

# CORRELATIVE NANOMECHANICAL MEASUREMENTS

**Douglas Stauffer, E. Hintsala, S.A.S. Asif**

Hysitron, Inc.

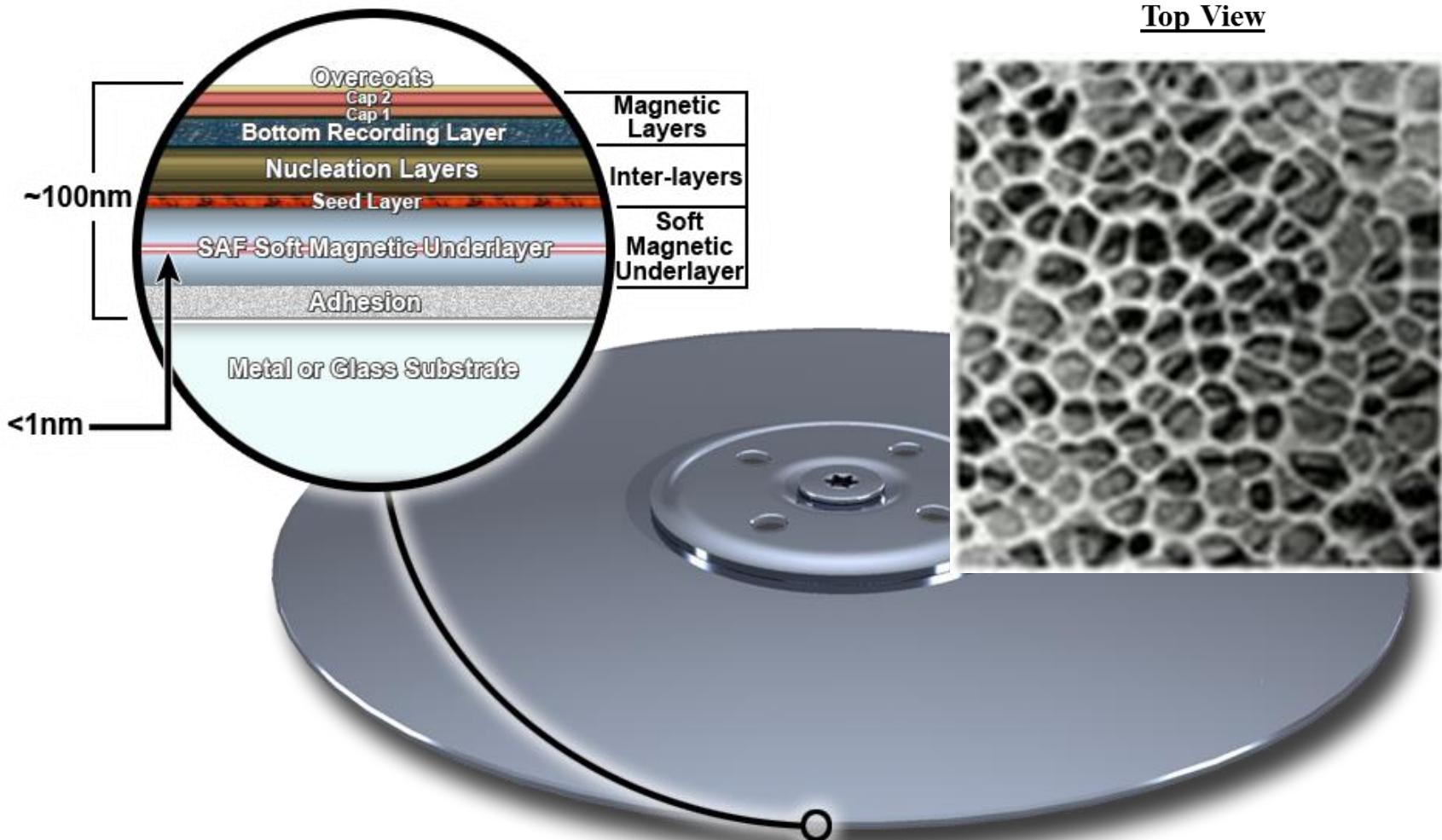
**Daniel Bufford, William Mook, Khalid Hattar**

Sandia National Laboratories

# CORRELATIVE NANOMECHANICAL MEASUREMENTS FOR COMPLEX ENGINEERED SYSTEMS

Anonymous Collaborators!!!

# Complex, Engineered, System



# I. Understanding damage mechanisms

## 1. Co-deposited films for a direct comparison

between *in* and *ex situ* mechanical testing

## 2. Indentation to look at controlled stress-field statistics

- Hardness and modulus as a function of depth/strain

## 3. *In Situ* TEM testing to look at deformation mechanisms

## 4. *In Situ* TEM scratch testing

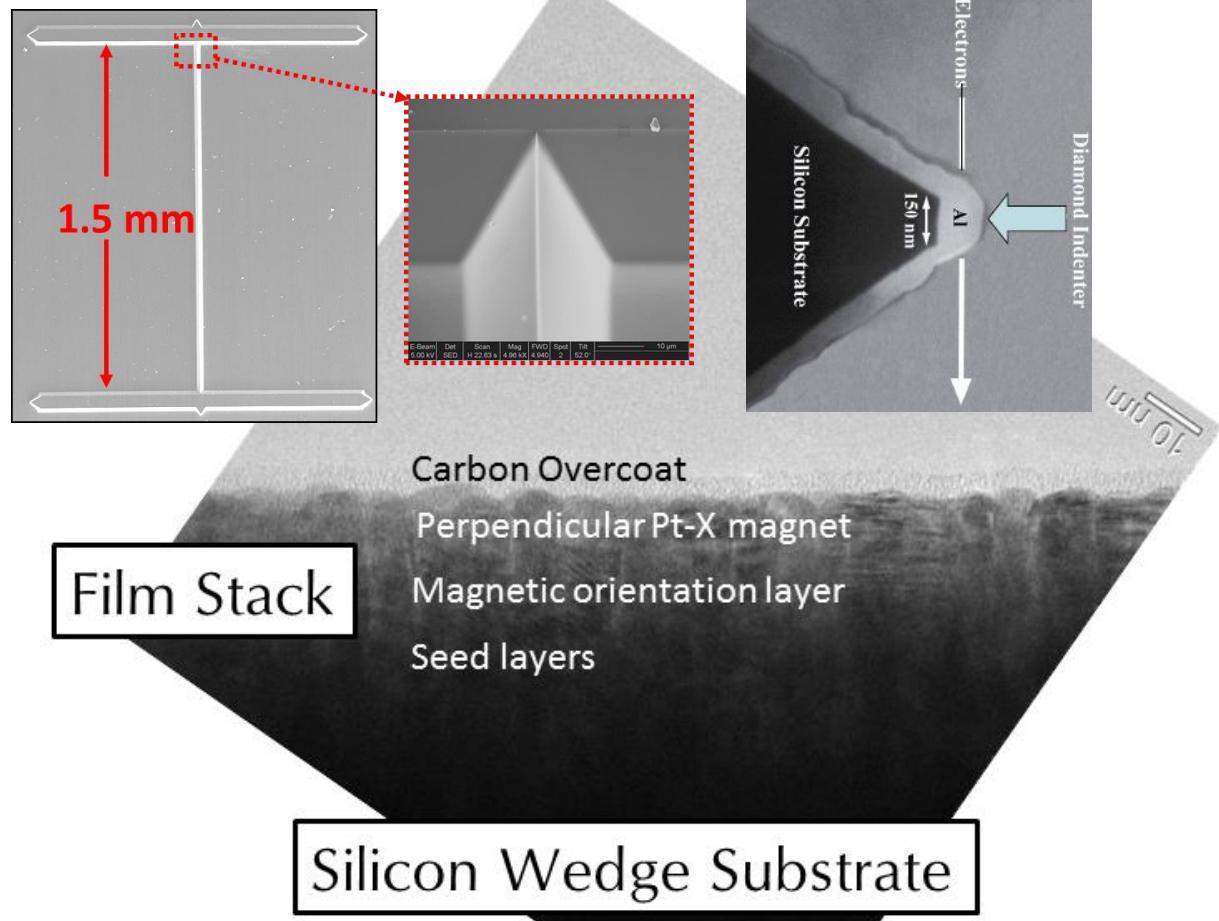
- – moving towards realism in measurements

# Film Deposition

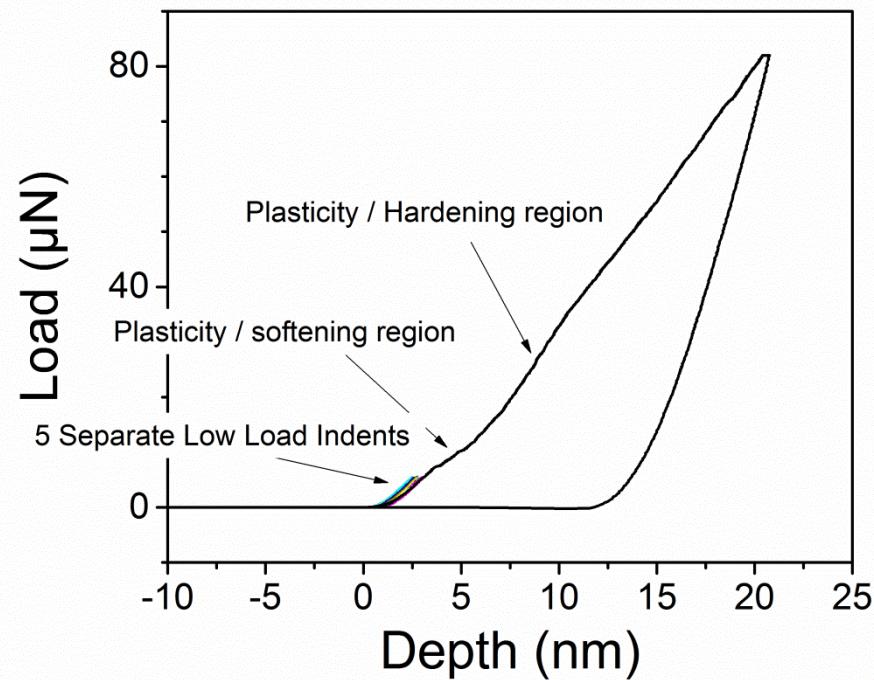
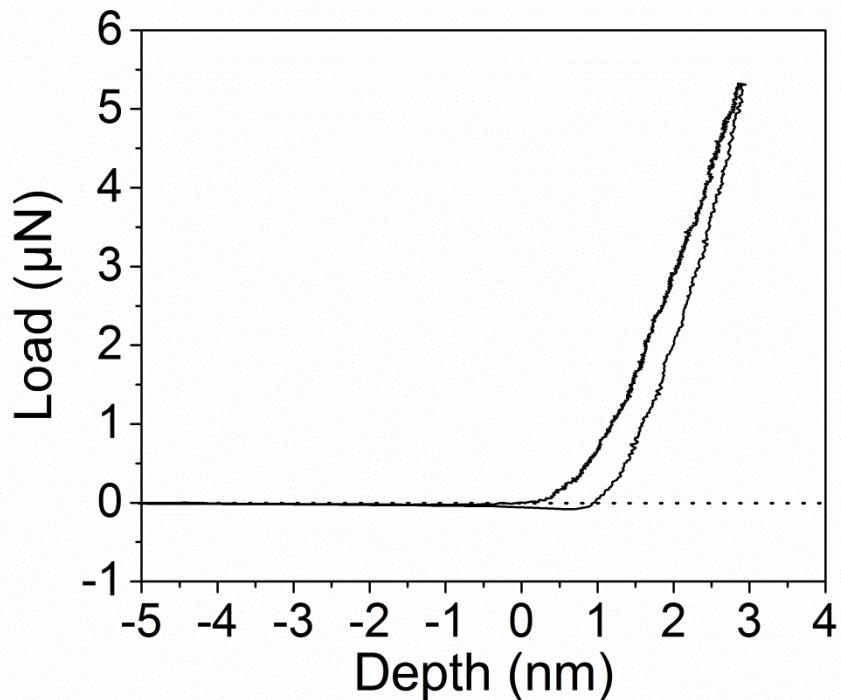
**TEM PicoIndenter®**



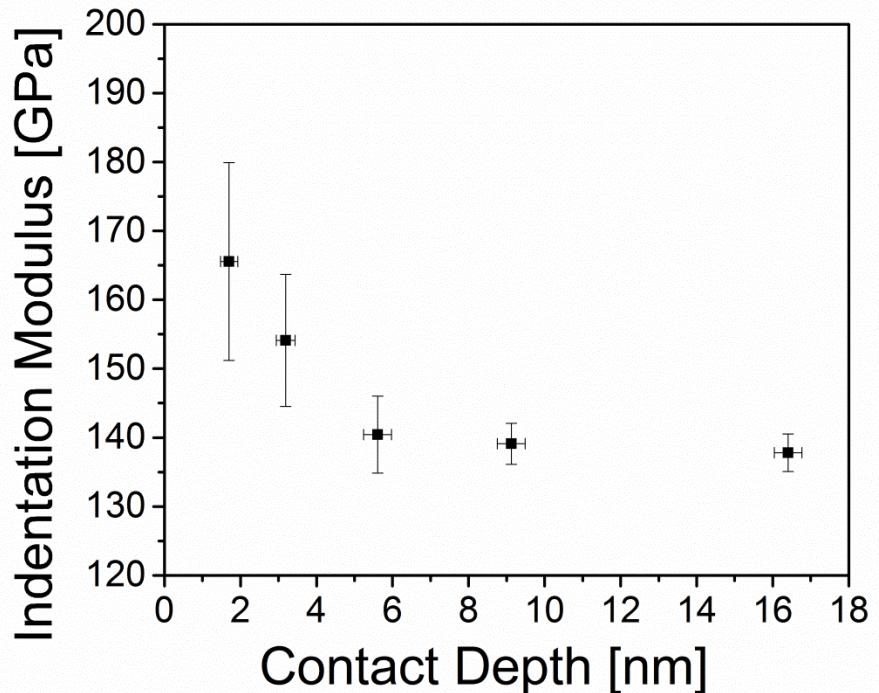
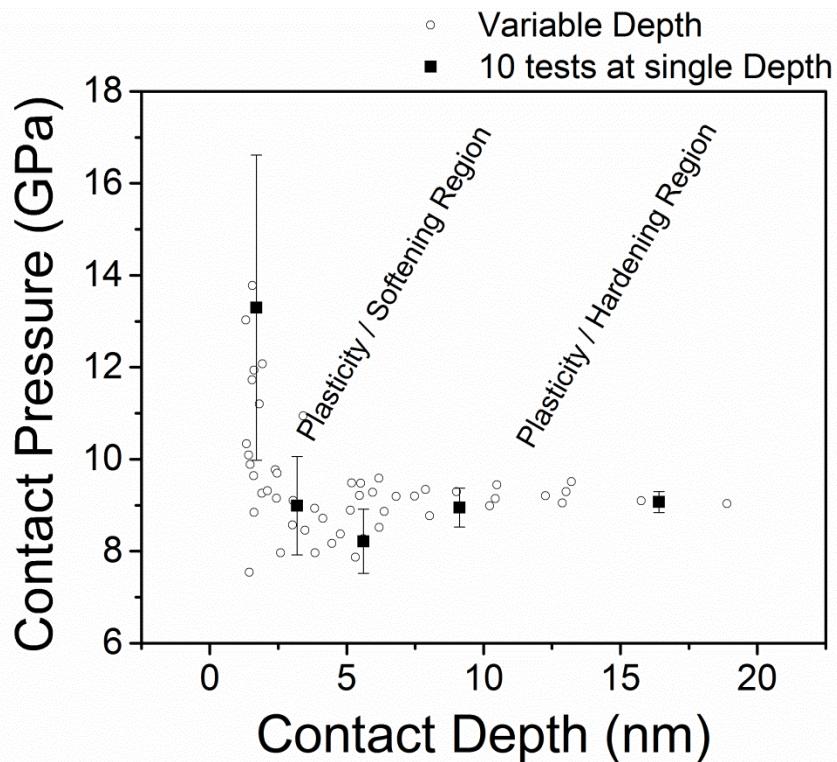
**PI 95**



# Indentation



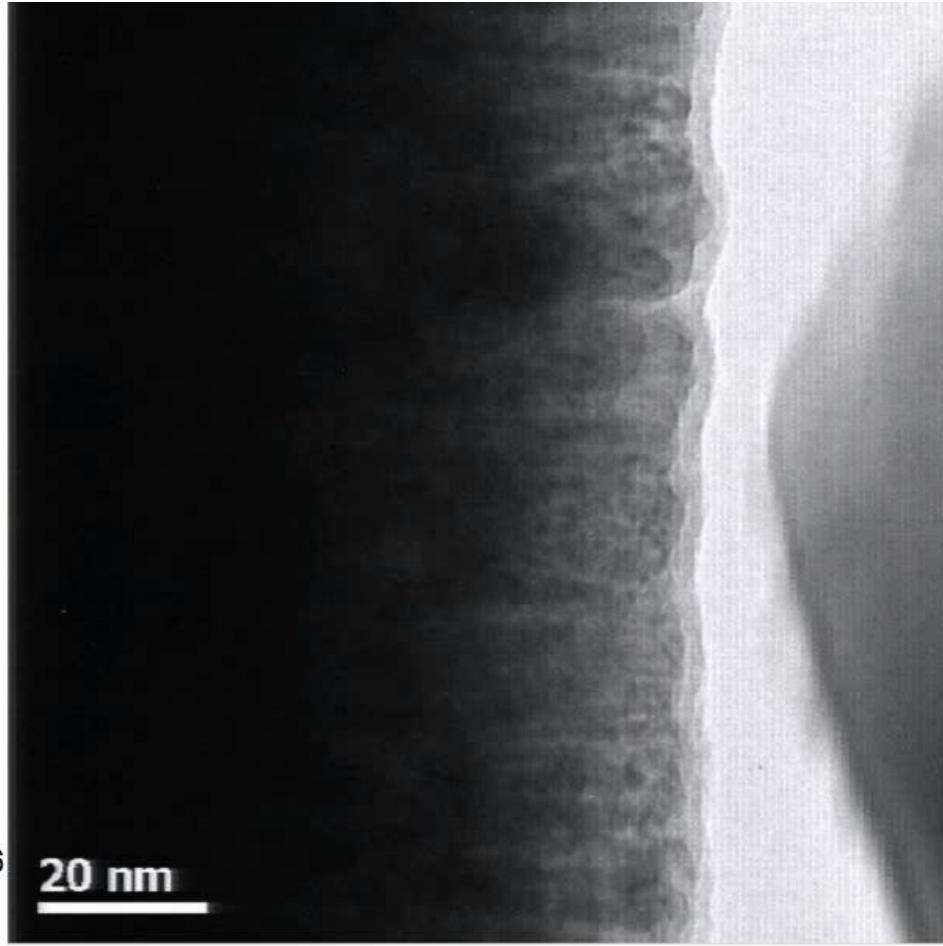
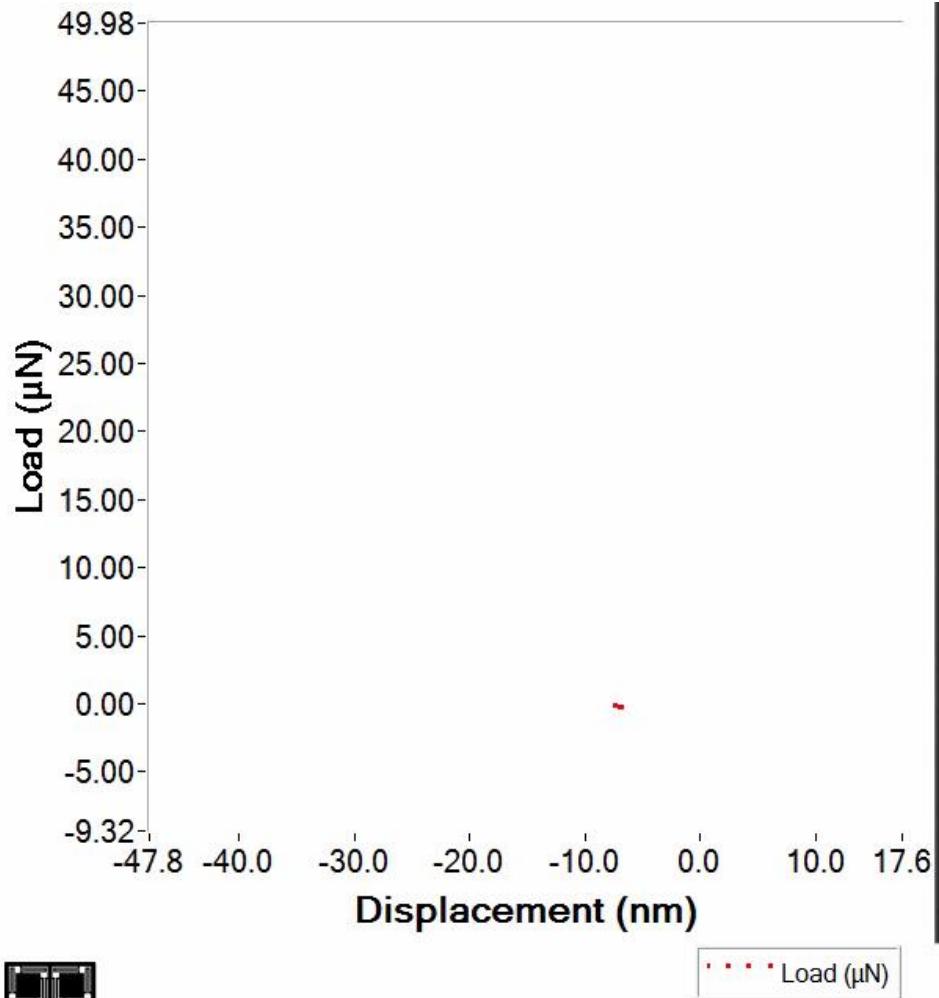
# Hardness/Modulus



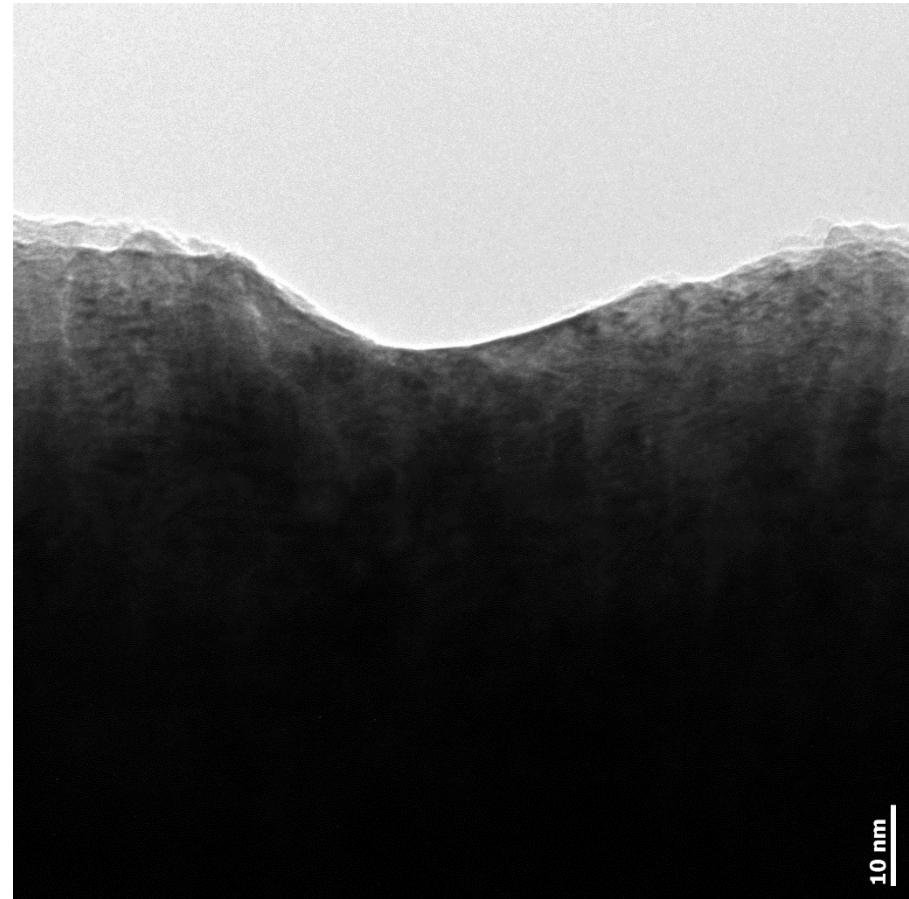
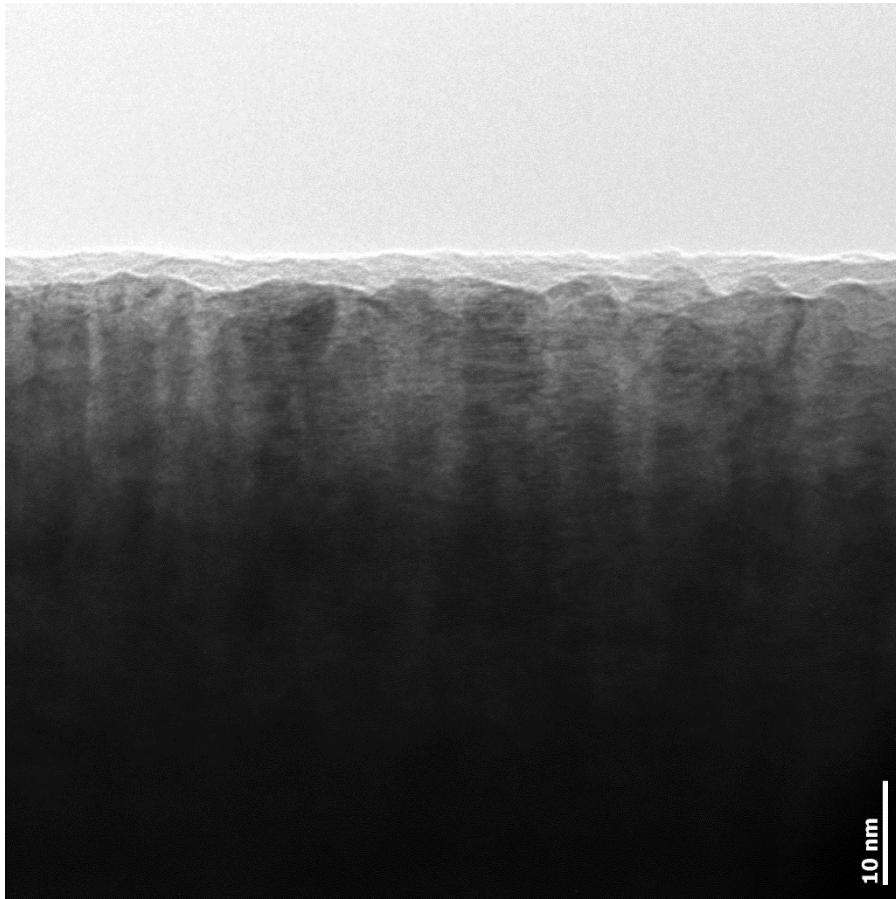
# *In Situ* - Low Load



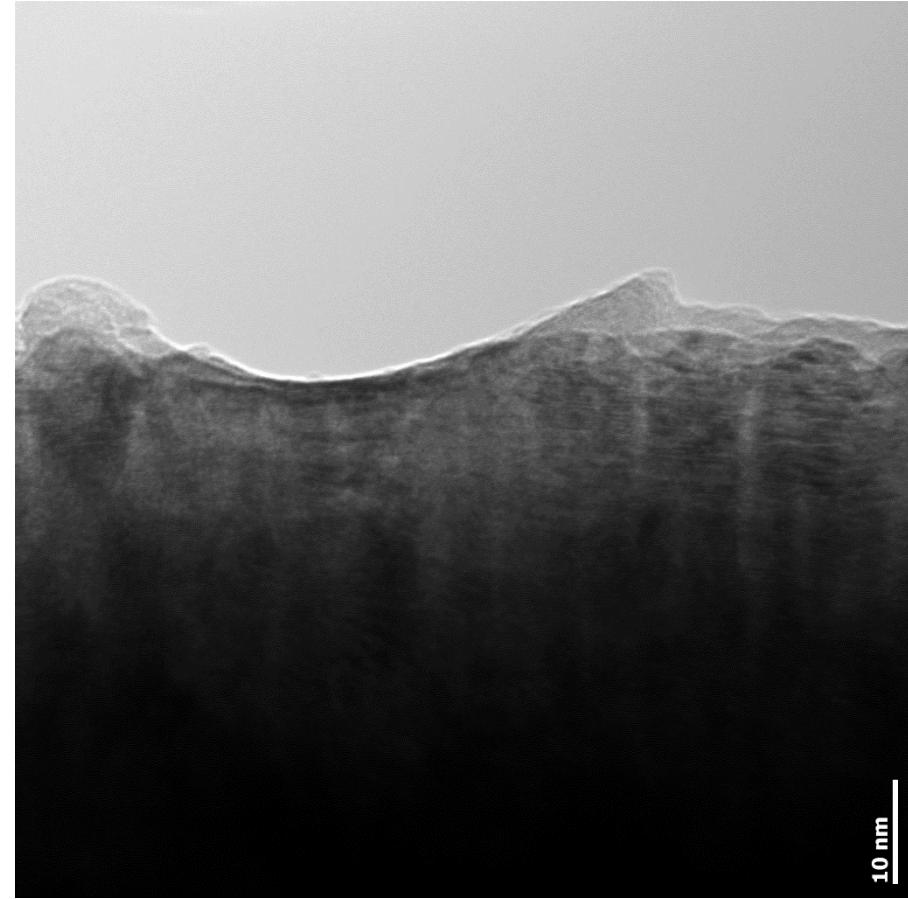
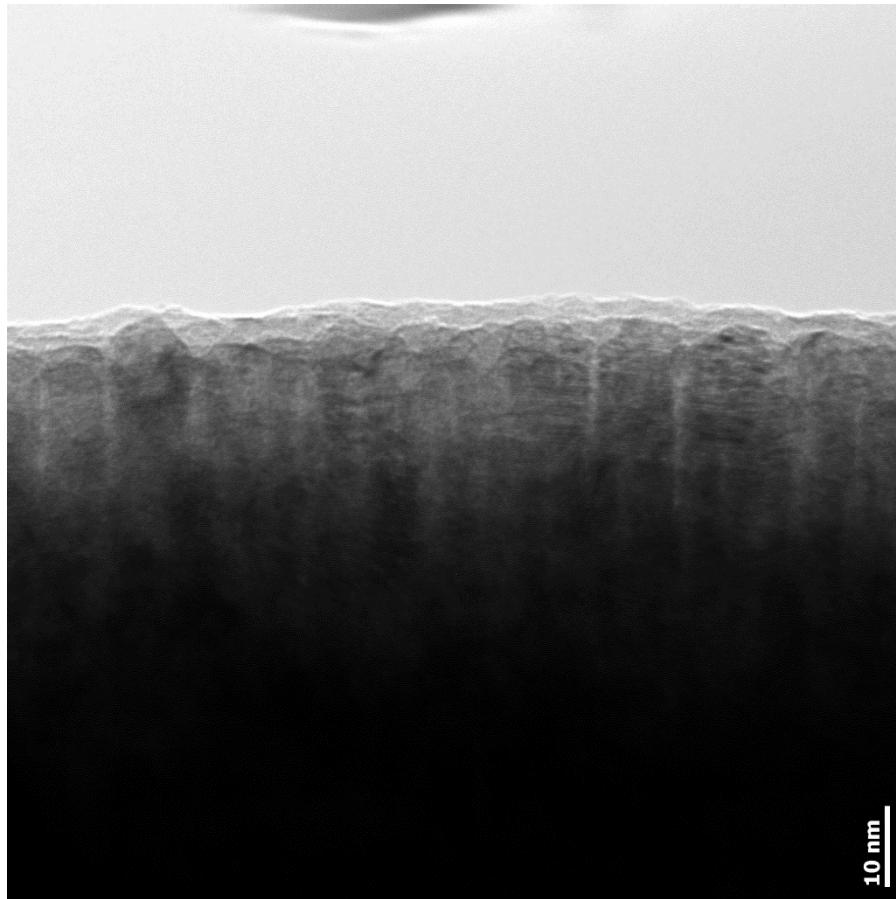
# *In Situ* - Increasing Load



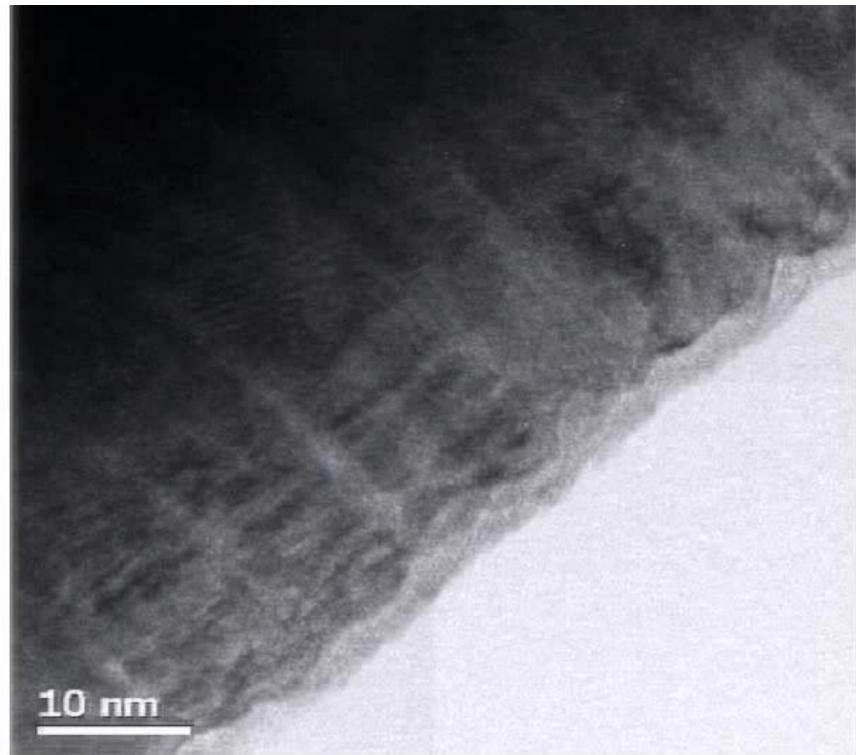
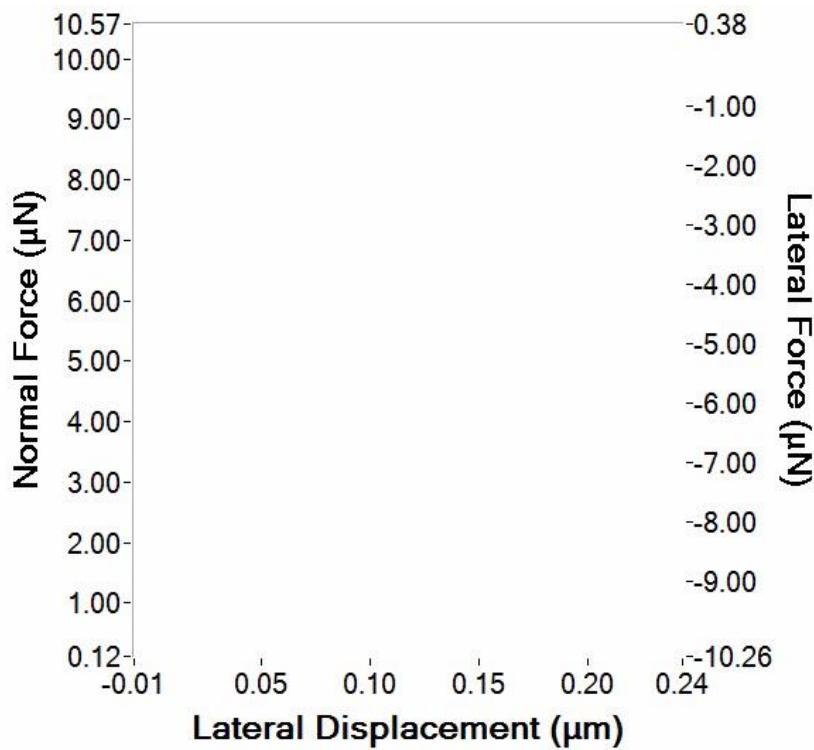
# *In Situ* – Increasing Load



# *In Situ* – Increasing Load

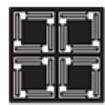


# *In Situ* - Scratch



 HYSITRON™

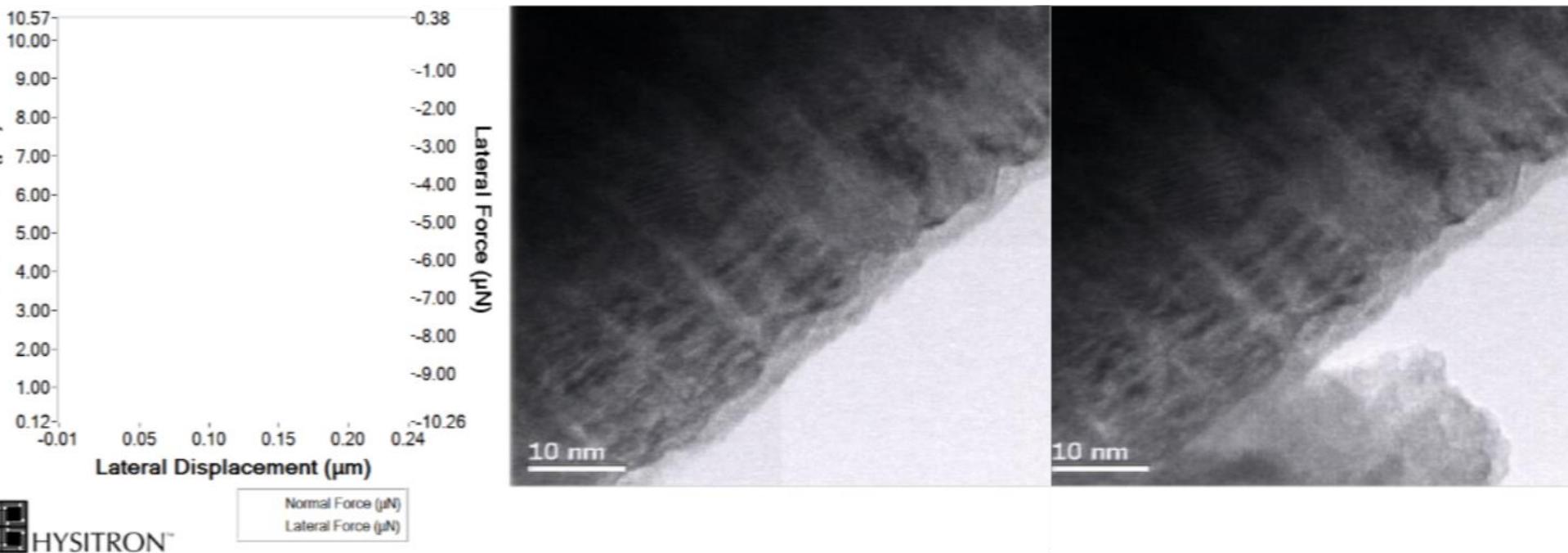
Normal Force (μN)  
Lateral Force (μN)

 HYSITRON

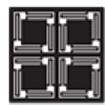
Correlative Nanomechanical Measurements

Douglas Stauffer

# Tribology - Grain Rotation



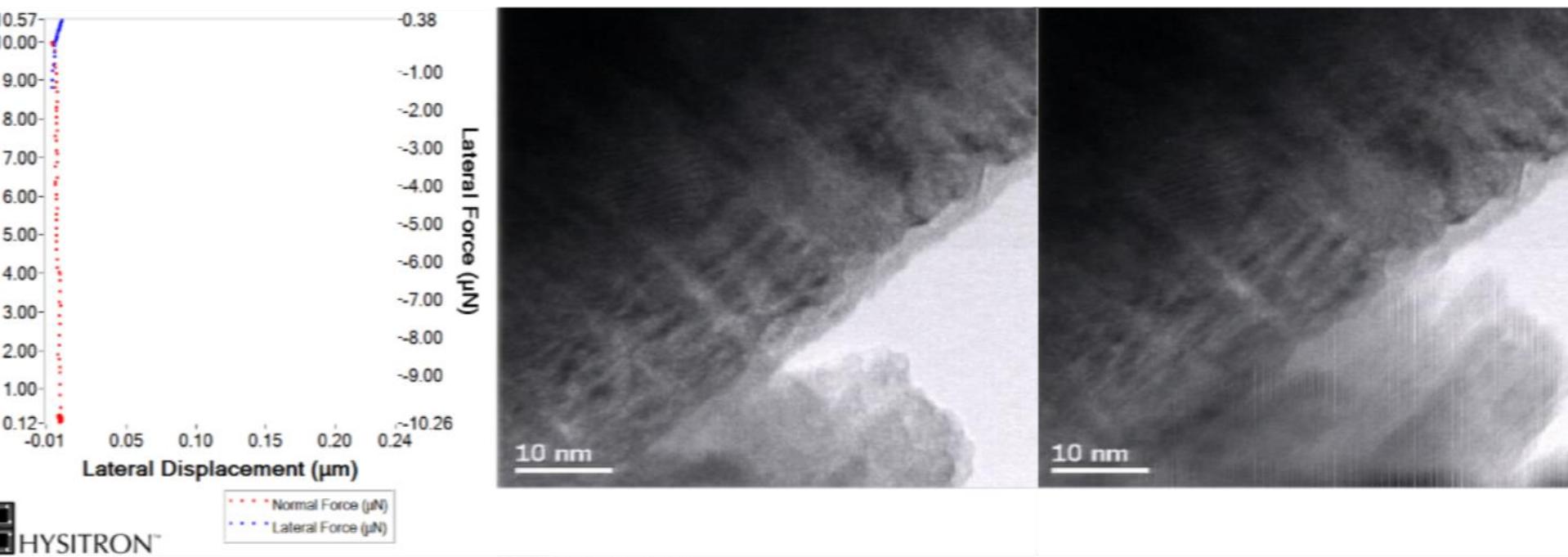
 HYSITRON™

 HYSITRON

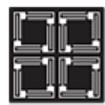
Correlative Nanomechanical Measurements

Douglas Stauffer

# Tribology - Grain Rotation



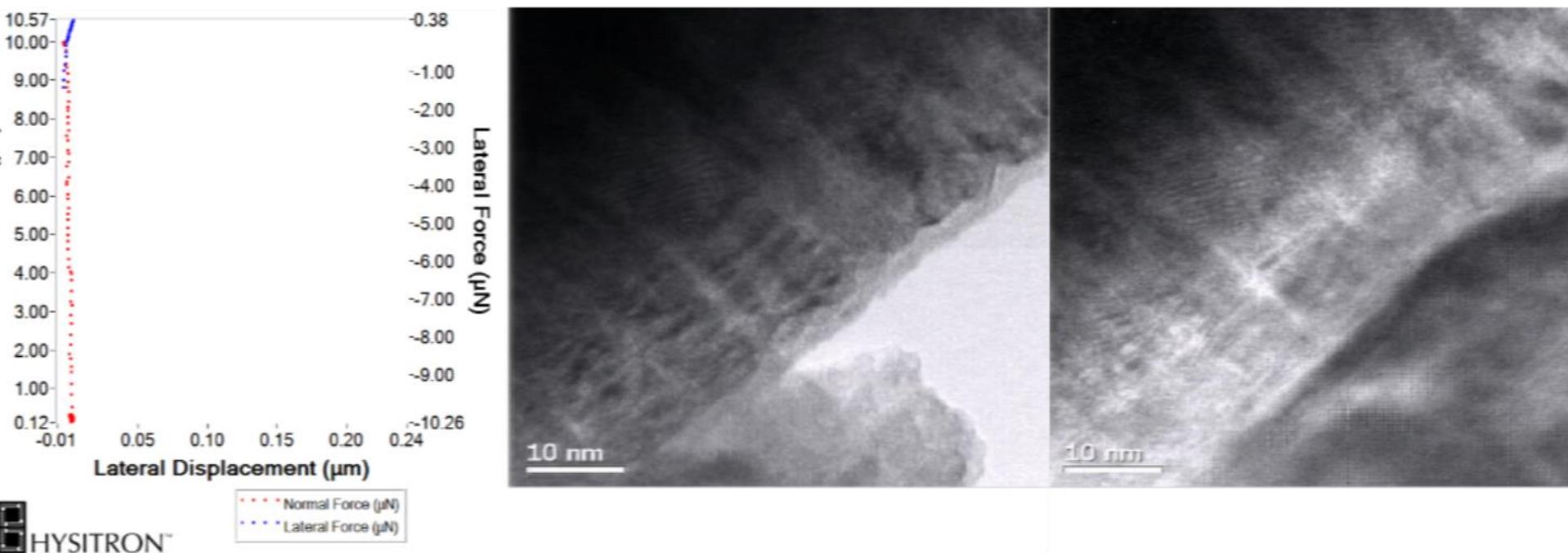
HYSITRON™

 HYSITRON

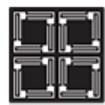
Correlative Nanomechanical Measurements

Douglas Stauffer

# Tribology - Grain Rotation



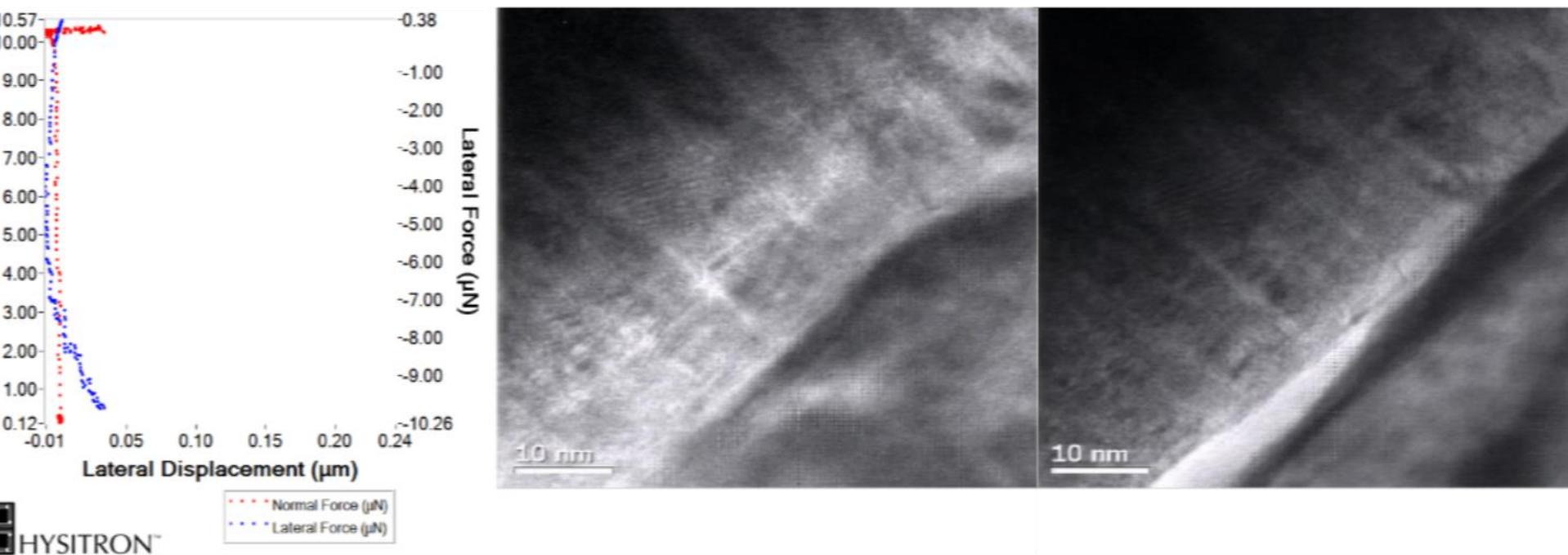
 HYSITRON™

 HYSITRON

Correlative Nanomechanical Measurements

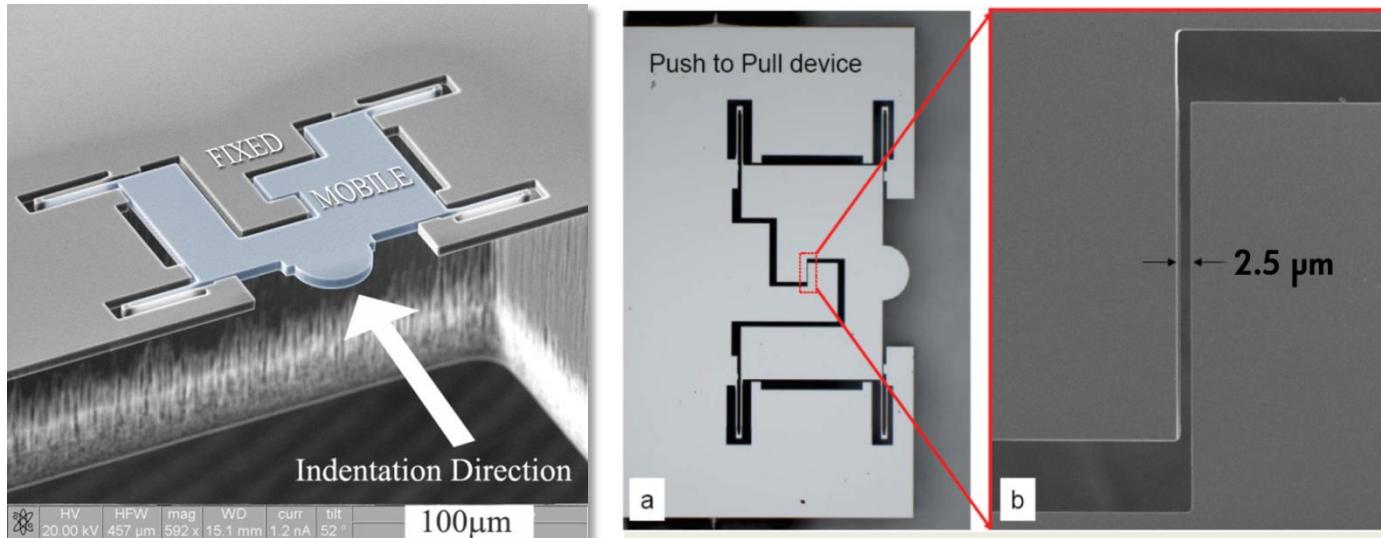
Douglas Stauffer

# Tribology - Grain Rotation

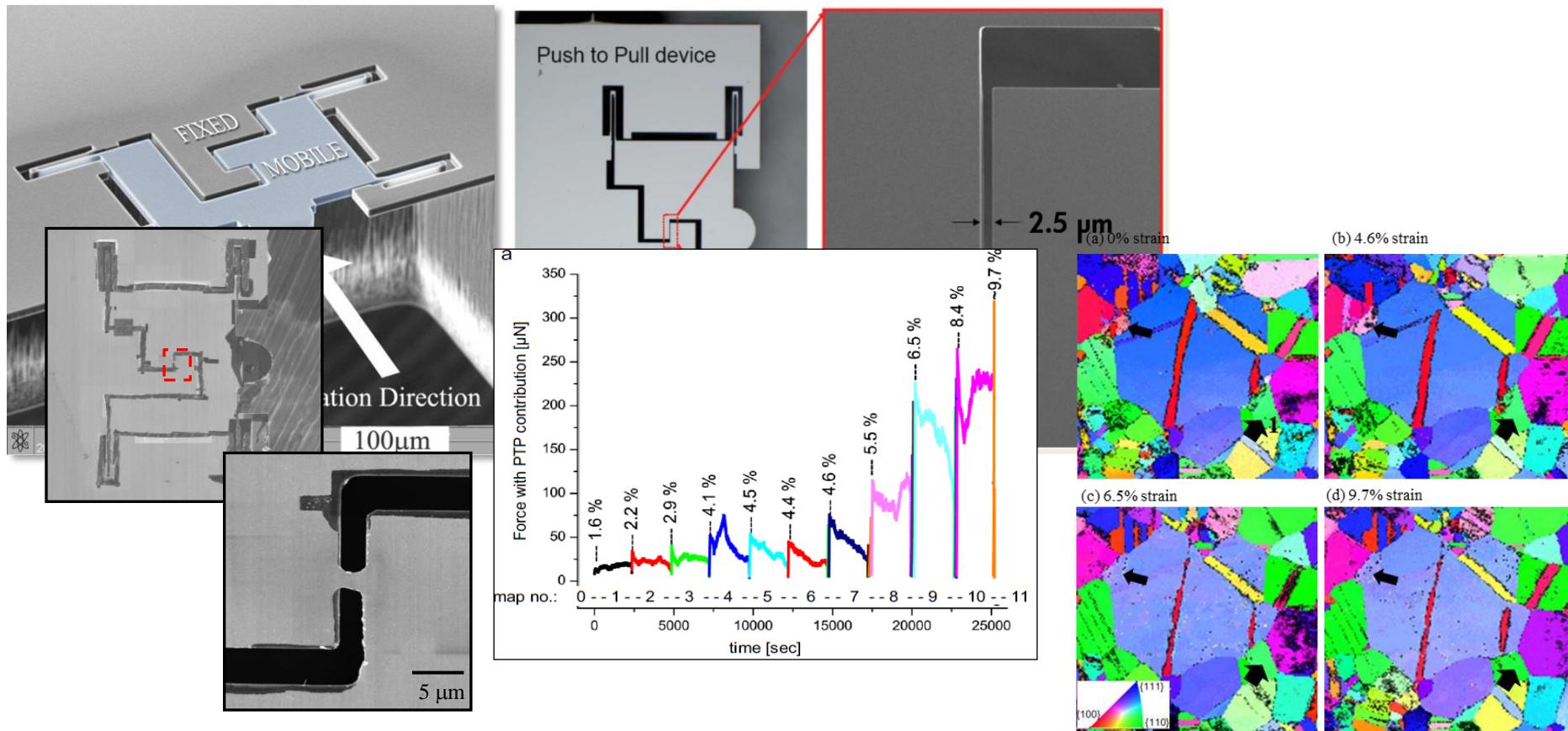


# I. Fatigue in Cu thin films

## *In Situ* TEM Tensile Testing



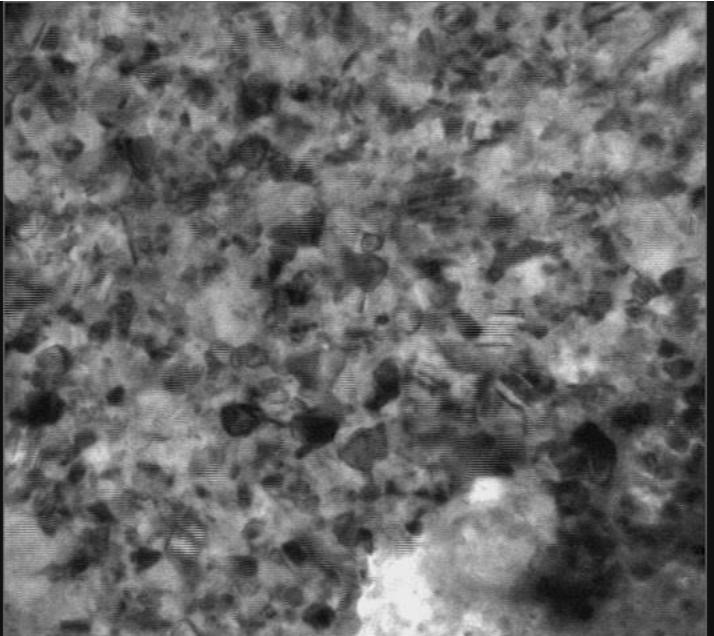
# *In Situ* TEM Tensile Testing



Courtesy Christian Kübel, Karlsruhe Institute of Technology / Karlsruhe Nano Micro Facility

**Combination of *in situ* straining and ACOM TEM: a novel method for analysis of plastic deformation of nanocrystalline metals**, A. Kobler, A. Kashiwar, H. Hahn, C. Kübel, *Ultramicroscopy*, 128, 68-81 (2013); DOI: 10.1016/j.ultramic.2012.12.019

# Crack Initiation



Pull in tension to  $150\mu\text{N}$

Cycle at 1 Hz

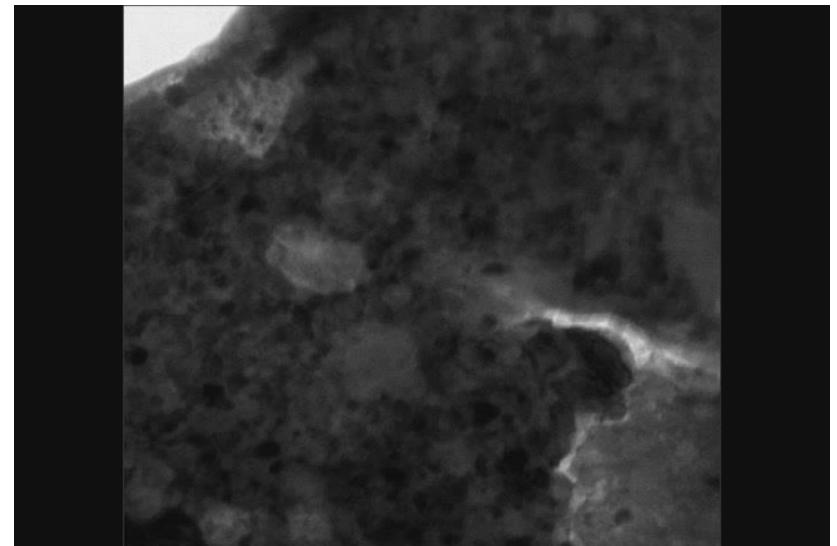
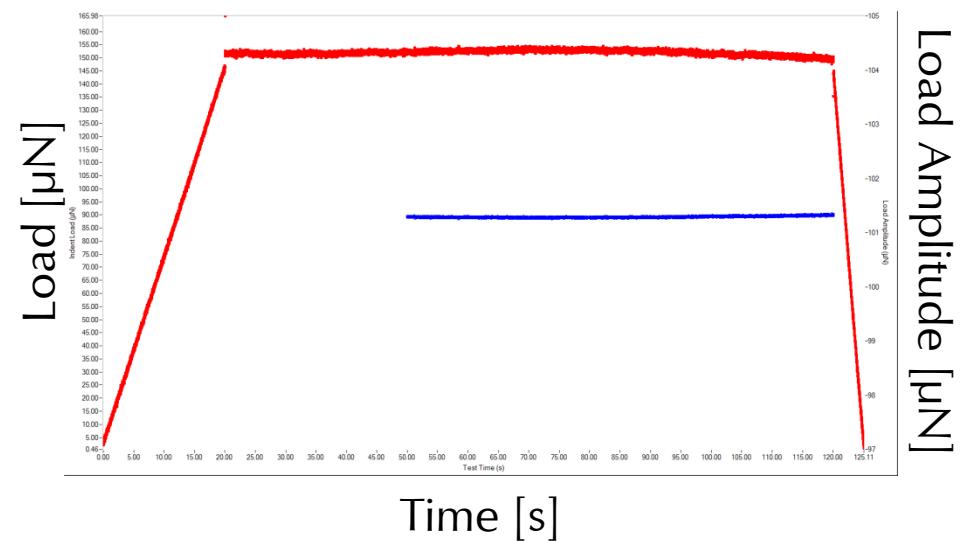
$$R = 0.5 = \frac{P_{min}/A}{P_{max}/A} = \frac{100\mu\text{N}}{200\mu\text{N}}$$



HYSITRON

# Crack Growth

$R = 0.5$  @200Hz,

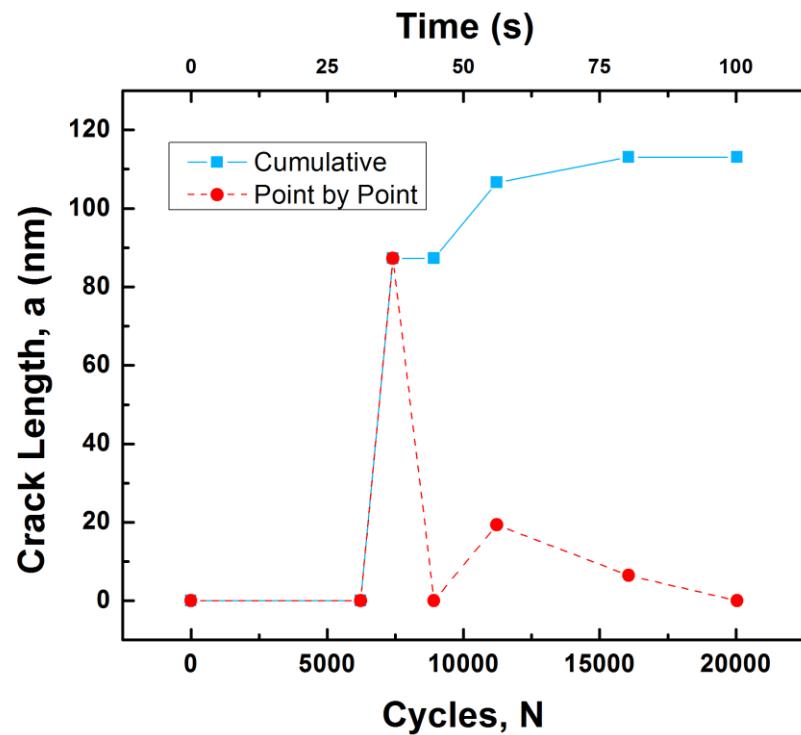
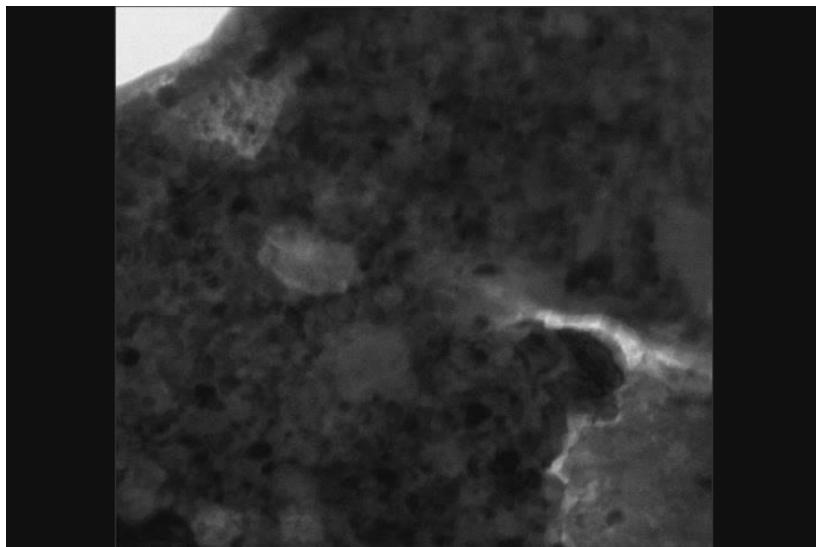


~257,000 cycles



HYSITRON

- 256,960 total cycles before this graph starts



# Acknowledgements

## *Anonymous Industrial Collaborators*

Parts of this work were carried out in the Characterization Facility, University of Minnesota, which receives partial support from NSF through the MRSEC program.



**Sandia National Laboratories**

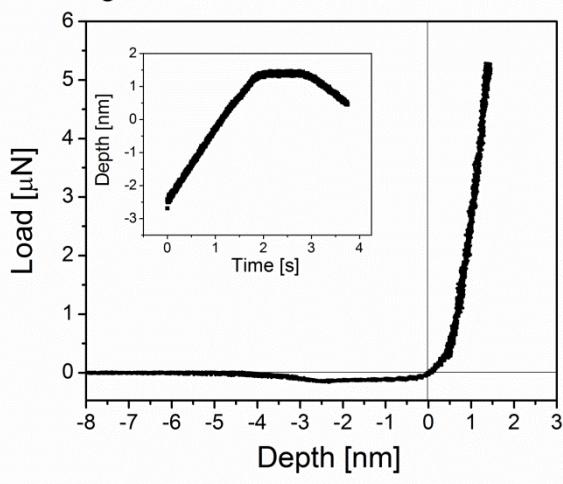


**U.S. DEPARTMENT OF  
ENERGY**

Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

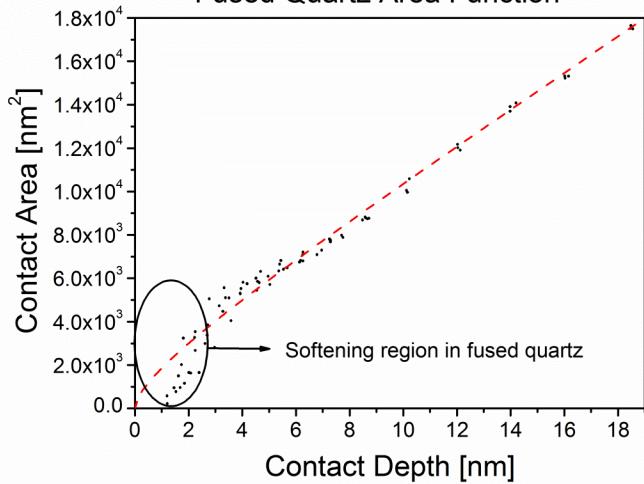
# Shallow Indentation: xProbe

Single 1.4nm Indent on 3nmDLC/Glass



Approach from Air  
Find positive deflection  
for zero point

Fused Quartz Area Function



Density of fused quartz changes at sub 4nm contact depths, need to use high resolution tip imaging to find the tip shape for shallow indents

Convoluted Image from Scan

