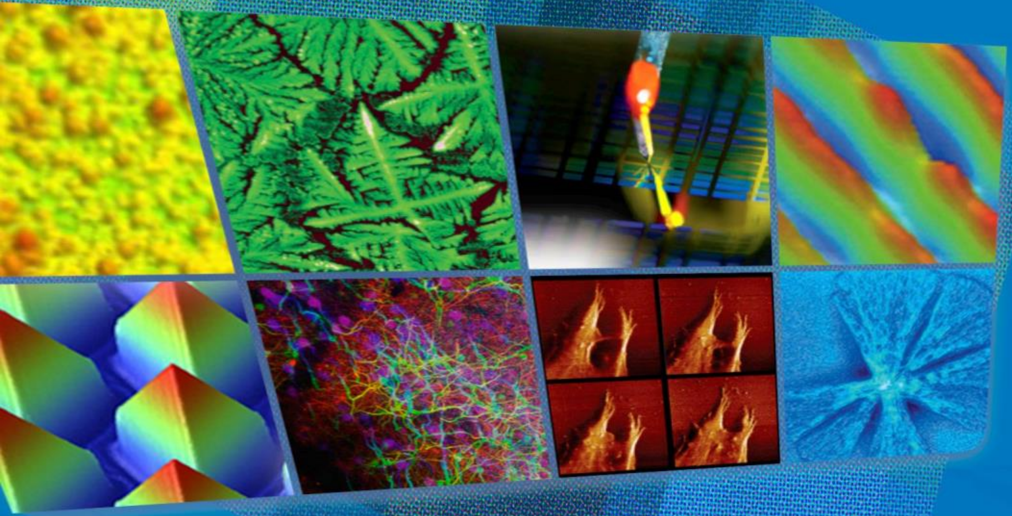
Advanced Nanomechanical Testing Methods

BRUKER



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

Bruker Nano Surfaces Division

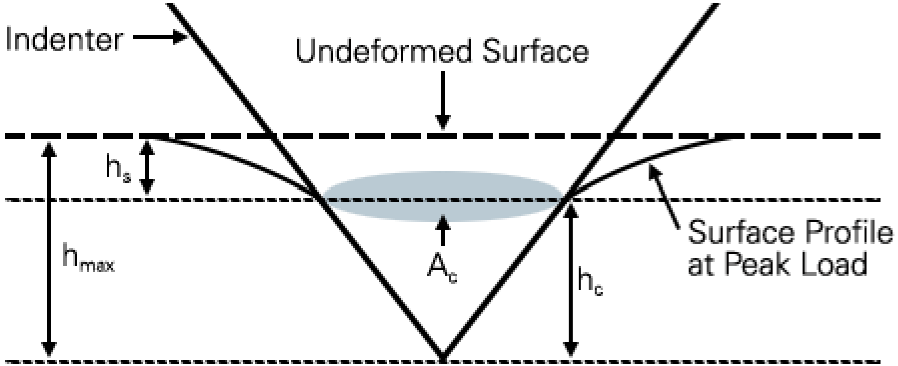
Innovation with Integrity

Radhika Laxminarayana

Outline

- 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

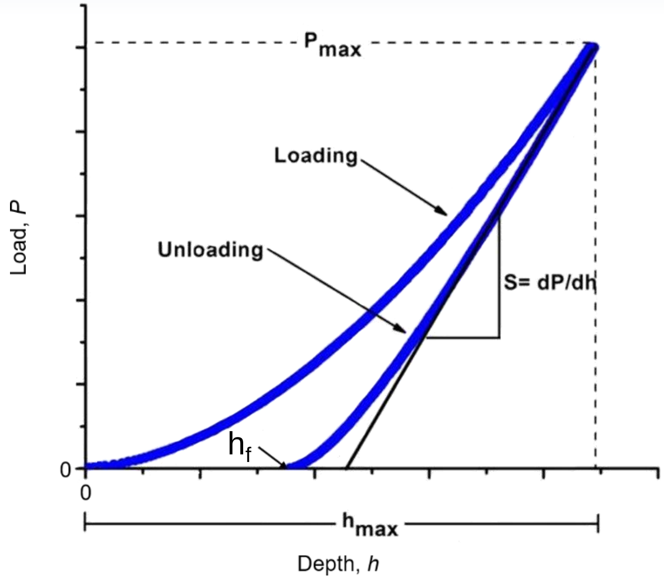


Reduced Modulus

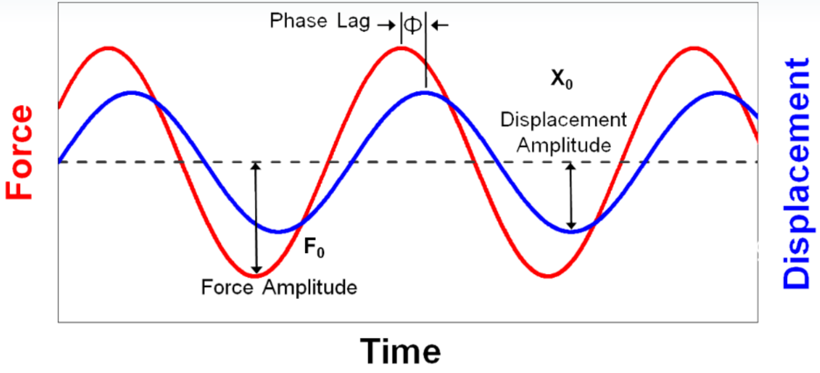
$$E_r = \frac{S\sqrt{\pi}}{2\sqrt{A}}$$

Hardness

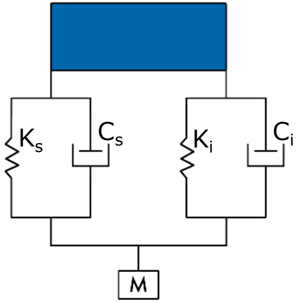
$$H = \frac{P}{A}$$

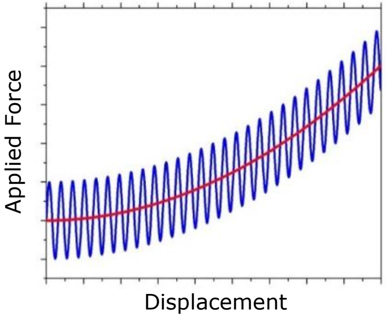


Brief Review of nanoDMA III



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





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

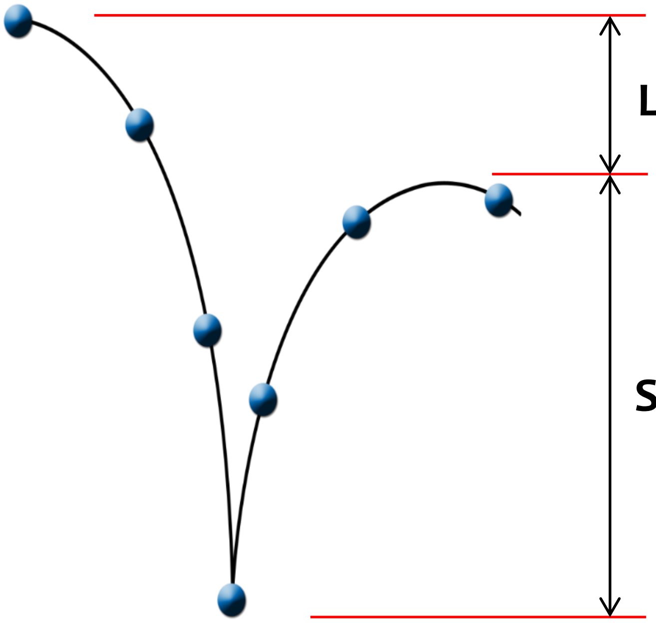
$$\tan \delta = \frac{\omega C_s}{k_s}$$

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

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

Storage and Loss Modulus

Bouncing Ball



Lost Energy

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

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

Stored Energy

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

Quasi-static vs. nanoDMA® III

Quasi-static Indentation

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

Dynamic Indentation

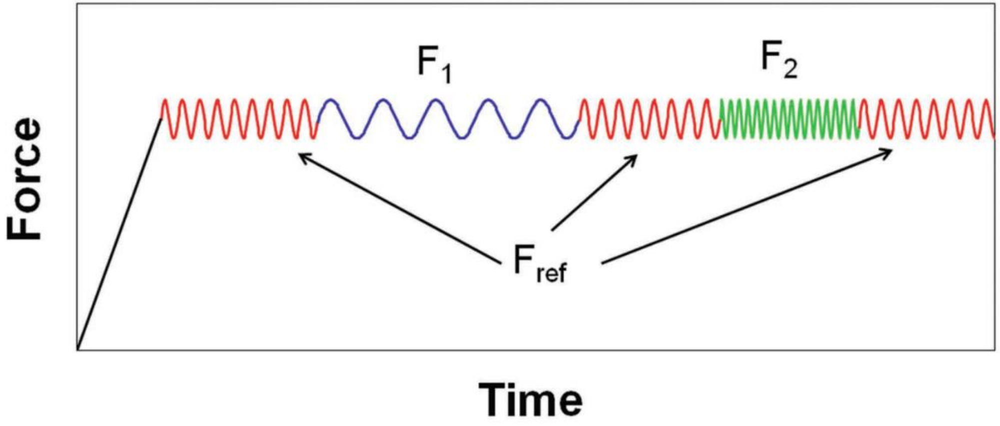
$$E' = \frac{k_s\sqrt{\pi}}{2\sqrt{A_c}}$$

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

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.

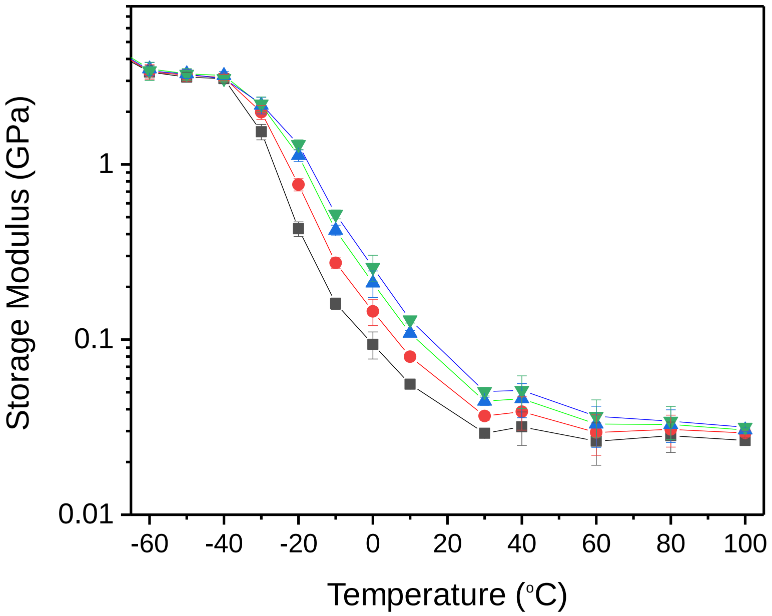


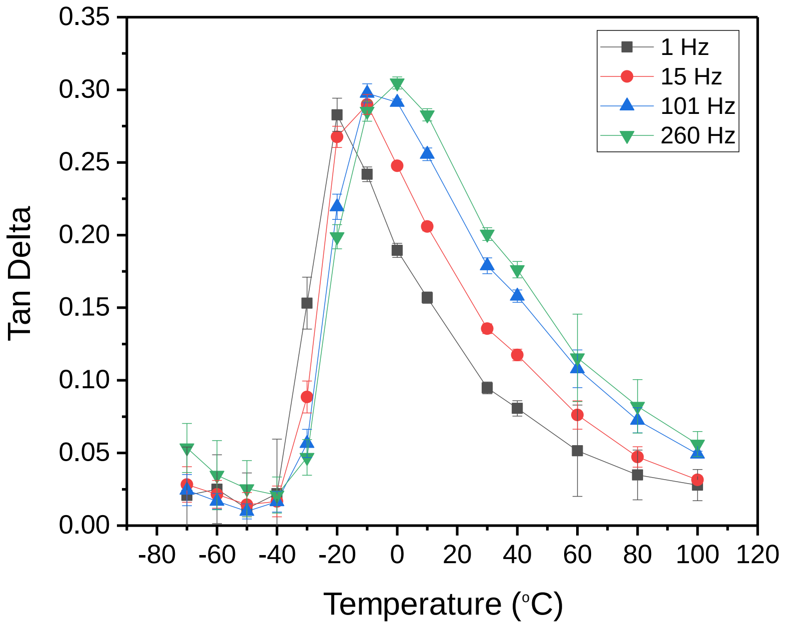
$$E' = \frac{k_s \sqrt{\pi}}{2\sqrt{A_c}}$$

$$k_s = k_s(F)$$

Amorphous Polymer Transitions

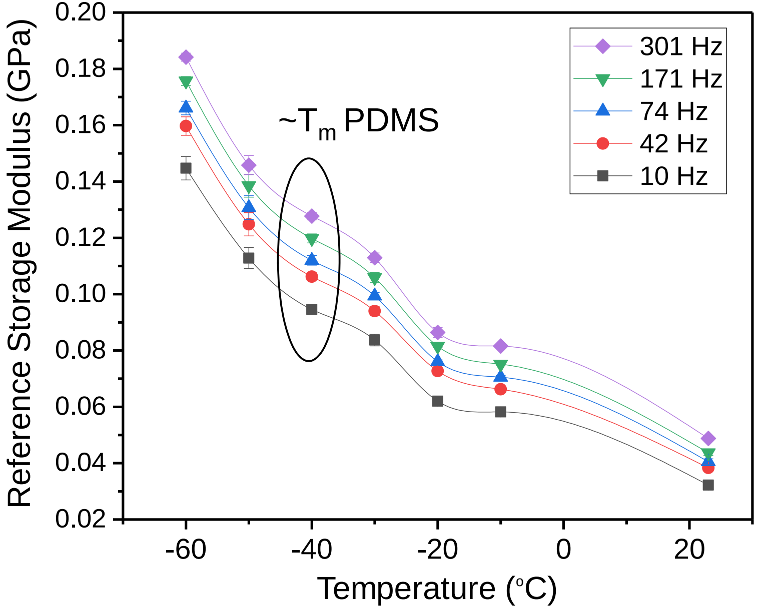
Polymer Film

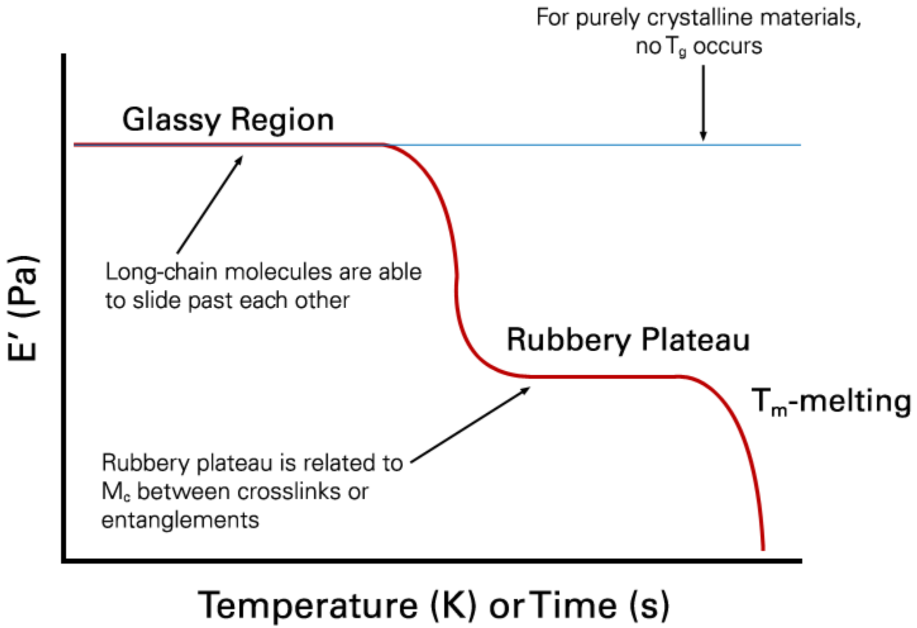




Amorphous Polymer Transitions

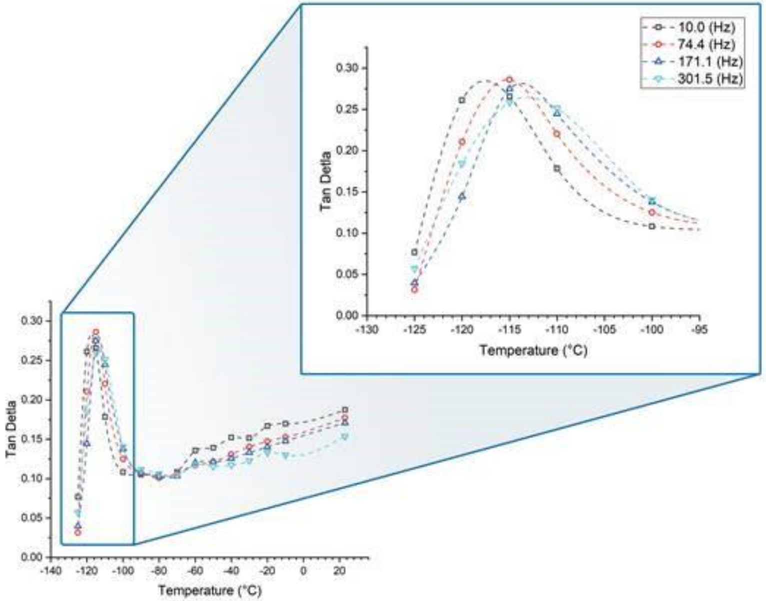
PDMS - Polydimethylsiloxane

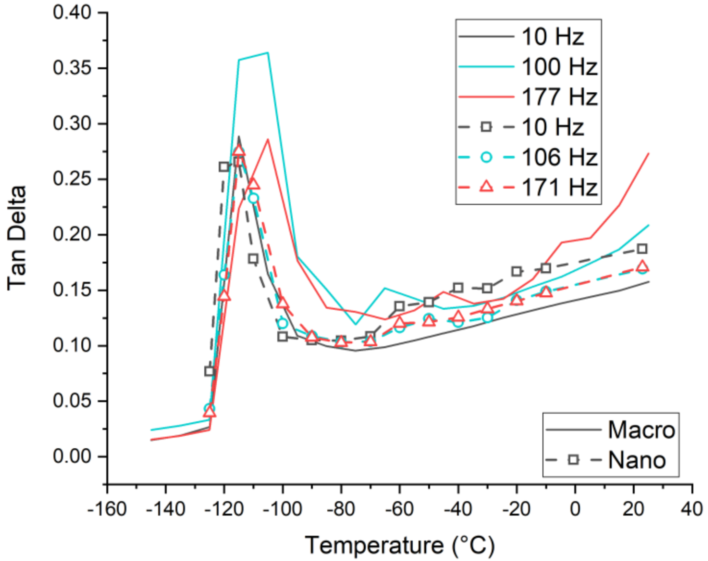




Amorphous Polymer Transitions

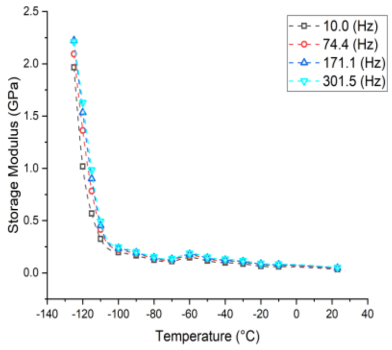
Tg - PDMS

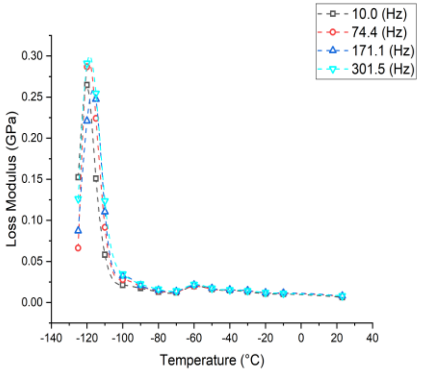




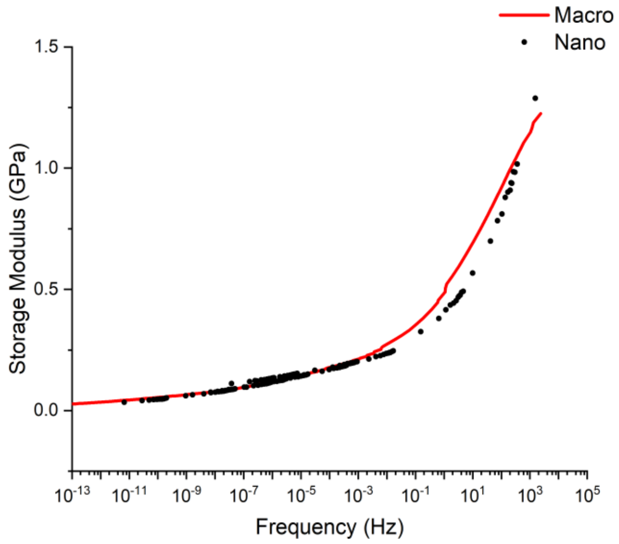
Time - Temperature Superposition (TTS)

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





TTS - Master Curve (PDMS)



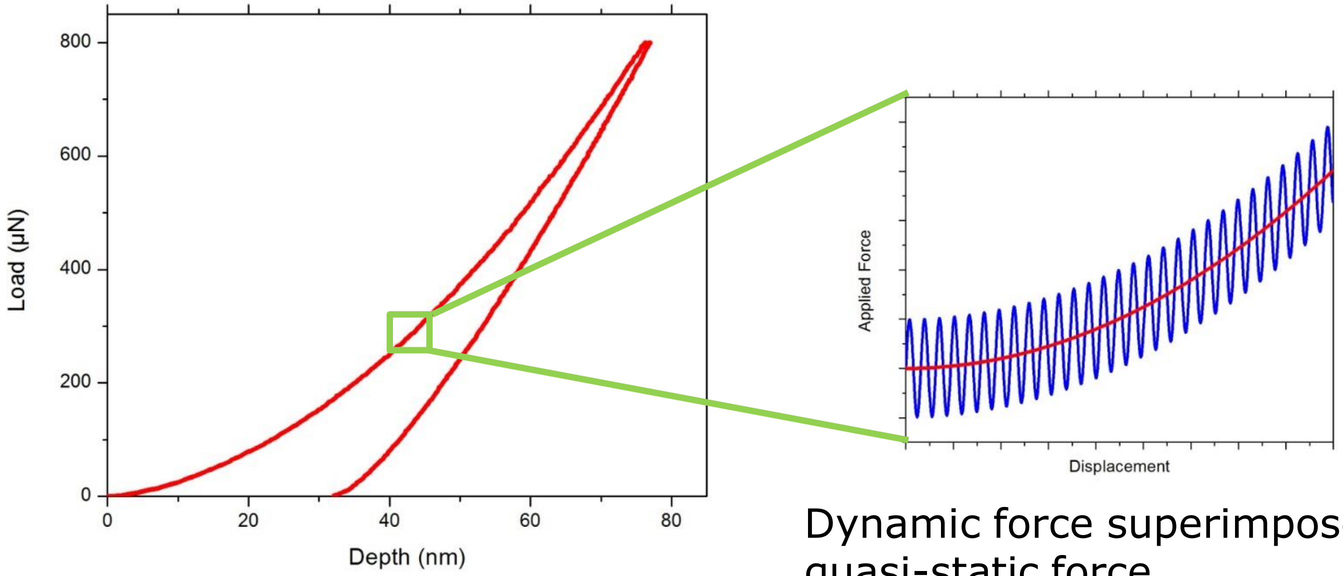
Time-Temperature Superposition using a WLF model.
Comparison between bulk and nanoDMA

- 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

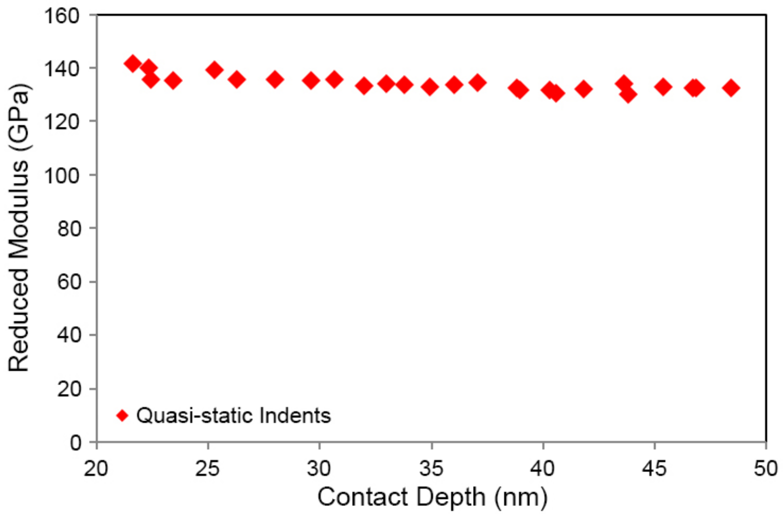
Continuously measure properties as a function of contact depth, frequency, and time.



Dynamic force superimposed on quasi-static force

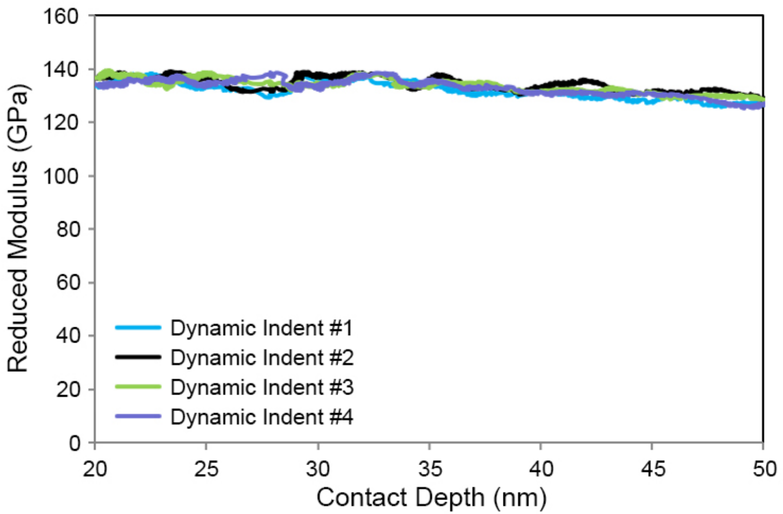
Quasi-static vs. CMX Depth Profiles

Quasi-static Indentation



134.04±2.7 GPa

Dynamic Indentation



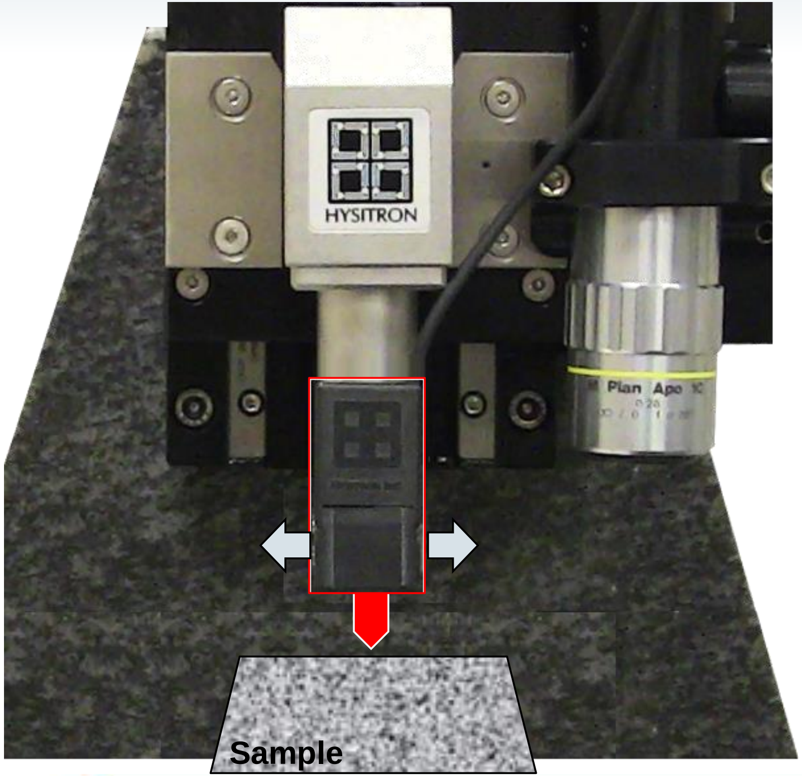
133.88±2.8 GPa

C. Packard and M. Kumar, Colorado School of Mines

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



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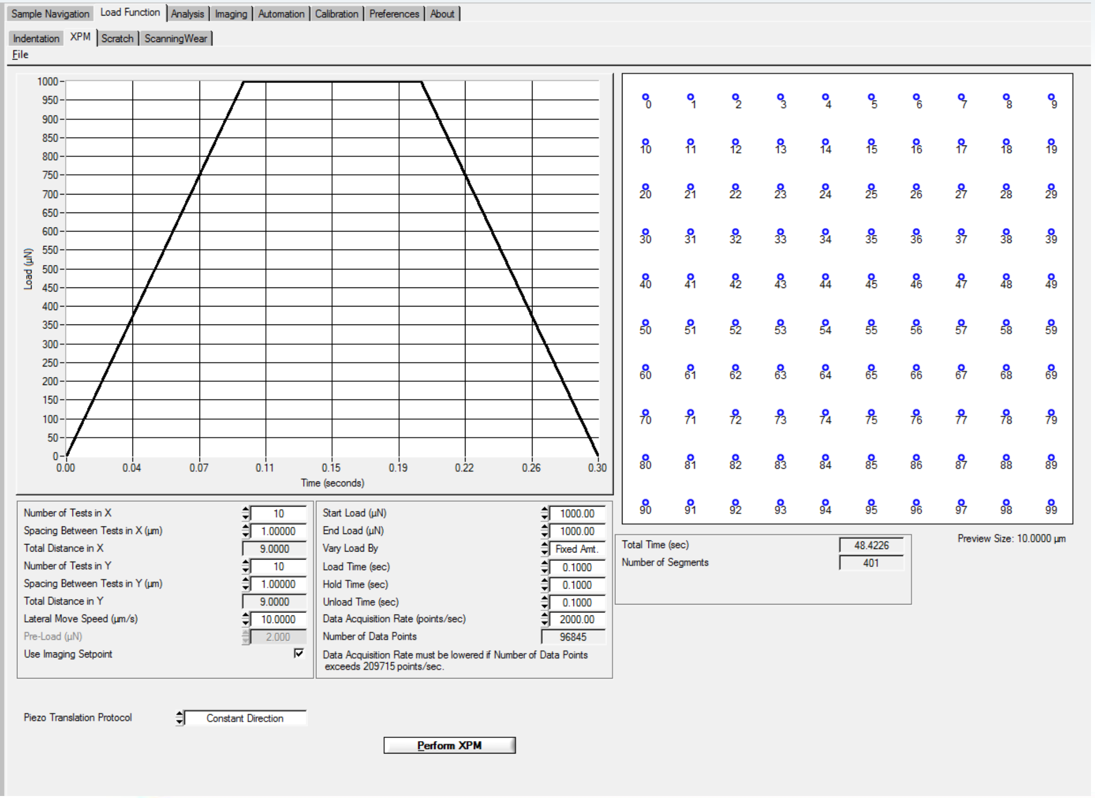
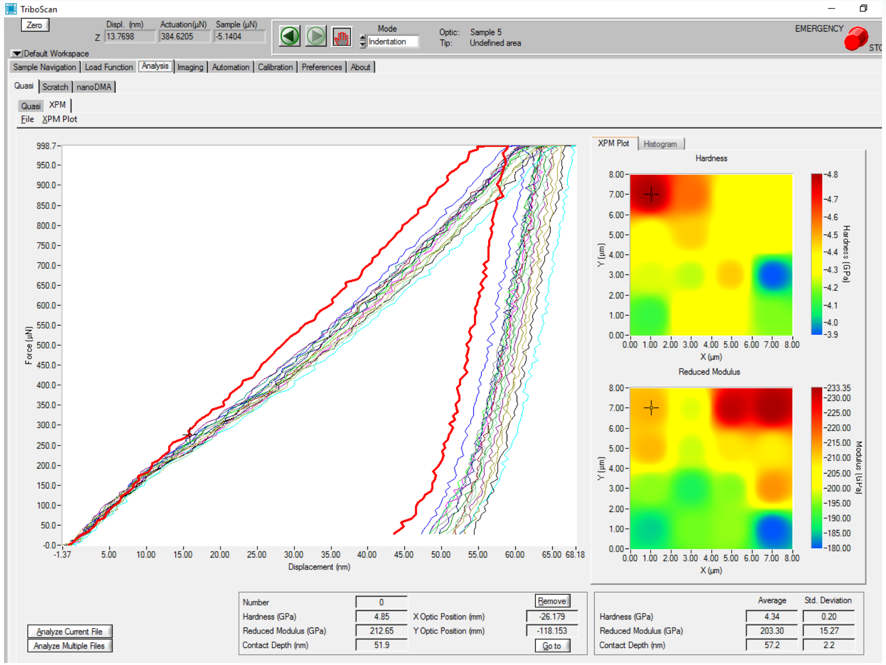
XPM Load Functions



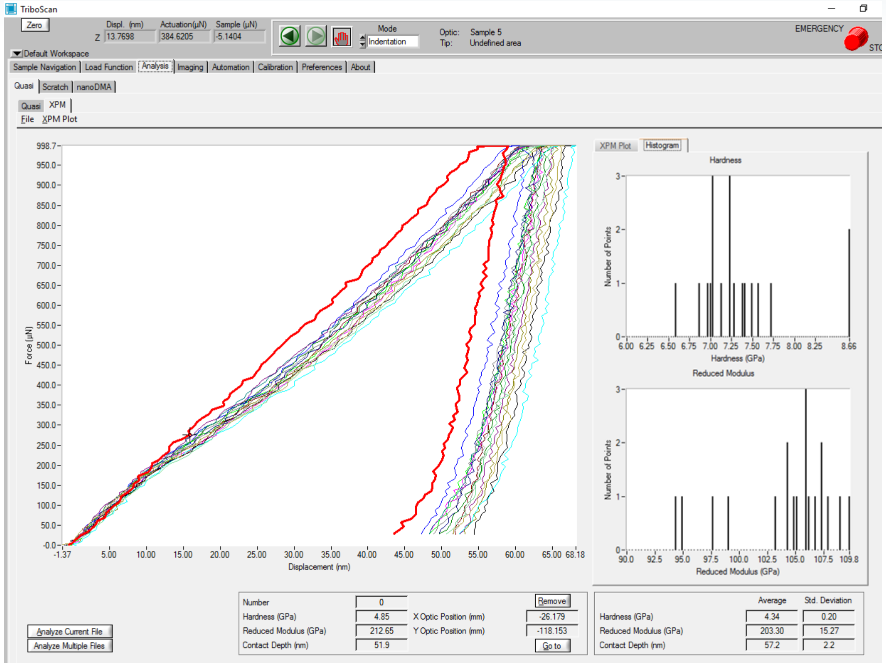
- Rectangular grids, can set spacing and number of indents
- Trapezoid load function only (default 0.1s load-hold-unload, can modify)
- Setpoint variation
- Can vary load linearly or by %
- Lateral move speed can be adjusted
- Limited by piezo scanner range (75 µm) and total number of data points (209715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



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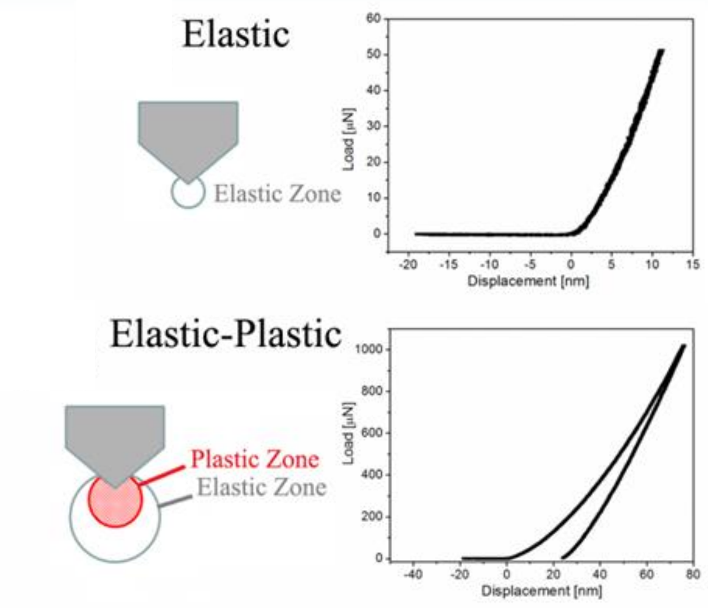
Best Practices for Mapping Parameters:

Indent Spacing and Tip Shape



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
- 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

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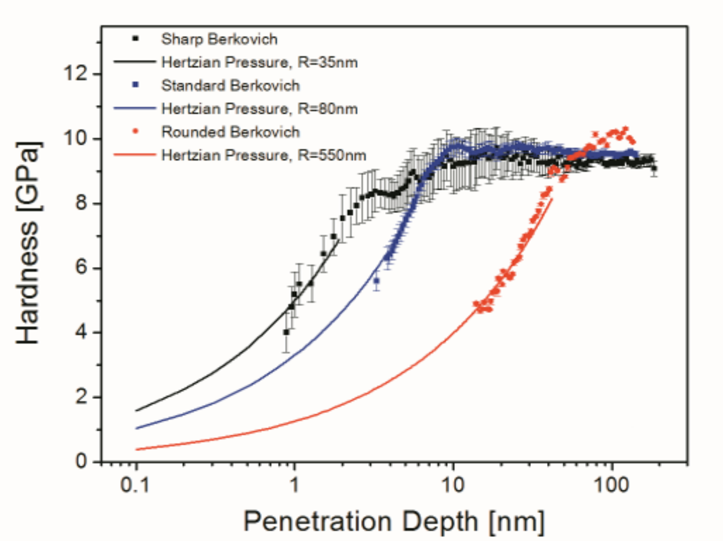

Hardness effect based on tip shape and depth

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

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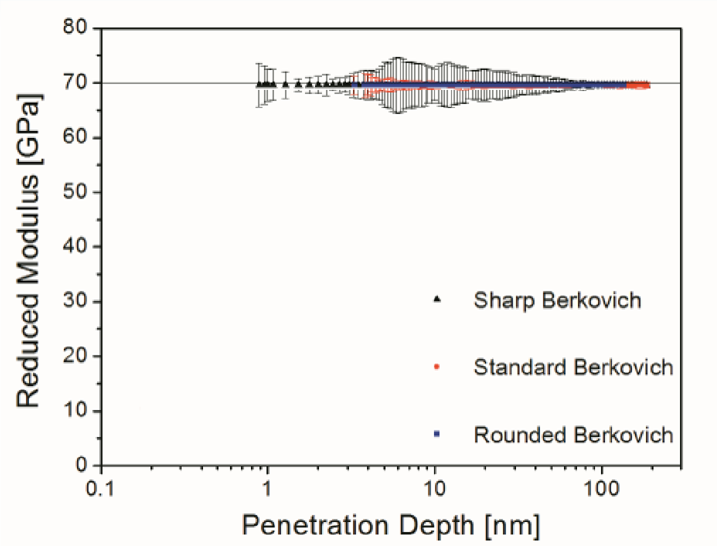

No modulus effect from tip shape

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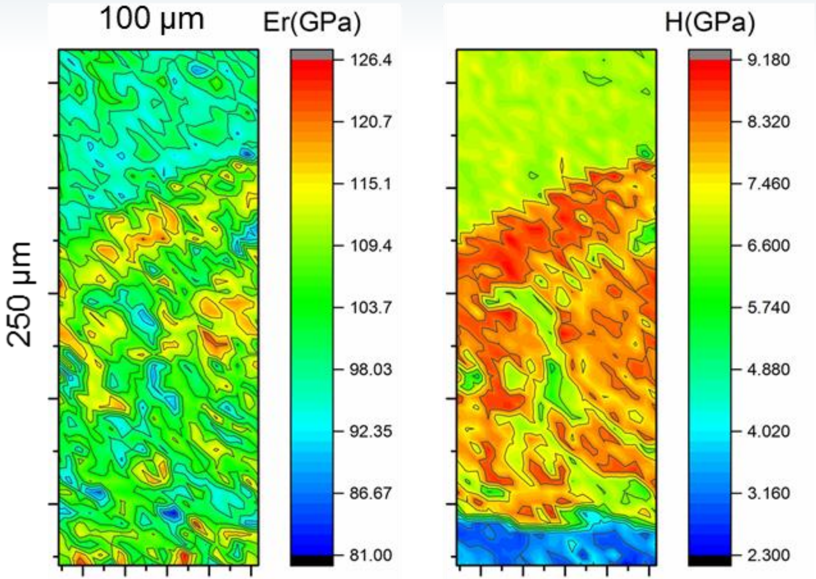
E.Hinstala, U.Hangen and D.Stauffer, Advances in Nanoindentation Techniques, 2018

Applications: Mapping Microstructural Features

Searching for Hard Intermetallic Phases in Weld Zone



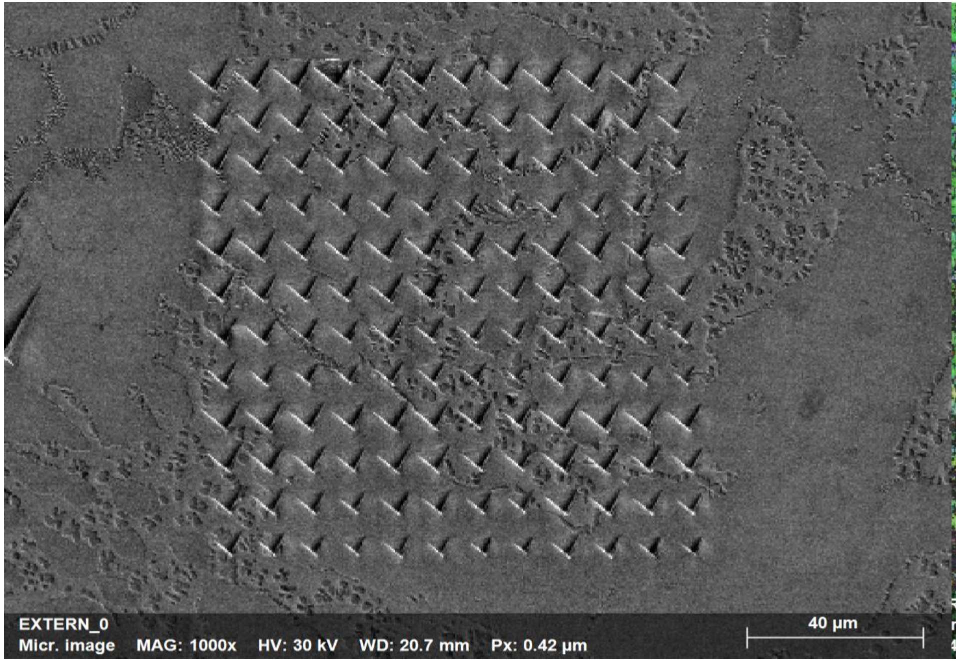
Ti - BMG Weld Zone

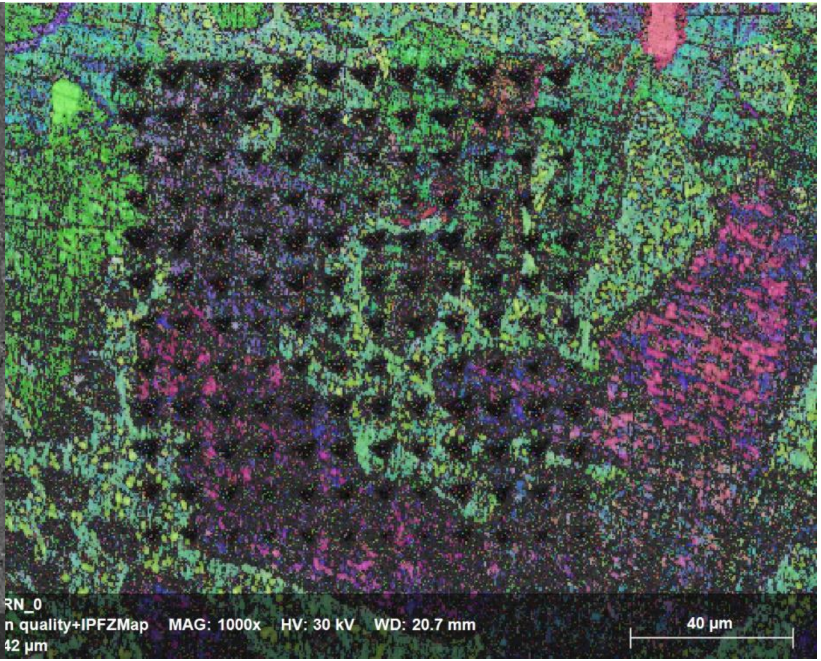
- XPM mapping allows one to explore the properties of different microstructural features of specimens
- It is especially powerful when combined with supplementary structural characterization such as diffraction techniques to make structure property maps
- Main applications is multiphase materials, weld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

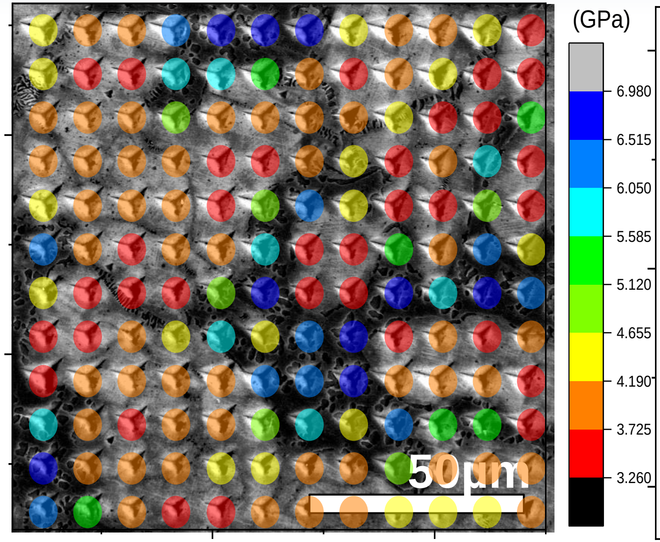




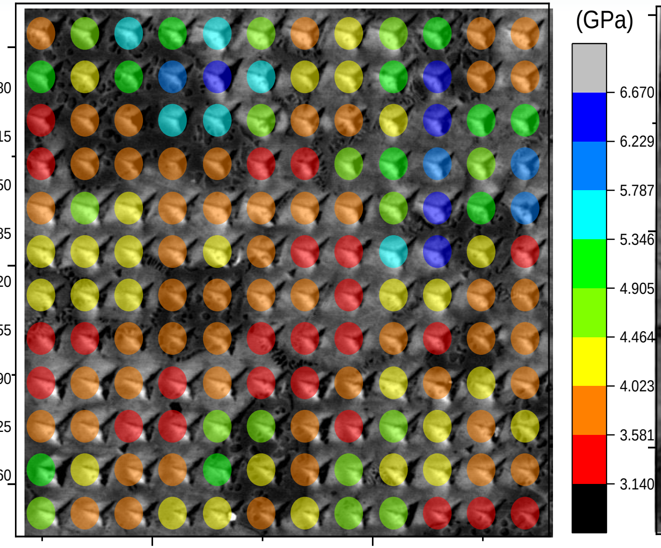
What do we do with this data?
"Cheap labor method"

100% Data usage

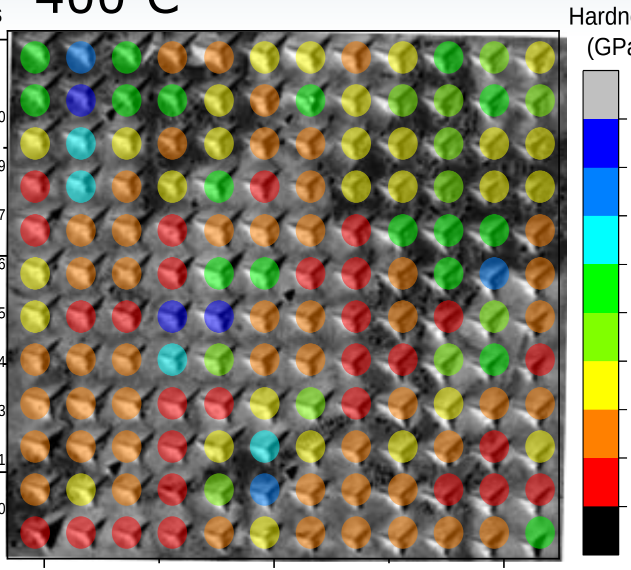
RT



300°C



400°C



FCC: 3.9± 0.3 GPa
BCC: 6.0± 0.6 GPa

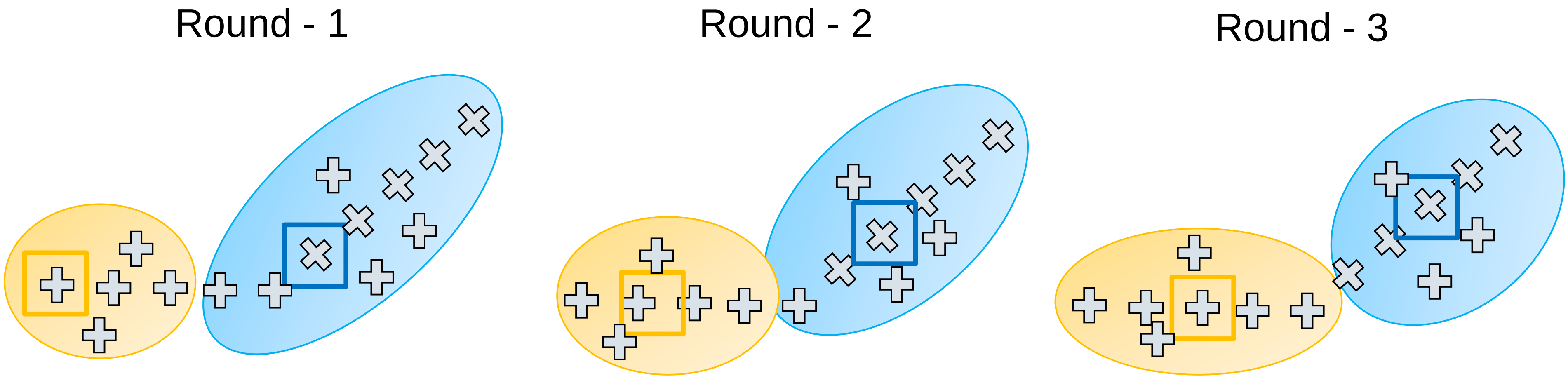
FCC: 3.9± 0.4 GPa
BCC: 5.5± 0.5 GPa

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

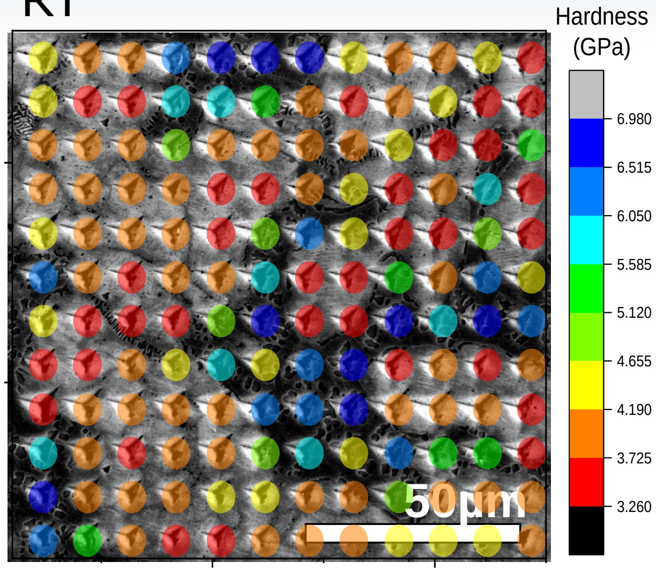


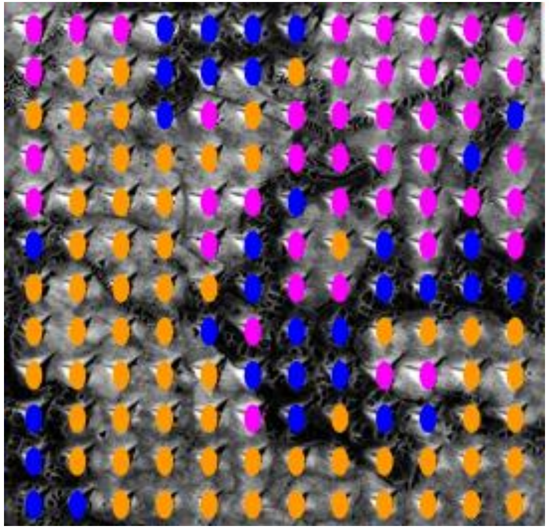
What do we do with this data?

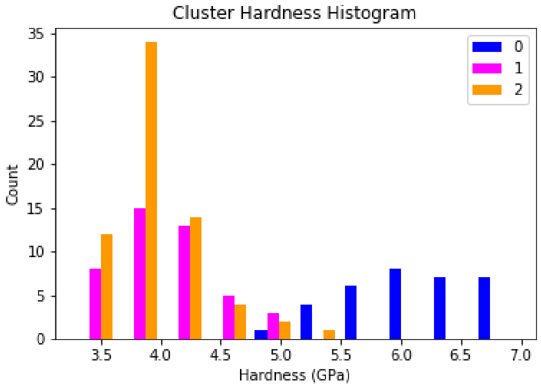
"3 clusters"

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.

Summary

- 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 Tg

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