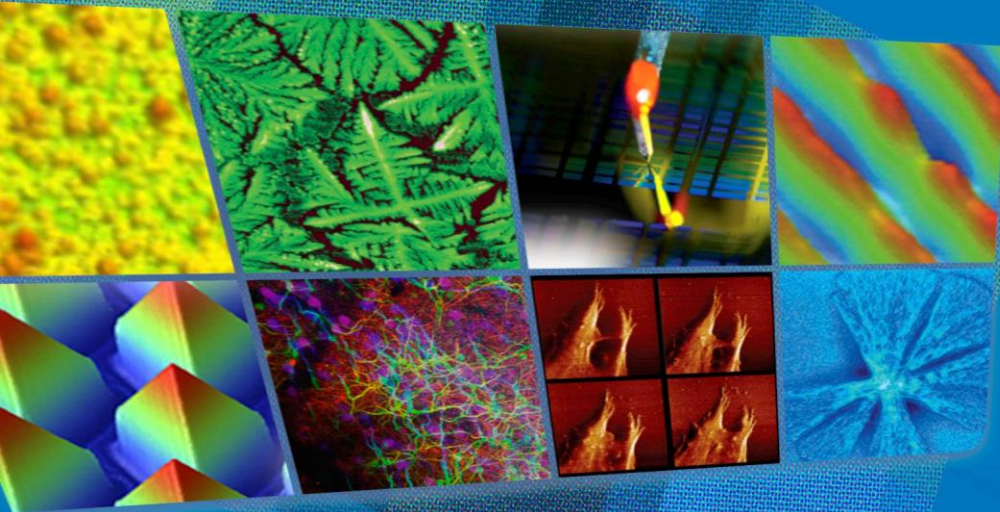


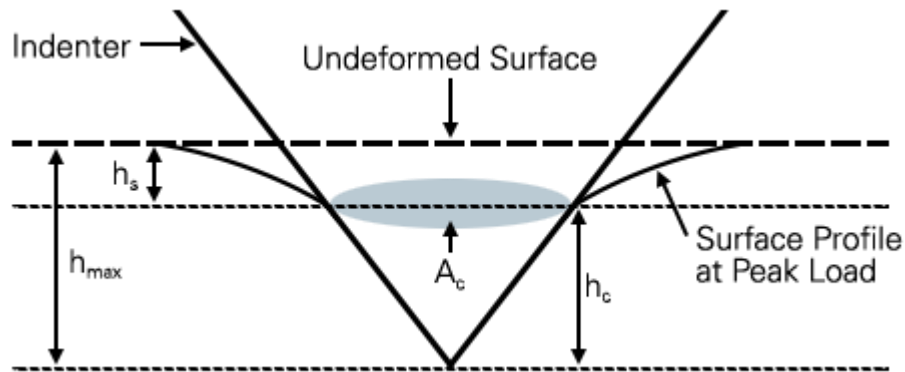
Advanced Nanomechanical Testing Methods



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

- Introduction: Brief overview of nanoindentation and nanoDMA III
- XPM™ Discussion:
 - What is it? How to do it? What can it do?
- Applications: Mapping microstructural features and interfaces, incorporating statistics
 - Pencil leads, *two phase materials*
 - PDMS, T_{melt}
 - Polymer thin film, T_g

Quasi-Static Nanoindentation

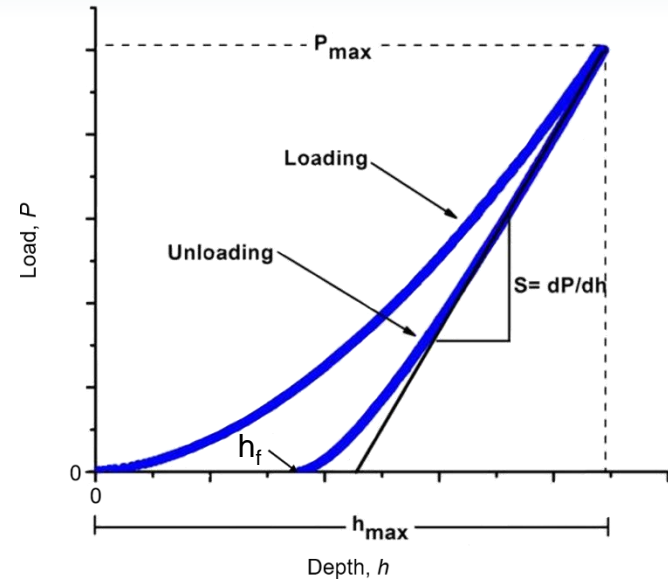


Reduced Modulus

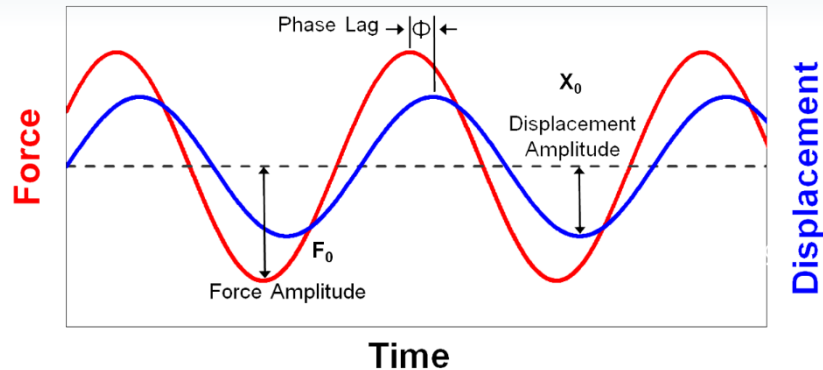
$$E_r = \frac{S\sqrt{\pi}}{2\sqrt{A}}$$

Hardness

$$H = \frac{P}{A}$$



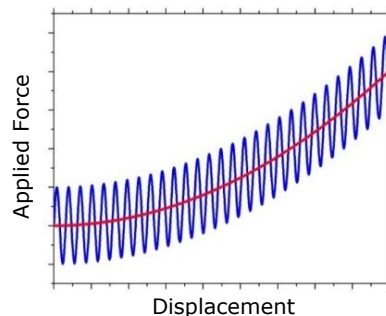
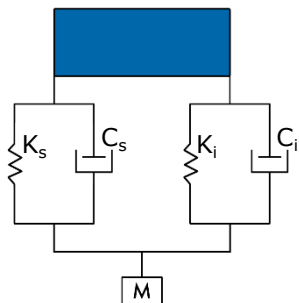
Brief Review of nanoDMA III



$$E' = \frac{k_s \sqrt{\pi}}{2\sqrt{A_c}}$$

$$\tan \delta = \frac{\omega C_s}{k_s}$$

$$m\ddot{x} + C\dot{x} + kx = F_0 \sin \omega t$$



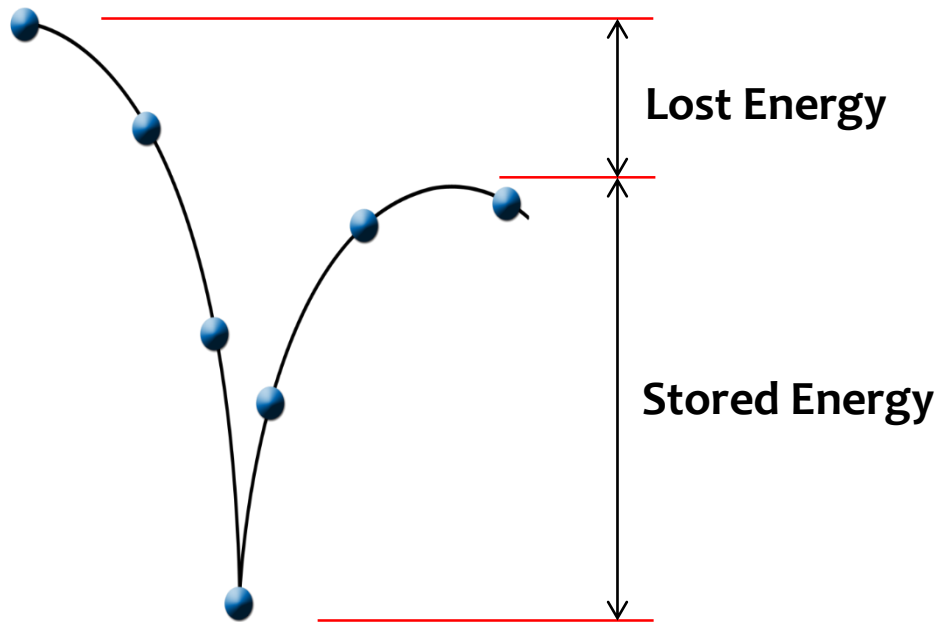
$$E'' = \frac{\omega C_s \sqrt{\pi}}{2\sqrt{A_c}}$$

S.A. Syed Asif, & J.B. Pethica, MRS Proceedings, 505,103 (1997)

Storage and Loss Modulus



Bouncing Ball



$$E'' = \frac{\omega C_s \sqrt{\pi}}{2\sqrt{A_c}}$$

$$\tan \delta = \frac{\omega C_s}{k_s}$$

$$E' = \frac{k_s \sqrt{\pi}}{2\sqrt{A_c}}$$

Quasi-static Indentation

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

Dynamic Indentation

$$E' = \frac{k_s\sqrt{\pi}}{2\sqrt{A_c}}$$

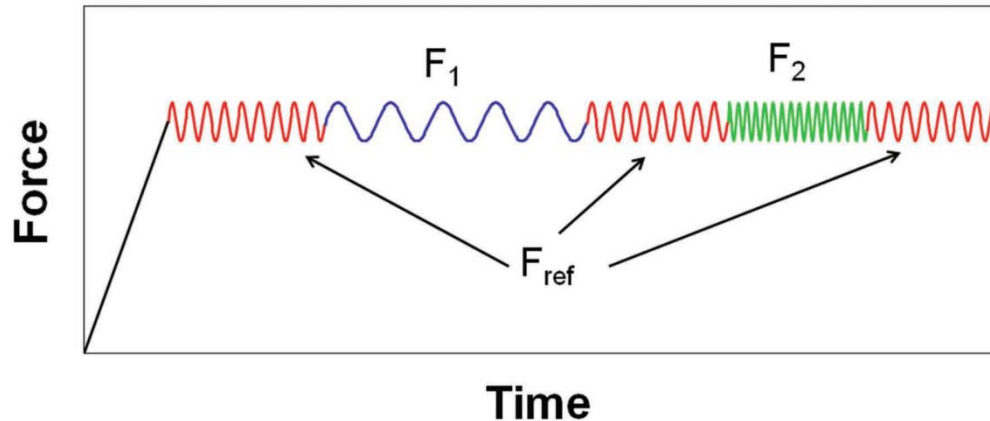
$$E'' = \frac{\omega C_s\sqrt{\pi}}{2\sqrt{A_c}}$$

S.A. Syed Asif, & J.B. Pethica, MRS Proceedings, 505,103 (1997)

Reference Frequency Sweep



For long durations, “reference” technique may be employed to measure the contact area periodically throughout the test.

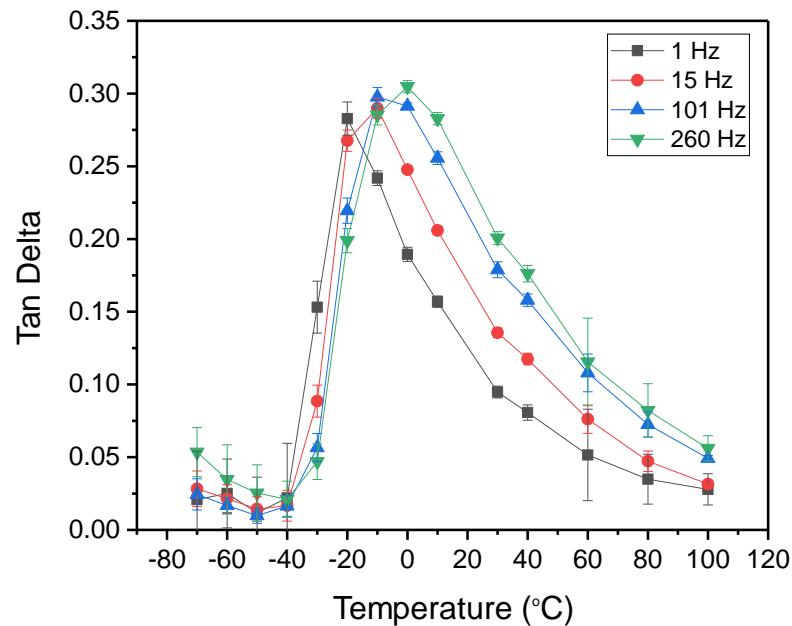
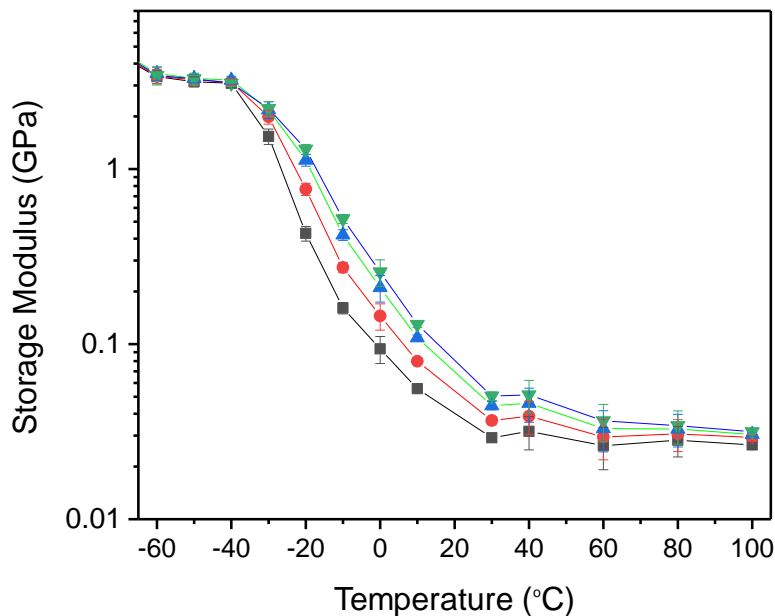


$$E' = \frac{k_s \sqrt{\pi}}{2\sqrt{A_c}}$$

$$k_s = k_s(F)$$

Amorphous Polymer Transitions

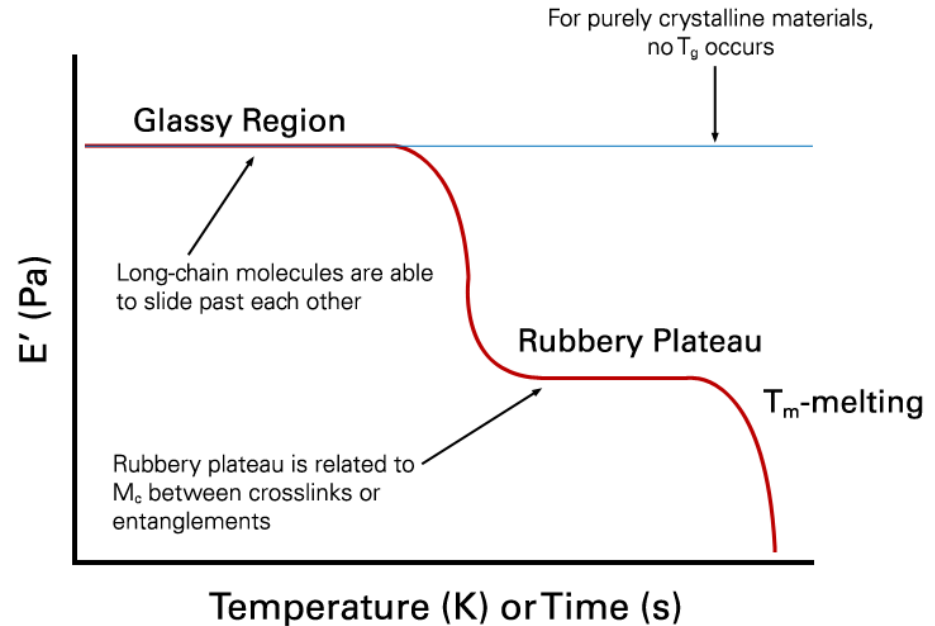
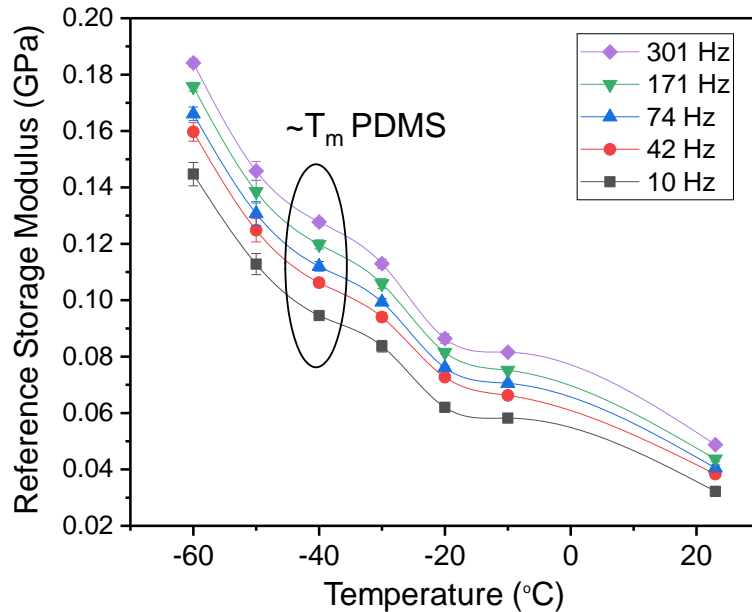
Polymer Film



Amorphous Polymer Transitions

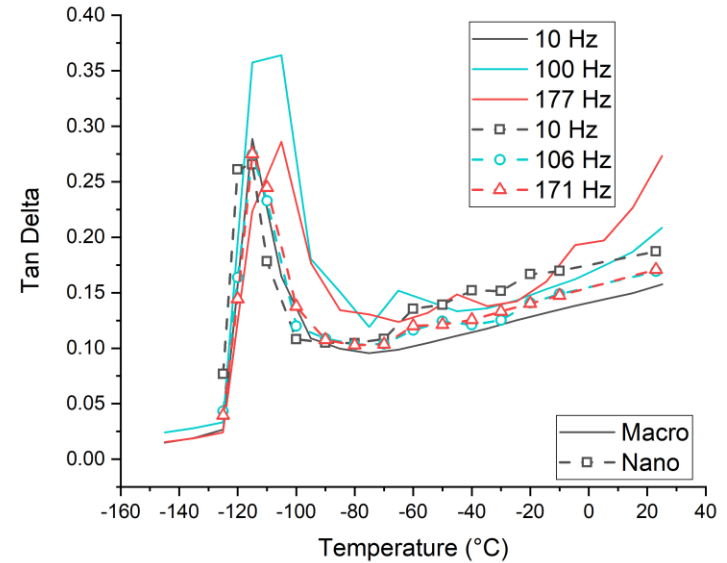
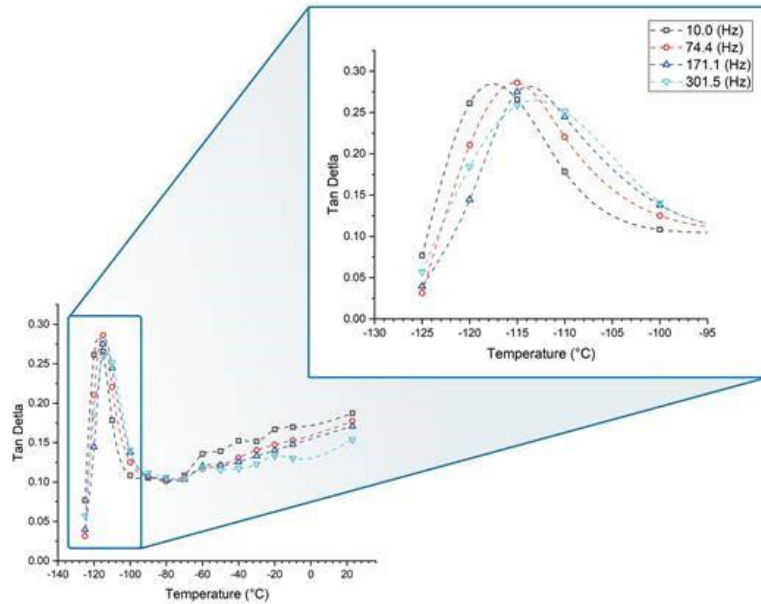


PDMS - Polydimethylsiloxane



Amorphous Polymer Transitions

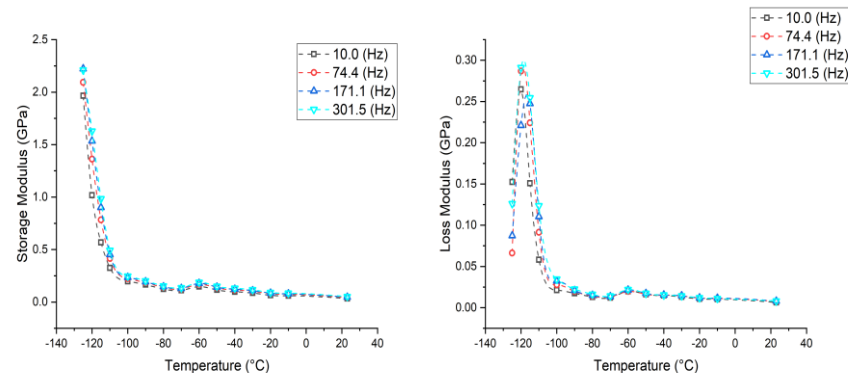
T_g - PDMS



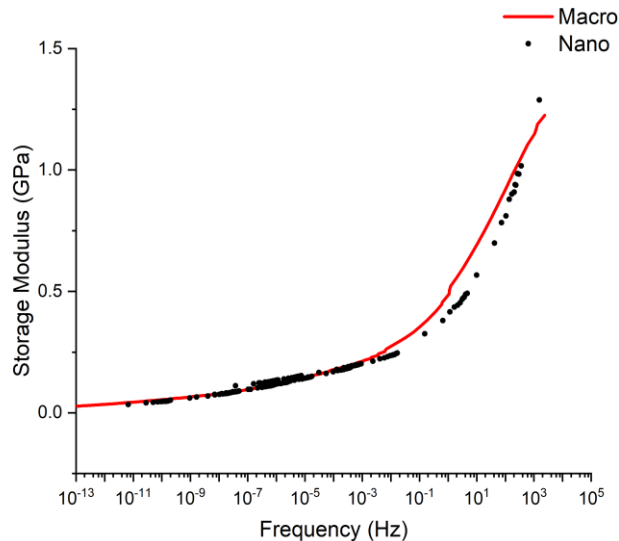
Time - Temperature Superposition (TTS)



- Shorter times (high frequencies) correspond to low temperatures and long times (low frequencies) correspond to high temperatures
- nanoDMA III frequency sweeps at varying temperatures - T_g



TTS - Master Curve (PDMS)



Time-Temperature Superposition using a WLF model.
Comparison between bulk and nanoDMA

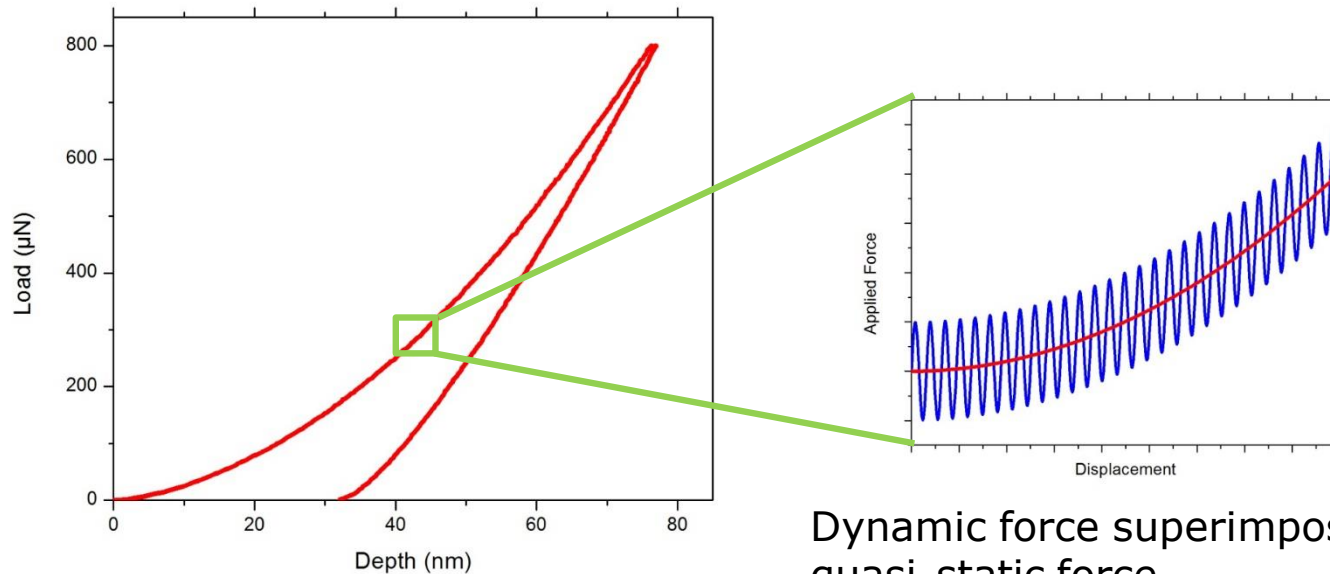
- Compares master curves at -115C created using TTS and the Williams-Landel-Ferry equation
- Frequency dependence from 10^{-11} Hz to 10^3 Hz
- Beyond instrumentation capabilities

Continuous Measurement of X

CMX



Continuously measure properties as a function of contact depth, frequency, and time.

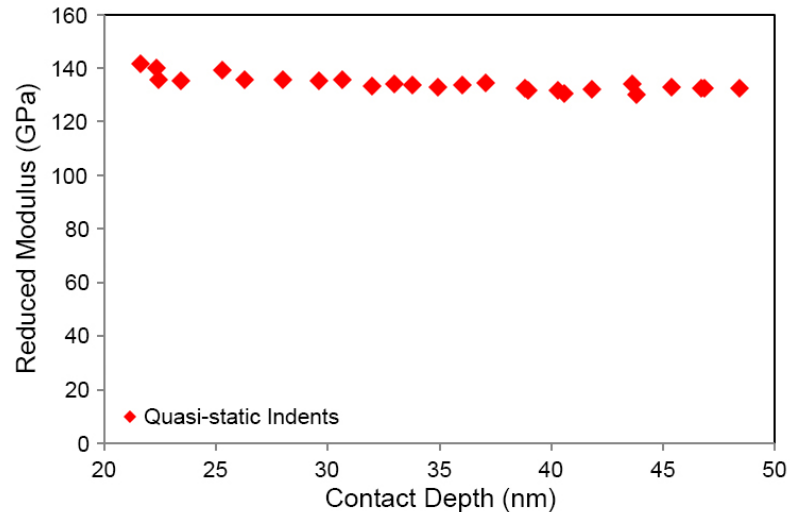


Dynamic force superimposed on quasi-static force

Quasi-static vs. CMX Depth Profiles

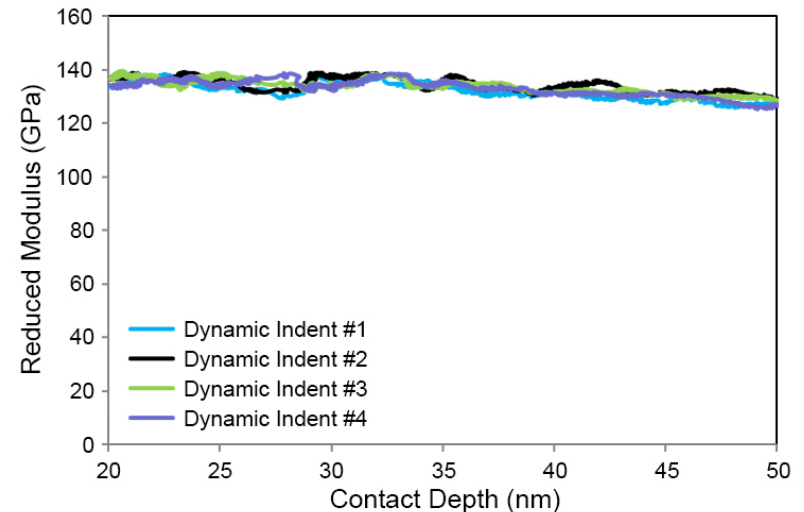


Quasi-static Indentation



134.04 ± 2.7 GPa

Dynamic Indentation



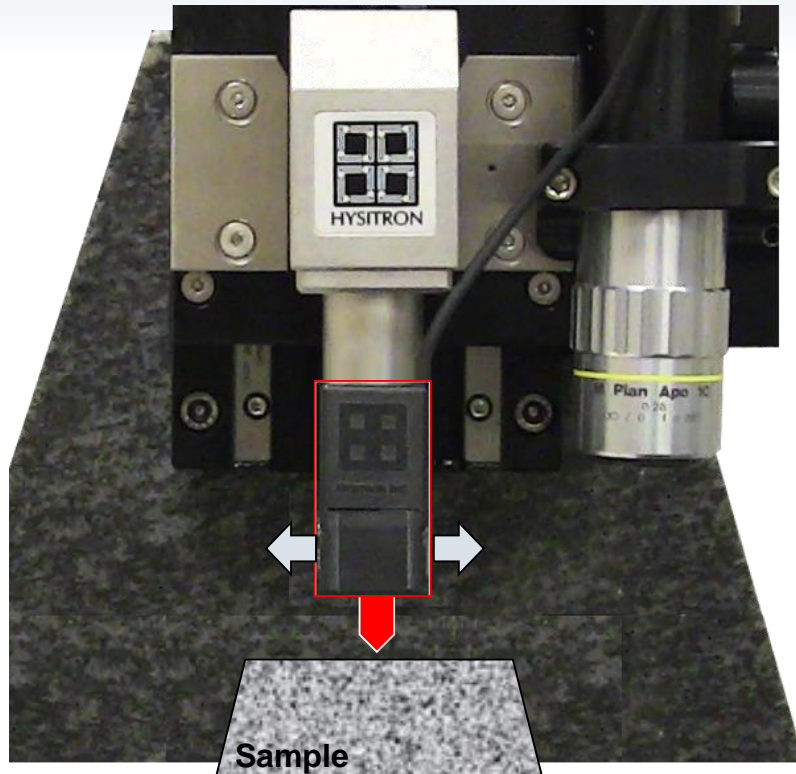
133.88 ± 2.8 GPa

XPM - Accelerated Property Mapping



- Available with the Hysitron® TI 980 TriboIndenter®
- Utilizes existing hardware with advanced software control
- How it works:
 - Approach routine makes contact with the sample
 - Electrostatic actuation to perform experiment and withdraw
 - Between indents, piezo is moved to next position

Large Arrays of Indents up to 6 indents/s



- How it works:
 - Approach routine makes contact with the sample
 - Electrostatic actuation to perform experiment and withdraw
 - Between indents, piezo is moved to next position

XPM Load Functions



Sample Navigation | Load Function | Analysis | Imaging | Automation | Calibration | Preferences | About

Indentation | XPM | Scratch | ScanningWear

File

Number of Tests in X	10	Start Load (µN)	1000.00
Spacing Between Tests in X (µm)	1.00000	End Load (µN)	1000.00
Total Distance in X	9.00000	Vary Load By	Fixed Amt.
Number of Tests in Y	10	Load Time (sec)	0.1000
Spacing Between Tests in Y (µm)	1.00000	Hold Time (sec)	0.1000
Total Distance in Y	9.00000	Unload Time (sec)	0.1000
Lateral Move Speed (µm/s)	10.0000	Data Acquisition Rate (points/sec)	2000.00
Pre-Load (µN)	2.0000	Number of Data Points	96845
Use Imaging Setpoint	<input checked="" type="checkbox"/>	Data Acquisition Rate must be lowered if Number of Data Points exceeds 209715 points/sec.	

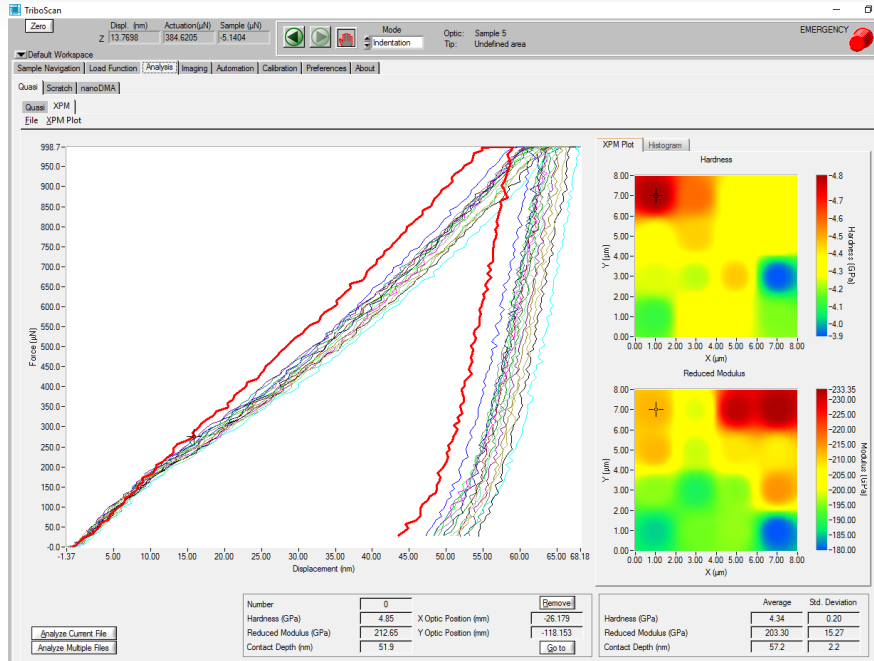
Piezoe Translation Protocol | Constant Direction

Perform XPM

Total Time (sec) | 48.4226
Number of Segments | 401
Preview Size: 10.0000 µm

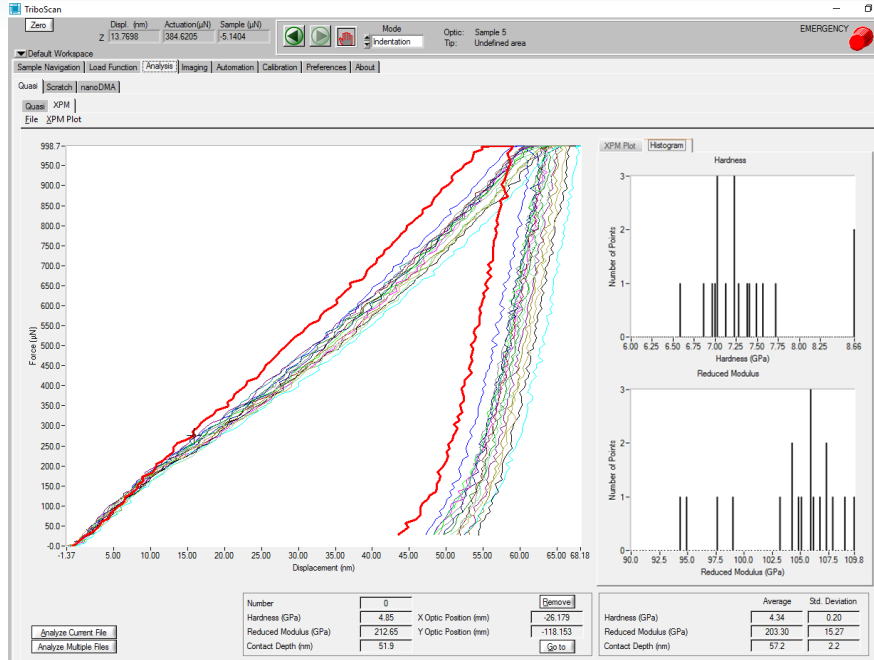
- Rectangular grids, can set spacing and number of indents
- Trapezoid load function only (default 0.1s load-hold-unload, can modify)
- Setpoint variation
- Can vary load linearly or by %
- Lateral move speed can be adjusted
- Limited by piezo scanner range (75 µm) and total number of data points (209715 points)

XPM Analysis



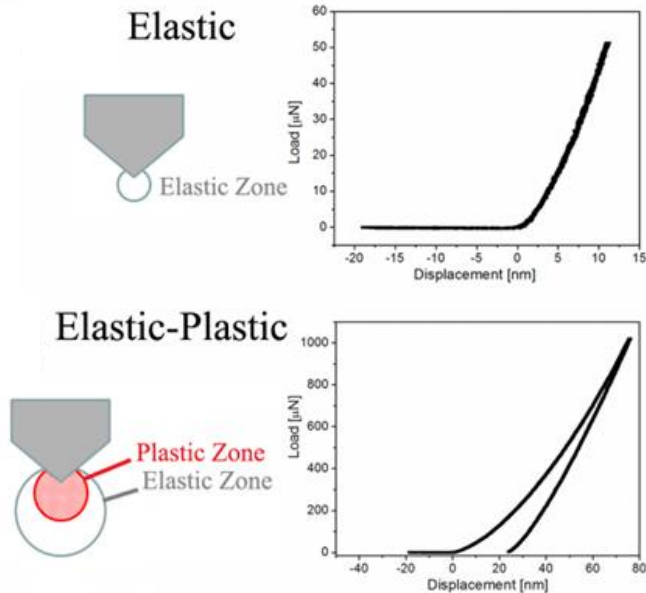
- Input preload, number of segments (2 or 3)
- Analysis using parameters from the quasi subtab
- Quasi subtab allows selection of area function, fitting range, etc.
- Several plotting options, plus histograms and basic statistical analysis
- Automatically generate text file complete with positions (can plot in origin)

XPM Analysis



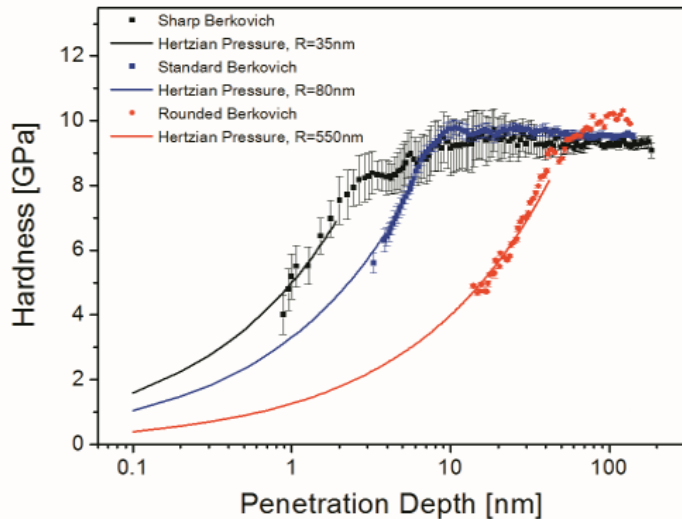
- Input preload, number of segments (2 or 3)
- Analysis using parameters from the quasi subtab
- Quasi subtab allows selection of area function, fitting range, etc.
- Several plotting options, plus histograms and basic statistical analysis
- Automatically generate text file complete with positions (can plot in origin)

Best Practices for Mapping Parameters: Indent Spacing and Tip Shape



- The volume of material whose stress exceeds the yield strength is in the plastic zone – always smaller than the elastic zone
- Elastic properties (Modulus) are not affected by plastic deformation
- Size of plastic zone is dependent on load/depth, indenter geometry and the material being indented
- Indentation size effects result in changes in hardness over shallow depths
- Best solution is to compare with single quasi-static indents

Best Practices for Mapping Parameters: Indent Spacing and Tip Shape

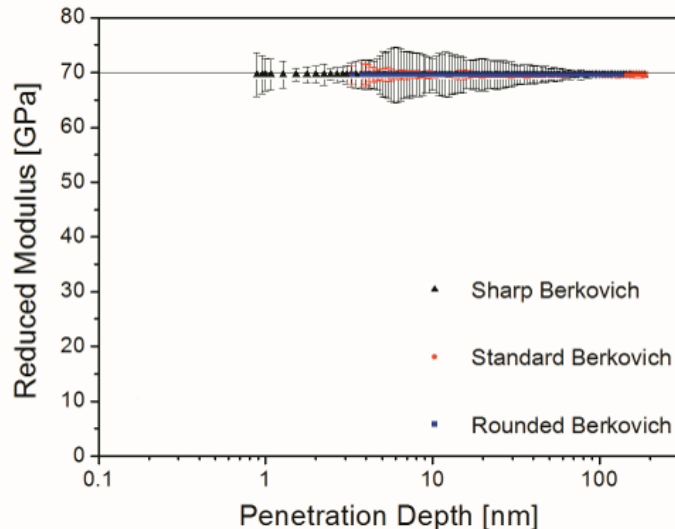


Hardness effect based on tip shape and depth

E.Hinstala, U.Hangen and D.Stauffer, *Advances in Nanoindentation Techniques*, 2018

- The volume of material whose stress exceeds the yield strength is in the plastic zone – always smaller than the elastic zone
- Elastic properties (Modulus) are not affected by plastic deformation
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Best Practices for Mapping Parameters: Indent Spacing and Tip Shape



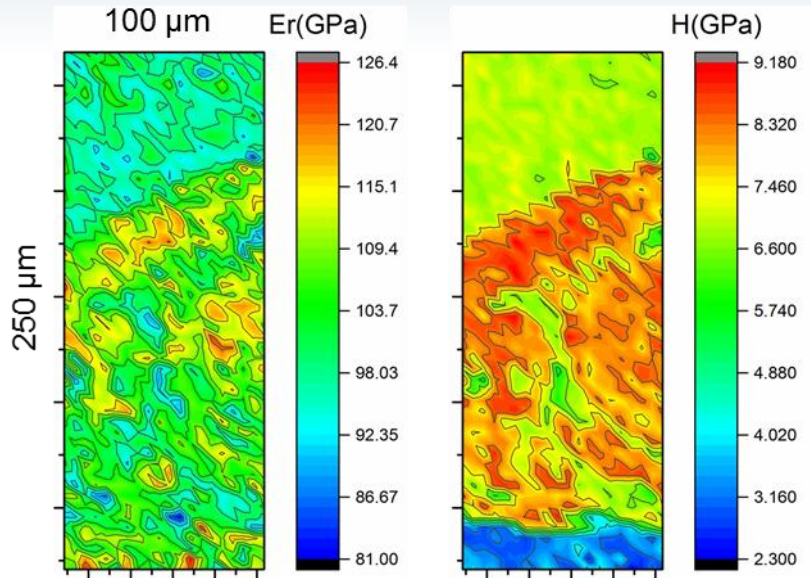
No modulus effect from tip shape

- The volume of material whose stress exceeds the yield strength is in the plastic zone – always smaller than the elastic zone
- Elastic properties (Modulus) are not affected by plastic deformation
- Size of plastic zone is dependent on load/depth, indenter geometry and the material being indented
- Indentation size effects result in changes in hardness over shallow depths
- Best solution is to compare with single quasi-static indents

Applications: Mapping Microstructural Features



Searching for Hard Intermetallic Phases in Weld Zone



Ti - BMG Weld Zone

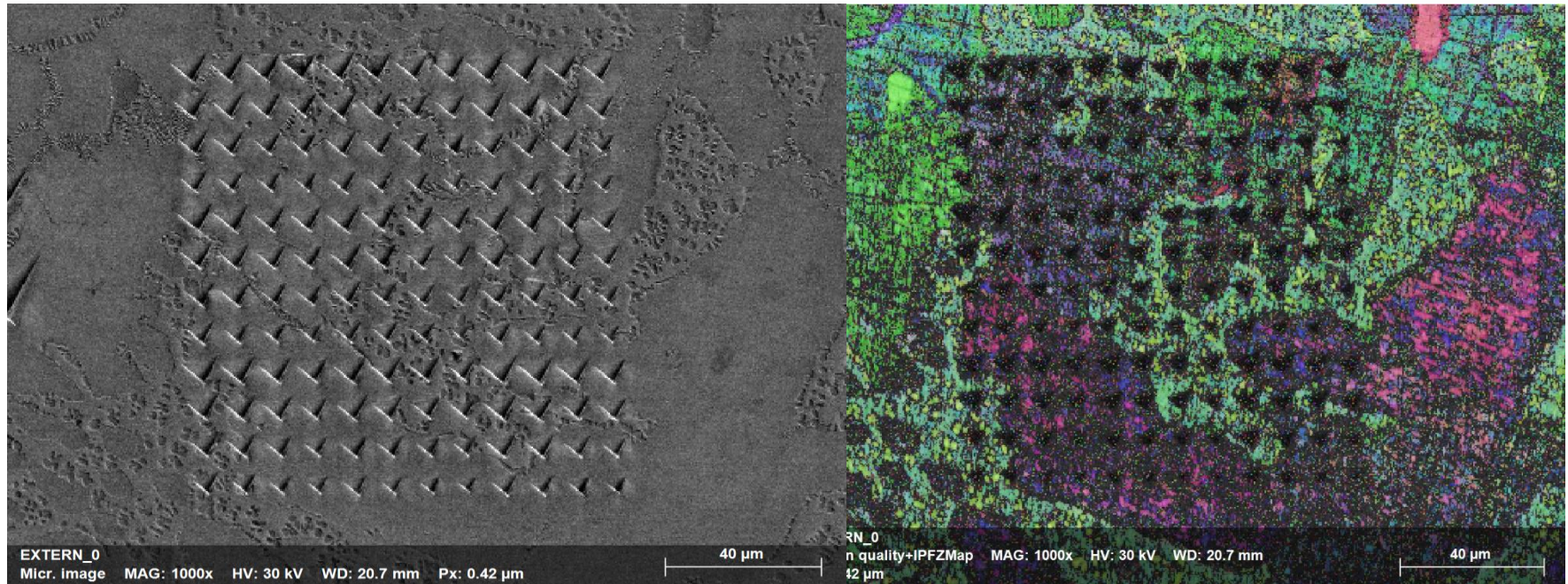
- XPM mapping allows one to explore the properties of different microstructural features of specimens
- It is especially powerful when combined with supplementary structural characterization such as diffraction techniques to make structure property maps
- Main applications is multiphase materials, weld interfaces, and composite materials

Sorensen, D., Pischlar, J., Stevick, J., Hintsala, E., Stauffer, D., Myers, J.C., Keenan, T. and Ramirez, A.J., 2019. Investigation of a dissimilar vitreloy 105 to grade 2 titanium laser weld. *Materials Science and Engineering: A*, 742, pp.33-43

What do we do with this data? "Correlative Microscopy"



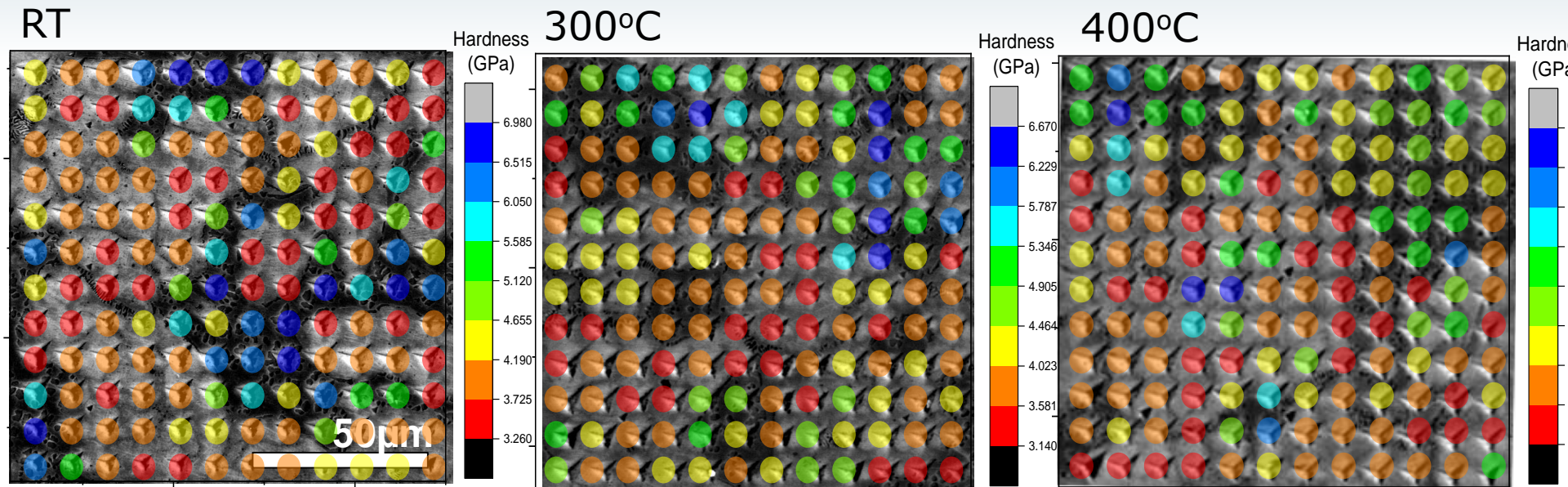
RT



What do we do with this data?

“Cheap labor method”

100% Data usage



FCC: 3.9 ± 0.3 GPa
BCC: 6.0 ± 0.6 GPa

FCC: 3.9 ± 0.4 GPa
BCC: 5.5 ± 0.5 GPa

FCC: 3.9 ± 0.4 GPa
BCC: 5.5 ± 0.6 GPa

K-means Clustering

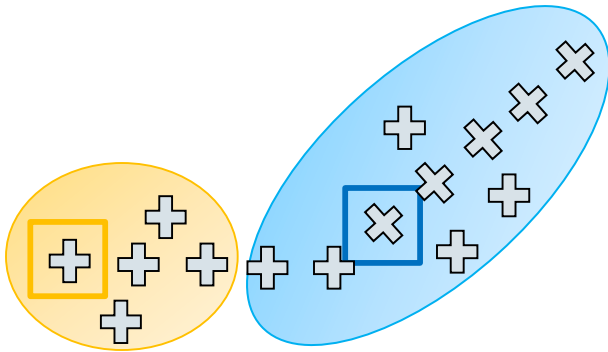


- Algorithm
- Input all points from data set
- How many clusters (k)?
- Starts by random k centroids
- Iteratively finds the closest centroid for each point
- Centroid position final when points don't migrate any further

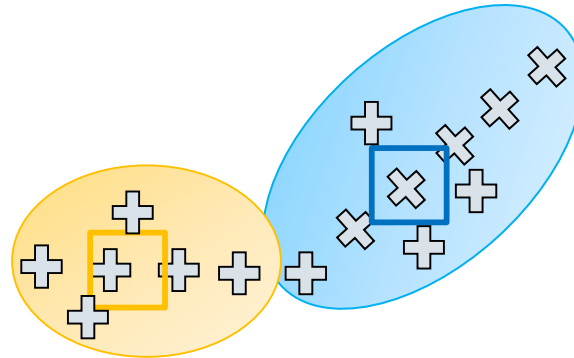
K-means Clustering



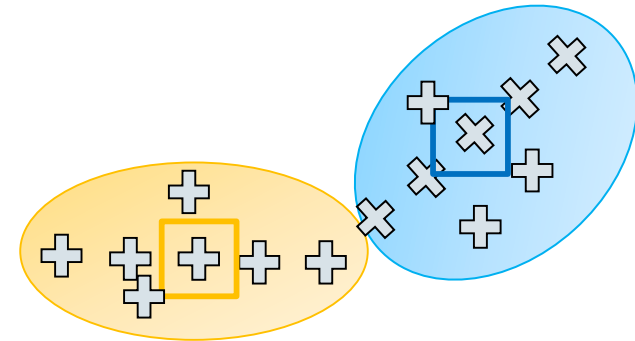
Round - 1



Round - 2



Round - 3



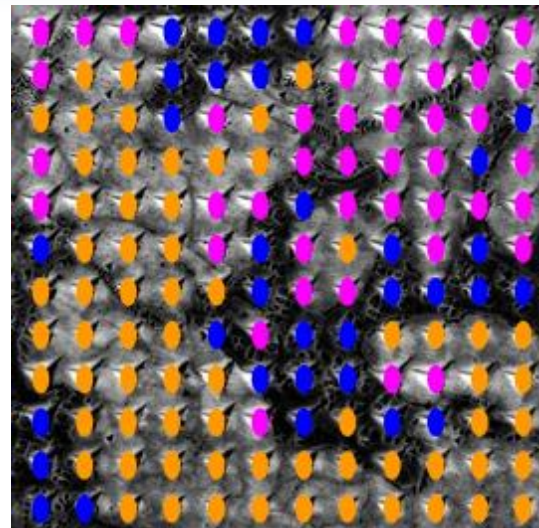
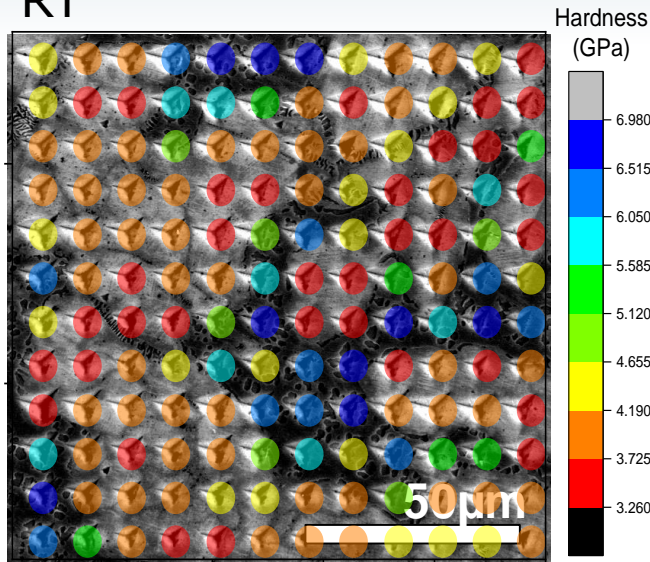
What do we do with this data?

"3 clusters"

72% Data usage: Cluster 1 = Interface

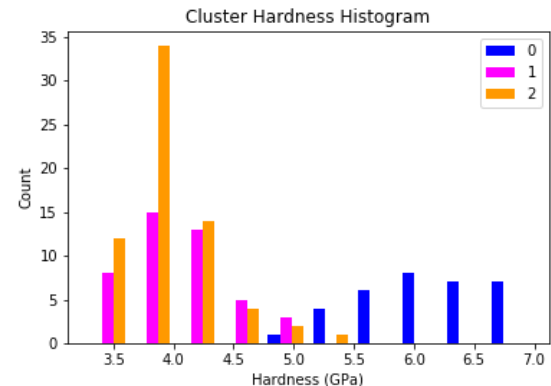
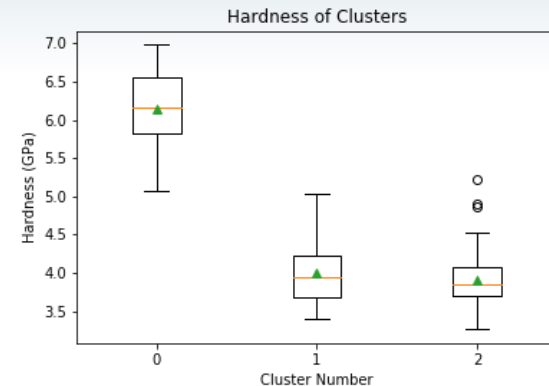


RT



FCC: 3.9 ± 0.3 GPa
BCC: 6.0 ± 0.6 GPa

FCC: 4.0 ± 0.4 GPa
BCC: 6.1 ± 0.5 GPa

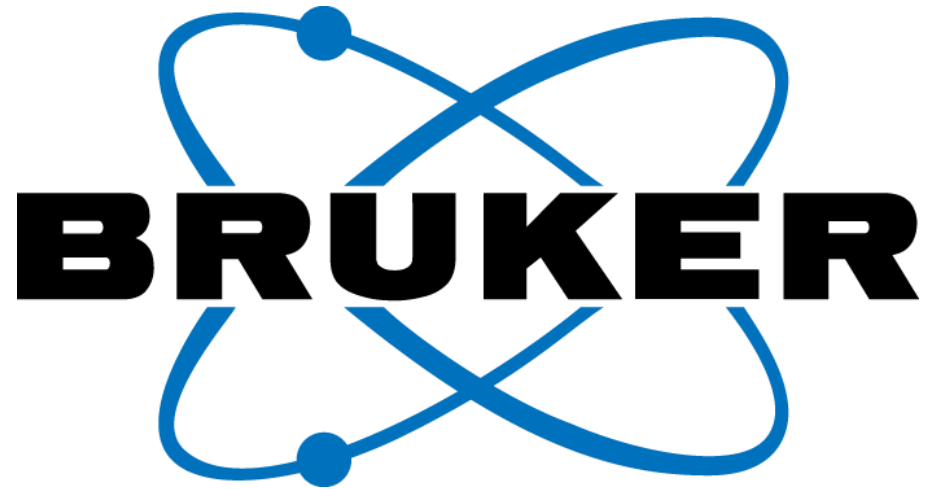


Summary



- The relation of a heterogenous microstructure and its mechanical properties can be assessed at scale with statistical relevance.
- XPM enables new techniques and studies that are impractical with standard indentation.
- The most ideal applications include high-resolution mapping of microstructure, statistical techniques (k-means clustering).
- A fast way to evaluate inhomogeneities and quickly obtain statistically significant data sets.

- Bruker's improved nanoscale dynamic mechanical testing enables:
 - A truly continuous measurement of x (x = hardness, storage modulus, loss modulus, complex modulus, tan-delta, etc.) as a function of contact depth, frequency, and time
 - Drift correction allows for long-duration frequency sweeps and creep tests to be reliably performed
 - PDMS through the melting temperature
 - Polymer film T_g



www.bruker.com