NANO-INDENTATION
Basics, theory, principles and techniques

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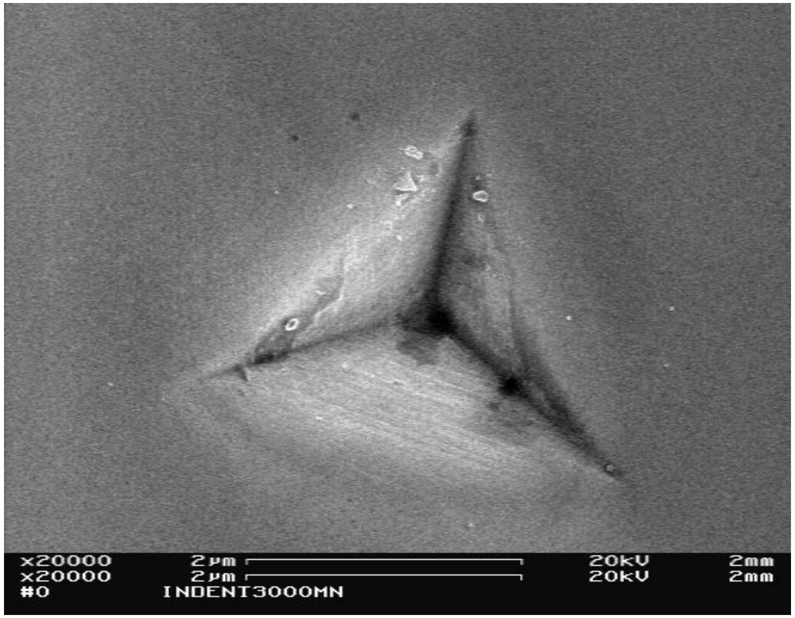
OUTLINE

- Why Nanoindentatioin?
- Principle of Nanoindentation
- Applications
- Instrumentation
- Theoretical analysis (indentation with conical and non-conical indenter, Oliver-Pharr method)
- Practical indentation

Hardness test on the nanoscale

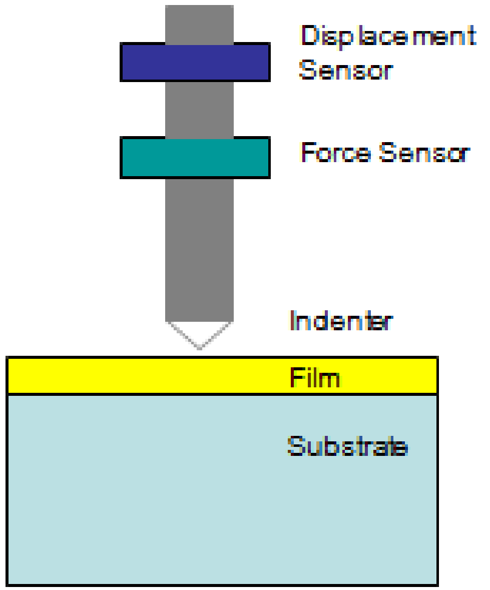


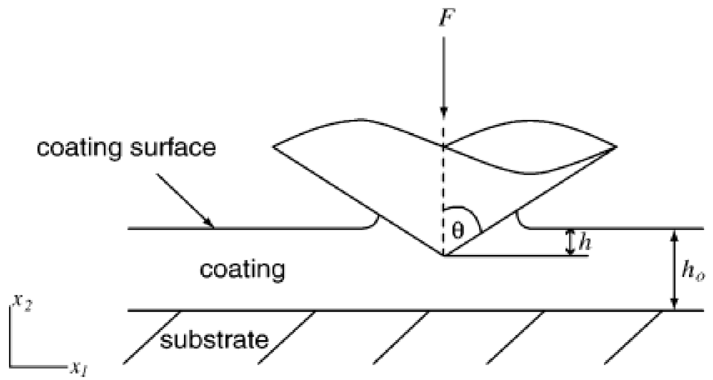
SEM micrograph of the indent made in nc-Cu by Berkovich indenter under the load of 3mN.

The idea of nanoindentation arose from the realization that an indentation test is an excellent way to measure very small volumes of materials. The only problem is determining the indentation area. For the indents below 1 μm in size the resolution of optical microscope is insufficient.
To solve this problem **depth sensing indentation method** was developed. In this method the load and displacement of the indenter are recorded during indentation. Mechanical properties of the indented volume can be determined without seeing the indent.

Why nanoindentation?

Definition: Mechanical probing of a material surface to nm-scale depths, while simultaneously monitoring LOAD and DEPTH.

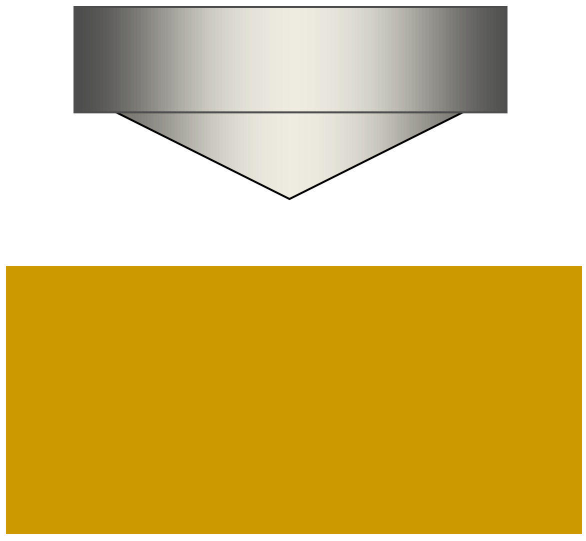


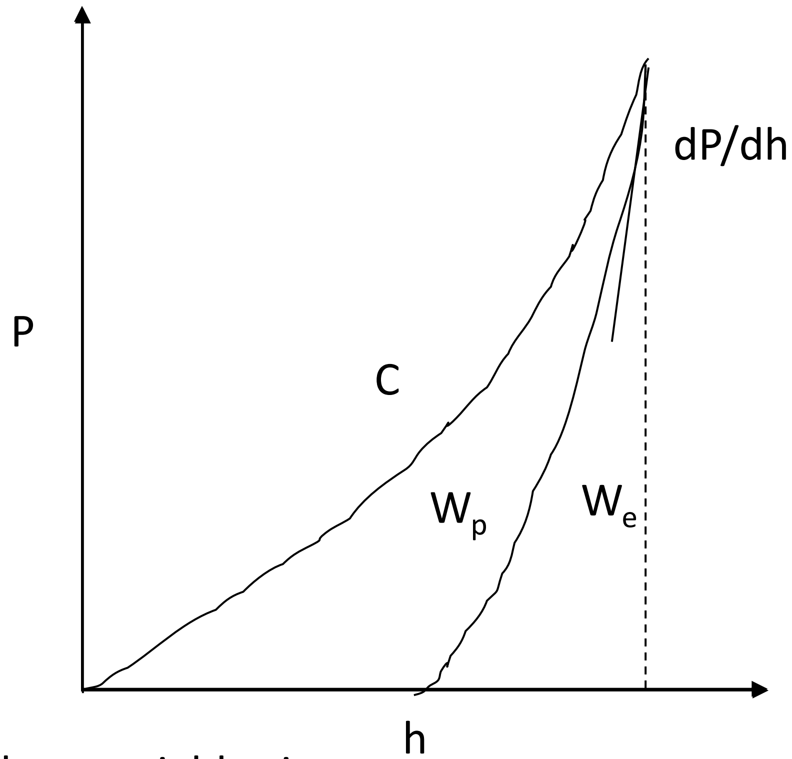


(a)

1. Materials engineering: Optimizing material composition, structure, processing for particular applications (bulk and thin films)
2. Mechanics of small volumes: Understanding whether thin films, lines, dots have different mechanical properties than bulk counterparts
3. Material physics: Measuring deformation processes such as dislocation nucleation, crack growth, etc.

What is nanoindentation?





Show variables in response:

C = loading curvature

dP/dh = unloading stiffness

P_{max}, h_{max}

W_p = plastic work of indentation

W_t = total work of indentation

Hysitron's Triboindenter

Current Nanoindentation application areas include…

- Automotive
- Bearings
- Biomedical Devices
- Ceramics
- Composites
- Contact Lenses
- Cutting Tools
- Hard Coatings
- Laminates
- Magnetic Disks
- Microelectronics
- Optical Coatings
- Optical Disks
- Packaging Materials
- Paints
- Paper Coatings
- Pharmaceuticals
- Photographic Film
- Polymers
- Powders
- Printing Plates
- Semiconductors
- Thin Film Adhesion
- Turbine Blades

…future application areas will be in?

Where does depth-sensing indentation fit in?

SPM

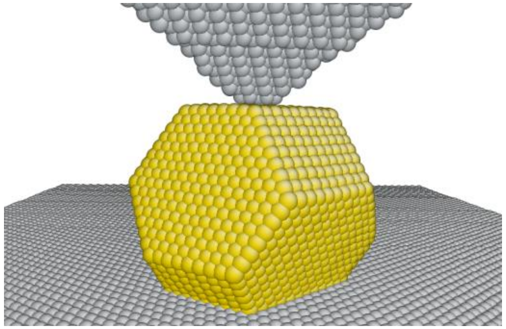
- *Surface*
- Friction
- Adhesion (local)
- (Wear)
- Topography
- Role of specific chemical interactions?

Nanoindentation

- *Surface/near-surface*
- Mechanical properties of thin films and coatings
- Hardness
- Modulus
- Creep
- Depth-profiling
- Wear resistance
- Interfacial adhesion
- Fracture toughness

Macro-hardness

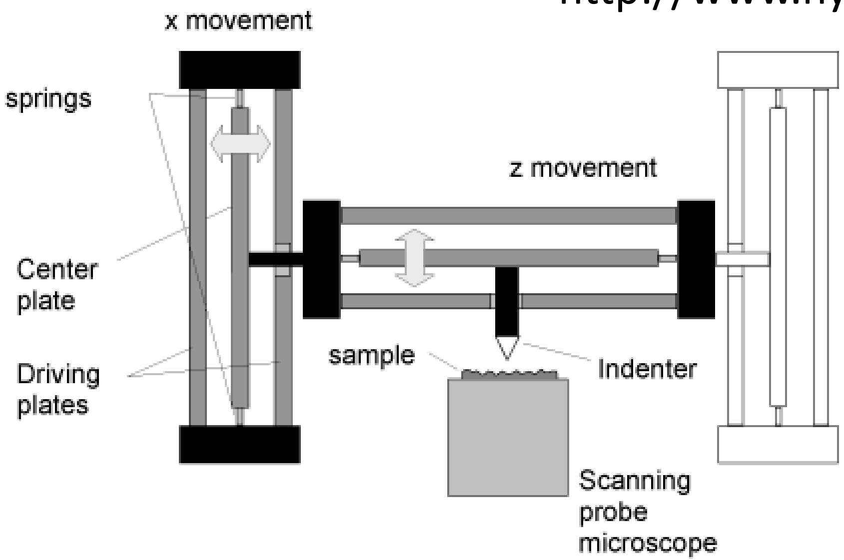
- *Bulk*
- Hardness of thick coatings and bulk materials
- Fracture toughness of bulk materials

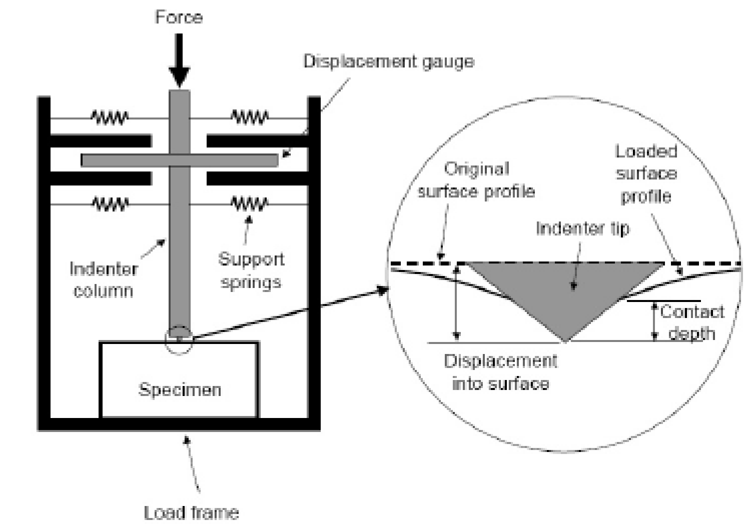


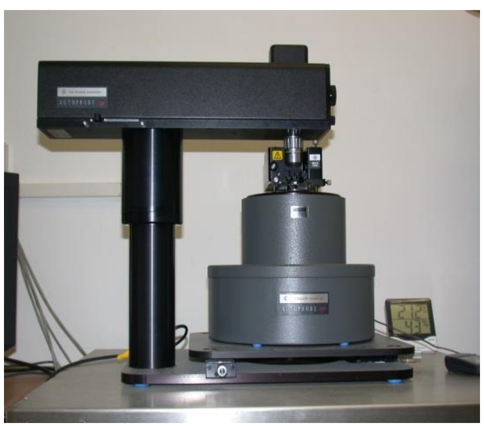
MD simulations of nanoindentation of gold nanoparticles (D. Mordehai)

Hysitron (US)

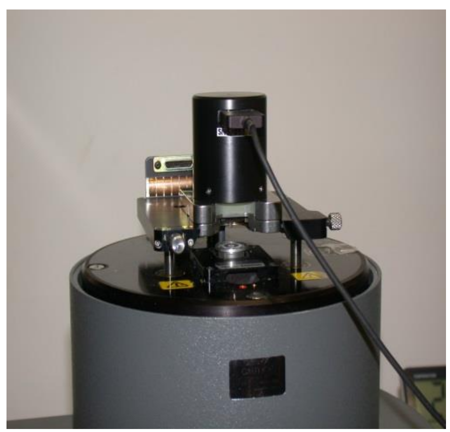
http://www.hysitron.com







SPM



SPM + Nanoindenter

The advantage of Hysitron is a combination of nanoindenting head with the SPM positioning stage. This allows a precise selection of the object to be tested.

The SPM is operating in the STM mode and the Control Unit is “cheated” by providing the voltage from capacitance sensor instead of tunnel current.

What is Measured?

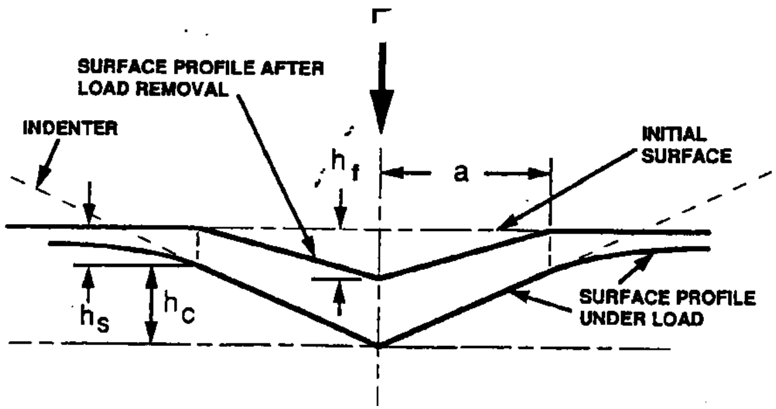
Techniques

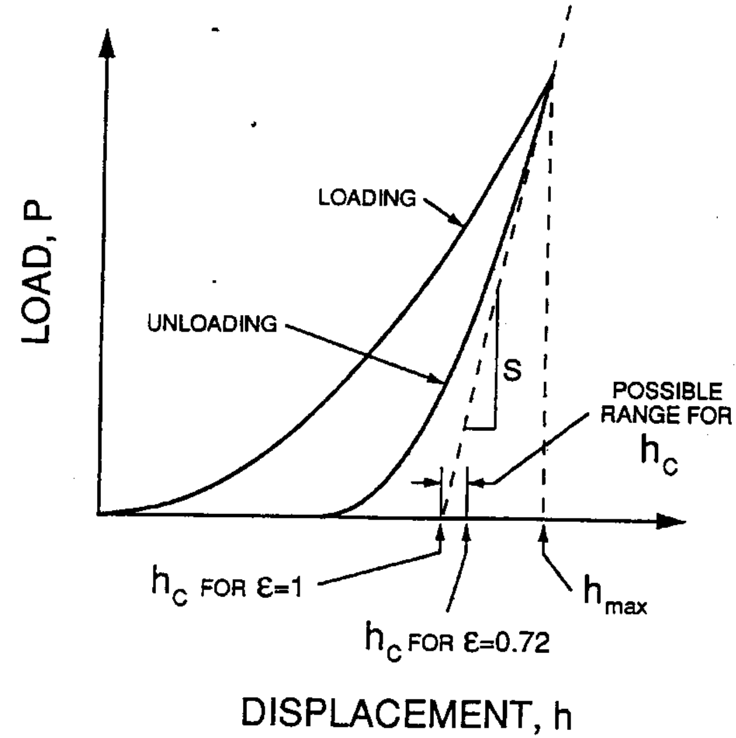
- Nanoindentation
- Microindentation
- Load-partial-unload
- High temperature testing
- Continuous compliance

Properties

- Hardness
- Elastic modulus
- Fracture toughness
- Adhesion
- Creep
- Elastic and plastic work
- Elastic recovery parameter
- Stress-strain

What is measured?





For calculation of nanohardness the knowledge of h_c (contact depth at maximal load) is necessary.

According to Oliver and Pharr (1992)

$$h_c = h_{max} - \varepsilon \frac{P_{max}}{S}$$

$$H = \frac{P_{max}}{A_c(h_c)}$$

where ε=0.72 for conical indenter and ε=0.75 for paraboloid of revolution. S is a contact stiffness.

$$Ac = 3\sqrt{3} h_p^{\ 2} \tan^2 65.3 = 24.5 h_p^{\ 2}$$ for Berkovich indenter

Young's Modulus, E

- E is calculated based on the Sneddon equation :

$$E_r = \frac{\sqrt{\pi}}{2\beta} \frac{dp}{dh} \frac{1}{\sqrt{A_c}}$$

Later, Pharr, Oliver and Brotzen where able to show that the equation is a robust equation which applies to tips with a wide range of shapes.

- where E_r is the composite (Reduced)Young's modulus,
- $\frac{dp}{dh}$ is the contact stiffness and
- β is a correction coefficient near to 1
- β=1.034 for a Berkovich tip
- *E* can be calculated as:

$$\frac{1}{E_r} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i}$$

Er is the reduced modulus of indentation contact,
E_i and v_i are the elastic modulus and poisson's ratio of the indenter respectively
E and v are the elastic modulus and Poisson's ratio of the test sample

Determination of Plasticity index (ψ)

- It is usually used to characterize
  - ✓ the elastic-plastic response of a material under external stresses and strains.
- The plasticity index can be calculated as follows:

$$\psi = \frac{A_1 - A_2}{A_1}$$

where A_1 is the area under the loading curve and A_2 is the area under the unloading curve

➢ the plasticity index is in the range of 0 < ψ< 1.

- Ψ= 0 represent the fully-elastic and
- Ψ= 1 denotes fully-plastic behavior of materials

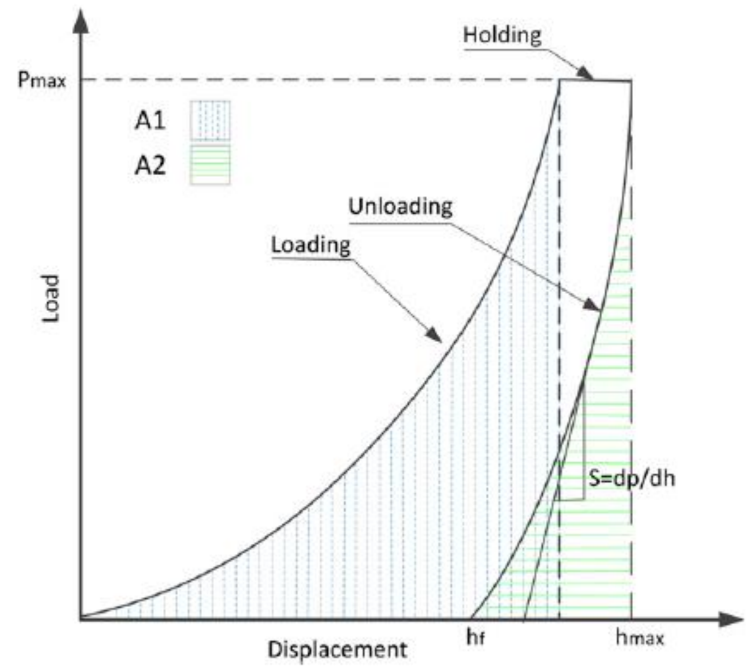
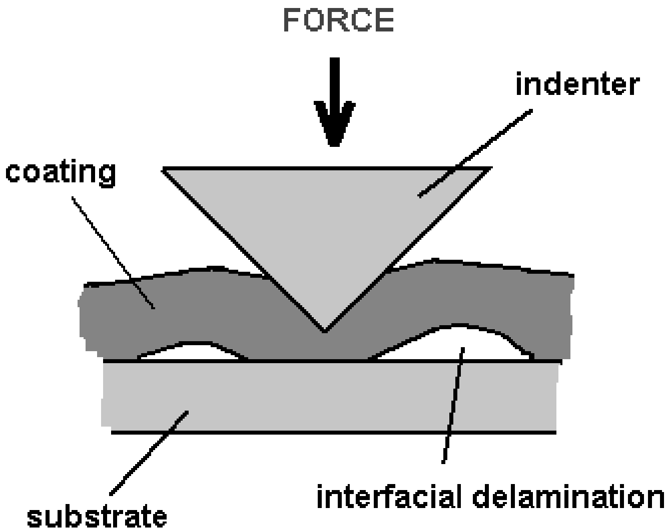
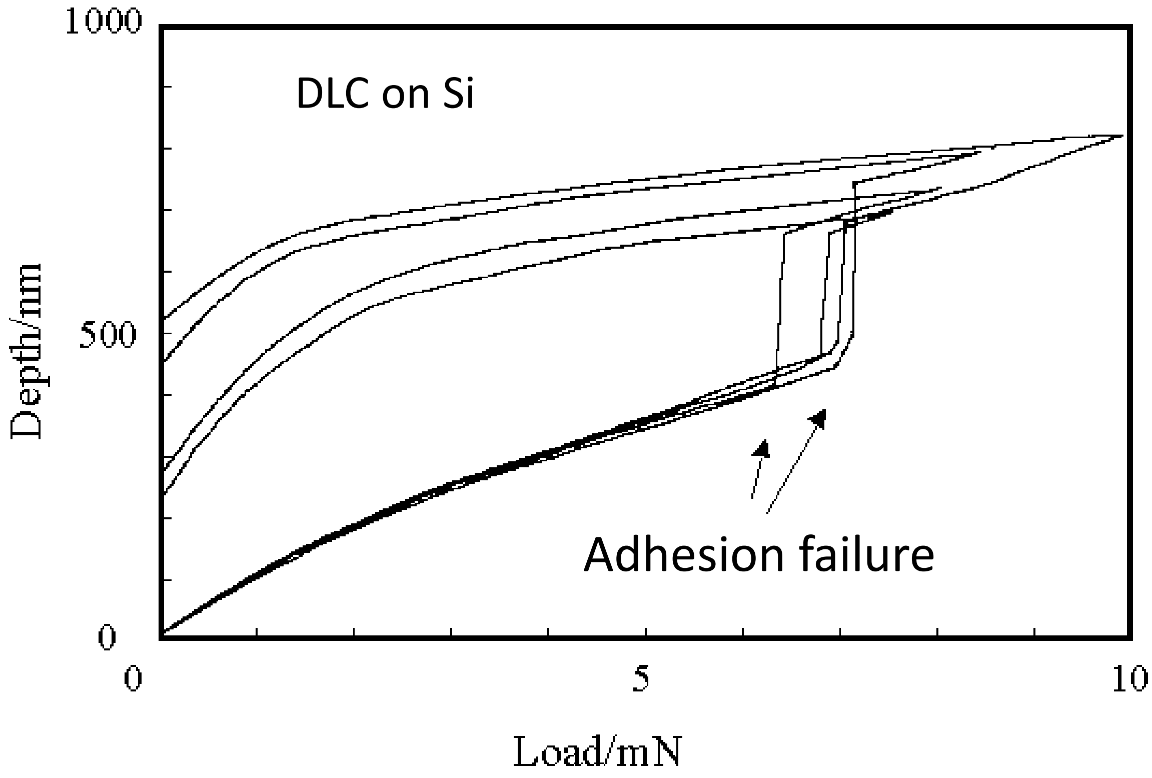


Fig. 1. The schematic load-displacement curve in nanoindentation test.

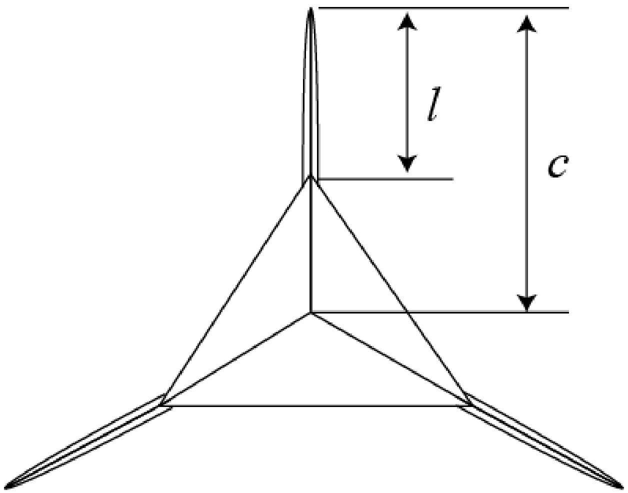
Nanoindentation-induced adhesion failure

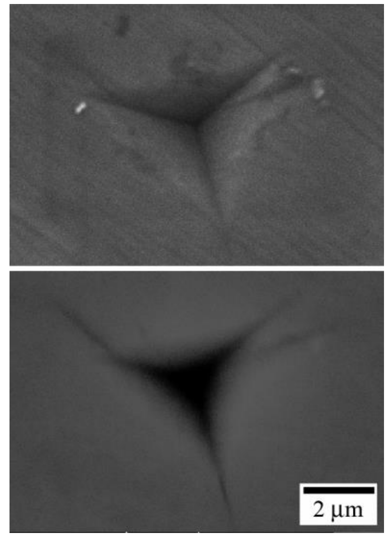


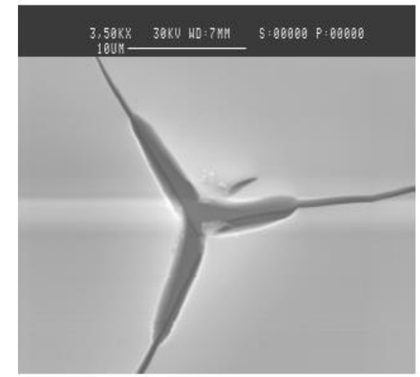


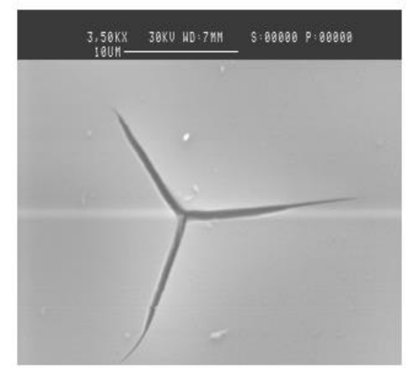
- indentation + residual stress = coating failure

Fracture toughness measurement









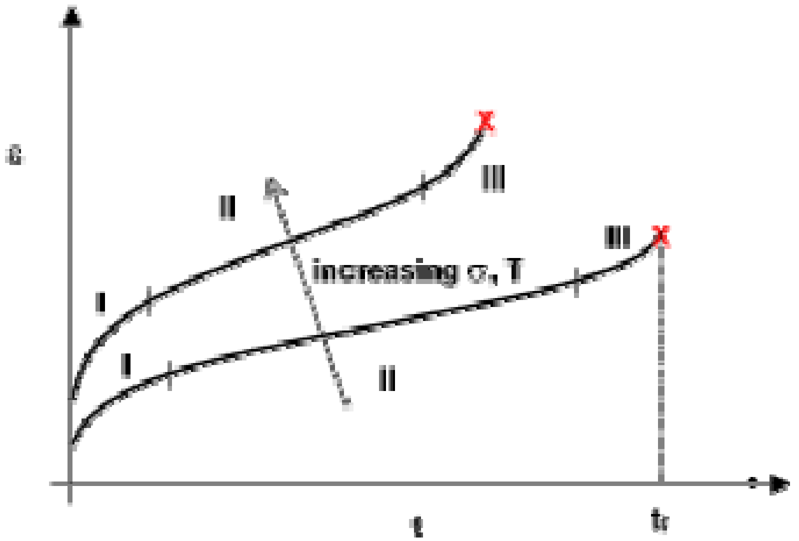
Combining of Laugier proposed toughness model and Ouchterlony's radial cracking modification factors, fracture toughness can be determined.

Fracture toughness expression

$K_c = 1.073\, x_v\, (a/l)^{1/2}\, (E/H)^{2/3}\, P / c^{3/2}$

$$K_c = \alpha \left(\frac{E}{H} \right)^{1/2} \left(\frac{P_{\max}}{c^{3/2}} \right)$$

Creep measurement

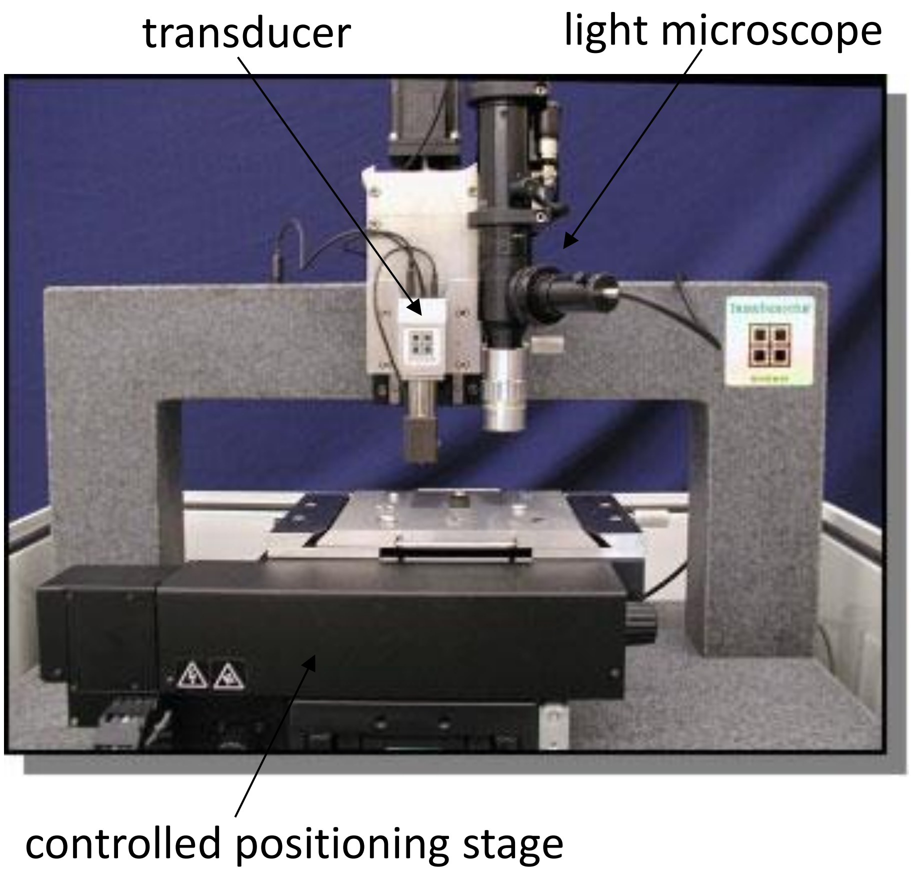


- **Plastic deformation in all materials is time and temperature dependent**
- **Important parameter to determine is the strain rate sensitivity**
- **The average strain rate can be given by**

$$\varepsilon_{ind} = \frac{1}{h_c}\frac{dh_c}{dt}$$

- **It can be done by experiments at different loading rate or by studying the holding segment of a nanoindentation.**

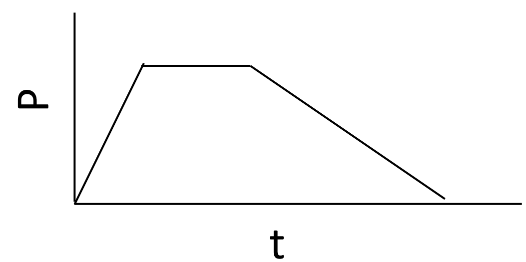
How does nanoindentation work?



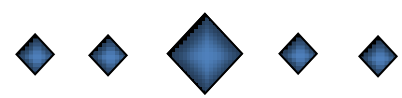
Procedure:

1. Prepare sample surface:
   Flat, Parallel faces, Smooth
2. Find region of interest on surface

3. Program load profile



4. Program spatial matrix



5. Execute experiment
6. Analyze data to obtain properties

Indenter Tips

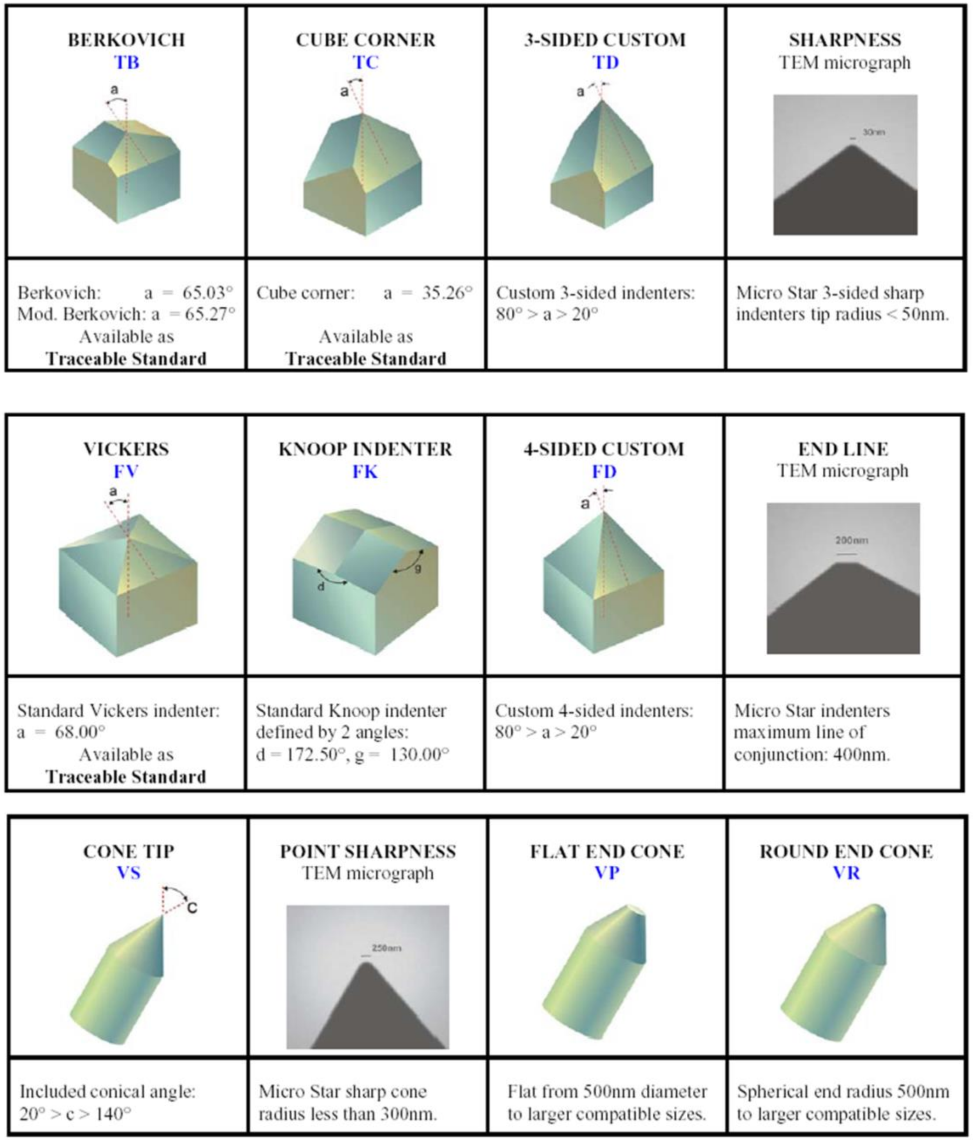
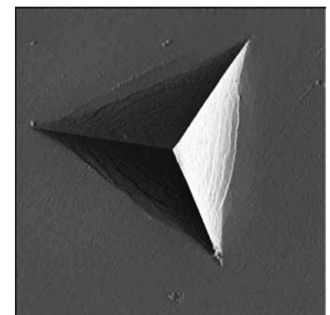


Figure 6 CONE INDENTERS

Sharp 3-sided indenters

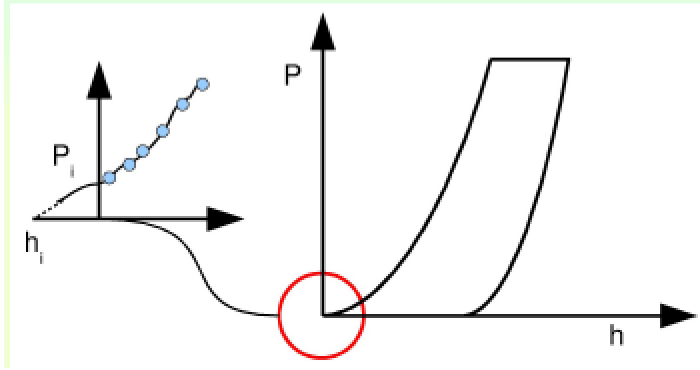


4-sided indenters

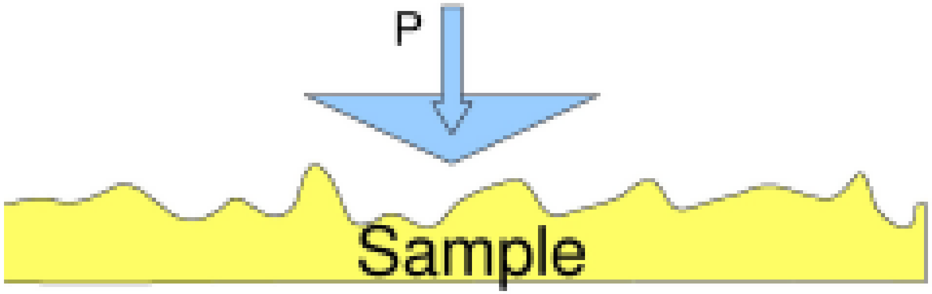
Indenter	Contact Area	Eq. cone angle
Berkovich	$3\sqrt{3}h^2 tan^2 65.27 = 24.5h^2$	70.3^o
Vickers	$4h^2 tan^2 68 = 24.5h^2$	70.3^o
Knoop	$2h^2 tan 86.25 tan 65 = 65.43h^2$	77.64^o
Cube corner	$3\sqrt{3}h^2 tan^2 35.26 = 2.6h^2$	42.278^o

Issues

- Initial penetration is an unavoidable part of the depth that is lost before the contact is detected. It can be neglected if the contact force is set small or estimated using extrapolation to the zero force level.

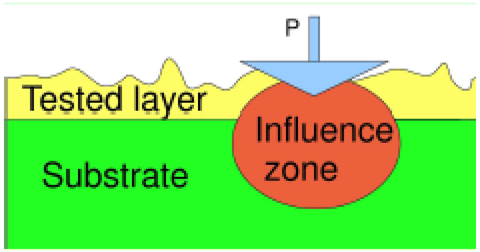


- Surface rougness. If the surface rougness is too large relative to the penetration depth than the contact equations become invalid.



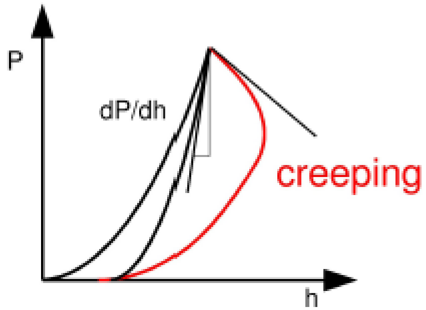
Other Issues

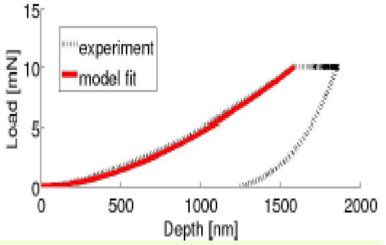
- The **layer thickness** must be larger (approx. 10) than the indent's depth
- Otherwise, the substrate influences the measurement and evaluation of material constants.



- **Material creep** can influence the evaluation of even the elastic properties.
- Using long dwell periods can minimize these efects.

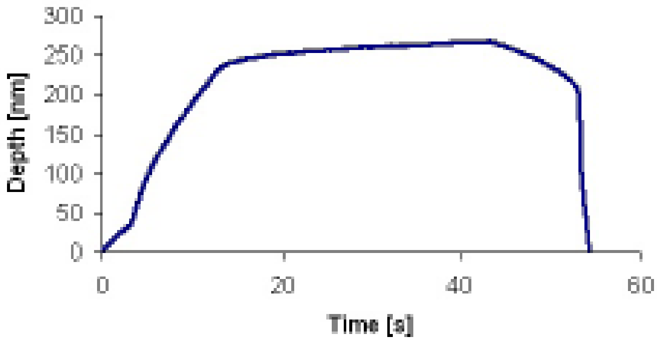
- If the dwell time is absent, the sample will continue to deform viscoplastically, while the load is being removed
- This will significantly distort the shape of the unloading curve leading to inaccurate modulus measurement

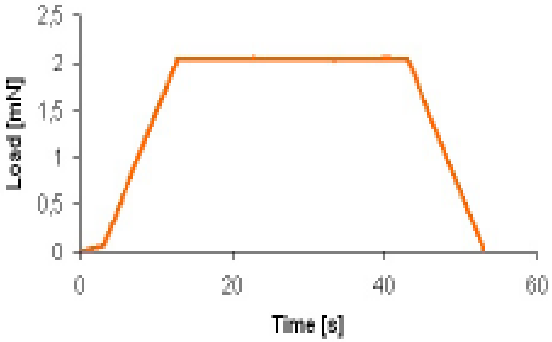


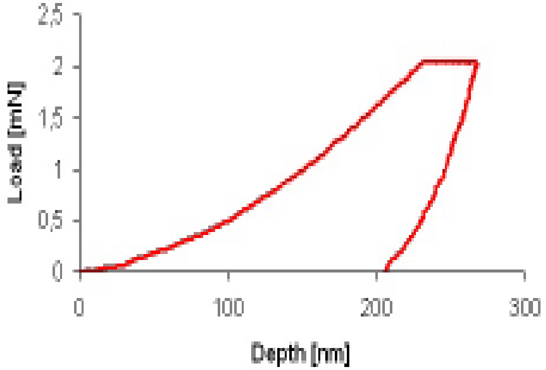


TIME-DEPENDENT MATERIALS

- Many materials (plastics, metals, wood, concrete, etc.) exhibit time-dependent behavior under steady load conditions called creep.
- During indentation test, creep acts during the whole loading history.
- It manifests itself mainly during the dwell phase and may be observed also at the unloading portion of the P-h curve.
- Standard elastic parameters such as hardness and modulus are affected by creep because the slope of the P-h curve computed from the upper part of the unloading curve contains not only elastic but also creep deformations.







Type of Sample

- Materials such as metals, semiconductors, ceramics, and polymers, as well as biomedical and biological samples.
- Samples may be in various formats, such as bulk materials, thin films, and composites.

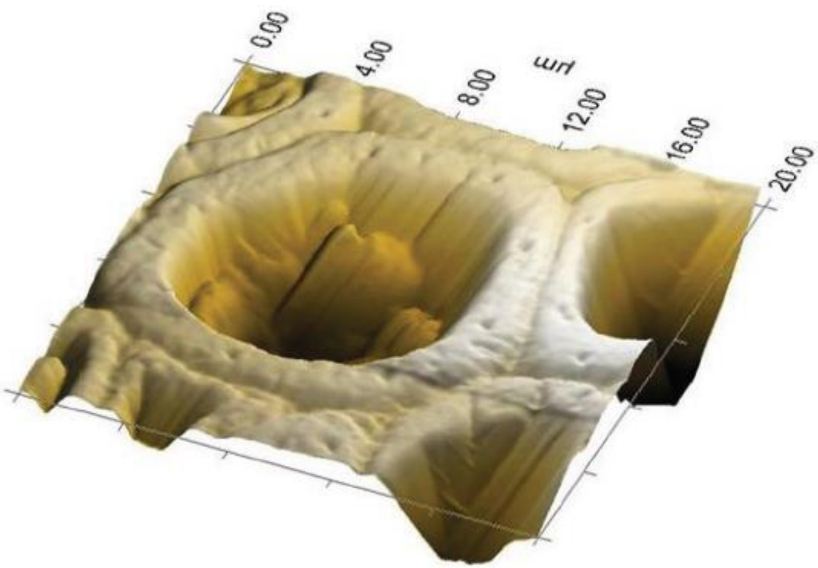
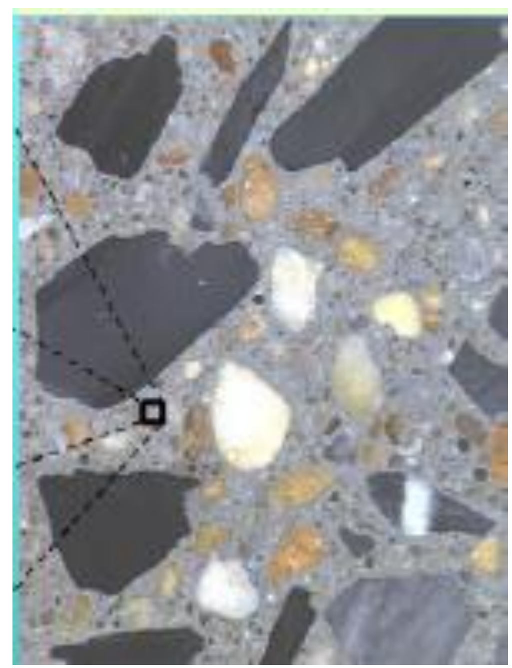


Figure 1: 20 µm scan of wood cells after nanoindentation tests around cell wall.



Sample Preparation

- **Sample surface must be:**

  - Flat (with plan-parallel top and bottom surfaces).
  - Clean (free of dust and adhesive particles that can contaminate (i) the measurement and (ii) the tip).
  - Smooth (surface roughness must be checked and kept much less than indentation depth).

- **Ways of preparation:**

  - Parallel cutting of both top and bottom surfaces (e.g. by precize diamond saw).

Sample Preparation

- Cleaning with pressurized air or washing in alcohol and in an ultrasonic bath.
- Polishing (using emery papers, diamond pastes and solutions, polishing on cloth)

- **Ways of checking:**
  - Optical or electron microscopy
  - AFM, 3-D image, exact roughness computation over the scanned area
  - Typical indentation depth 100 - 500nm requires roughness 10 - 50nm.

In-Situ Testing Techniques Used:

***In-situ* SPM imaging**: for qualitative surface analysis before and after testing and quantifying surface roughness

- **Nanoindentation:** for quantitative material properties including hardness, modulus, and stiffness; depth profiling
- **Nanoscratch:** ramping force nanoscratch for quantification of film delamination/breakthrough
- **Nanoscratch:** reciprocating nanoscratch for tribology, friction, and wear failure
- **ScanningWear:** for wear resistance and wear volume quantification

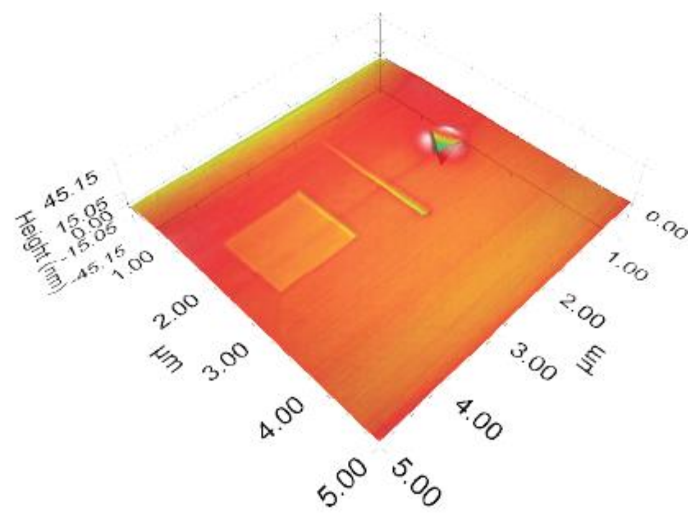
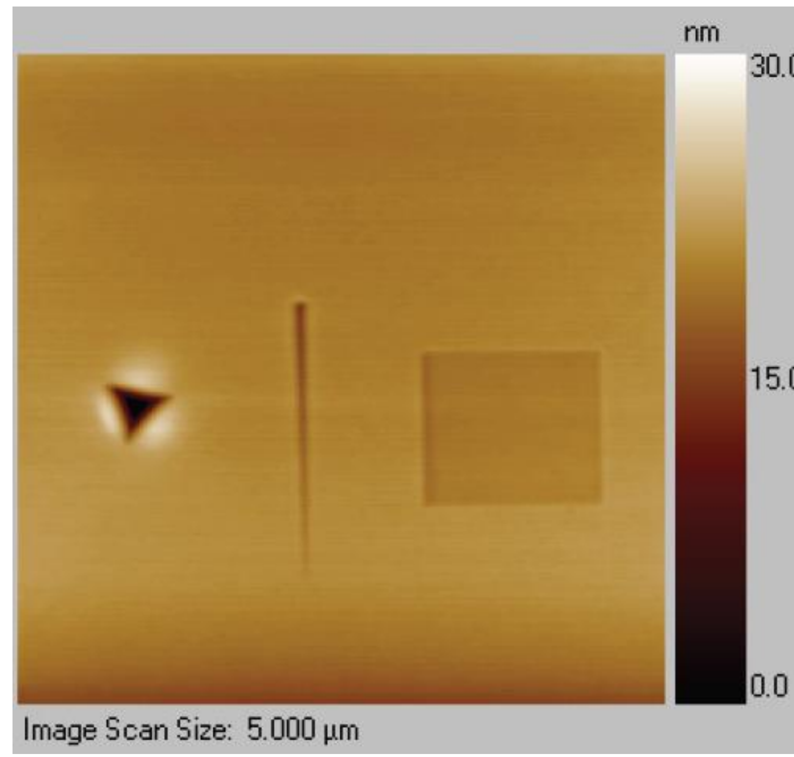


Figure 4. 3D topographical image of indent, scratch, and wear test on DLC film created using TriboView

NanoScratch Testing:

- Nanoscratch Resistance
- Interfacial Adhesion of Thin Films
- Delamination
- Lateral Force Measurement
- Mar Resistance



Post-test *in-situ* SPM image of an indent, scratch, and ScanningWear test within a 5 µm test area.

Principle of Scratching

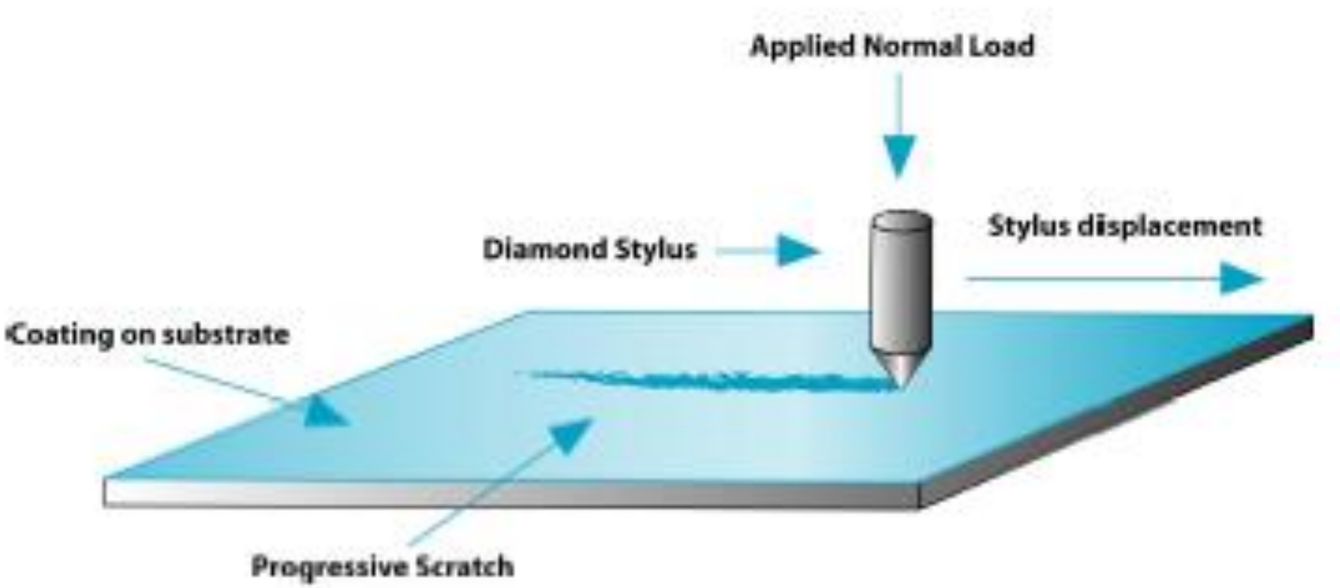
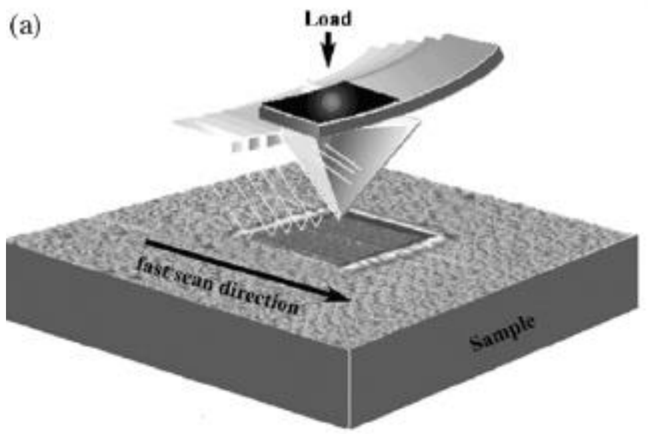


Figure 1 : Principle of scratch testing

Nano Wear



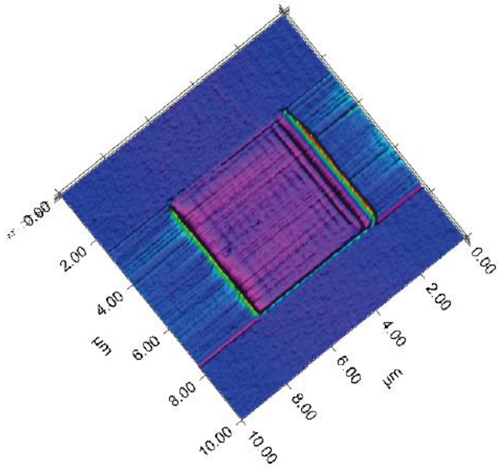
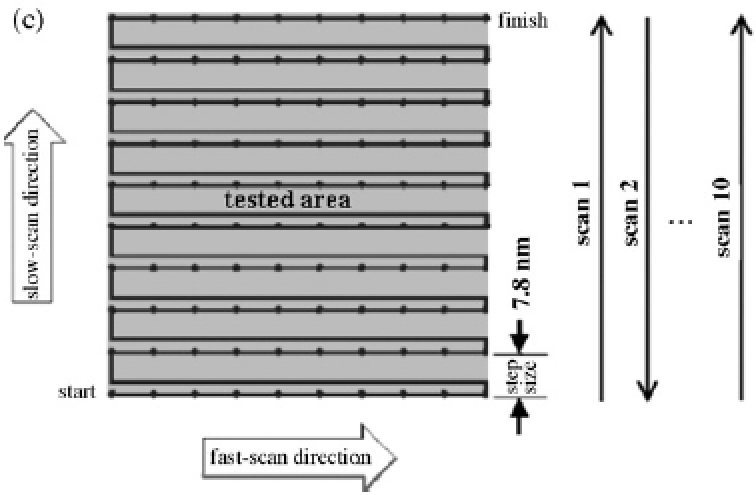
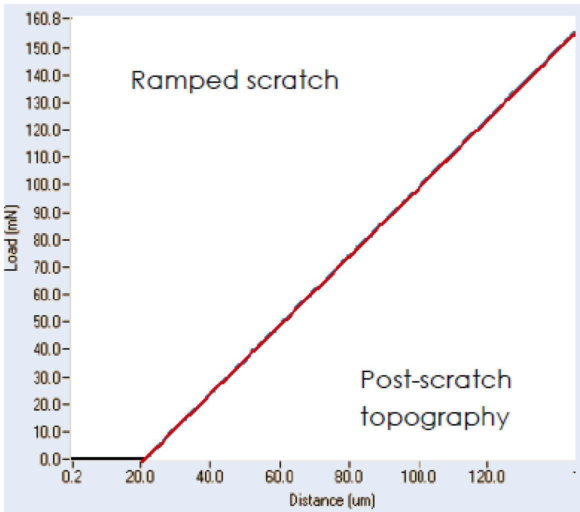


Figure 2: *In-situ* SPM image of wear track made on DLC film to determine wear resistance of coating.





ScanningWear Features

- Programmable forces for precise load control within a wear box
- Adjustable wear track size from <1 µm to 100 µm
- Single or multiple pass wear testing capabilities
- Wear volume easily measured using *in-situ* SPM imaging
- Roughness measurement utility as standard
- Sensitive enough to measure <1 nm of material removal
- Ability to test in fluid medium such as lubricants and oils.

Ultra-Thin Film Applications:

Optimizing thin film properties requires a well-understood relationship between processing parameters, modeling, and model confirmation through novel testing techniques.

- Microelectronics
- Optics and optical coatings
- MEMS devices
- Hard and corrosion resistant coatings
- Photovoltaics
- Shape memory alloys

Effect of Surface Roughness

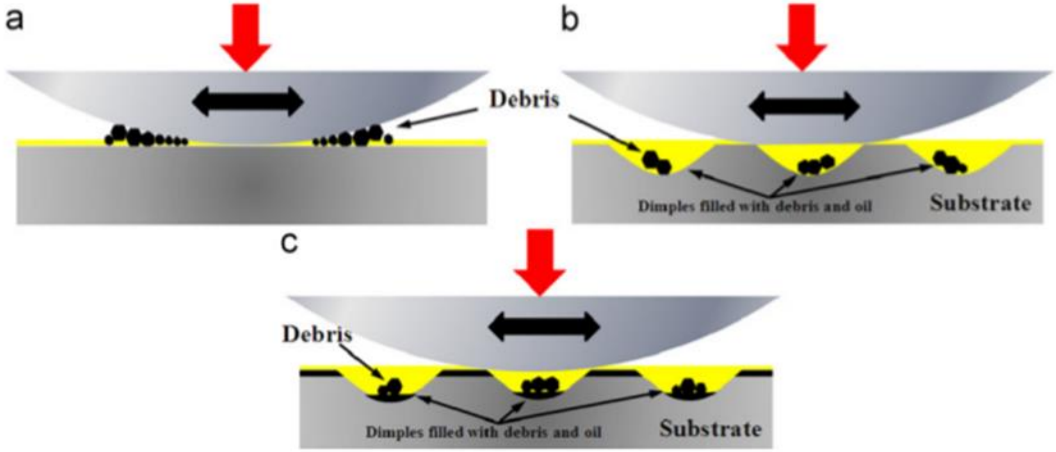


Fig. 12. Cross-sectional schematic view of the wear mechanism model for the polished (a), dimpled (b) and over-coated dimpled specimens.

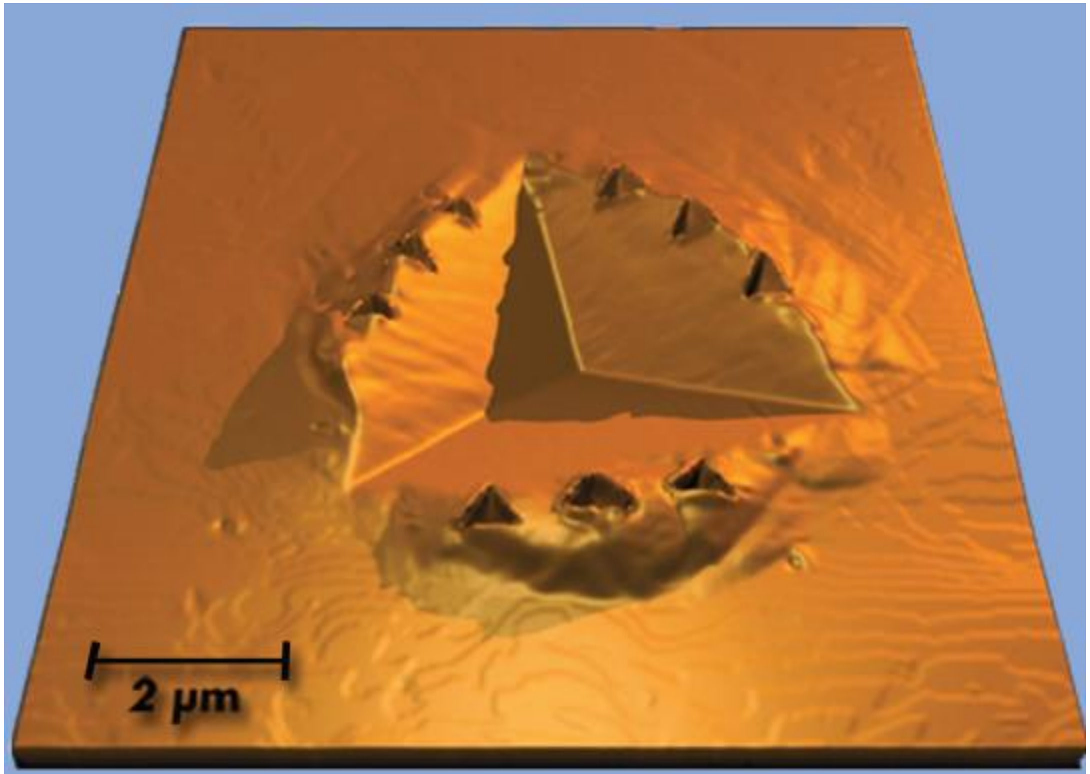


Image showing a residual high-load indent impression with low-load indentation tests placed along the pile-up.

In-situ SPM Imaging

- The *in-situ* images are obtained by raster scanning the indenter probe over the sample surface to allow for pre- and post-test observation of the material surface
- The **TI 950** offers quantitative topographical imaging with an extremely low imaging contact force (≤70 nN).

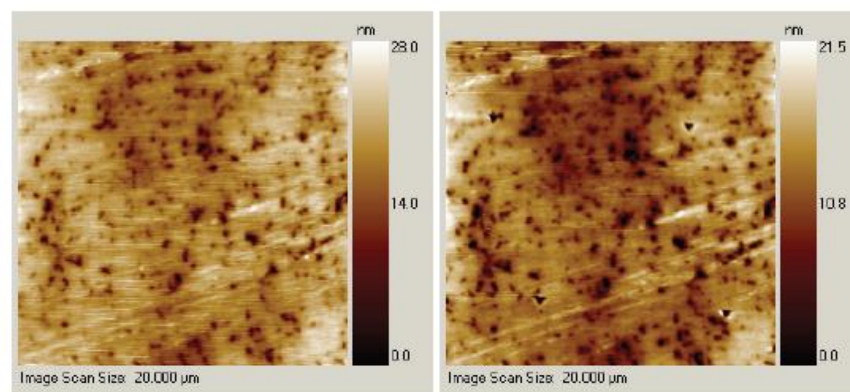


Figure 1: SPM image of polished nitride hard coating.
(left: before test, right: after test, image scan size: 20μm)

In-situ SPM imaging

- *In-situ* SPM imaging allows observation and quantification of material deformation, such as:
    - ❖ pile-up,
    - ❖ wear volume,
    - ❖ crack length and
    - ❖ scratch morphology, incurred during testing

Hardness Mapping: Example

- A 10x10 automated grid of indents was performed on a defect-free region of the sample identified by SPM imaging and indents were spaced 1 µm apart.

- Indentation results were analyzed and mapped using the Hysitron **TriboAnalysis**™ software package.

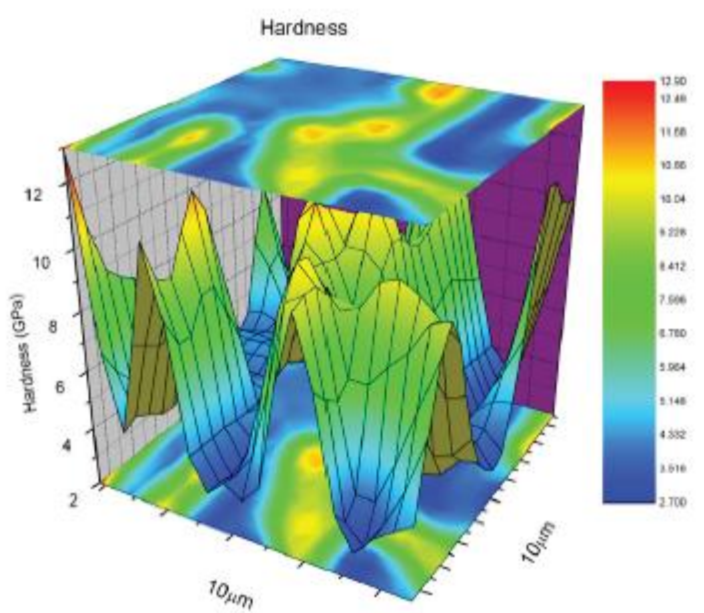


Figure 1: Hardness map showing distinct ferrite and martensite phases.

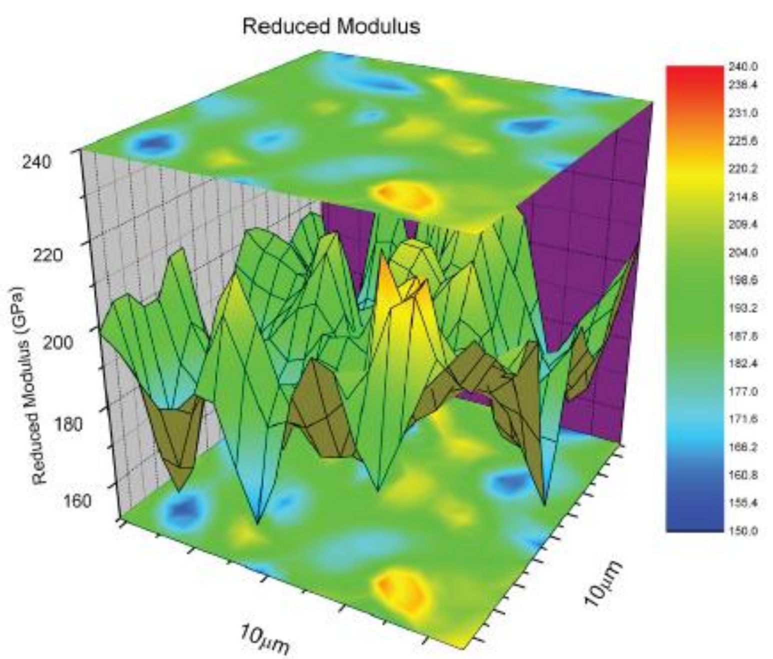


Figure 2: Reduced modulus map of a martensitic/ferritic dual-phase steel.