Nanoindentation:
Materials Mechanics at Small Length Scales

Atomic Force Microscopy
3D Optical Microscopy
Fluorescence Microscopy
Tribology
Stylus Profilometry
Nanoindentation
Traditional Mechanical Test

Tensile & Compression Testing

- Stringent requirements for sample geometry
- Length scale limitations for testing thin films
- Measures bulk properties along entire gauge length

\[ \sigma = E \varepsilon \]
How do I test...?

Small Feature Position Accuracy
Basic Nanoindentation Principles – Quasi-Static Nanoindentation

Reduced Modulus

\[ E_r = \frac{S \sqrt{\pi}}{2 \sqrt{A}} \]

Hardness

\[ H = \frac{P}{A} \]
Electrostatic Actuation / Capacitive Displacement Sensing

- Load or Displacement Control
- 78 kHz Feedback Loop Rate
- 38 kHz Data Acquisition Rate
- Enhanced Testing Routines
- Digital Signal Processor (DSP) + Field Programmable Gate Array (FPGA) + USB Architecture
- Modular Design
- Low Moving Mass / Inertia
- Low Intrinsic Dampening

Transducer Stability Specs
- 0.1 nm Displacement Noise Floor
- 20 nN Force Noise Floor
- <0.05 nm/sec Thermal Drift
- *Specs Guaranteed On-Site*
What that looks like...

- In-situ SEM indentation with Bruker’s Hysitron PI 88: Multilayer coating
Creative Geometries

Nanoindentation

Compression

Bend

Tension

Fatigue

Tribology
Speeding up the Process: XPM

- Performech II controller + Triboscan 10 software 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
Speeding up the Process: XPM
Test Outputs:
Modulus, Hardness, Position

20x20 Nanoindentation Grid
400 Indents in ~100 Seconds

Optical – SPM – XPM Indentation
Ceramic Matrix Composite – SiC/SiC
xSol® - Heating, Cooling, Humidity
Heating: 800°C • Cooling: -100°C • Humidity: 95% RH

- Temperatures from -140 to 800°C
  - Using xSol technology
- O₂ and H₂O < 1ppm [P₀₂ ~ 10⁻³ Torr]
SiC Fiber-Matrix Composite

Fiber Matrix 400°C
SiC Fiber-Matrix Composite

Fiber

Matrix

Fiber

Matrix

Fiber

Matrix
Temperature Effects on 1018 Steel

1018 is a very common structural material: think nuts and bolts
Temperature Effects on 1018 Steel

Mapping 3 Indents/s at 0°C

SPM with the Cooling System at 0°C
Temperature Effects on 1018 Steel

Mapping 3 Indents/s at 0°C

Hardness Contour Map
1018 Steel

Hardness Frequency

Count

H(GPa)

3.0 3.5 4.0 4.5 5.0 5.5 6.0
Ferrite Phase: Sweeping Temperature

0.5s\(^{-1}\) constant \(\dot{\varepsilon}\)
Comparison of Three Steels

- Nitronic 50 shows no transition down to -73°C.
- Sandvik 1RK 91 exhibits a transition between -15°C and -25°C.
Dynamic Techniques

Key Model Assumptions

- Linear Elasticity/Viscoelasticity
- Oscillation amplitude is small, such that contact area change during oscillation is insignificant

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

\[ X_0 = \frac{F_0}{\sqrt{(k - m\omega^2)^2 + [(C_1 + C_s)\omega]^2}} \]

\[ \phi = \tan^{-1}\left(\frac{(C_1 + C_s)\omega}{k - m\omega^2}\right), \quad k = K_s + K_i \]
nanoDMA III Principles

Dynamic Measurement

- Hardness
- Storage Modulus
- Loss Modulus
- Tan Delta
- Stiffness

\[ E' = \frac{K_s \sqrt{\pi}}{2\sqrt{A_c}} \quad E'' = \frac{\omega C_s \sqrt{\pi}}{2\sqrt{A_c}} \quad \tan \delta = \frac{\omega C_s}{k_s} = \frac{E''}{E'} \]

1. Frequency/time dependent measurements (e.g. polymer response).
   S.A. Syed Asif, J.B. Pethica, MRS proceeding, 505,103 (1997)

2. Less influence from thermal drift (excellent for nanoscale creep).
   S.A. Syed Asif, J.B. Pethica, Phil. Mag.,76 (1997) 1105
nanoDMA® – CMX Test

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

Dynamic force superimposed on quasi-static force
Through thickness comparison

**Quasi-static Indentation**

- Reduced Modulus (GPa): 134.04 ± 2.7 GPa

**Dynamic Indentation**

- Reduced Modulus (GPa): 133.88 ± 2.8 GPa
Substrate Effect

• Zone of elastic deformation extends far beyond the depth of the indenter.

• If the elastic zone passes the film/substrate boundary, measurement will be influenced by substrate properties.

• CMX tests allow easy detection of substrate influence for thin film measurements
Substrate Effect

Substrate effects can occur at small depths.

CMX tests performed on 400 nm SiO₂ on Si

Modulus vs. Displacement
Frequency Sweeps

Quasistatic Load, DC = 1000 µN
Load Amplitude, AC = 50 µN
Understanding fatigue in nanocrystalline metals

A nanoDMA modified PI-95 for performing tensile fatigue

Daniel Bufford\textsuperscript{2}, Douglas Stauffer\textsuperscript{1},
William Mook\textsuperscript{2}, S.A. Syed Asif\textsuperscript{1},
Brad Boyce\textsuperscript{2}, Khalid Hattar\textsuperscript{2}

1. Bruker Nano, Inc.
2. 2. Sandia National Laboratories
Nanocrystalline metals

nc Metals exhibit increased strength

G.B. stability can be obtained by alloying

Stabilizing structures under fatigue? Grain or crack growth?


Converting Forward Actuation into Tension

\[ F_{\text{applied}} = F_{\text{measured}} - F_{\text{spring}} \]

\[ F_{\text{spring}} = S\delta = 336.5 \, \text{N/m} \cdot \delta \]

C. Chisholm et al.  

S. Kaps et al.  
ACS Omega 2, 6, (2017) 2985-2993
In-Situ TEM Tensile Testing of Thin Films

Monotonic Loading of Cu Thin Films

Thickness: 75 nm  |  Grain size of 27±9 nm  |  Range: 20-110 nm
Crack (void) Initiation at Cu/CuO\(_x\) Interface

1 Hz Fatigue Loading \(\mid\sigma_{\text{mean}} = 910\ \text{MPa}\) \(\mid R = 0.46 = \frac{P_{\text{min}}/A}{P_{\text{max}}/A} = \frac{63\mu\text{N}}{136\mu\text{N}}\)
Crack Propagation from Cu/CuO\textsubscript{x} Interface

15fps, now ~13 cycles/frame | 256,000 total cycles before this video starts
Crack Growth, After 256k Cycles

- Average crack extension of 6pm/cycle
- Initiation / Stage I Regime for fatigue
- Reduction in dynamic stiffness can be used to monitor crack growth
Contrast at Crack Tip

Contrast change at crack tip.

Is this grain growth due to enhanced stress in front of the advancing crack?

Bufford, et al. Nanoletters 16 2016 4946-4953
Exploring the Grain Growth Hypothesis
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