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NIST
...working with industry to foster innovation, trade, security and jobs

The National Nanotechnology Initiative and Nano-scale Length Metrology

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Simposio de Metrologia
Querétaro, Mexico
October 22, 2008

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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

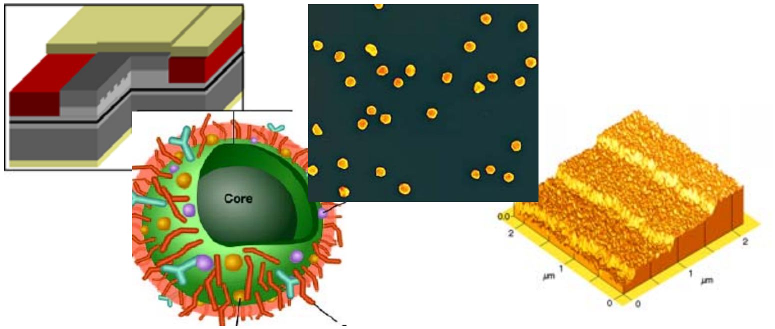
Certain Materials Provided by:

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*

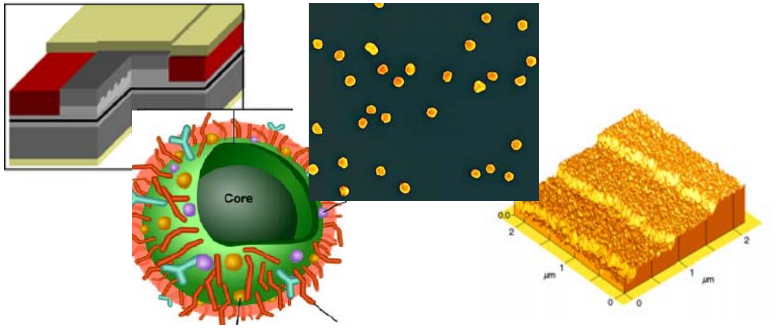


What is the NNI?

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

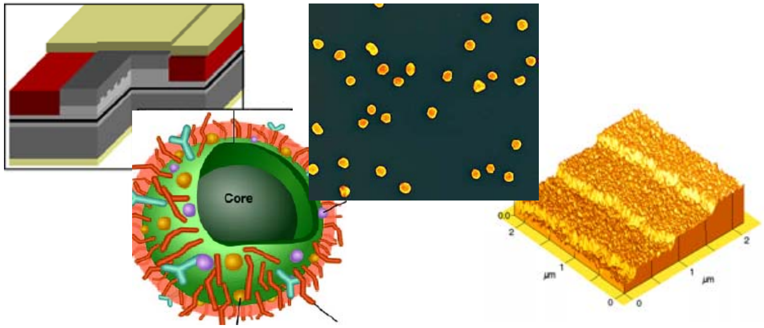
- Coordinate Federal nanotechnology R&D by serving as a central locus for communication, cooperation, and collaboration among agencies that wish to participate (currently 25)
- Provide a vision of long-term opportunities and benefits of nanotechnology

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.



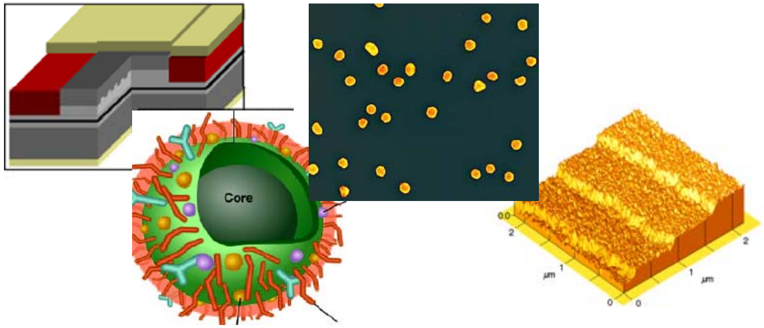
Vision of the NNI:

"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



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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)

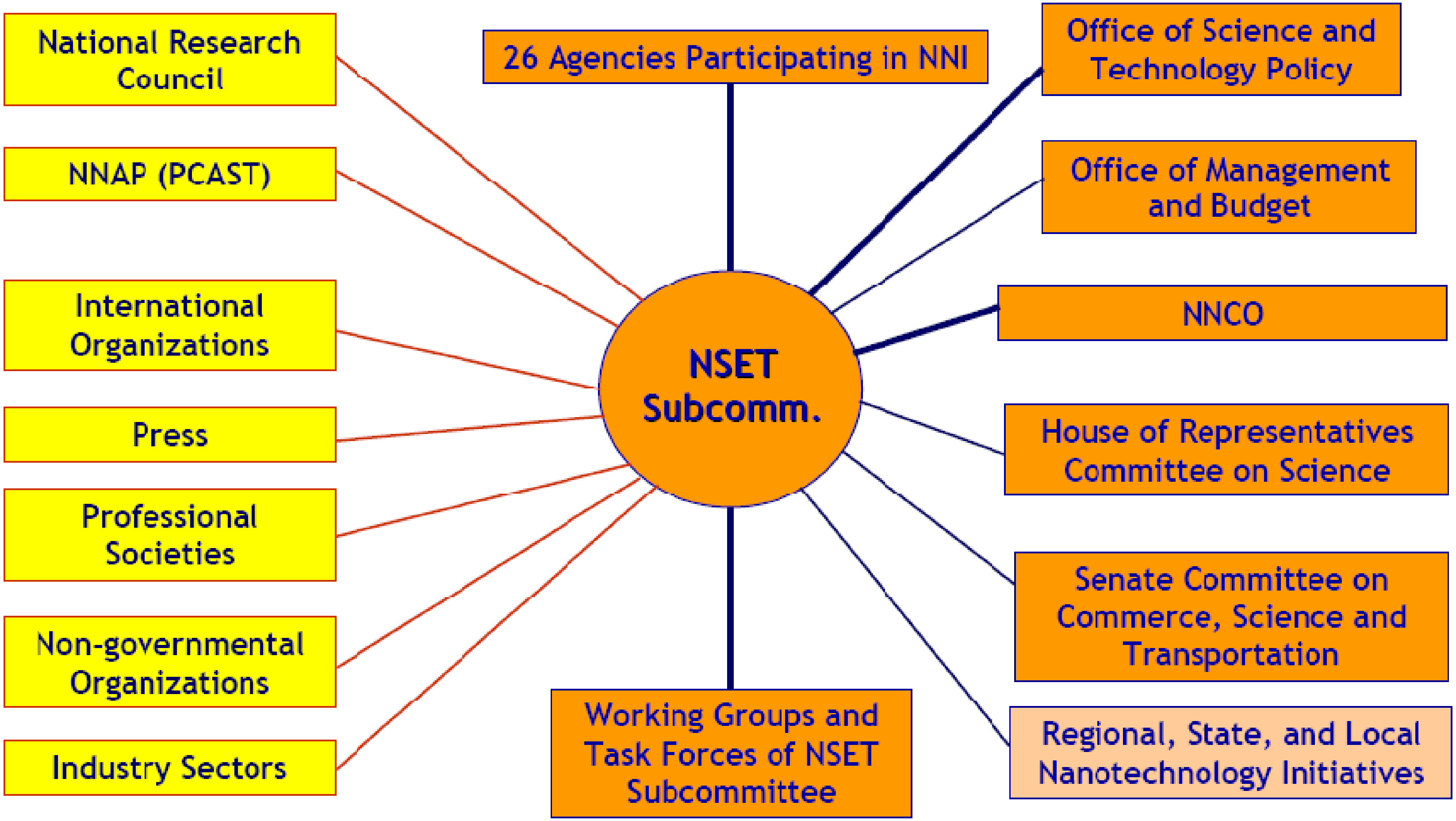
•Major Research Facilities and Instrumentation Acquisition – infrastructure

•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

NSET Subcomm.

Working Groups and Task Forces of NSET Subcommittee

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

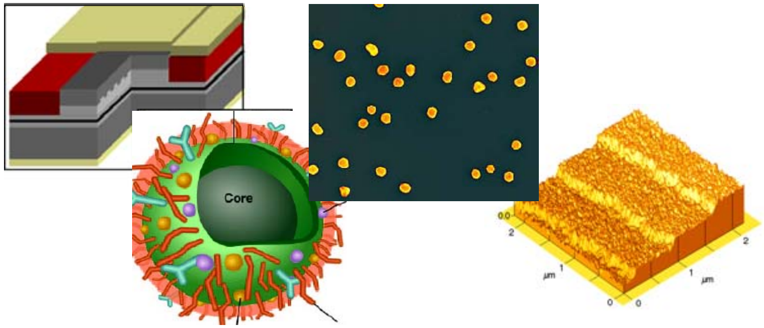
Regional, State, and Local Nanotechnology Initiatives

Global Issues in Nanotechnology Working Group

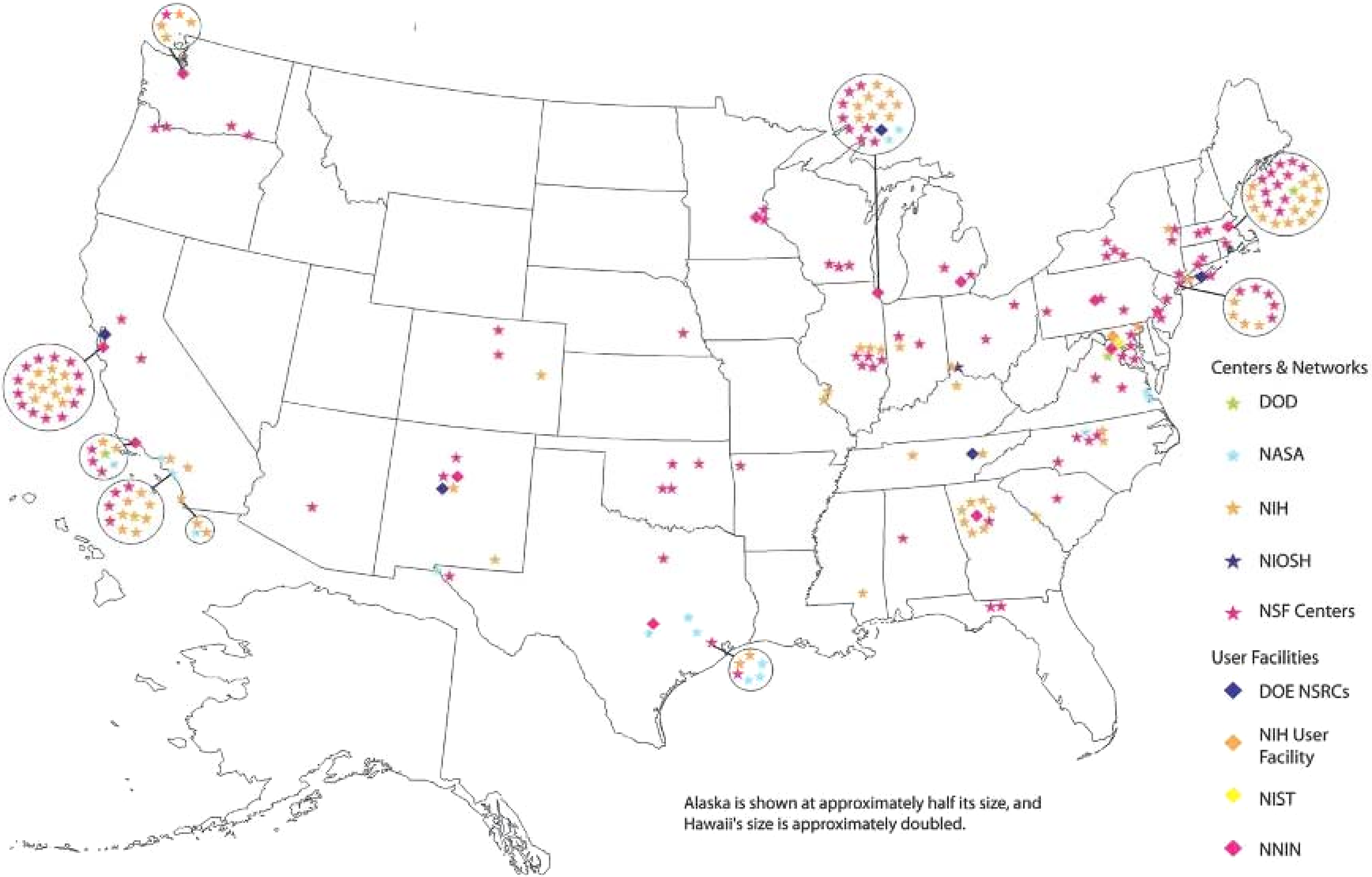
- Coordinates international activities of the agencies in nanotechnology
- Monitors foreign nanotechnology programs
- Seeks to broaden international cooperation and communication with respect to nanotechnology R&D

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.

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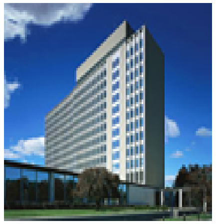
Center for Nanoscale Science and Technology

photo courtesy HDR Architecture, Inc.
Steve Hall © Hedrich Blessing

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

cnst.nist.gov

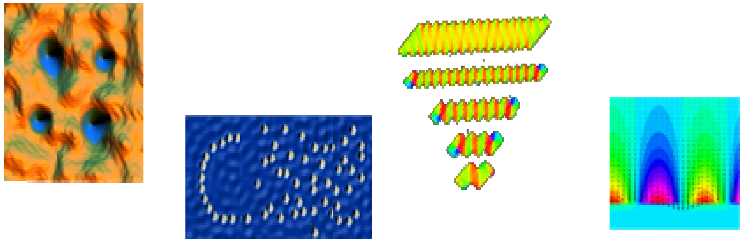


Collaborations

Nanofabrication

Future Electronics

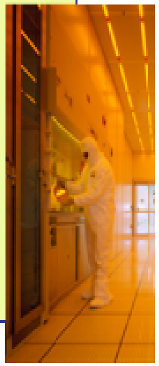
Energy



740 m^2 Class 100 Clean-room includes

- Atmospheric furnaces
- LPCVD
- Plasma etching
- Photolithography
- Metallization
- Nano-imprinting
- Wafer dicing
- E-beam lithography
- AFM
- FIB
- FESEM

Process engineering and nanofabrication experts on staff.



Open Access to Nanofabrication and Measurement

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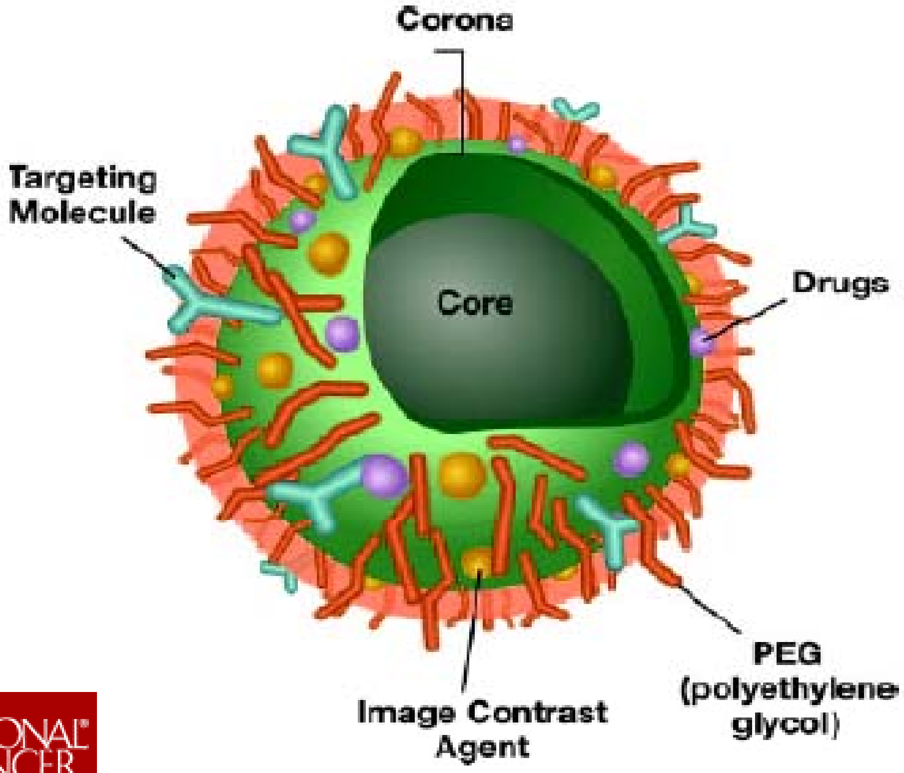
Applications at NIST:
Particle Standards for Medical Industries

Particle-based Delivery Systems in Medicine for Diagnosis and Treatment

⇒ Size matters



From D. Kaiser

Part of Joint Program with FDA NATIONAL CANCER INSTITUTE

NIST National Institute of Standards and Technology

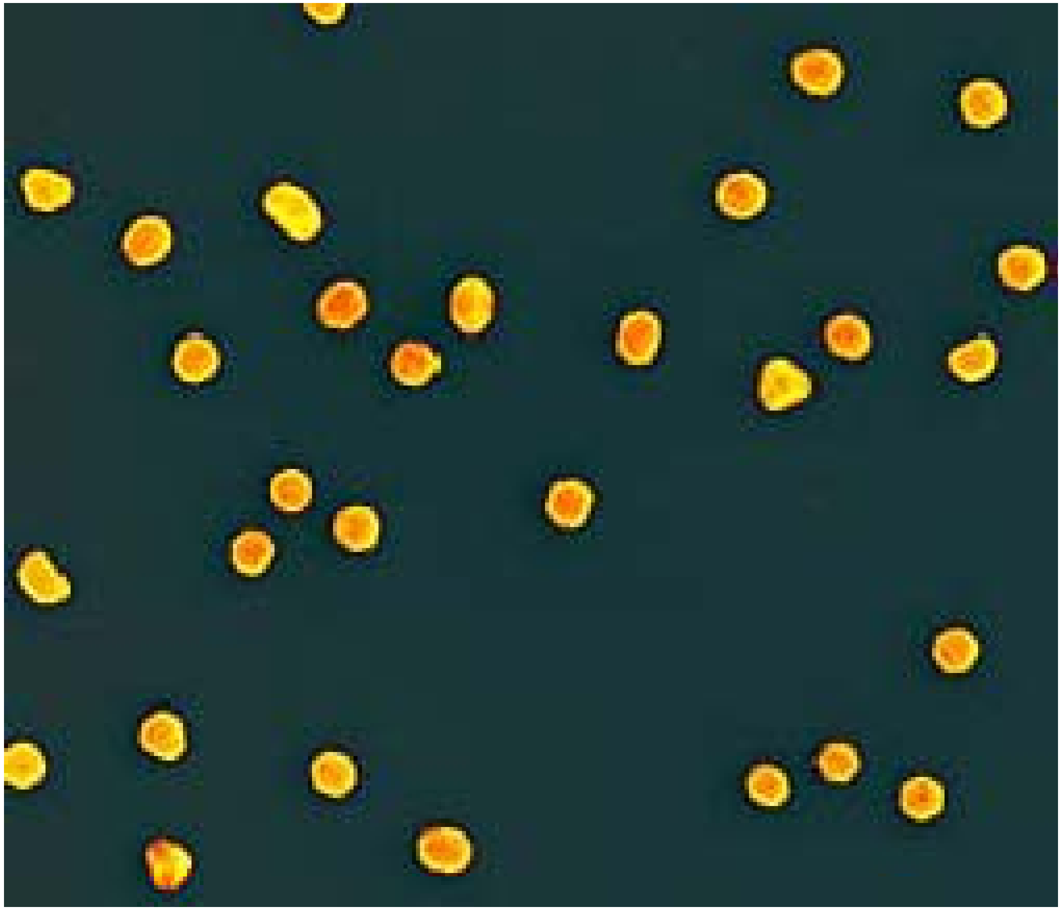
NIST Reference Materials (RM) 8011, 8012, 8013 – Gold Nanoparticles

With Mean Sizes

8011 – 10 nm

8012 – 30 nm

8013 – 60 nm



A. Vladar, SEM of RM 8012 particles

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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

Technique	Analyte Form	Particle Size (nm)
Atomic Force Microscopy	dry, deposited on substrate	24.9 ± 1.1
Scanning Electron Microscopy	dry, deposited on substrate	26.9 ± 0.1
Transmission Electron Microscopy	dry, deposited on substrate	27.6 ± 2.1
Differential Mobility Analysis	dry, aerosol	28.4 ± 1.1
Dynamic Light Scattering	liquid suspension	
173° scattering angle (backscatter)		28.6 ± 0.9
90° scattering angle		26.5 ± 3.6
Small-Angle X-ray Scattering	liquid suspension	24.9 ± 1.2

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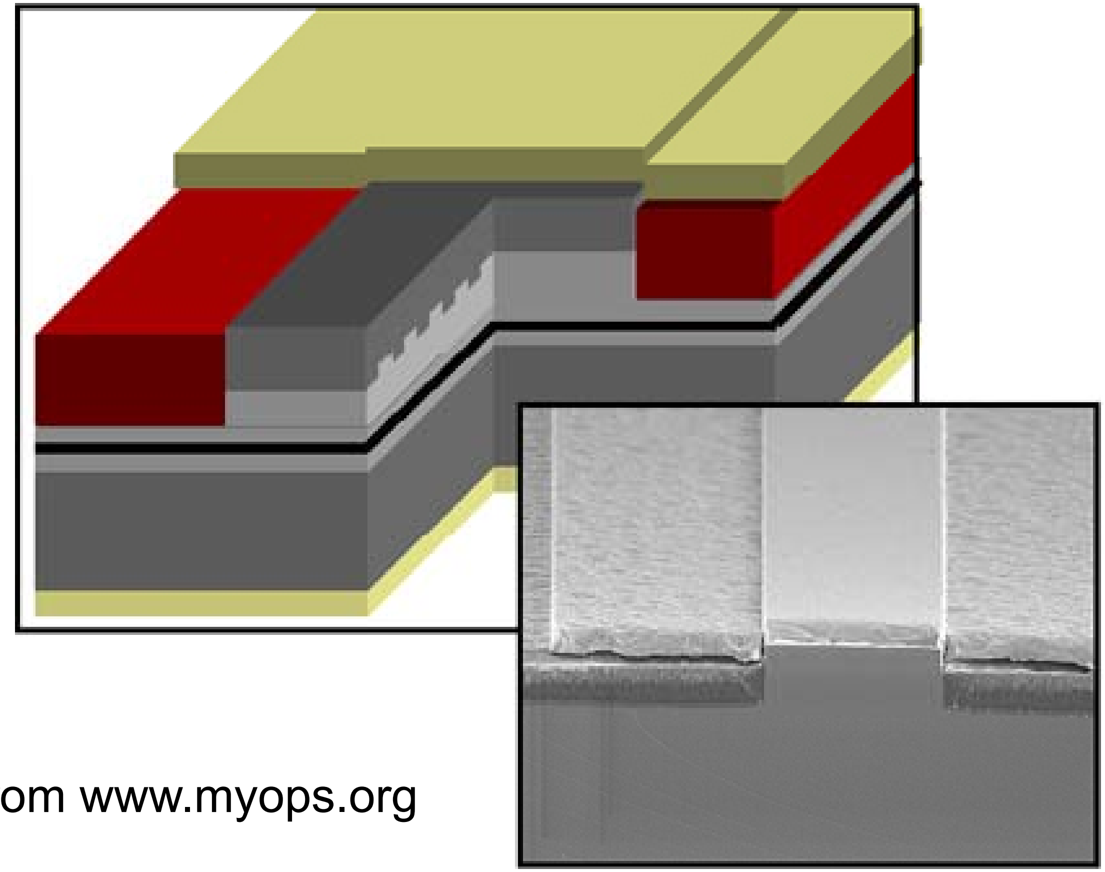
Applications at NIST:

Length Metrology for Semiconductor Manufacturing

- Linewidth
- Step Height
- Line Edge Roughness
- Pitch

In collaboration with

SEMATECH



From www.myops.org

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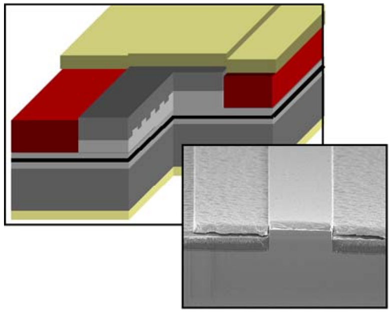
Motivation:
Decreasing Critical Dimensions in Semiconductor Manufacturing

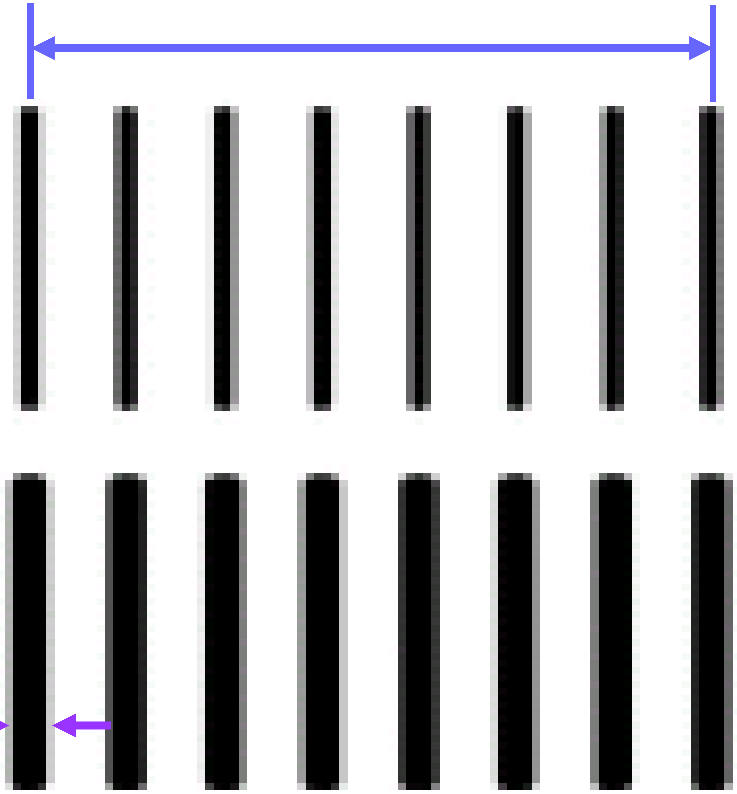
Technology Node	130nm	115nm	100nm	90nm	65nm	45nm	32nm	22nm	18nm
Year of Production	*2001*	*2002*	*2003*	*2004*	*2007*	*2010*	*2013*	*2016*	*2018*
DRAM 1/2 pitch [nm]	130	115	100	90	65	45	32	22	18
MPU printed gate length [nm]	90	75	65	53	35	25	18	13	10
MPU etched gate length [nm]	65	53	45	37	25	18	13	9	7

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



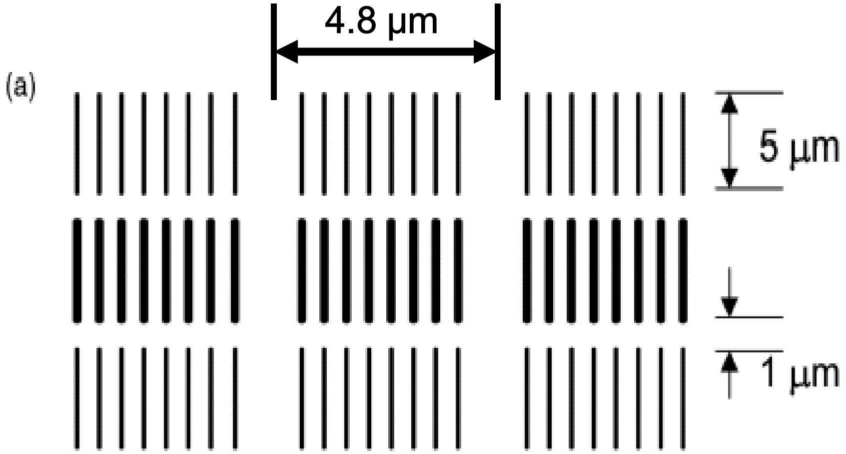
For length (or width) measurements the resolution of the measurement probe must be minimized or accounted for



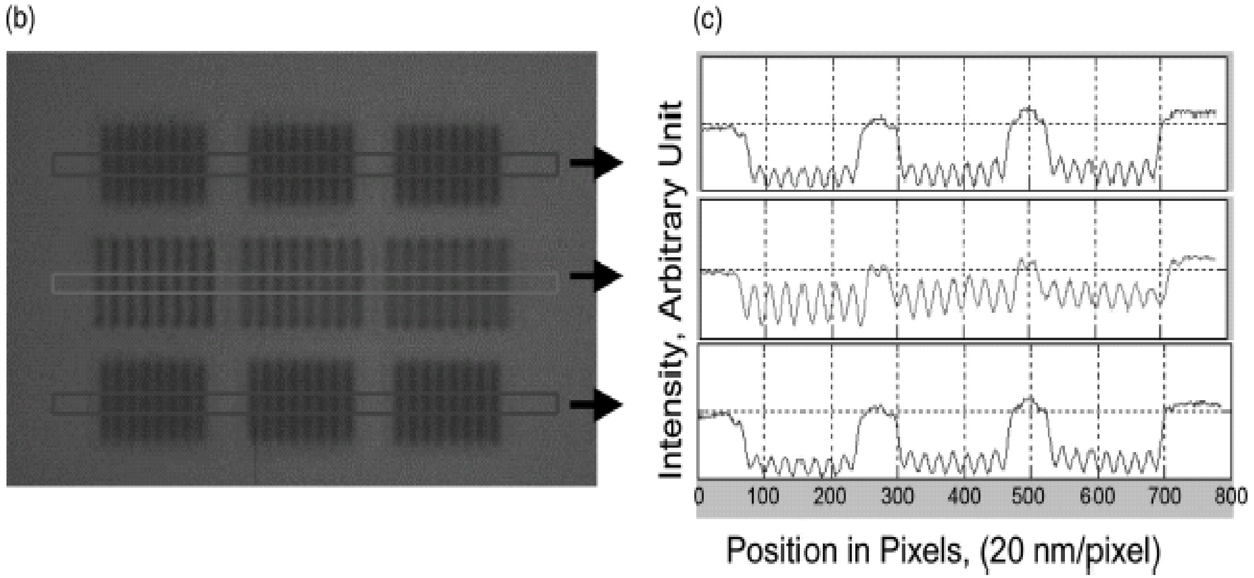
Spacing measurements are relatively unaffected by the resolution of the measurement probe

Length or width measurements are affected by the resolution of the measurement probe

Optical Microscopy: Diffraction Limit

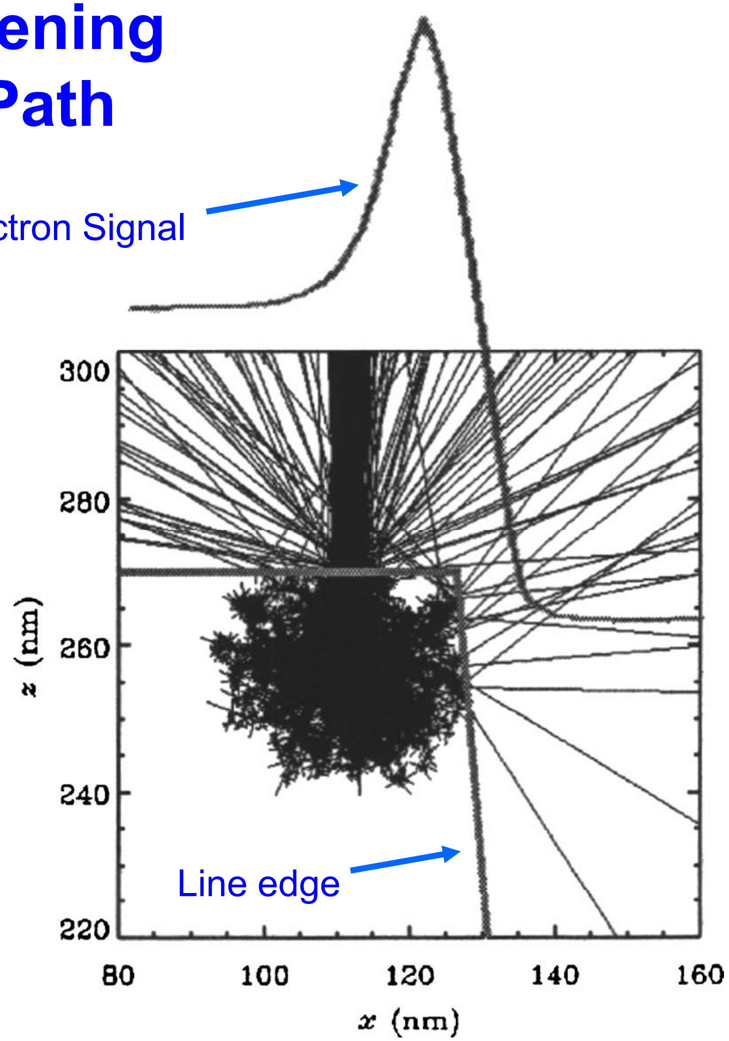


Optical Microscopy of Narrow Lines on Si



R. Silver et al.
Appl. Opt. **46**,
4248 (2007)

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.

M. Postek et al., J. Vac. Sci. Technol. B23, 3015 (2005)

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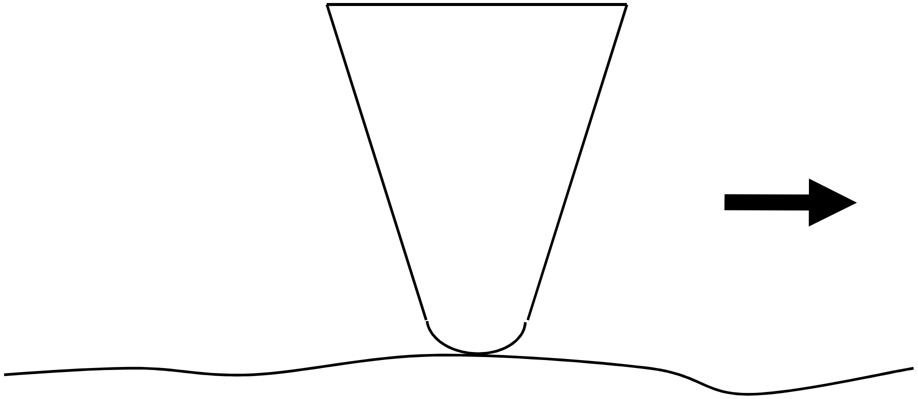
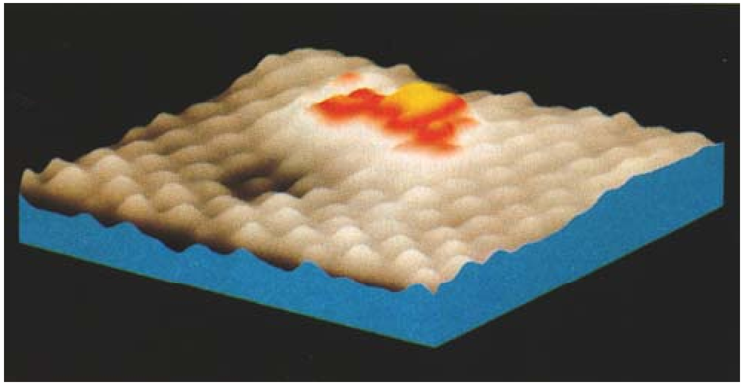
Probe microscopy has a short range interaction, < 1 nm, and under certain conditions is capable of measuring surfaces with atomic resolution



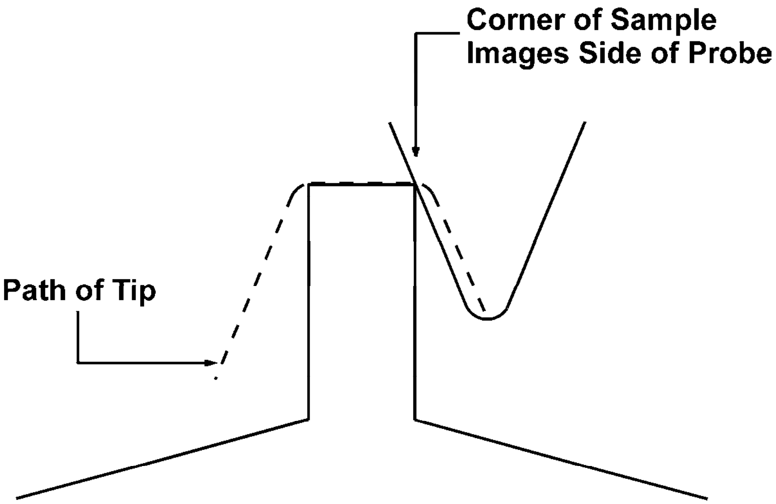


2.5 nm x 2.5 nm AFM image of sorbic acid molecule on graphite, measurement by T. Albrecht et al., in D. Rugar and P. Hansma, *Physics Today*, p. 20 (Oct. 1990).

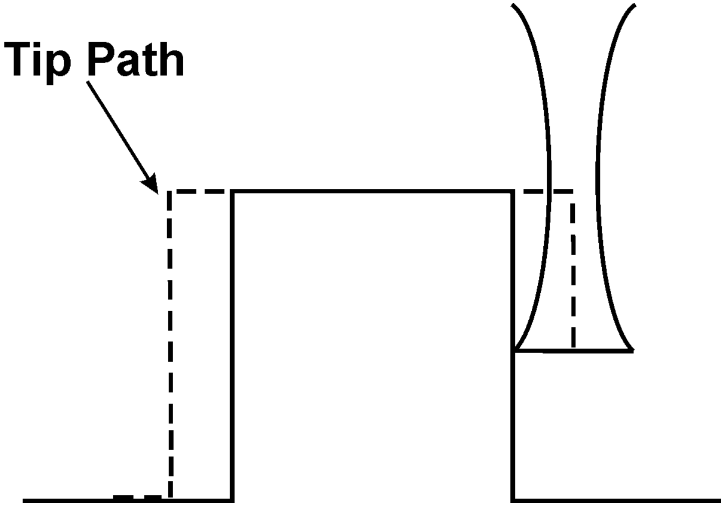
Conventional AFM versus Critical Dimension AFM (CD-AFM)

$W_{apparent}(h) = W_{top} + TW(h)$

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

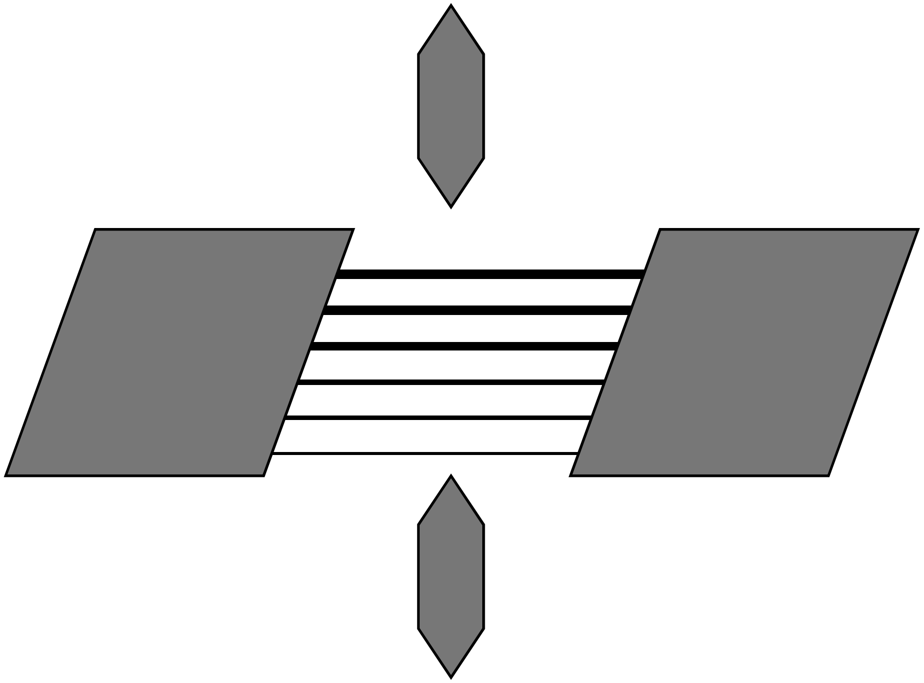


Conventional AFM



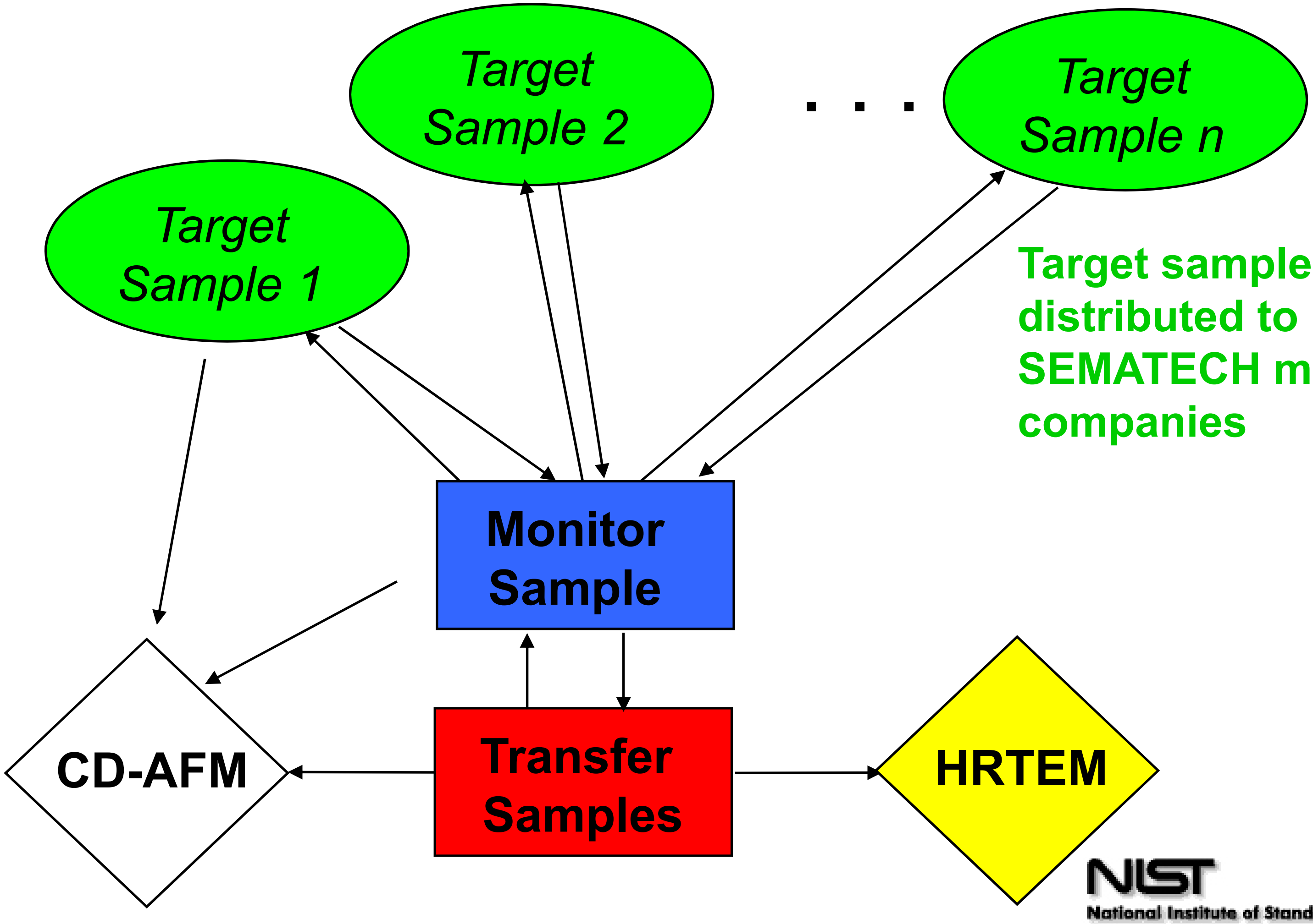
CD-AFM

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

. . .

Target Sample n

Target samples distributed to SEMATECH member companies

Monitor Sample

CD-AFM

Transfer Samples

HRTEM

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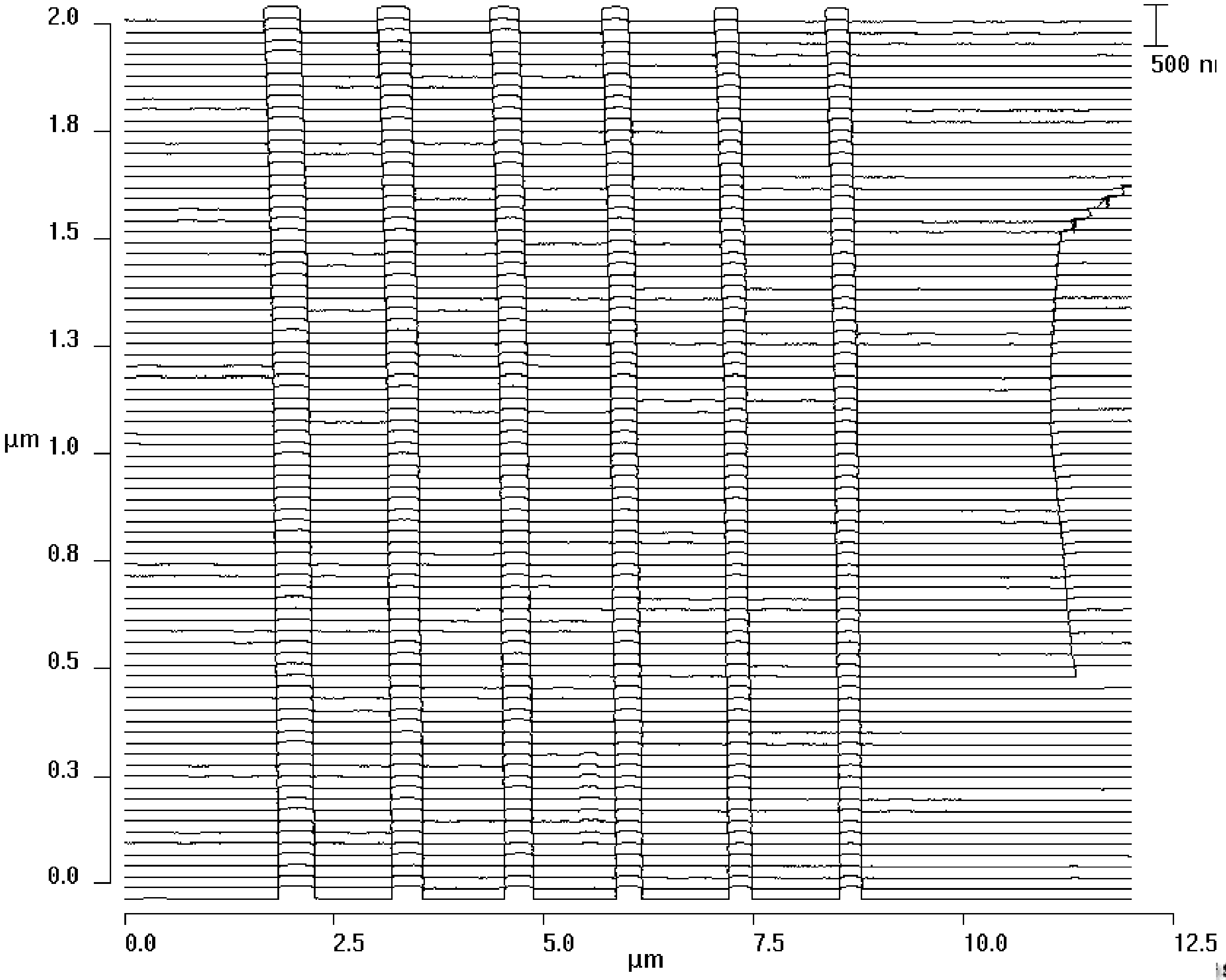
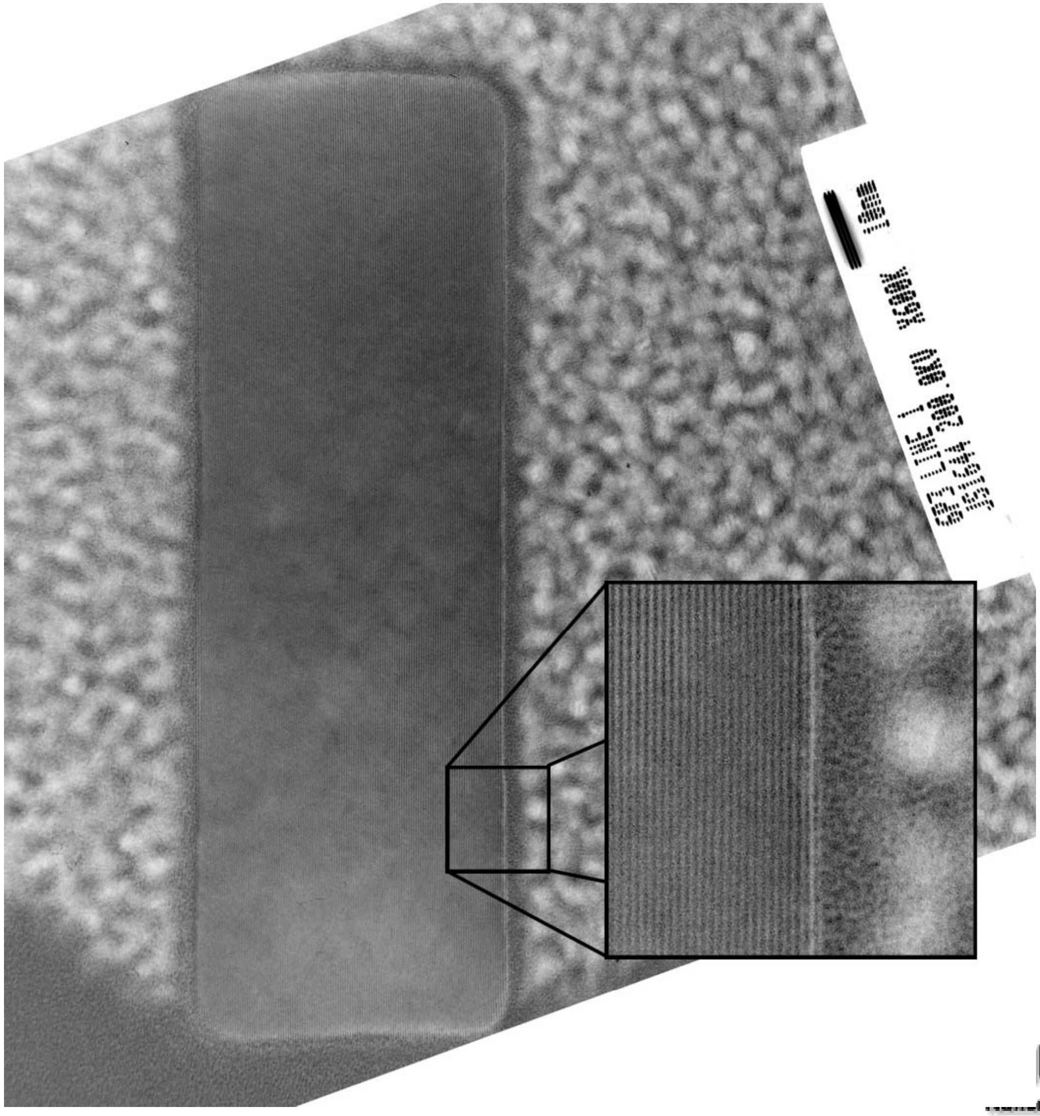
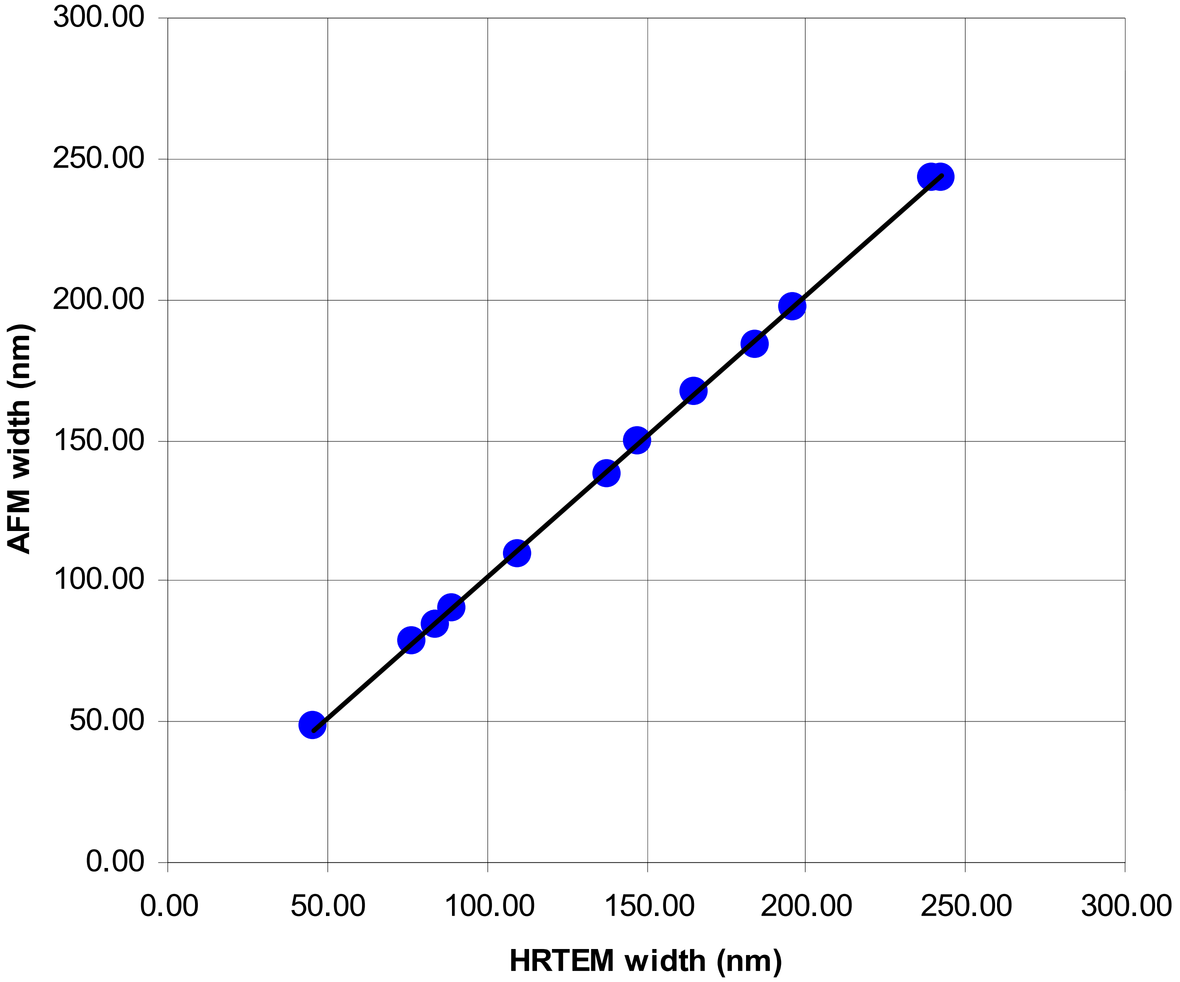
Example of CD-AFM Data



Example of HRTEM Image

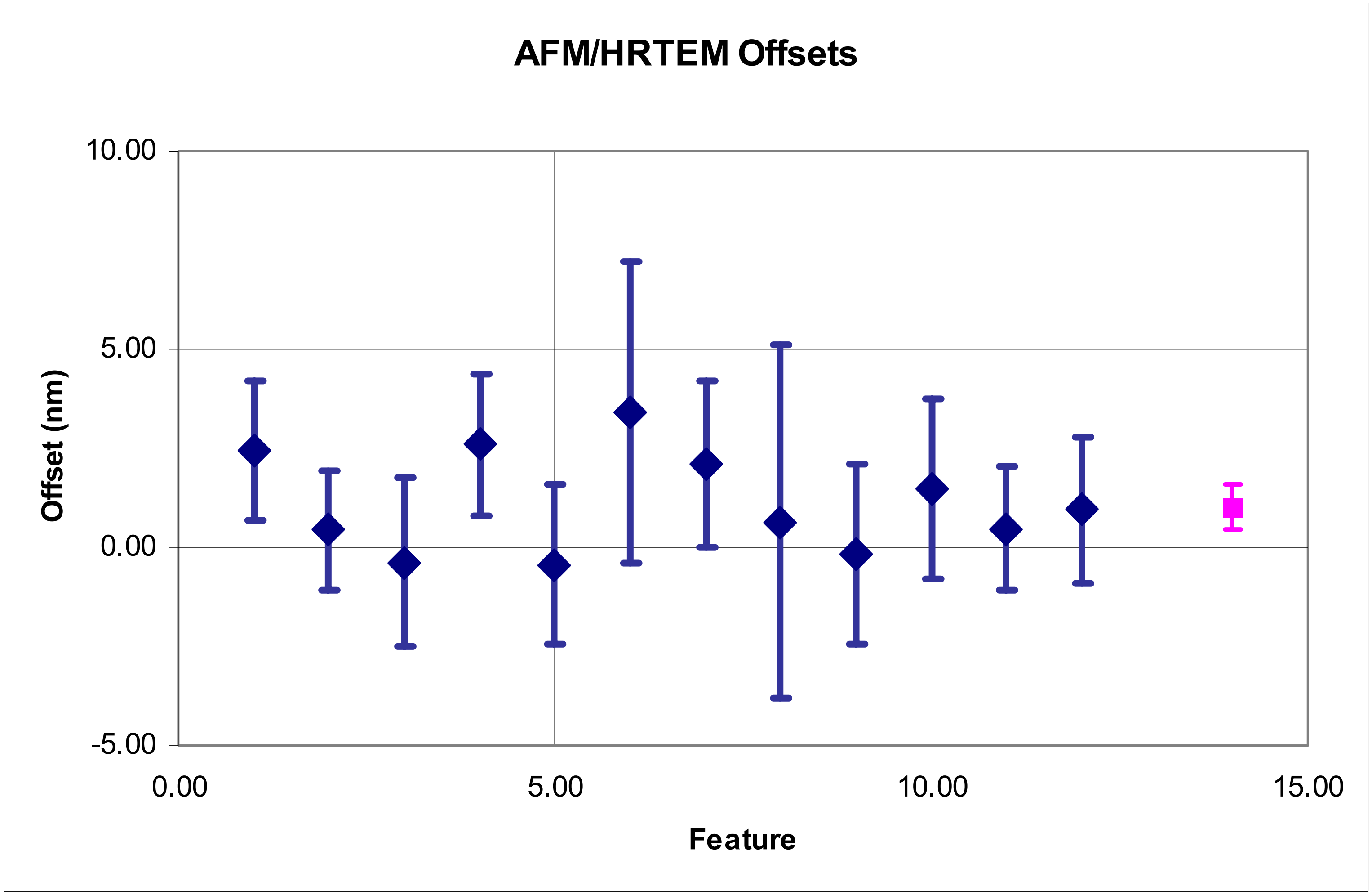


AFM/HRTEM Regression:



Slope: 0.996 +/- 0.0053 (k = 1)

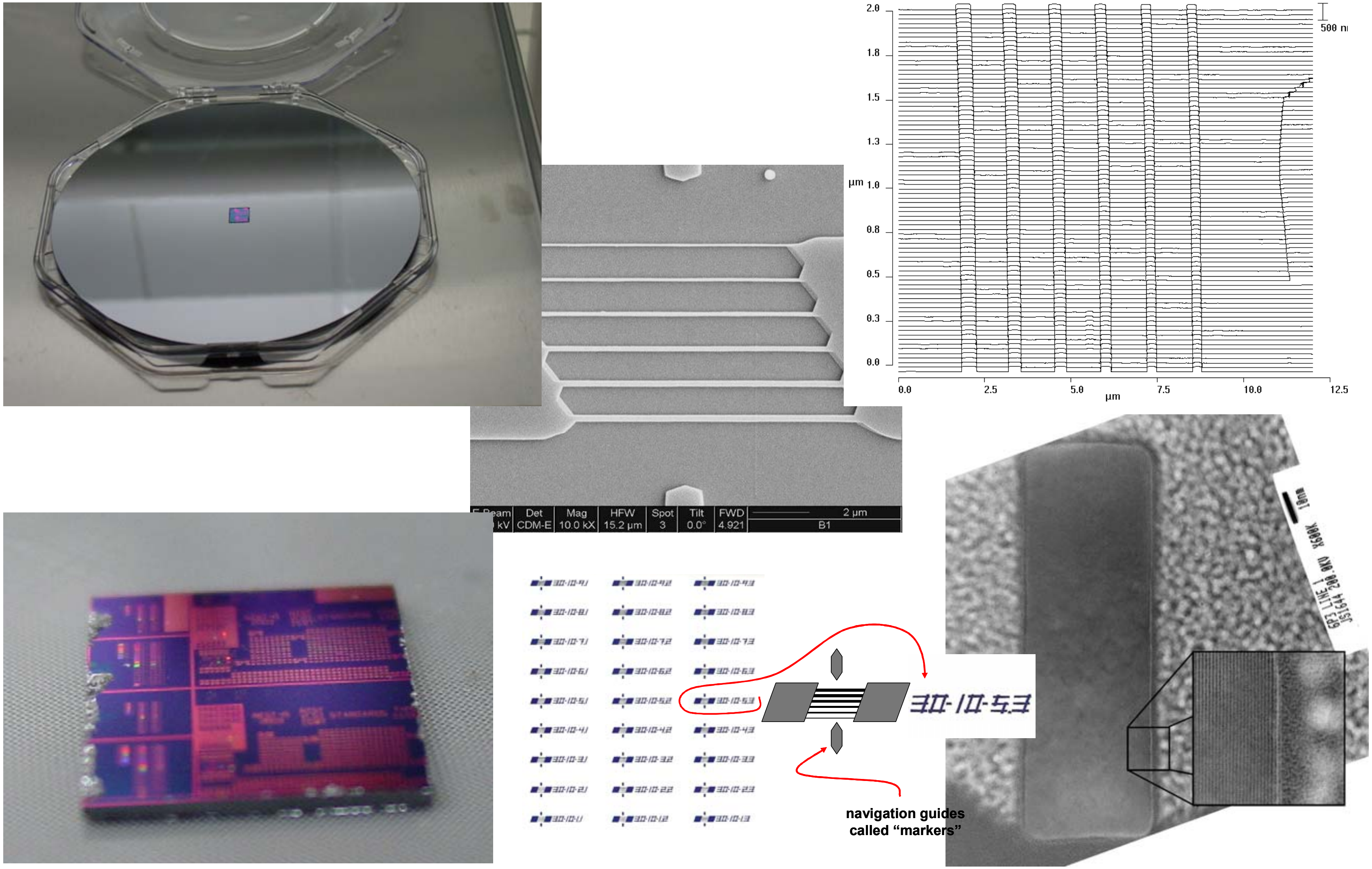
Distribution of AFM/HRTEM Offsets



Final Offset and Expanded Uncertainty (k=2) : 1.03 nm +/- 0.58 nm

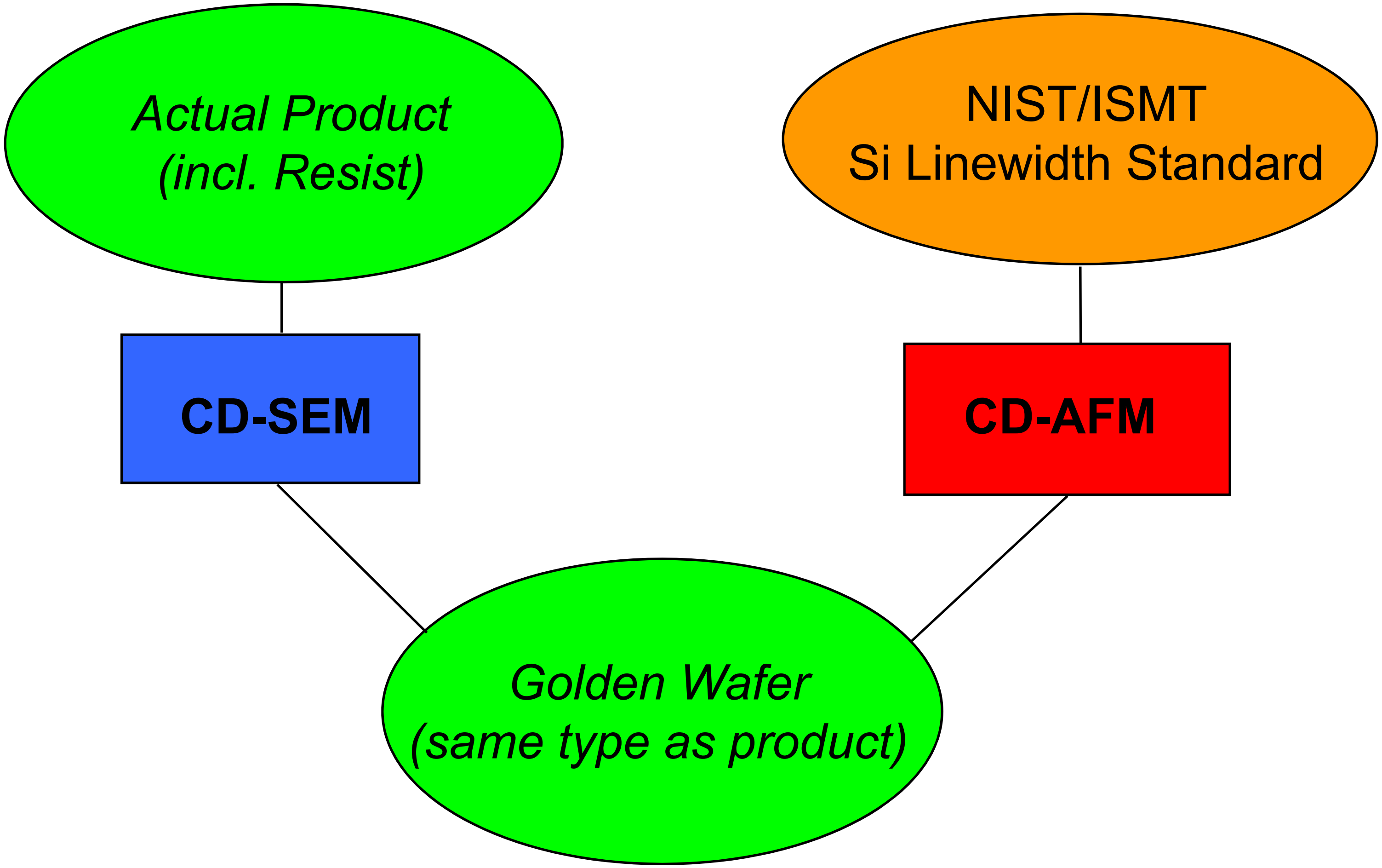
(from weighted averaging of individual offsets)

The SCCDRM



navigation guides
called “markers”

Recommended Use of SCCDRM



Actual Product
(incl. Resist)

NIST/ISMT
Si Linewidth Standard

CD-SEM

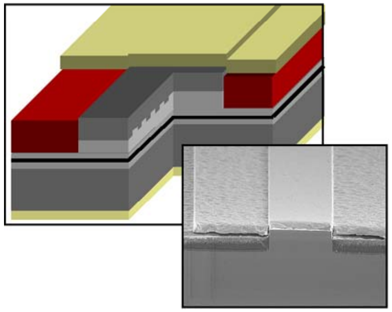
CD-AFM

Golden Wafer
(same type as product)

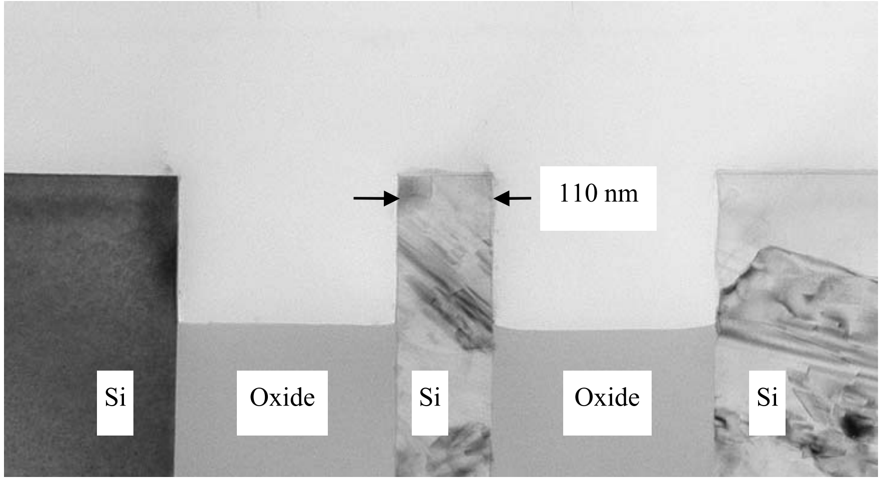
How to Verify the Claimed Uncertainty?

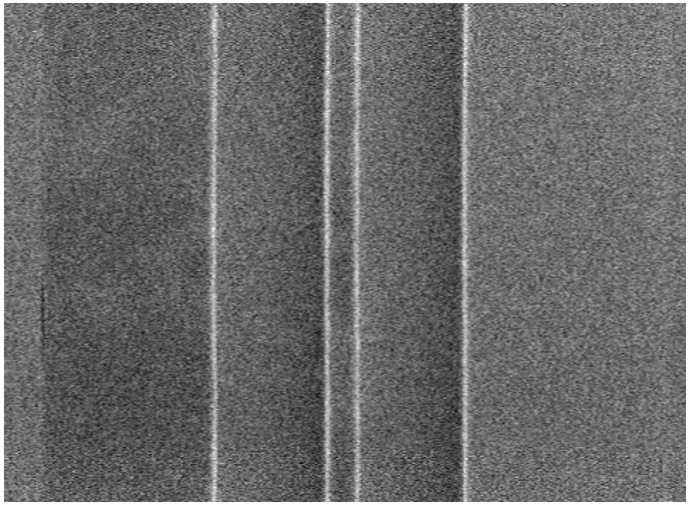
Two Comparisons with Independent Measurements

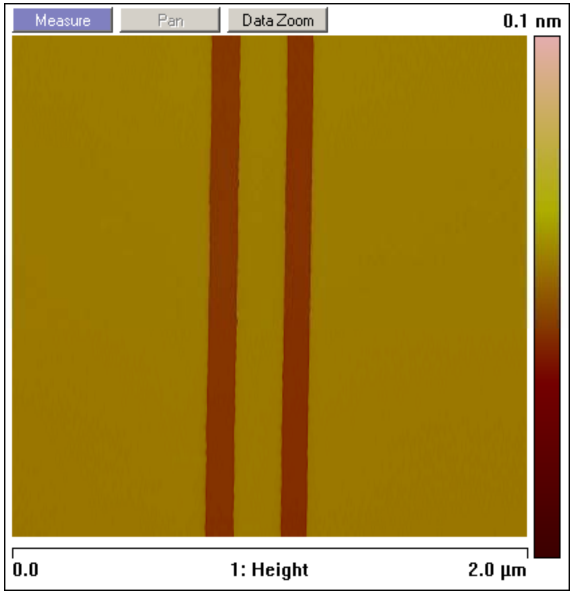
- **Commercial Linewidth Standard, independently calibrated –** R.D. Dixson & N.G. Orji, Proc. SPIE **6518**, 651816 (2007).
- **Annular Dark Field TEM (ADF-TEM), a different TEM method –** N.G. Orji et al., Proc. SPIE, **6518**, 651810 (2007).

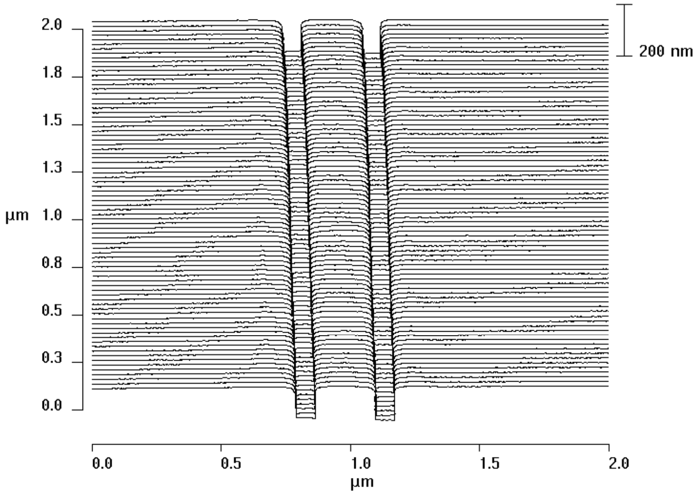


Commercial Critical Dimension Standard (CCDS)









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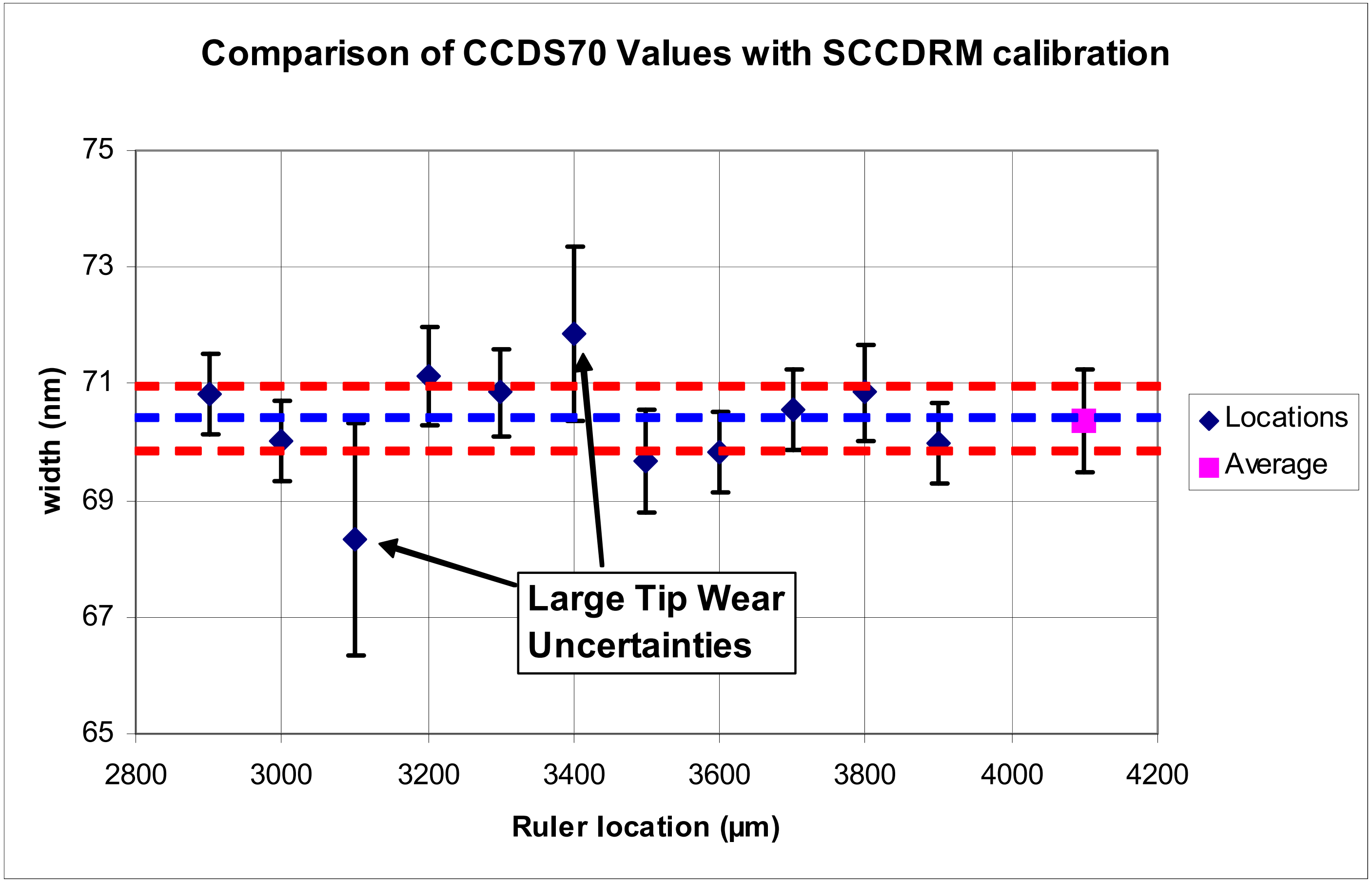
SCCDRM vs. CCDS Comparison



Large Tip Wear Uncertainties

Measurements performed using SXM320 in NIST/AML.

R.G. Dixson & N.G. Orji, SPIE **6518** (2007) doi: 10.1117/12.714032

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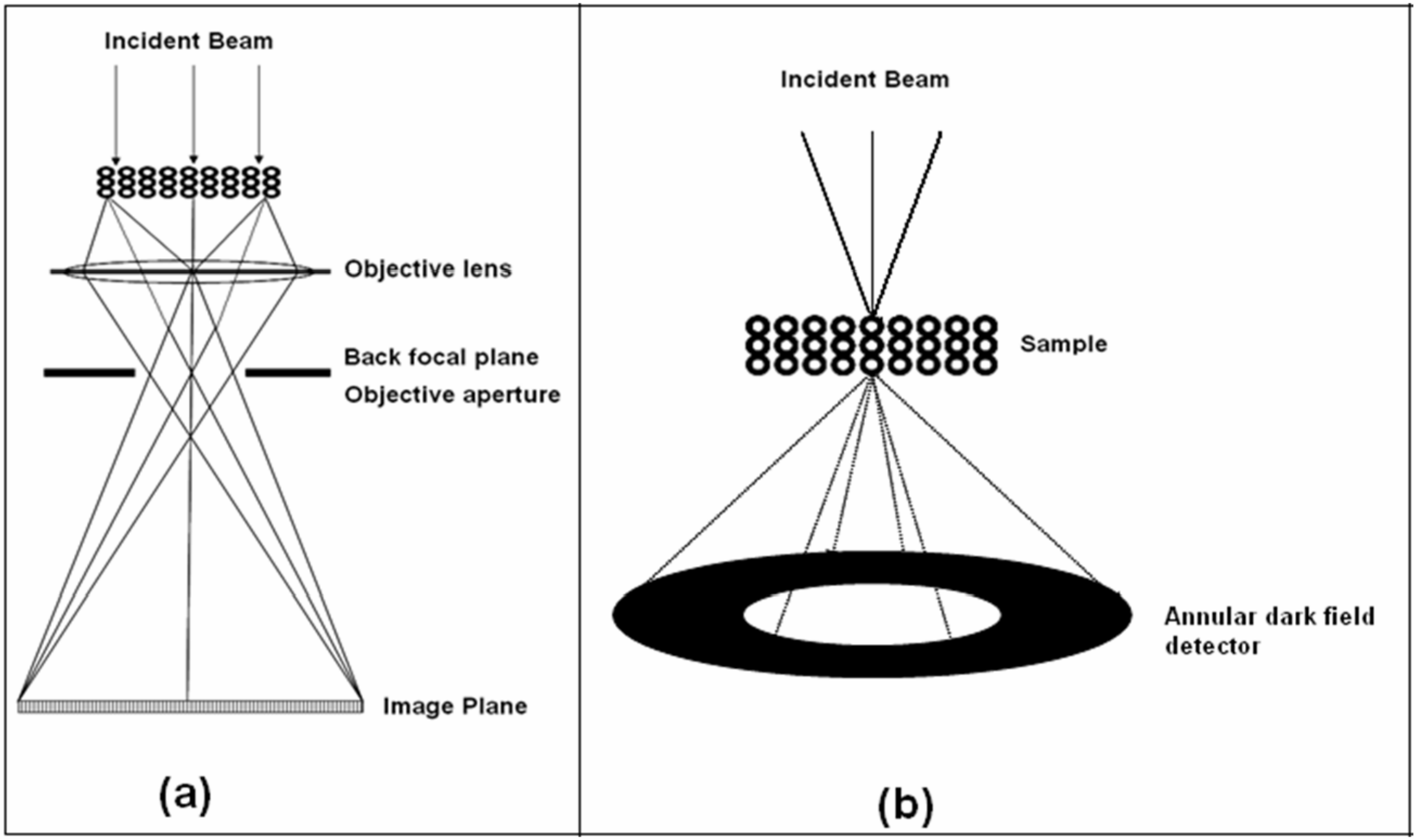
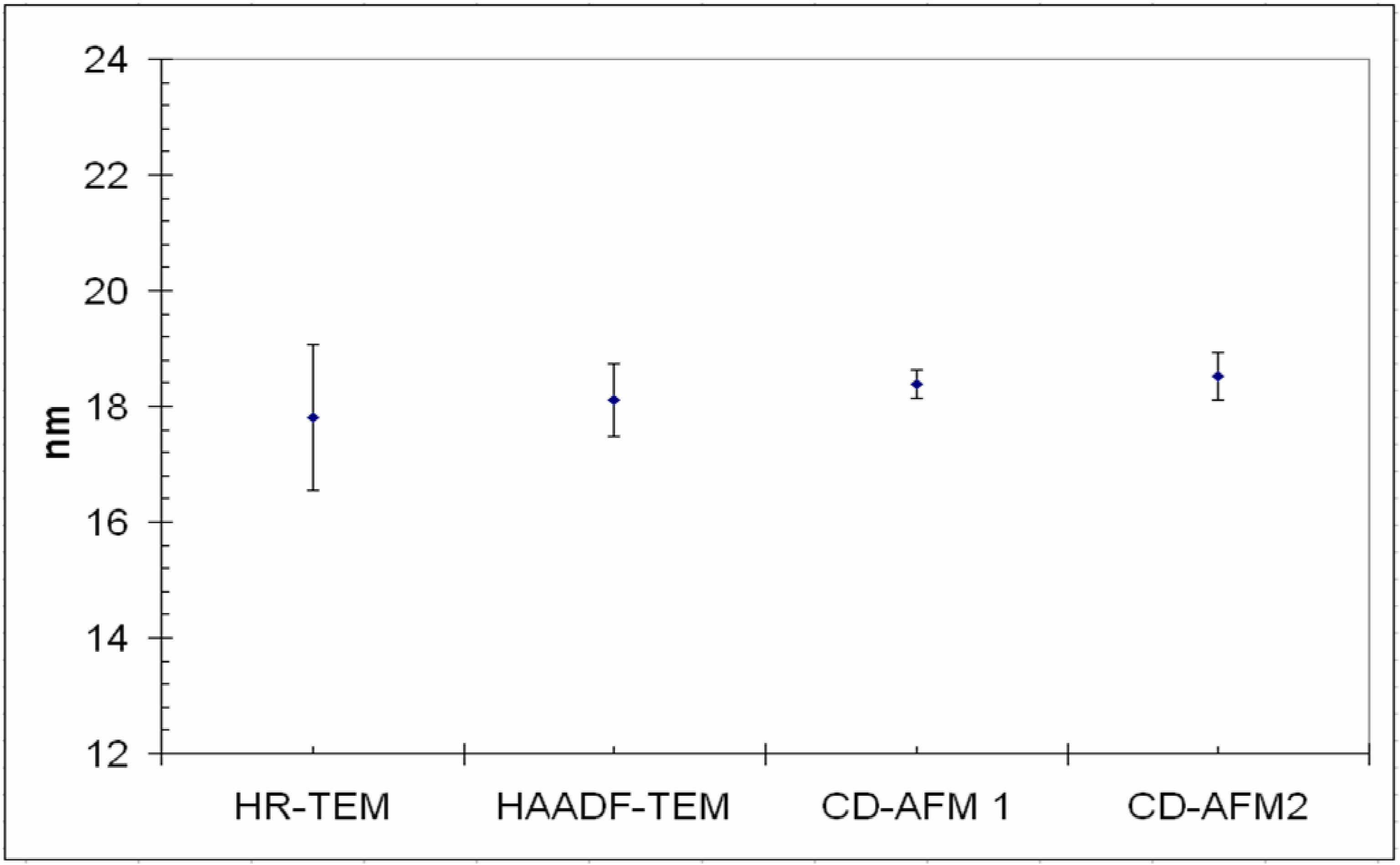
HRTEM

Annular Dark Field TEM





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.***

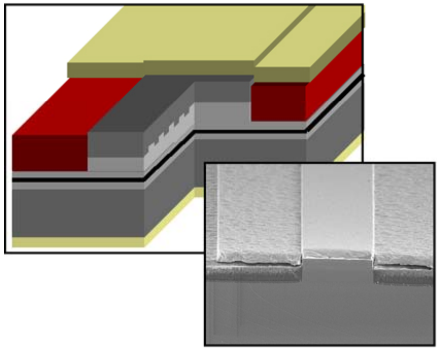
- ***The results agree with two sets of independent measurements***

- Have Initiated a project to calibrate and distribute additional SCCDRMs as a Standard Reference Material

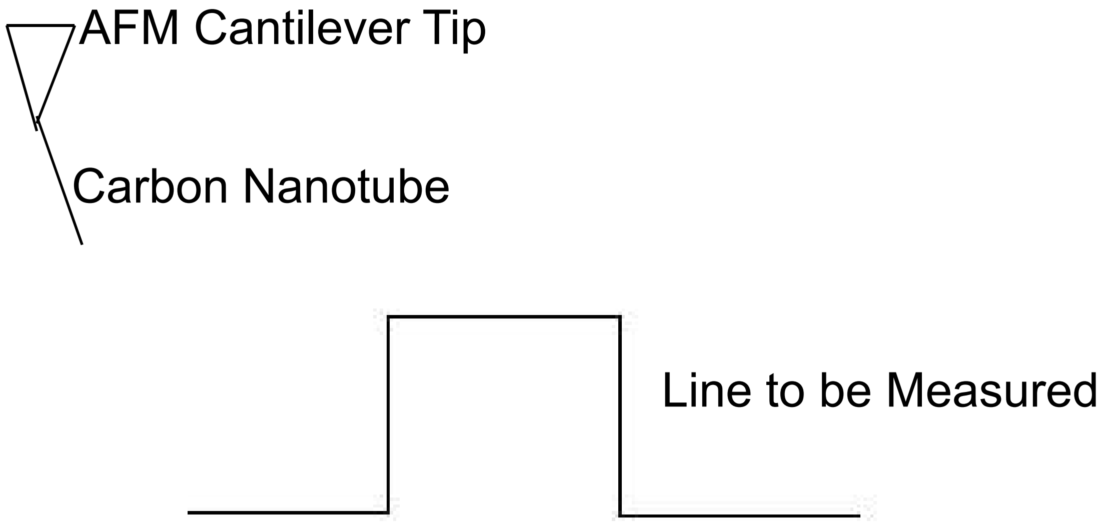
Image Stitching Linewidth Measurement:

Goal

An independently traceable probe-based method for measurement of linewidth



AFM Image Stitching for Linewidth Measurement



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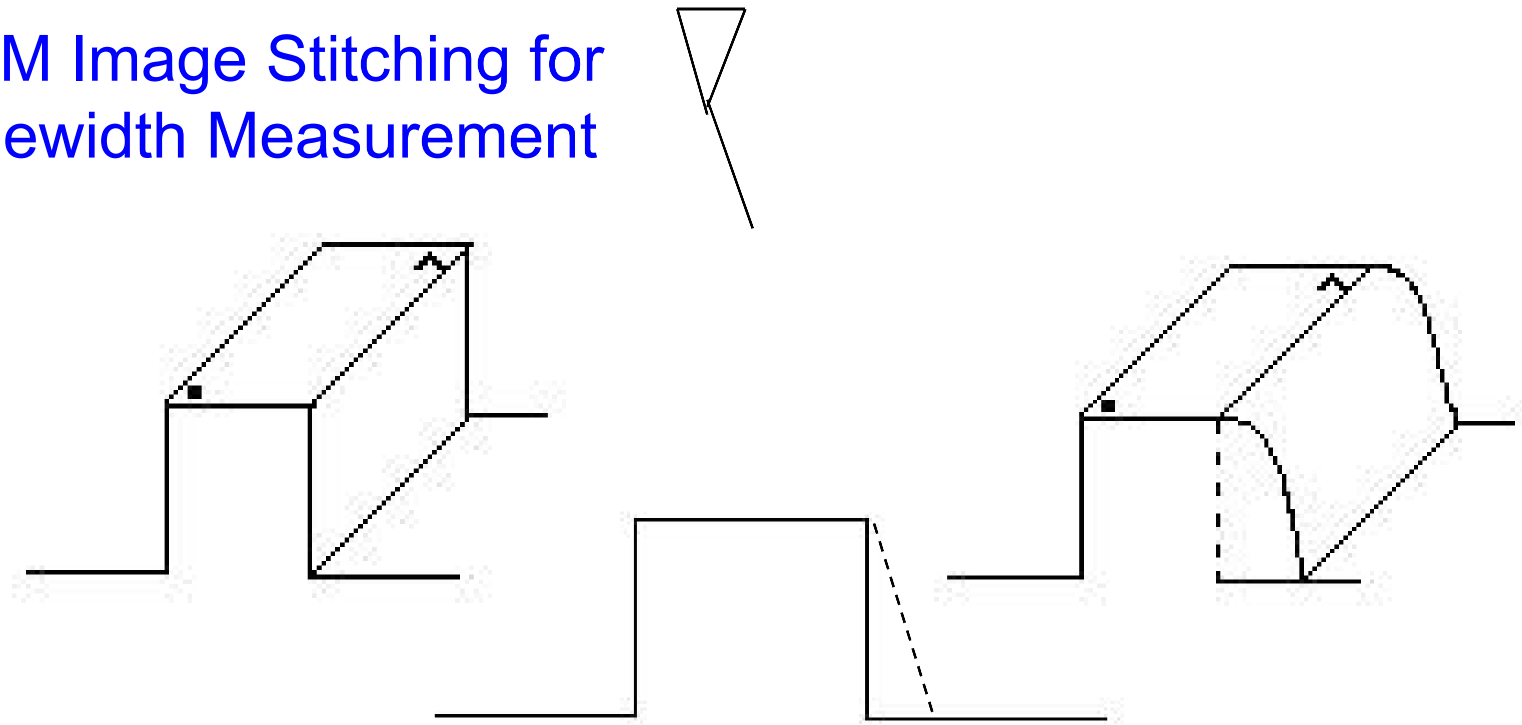

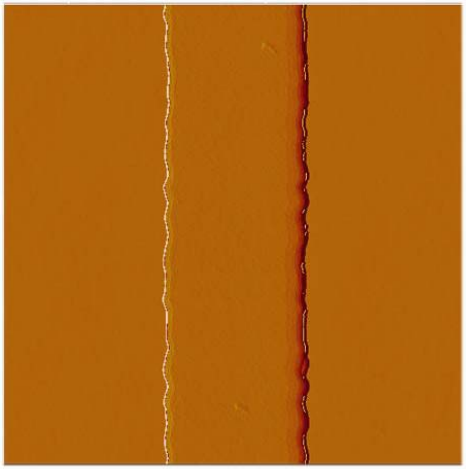

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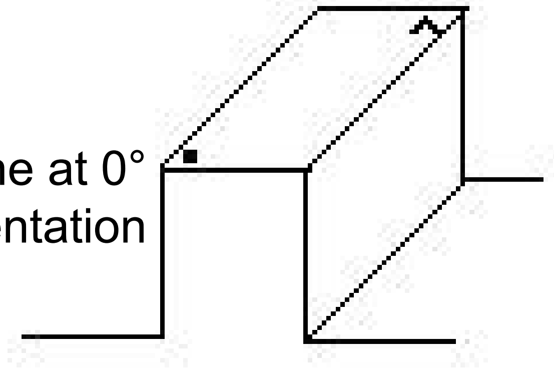
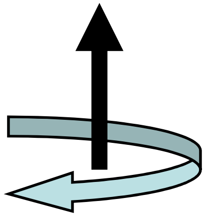
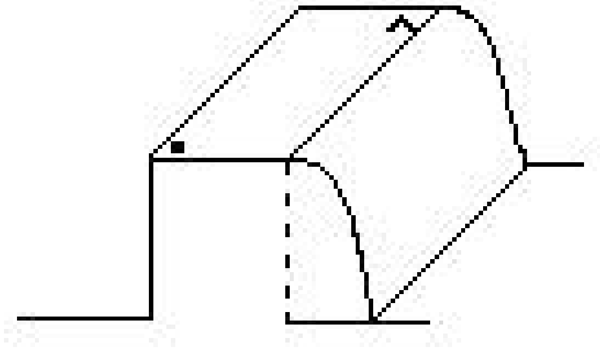
AFM Image Stitching for Linewidth Measurement



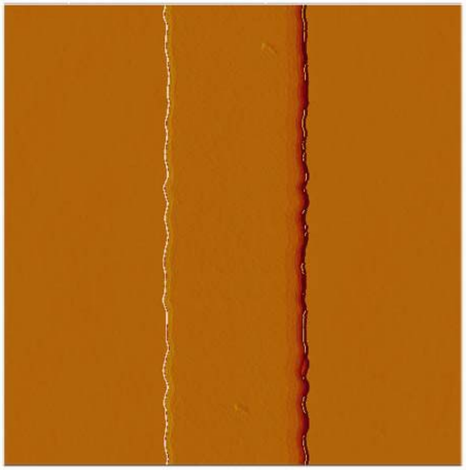
Line at 0° Orientation



Rotate Surface by 180°

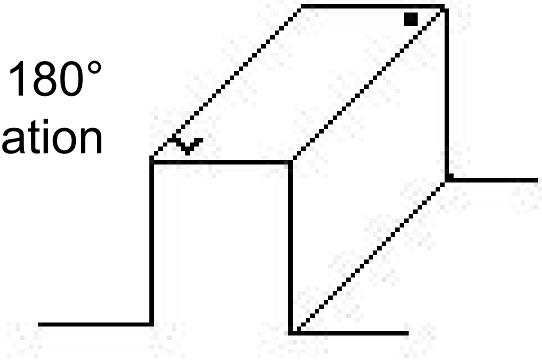


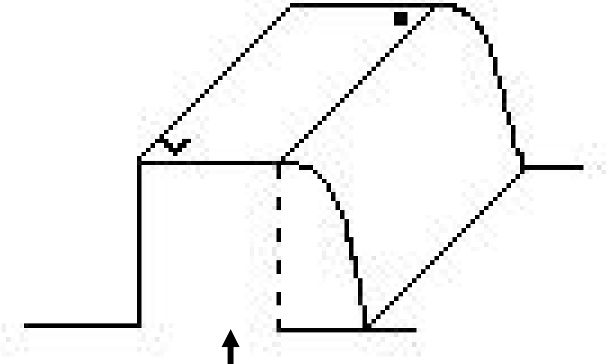
AFM Topography Image at 0° Orientation



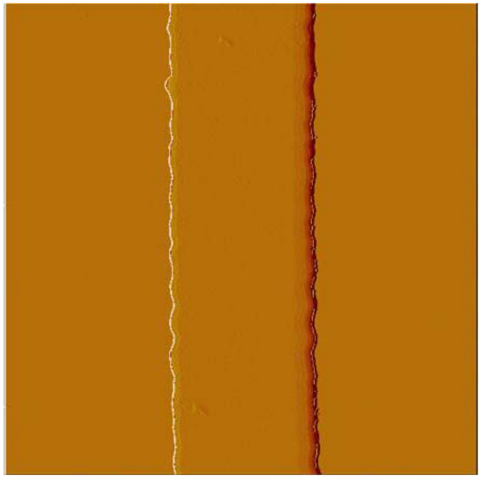
Generate Second Topographic Image of Rotated Line

Line at 180°
Orientation

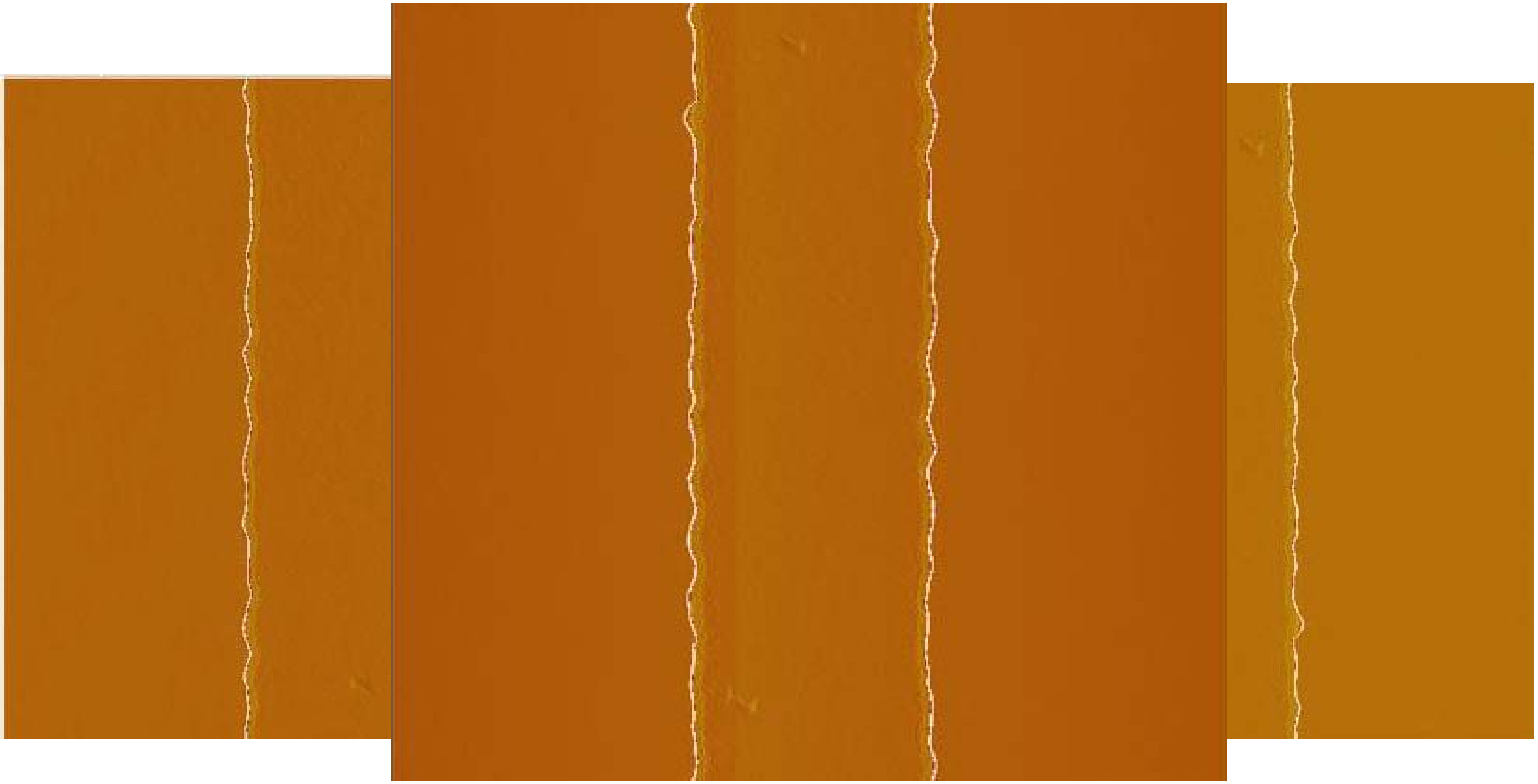




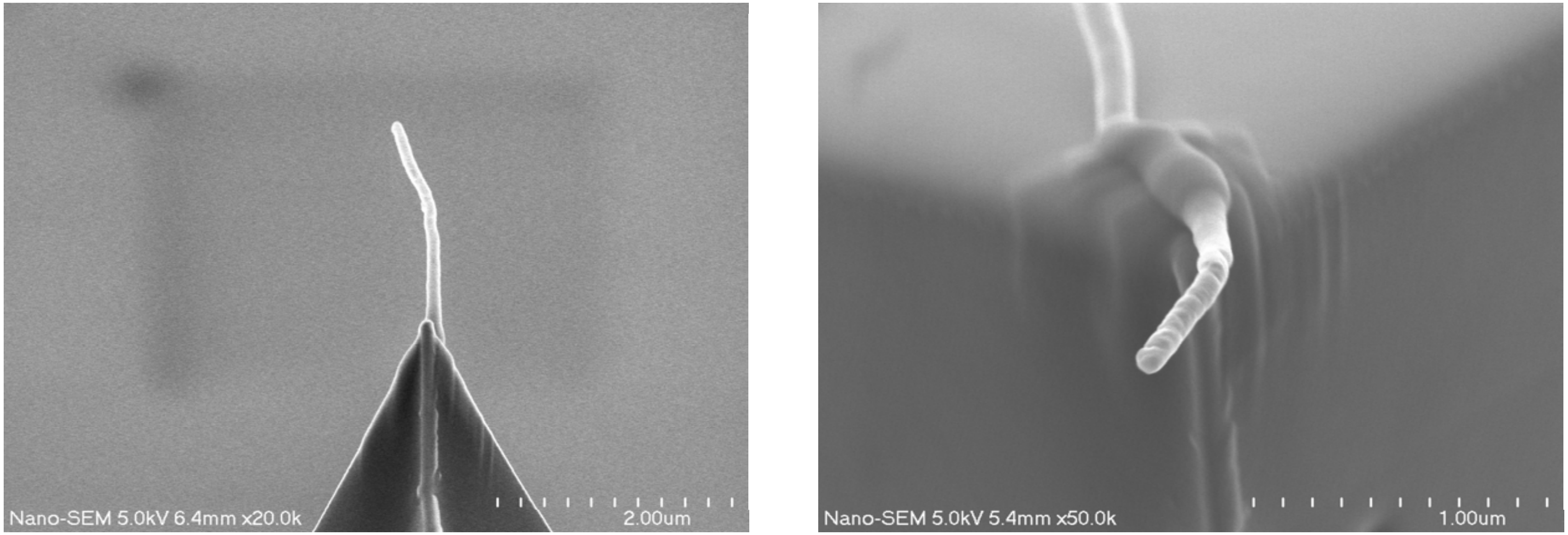
AFM Topography Image
at 180° Orientation



Form Composite Image from the Undistorted Data



SEM micrographs of multi-walled carbon nanotube tip used to collect images #1 and #2

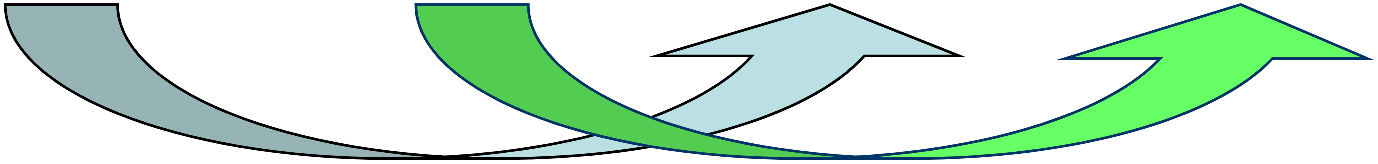


Side view

Top view

Linewidth Measurement Results (after nanotube radius correction)

Nanoscope AFM Image Stitching				CD-AFM			
Sample #1		Sample #2		Sample #1 (using X3D)		Sample #2 (using SXM)	
Top	Bottom	Top	Bottom	Top	Bottom	Top	Bottom
536.6 nm	579.9 nm	574.9 nm	593.2 nm	557.1 nm	539.5 nm	577.5 nm	554.8 nm



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)

Contents:

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

- Length Measurement in the NNI

- 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**

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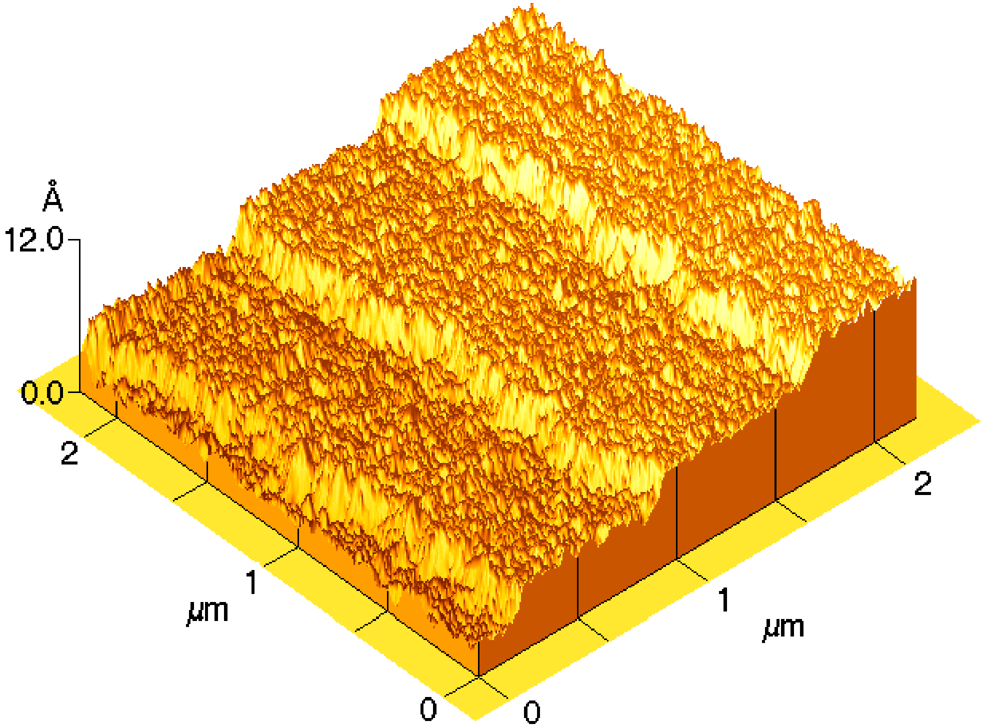
AFM image of silicon (111) atomic step specimen with native oxide



See N.G. Orji et al. *Wear* **257**, 1264 (2004).

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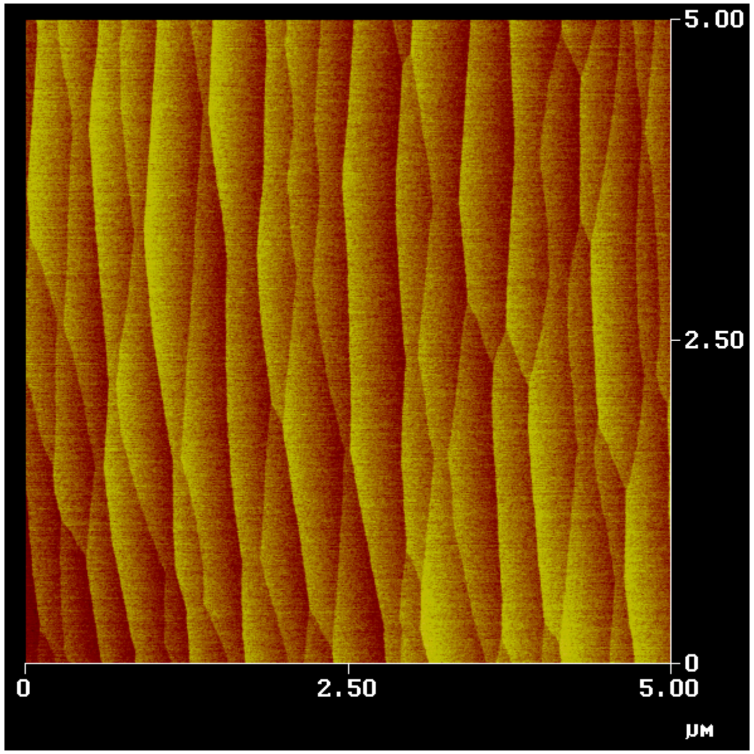
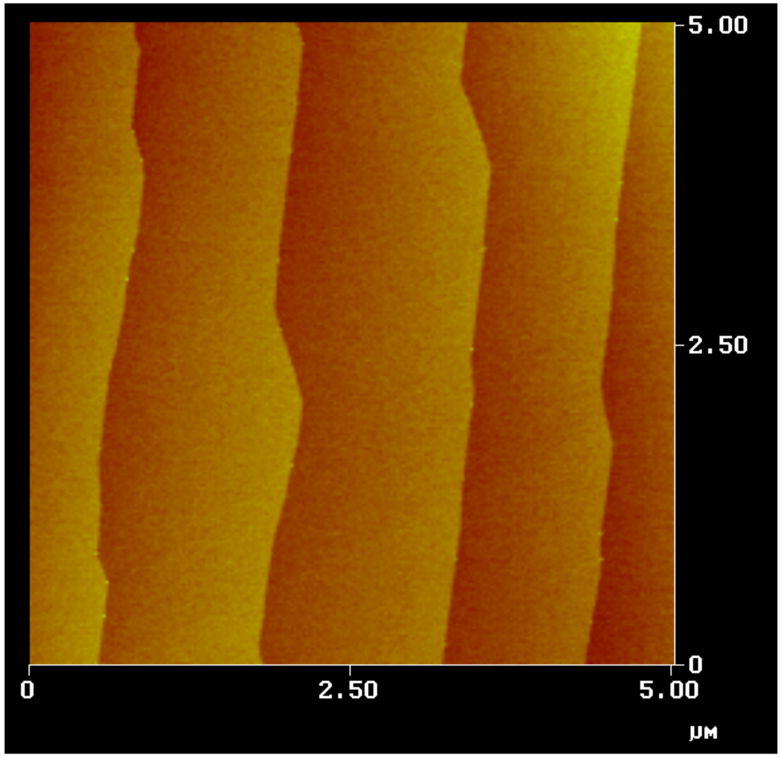
Relation between miscut angle and average width of terraces



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.

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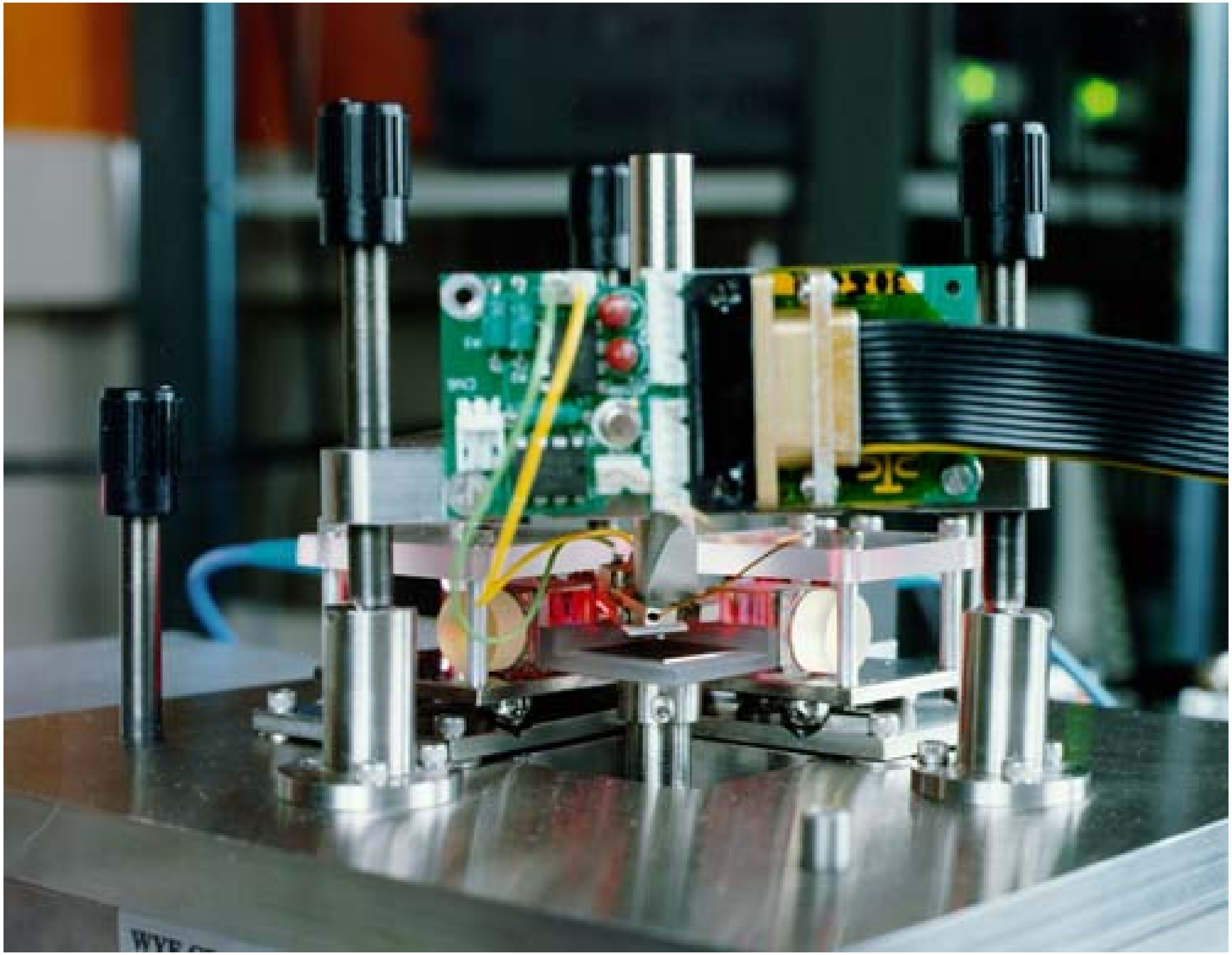
Calibrated Atomic Force Microscope with AFM Sensor on Kinematic Mount



Comparison of Silicon Step Height Results

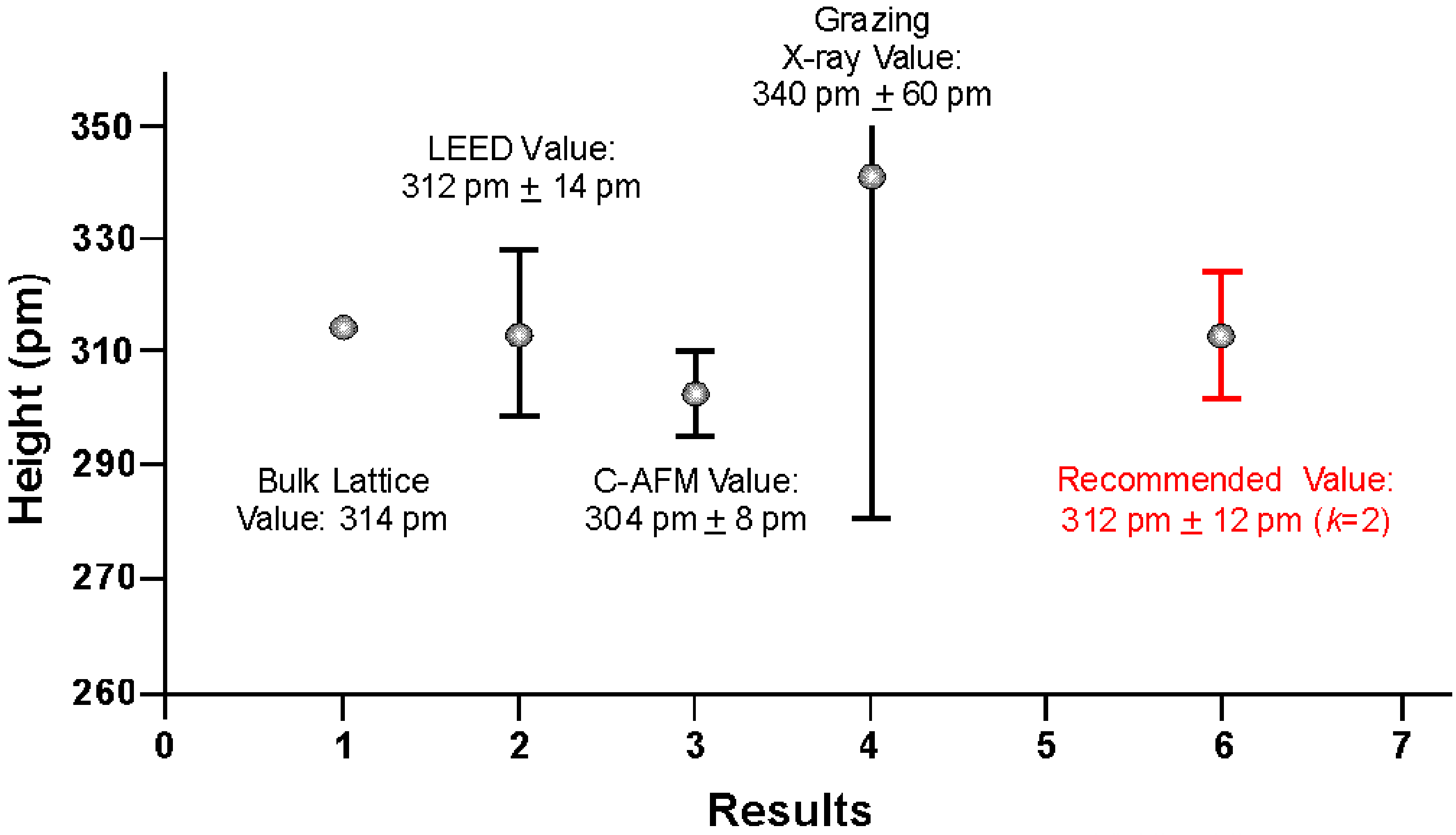


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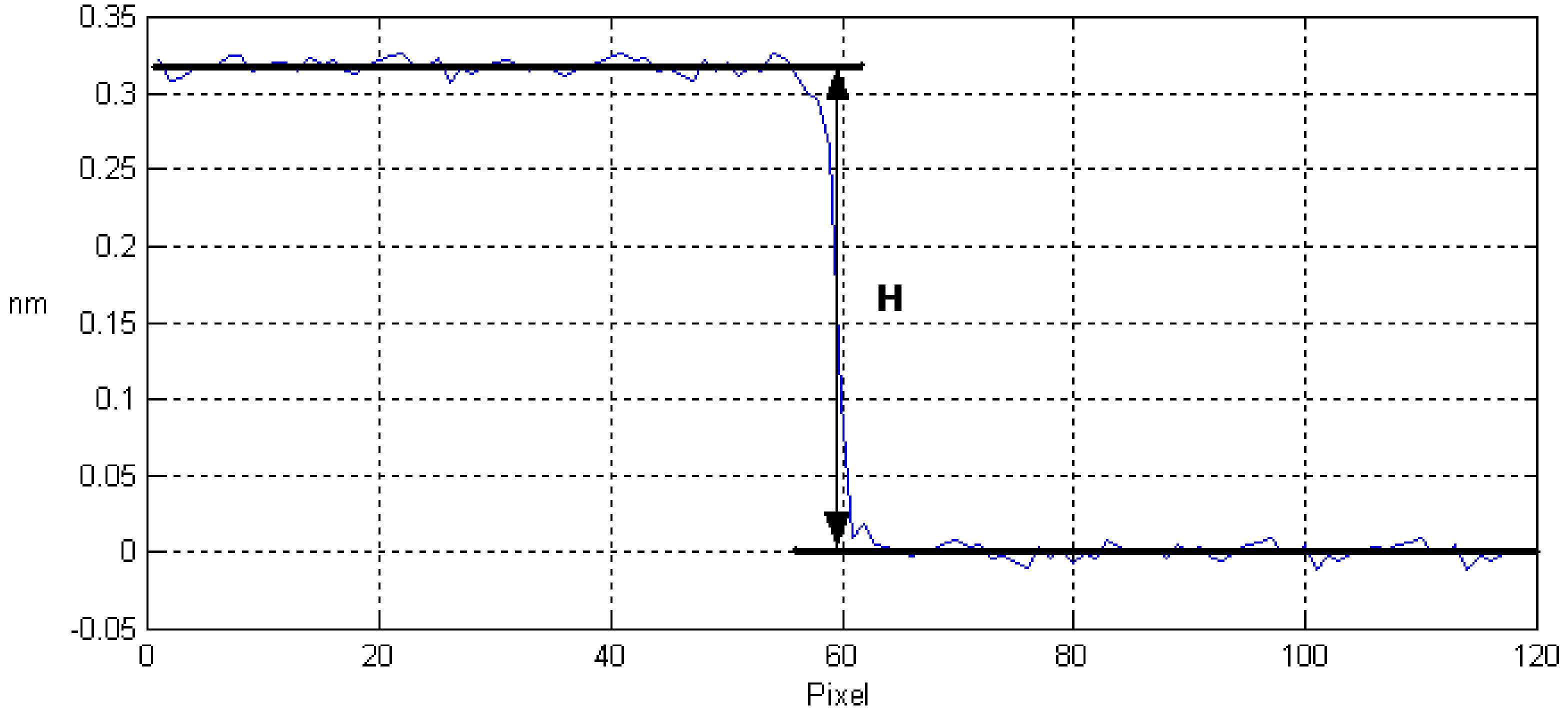
Illustration of Step Height Calculation for a Single Profile



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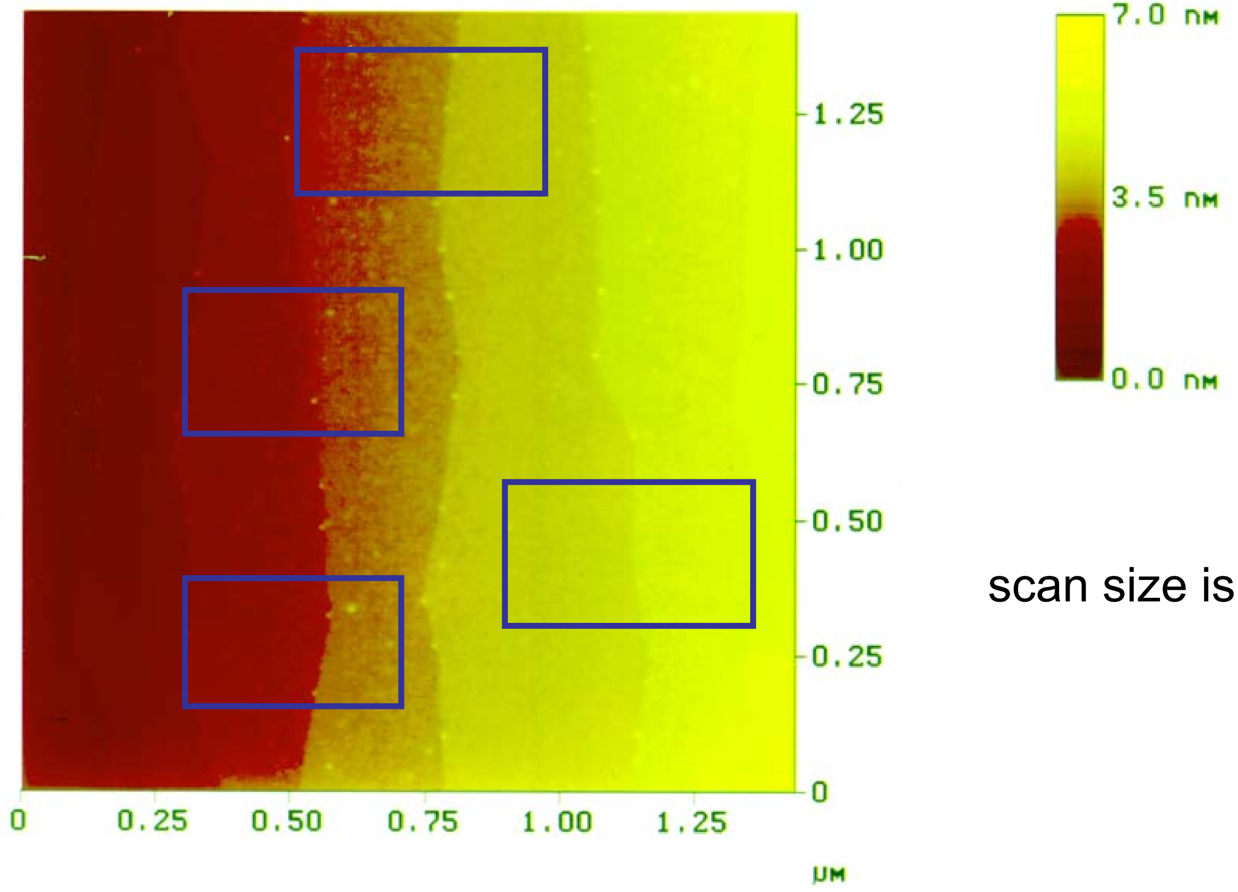
Typical Area Selection for Step Height Analysis on Si (111)

What is the expected variability for a user?



scan size is 1.44 µm

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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

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

Reference: R. Dixson et al. *SPIE Proc.* **4344**, 157 (2001)

Designation: E 2530 – 06

Developed by ASTM SC E42.14, STM/AFM

Standard Practice for Calibrating the *Z*-Magnification of an Atomic Force Microscope at Subnanometer Displacement Levels Using Si (111) Monatomic Steps[1]

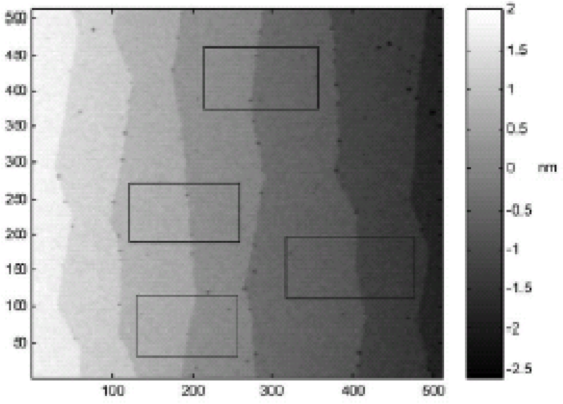

FIG. 2 Illustration of Selected Areas

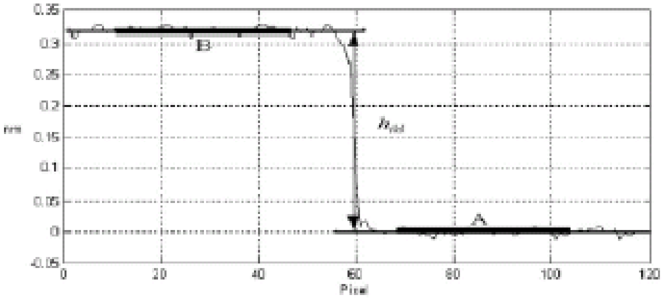

FIG. 3 Illustration of the Step-Height Algorithm Used

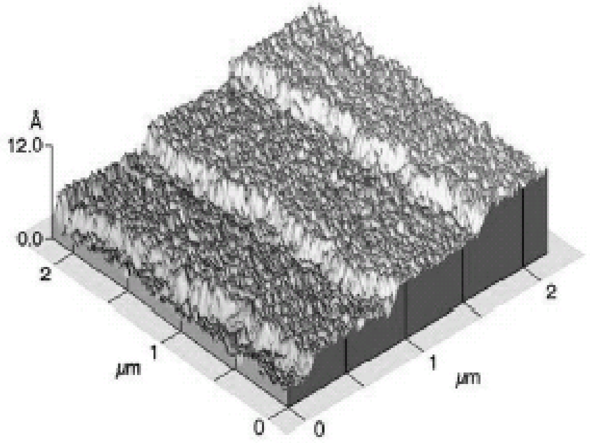

FIG. 1 Image of a Silicon Monatomic Stepped Surface

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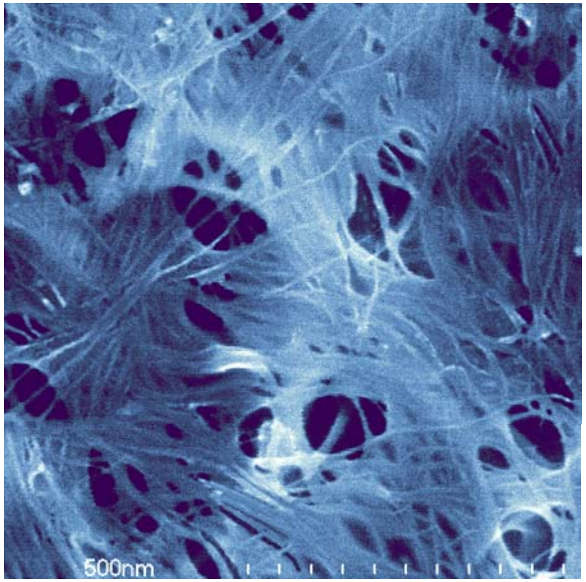
Observations

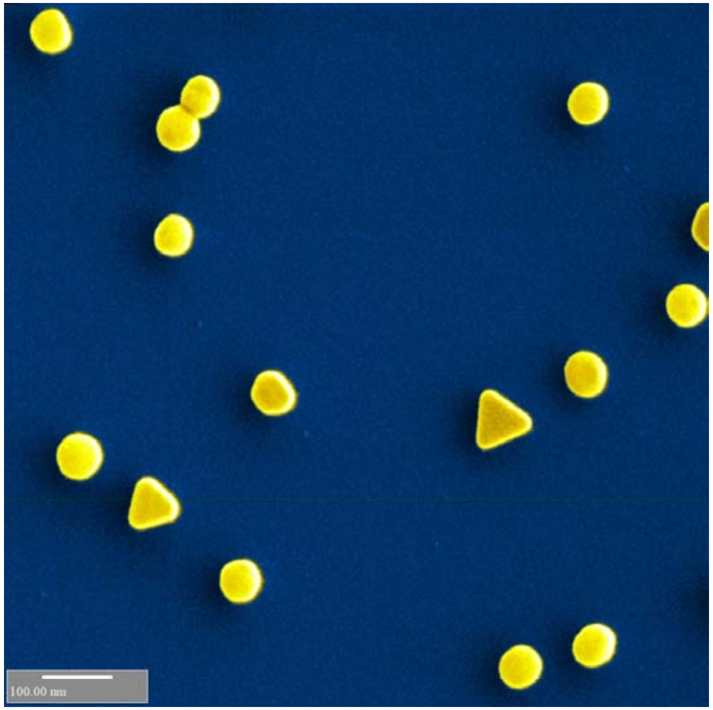
- The structure of NNI provides for strong *coordination* among agencies with different missions
- Nanoscale length metrology plays an important role in the semiconductor industry and in particle based nano-technology and will be important in the manufacture of many other nano-scale components and functional 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

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