Nanomechanics
Measurements and Standards
at
NIST

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NIST Mission

Promote U.S. innovation and industrial competitiveness by

advancing measurement science, standards, and technology in ways that

enhance economic security and improve our quality of life.

(What)  (How)  (Why)
Nanomechanics Measurement Needs

Develop ultra-small scale mechanical measurement techniques and underlying metrology to enable commercial innovation and advanced manufacturing.

Micro-electromechanical Systems (MEMS): elasticity, plasticity, fracture, and adhesion of MEMS elements critical to performance and reliability
Develop ultra-small scale mechanical measurement techniques and underlying metrology to enable commercial innovation and advanced manufacturing.

**Magnetic recording head**: deformation and adhesion at the head-media interface critical to data storage performance.
Nanomechanics Measurement Needs

Develop ultra-small scale mechanical measurement techniques and underlying metrology to enable commercial innovation and advanced manufacturing.

**Nanoparticle Structures:**
forces between the particle and the substrate critical in particle manipulation.
Nanomechanics

Nanomechanics (definition): Analysis of the actions of forces and displacements (mechanics) at the nano-scale (< 100 nm).

Nanomechanics (implementation): Nano-scale measurement, calibration, and control of mechanical properties and behavior of materials and structures.

Bulk behavior and properties: Measurement, calibration and control of mechanical properties that have macroscopic analogs (e.g., modulus, hardness, and toughness, or stiffness, collapse load, and failure load). At issue here is performing measurements at small scales.

Surface + bulk behavior and properties: Measurement, calibration and control of mechanical behavior and properties that are intrinsic to the nano-scale. Forces associated with interactions between surfaces become comparable to those associated with bulk deformation at small scales and such forces become quantized as intrinsic material and system length scales are approached. At issue here is performing measurements on small-scale phenomena.
Nanomechanical Properties Group

Nanomechanical Measurement Science (Metrology)
Elucidation of scientific principles and laws related to nano-scale mechanical properties measurements so as to enable interpretation of measurements and development of measurement standards and technologies.

*Example:* Deconvolution of AFM-based adhesion measurements
MEMS, NEMS, Magnetic Storage, Nanoparticle Sensor Industries

Nanomechanical Measurement Standards
Development of standards, in the form of physical artifacts that enable calibration of nanomechanical instruments and comparisons of nanomechanical properties of materials and devices.

*Example:* Calibrated Reference Cantilever Array SRM
MEMS, SPM industries (Veeco, Asylum)

Nanomechanical Measurement Technology
Development of instruments and methodologies that enable nano-scale measurements of mechanical properties and performance of materials and devices.

*Example:* Development of “theta”-like ultra-small tensile specimen
MEMS Industries (Sandia National Laboratory)
# Nanomechanical Properties Group: Project Scope and Support

## Scanned Probe Microscopy Measurements and Standards
- **AFM-based adhesion and friction measurements** (meniscus, van der Waals, electrostatic)
- **Contact resonance AFM modulus measurements** (nanowires, topography)
- **AFM dimension measurements** (nanoparticles)
- **Flexural and torsional reference cantilevers**
  
  *(Six AFMs)*

*Support from WCF, SD, NCI*

## Nanoparticle Measurements and Standards for Biomedical Applications and Health
- **Nanoparticle standards**
- **DLS, SANS, and USAXS measurements of dimensions, hydrodynamic properties, and stability of nanoparticles and nanotubes**
- **Nanoparticle zeta-potential measurements**

*Support from NCI, Nanotube IMS, WCF*

## Nanoindentation Measurements and Standards
- **Small force reference transducer**
- **Break-junction measurements of atomic-scale forces**
- **IIT measurements and standards**
- **IIT reference instrument**
  
  *(Four Nanoindenter)*

*Support from Intrinsic Force IMS, MSAG*

## MEMS and NEMS Mechanical Reliability Measurements
- **“Theta”-like ultra-small tensile specimen**
- **Cantilever Array Discovery Platform**

*Support from DOE, TD*

## Piezospectroscopy Measurements and Standards
- **Nano-scale strain mapping using Raman spectroscopy and EBSD**
- **Stress standards**

*Exploratory project with CSTL*

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**AFM**: Atomic Force Microscope  
**MAA**: Multiscale Adhesion Apparatus  
**DLS**: Dynamic Light Scattering  
**SANS**: Small Angle Neutron Scattering  
**USAXS**: Ultra-Small Angle X-ray Scattering  
**IIT**: Instrumented Indentation Testing  
**EBSD**: Electron Backscatter Diffraction  

**NCI**: National Cancer Institute  
**MSAG**: Measurement Services Advisory Group  
**IMS**: Innovations in Measurement Science  
**WCF**: Working Capital Fund  
**SD**: Service Development  
**DOE**: Department of Energy  
**TD**: Treasury Department (US Mint)
# Nanomechanical Properties Group: Standards Development and Activities

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**National Institute of Standards and Technology**
Materials Building Facilities

Nano-scale Contact Laboratory
Nanoparticle Metrology Laboratory
Optical Microscopy and Fractography Laboratory
Center for Theoretical and Computational Materials Science
AFM Laboratory (with NIST Fellow, S. Wiederhorn)
Micro-scale Hardness and Toughness Standards Laboratory
Stress-Optical Laboratory (under construction, with CSTL)
Advanced Measurement Laboratory Facilities

Class 1000
±0.01 °C

Class 1000
±0.1 °C

Class 10000
±0.25 °C

Class 10000
±0.25 °C

Building 218, 150 m²

Gowning Area

National Institute of Standards and Technology
Advanced Measurement Laboratory Facilities

- NIST Multiscale Adhesion Apparatus
- Veeco CR-FAM
- Hysitron Nanoindenter
- Veeco AFM
- Surface Preparation
- RHK UHV STM, AFM
- MTS Nanoindenter
Nanomechanics Research at NIST

- **Materials Science and Engineering Laboratory**
  - Nanomechanical Properties Group (R. Cook)
  - Nanoscale Reliability Group (R. Keller)

- **Manufacturing Engineering Laboratory**
  - Electrostatic Force Balance (force metrology, SI-traceable μN to mN, J. Pratt)
  - Atomic Force Microscopy (dimensional metrology, SI-traceable nm, T. Vorberger)

- **Chemical Science and Engineering Laboratory**
  - Raman Spectroscopy (stress mapping, S. Stranick)

- **Electronics and Electrical Engineering Laboratory**
  - Microelectromechanical Systems (reliability and standards, J. Marshall),

- **Building and Fire Research Laboratory**
  - Instrumented Indentation Testing (coating integrity, scratch resistance, A. Forster)
Nanomechanics Program: Standards Impact Path

SI-traceable force and stiffness (822)

SI-traceable dimensions (821)

Break Junction Stiffness and Strength

Reference Cantilever SRM, RM

AFM Cantilever Stiffness Calibration

AFM, MAA, IIT: Adhesion, Friction Modulus, Hardness

Small Force Transducer SRM

IIT Calibration

IIT SRD

Theta-like Stiffness and Strength

Customers

Nanoparticle RM

AFM, MAA, IIT: Adhesion, Friction Modulus, Hardness

National Institute of Standards and Technology
Measurements and Standards Needs

• Commercial need for accurate and precise measurements of mechanical properties at the micro and nano scales
  ➢ Increased device functionality and decreased development costs
  ➢ Increased device manufacturing yield
  ➢ Increased device operational reliability

• Standards are required to enable measurement accuracy and precision
  ➢ Physical Artifacts (Reference Materials): Instrument calibration (enables accuracy, enables estimates of precision)
  ➢ Information (Reference Data): Instrument calibration (enables accuracy, enables estimates of precision)
  ➢ Procedures (Test Methods): Instrument calibration and operation (enable greatest accuracy and precision of measurement)

• NIST should assist in development of standard test methods for micro- and nano-mechanical measurements
  ➢ Standard reference materials and data are best utilized
AFM Cantilever Calibration

Measurement calibration needs for AFM tip-surface interactions:

Normal (z) force

Lateral (y) and Torsional (τ) force

- Methods for optimal calibration accuracy and measurement precision. Published 2007
- “Hammerhead” lateral force cantilever prototype. Planned for September 2009
- Standards development through VAMAS and ISO

VAMAS

ISO International Organization for Standardization

National Institute of Standards and Technology
Standards Opportunities

• ASTM committee C28 is perfectly placed to develop standard test methods for advanced materials at the micro and nano scales
  ▫ Large depth of experience and strong technical expertise within C28 in developing test methods for elastic, plastic, and fracture properties of advanced ceramics—brittle materials
  ▫ Majority of materials used in microelectronics, magnetic storage, microelectromechanical systems, and nanoparticle systems are brittle at the length scales of application

• Need to expand idea of what materials are considered advanced ceramics:
  ▫ Si (single- and poly-crystalline) (also SiC, poly-diamond, … )
  ▫ Non-structural ceramics that must exhibit structural integrity (dielectrics, ferroelectrics, thermoelectrics, … )

• Need to consider wide range of geometries
  ▫ Non-monolithic (film-on-substrate)
  ▫ Non-bulk (films, wires, dots)
Workshop Outcomes

• Greatest opportunity for C28 lies in development of standards for fracture testing methods:
  • Strength, strength distribution
  • Defect identification
  • Toughness
  • Lifetime, reliability
  • Fatigue

• Possibility: Round Robin on fracture strength, strength distribution, and defect identification of a simple, micro-scale test geometry