In-Situ Nanomechanics in the Transmission Electron Microscope (TEM)

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

- Hysitron® PI 95 TEM PicoIndenter®
- Sample Preparation and Testing Techniques
- Advanced Applications: A. Heating, B. Electrical, C. nanoDynamics, and D. Tribology
Nanomechanics:
- Elasticity, plasticity, fracture, fatigue, creep, wear, strain rate effect of materials from the micrometer down to the tenths of nanometer length scale
- Miniaturized test rigs and MEMS-based devices

Why In-Situ TEM?
Direct observation of phenomena:
- Phase transformations
- Dislocation bursts and other defect nucleation
- Fracture
- Deformation mechanism

M. Uchic, et al., Science 2004

Greer and De Hosson, Progress in Materials Science, 56 (2011) 654–724.
Quantitative In-Situ TEM Nanomechanical Testing Instruments

Commercial PI 95 Units Available for 4 Major TEM Brands

Seeing is Believing®
Quantitative Nanomechanical Testing Inside Your Electron Microscope

Sanjit Bhowmick, Ph.D. | Bruker
In-Situ Nanomechanics in the Transmission Electron Microscope (TEM) | 10/18/2018
Small nanoindentation transducers with independent electrostatic actuator and capacitive displacement sensor.

- 1 mN max. load
- 2 μm max. displacement
- <200 nN force sensitivity
- <1 nm displacement sensitivity
- Interchangeable transducer
- 1 US Patent # 8,161,803

Miniature transducer¹
(JEOL TEMs)

- 1.5 mN max. load
- 5 μm max. displacement
- <200 nN force sensitivity
- <1 nm displacement sensitivity
- Interchangeable conductive indenter probe
- ¹US Patents # 8,456,498 & 7,798,011

MEMS transducer²
(FEI/Hitachi/Zeiss TEMs)

- 1 mN max. load
- 2 μm max. displacement
- <200 nN force sensitivity
- <1 nm displacement sensitivity
- Interchangeable transducer
- ²US Patent # 8,161,803
Precise Positioning

Three Levels of Control for Tip Positioning and Mechanical Testing

1. **Manual Control:**
   - Three-dimensional movement
   - Range: μms to mms

2. **Piezoelectric Actuation:**
   - Three-dimensional movement
   - Computer controlled
   - Range: nm to μm

3. **Transducer:**
   - Indentation direction
   - Computer controlled
   - Range: nm to μm

Sample remains stationary during testing

Piezo actuator used only for fine positioning
Testing Techniques and Sample Preparation

Nanoindentation

Compression

Bending

Tension

Fatigue

Tribology
A. Nanoparticles and Thin Films

Silicon Wedges

- 150 nm narrow Si wedges are used as a template for thin film deposition.
- 1 micron plateau Si wedges are used as substrates for nanoparticle deposition.

Schematic of narrow Si wedge and 1 micron plateau.

**Left:** Thin Al film on narrow Si wedge.
**Right:** Gold nanoparticles on 1 micron Si wedge.
B. Tensile Testing
Push-to-Pull (PTP) Device

US Patent # 8,434,370

Tensile Testing of Nanowires or Thin Films

PTP device mounts to PicoIndenter sample stage.

Nominal Stiffness

- 15 N/m
- 150 N/m
- 450 N/m
B. Tensile Testing
Push-to-Pull (PTP) Device

Nanowire Mounting:

A nanowire is picked by nanomanipulator inside FIB-SEM, placed on E-PTP, and welded using Pt gas injection system. Other leads are also connected by Pt.

- FIB-SEM
- Manipulator
- Gas injection system, Pt, W
B. Tensile Testing

Push-to-Pull (PTP) Device


**Thin Film Mounting:**

**Method A:**
- Deposit thin film on NaCl crystals. Dissolve NaCl in water and collect free standing thin film on PTP. FIB cut thin film which is deposited on PTP springs. Leave a part of the film on the sample mounting gap.
- Carbon coated Mica can also be used as a substrate.

**Method B:**
- Pattern transfer after depositing a film on standard holey carbon grid.
Unique Method for Quantitative In-Situ Nano-Tensile Testing Inside TEM

Cu nano-tensile sample with diameter 100-200 nm.

C. Bulk Sample and Half-Grid

Indentation, Bending, and Pillar Compression

Electron transparent thin region
Approximate length 5-10 micron, width 2-3 micron, thickness <100 nm

Bending

Pillar
The four-screw designed front-end was developed for multiple contact sample mount geometries.

Front-ends are compatible with bulk sample mount, Si wedge mount, PTP mount, Electrical Characterization Module (ECM) mount, and 400°C MEMS heater.
TriboScan™ Software

- Tabbed scheme for quick access to stage control, transducer calibration, test setup, and data analysis.
- Flexible load function generator w/ load- and displacement-controlled modes.
- Integrated TEM/SEM video capture and synchronization for side-by-side analysis.
- Empowering for **Operator** and **Expert** level users alike.
Simultaneous complementary techniques can be coupled to in-situ mechanical tests for an even more robust examination of material properties.

Applications: Hybrid Techniques

Electron Microscope Imaging

Nanomechanical Testing Technique

(Indentation, compression, etc.)

Complementary Technique

(I. Heating, II. Electrical, III. nanoDynamic, and IV. Tribology)
I. High Temperature Testing
In-Situ Heating to 400°C

Closed-Loop, Software-Controlled Heating up to 400°C

- Low-power, ultra-stable heating design for both SEM and TEM nanomechanical testing.
- Direct correlation between temperature and mechanical properties.
- Sample is mounted in close proximity of heating element and sensor.
- Combined heating element and temperature sensor.
- US and International Patents Pending.
II. Electrical Characterization

**Materials**
- PZT Materials
- Phase Change Memory
- Bulk metallic glasses
- CNT composites
- Silicon
- Battery & Energy Storage

**Properties**
- Mechanical
- Electrical

**Testing Modes**
- Constant Voltage
- Constant Current
- Voltage Sweep

**Specifications**
- Current Noise Floor: 20 pA
- Current Resolution: 5 pA
- Voltage Noise Floor: 10 μV
- Voltage Resolution: 5 μV
II. Electrical Characterization
Option: Electrical + Push-to-Pull (E-PTP)

Electromechanical Characterization of One-Dimension Solids
Source and sense connections shown by arrows

Sanjit Bhowmick, Ph.D. | Bruker  
In-Situ Nanomechanics in the Transmission Electron Microscope (TEM) | 10/18/2018
II. Electrical Characterization

Electromechanical Properties of ZnO Nanowires

ZnO Nanowire Mounted on E-PTP Inside FIB using Manipulator and GIS

Conductivity increases to 14.2% and 13.9% at 4.25% strain

Bhowmick, et. al., In Situ Electromechanical Study of ZnO Nanowires, Microscopy & Microanalysis, 2013, 19, p. 434.
Dynamic force superimposed on quasi-static force.
III. nanoDynamic™ Mode

- Cyclic loading up to 300 Hz.
- Efficient mode for in-situ fatigue testing in the TEM.
- Quantitatively measure stiffness as a function of time (cycles) and correlate to imaging information.
- Couple with PTP to apply tensile fatigue stresses to thin films or nanowires.

*in situ* dynamic loading

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IV. Tribology

nanoScratch Option for the TEM PicoIndenter

- Simultaneous normal/lateral force sensing.
- Tribology, sliding contacts, friction, and wear.
- Compatible with FEI, Zeiss, Jeol, and Hitachi TEMs.
IV. Tribology

2D Force and Displacement sensing: Hysitron Transducer

3D Piezoelectric Element:
- Normal Force
- Lateral Force
- Normal Displacement

MEMS Transducer:
- Lateral Displacement

Sample (stationary)
Probe (usually diamond, but customizable)

Scratch Action: 3D Piezoelectric Element
Films deposited on Si Wedge sample mount of Hysitron PI 95.
Wedge shaped substrate allows for electron transparency at the tip, and also serves as rigid substrate for mechanical test.
IV. Tribology

2D MEMS Application: Tribology of HDD Film Stack
Two different types of deformation were observed in PMR as a function of normal load.

- At low normal loads of 1, 2 and 5 μN, the asperities of the columnar grains were flattened.
- At high normal loads of 10 and 20 μN, the grains underwent irreversible plastic bending, which would trigger data loss.

20 μN normal force scratch experiment. Grain reorientation happened.
IV. Tribology

**Single WS₂ Particle**
- Coefficient of friction was correlated to exfoliation of particles

![Image of WS₂ particle with friction curve](attachment:image1.png)

**Olivine**
- Repeated scratches on the olivine specimen showed substantial plasticity.
- Each pass was observed to nucleate more defect zones which were arranged in a symmetric array along the wear path.

![Image of olivine specimen with wear path](attachment:image2.png)

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Hysitron PI 95 TEM PicoIndenter
Quantitative Nanomechanical Testing Inside Your Electron Microscope

https://www.bruker.com/nanomechanical-testing
In Situ Straining in the TEM
Ankush Kashiwar, Aaron Kobler, Christian Kübel
Karlsruhe Institute of Technology

External Stimuli
- Biasing
- Heating
- Liquid
- Gas
- Deformation

Synthesis/Processing

Structure

Properties

Performance

In Situ Nanomechanics in the Transmission Electron Microscope (TEM)  |  10/18/2018
Christian Kübel, Ph.D.  |  Karlsruhe Institute of Technology
Motivation
Deformation of Nanocrystalline Metals


Nanocrystalline Metal
↑ High strength
↑ Good fatigue properties
↓ Poor ductility

Coarse Grained Metal
→ Limited strength
→ Limited fatigue properties
↑ Good ductility
Motivation
Structure & Defects in Nanocrystalline Metals

Modified from E.A. Lazar et al., Acta Mat. 2011, 59, 6837

Dislocation and Grain Boundary Based Deformation Processes
Motivation
Deformation at Small Length Scales

Deformation in Individual Grains
UFG Al (d = 250 nm)
Mompiou et. al., Acta Mat. 2013, 61, 205.

Deformation at Atomic Resolution
Au nanocrystal (sub 10 nm)
Motivation
Deformation of Nanocrystalline Metals


Atomic resolution image of the complex S9 and S3 twin boundary system.
Motivation

Deformation of Nanocrystalline Metals


Atomic resolution image of the complex S9 and S3 twin boundary system.
Motivation

Deformation of Nanocrystalline Metals


Atomic resolution image and $e_{xx}$ strain component in a grain with a complex S9 and S3 twin boundary system.
Methodology

Automated Crystal Orientation Mapping

1. Crystallographic information
2. Large field of view and nm resolution
3. Statistically significant area
Methodology

*In situ* ACOM Straining


**Combined Techniques**
- *In situ* straining
- mp-STEM imaging
- ACOM-TEM

**Advantages**
- Correlation of mechanical data and image information
- Reference imaging with µp-STEM: fast overview
- Crystallographic information with ~1 nm resolution
- Local information with decent statistic

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Christian Kübel, Ph.D. | Karlsruhe Institute of Technology | In-Situ Nanomechanics in the Transmission Electron Microscope (TEM) | 10/18/2018
Methodology
Sample Preparation


Au frame on holy carbon grids with additional C-film and sputtered nc Pd

FIB to prepare ‘dog-bone’ shape and cut transfer frame

Frame + sample placed on PTP
**In situ** Mechanical Testing of nc Pd Stress-Strain Curves


### Load Controlled Experiment

![Strain-force curve](image)

- **F with PTP [μN]**
  - 0
  - 50
  - 100
  - 150
  - 200
  - 250
  - 300

- **time [h]**
  - 0
  - 1
  - 2
  - 3
  - 4
  - 5
  - 6

- **Fracture**

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**Experimental time-force curve**

- 1
- 2
- 3
- 4
- 5
- 6
- 7

**100 nm**

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Christian Kübel, Ph.D. | Karlsruhe Institute of Technology

In-Situ Nanomechanics in the Transmission Electron Microscope (TEM) | 10/18/2018
**In situ** Mechanical Testing of nc Pd Stress-Strain Curves


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**Load Controlled Experiment**

![Strain-force curve](image)

- **Creep**
- **Load plateaus**
- **Fracture**

**Diagram Details:**
- **Y-axis:** Force without PTP [μN]
- **X-axis:** Strain [%]
- **Scale:** 100 nm

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**Image Description:**
- Nanomechanical data from in-situ testing in the TEM, showing load-controlled experiment results with strain-force curve and associated phenomena.

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**References:**
- **Christian Kübel, Ph.D. | Karlsruhe Institute of Technology**
- **In-Situ Nanomechanics in the Transmission Electron Microscope (TEM)**

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**Date:** 10/18/2018
In situ Straining of Annealed nc Pd

In situ ACOM STEM Characterization


Grain recognition reliable for 90-95% of area ⇒ results representative for sample.
In situ Straining of Annealed nc Pd Twin Boundary Motion

In situ Straining of Annealed nc Pd Grain Boundary Motion

In situ Straining of Annealed nc Pd Grain Boundary Motion

**In situ** Straining of Annealed nc Pd Crystal Rotation Map - 65°


Ambiguity correction for all grains throughout the complete ACOM straining series needed to prevent detection of false positive S3 and S9 changes!
Mechanically Driven Grain Refinement in Nanocrystalline Palladium

In situ Straining of Annealed nc Pd Crystal Rotation Map - 12°

In situ Straining of Annealed nc Au$_{72}$-at\%Pd$_{28}$-at\%
Crystal Rotation Map - 12°
In situ Straining of Annealed nc Au$_{72}$-at% Pd$_{28}$-at%

Intra Grain Rotation - Small Angle Boundaries

A. Kobler & C. Kübel, unpublished
In situ Straining of Annealed nc Au$_{72}$-at% Pd$_{28}$-at%

Intra Grain Rotation - Small Angle Boundaries

A. Kobler & C. Kübel, unpublished
In situ Straining of nc Au$_x$Pd$_y$

Grain Rotation Histograms


- **Aged nc Au**:
  - $d_N = 38$ nm
  - $d_A = 95$ nm

- **Annealed nc Pd**:
  - $d_N = 12$ nm
  - $d_A = 25$ nm

- **nc Pd**:
  - $d_N = 7$ nm
  - $d_A = 10$ nm

- **Annealed nc Au$_{72}$Pd$_{28}$**:
  - $d_N = 12$ nm
  - $d_A = 25$ nm
In situ Straining of Annealed nc Pd
‘Reversible’ Plastic Deformation

Background

Bauschinger Effect in Nanocrystalline Metals


- Dislocation pile up at grain boundaries and interfaces
- Stress differences between grains
Background

Bauschinger Effect in Nanocrystalline Metals

F. Mompiou et al., *Acta Materialia* 60 (2012) 3402–3414

- Plastic recovery in UFG metals
- Reverse motion of dislocation
In situ Straining

Bauschinger Effect

A. Kashiwar & C. Kübel, unpublished results

In situ Straining
10 nm/sec. displacement controlled

![Strain vs Stress Graph with annotations](image)

Playback speed x17.5

200 nm

Playback Speed x22.5

Fracture

2x PTP

Cycle #1

#2

#3

#4

#5

#6

Strain rate ~7*10^{-4} s^{-1}

0 1 2 3 4 5 6

0 500 1000 1500

Stress [MPa]

Strain [%]
In situ Straining
Reversible Grain Rotation

A. Kashiwar & C. Kübel, unpublished results
In situ Straining
Reversible Grain Rotation

A. Kashiwar & C. Kübel, unpublished results
Conclusion

Methodology
Combination of *in situ* straining and ACOM-TEM
- Real time orientation mapping of nc metals
- Reliable quantitative analysis: global and local
- Challenges: projection effects and film bending

Deformation mechanism in nc Pd<sub>x</sub>Au<sub>y</sub>
- Significant dislocation contribution
  - S3 and S9 boundary formation
  - Grain rotation
  - Grain refinement

Bauschinger Effect in nc Pd<sub>x</sub>Au<sub>y</sub>
- Deviation from ideal elastic unloading
- Partially reversible grain rotation, sub-grain boundary motion and grain development
Acknowledgement

Electron Microscopy Group

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KIT
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Bruker (Formerly Hysitron)
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NanoMegas
Edgar Rauch, Muriel Veron

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