COVALENT METROLOGY

Welcome

NANOINDENTATION WITH THE UNHT^3 INDENTATION TESTER: NANO-MECHANICAL ANALYSIS WITH A BIG IMPACT

SPEAKERS:

Patrick O'Hara
Technical Sales Representative,
Anton Paar

Austin Barnes, PhD
Member of Technical Staff,
Covalent Metrology

June 17, 2021 | 11AM PT

Anton Paar

COVALENT
METROLOGY

COVALENT
ACADEMY

Advancements in
Instrumentation Series

Episode 23

COVALENT METROLOGY

Silicon Valley-based analytical labs and platform delivering quality data and expert analysis for advanced materials and device innovation

Comprehensive Solutions Stack

- *40+ cutting-edge instruments, offering 100+ Techniques*
- *Analytical Services*
- *Advanced Modeling*
- *Method Development*
- *Temp. Staffing Solutions*

Affordable and Fast

- *Fast Turnaround Times, No Expedite Fees*
- *Volume Savings*
- *Instant Access to Data and Reports in Secure Portal*

Flexible Business Model

- *Custom Consulting Solutions and Certified Onsite Support*
- *Training and Certification on Instrumentation*
- *Co-op and Tool-Share Opportunities*
- *Laboratory Audits*

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PVA TePla
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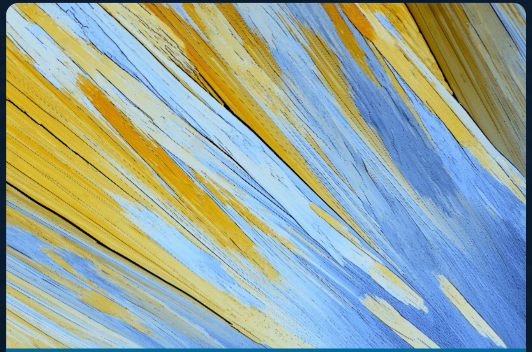
ASYLUM RESEARCH an Oxford Instruments company

Rich Network of Partnerships

- *Partner to World's Leading Instrument Manufacturers and Labs*
- *Expanding Instrumentation, Lab Connections and Learning*



Who We Are, Who We Serve

- *500 Clients + 40-60 New Clients/ Quarter*
- *40 People, 13 PhDs*
- *Cutting-edge Analytical Capabilities*
- *Lab Locations: Sunnyvale, CA*

Covalent Analytical Service Categories

COVALENT METROLOGY

PCBA, Semiconductor, and Electronic Device Metrology & Failure Analysis

- DPA / Mechanical Cross-section
- Dye & Pry Test
- EBIC / OBIC failure analysis
- Hot Spot Detection
- IR Imaging / Emission Microscopy
- NIR Imaging
- Root-Cause Failure Analysis

Electron Microscopy and Scanning Probe Microscopy

- AFM & Advanced AFM Modes (EFM, KPFM, MFM, PFM)
- Scanning Acoustic Microscopy (SAM)
- SEM (+ EDS)
- FIB-SEM (+ EDS)
- S/TEM (+ EDS / + EELS)
- **Nano-indent / Nano-scratch** New Tool !

Optical Microscopy & Spectroscopy

- Chromatic Aberration
- Digital Optical Microscopy
- **FTIR and ATR-FTIR** New Tool !
- Laser Scanning Confocal Microscopy
- Spectral Ellipsometry
- UV-Vis-NIR Spectroscopy
- White Light Interferometry

X-Ray Characterization

- X-Ray Diffraction (XRD)
- X-Ray Reflectometry (XRR)
- Micron-spot ED-XRF
- WDXRF
- Micro-computed X-ray Tomography (Micro-CT)
- 2D X-ray Inspection & X-ray Radiography

Elemental / Chemical Composition Analysis

- EPMA
- **GD-OES** New Tool !
- GC-MS
- **ICP-MS and LA-ICP-MS** New Tool !
- **Raman Microscopy & Spectroscopy** New Tool !
- NMR (1D or 2D; solid / liquid)

Particle Analysis

- Dynamic Light Scattering (DLS)
- Laser Diffraction Particle Size Analysis (PSA)
- Particle Zeta Potential

Material Property Characterization

- DSC
- DMA & TMA
- Rheometry
- TGA
- Surface Zeta Potential

Coming Soon:

- *Porometry / Porosity*
- *Gas Adsorption*
- *Gas Pycnometry*
- *Foam Density*
- *Tap Density*

Surface Spectroscopy Analysis

- Dynamic-SIMS
- ToF-SIMS (Static-SIMS)
- Ion Scattering Spectroscopy (ISS)
- Ultraviolet Photoelectron Spectroscopy (UPS)
- X-ray Photoelectron Spectroscopy (XPS)

Introducing

Austin Barnes, PhD

Member of Technical Staff, Scanning Probe Microscopy
Covalent Metrology

- Austin is the technical lead for the SPM group at Covalent Metrology (atomic force microscopy, nano-indentation, and scanning acoustic microscopy).
- Austin Barnes is a physical chemist with over 10 years of experience in scanning probe microscopy (SPM) with a wide range of expertise in scanning probe methodologies.
- Austin holds a PhD in Chemistry from the University of California, Santa Barbara and a B.S in Physics from the University of Massachusetts, Amherst

Introducing

Patrick O’Hara

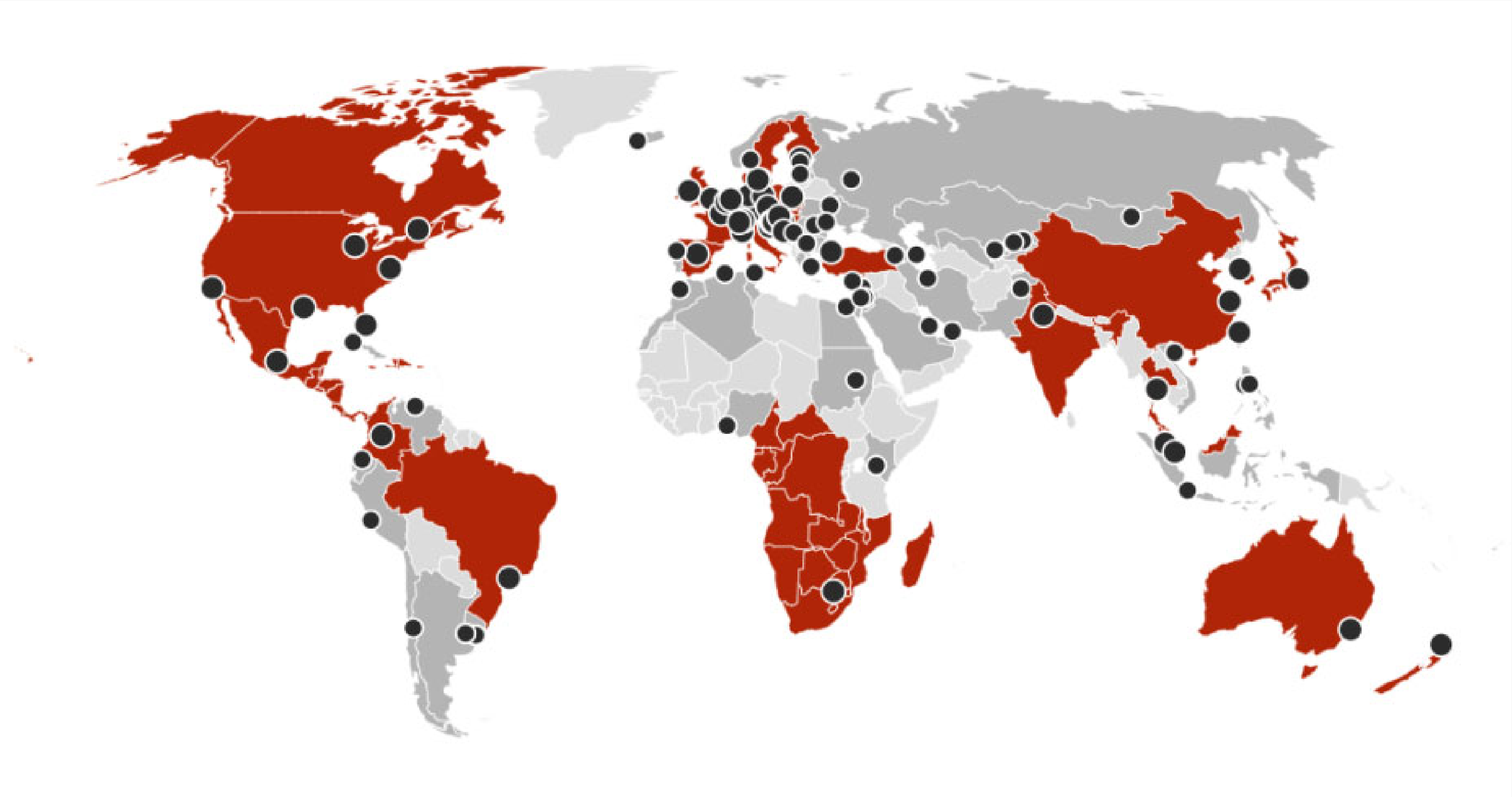
Technical Sales Representative
Anton Paar

- Patrick has worked in the surface characterization industry for more than 20 years
- He has been a Technical Sales Representative with Anton Paar for the past 2 years
- Experience in engineering, sales, marketing, and manufacturing of a variety of surface characterization instruments including:
  - Atomic force microscopes
  - White light interferometers
  - Indentation and scratch instruments
  - Stylus profilometers

Anton Paar

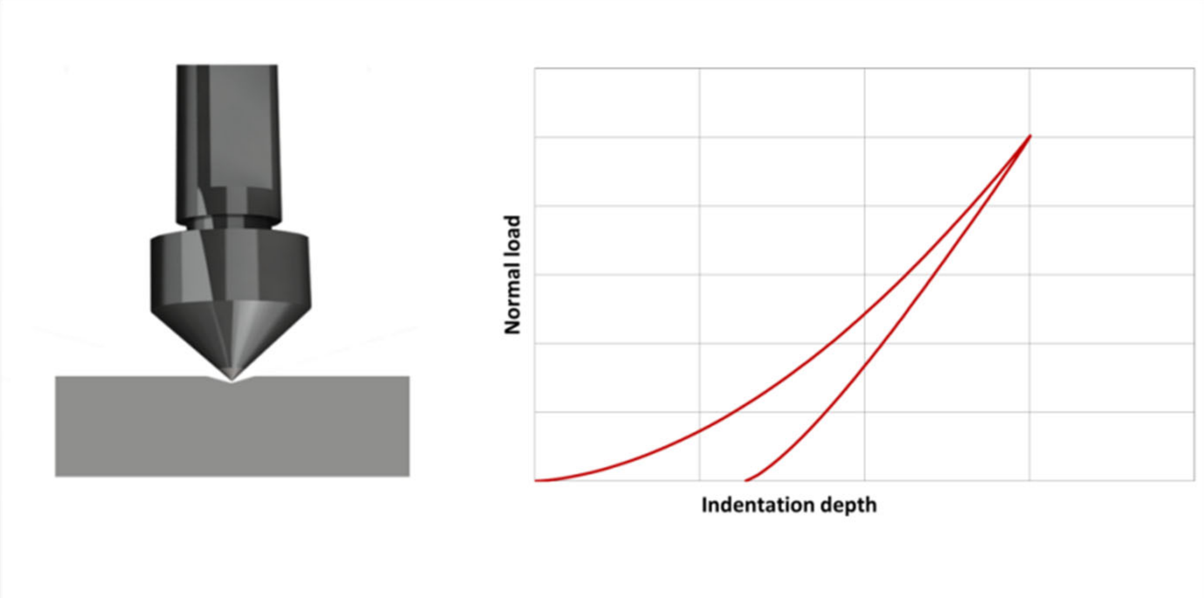
Anton Paar Overview

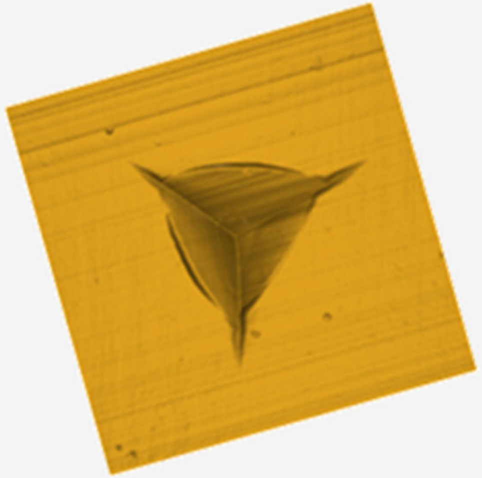
- Headquartered in Graz, Austria
- 3,400+ employees
- 33 sales subsidiaries worldwide
- 4 USA regional offices:
  - Ashland, VA
  - Torrance, CA
  - Houston, TX
  - Vernon Hills, IL



What is indentation testing ?

- Mechanical test derived from conventional hardness testing (Vickers, Brinell, etc.)
- An indenter of known geometry is pressed into a sample surface.
- Normal force and penetration depth are measured to get an indentation curve
- From the curve, several mechanical material parameters can be calculated



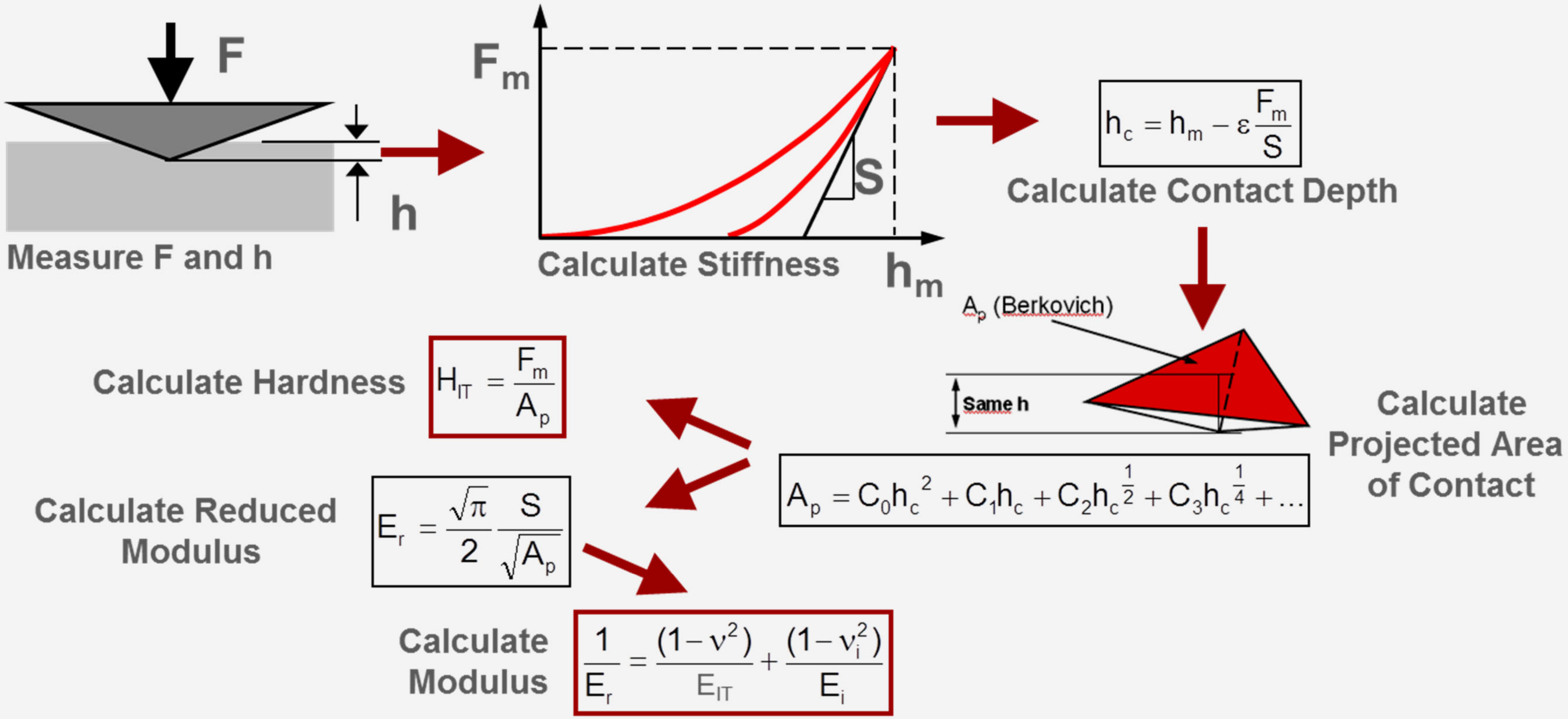


What information can I get ?

- **Hardness** : ability of a material to resist a permanent deformation
- **Elastic modulus** : resistance of a material to being elastically deformed
- **Creep** : time dependent permanent deformation under a certain applied load
- **Viscoelasticit**y : property of a sample that exhibits both viscous and elastic properties
- **Many other parameters** : fracture toughness, breaking force, deformation energies, etc.

Indentation calculations

Oliver and Pharr model



Measure F and h

Calculate Stiffness

$$h_c = h_m - \varepsilon \frac{F_m}{S}$$

Calculate Contact Depth

Calculate Projected Area of Contact

$$A_p = C_0 h_c^{\,2} + C_1 h_c + C_2 h_c^{\frac{1}{2}} + C_3 h_c^{\frac{1}{4}} + \ldots$$

Calculate Hardness

$$H_{IT} = \frac{F_m}{A_p}$$

Calculate Reduced Modulus

$$E_r = \frac{\sqrt{\pi}}{2} \frac{S}{\sqrt{A_p}}$$

Calculate Modulus

$$\frac{1}{E_r} = \frac{(1-\nu^2)}{E_{IT}} + \frac{(1-\nu_i^2)}{E_i}$$

from properties of indenter tip and sample Poisson's ratio ν

Why should I choose indentation testing ?

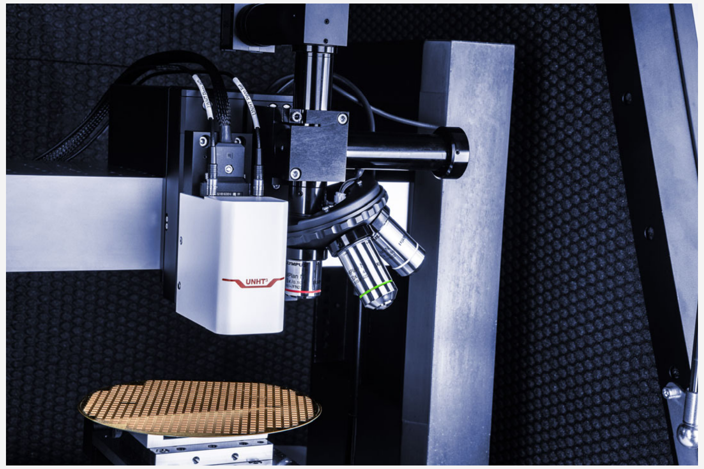
- Automated measurement : no optical analysis, measurement of pre-defined matrices
- Calculation of several mechanical properties in a single measurement (hardness, elastic modulus, loss modulus, etc.)
- Fast measurements : only a few seconds to get a measurement
- Only reliable method of measuring hardness of coatings
- Measurements can be made on small features in precise locations

Challenges with instrumented indentation

- Tip area calibration is important to making accurate and repeatable measurements
- Dirty indenters and surfaces can cause inaccurate measurements
- Substrate effects can influence the validity of mechanical property measurements of thin films
- Similarly surface roughness should be much less than the depth of the indent to acquire accurate measurements
- Sample mounting is critical to avoid issues related to compliance

ULTRA NANOINDENTATION TESTER
UNHT^3

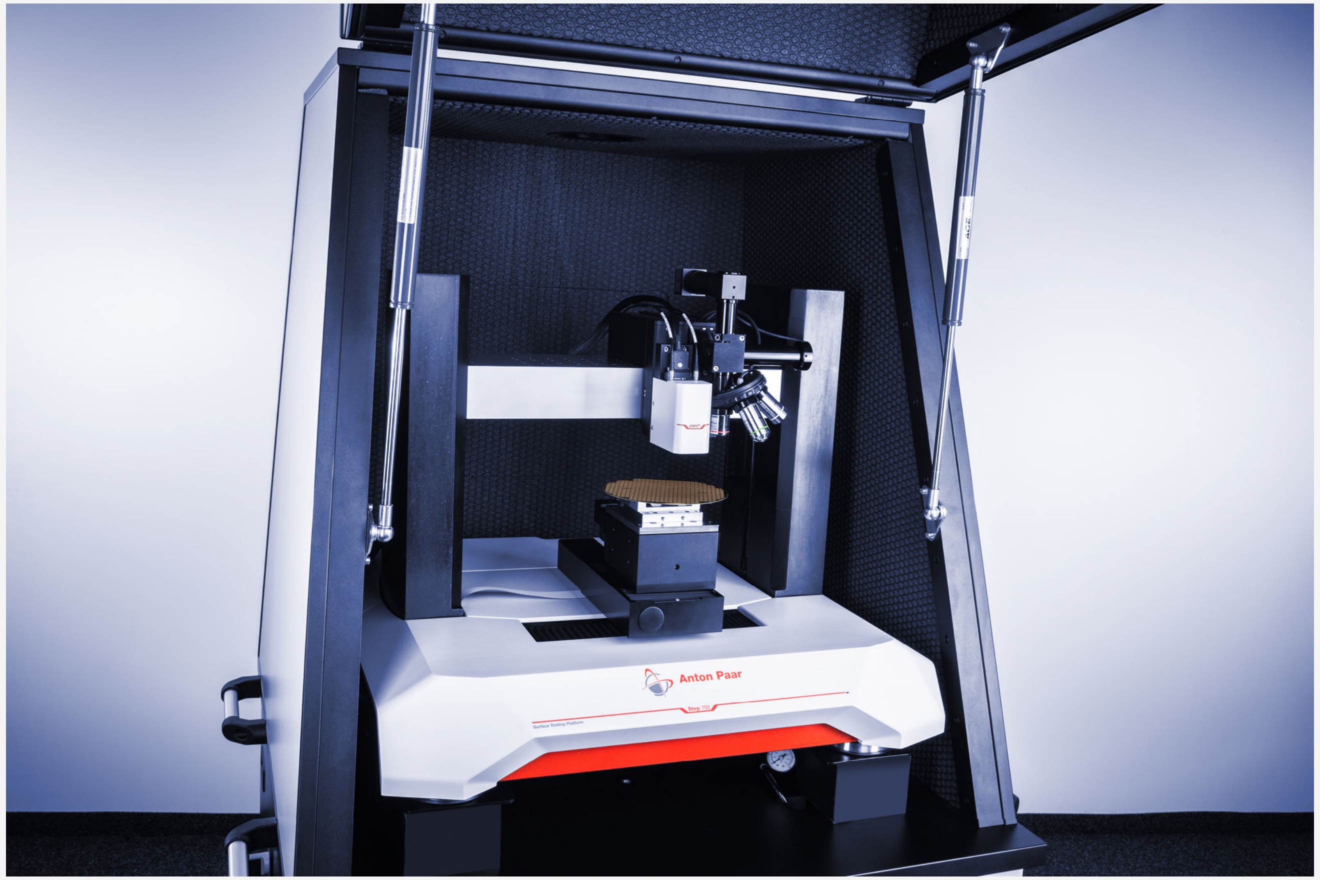
High resolution nanoindentation tester



Ultra Nanoindentation Tester - UNHT^3

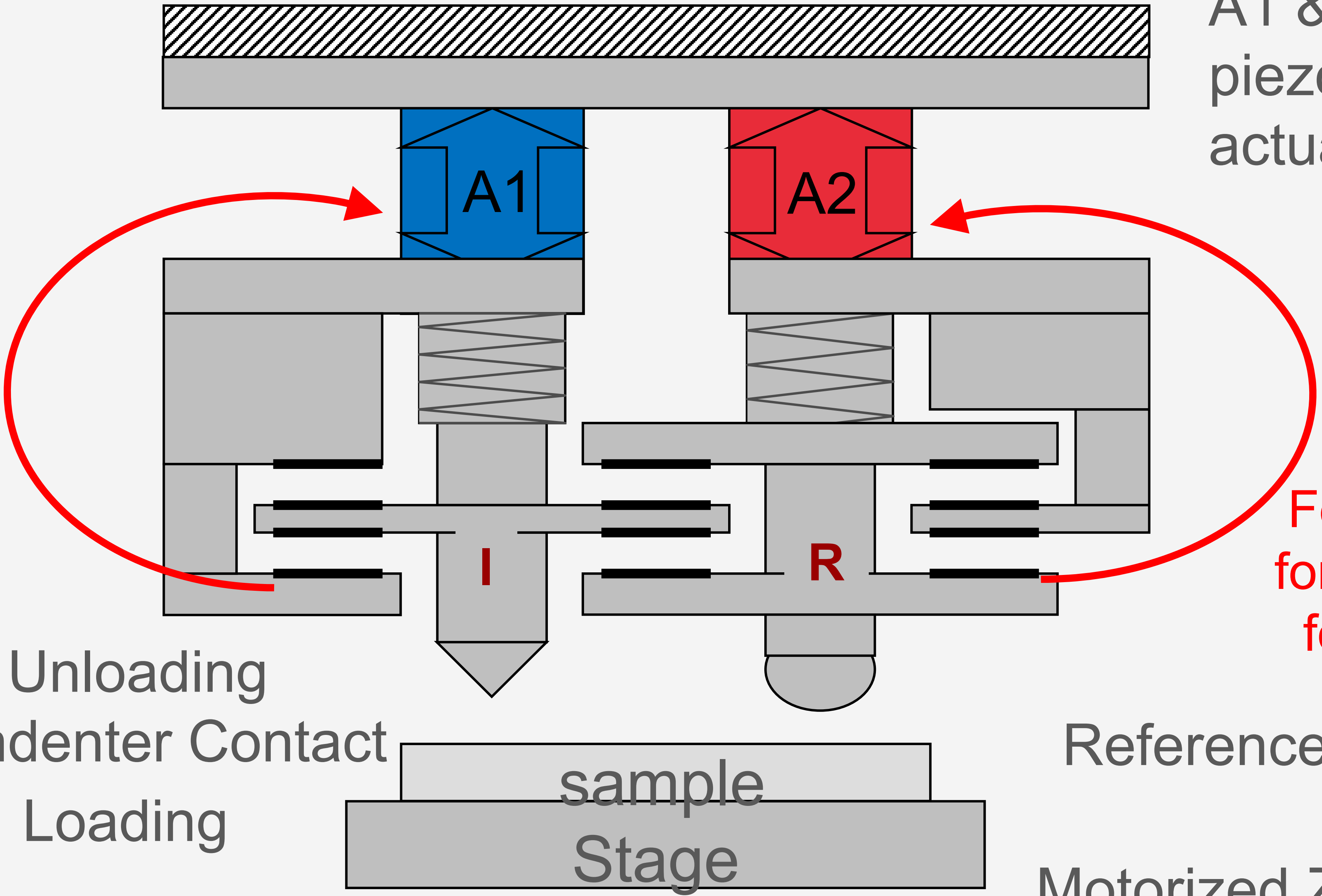
High resolution nanoindentation tester

UNHT^3	
Load range	0.01 mN – 100 mN
Load resolution	0.003 µN
Load noise floor	< 0.05 µN
Maximum penetration depth	100 µm
Depth resolution	0.003 nm
Depth noise floor	< 0.03 nm



UNHT^3 Design

Patented active top referencing



A1 & A2: piezoelectric actuators

Feedback loop on force sensor

Feedback loop for accurate low force sensing

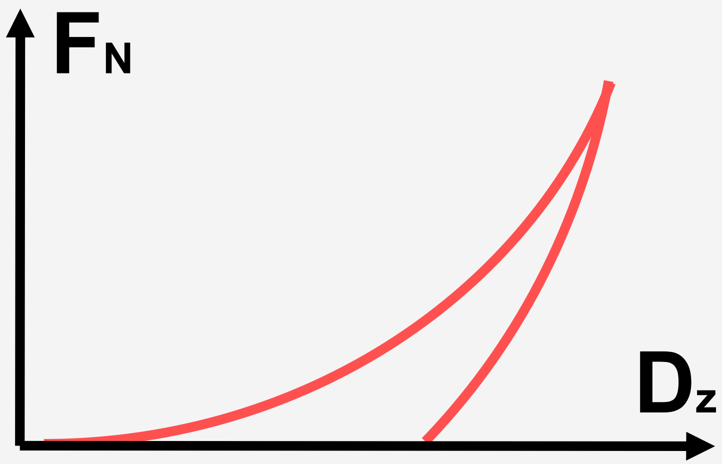
Unloading

Indenter Contact

Loading

Reference contact

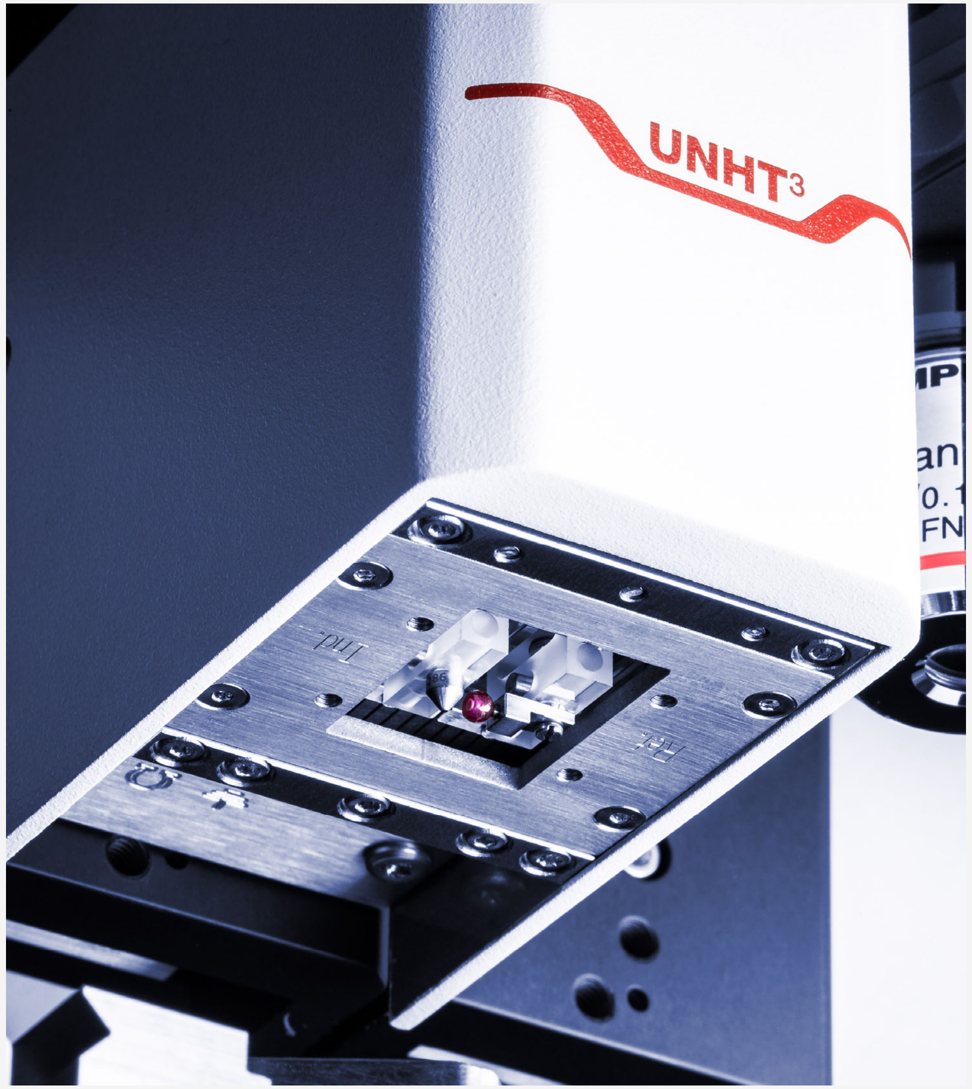
Motorized Z table



Load-depth curve

UNHT^3

- The most accurate nanoindentation tester
  - Two independent depth and load sensors provide true control of forces and indentation depth
- The nanoindenter with the highest stability on the market
  - Unique patented active top surface referencing
  - Negligible thermal drift down to 10 fm/sec without any depth correction
  - Only nanoindentation tester that can be used for long-time measurements such as creep tests.
- High throughput and measurement speed (>600 measurements per hour)
  - A sample can be measured immediately after installation without waiting hours for thermal stabilization
  - Quick Matrix mode : more than 600 measurements per hour with real indentation
- Additional dynamic mechanical analysis (DMA) with “Sinus Mode”



UNHT^3 – Typical Applications

- **Thin films** : high resolution for low penetration depths
- **Glass Industry** : excellent combination with NST (anti-reflex, anti-scratch coatings, etc.)
- **Polymers** : unique thermal stability to measure creep
- **Microelectronics**

Anton Paar

Tip Area Calibration

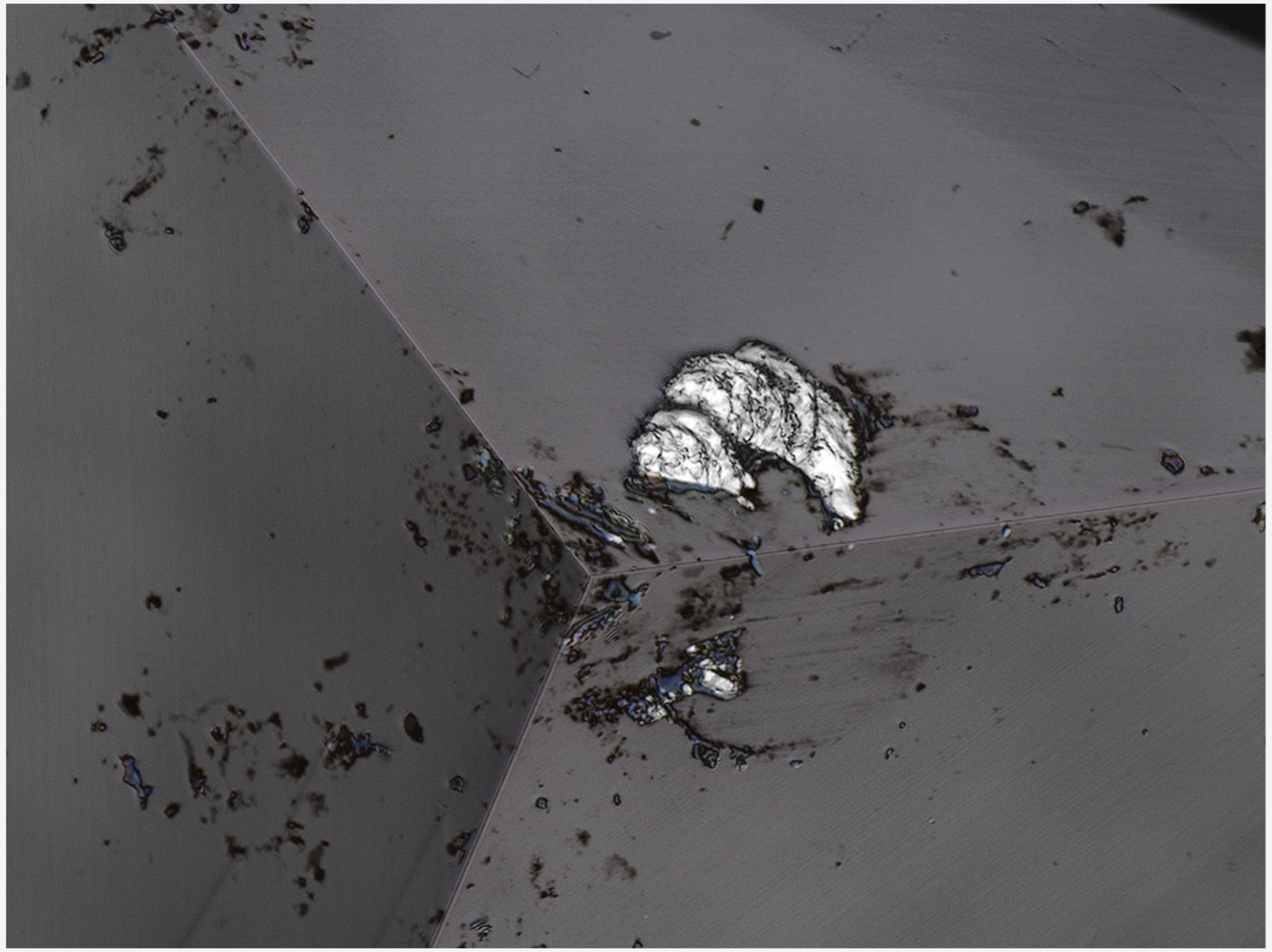
- Tip area is a function of contact depth

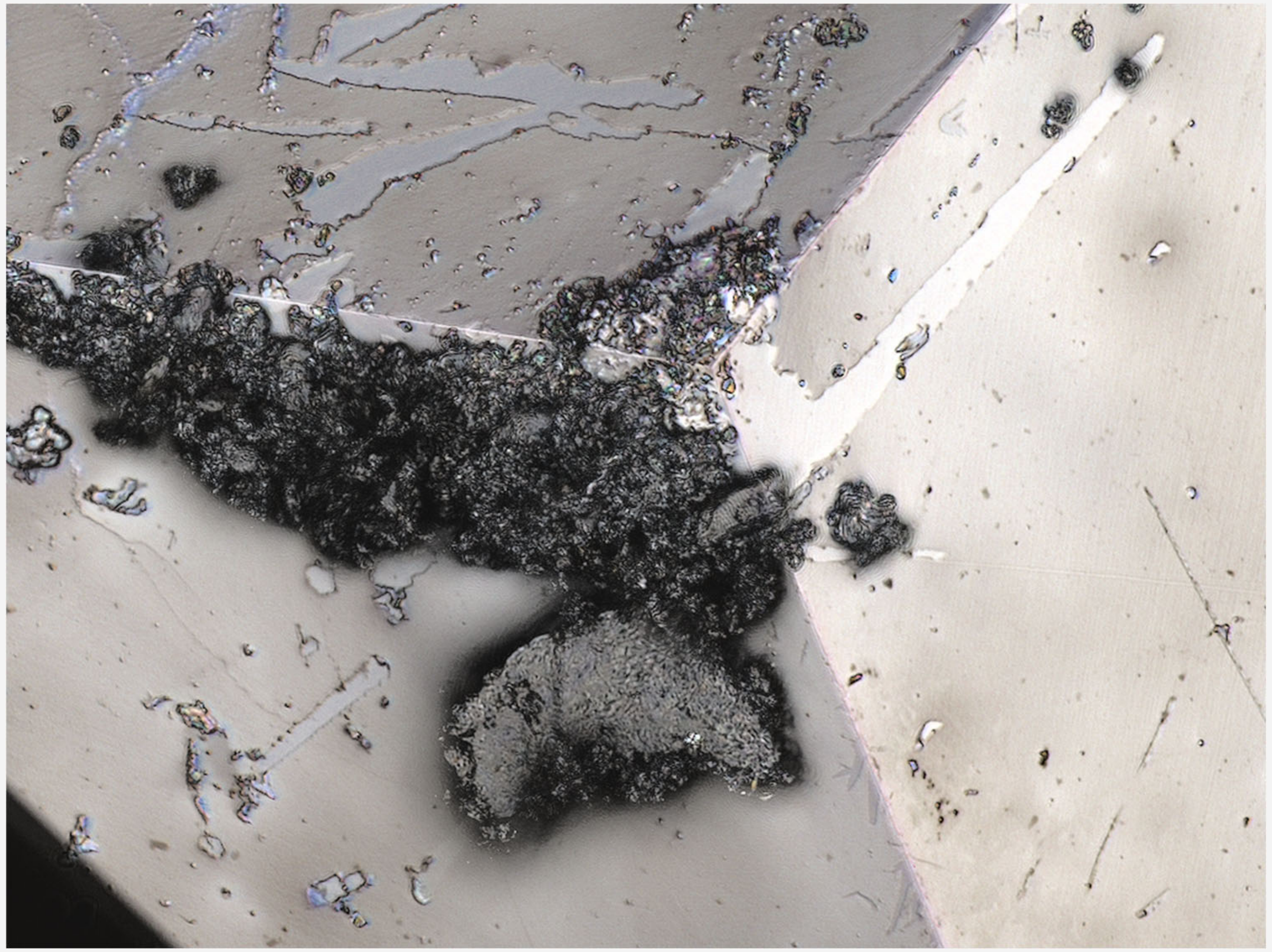
$$\mathbf{Area} = \mathbf{24.5} \times (\boldsymbol{Depth})^2$$

for an ideal Berkovich tip

- Accuracy of hardness and modulus is influenced by how well the tip area is calibrated
- At low contact depths (less than 40 nm) tip area deviates from an ideal Berkovich
- This limitation is typically resolved by generating a separate calibration curve to very low contact depths

Tip Area Calibration and Tip Cleanliness



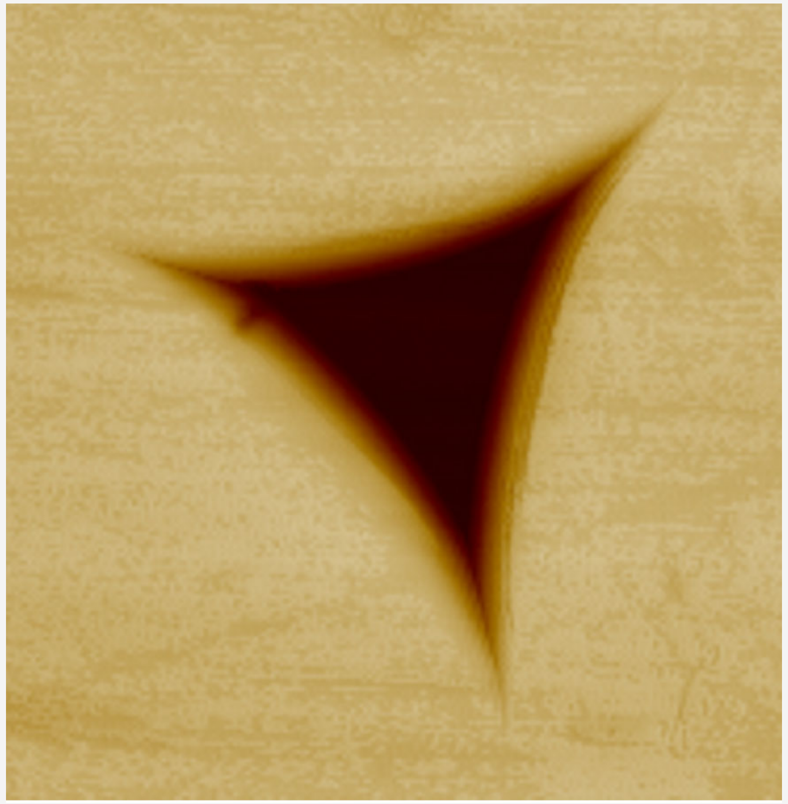


Substrate Effects and the 10% Rule

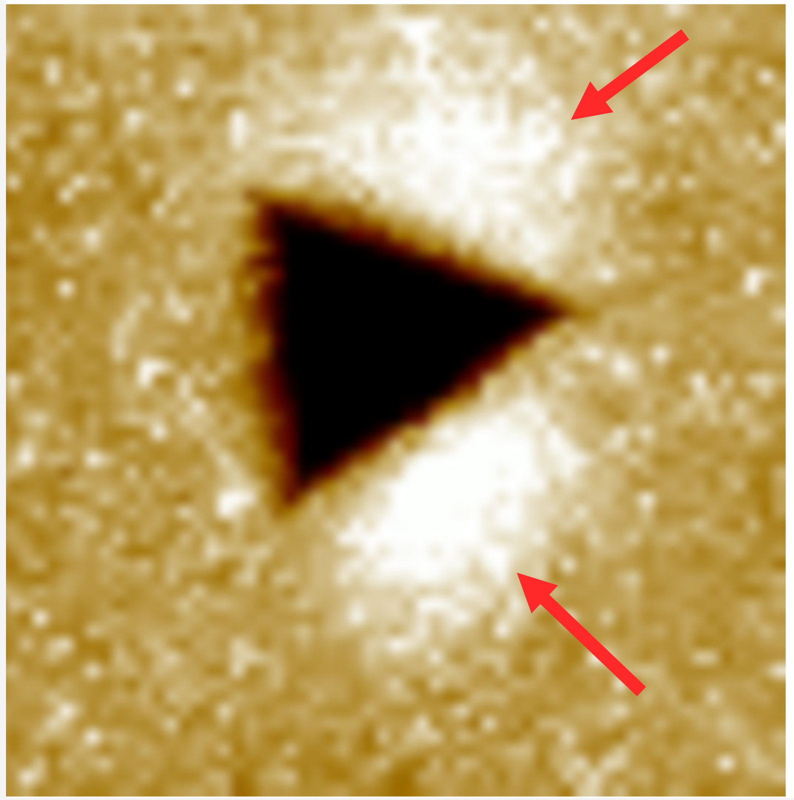
- **10% Rule:** Contact depths should not exceed 10% of the coating thickness
- Can get an idea of necessary contact depths if coating thickness is known
- Progressive multi-cycle indentations will measure modulus vs. depth and show at what depth substrate effects dominate

Material Pile-up and its Influence on Hardness and Modulus

Fused Quartz



Aluminum



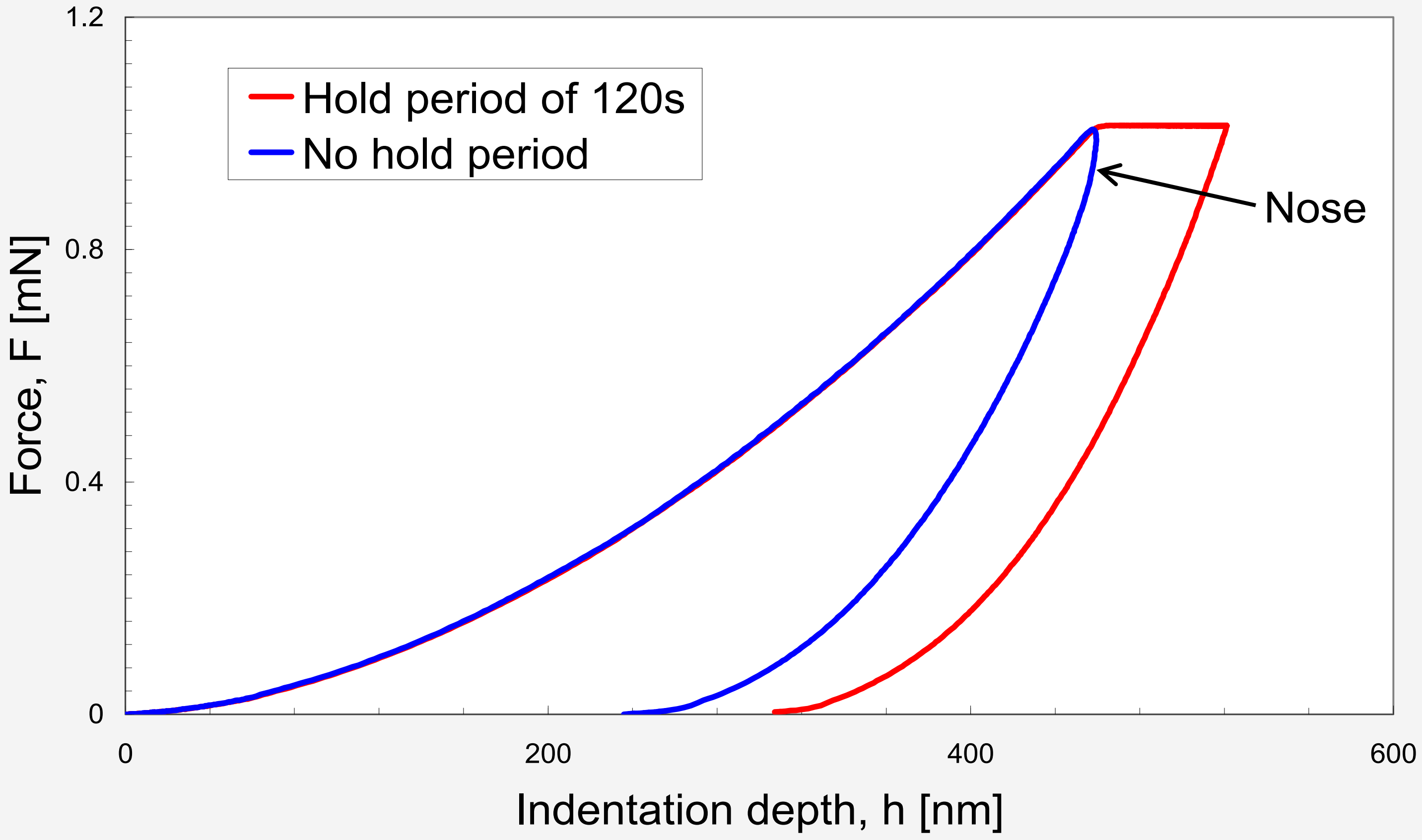
- AFM images show how different materials deform under similar load
- In some cases, pile-up can lead to significant source of error in tip surface area calculation
- This highlights the importance of being able to take an image of the indents after testing

Surface Roughness Effects

- Surface roughness contributes to variance in hardness and modulus
- Indenting on a peak (versus a valley) influences the area sampled by the tip
- Greater contact depths reduce variance
- Typically contact depths should be greater than 10 × R_a

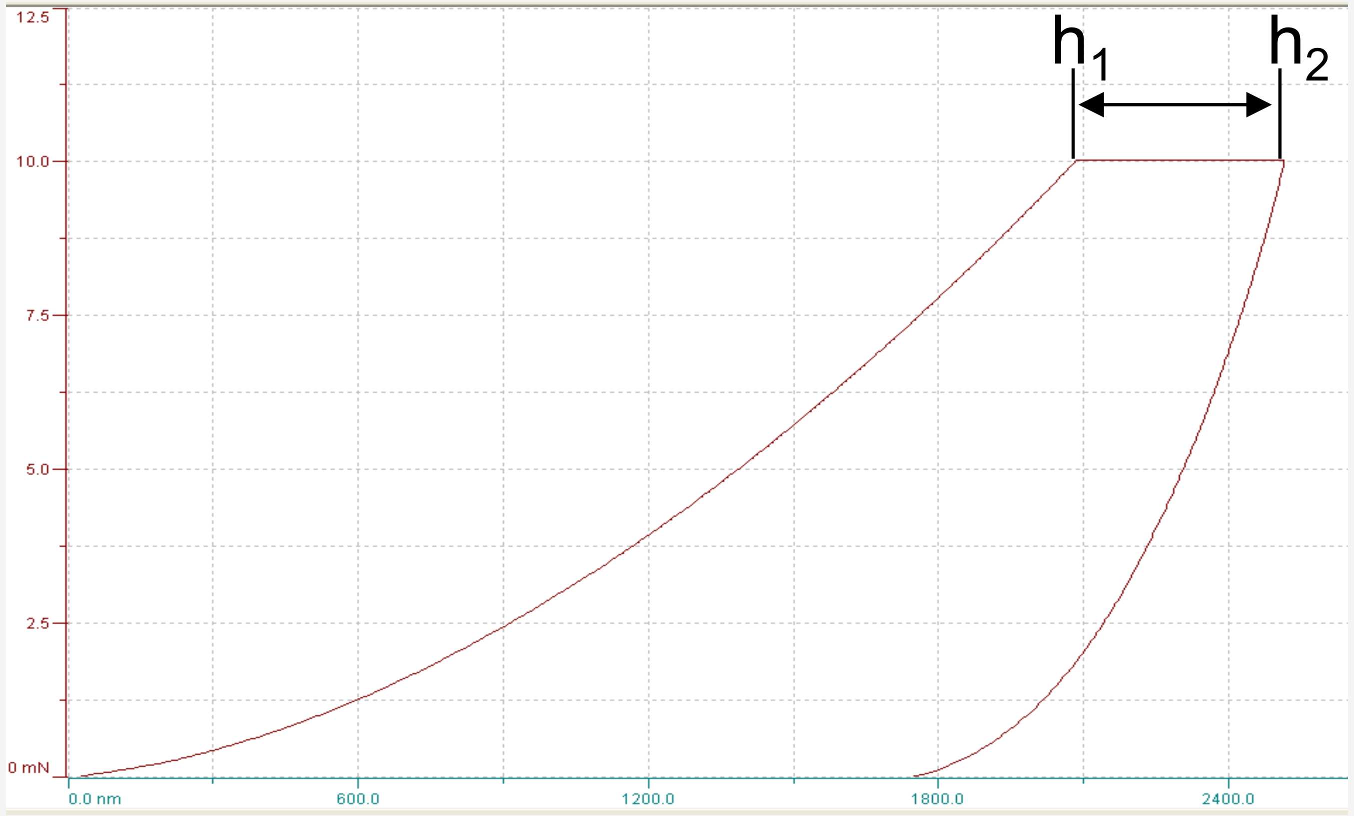
Creep During Nanoindentation

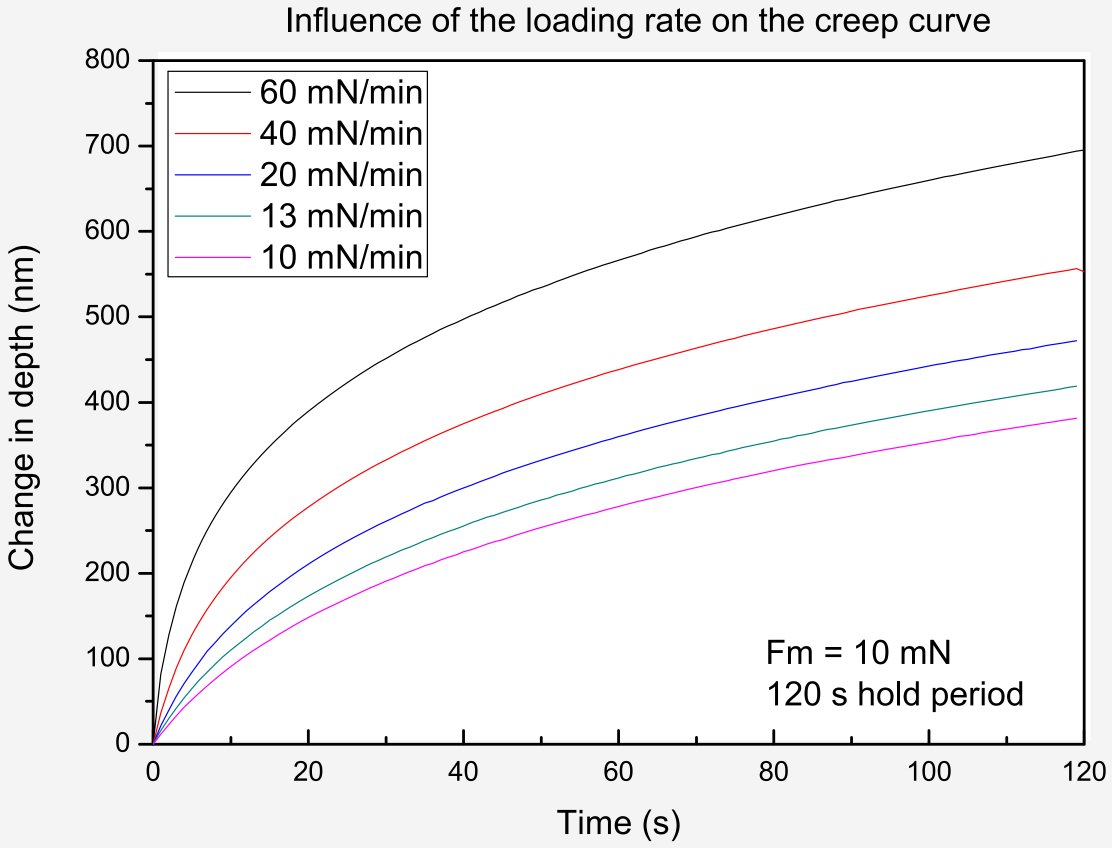
- In an indentation test, creep often manifests itself as a bowing out or “nose” in the unloading portion of the force-displacement curve
- For such material, when the force is held during a certain time at the maximum force, the indenter continues to penetrate



Creep Procedure for Nanoindentation

- Recommended procedure to analyze creep is fast loading (~2 to 10 seconds) followed by long hold (30 to 120 seconds) at constant force
- Fit depth increase during hold at constant force by using Oyen approach (Indentation software)
- Simpler form of creep (C_{IT}) is defined by ISO 14577: $C_{IT} = \frac{h_2 - h_1}{h_1}$

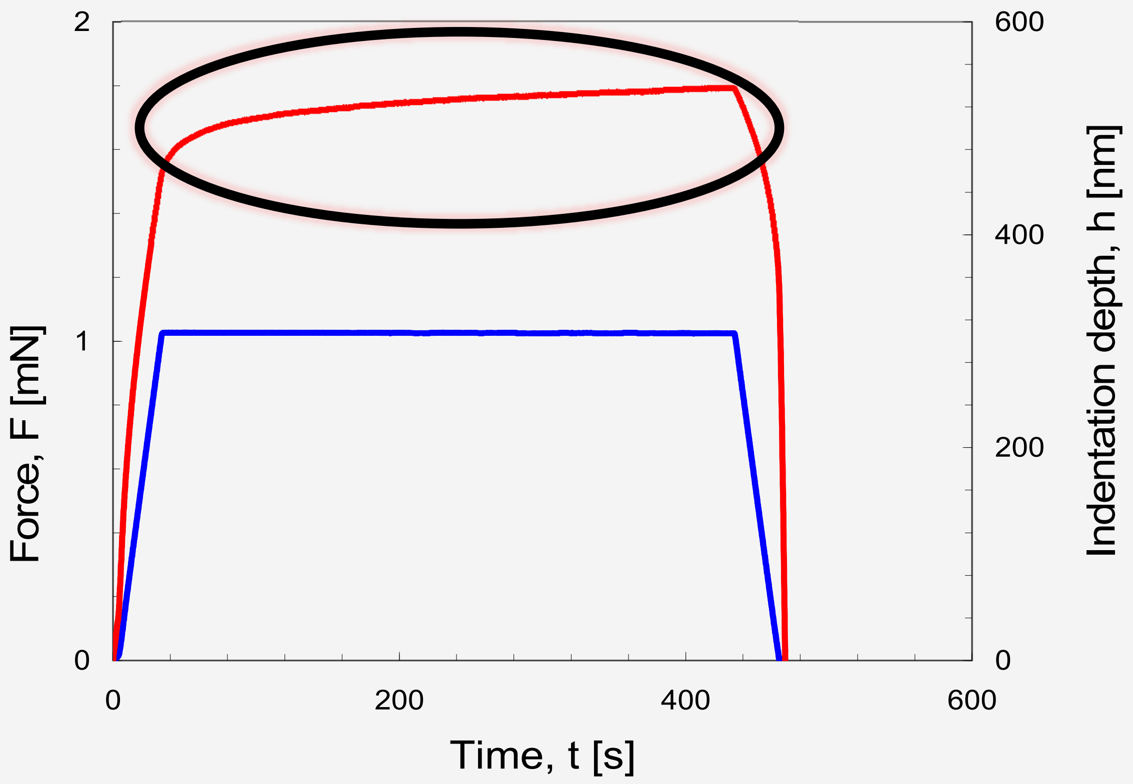




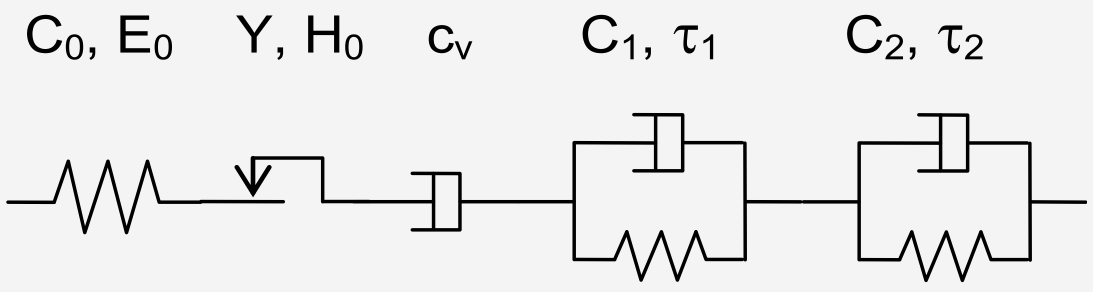
Oyen, Michelle L. “Spherical Indentation Creep Following Ramp Loading.” *Journal of Materials Research* 20, no. 08 (2005): 2094–2100.

Spherical Indenters – Creep Analysis

Fit of displacement data during hold period



$$[h(t)]^m = K \cdot F\left\{C_0 + \sum C_j\left[1-\rho_j e^{-\frac{t}{\tau_j}}\right]\right\}$$



Kelvin-Voigt Model (usually 2 to 3 bodies)

C_1, C_2, .. , C_n: compliances

t_j : relaxation times

Obtained by fitting

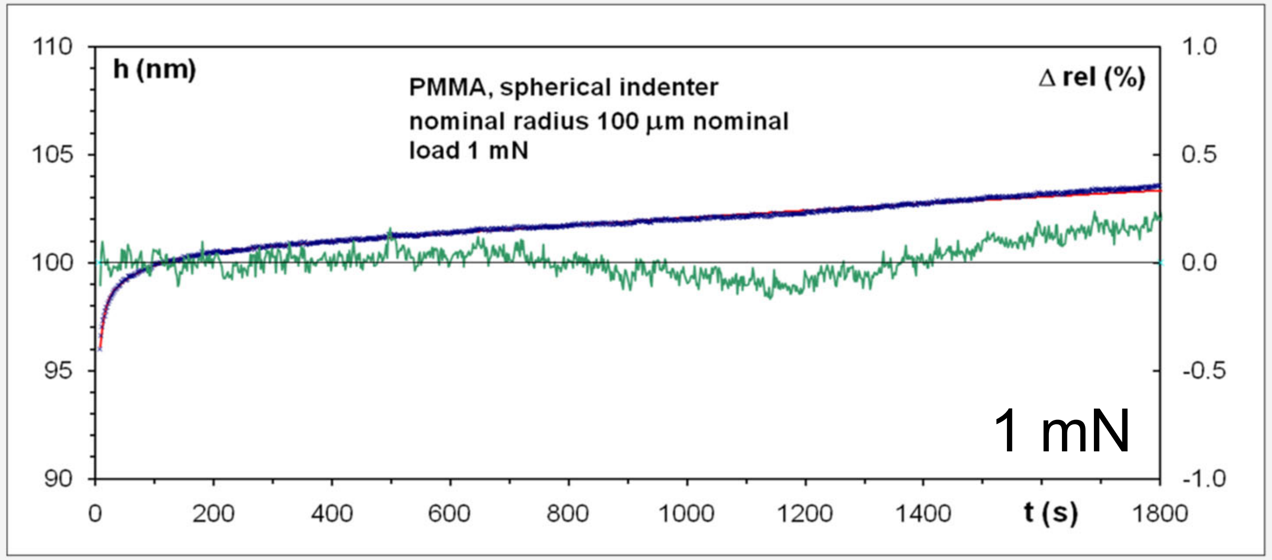
Shear modulus, elastic modulus, bulk modulus:

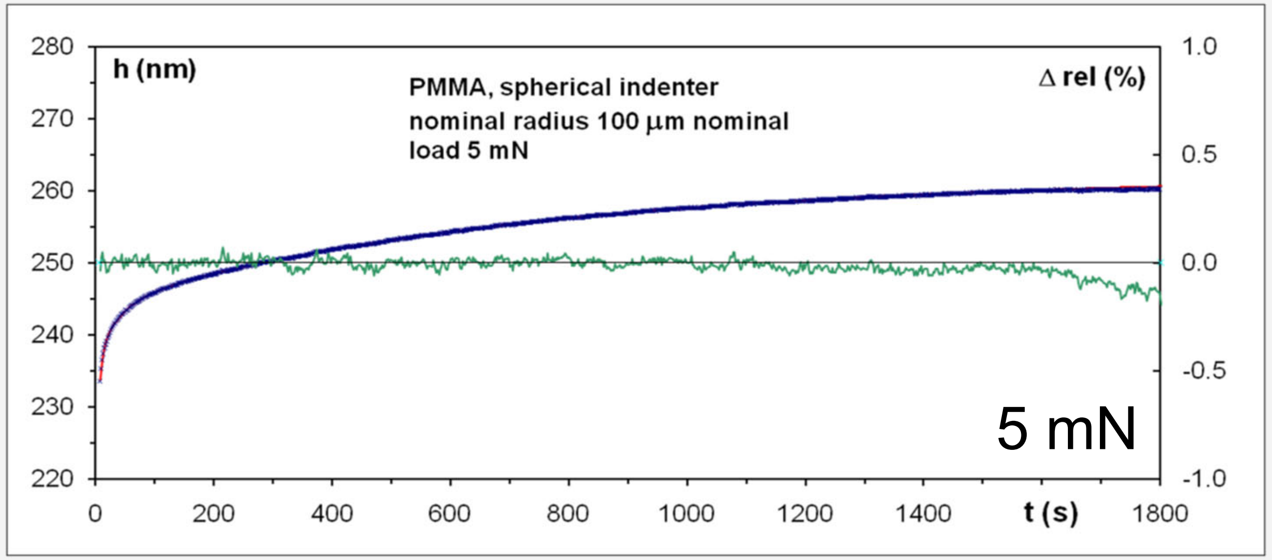
$$G = \frac{1}{2\left(C_0 - \sum C_i\right)} \qquad E = 2G(1+\nu) \qquad K = E/3(1-2\nu)$$

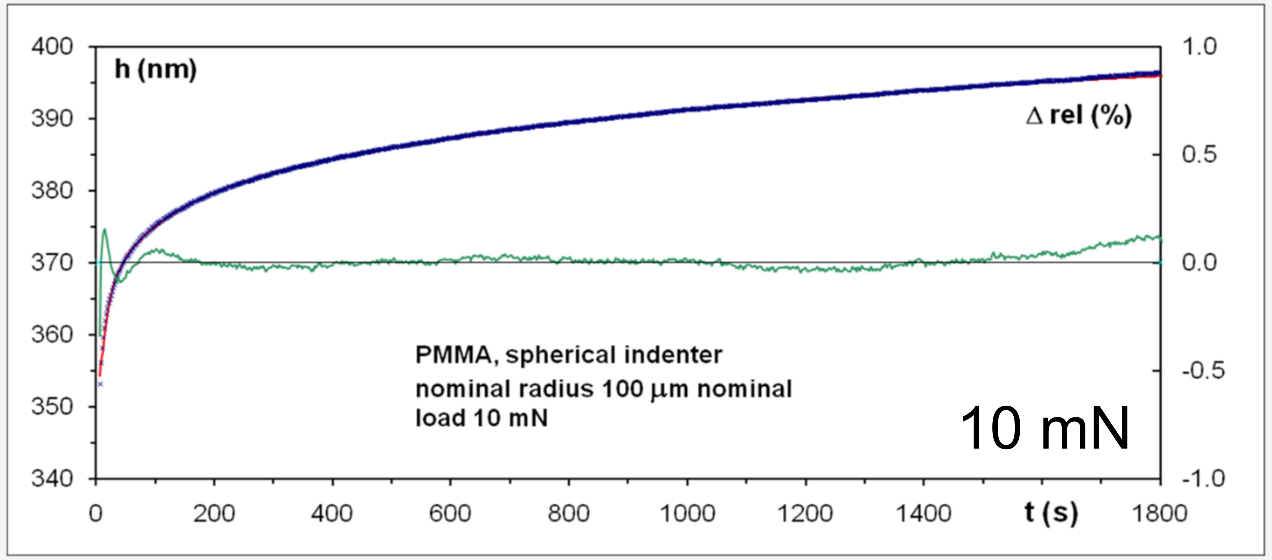
Excellent thermal stability needed

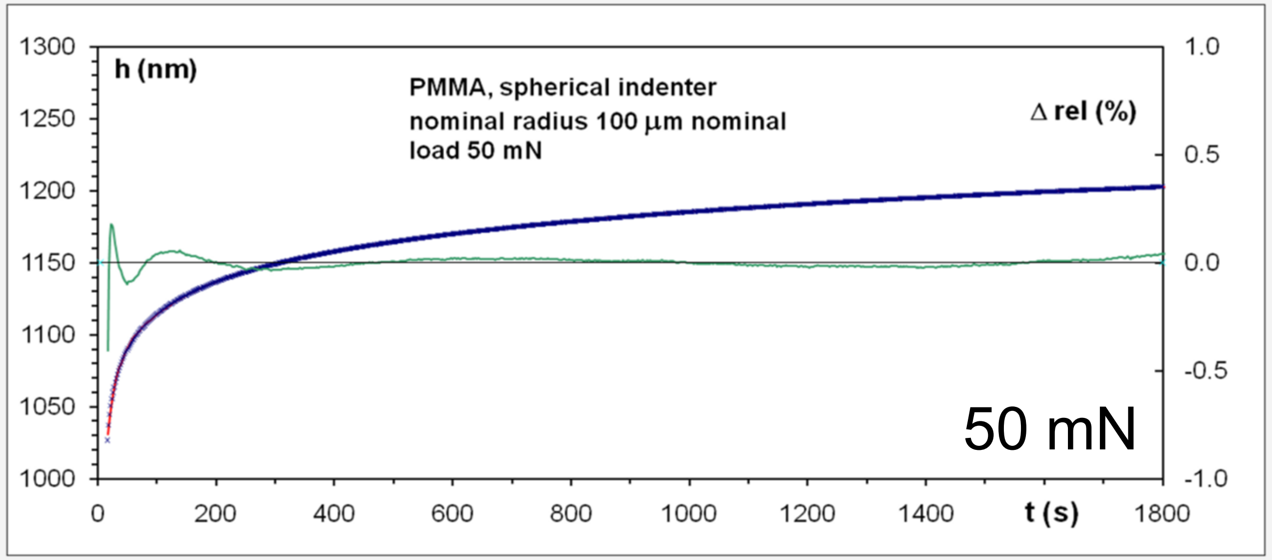
*J. Mencik, L.H. He, M. Swain Journal of the Mechanical Behavior of Biomedical Materials 2 (2009) p. 318

Fitting of PMMA creep data (UNHT)









Red line: measurement Blue line: creep compliance function fit Green line: relative error

Nohava, Jiří, and Jaroslav Menčík. “A Contribution to Understanding of Low-Load Spherical Indentation—Comparison of Tests on Polymers and Fused Silica.” Journal of Materials Research 27, no. 1 (January 14, 2012): 239–44. https://doi.org/10.1557/jmr.2011.267.

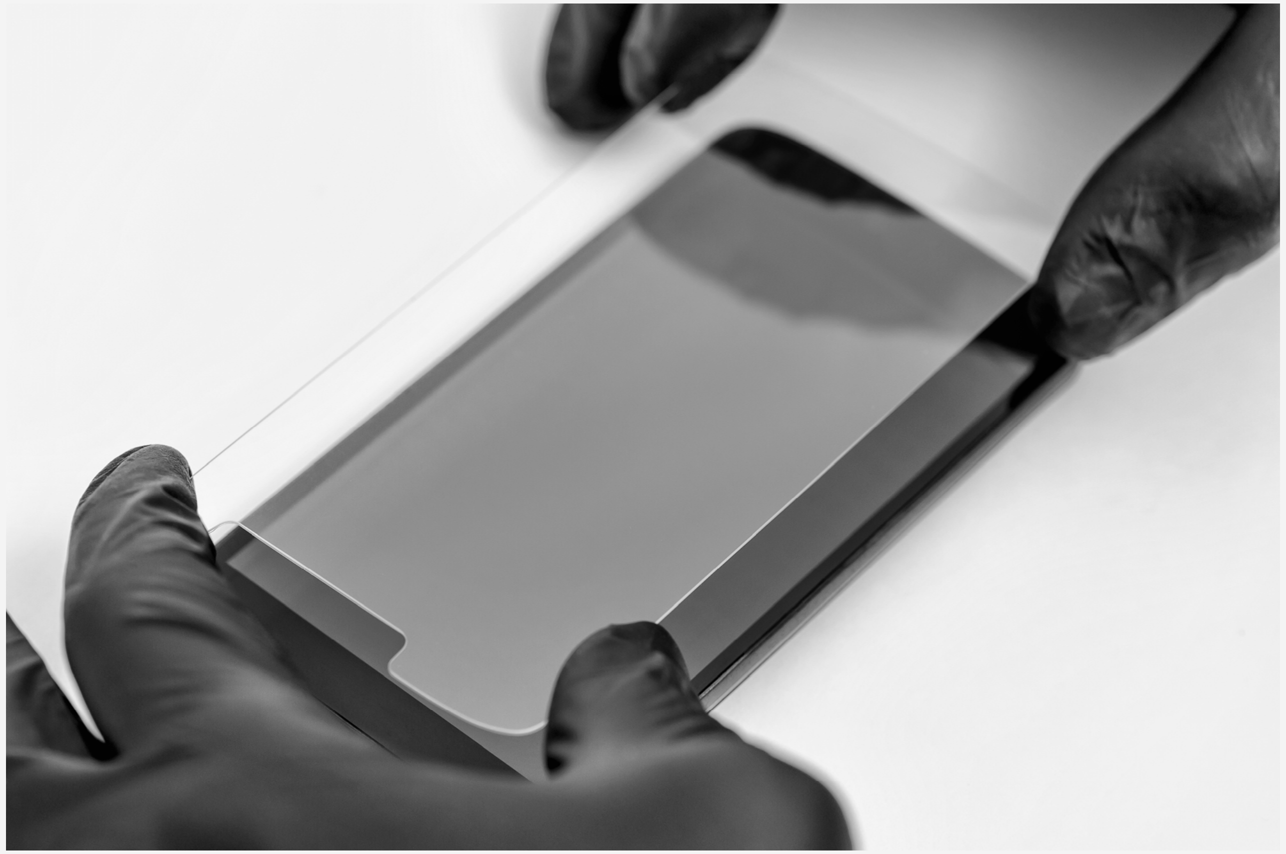
Properties of Thin Protective Layers on Displays

“I want to know the hardness of a very thin protective layer.”

Hardness of protective layers is very important for scratch and fracture resistance. This is why we need to measure hardness of these layers.

Protective transparent organic film on glass substrate:

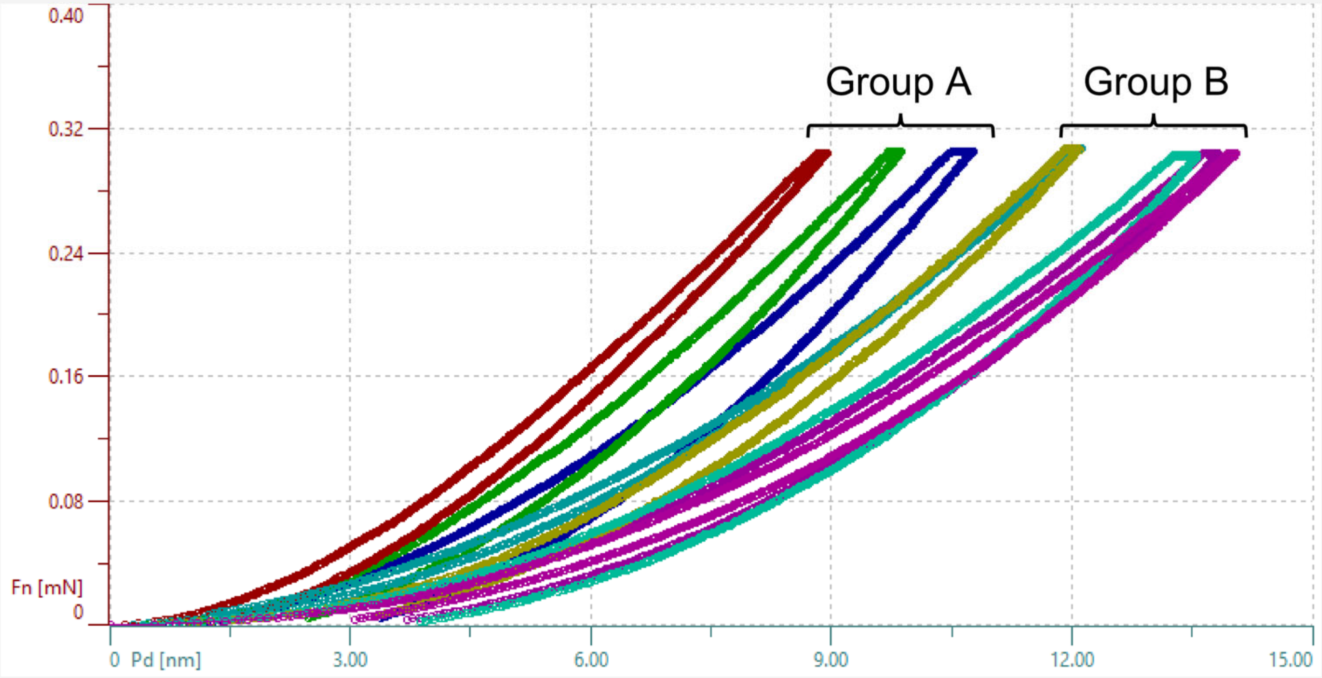
- Only 100 nm thickness
- UNHT³ for hardness and elastic modulus



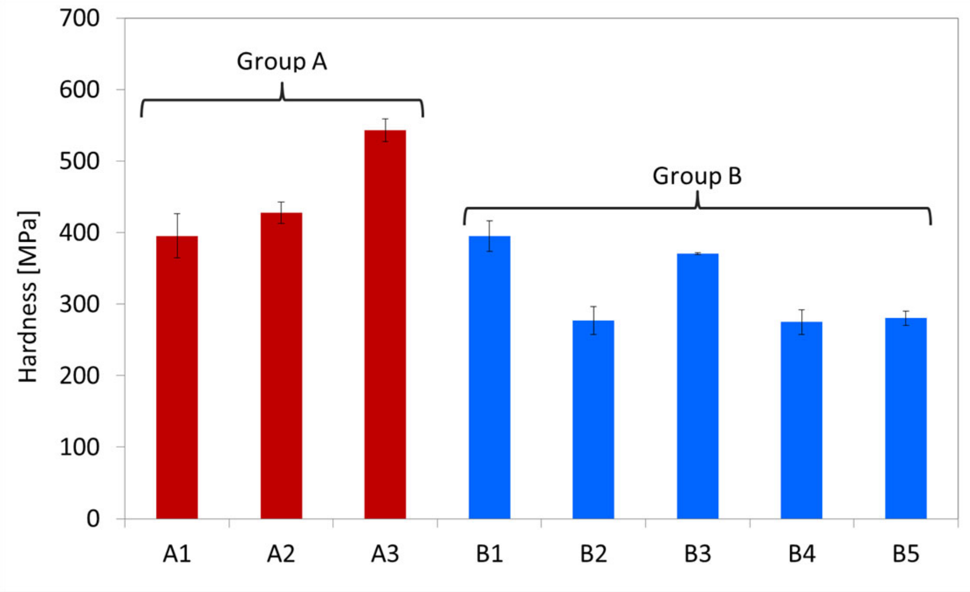
Hardness of Thin Protective Layers on Displays

“I want to know the hardness of a very thin protective layer.”

- Here we have examples of UNHT³ hardness measurements on eight thin layers. The UNHT³ can easily measure such thin layers
- We found differences between two groups of the layers and we could also differentiate layers in each group



- Group A: layers A1, A2, A3
- Group B: layers B1 to B5
- Maximum penetration depth ~10 nm



Layers in group A have higher hardness
→ they have better resistance to scratching

Layers in group B have lower hardness
→ they are less wear resistant

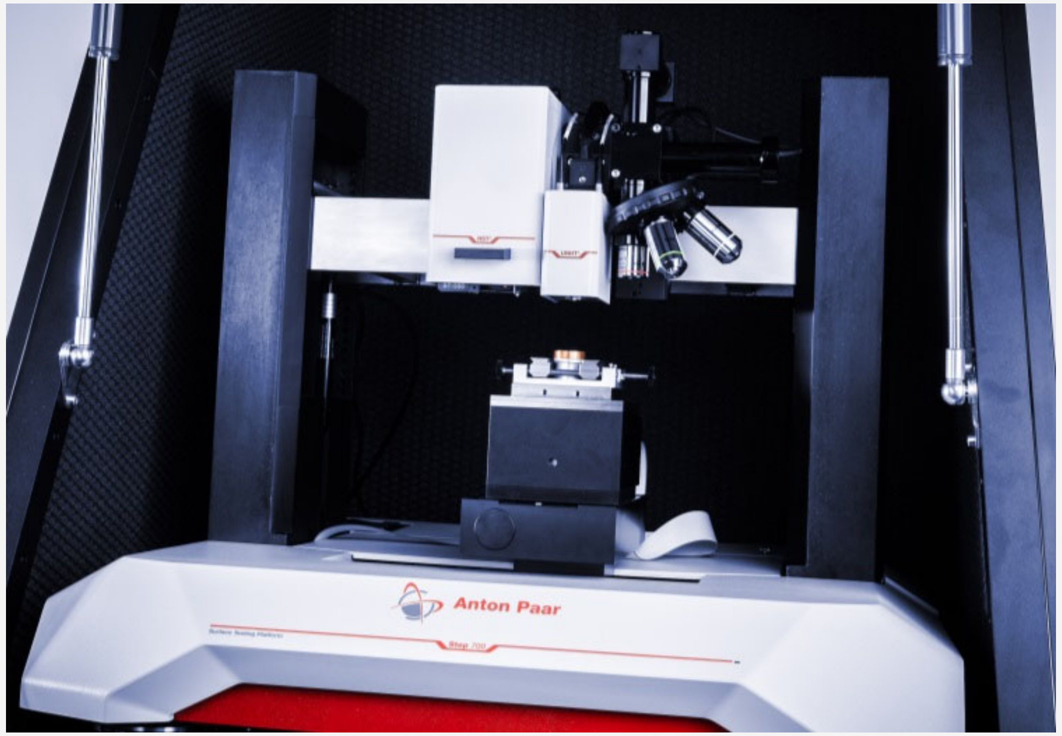
Nanoindentation of Thin Layers on Displays I

Organic film on glass substrate

- 100 nm thickness
- Ultra low load nanoindentation (UNHT) for hardness and elastic modulus

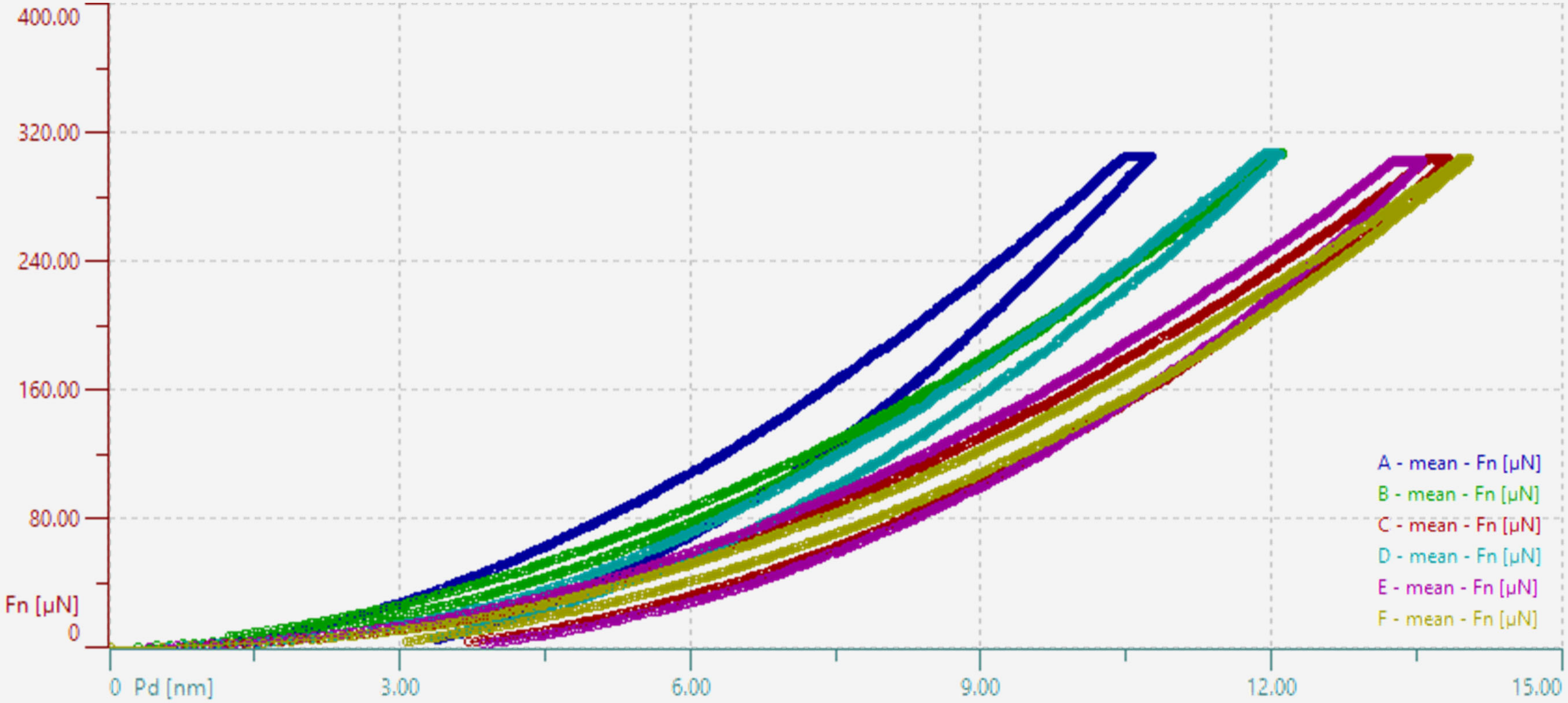
Nanoindentation

- Depth limited to ~10% of the thickness: ~12 nm
- Linear loading up to 300 µN
- Smooth surface: five indentations per sample



Nanoindentation – Comparison of Indentations

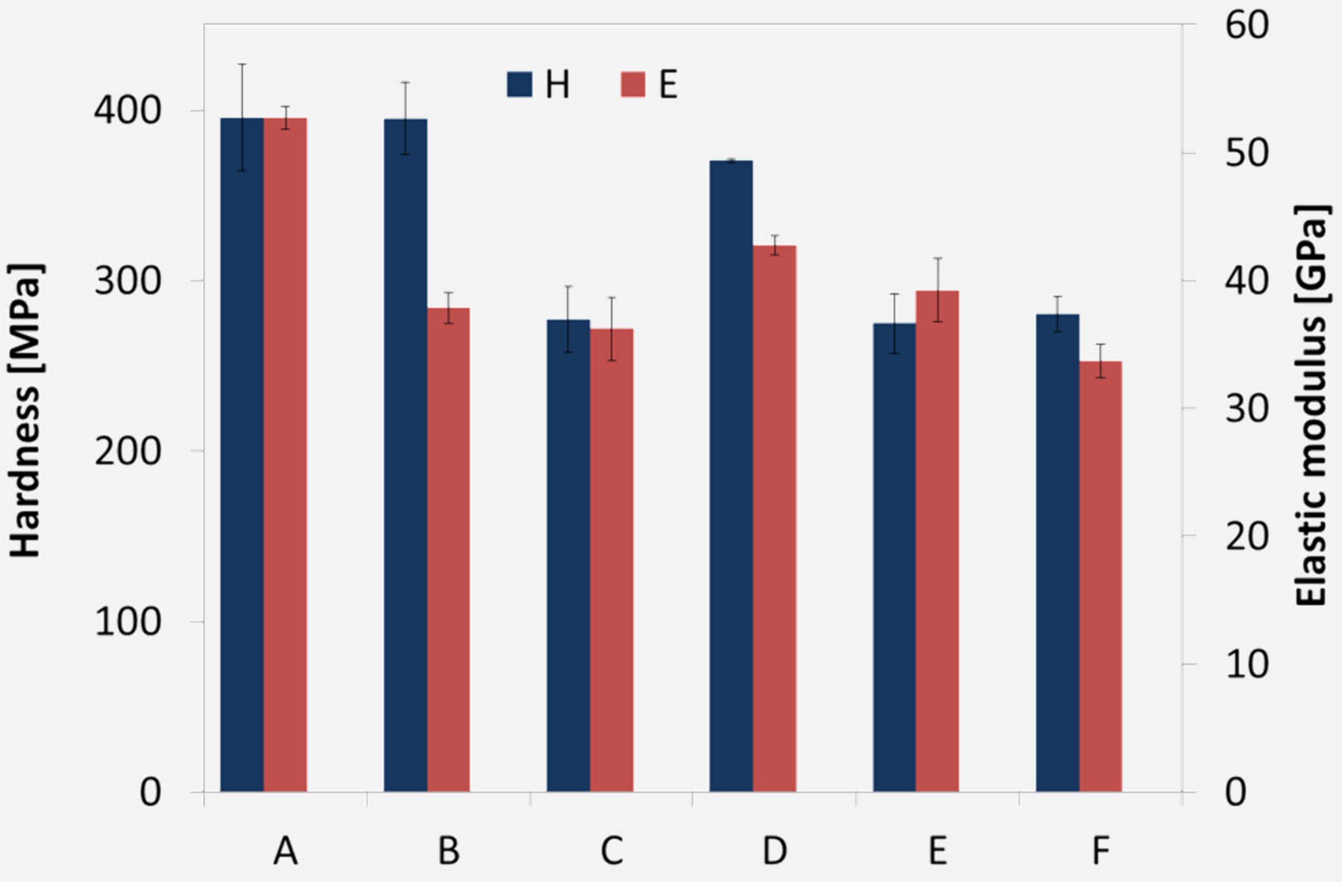
Maximum Indentation Depth ~14 nm



Nanoindentation – Results

Homogeneous layer with smooth surface

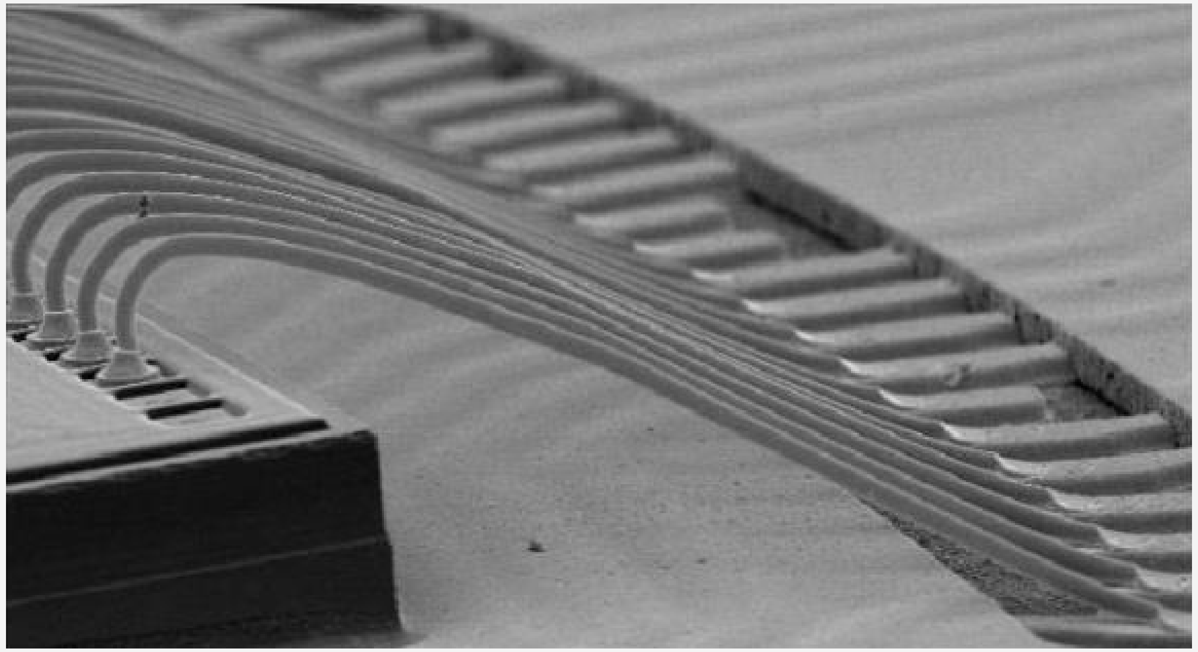
→ very good repeatability despite low penetration depth (~12 nm)



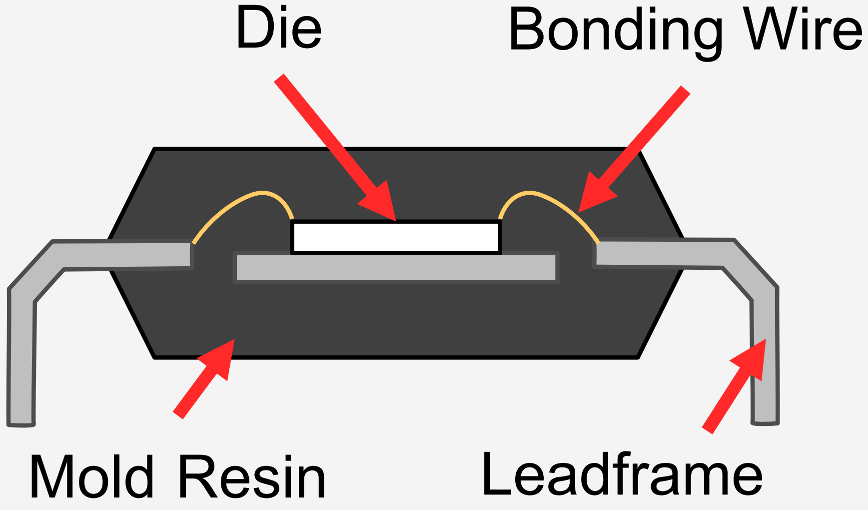
		A	B	C	D	E	F
HIT (O&P)	Mean	**395.43**	**395.03**	**277.13**	**370.32**	**274.81**	**280.27**
[MPa]	Std Dev	31.21	21.17	19.33	1.13	17.54	10.15
EIT (O&P)	Mean	**52.70**	**37.84**	**36.21**	**42.77**	**39.23**	**33.69**
[GPa]	Std Dev	0.90	1.18	2.50	0.75	2.48	1.29
hm (O&P)	Mean	**10.73**	**12.05**	**13.84**	**12.10**	**13.57**	**14.03**
[nm]	Std Dev	0.71	0.53	0.50	0.17	0.47	0.26

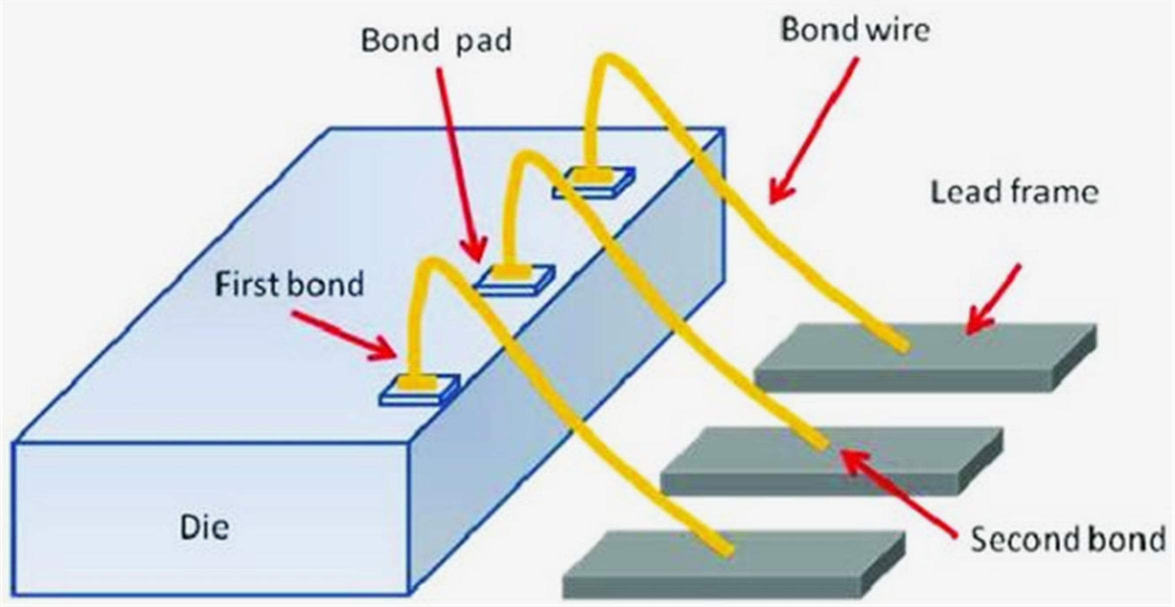
Hardness of bonding pads

- Bonding pads: electric connection of PCB to the outside
- Connections by soldering, wirebonding etc.
- Pure metals (Al) or intermetalics (AlSiCu, NiPdAu)
- Quality of soldering depends of mechanical properties of the bond pad (related to structure)



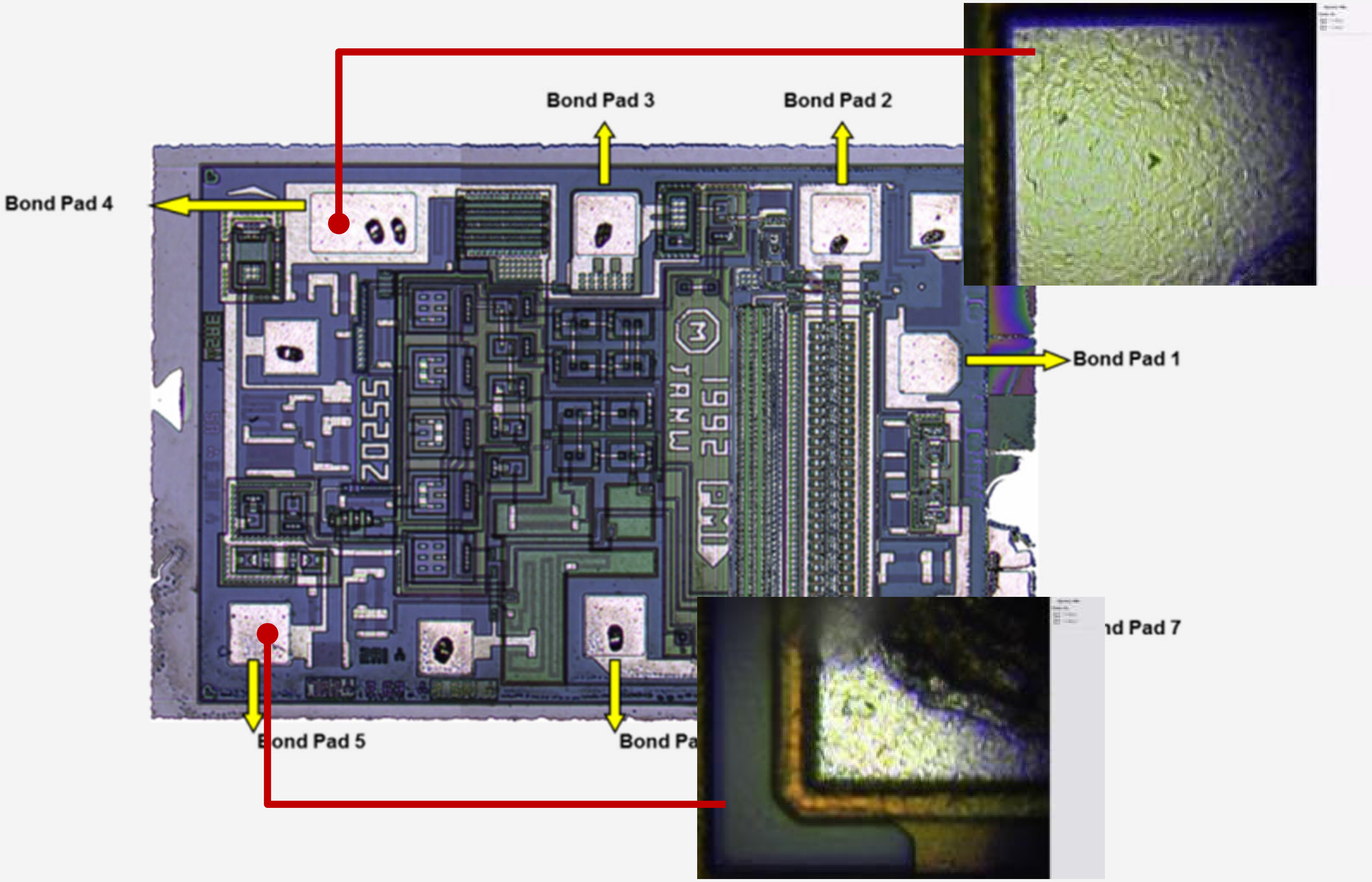
DIP

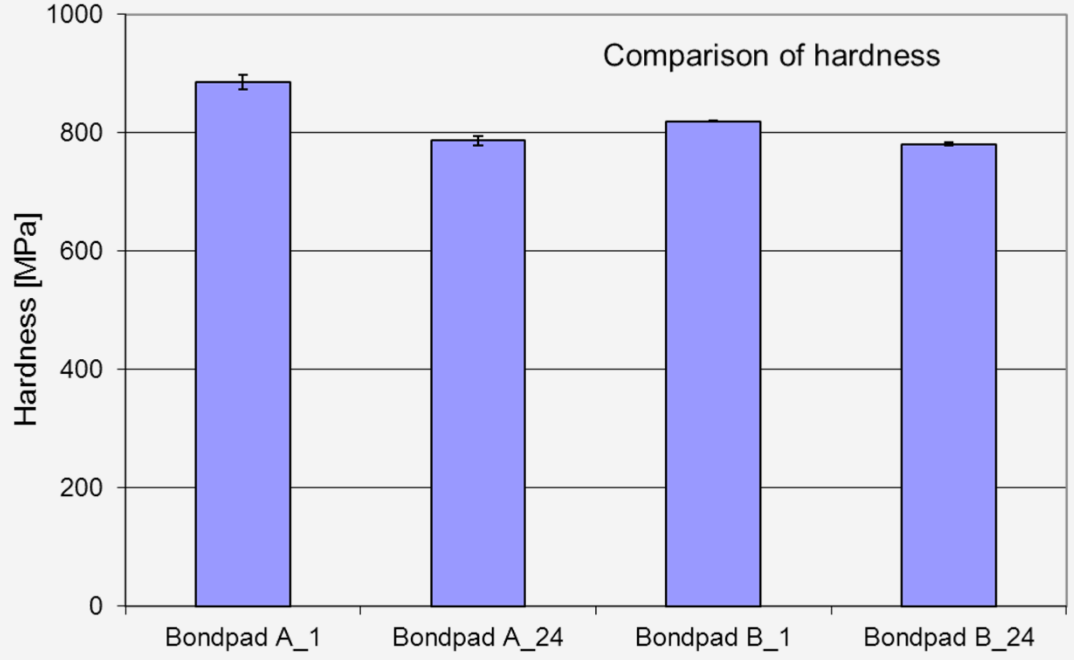




Hardness of bonding pads

- Characterization of mechanical properties on a microscale
- Nanoindentation Tester: very local indentations
- Miniature bond pads: Ultra Nanoindentation Tester





Hardness of different bond pads on a PCB

Nanoindentation helps in deciding whether the bond pad have good structure and composition

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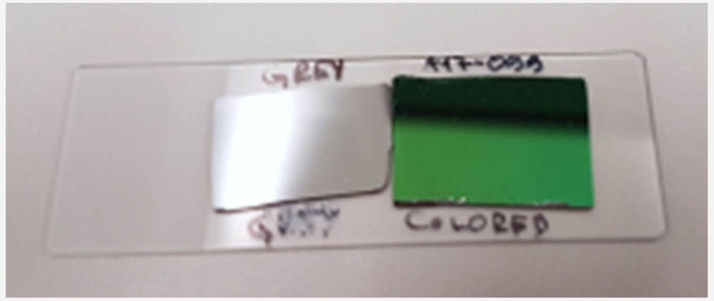
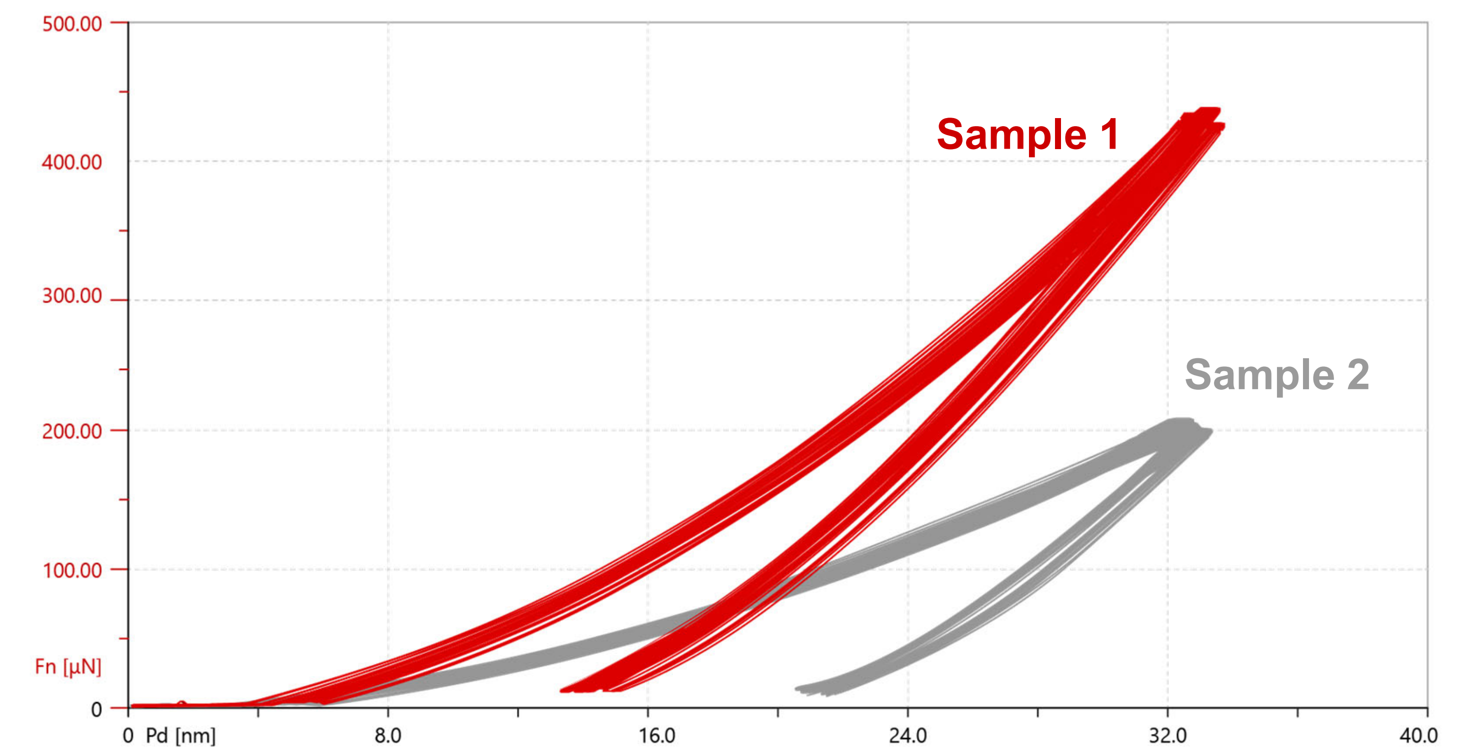
Hardness and elastic modulus of Si_3N_4 layer

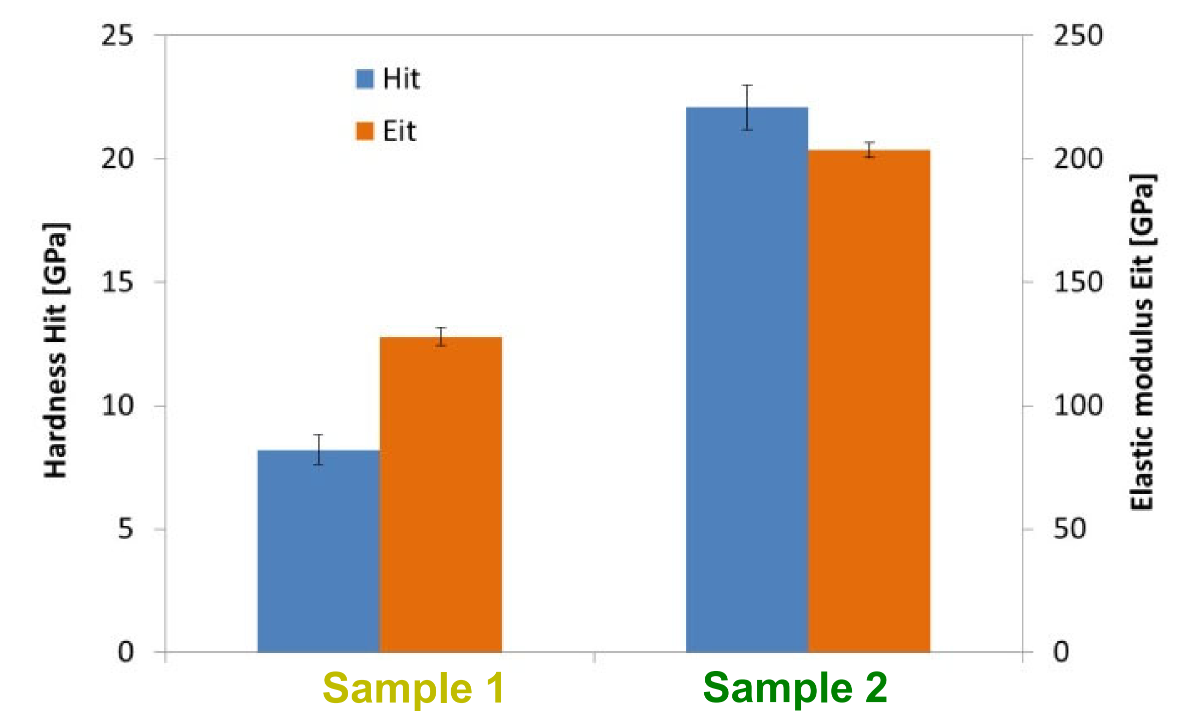
Ultra Nanoindentation tests for mechanical properties

- Two samples with Si_3N_4 layer: different methods/parameters of deposition
- Coating thickness 300 nm
- **Maximum depth ~33 nm for a force applied of ~ 400 µN**
- Clear difference in hardness and elastic modulus: Sample 1 harder than Sample 2





Comparison of indentation curves obtained on Sample 1 and Sample 2



Comparison of hardness and elastic modulus of both samples

AK1

Nano Scratch and Ultra Nanoindentation Testers

Mechanical properties of thin films

Nano Scratch Tester for adhesion and scratch resistance of thin films:

Fast response time and high precision on small forces applied (µN force range)

Ultra Nanoindentation Tester for hardness and elastic modulus of thin films:

From low loads (10 µN) to higher loads (up to 100 mN), the best metrological nanoindenter at low forces

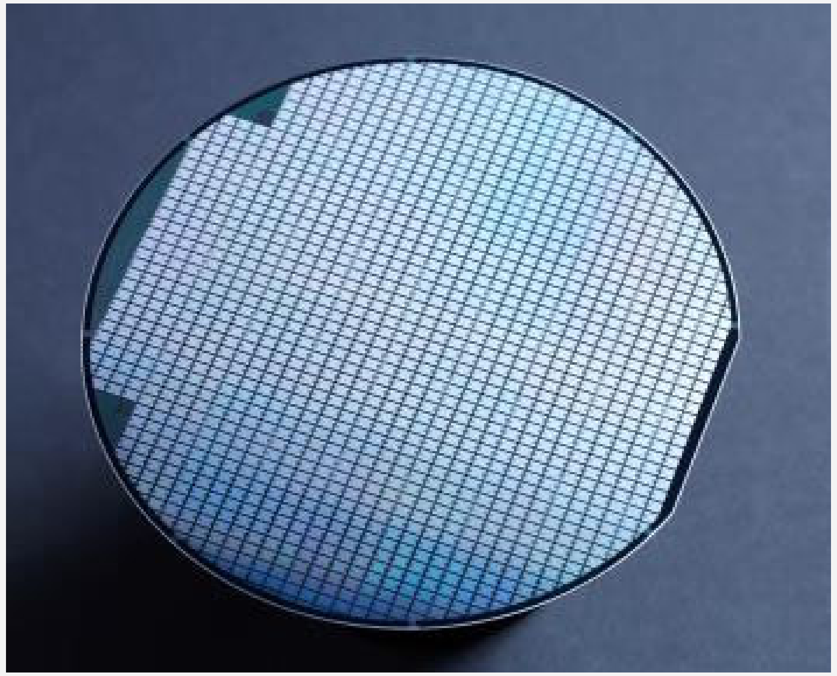
Slide 36

AK1 Propose reordering ahead of Slide 33?
Aleia Kim, 6/11/2021

Hardness & Adhesion of Si_3N_4 Layers on Si Wafer

Layers deposited on wafers and packaging of semiconductor components

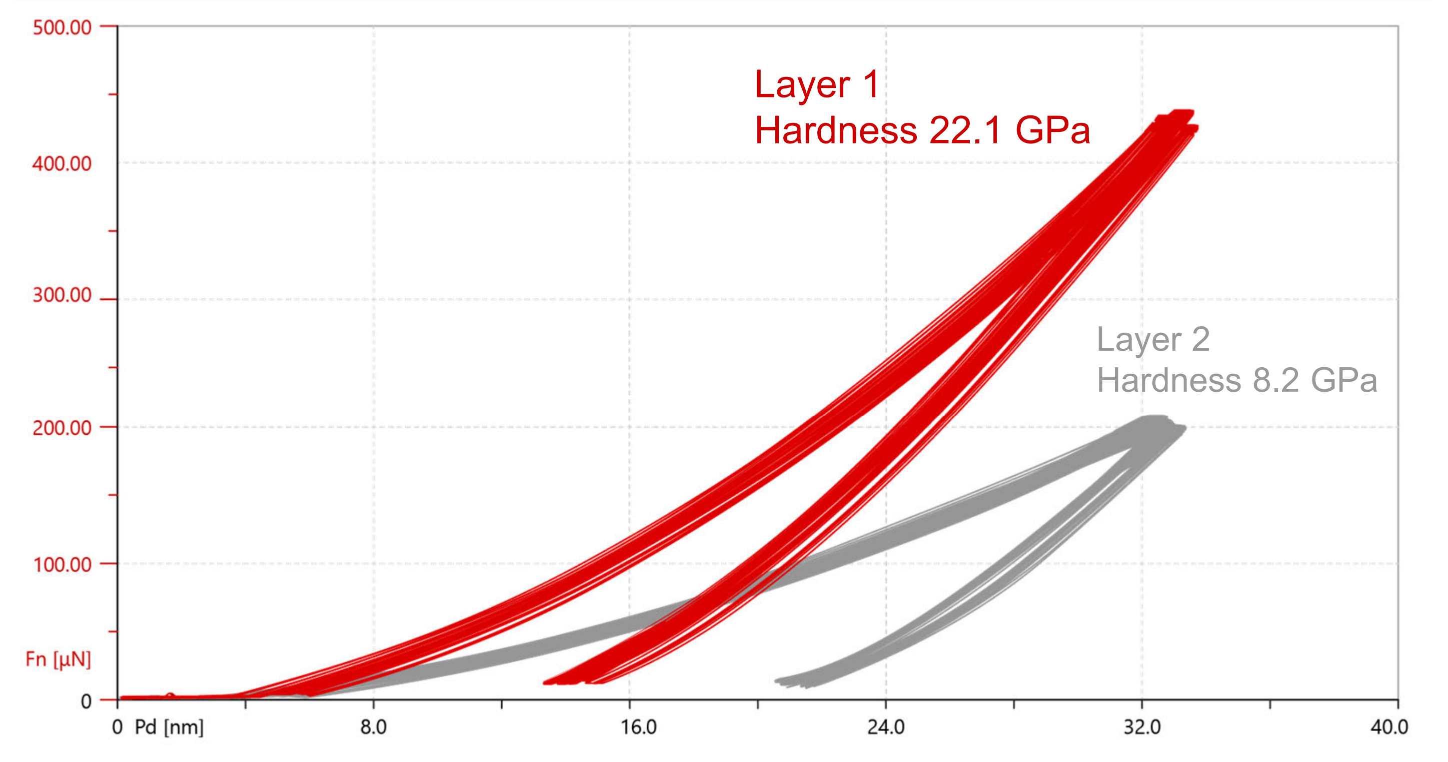
Silicon nitride: common layer, used for passivation or as a hard mask for etching



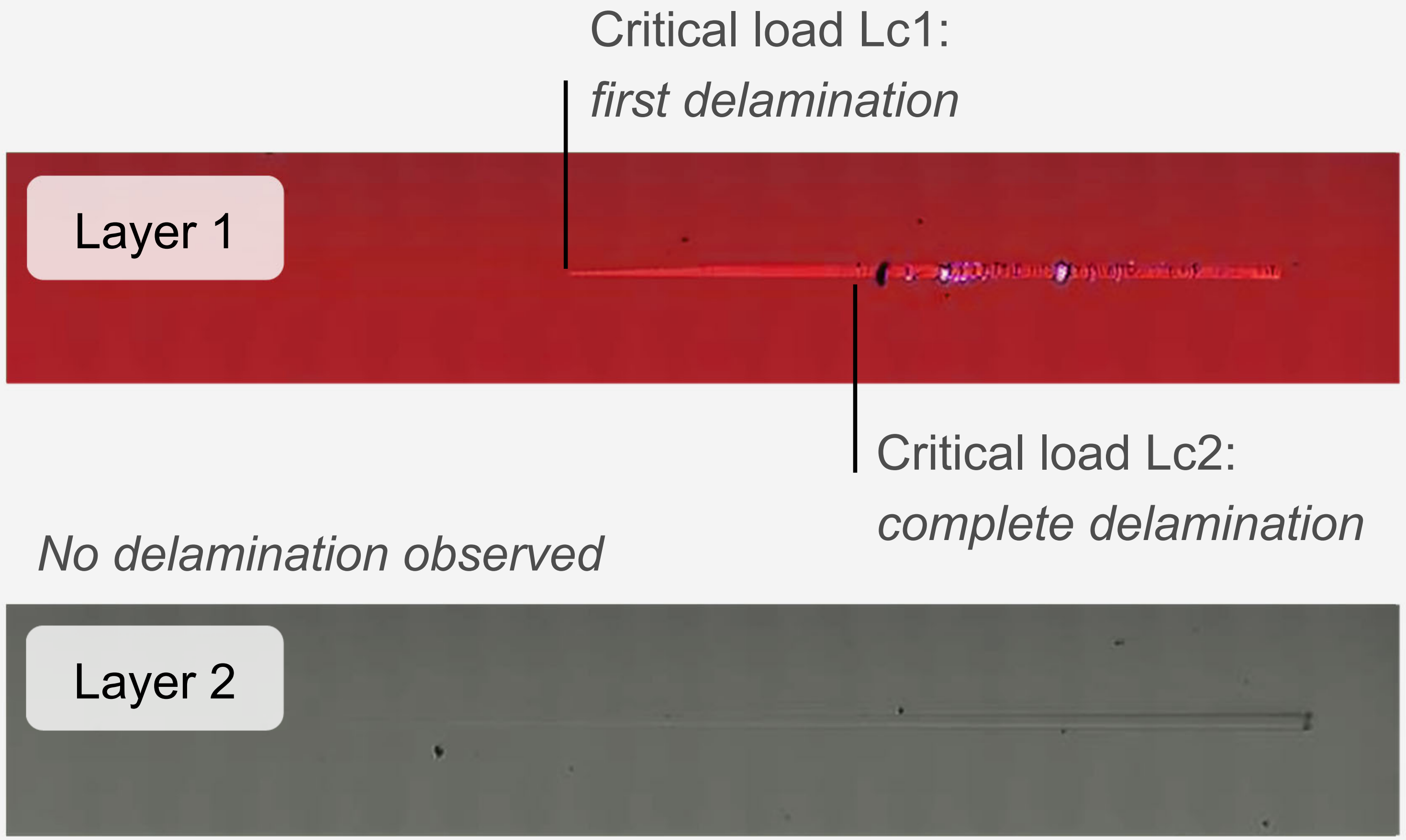
- Hardness of Si_3N_4 Layers deposited with a new deposition process is related to its durability
- Ultra low load nanoindentation
- Adhesion by nanoscratch testing

Hardness & Adhesion of Si_3N_4 Layers on Si Wafer

Indentation measurements:
Layer 1 is harder than Layer 2



Scratch measurements:
Layer 1 delaminated earlier than Layer 2

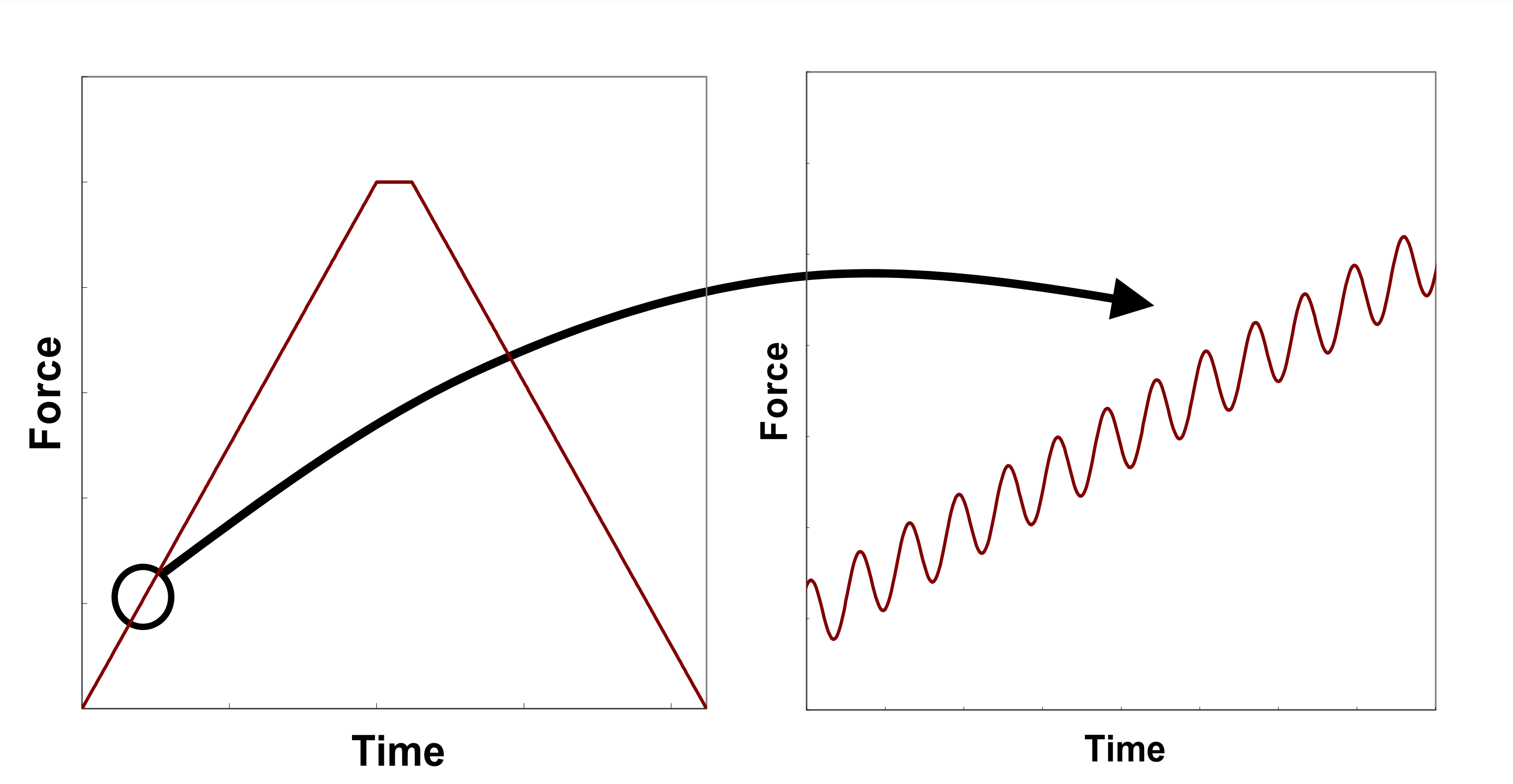


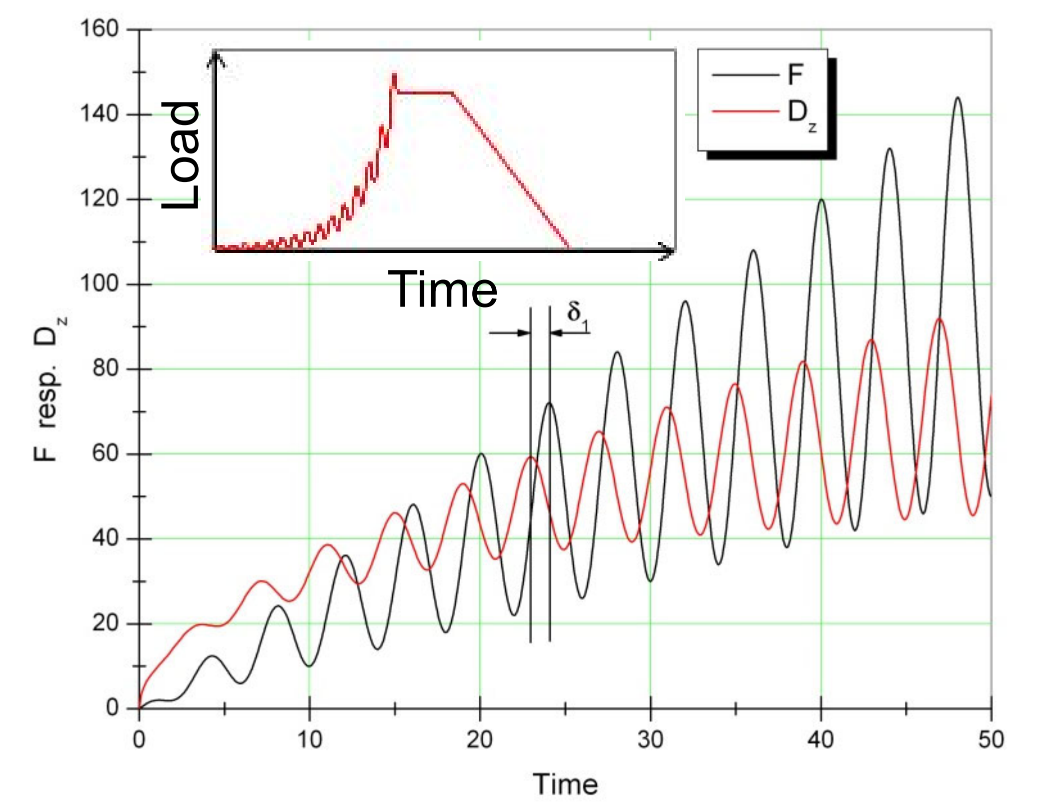
✓ Optimal layer should have hardness between the two layers and good adhesion

Dynamic Mechanical Analysis (DMA)

Depth profiling with Sinus mode measurements

- Small oscillations in a sinus wave are superimposed to the increasing force
- It allows a determination of the mechanical properties as a function of depth in materials



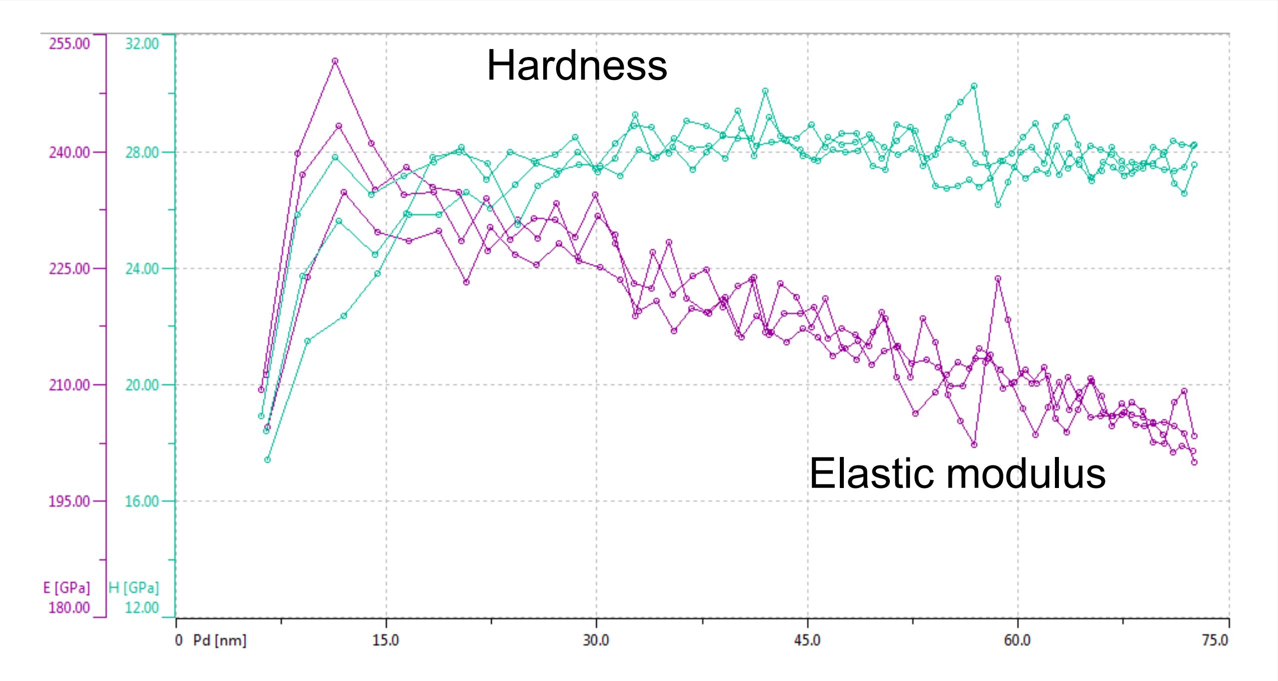


Hardness and elastic modulus of Si_3N_4 layer

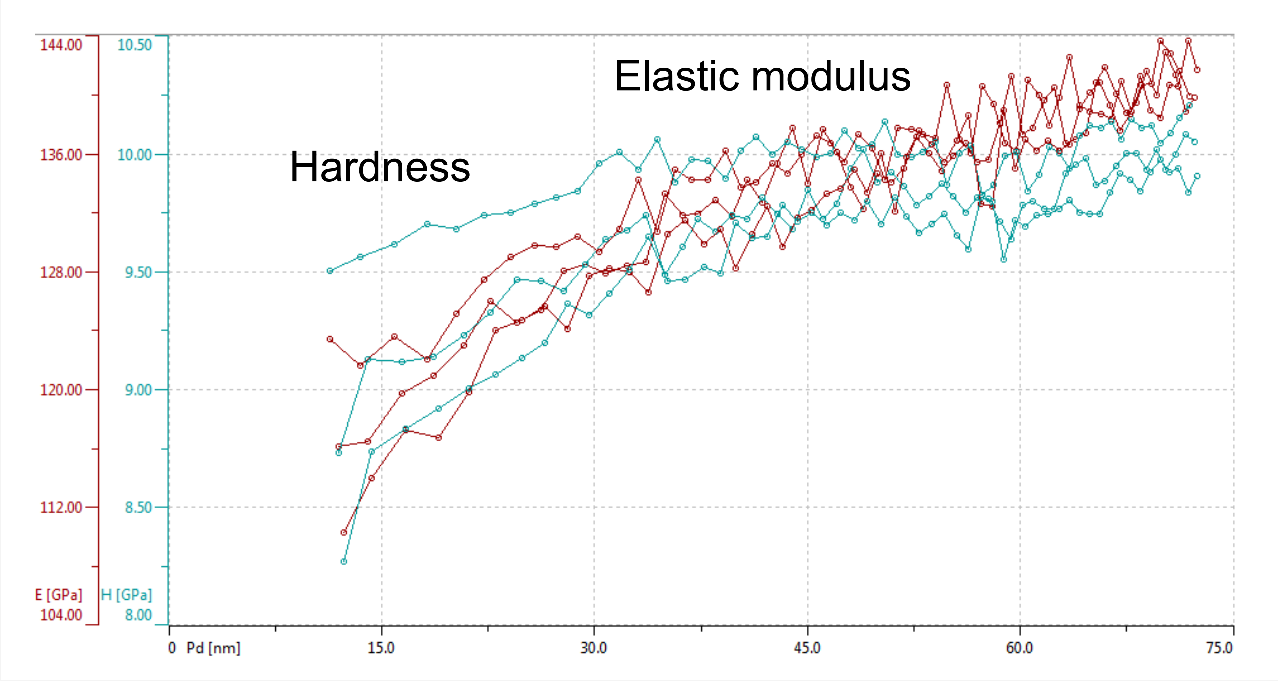
Ultra Nanoindentation tests for mechanical properties

Sinus mode for depth profile of hardness and elastic modulus

- Maximum load 2 mN (depth ~75 nm), frequency 10 Hz
- Variations of hardness and elastic modulus are different between both samples

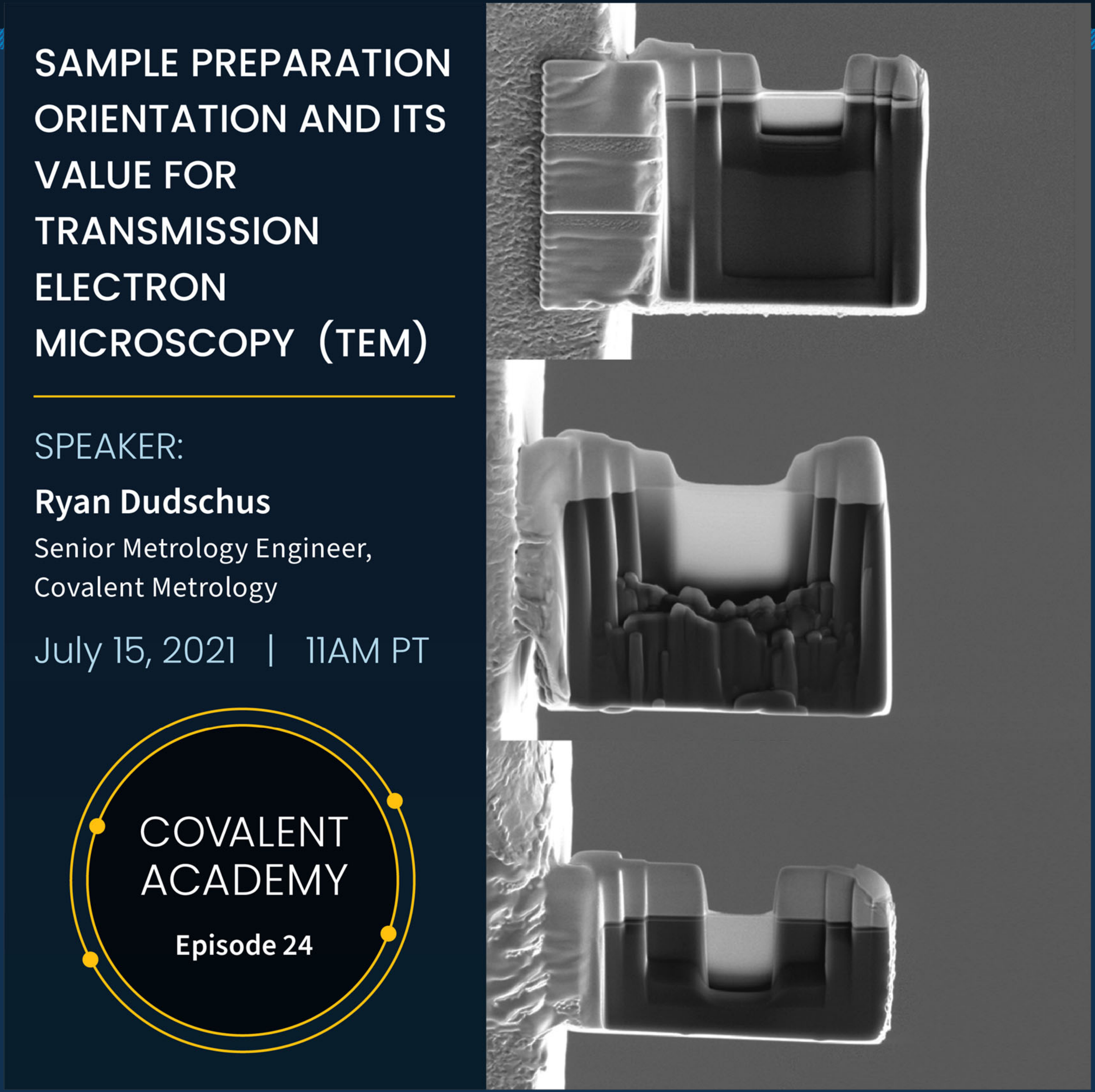


Sinus measurement (depth profile) on Sample 1



Sinus measurement (depth profile) on Sample 2

Coming Up…



SAMPLE PREPARATION ORIENTATION AND ITS VALUE FOR TRANSMISSION ELECTRON MICROSCOPY (TEM)

SPEAKER:

Ryan Dudschus

Senior Metrology Engineer,
Covalent Metrology

July 15, 2021 | 11AM PT

COVALENT ACADEMY

Episode 24

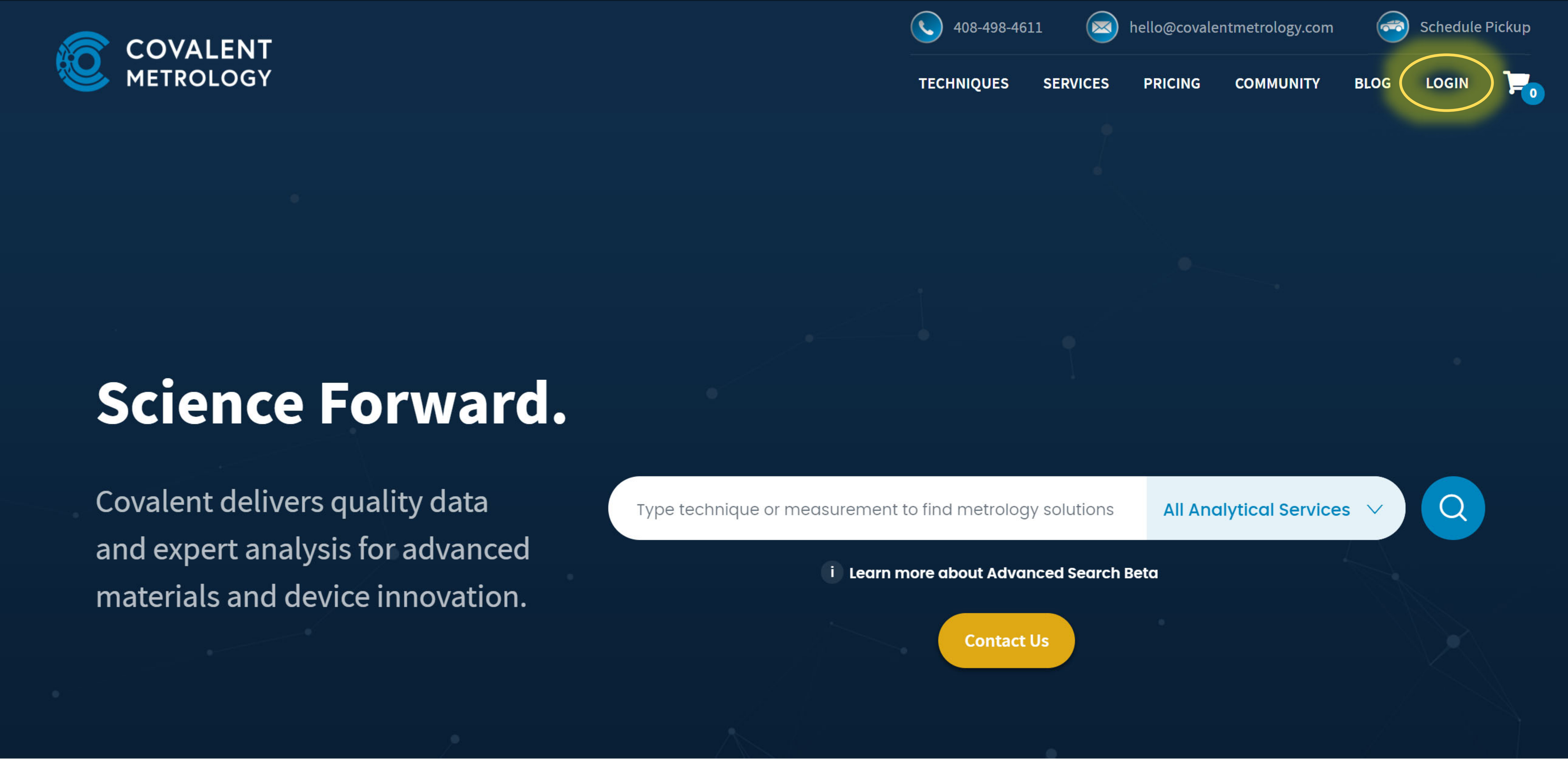
Survey and Sign-Up for Calendly Appointments

Want to learn more about Covalent's
Nanoindentation and Nanomechanical Testing Services?

Talk with a Covalent Expert!

Schedule your Appointment Now with Calendly
- link is in the chat -

Covalent Community



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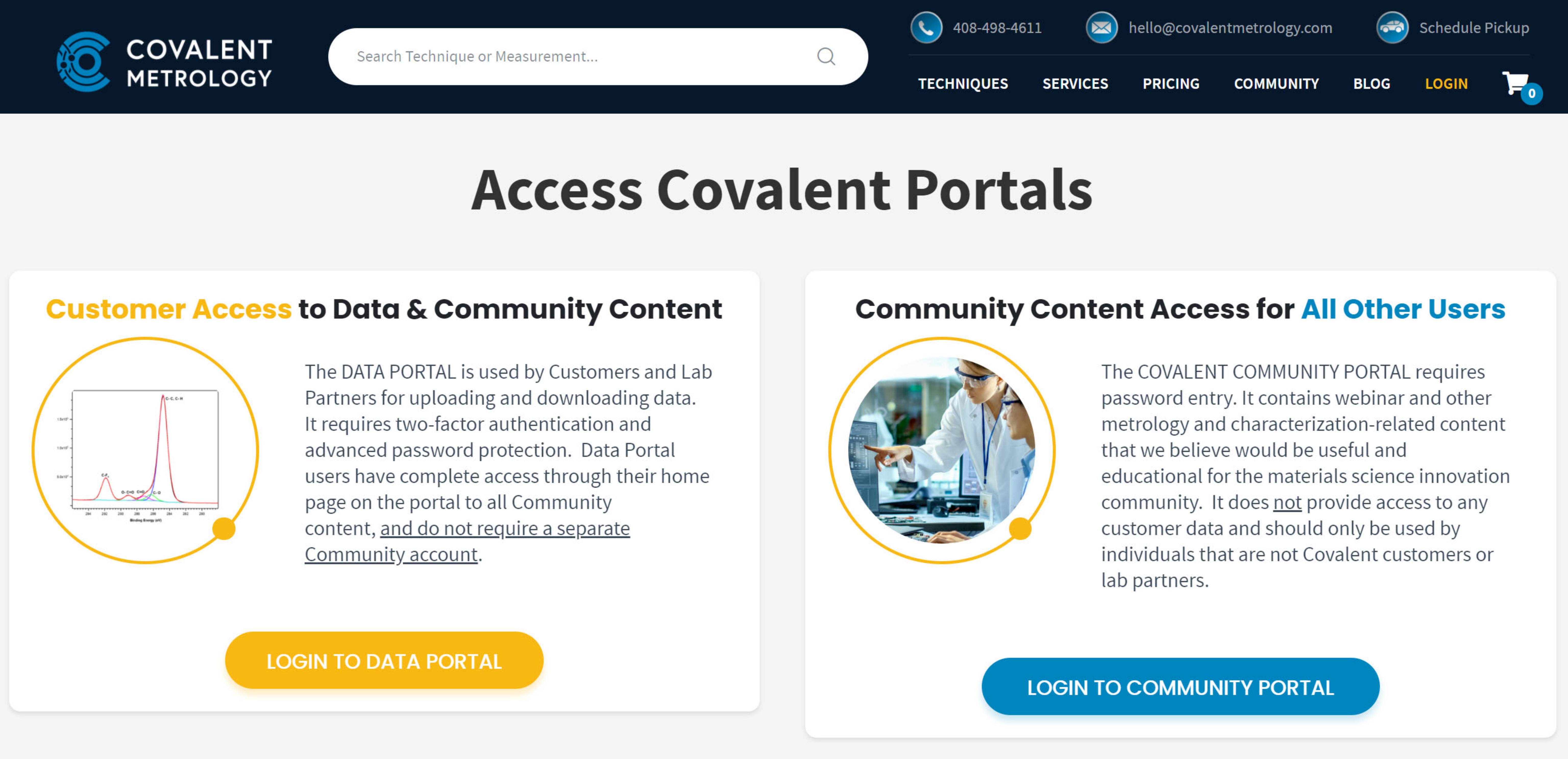
Covalent Community



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Q & A
Session

COVALENT METROLOGY

Thank you