The National Nanotechnology Initiative and Nano-scale Length Metrology

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Contents:

• The U.S. National Nanotechnology Initiative (NNI)
  - Goals
  - Activities
  - Organization…

• Length Measurement Applications in the NNI
  - Gold Nanoparticle Reference Materials

• Probe-based Nano-scale Length Measurement
  - Critical Dimension Atomic Force Microscopy (CD-AFM) of Linewidth
  - Image Stitching Linewidth Measurement
  - Si(111) Atomic Steps for Z-calibration of AFMs
Funding:
NIST Manufacturing Engineering Laboratory
Nanomanufacturing Program,
NIST Office of Microelectronics Programs
National Cancer Institute

Certain Materials Provided by:
Michael Postek, Debra Kaiser, Andras Vladar (NIST)
E. Clayton Teague (NNCO)
What is Nanotechnology?

The understanding and control of matter at dimensions between approximately 1 nm and 100 nm, where unique phenomena enable novel applications… Nanotechnology involves imaging, measuring, modeling, and manipulating matter at this length scale.

From The National Nanotechnology Initiative – Strategic Plan December 2007
What is the NNI?

A collaborative, cross-cut program established in 2001 to:

• Coordinate Federal nanotechnology R&D by serving as a central locus for communication, cooperation, and collaboration among agencies that wish to participate (currently 25)

• Provide a vision of long-term opportunities and benefits of nanotechnology

The NNI creates a framework for a comprehensive nanotechnology R&D program by establishing shared goals, priorities, and strategies. The NNI as a program does not fund research but it informs and influences the Federal budget and planning processes through its member agencies.
Vision of the NNI:

“A future in which the ability to understand and control matter at the nanoscale leads to a revolution in technology and industry that benefits society”… expedited through a program of coordinate R&D aligned with missions of participating agencies.
Goals of the NNI:

1) Advance a world-class nanotechnology R&D program

2) Foster the transfer of new technologies into products for commercial and public benefits

3) Develop and sustain educational resources, a trained workforce, and the supporting infrastructure and tools to advance nanotechnology

4) Support responsible developments of nanotechnology
Program Component Areas:
Fundamental Nanoscale Phenomena and Processes – knowledge, new phenomena

- Nanomaterials – nanostructured materials with targeted properties
- Nanoscale Devices and Systems – novel devices
- Instrumentation Research, Metrology, and Standards for Nanotechnology – tools for characterization, synthesis, design (NIST is lead)
- Nanomanufacturing – scaled up, cost effective manufacturing (NIST is co-lead)
- Major Research Facilities and Instrumentation Acquisition – infrastructure
- Environment, Health, and Safety – assessing and managing risk
- Education and Societal Dimensions – includes outreach, studying implications for society

2009 Aggregated Budget Request $1.5 B
Structure of NNI

- National Research Council
- NNAP (PCAST)
- International Organizations
- Press
- Professional Societies
- Non-governmental Organizations
- Industry Sectors
- 26 Agencies Participating in NNI
- Office of Science and Technology Policy
- Office of Management and Budget
- NNCO
- House of Representatives Committee on Science
- Senate Committee on Commerce, Science and Transportation
- Regional, State, and Local Nanotechnology Initiatives
- NIST

Working Groups and Task Forces of NSET Subcommittee
Global Issues in Nanotechnology Working Group

• Coordinates international activities of the agencies in nanotechnology

• Monitors foreign nanotechnology programs

• Seeks to broaden international cooperation and communication with respect to nanotechnology R&D

Issues include:
- Environmental health and safety
- Trade and commercial interests
- Cooperation with other nations on joint R&D goals
64 major NNI centers, networks, user facilities

Alaska is shown at approximately half its size, and Hawaii’s size is approximately doubled.
Center for Nanoscale Science and Technology

providing measurement methods, standards and technology to support nanotechnology development from discovery to production

cnst.nist.gov

Collaborations

Nanofabrication
Future Electronics
Energy

Open Access to Nanofabrication and Measurement

Class 100 Cleanroom includes:
- Annealing furnaces
- UHV
- Plasma etching
- Photolithography
- Metalization
- Nano-imprinting
- Wet etching
- Ebeam lithography
- AFM
- STEM
- FESEM

Process engineering and nanofabrication experts on staff
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Applications at NIST:
Particle Standards for Medical Industries

Particle-based Delivery Systems in Medicine for Diagnosis and Treatment

⇒ Size matters

Part of Joint Program with FDA
From D. Kaiser
NIST Reference Materials (RM) 8011, 8012, 8013 – Gold Nanoparticles

With Mean Sizes

8011 – 10 nm
8012 – 30 nm
8013 – 60 nm

A. Vladar, SEM of RM 8012 particles
# Report of Investigation

Reference Material 8012

Gold Nanoparticles, Nominal 30 nm Diameter

## Table 1. Reference Value Mean Size and Expanded Uncertainty (a)

Average Particle Size (Diameter), in nm

<table>
<thead>
<tr>
<th>Technique</th>
<th>Analyte Form</th>
<th>Particle Size (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Atomic Force Microscopy</td>
<td>dry, deposited on substrate</td>
<td>24.9 ± 1.1</td>
</tr>
<tr>
<td>Scanning Electron Microscopy</td>
<td>dry, deposited on substrate</td>
<td>26.9 ± 0.1</td>
</tr>
<tr>
<td>Transmission Electron Microscopy</td>
<td>dry, deposited on substrate</td>
<td>27.6 ± 2.1</td>
</tr>
<tr>
<td>Differential Mobility Analysis</td>
<td>dry, aerosol</td>
<td>28.4 ± 1.1</td>
</tr>
<tr>
<td>Dynamic Light Scattering</td>
<td>liquid suspension</td>
<td>28.6 ± 0.9</td>
</tr>
<tr>
<td>173° scattering angle (backscatter)</td>
<td></td>
<td>26.5 ± 3.6</td>
</tr>
<tr>
<td>90° scattering angle</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Small-Angle X-ray Scattering</td>
<td>liquid suspension</td>
<td>24.9 ± 1.2</td>
</tr>
</tbody>
</table>
Applications at NIST: Length Metrology for Semiconductor Manufacturing

- Linewidth
- Step Height
- Line Edge Roughness
- Pitch

In collaboration with

From www.myops.org
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Motivation:
Decreasing Critical Dimensions in Semiconductor Manufacturing

<table>
<thead>
<tr>
<th>Technology Node</th>
<th>130nm</th>
<th>115nm</th>
<th>100nm</th>
<th>90nm</th>
<th>65nm</th>
<th>45nm</th>
<th>32nm</th>
<th>22nm</th>
<th>18nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>DRAM 1/2 pitch [nm]</td>
<td>130</td>
<td>115</td>
<td>100</td>
<td>90</td>
<td>65</td>
<td>45</td>
<td>32</td>
<td>22</td>
<td>18</td>
</tr>
<tr>
<td>MPU printed gate length [nm]</td>
<td>90</td>
<td>75</td>
<td>65</td>
<td>53</td>
<td>35</td>
<td>25</td>
<td>18</td>
<td>13</td>
<td>10</td>
</tr>
<tr>
<td>MPU etched gate length [nm]</td>
<td>65</td>
<td>53</td>
<td>45</td>
<td>37</td>
<td>25</td>
<td>18</td>
<td>13</td>
<td>9</td>
<td>7</td>
</tr>
</tbody>
</table>

From the 2003 International Technology Roadmap for Semiconductors (ITRS) for CD Metrology

Metrology techniques and physical standards with nanometer scale uncertainty are required to support these specifications
For length (or width) measurements the resolution of the measurement probe must be minimized or accounted for.

Spacing measurements are relatively unaffected by the resolution of the measurement probe.

Length or width measurements are affected by the resolution of the measurement probe.
Optical Microscopy: Diffraction Limit

Optical Microscopy of Narrow Lines on Si

R. Silver et al.
SEM Metrology: Line Broadening due to Electron Mean Free Path

Monte Carlo simulation of the electron beam interaction at the edge of a silicon line. The simulation yields the predicted “secondary” electron signal showing the offset of the peak of the signal relative to the edge.

Probe microscopy has a short range interaction, < 1 nm, and under certain conditions is capable of measuring surfaces with atomic resolution.

2.5 nm x 2.5 nm AFM image of sorbic acid molecule on graphite, measurement by T. Albrecht et al., in D. Rugar and P. Hansma, *Physics Today*, p. 20 (Oct. 1990).
Conventional AFM versus Critical Dimension AFM (CD-AFM)

\[ W_{\text{apparent}}(h) = W_{\text{top}} + TW(h) \]

\[ W_{\text{apparent}}(h) = W(h) + TW_{\text{zeroth order}} + \varepsilon_{\text{higher order}} \]

Conventional AFM

CD-AFM
SCCDRM Project
(Single Crystal Critical Dimension Reference Materials)

• Fabricate chips with linewidth structures having vertical sidewalls and uniform linewidths using lattice plane selective etching.

  • Linewidths range from 50 nm to 250 nm.
  • Use CD-AFM as the transfer metrology for the distributed wafers.
  • Calibrate the CD-AFM tip offsets with lattice plane counts from HRTEM (High-Resolution Transmission-Electron Microscopy) images.
Use of CD-AFM for Calibration of SCCDRM Linewidth Standards

Target Sample 1

Target Sample 2

\ldots

Target Sample n

Target samples distributed to SEMATECH member companies

Monitor Sample

CD-AFM

Transfer Samples

HRTEM

Transfer Samples

Target samples distributed to SEMATECH member companies
Example of CD-AFM Data
Example of HRTEM Image
AFM/HRTEM Regression:

Slope: 0.996 +/- 0.0053 (k = 1)
Final Offset and Expanded Uncertainty (k=2) : 1.03 nm +/- 0.58 nm
(from weighted averaging of individual offsets)
The SCCDRM

navigation guides called "markers"
Recommended Use of SCCDRM

- Actual Product (incl. Resist)
- NIST/ISMT Si Linewidth Standard
- CD-SEM
- CD-AFM
- Golden Wafer (same type as product)
How to Verify the Claimed Uncertainty?

Two Comparisons with Independent Measurements


• Annular Dark Field TEM (ADF-TEM), a different TEM method – N.G. Orji et al., Proc. SPIE, 6518, 651810 (2007).
Commercial Critical Dimension Standard (CCDS)
SCCDRM vs. CCDS Comparison

Comparison of CCDS70 Values with SCCDRM calibration

Large Tip Wear Uncertainties

Measurements performed using SXM320 in NIST/AML.
HRTEM

(a)

Annular Dark Field TEM

(b)
N.G. Orji et al. SPIE (2007)
Summary: CD-AFM Linewidth Measurements

• Measurement of Transfer Specimens with both CD-AFM and HR-TEM provides calibration of CD-AFM tip widths and SCCDRMs

• SCCDRM chips with calibrated linewidths in pocket wafers were released to SEMATECH Member Companies in 2004.

• The final widths ranged from about 50 nm to 250 nm, with expanded uncertainties (k=2) typically ~ 2 nm.

• CD-AFM tip width calibration with 1 nm (k=1) uncertainty is now possible.

• The results agree with two sets of independent measurements

• Have Initiated a project to calibrate and distribute additional SCCDRMs as a Standard Reference Material
Image Stitching Linewidth Measurement:

Goal
An independently traceable probe-based method for measurement of linewidth
AFM Image Stitching for Linewidth Measurement

AFM Cantilever Tip

Carbon Nanotube

Line to be Measured
AFM Image Stitching for Linewidth Measurement
AFM Image Stitching for Linewidth Measurement

Line at 0° Orientation

Rotate Surface by 180°

AFM Topography Image at 0° Orientation
Generate Second Topographic Image of Rotated Line

Line at 180° Orientation

AFM Topography Image at 180° Orientation
Form Composite Image from the Undistorted Data
SEM micrographs of multi-walled carbon nanotube tip used to collect images #1 and #2

Side view

Top view

NIST
National Institute of Standards and Technology
## Linewidth Measurement Results
(after nanotube radius correction)

<table>
<thead>
<tr>
<th>Nanoscope AFM</th>
<th>CD-AFM</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Sample #1</td>
</tr>
<tr>
<td></td>
<td>Top</td>
</tr>
<tr>
<td>Sample #1</td>
<td>536.6 nm</td>
</tr>
<tr>
<td>Sample #2</td>
<td>(using X3D)</td>
</tr>
</tbody>
</table>
Preliminary Uncertainty Budget for Image Stitching Linewidth Measurement

Currently all components are type B

- Scale Calibration (1 %) 7 nm
- Template Matching (3 pixels) 14 nm
- Nanotube Radius (10 % of 80 nm) 8 nm
- Probe Compliance 3 nm

Quadratic Sum ±18 nm (k = 1)
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AFM image of silicon (111) atomic step specimen with native oxide

Relation between miscut angle and average width of terraces

Si(111) with 0.09° miscut

Si(111) with 0.015° miscut

Samples after heating in UHV reveal single atomic terraces, and the average width of the terraces is 0.314 nm/tanθ, θ the miscut angle.
Calibrated Atomic Force Microscope with AFM Sensor on Kinematic Mount
Comparison of Silicon Step Height Results

- **LEED Value:** 312 pm ± 14 pm
- **C-AFM Value:** 304 pm ± 8 pm
- **Grazing X-ray Value:** 340 pm ± 60 pm
- **Recommended Value:** 312 pm ± 12 pm (k=2)
Illustration of Step Height Calculation for a Single Profile
Typical Area Selection for Step Height Analysis on Si (111)

What is the expected variability for a user?

scan size is 1.44 µm
Results of Industrial Measurement Comparison

Five participants, including NIST (using Nanoscope 3, not C-AFM)

Table I. Summary of Final Results for Silicon Step Height Samples

<table>
<thead>
<tr>
<th>Participant</th>
<th>Number of Topographic Images</th>
<th>Average Step Height Value (pm)</th>
<th>One Standard Deviation (pm)</th>
<th>Average Number of Steps in Each Image</th>
<th>Number of Areas Measured</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>7</td>
<td>325</td>
<td>12</td>
<td>6</td>
<td>268</td>
</tr>
<tr>
<td>B</td>
<td>9</td>
<td>302</td>
<td>8</td>
<td>4</td>
<td>90</td>
</tr>
<tr>
<td>C</td>
<td>10</td>
<td>317</td>
<td>4</td>
<td>7</td>
<td>100</td>
</tr>
<tr>
<td>D</td>
<td>4</td>
<td>306</td>
<td>3</td>
<td>6</td>
<td>180</td>
</tr>
<tr>
<td>E</td>
<td>6</td>
<td>311</td>
<td>3</td>
<td>6</td>
<td>60</td>
</tr>
</tbody>
</table>

Standard Practice for Calibrating the \( Z \)-Magnification of an Atomic Force Microscope at Subnanometer Displacement Levels Using Si (111) Monatomic Steps

FIG. 1 Illustration of Selected Areas

FIG. 3 Illustration of the Step-Height Algorithm Used

FIG. 4 Image of a Silicon Monatomic Stepped Surface
Observations

• The structure of NNI provides for strong *coordination* among agencies with different missions.

• Nanoscale length metrology plays an important role in the semiconductor industry and in particle based nanotechnology and will be important in the manufacture of many other nano-scale components and functional features.

• Probe-based Microscopy is capable of nm uncertainties for width and pm-scale uncertainties for height of features on surfaces.
Probable Future Developments

Probe-based microscopy to improve its capability for high-resolution probing of soft structures

SEM image of Buckypaper, Chastek/Talbott NIST

A. Vladar, He ion microscopy of 60 nm gold

He ion microscope under development for nanoscale length measurement