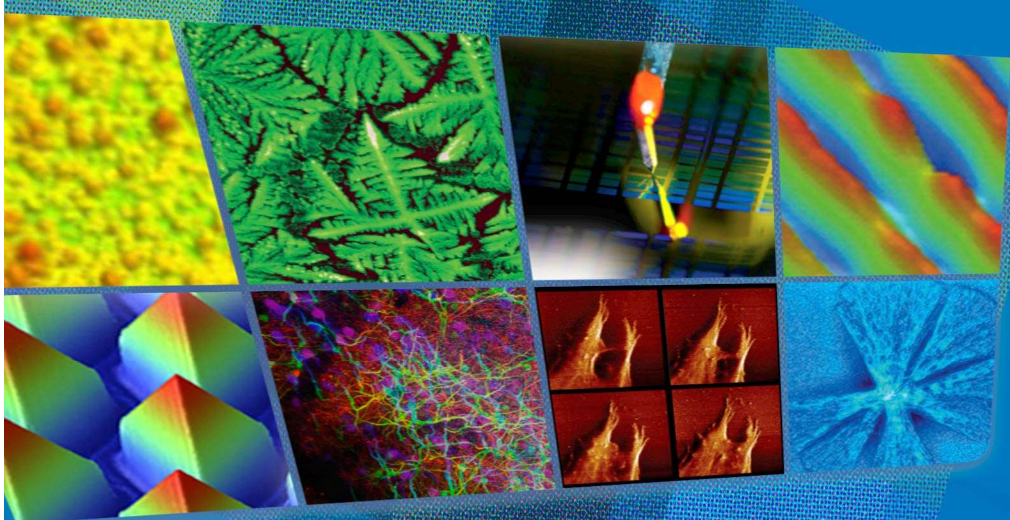
Bruker (Hysitron) Nano Mechanical Technology



Atomic Force Microscopy
3D Optical Microscopy
Fluorescence Microscopy
Tribology
Stylus Profilometry
Nanoindentation

Jimmy Juan
2017 .3.28

Bruker Nano Surfaces Division

Innovation with Integrity

Bruker NI (Hysitron) Overview

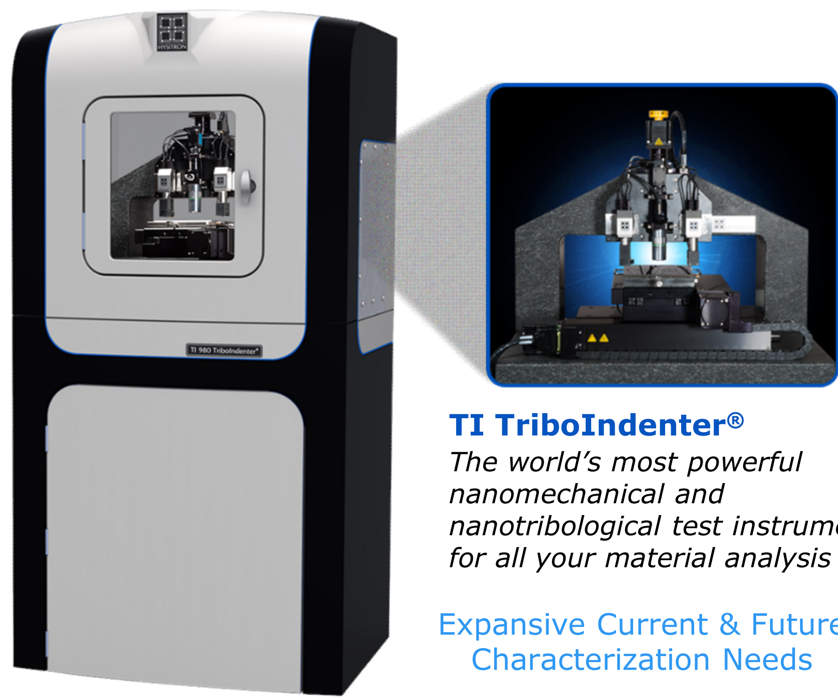
Founded: 1992
Headquarters: Minneapolis, MN USA

Hysitron Inc. Designs, Manufactures, and Services Leading Edge Nanomechanical Test Equipment for Materials Research, Development, and Process Monitoring

- 70+ employees headquartered in MN with extension applications laboratories in Berkeley CA, Germany, Czech Republic, India, China, Taiwan, and Japan
- World-Wide Representative and Distributor Network

Product Portfolio
Hysitron Stand-Alone Nanoindenter Platforms

Stand Alone Nanomechanical and Nanotribological Test Systems



TI TriboIndenter®
The world's most powerful nanomechanical and nanotribological test instrument for all your material analysis needs

Expansive Current & Future Characterization Needs

TI Premier™
Compact instrument platform configured for dedicated characterization needs

Dedicated Characterization Needs

Product Portfolio
Hysitron Process Metrology

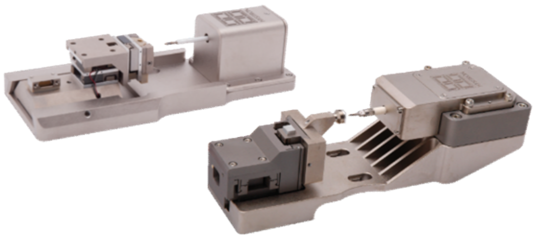
Fully Automated and Semi Automated Nanomechanical Metrology Systems for In-Line and Near-Line Process Monitoring of Thin Film Mechanical Properties and Interfacial Adhesion

ATI 8800

TI 950A

Product Portfolio
Hysitron Microscopy Products

Scanning Electron Microscopes (SEM)



PI 85L/88 SEM PicoIndenter®

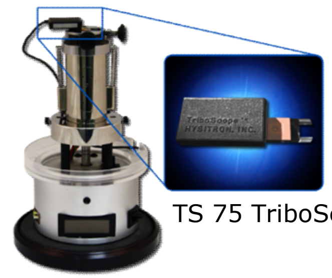
Transmission Electron Microscopes (TEM)

PI 95 TEM PicoIndenter®

X-Ray Microscopes (XRM) Beamline/Synchrotron Sources

IntraSpect™ 360

Atomic Force Microscopes (AFM)



TS 75 TriboScope®

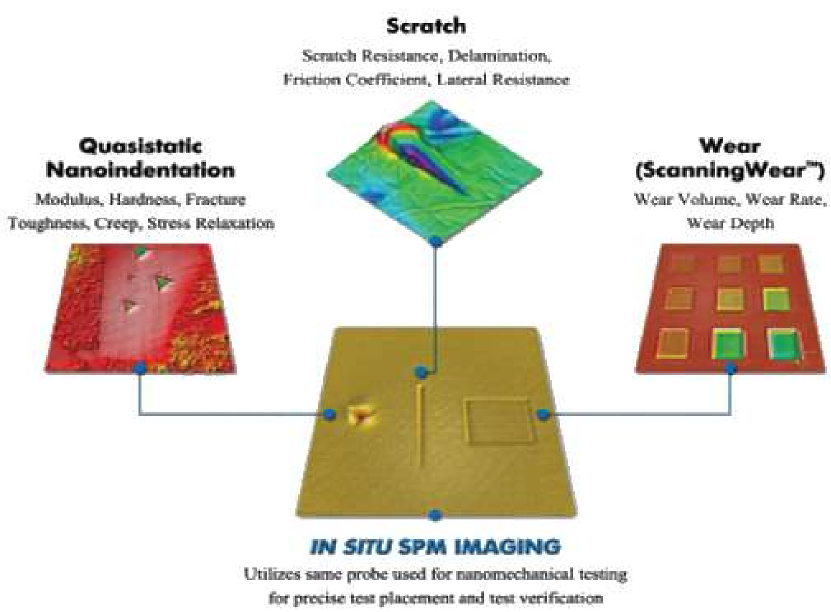
Inverted Optical Microscopes

BioSoft™

Delivering Quantitative Nanomechanics to the World of Microscopy

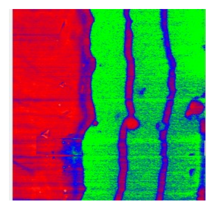
Hysitron Nanomechanical Techniques

Basic Indentation Techniques:



Positioning accuracy of +/- 10 nm when using in-situ SPM

Advanced SPM Imaging-based Techniques:



Modulus Mapping

Friction Imaging

Conductive Imaging

Other Novel & Combinatory Techniques:

- Dynamic mechanical analysis (nanoDMA®, CMX)
- Acoustic emission monitoring
- Heating/cooling: -150°C to >800°C
  Humidity control to 80% R.H.
- Electrical characterisation
- Fluorescence microscopy
- Co-localised Raman spectroscopy
- Testing in electrochemistry (EC) environment

Summary

In-Situ Technology

Hybrid Technology

HYSITRON®

Mechanical

- Nanoindentation
- NanoScratch Microscope
- Nano Wear

XCT | SEM | TEM | SPM

Nano Surface
- SPM
- XProbe
- Optical Microscope

Electrical
- Nano ECR
- EPTP

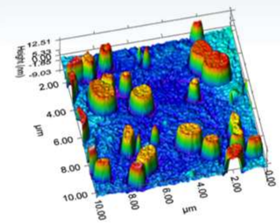
Environmental
- Xsol Stage 800
- Cooling
- Humidity

Chemical
- EC Cell
- Raman

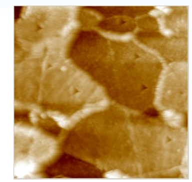
Optical
- Raman Spectroscope
- Fluorescence Microscope
- Optical Microscope

Example Applications

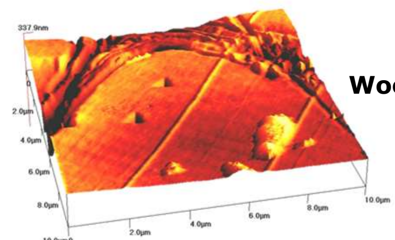
- Ceramics
  - Protective coatings, ceramic composites, tribofilms
- Metals
  - Individual grains, metal films, recording media
- Polymers
  - Low-k materials, polymer coatings, elastomers
- Biological/Biomaterials
  - Bones/teeth, tissue, implantable devices
- MEMS Devices
  - Actuation force, stiffness, deflection range



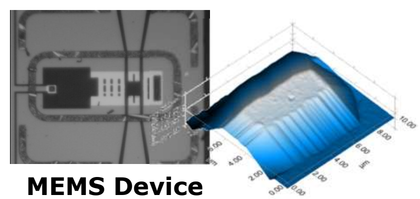
DLC Film on AlTiC Phases



Individual Metal Grains



Wood Cell

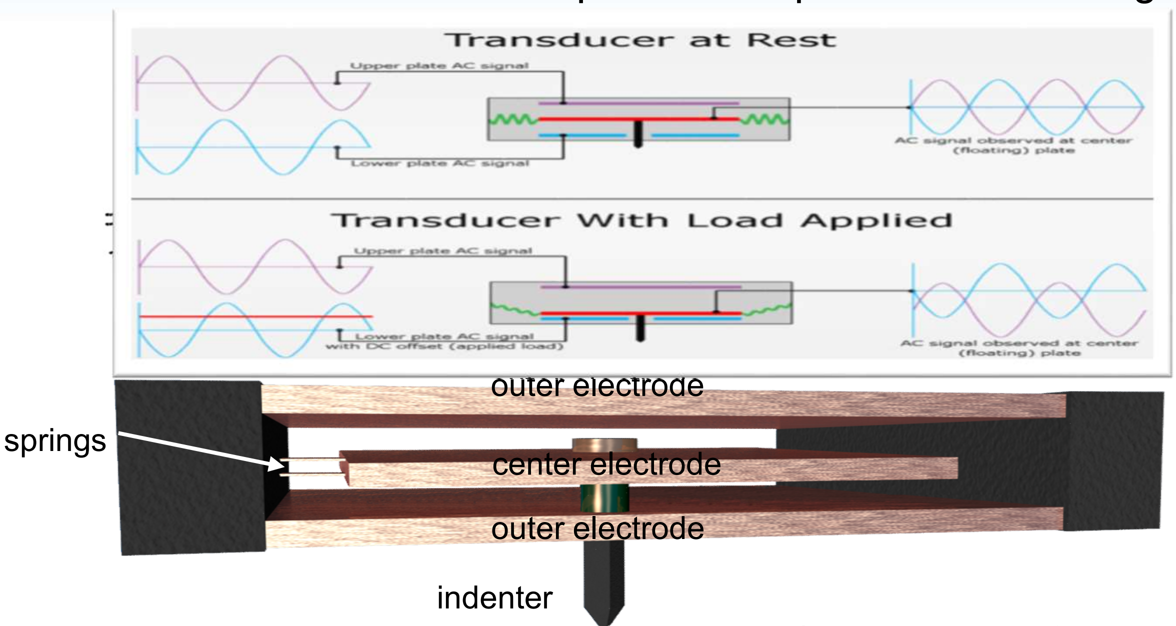


MEMS Device

Red Blood Cell

Capacitive Transducer

Electrostatic Actuation / Capacitive Displacement Sensing



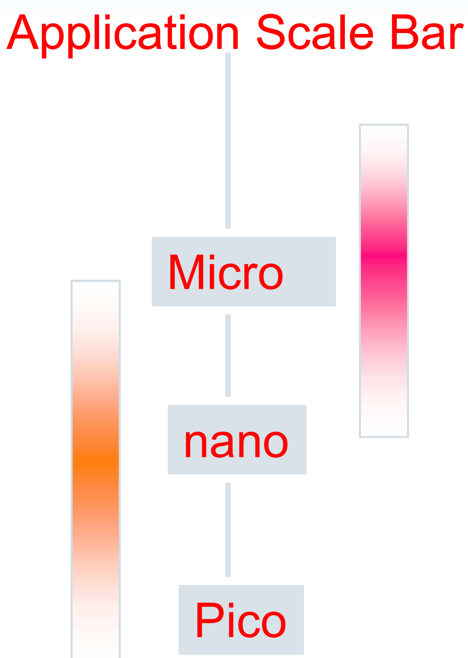
<0.05 nm/sec thermal drift

- ***Specs guaranteed on-site***

- **The outer 2 electrodes are fixed in space with AC signals 180 degrees out of phase applied**
- **Offsetting AC signals set up an electric field potential between the plates (0 at center)**
- **DC offset voltage is applied on outer plates, which electro-statically drives tip up or down**
- **Capacitive field is calibrated to force and displacement**
- **Hysitron has patented electro-static actuation, which leads to very low heat generation and low thermal drift**

US Patent #s: 5553486, 5576483, 5661235, 5869751, 6026677

Two Heads Designed for Best Performance

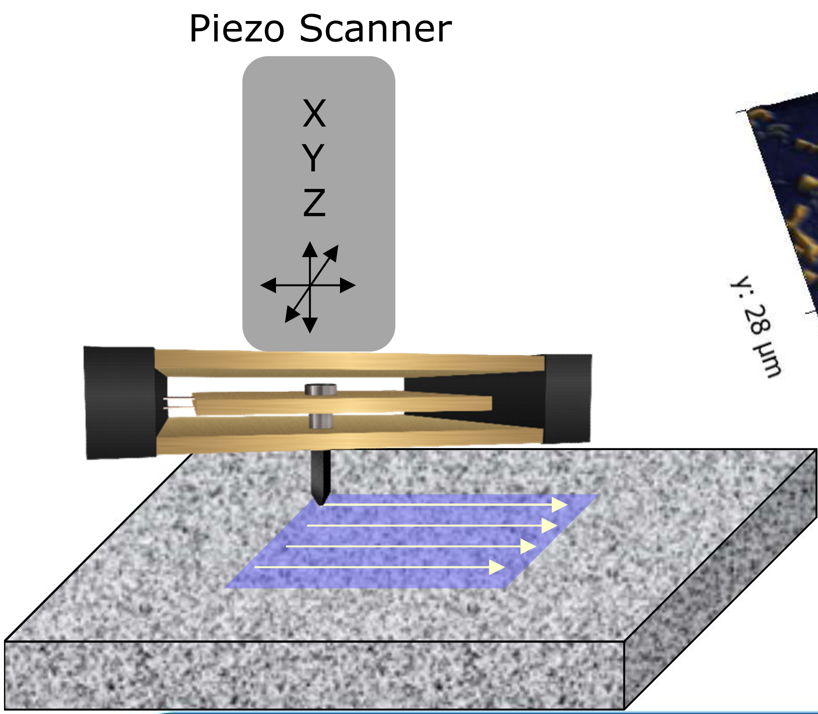


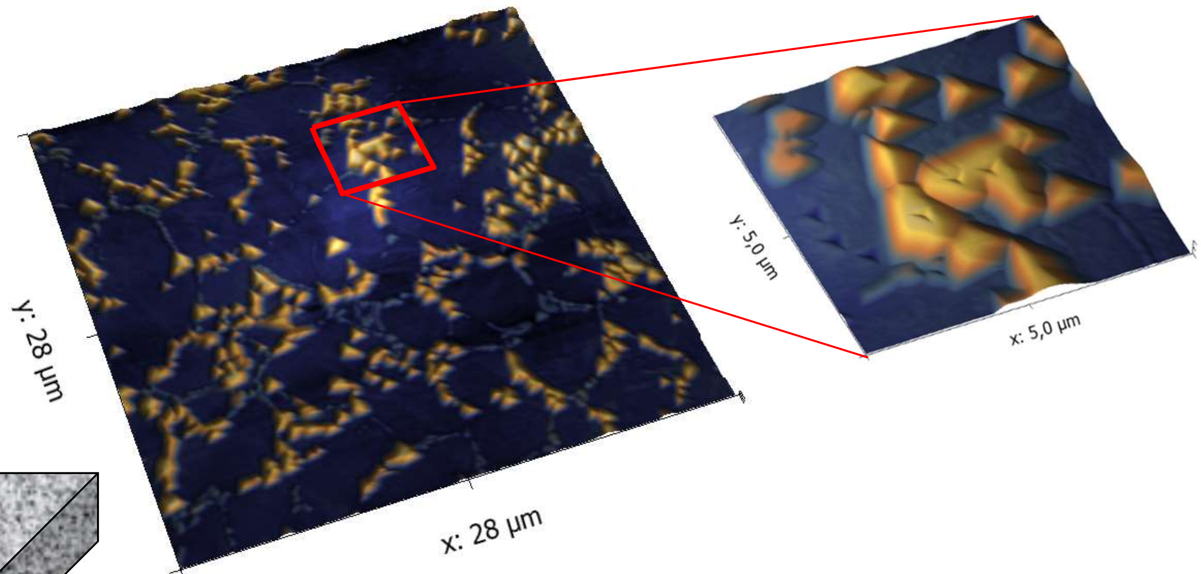
10mN Transducer
- True nano-indentation
- Max. Force: 10 or 30mN
- Force Noise Floor: 20nN
- Displacement Noise Floor: 0.1nm

3D Omniprobe
- Scale Connectivity; nano to micro
- Max. Force:500mN or 10N
- Force Noise Floor: 10μN – dep. on Fmax
- Displacement Noise Floor: 0.2nm

In-Situ SPM for Targeting Indents
Steel Sample with Precipitate

Position indents within +/- 10nm

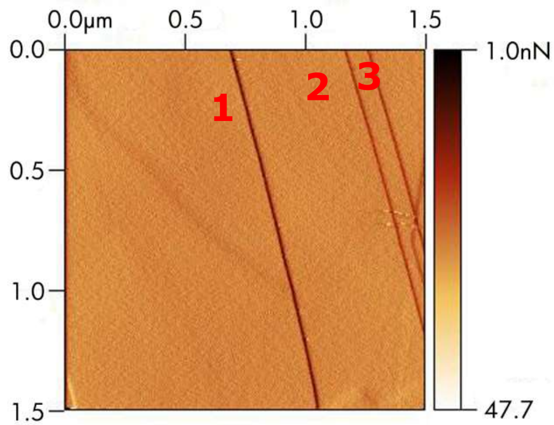


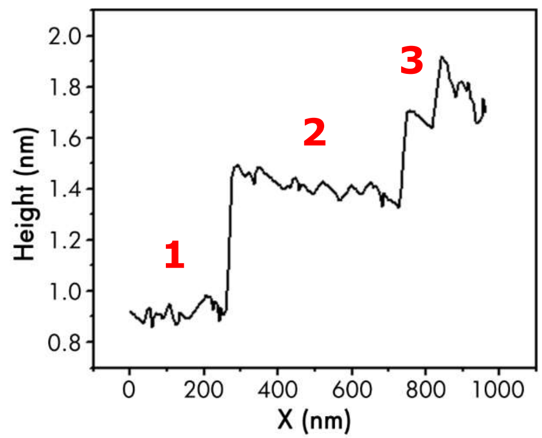


Residual indents clearly visible in post-test SPM image

xProbe
Low Force in-situ SPM Imaging

- Unparalleled sensitivity enables high resolution imaging and the ability to detect the height of **single atomic steps** of HOPG *(single layers = Graphene)*

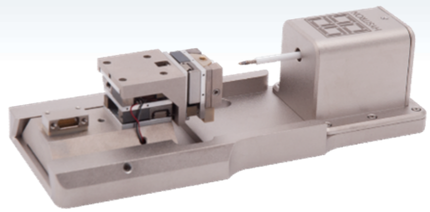


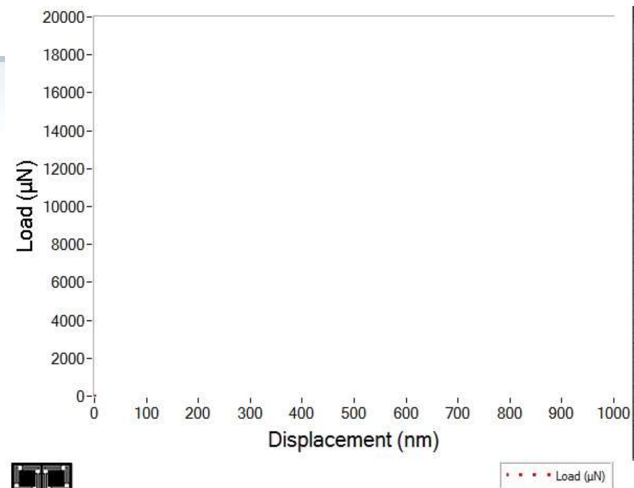


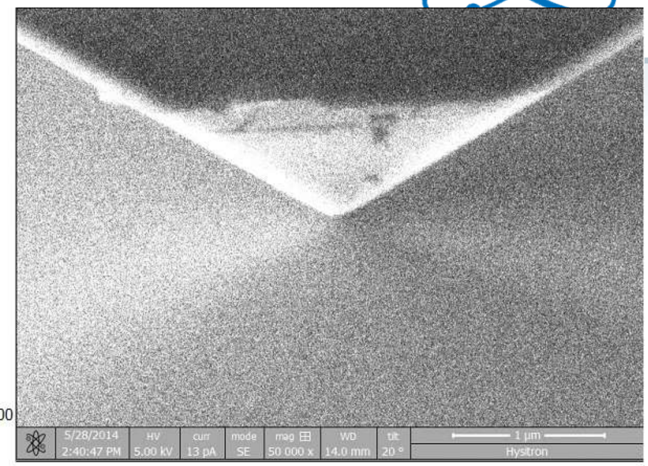
- A double layer step of HOPG followed by two single layer (graphene) steps is imaged at **5nN** with a **50nm** radius probe.
- Step heights determined from the line profile drawn across the steps in the graphene layers.

In Situ Indentation of Fracture Toughness of DLC film

BRUKER

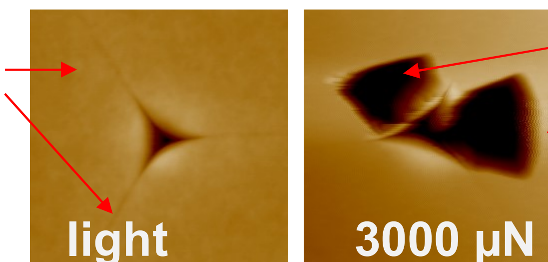






HYSITRON™

Cracking



Mechanical Data

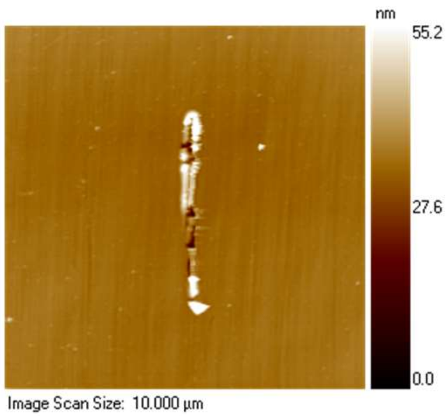
material failure

- Low K Film Fracture Toughness

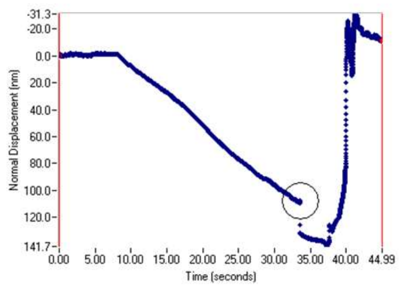
force.

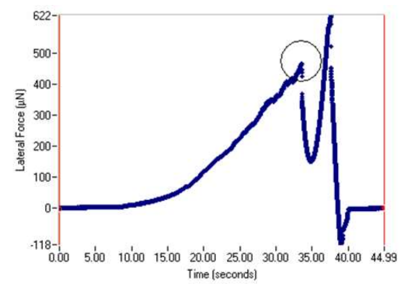
Interfacial Adhesion Characterization of a Thin Film

- Ramping force nanoscratch of a 150 nm titanium nitride thin film
- The loading profile is designed to induce breakthrough event or film delamination



Post-scratch in-situ SPM image for visual analysis and test site verification





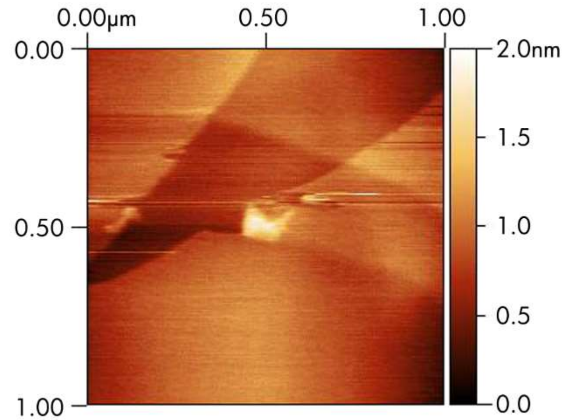
Distinct changes in curve profiles corresponding to breakthrough / delamination events → ***critical load (P_{crit})*** *and* ***critical depth (h_{crit})***

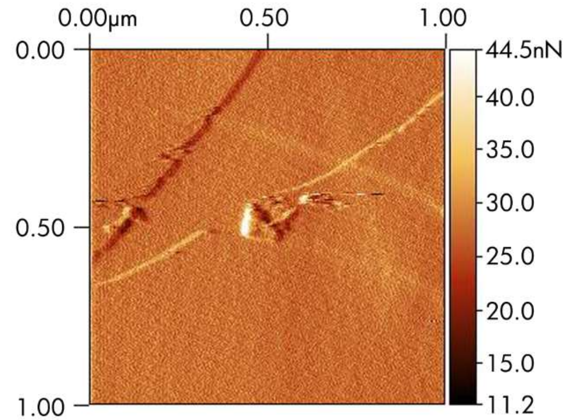
Scratch testing has been widely accepted as a way of evaluating **interfacial adhesion** of thin film/substrate systems. Failure events may be found where the probe produces delamination, debonding, cracking, fracture, or breakthrough at the film/substrate interface.

xProbe 2D

Low Force SPM Imaging - and scratch testing!

- 5 µN load horizontal scratch across a single atomic step of HOPG showing delaminating and tearing.

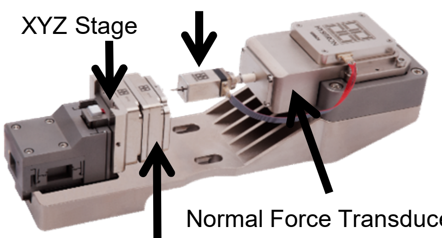


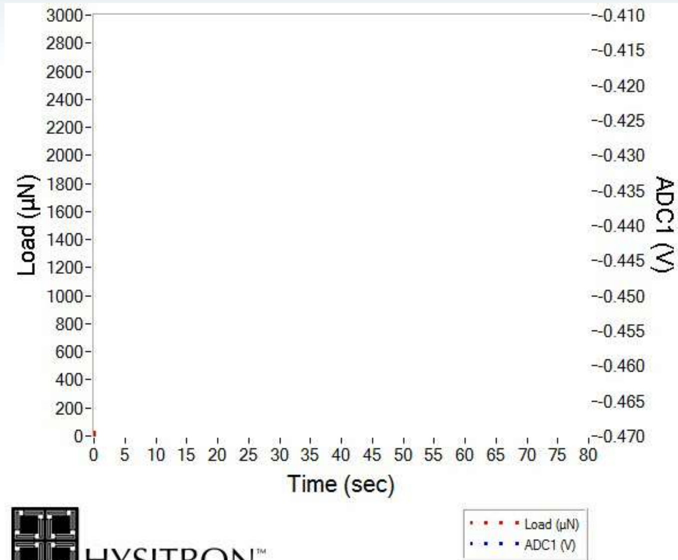


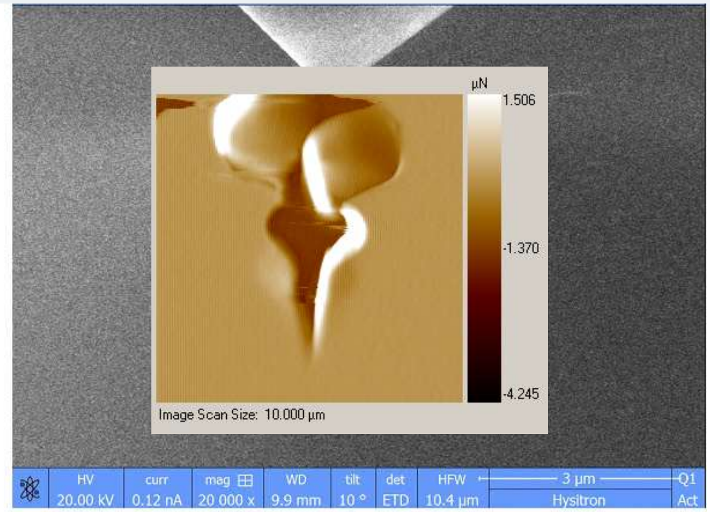
- The xProbe Transducer enables scratching of monolayer sheets of graphene with high resolution imaging to observe the delaminating and tearing of the monolayer.
- In order to delaminate the layers, a scratch across the horizontal center line was performed, while the load was increased from 5 nN to 10nN, 50nN, 100nN, 500nN, 1 µN etc.
- An in-situ SPM scan was performed to determine failure between each scratch iteration.
- **Failure of the interface was accompanied by the tearing of a single layer at 5 µN load in the Z-direction.**

In-Situ Nano Scratch in SEM

Delamination and failure in a low-k dielectric film







HYSITRON™

Specifications

- Lateral actuation distance: 30 µm
- Maximum lateral force: 30 mN
- Lateral force noise floor: 3 µN

Correlation with SPM Image and Force Displacement with ATI8800 or TI 950

Friction Experiment

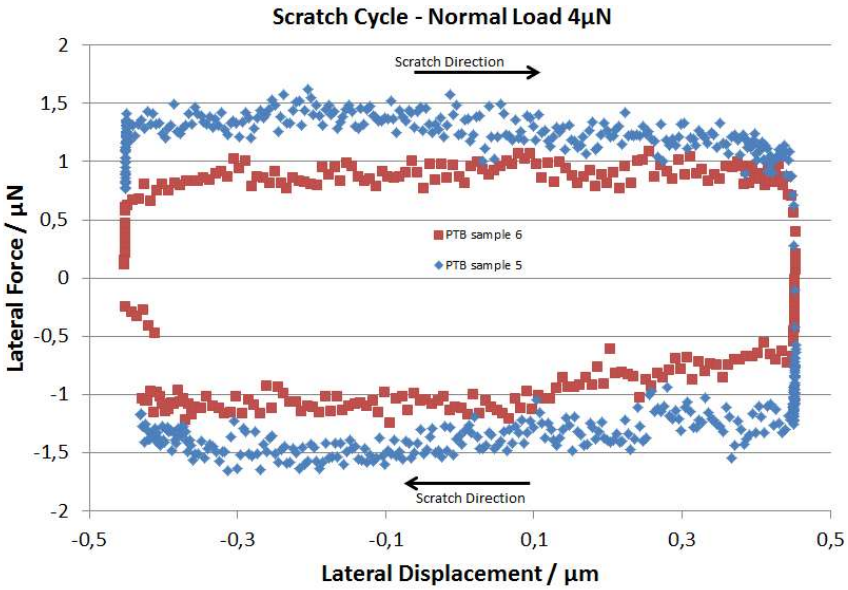
Scratch Cycle on Two Polymers

- Normal Load 4μN
- Lateral force 1μN

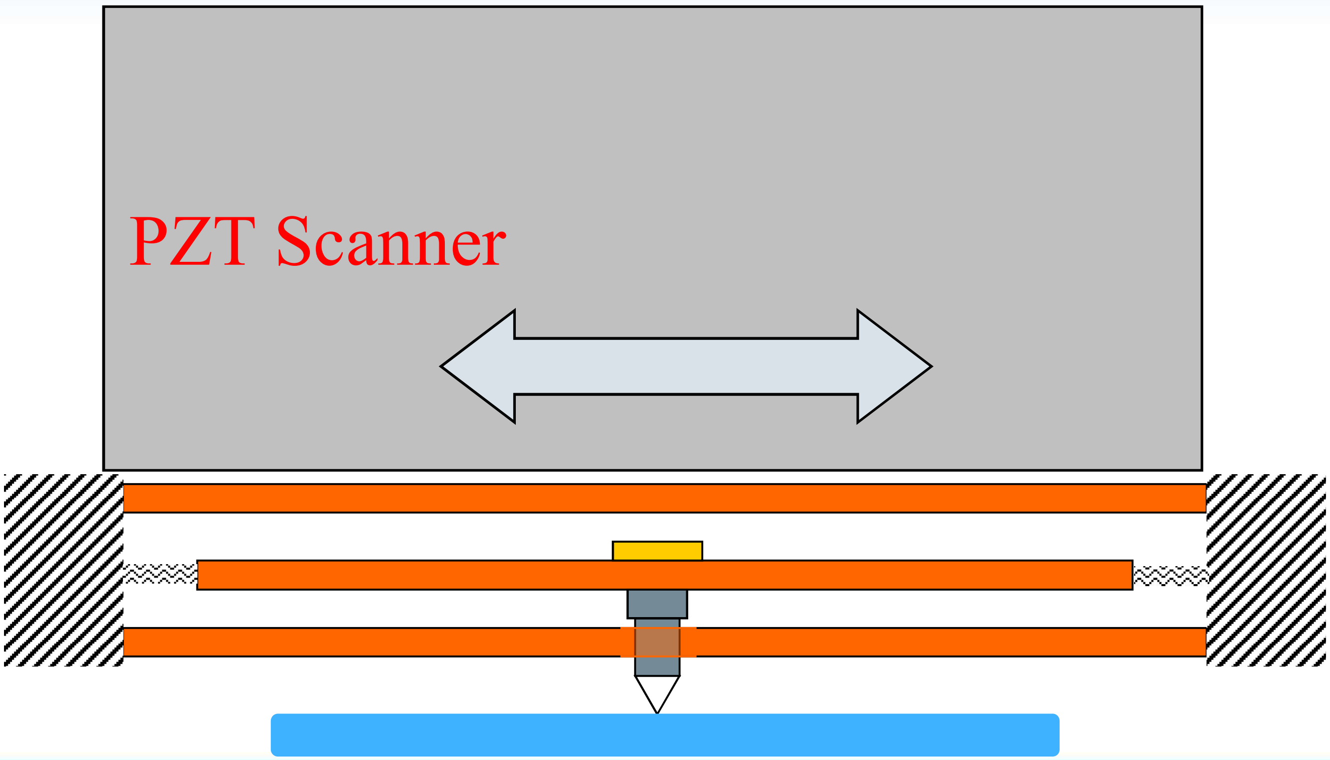
Differences in Friction can be Detected

- The lateral displacement ranges from a few nm to 15μm.
- Fretting experiments at the nanoscale are also possible.

10mN Transducer 2D

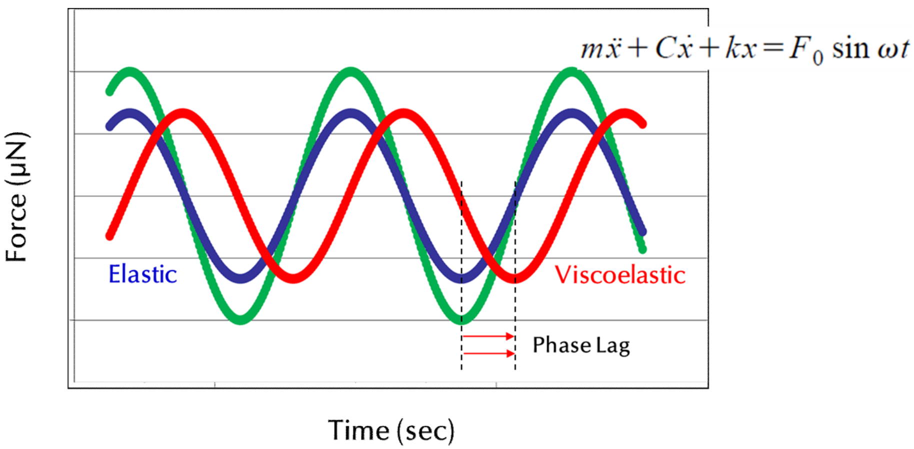


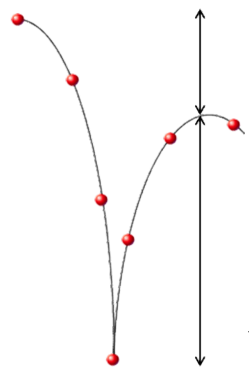
Nano Wear™



nanoDMA® III

The Next-Generation of Dynamic Nanoscale Mechanical Property Characterisation



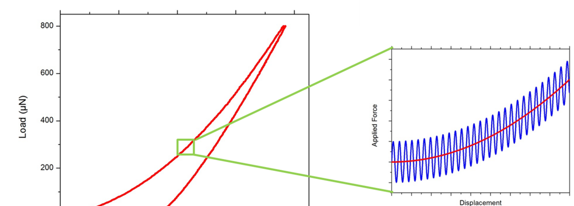


$$E_r'' = \frac{\omega C_s \sqrt{\pi}}{2\sqrt{A_c}}$$ *Energy loss*

$$E_r' = \frac{k_s \sqrt{\pi}}{2\sqrt{A_c}}$$ *Elastic response*

$$\tan\delta = \frac{E_r''}{E_r'} = \frac{\omega C_s}{k_s}$$ *Damping*

- Dynamic Measurement of Hardness, Modulus, Storage Modulus, Loss Modulus, Tan Delta
- Continuously measure properties as a function of contact depth, frequency and time

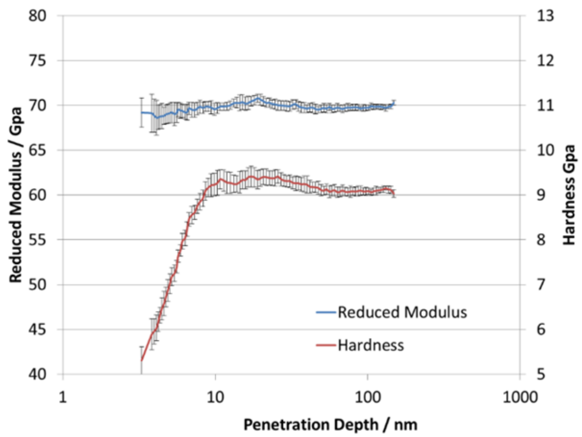


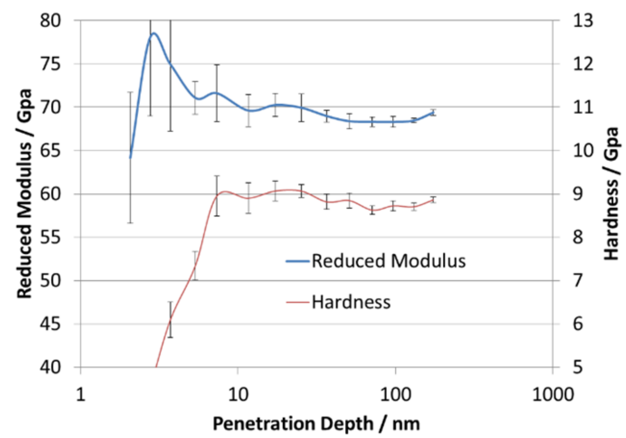
Dynamic force superimposed on quasi-static force

Fast Depth Profiling Using CMX

CMX - Continuous Measurement of X (X = Hardness, E', E", tanδ, etc.)

Continuously measure mechanical properties as a function of indentation depth, frequency, and time





CMX Test:

6 CMX curves, averaged at 100 different loads

Quasi-Static Test:

14 loads applied, average of 10 different measurements each

Wear + CMX

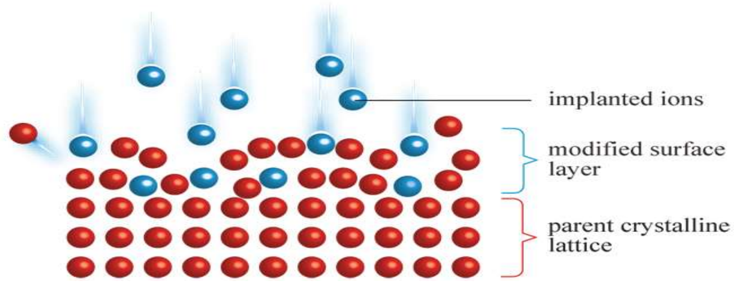
Tooling coating or Ion-Implanted Steel

- Implanting depth: ~50 nm
- Ions: Cr, Ti, N .....

Issues :

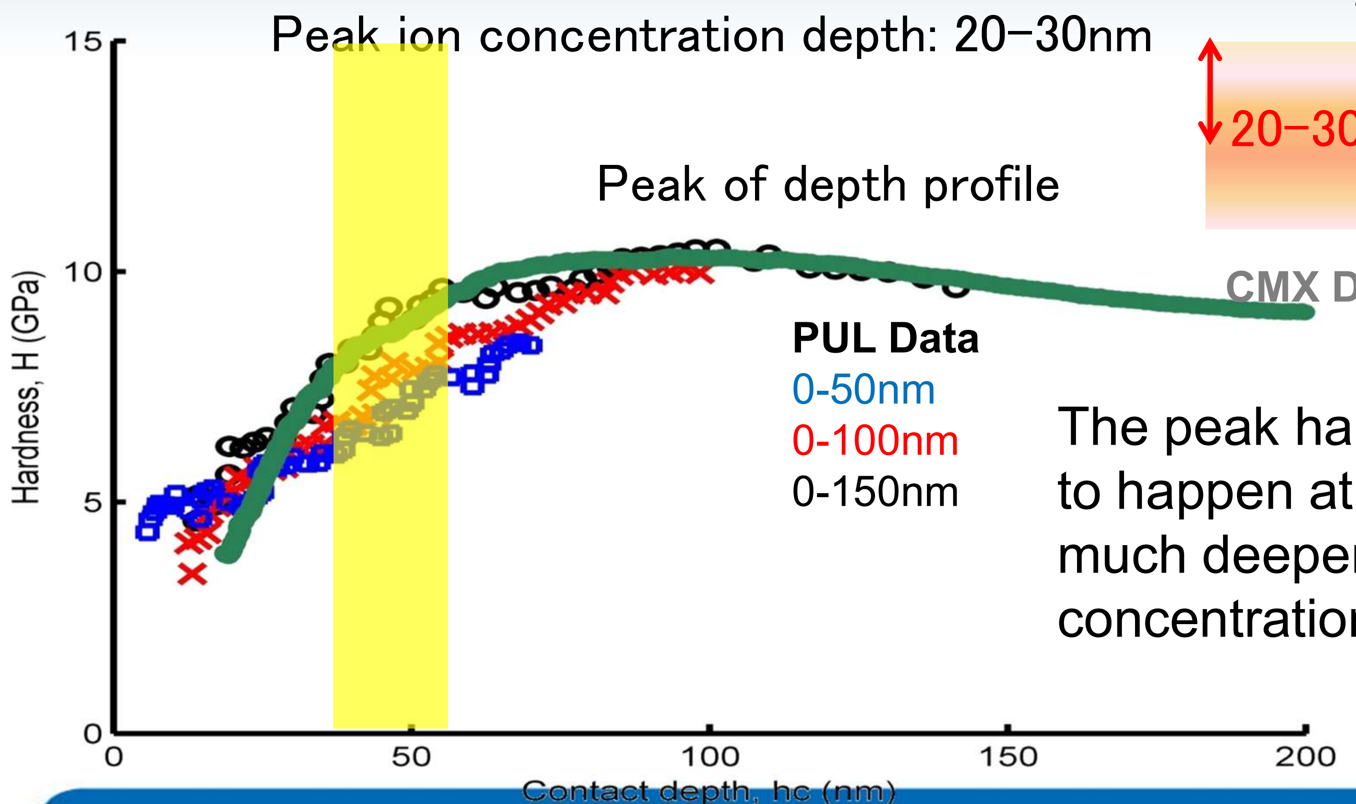
There is no interfaces of ion implanted sample

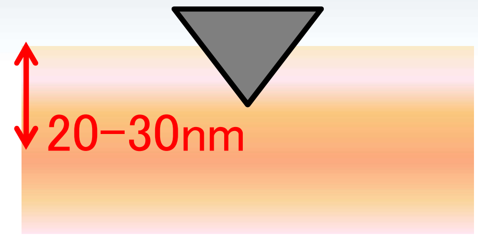
And Penetrate depth is too shallow



Ion-Implanted Steel

Depth Profiles



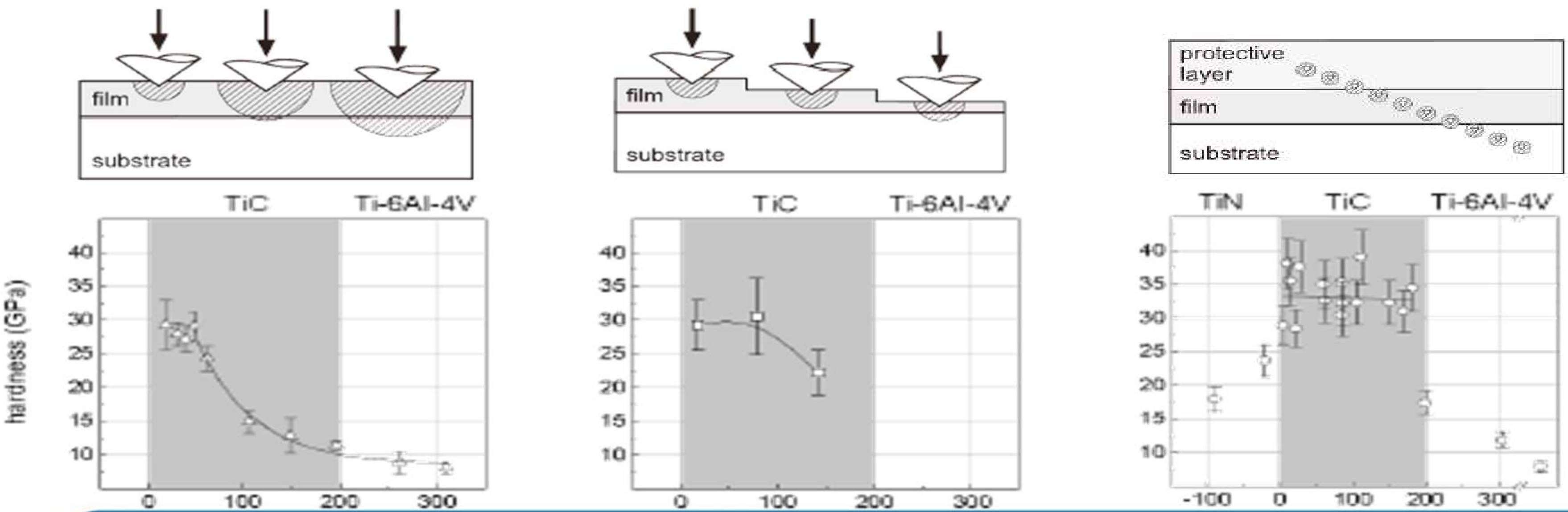


The peak hardness was found to happen at 70-80nm, which is much deeper than the peak ion concentration depth of 20-30nm.

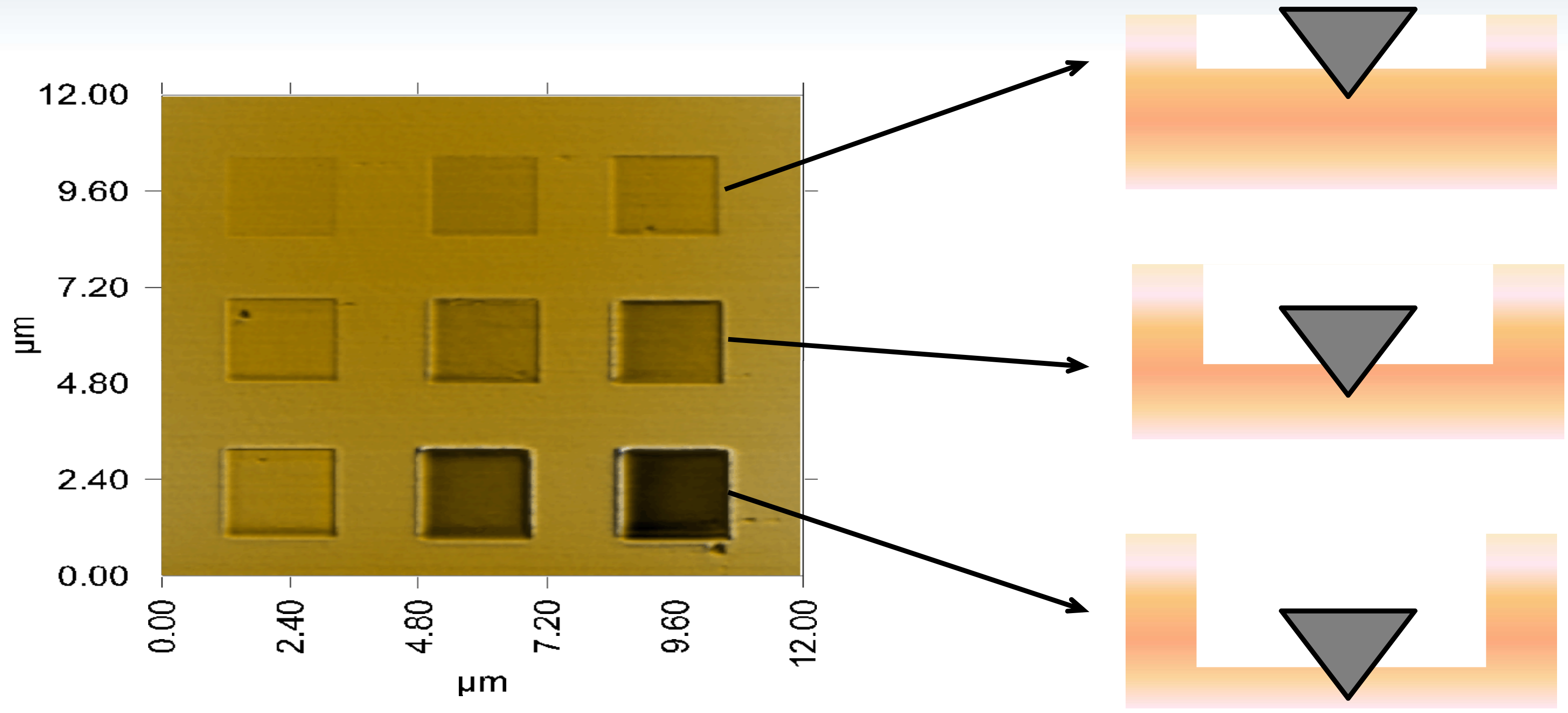
Hardness Transition

Transition features:

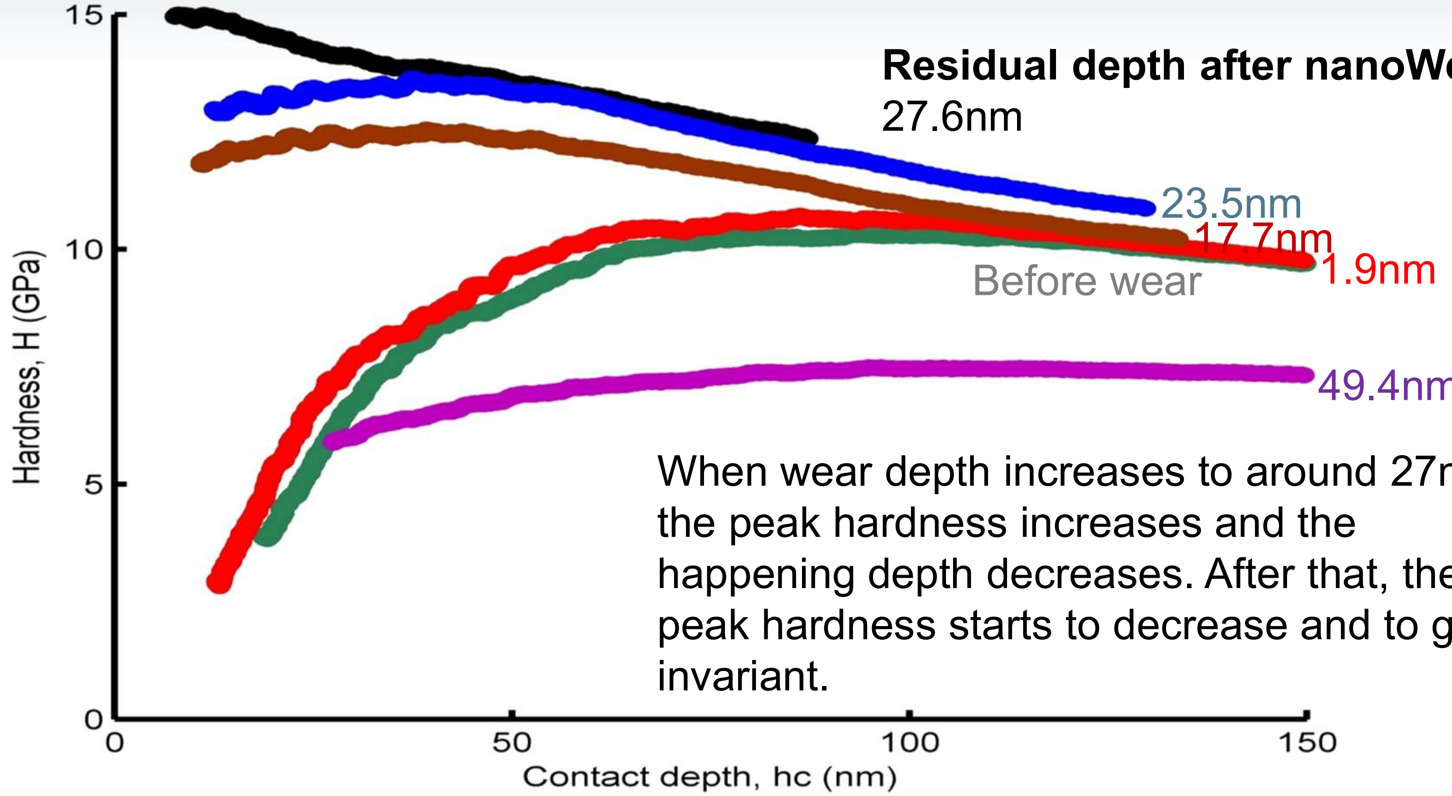
1. Effective hardness gradually transits from film hardness (at shallow depth) to substrate hardness (at large depth).
2. The transition behavior depends on film and substrate combination.
3. Multi-layered or gradient coating case is more complicated.



Metrology Concept

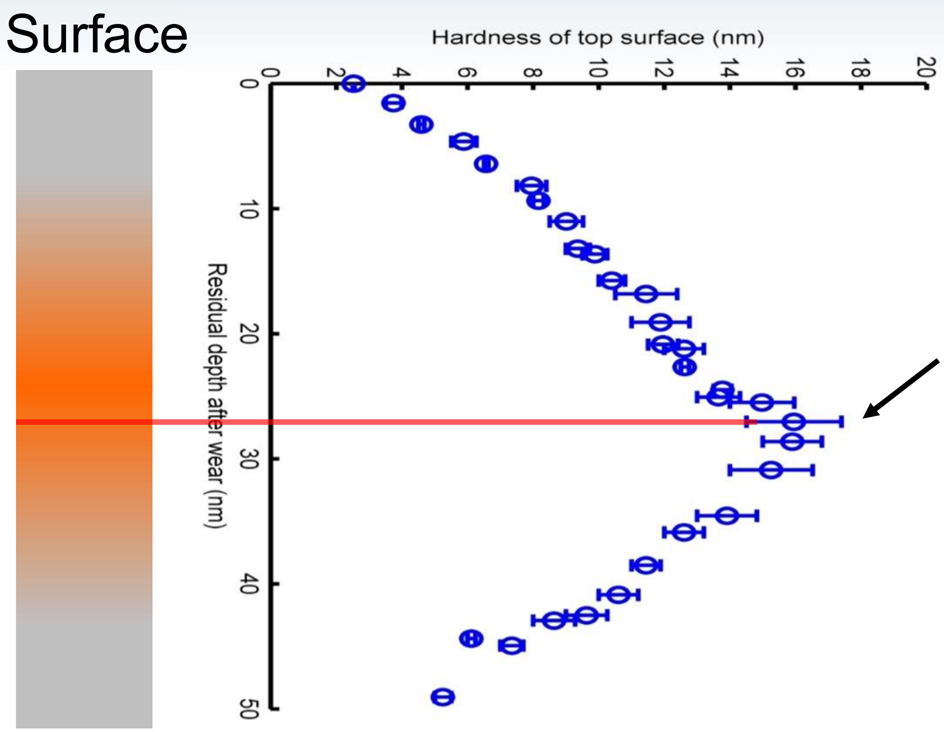


Depth Profiles after Wear

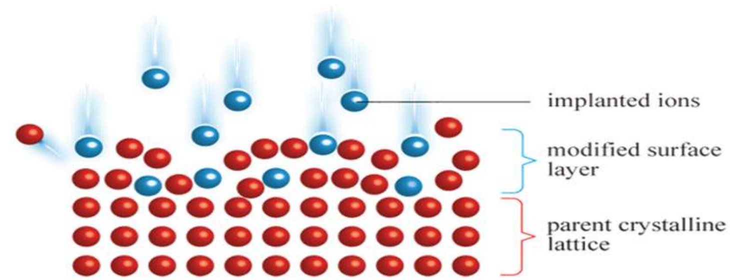


When wear depth increases to around 27nm, the peak hardness increases and the happening depth decreases. After that, the peak hardness starts to decrease and to go invariant.

Surface Hardness after Wear



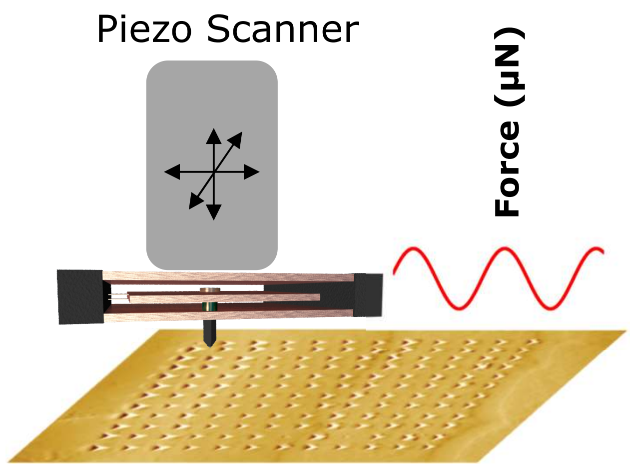
Peak hardness was found at wear depth of around 27nm



High speed Indentation (XPM)

XPM – Extreme High-Speed Nanomechanical Property Mapping

- 500x faster than regular nanoindentation – up to 6 indents per second!
  - Perform a 400 point quantitative mechanical property map in only 67 seconds!



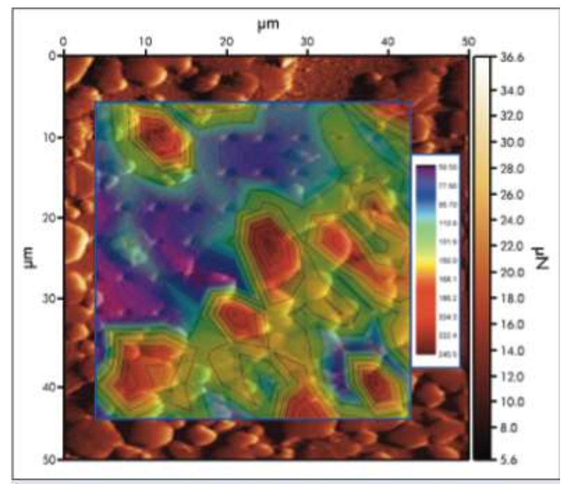
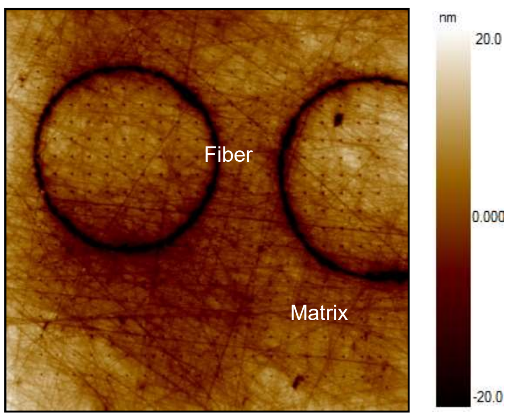
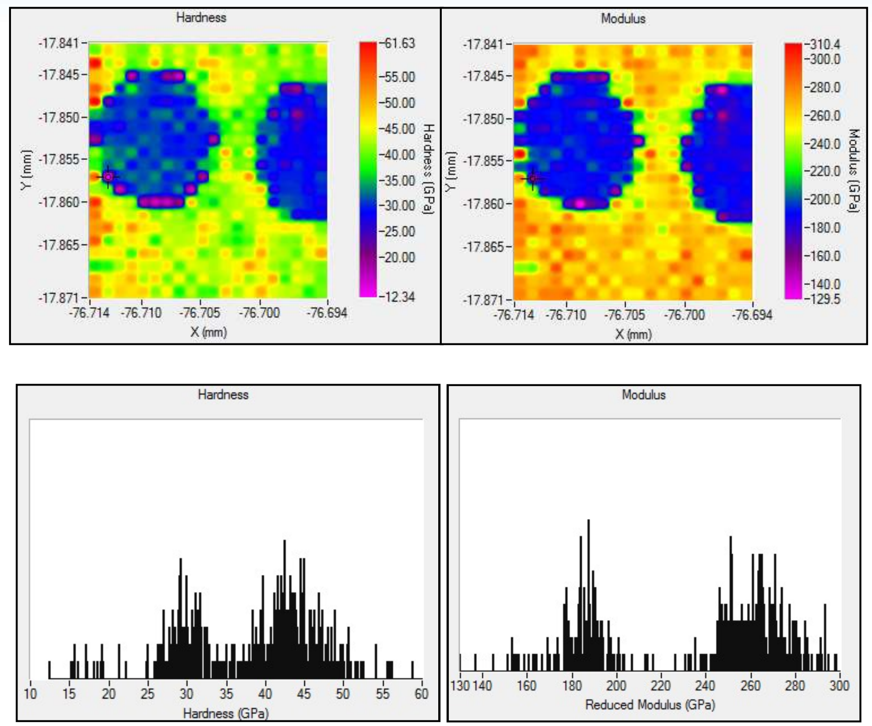


Figure 3: Cu-W alloy modulus map overlaid on an in-situ SPM image.

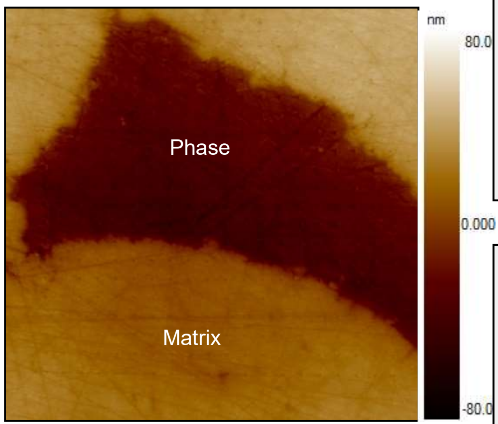
Quantitative Maps of Hardness and Elastic Modulus



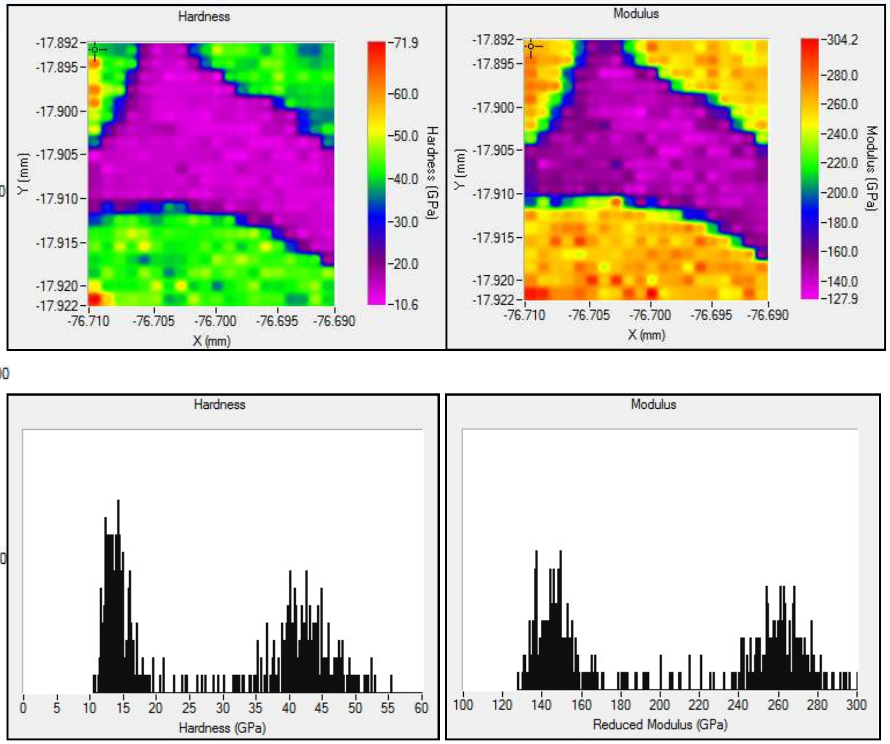
Scan Size = 20 µm



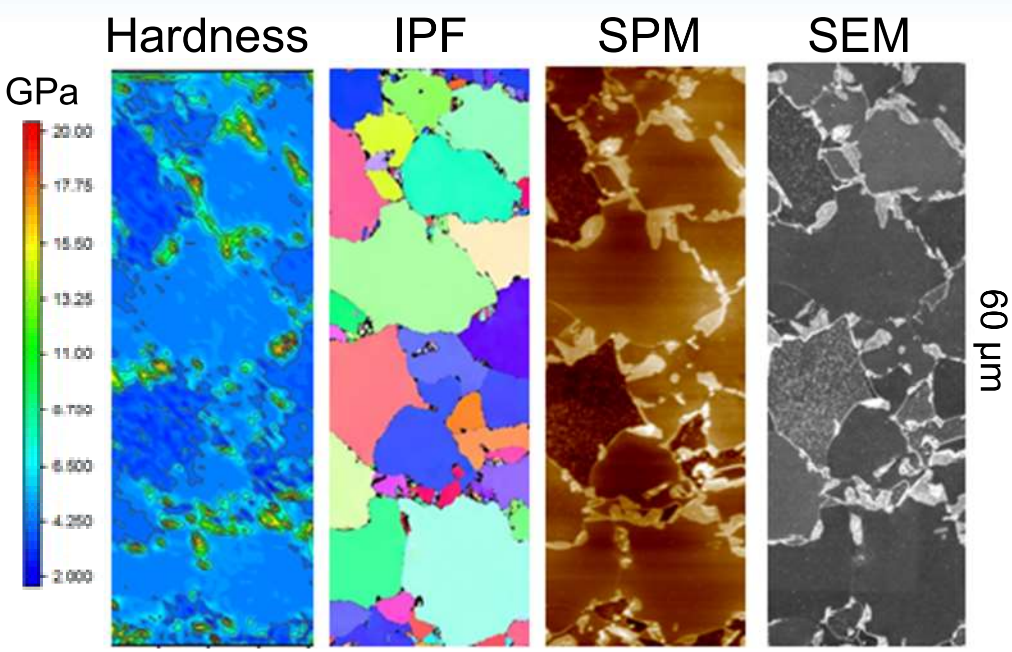
Nanomechanical Property Maps of the Composite



Scan Size = 20 μm



Correlated XPM/EBSD of Railway Weld

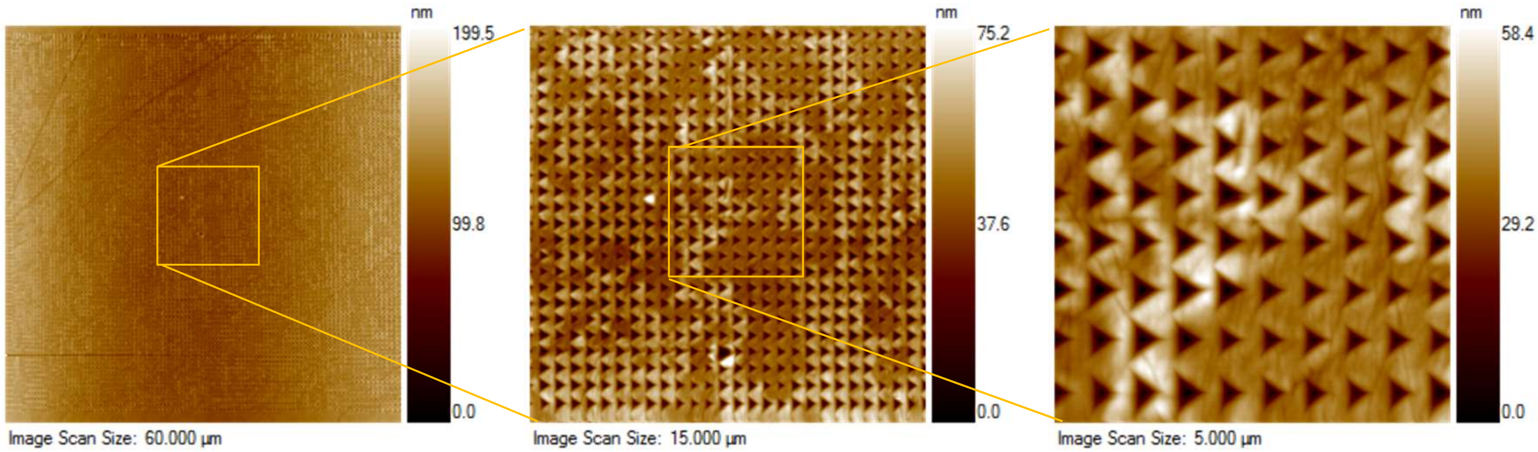


Inverse pole figure (IPF) orientation component uses a basic RGB colouring scheme, fit to an inverse pole figure. For cubic phases, full red, green, and blue are assigned to grains whose <100>, <110> or <111> axes, respectively, are parallel to the projection direction of the IPF.

- High hardness in martensitic islands at grain boundaries
- Slight hardness variations within grains correlated with orientation

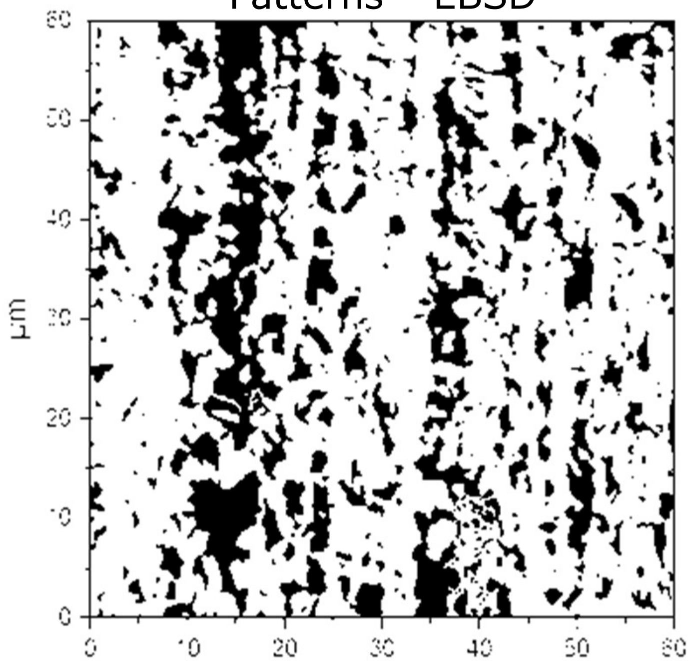
Hardness Mapping Steel – DP980

- Hardness Mapping on the cross section of a cold rolled steel sheet.
- Mapping at half thickness.
- Image size: 60x60µm; 100x100 grid array of indents = 10,000 indents in only ~40 minutes!



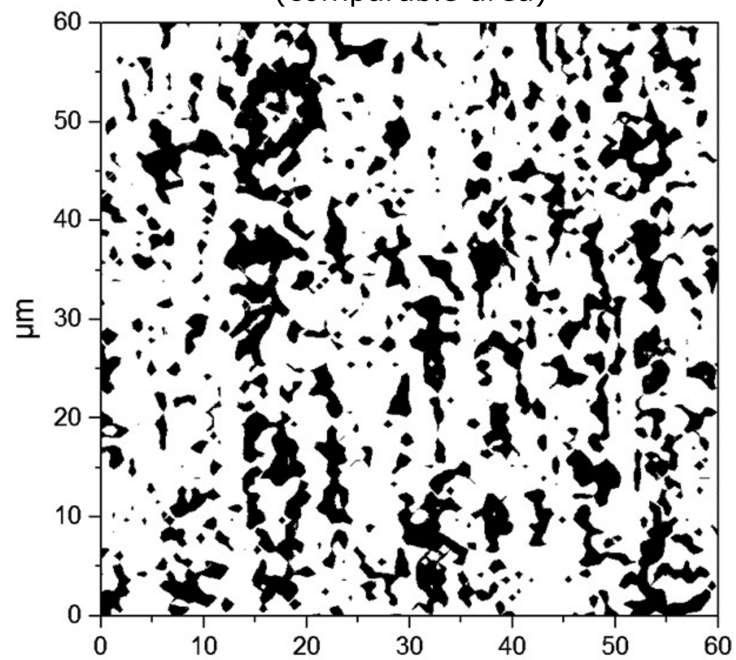
Phase Distribution – DP980

From Electron Diffraction Patterns – EBSD



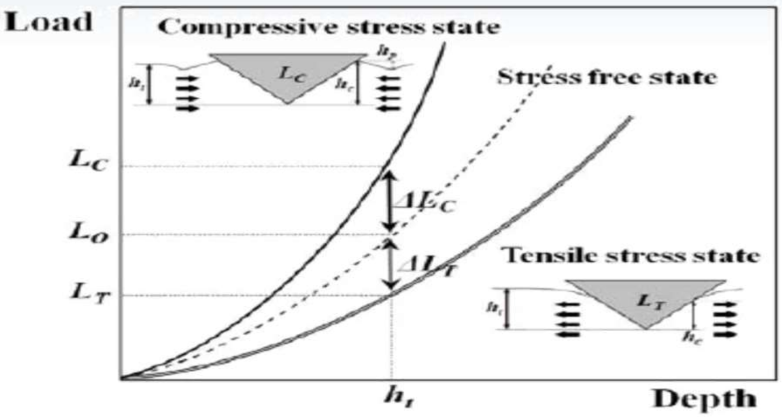
Black – Martensite
White - Ferrite

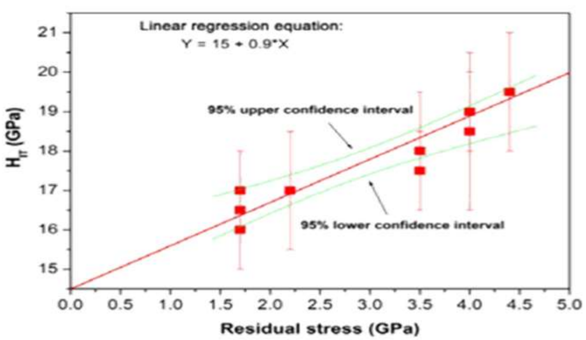
From Nanoindentation Testing
(comparable area)



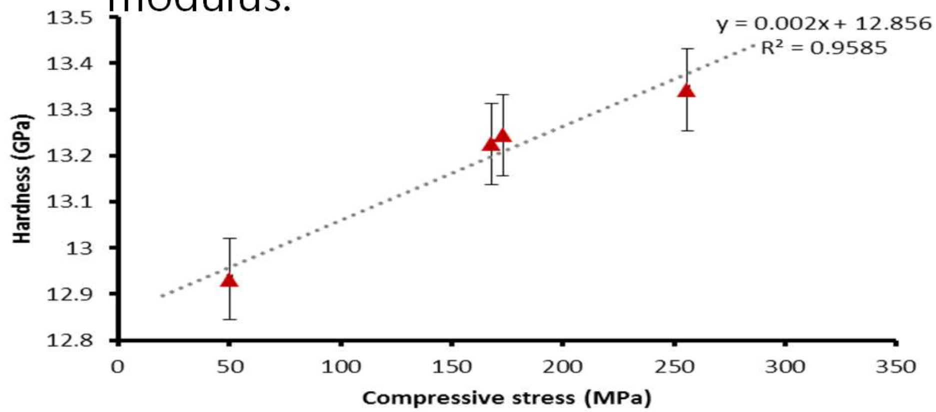
Excellent correlation between techniques for phase distribution comparison

Index of Residual Stress: Hardness Annealed Silicon Oxide Films

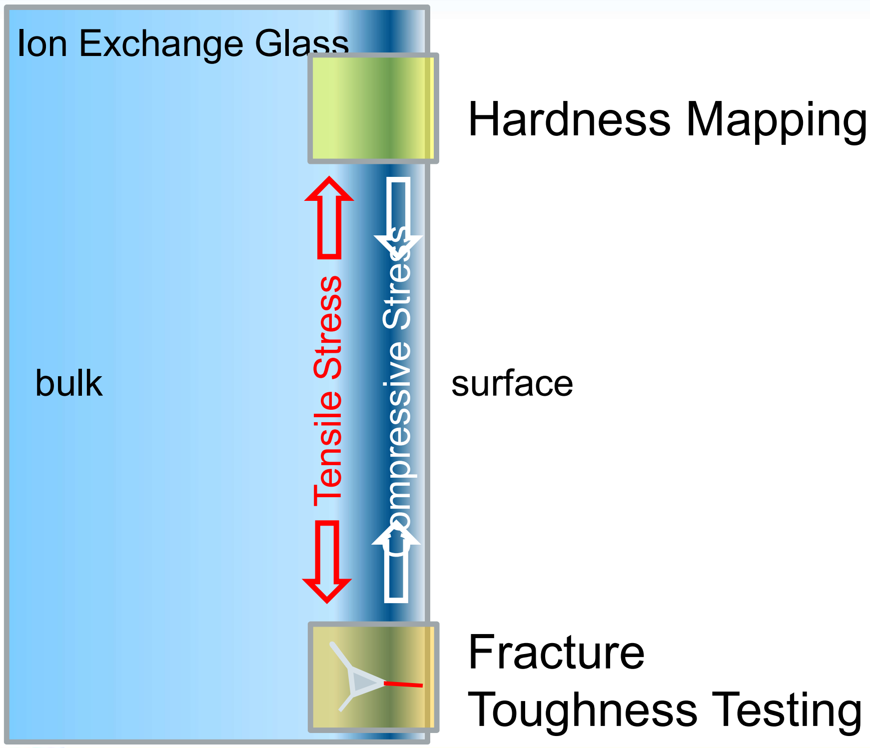




Indentation hardness of film is affected by residual stress. Compressive residual stress was found to linearly increase indentation hardness of film, but to have almost zero effect in indentation modulus.



Investigation of Ion-Exchange Zone



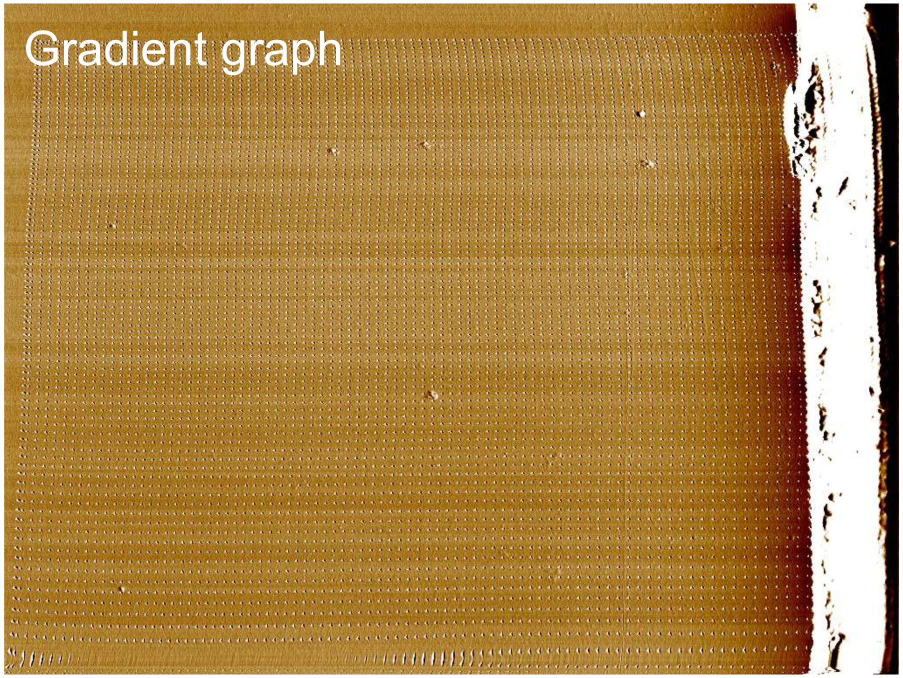
Ion-exchange glass contains engineered compressive stresses and exhibits an increased surface hardness

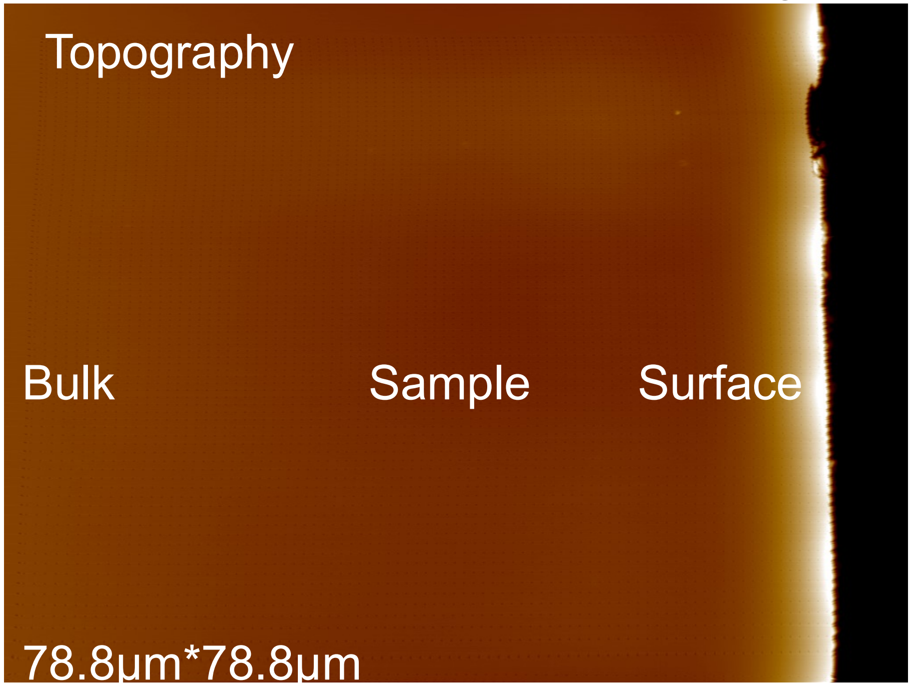
-Hardness Mapping with a Berkovich indenter allows measuring the different hardness and modulus of the glass vs. Depth.

-Cube Corner indentation initiates cracks and the effect of toughening and tensile stress are observed directly.

100 *100 XPM Map

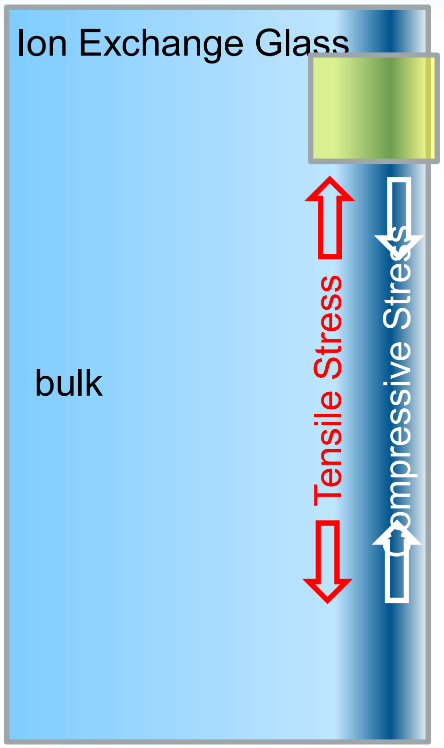
The in-situ SPM image confirms an excellent sample preparation with a sharp edge.

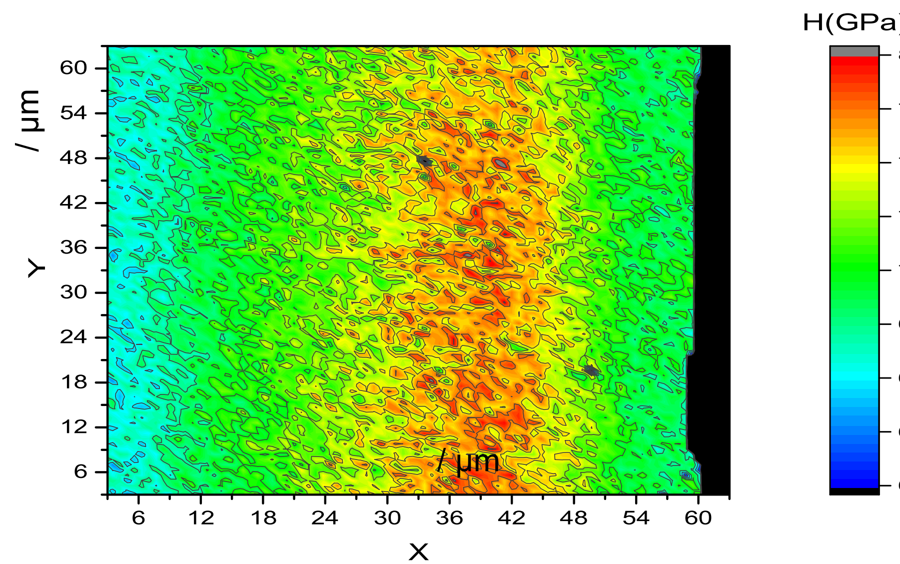




XPM Map Results

100 *100 XPM map with a penetration depth of 25nm



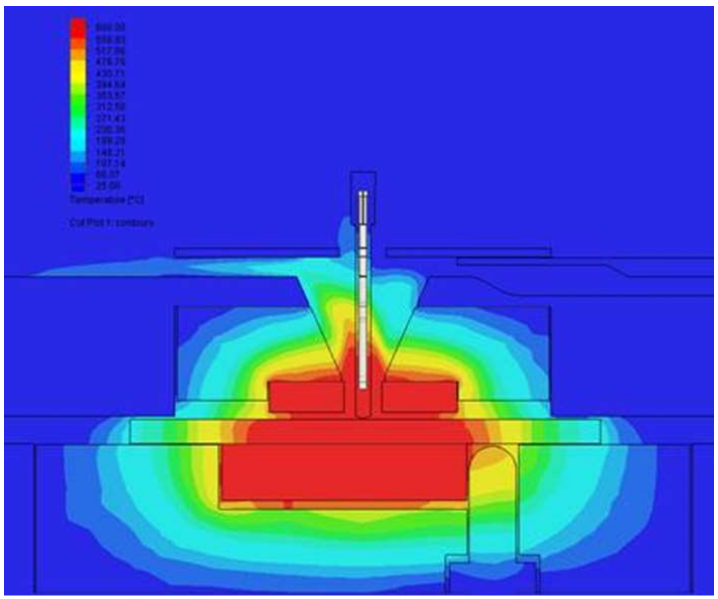


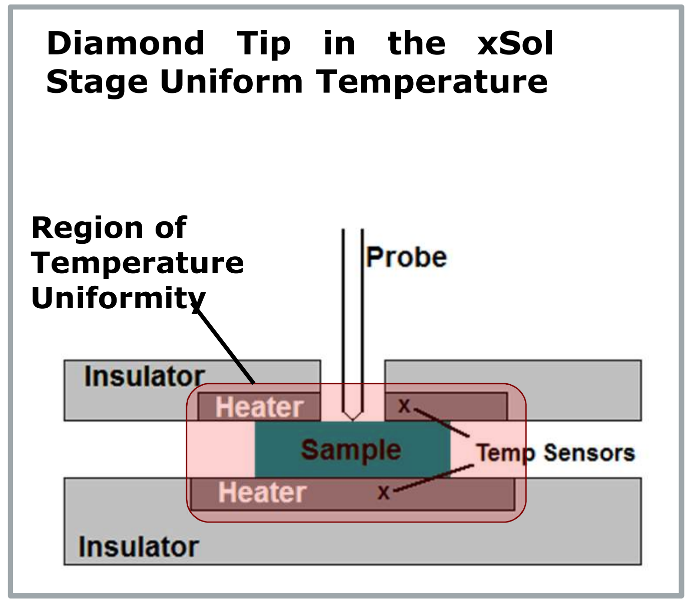
Hardness map (10.000 single indentations)

High Temperature Nano Indentation

Up to 800 degree C

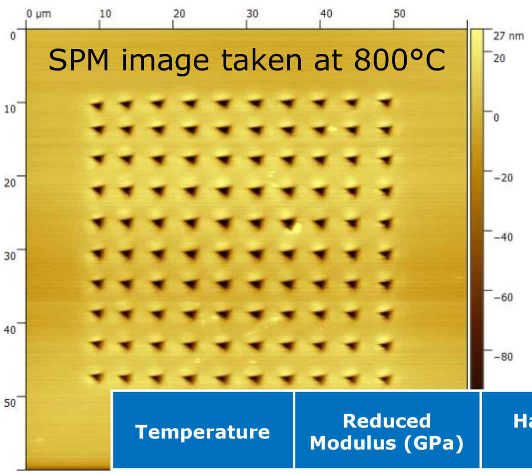
Micro Chamber design with passive Tip heating



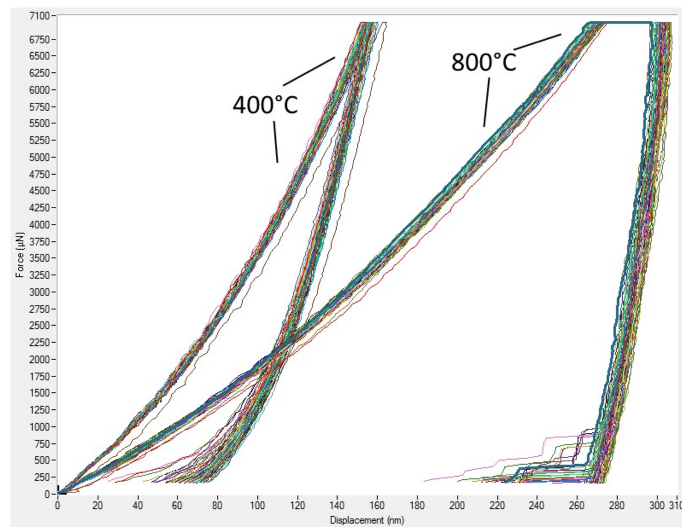


High Temperature + XPM on Silicon

- Silicon sample tested at 400°C and 800°C using XPM, 10x10 array
- Tested under inert 95% Argon / 5% hydrogen atmosphere

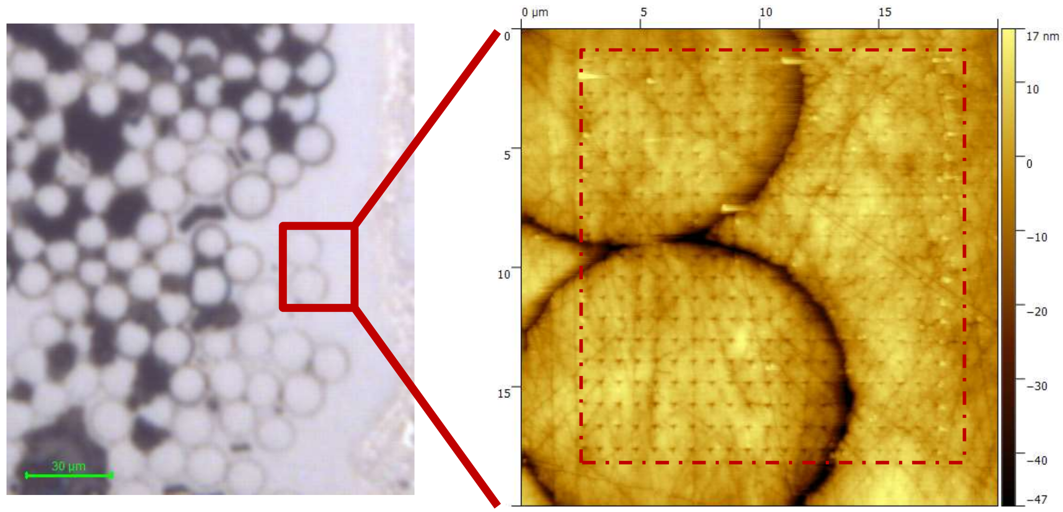


Temperature	Reduced Modulus (GPa)	Hardness (GPa)
400°C	157.2±3.1	10.67±0.24
800°C	153.0±9.2	2.350±0.035



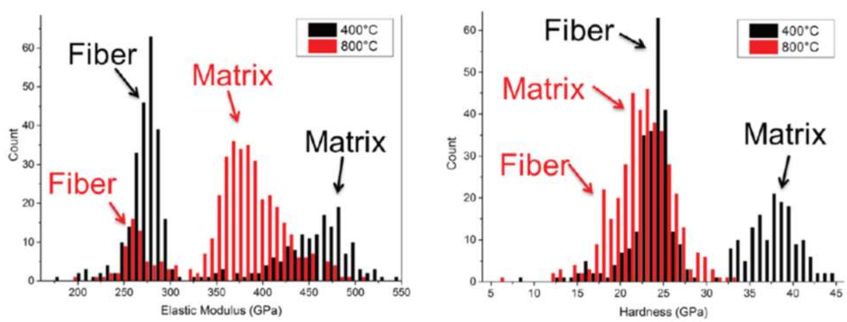
High Temperature XPM on SiC Fiber-matrix Composite

- Silicon Carbide CMC sample tested at 400°C and 800°C using XPM high throughput indentation
- 20x20 grid performed in 100 seconds

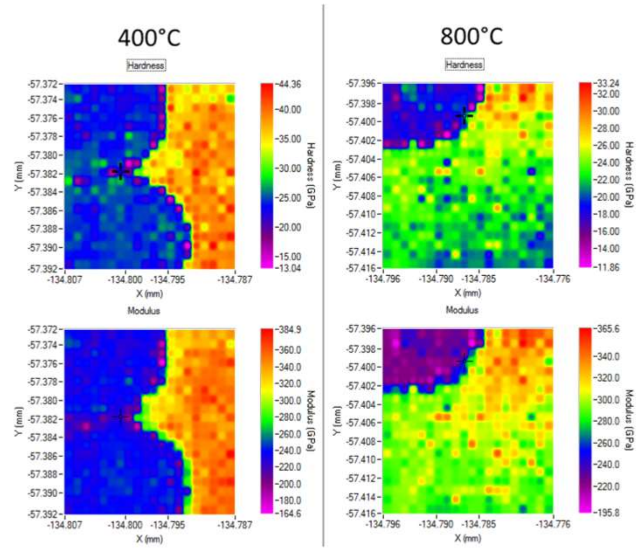


High Temperature XPM on SiC Fiber-matrix Composite

- Silicon Carbide fibers embedded within SiC matrix sample tested at 400°C and 800°C
- 20x20 grid performed in only 100 seconds – reduces tip degradation at high temperature!



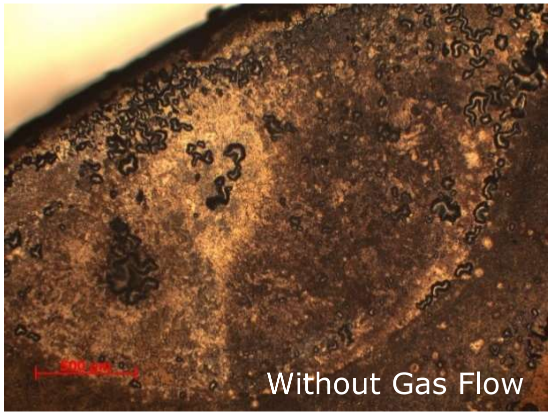
Histograms of silicon carbide fibre-matrix composite hardness and elastic modulus results obtained from XPM indentation testing at 400°C and 800°C

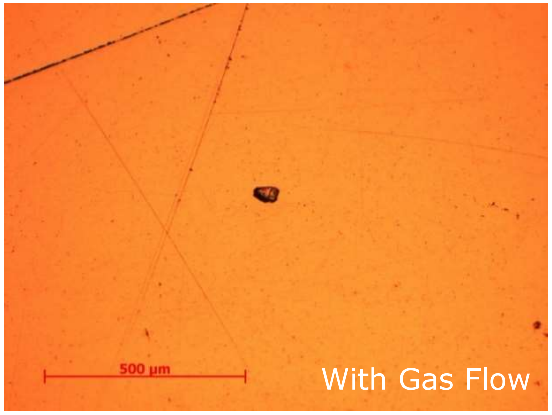


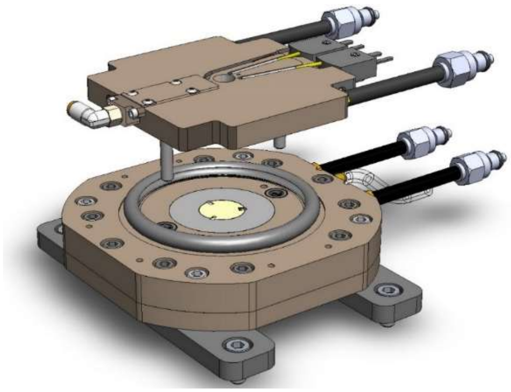
Fiber hardness and modulus, while lower, are also retained better at high temperature

xSol Atmosphere Control

The use of “shield gas” to prevent or slow oxidation is possible because of the closed design of the stage.







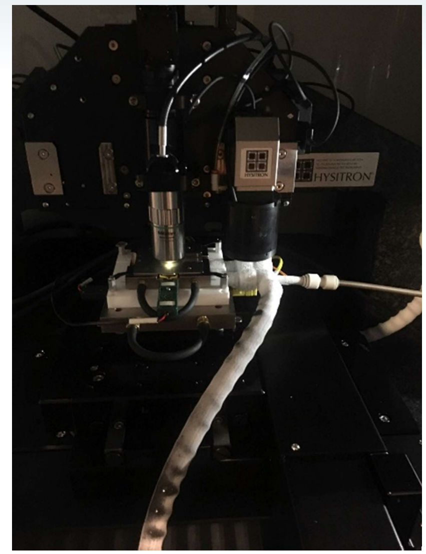
- Fast Warm Up and Stabilization
- No vacuum needed

xSol Cooling Stage System Overview

Compressed Nitrogen

Nitrogen Coil in LN_2 Tank

TI 980 version of Cooling port

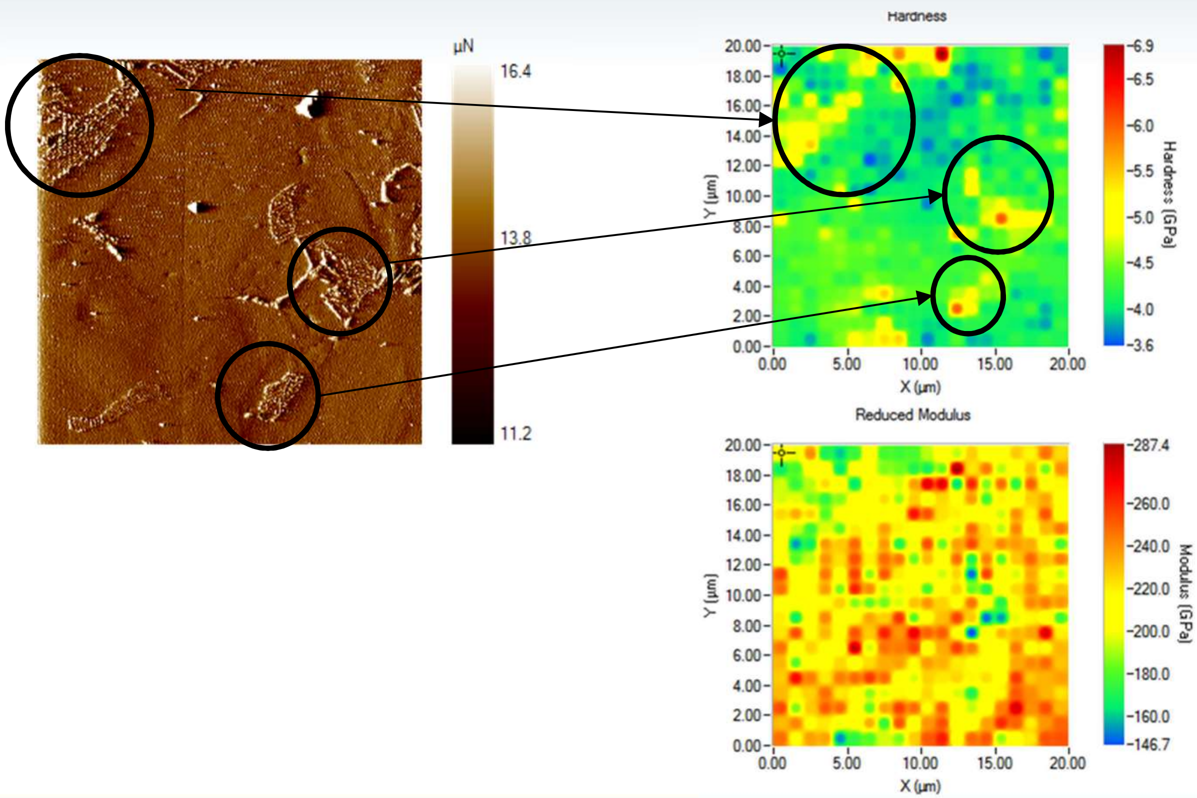


Internal heat exchanger/sample chamber

- Nitrogen gas passes through liquid N2 to cool
- Cold gas enters the chamber through a port on the side of the enclosure to passively cool the chamber
- A heater is used to actively hold the temperature steady at the set temperature
- Achieves cooling capabilities down to -160°C

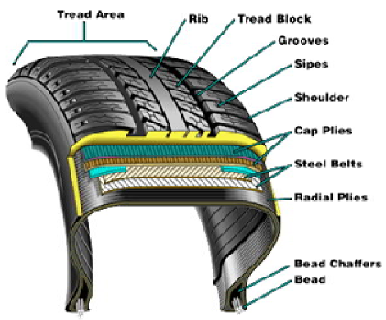
XPM Tests at -100° C

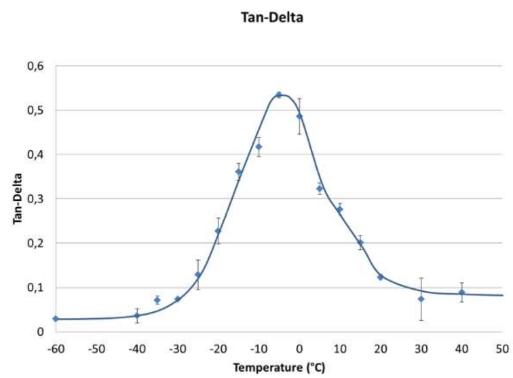
- Low carbon steel with Pearlite phase and Ferrite phase shown on the SPM image.
- Low temperature XPM shows hardness variation between those two phase where Pearlite phase exhibits ~ 20% more hardness value than the Ferrite phase.

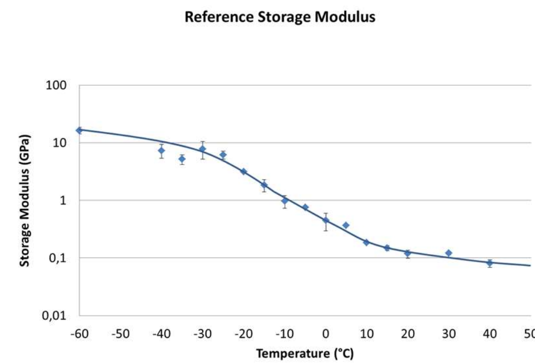


xSol Heating & Cooling on Tire Rubber

- nanoDMA III testing of tread materials used in winter tires
- A cold and dry gas environment around the sample was generated by the controlled flow of gas from a dewar filled with Li N_2
- Controlling sample temperature by xSol heaters

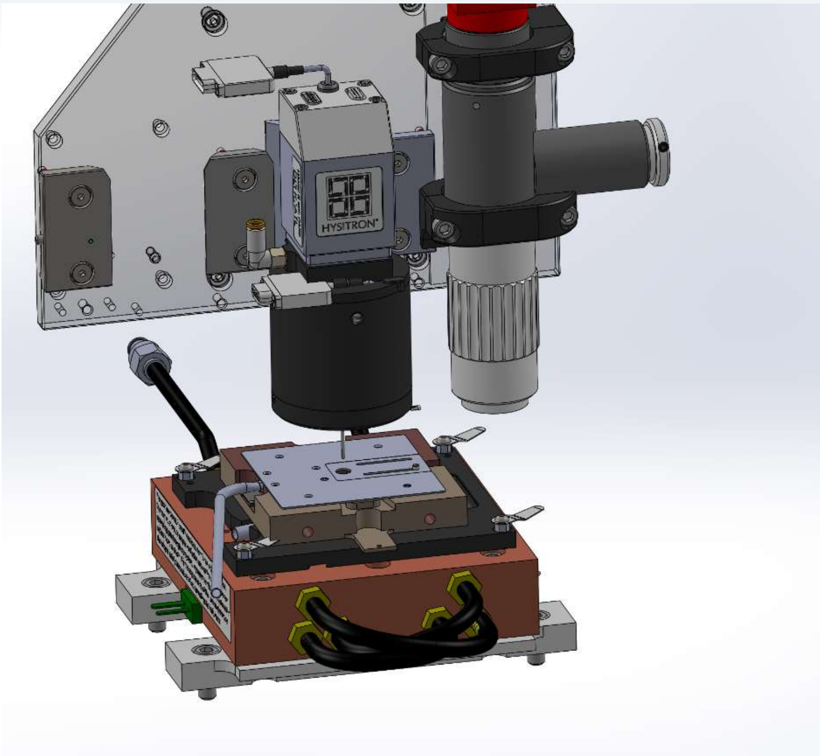


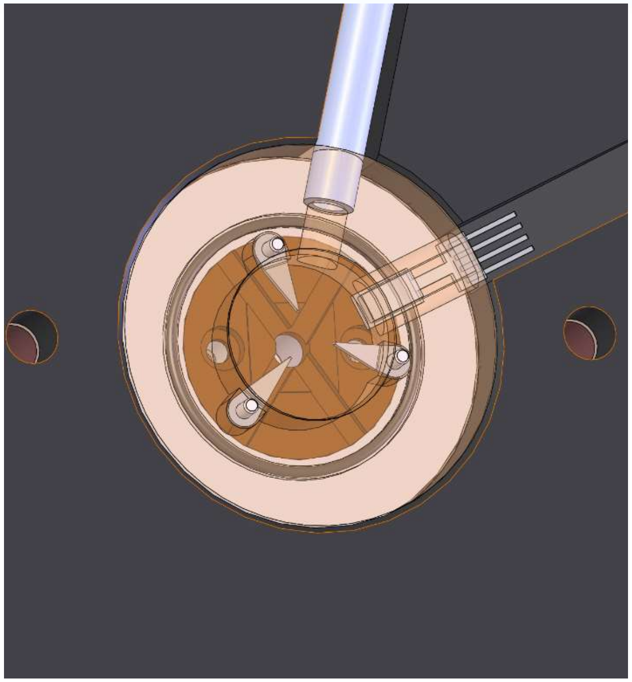




- The tread material of a winter tire shows a peak in the tan(d) at a temperature of -5°C which indicates that the compound undergoes a glass transition
- The storage modulus drops from a value around 10 GPa at -60°C of less than 100 MPa at 40°C which is a typical observation if a polymer undergoes glass transition

Humidity & Temperature Control





Quantitative *In-Situ* TEM Nanomechanical Testing Instruments

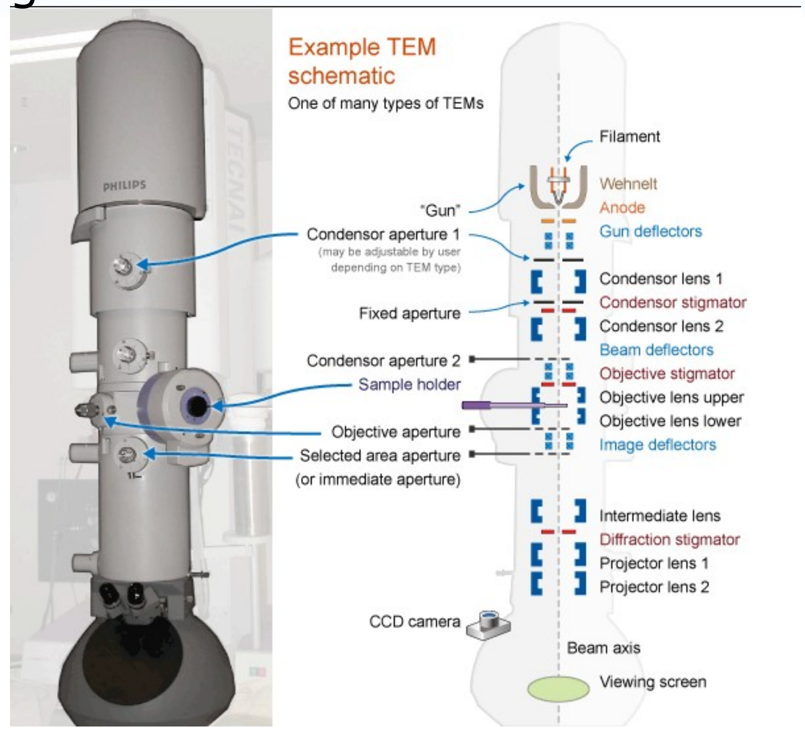
TEM Mechanical testing Holder

Zeiss

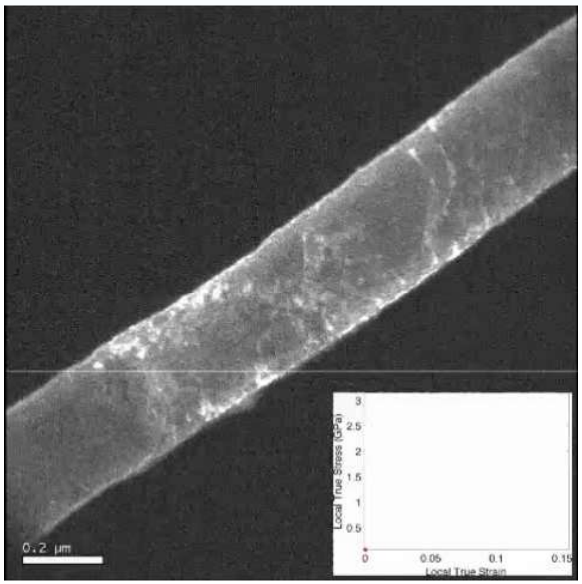
JEOL

FEI/Philips

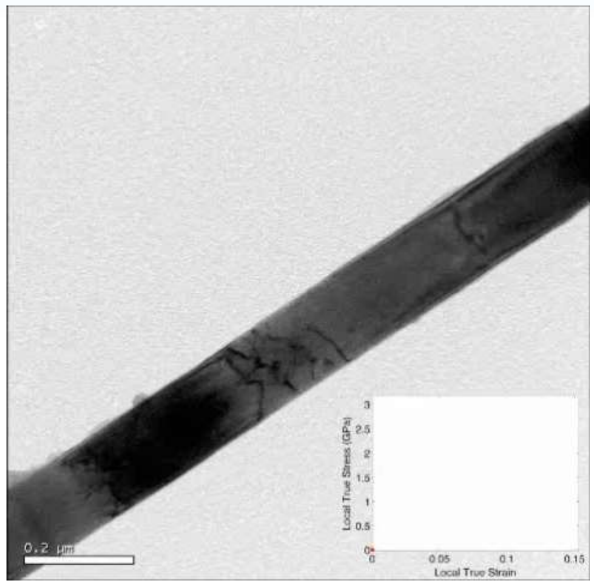
Hitachi



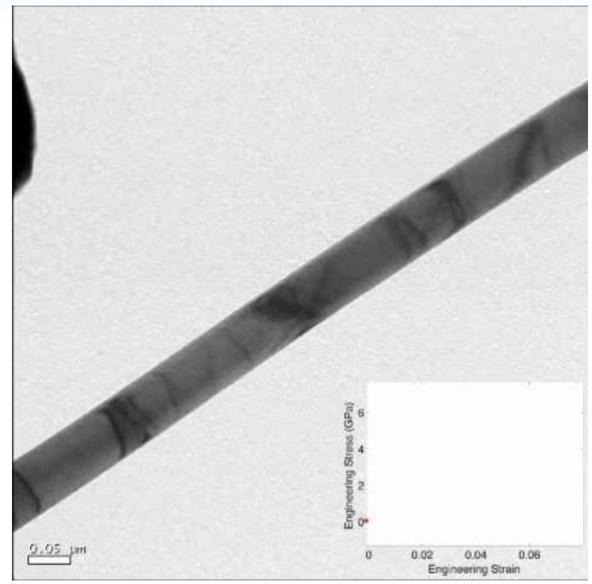
PTP Applications:
Tensile Testing of Mo-alloy nano-fibers



15%
Pre-strain



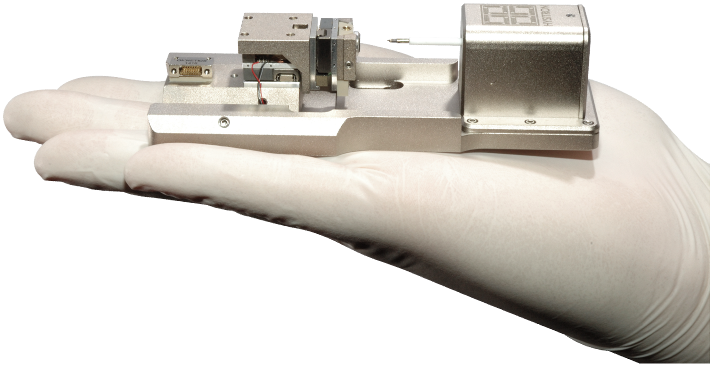
0% Pre-strain
(not dislocation-free)

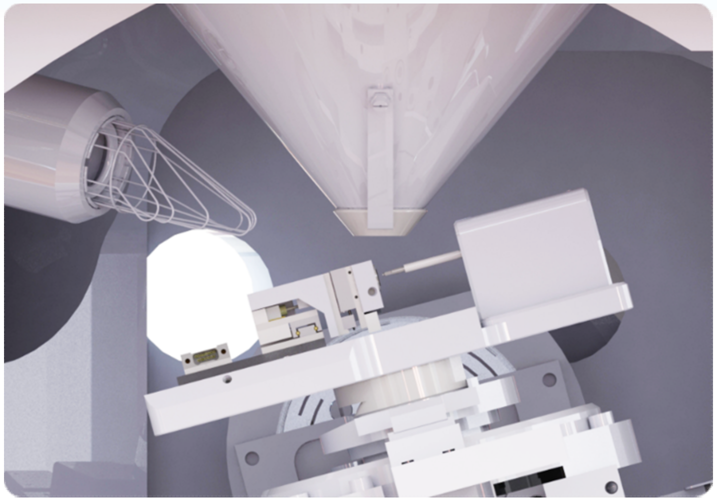


0% (dislocation-free)

In Situ SEM Nano indentation

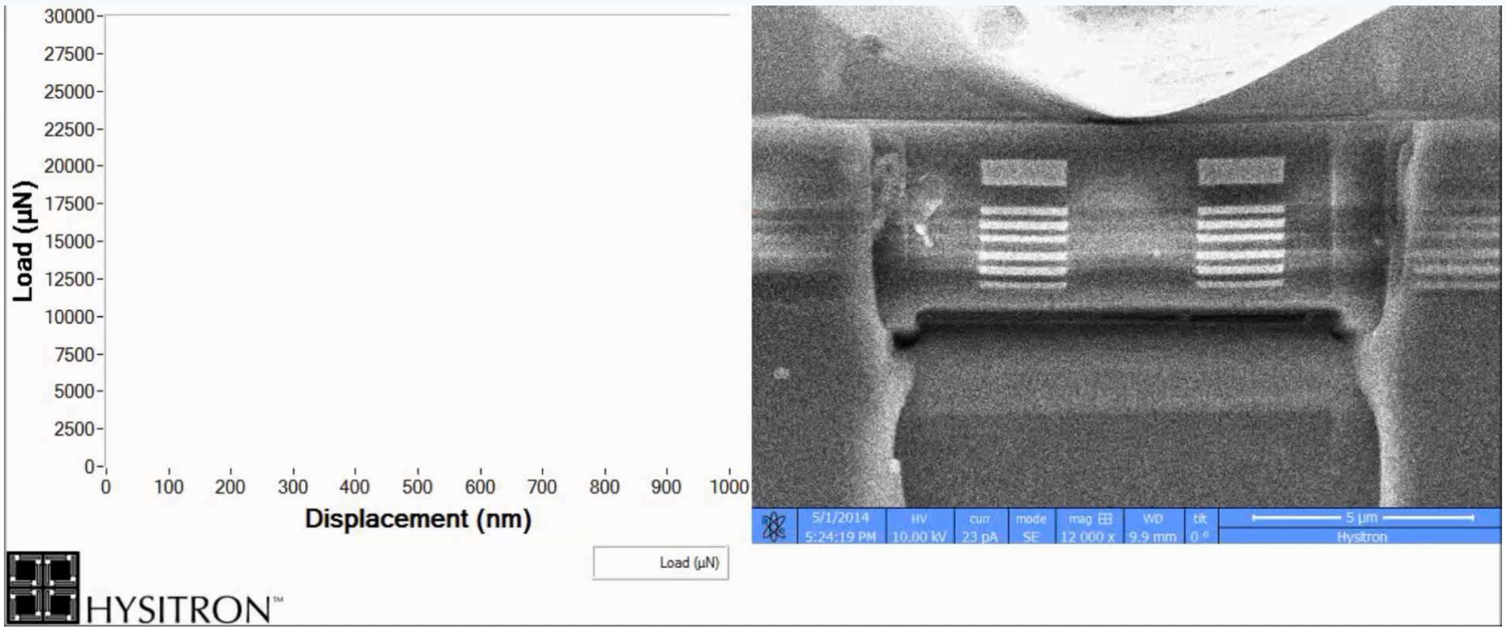
Small-volume system





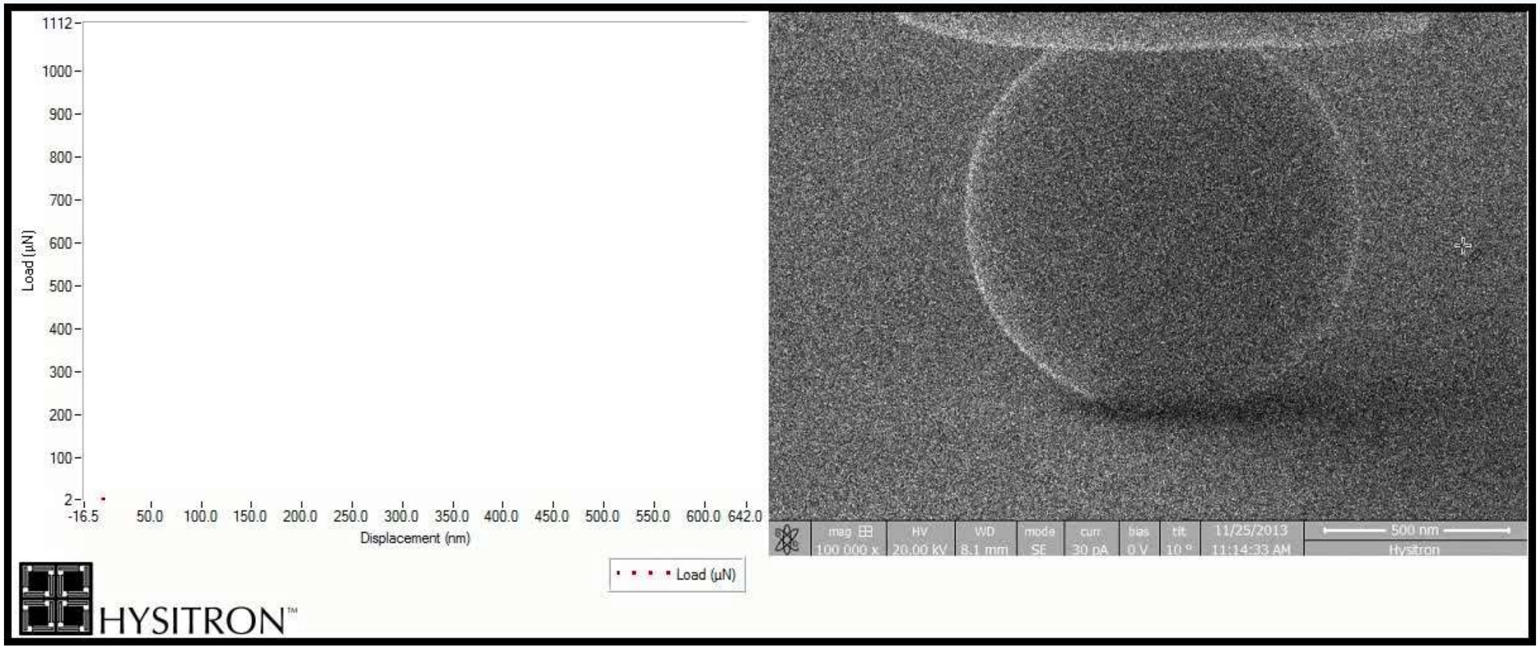
- Compact system designed to fit standard SEM and FIB/SEM chambers
- Enables high tilt of the microscope stage for specimen imaging during testing
- Compatible with most FEI, Hitachi, JEOL, TESCAN, Zeiss, and more…
- Formal compatibility check with your SEM

Applications: Failure analysis of BEOL Materials by Microbeam Bending Technique



"In-situ scanning electron microscopy study of fracture events during back-end-of-line microbeam bending tests" K. Vanstreels, I. De Wolf, H. Zahedmanesh, H. Bender, M. Gonzalez, J. Lefebvre, S. Bhowmick; Applied Physics Letters, 105, 213102 (2014), DOI: 10.1063/1.4902516

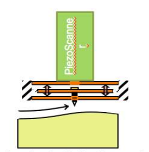
nanoDynamic™- SEM PicoIndenter



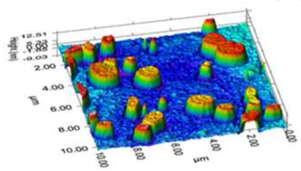
1 µm Silica particle compression with 1 mN DC, 100 µN @ 10 Hz AC load

Nano mechanical Hybrid

表面性質



SPM (Scanning Probe Microscope)
X probe (MEMS Transducer)
Optical Microscope
Modulus Mapping

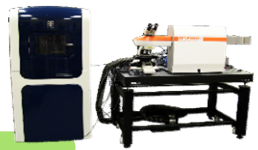


力

HYSITRON®

化學性質

EC Cell
Raman Microscope



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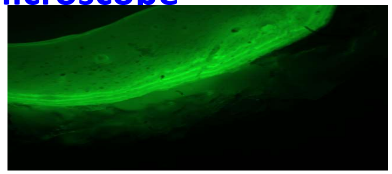
Raman Spectroscope
Fluorescence Microscope
Optical Microscope

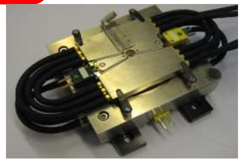


電

NaNo ECR

熱

Xsol Stage 800
Humidity
Cooling



www.bruker.com