep rate to stabilize or relaxation rate to decrese

MATERIALS, DESIGN AND MANUFACTURING FACILITY(CWB)

How to access ?

You can reserve a timeslot of our machines via following link :
https://emz075.ust.hk/MachineBooking/CAD/CAD_MachineBookingForm.html
https://emz075.ust.hk/MachineBooking/MAT/MAT_MachineBookingForm.html

MATERIALS, DESIGN AND MANUFACTURING FACILITY(CWB)

MDMF – MAT Unit

Non Detructive Inspection Machine	
3D Surface Metrology : BrukerNPFLEX-1000	Delivers the accurate and repeatable surface texture and roughness metrology
Contact Angle Meter : Biolin Theta	Contact angle, θ, is a quantitative measure of wetting of a solid by a liquid.
Particle Size Analyzer	Measure the size of particle distribution
Scanning Acoustic Microscope (C-SAM)	Using ultrasonic to detect Delamination and crack, Failure analysis, Transducer 110 MHz, 50 MHz scanning area (350 x 350 mm)
Surface Area and Pore Size Distribution Analyzer	Using gas adsorption method to measure Surface area
TecScan 7 Axis Immersion Scanner	Crack, Delamination Detection, Failure analysis, 1Mhz Transducer (Tank dimension: 2.0 X 1.2 X 1.2 m deep)
X-Ray with CT Imaging System	CT, Failure analysis (Recommend 5 x 5 mm)
ZetaPlus (Zeta Potential Nano-particle Analyzer)	Measure the magnitude of the electrostatic repulsion/attraction between particles and is one of the fundamental parameters known to affect stability of dispersed systems. Its measurement brings detailed insight into the causes of dispersion, aggregation or flocculation, and can be applied to improve the formulation of dispersions, emulsions and suspensions.