Metrology, Nanocharacterization, and Instrumentation for Emerging Nanotechnology and Nanoelectronics

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Agenda

- **Electrical and Optical Characterization (Seiler)**
  - Backdrop: Metrology and Characterization at the Molecular Level
  - NIST Roles
  - Workhorse Characterization Tools and Issues
  - Scanning Probes
  - Electrical Characterization for Molecular Electronics and Gate Dielectrics
  - Optical Techniques and Applications
  - Nanopore Metrology
  - Spintronics
  - Instrumentation and Metrology
- **Particle Beam and Scanned Probe Instrumentation (Postek)**
  - Introduction
  - Nanometrology Challenges
  - Optical Microscopy
  - Scanning Electron Microscopy
  - Atomic Force Microscopy
  - Focused Ion Beam/Dual Beam Microscopy
  - Environmental Scanning Electron Microscopy
  - Transmission Electron Microscopy
  - Helium Ion Microscopy
  - Conclusion

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–
Electrical and Optical Characterization

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Washington D.C.
December 2007

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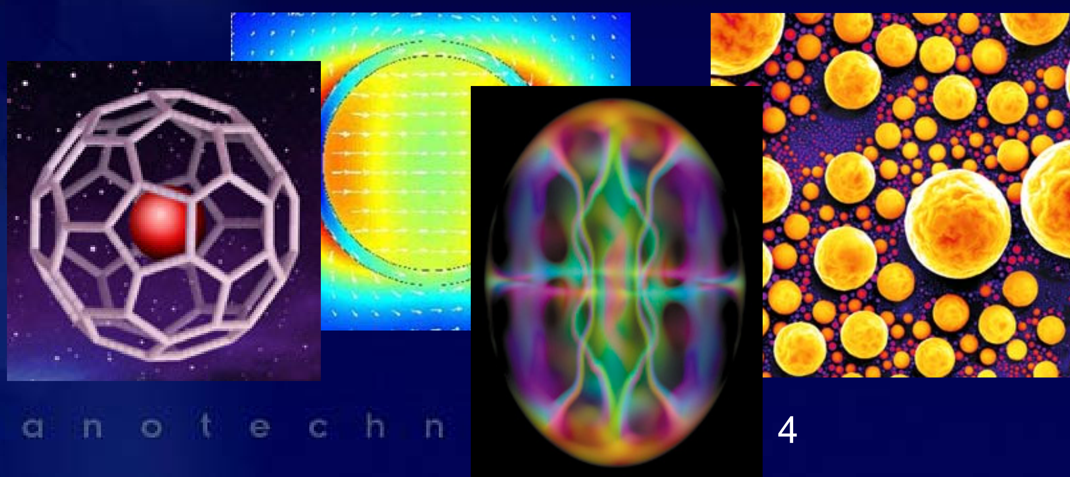
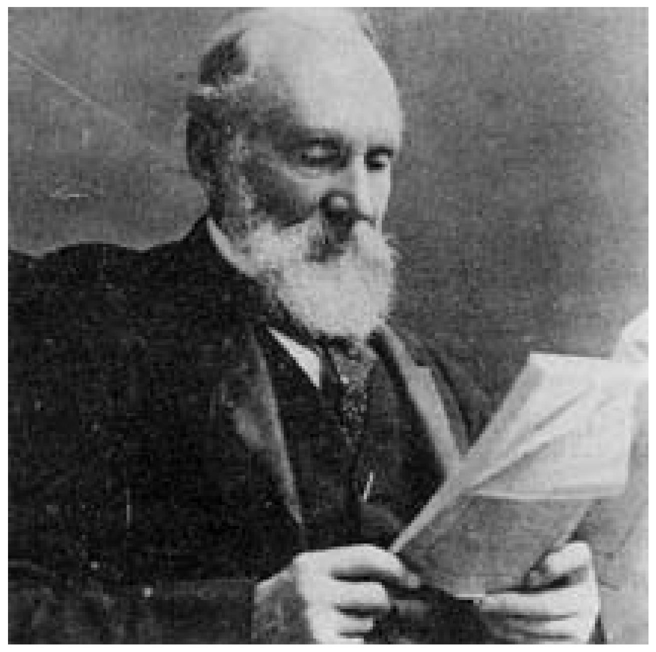
Science and Engineering at the Atomic and Molecular Level

- Nanoscale Science and Technology Involves the Atomic and Molecular Level
  - fundamental understanding of phenomena and materials at the nanoscale
  - creation and use of structures, devices, and systems that have novel properties and functions because of their small size
  - control of matter and processes at the atomic and molecular level
  - Integration of nanoscale materials and structures into larger materials components, systems, and architectures



Metrology

The science of measurement; a system of measures



"When you can measure what you are speaking about, you know something about it. But when you cannot measure it, your knowledge is of a meager and unsatisfactory kind. It may be the beginning of knowledge, but you have scarcely advanced to the stage of science."

William Thomson, Lord Kelvin

- The semiconductor industry most often refers to measurements used in process control as *metrology*.
- Engineers often use the word to describe procedures, such as critical dimension measurements, which routinely monitor lithography processes inside the cleanroom.
- Others generalize it to all in-line measurements.

Semiconductor Characterization

- An integral part of research, process development, and manufacturing
- Best described as a wide range of interdisciplinary activities that determine the structure, composition, properties, and performance of materials and devices, and the relationships among them*

Thus, characterization measurements address quality assurance of incoming materials, wafer screening methods, control and monitoring of equipment and manufacturing processes, diagnostic and failure analysis, and end device production in light of intended design and function.

** Office of Science and Technology Policy, Advanced Materials and Processing: Fiscal Year 1993 Program*

NIST Core Roles

- measurements and standards
  - methods
  - techniques
  - instruments and tools
  - reference materials
  - traceability

- critically evaluated data
  - foundation for models and simulations
    - predict properties and function of materials
    - scaleable models
  - tools for validation

- *basis for nanoscience to move forward*
  - *assess quality of results*
  - *predict novel outcomes*

Path of Impact for Workhorse Tools

Grand Challenges	Metrology Drivers	Workhorse Analytical Tools
Affordable Scaling	Small Geometries Film Thickness Surfaces and Interfaces	**OM**, CD-SEM, HRTEM, AFM **SE**, **FTIR**, **PR**, **PA**, SEM, HRTEM, SEM AES, XPS, ToF-SIMS, AFM, HRTEM, FIB
New Materials and Structures	Composition & Bandgap Dopants Crystallinity	**FTIR**, **PL**, **PR**, EMP, RBS, HIBS, XRD, AES **FTIR**, **PL**, SIMS, ToF-SIMS, NAA, SSMS **SE**, **Raman**, XRD, XRT, XRRC, FIB
Yield and Reliability	Contamination Impurities Physical Defects	**FTIR**, **PL**, AES, ToF-SIMS, EMP, XPS, TXRF **FTIR**, **PL**, SIMS, ToF-SIMS, NAA, SSMS **OM**,**SCT**,**PL**,SEM,HRTEM,XRT,AFM,FIB
GHz Frequency Operation	< Pico-second Analysis	Laser-based probes, high speed electrical
Faster Time-to-Market	Integrated Metrology	In line/in situ: **OM**, **OES**, **SE**, **PA**, XRF

* Optical techniques are shown in bold type

Path of Impact for Workhorse Tools - Acronyms

AES	Auger Electron Spectroscopy
AFM	Atomic Force Microscopy
CD-SEM	Critical Dimension SEM
DCT	Double Crystal X-ray Topography
FIB	Focused Ion Beam (sectioning)
EMP	Electron Microprobe
FTIR	Fourier Transform Infrared Spectroscopy
HIBS	Heavy Ion Backscattering Spectrometry
HRTEM	High Resolution TEM
NAA	Neutron Activation Analysis
OES	Optical Emission Spectroscopy
OM	Optical Microscopy
PA	Photo-Acoustic
PL	Photoluminescence
PR	Photoreflectance
Raman	Raman Spectroscopy
RBS	Rutherford Backscattering Spectrometry
SCT	Scatterometry
SE	Spectroscopic Ellipsometry
SEM	Scanning Electron Microscopy
SIMS	Secondary Ion Mass Spectrometry
SSMS	Spark Source Mass Spectrometry
TEM	Transmission Electron Microscopy
ToF-SIMS	Time-of-Flight SIMS
TXRF	Total Reflection X-ray Fluorescence
XPS	X-ray Photoelectron Spectroscopy
XRD	X-ray Diffraction
XRF	X-ray Fluorescence
XRRC	X-ray Rocking Curves
XRT	X-ray Topography

Variety of Tools Needed for Metrology

Example: Physical Characterization Tools for Thin Dielectrics

- C. A. Richter et al., Proc., 1998 International Conference on Characterization and Metrology for ULSI Technology, pp. 185-189 (01-JUL-1998)
- C. A. Richter et al., Characterization and Metrology for ULSI Technology: 2000, pp. 134-139 (01-FEB-2001)
- J. R. Ehrstein et al., Characterization and Metrology for ULSI Technology: 2003, pp. 331-336 (30-SEP-2003)

Thickness:

Ellipsometry
TEM
Electrical (CV/IV)
Neutron Reflectometry
X-ray Reflectometry
MEIS

Roughness:

AFM/STM
X-ray Scattering
Weak-Localization
TEM
SHG
Light Scattering

Structure/Composition:

XPS
Auger
SIMS
FTIR
MEIS
TEM
Ellipsometry

Strain:

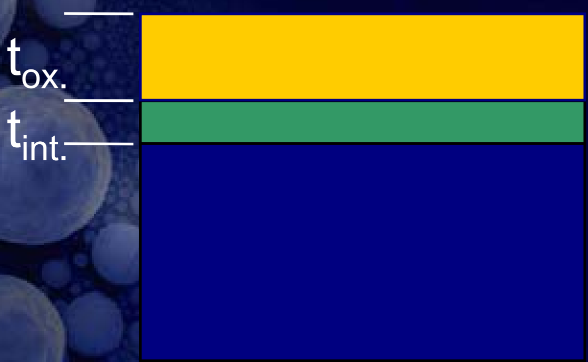
X-ray Diffraction
Raman
Ellipsometry
SHG

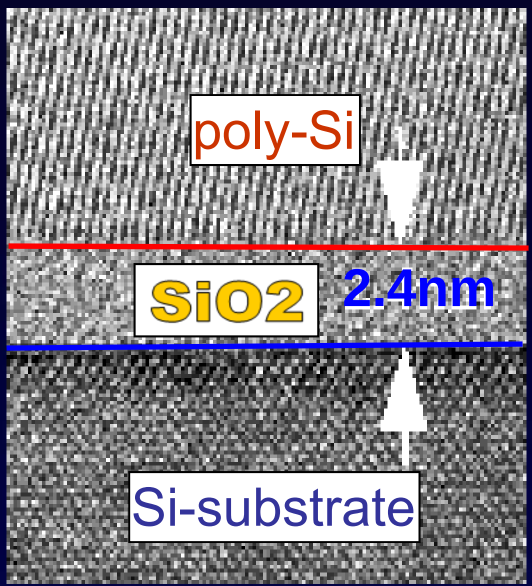
Variety of Tools Needed for Metrology (continued)

Which set of characterization tools is optimal to provide the necessary understanding and form the basis to address industry's needs?

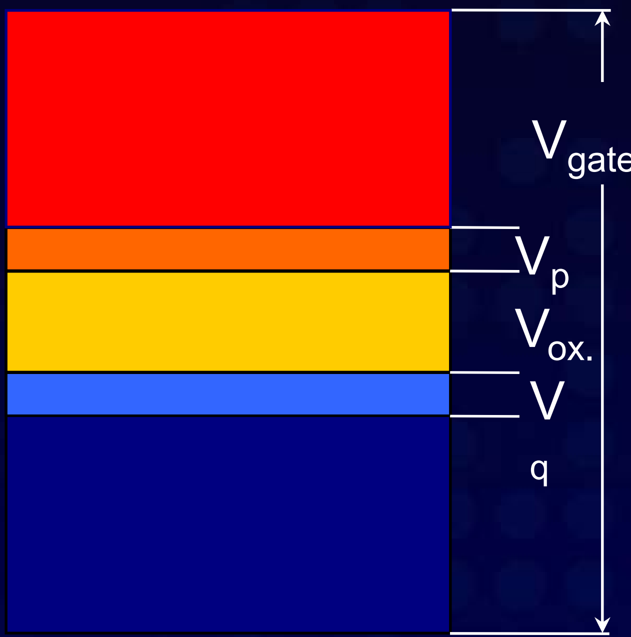
- Answer is Application and Cost Dependent:
  - Diagnostics/Failure Analysis
  - Process Development
  - Improve Optical & Electrical Models
  - Improve Basic Understanding
- Assertations:
  - Individual methods may not be sufficient
  - More cross comparisons needed
- NIST Role:
  - To understand differences and harmonize results

Differing Views of Gate Dielectrics





B. Weir, et al, IEDM 1997, p.73



Optical ⟷ Physical ⟷ Electrical

(Process Monitoring)

(Device Performance/ Reliability)

Ellipsometry

Physical Characterization

E-test

Metrology and Characterization Resources

- Hard-bound proceedings from semiconductor characterization and metrology conferences published by AIP
  - Ordering information: http://proceedings.aip.org/proceedings/
- High-level Metrology Presentations available on-line from 2000, 2003, 2005, and 2007 meetings: www.eeel.nist.gov/812/conference/
  - Approximately 140,000 hits from April 2007 – August 2007
- 2009 Meeting to be held May 11-15, 2009, at the College of Nanoscale Science and Engineering, University at Albany, Albany, NY

Other Resources

A.C. Diebold, ed., Handbook of Silicon Semiconductor Metrology, Published by Marcel Dekker, Inc. (2001)

S. Perkowtiz and D. G. Seiler, Optical Characterization in Microelectronics Manufacturing, Journal of Research of the National Institute of Standards and Technology, Vol. 99, No. 5, pp. 605-639 (1994)

D. G. Seiler, S. Mayo, and J. R. Lowney, Hg-1.xCdxTe Characterization Measurements: Current Practice and Future Needs," Semiconductor Science Technology, Vol. 8, pp. 753-776 (1993)

T. J. Shaffner, Semiconductor Characterization and Analytical Technology, Proceedings of the IEEE, Vol. 88, No. 9, pp. 1416-1437 (2000)

Critical Interplay Between Modeling and Characterization

Two categories of modeling/characterization interactions:

1. Model necessary to interpret a physical or electrical measurement.
2. Physical or electrical characterization necessary to confirm a model of a novel material, device structure, or process.

Characterization and modeling/simulation synergy is necessary for success.

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Correlating Physical Characterization and Test Structure Results Critical

Correlation of final device performance with underlying physical properties.

Structure ⟷ Function

Fundamental understanding of carrier transport in the various nanostructures.

- Assessment of the viability of new technologies
- Enables the design of improved devices
- Understand failure and reliability
- Needed to optimize process monitoring metrology

Interdisciplinary team needed to achieve optimal results !

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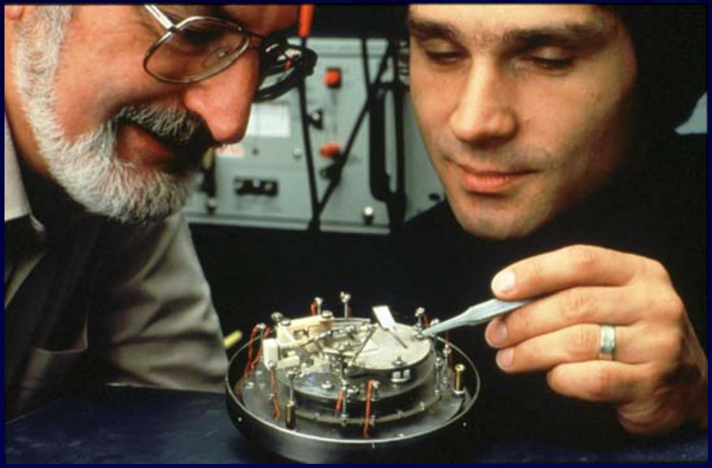
Scanning Probe Microscopy

- One key enabling invention creating technological opportunity in nano S&T is the development in the 1980s (with continuing improvements through the present) of scanning probe microscopy:
  - Ask a dozen surface scientists to identify key developments in instrumentation that are responsible for catapulting nanotechnology to the front lines of physical science research. Nearly all of them will point to the advent of the scanning probe microscopy. The scanning tunneling microscope was the first instrument to enable scientists to obtain atomic-scale images and ultimately to manipulate individual atoms on the surface of materials.



Nobel laureates Heinrich Rohrer (left) and Gerd Binnig (right) of IBM's Zurich Research Laboratory, shown here in 1981 with a first-generation scanning tunneling microscope (STM).

Continued Advances in AFM Technology (FIRAT Probe), 2006

- “Georgia Tech researchers have created a highly sensitive atomic force microscopy (AFM) technology capable of high-speed imaging 100 times faster than current AFM. This technology could prove invaluable for many types of nano-research, in particular for scanning integrated circuits for mechanical and material defects ... even translating into movies of molecular interactions in real time.”

- “FIRAT (Force sensing Integrated Readout and Active Tip) can capture other measurements never before possible with AFM, including material property imaging ... It could also speed up semiconductor metrology and even enable fabrication of smaller devices.”
- “FIRAT solves two of AFM's chief disadvantages as a tool for examining nanostructures: AFM doesn't record movies and it can't reveal information on the physical characteristics of a surface...”

- from “Faster Technology Lets Atomic Force Microscope Capture Nano Movies",” KurzeweilAI.net, Feb. 10, 2006

Scanning Probe Microscopy (SPM)

Joseph Kopanski (joseph.kopanski@nist.gov)

Goals/Principle/Method

- Techniques such as scanning tunneling microscopy, atomic force microscopy, and their many variants.
- Non-destructive technique for the measurement of surface features with spatial resolution to the nm scale.
- A sub-set of techniques can achieve atomic resolution.

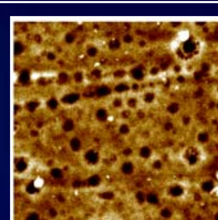
Scanning Probe Microscopy: Electrical and Electromechanical Phenomena at the Nanoscale, S. Kalinin and A, Gruverman, Eds., (Springer, NY 2007).

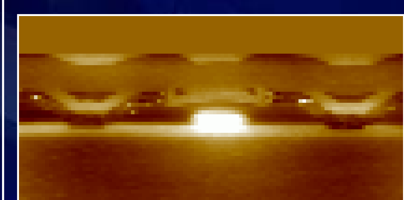
Experimental/Equipment

- Commercial systems with a range of capabilities from table top AFMs for surface roughness, to UHV STMs with atomic resolution, to in-line tools.
- NIST has specialized SPMs sensitive to capacitance, resistivity, and work function.
- SPM based high-frequency (>1 GHz) electrical measurements and near field optical probes an emerging research interest.

Data/Results

- Surface roughness, high temperature annealed SiC.





- Cross-sectioned MOSFET showing 1-µm gate and contact metallizations.

Impact/Importance

- One of the most important techniques to visualize and characterize nanostructures.
- A wide range of applications for integrated circuit & nanoelectronic characterization: surface roughness, step height, line width, contamination and defect characterization, and failure analysis.
- Electrical measurements within the active regions of FINFETs, nanowires, and other nano structures.

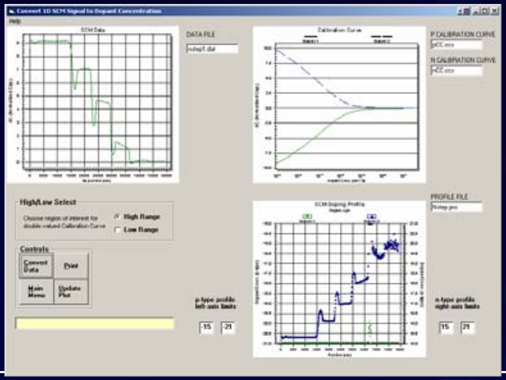
Scanning Capacitance Microscopy (SCM)

Joseph Kopanski (joseph.kopanski@nist.gov)

Goals/Principle/Method

- Combines a 1 GHz differential capacitance measurement with an atomic force microscope.
- Capacitance images can be interpreted as two-dimensional dopant profiles with spatial resolution approaching a few nanometers.

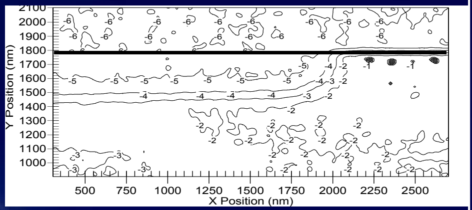
Experimental/Equipment



NIST ***FASTC2D*** software for dopant profile extraction from SCM images.

Data/Results

Two-dimensional dopant profile contours of shallow boron implant.



J. J. Kopanski, J. F. Marchiando et al, J. Vac. Sci. Technol B **22**, 399 (2004).

Impact/Importance

- Quantitative measurement of two-dimensional dopant profiles.
- Qualitative images of junction shape and proper type of implanted dopants for failure analysis.

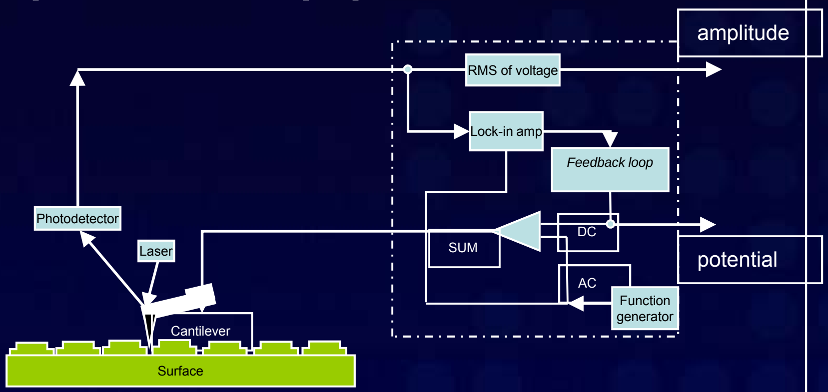
Scanning Kelvin Force Microscopy (SKFM)

Joseph Kopanski (joseph.kopanski@nist.gov)

Goals/Principle/Method

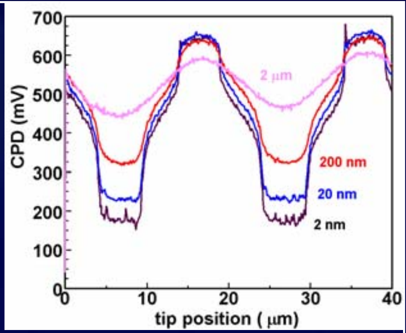
- Measurement of electrostatic force, surface potential, contact potential difference, work function.
- Basic SKFM is a “simple” modification of atomic force microscopy, but high spatial resolution requires more sophisticated measurement techniques.
- Work function measurements of candidate gate metals for high-k stacks, interface and potential measurements of nanostructures.

Experimental/Equipment



Data/Results

Tip-to-sample contact potential difference of alternating Al and Au lines.



J. J. Kopanski et al, *2007 International Conference on Frontiers of Characterization and Metrology for Nano-electronics*, AIP, Gaithersburg, MD (2007).

Impact/Importance

- Quick work function measurements that can be correlated to work function from flatband voltage versus oxide thickness measurements and other techniques.
- Surface potential measurements within the active regions of nanoscale devices.

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Molecular Electronic Test Structures

Curt Richter (curt.richter@nist.gov)

Goal

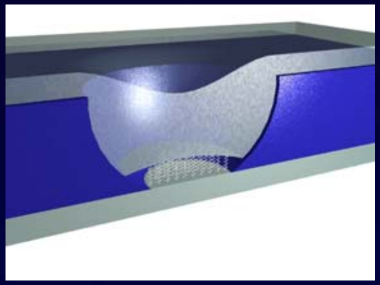
- Reliable, Reproducible Electrical Measurements of molecules.

Approach

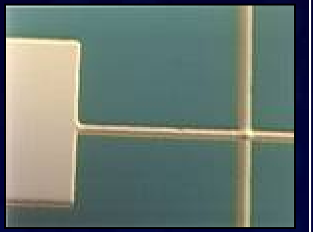
- Design and fabricate device prototypes that enable robust electrical measurements of electrically active molecules.

Example Test Structures

nanopore

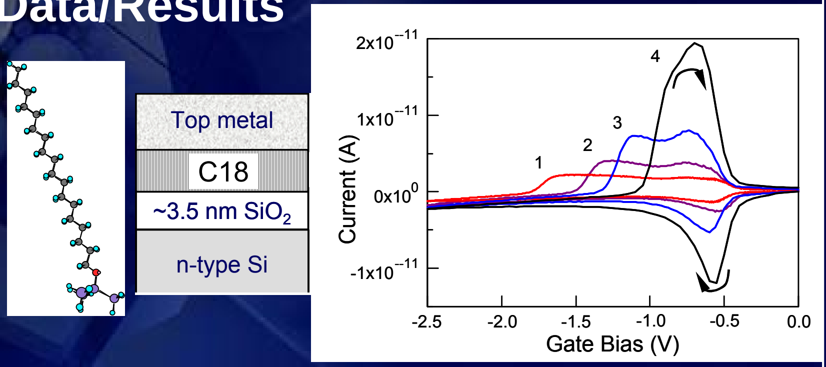


crossbar



Wang et al., Appl. Phys. Lett. 89, 153105 (2006)

Data/Results



Richter, et al. Solid State Electronics, 2006

Impact/Importance

- Assess this promising new technology
- Screening of candidate molecular electronic molecules
- Determine conduction mechanisms
- Assess failure and reliability

C.A. Richter, et al, Applied Phys. A 2005

NIST nanotechnology

Advanced Electrical Characterization Methods for Molecular Electronics

Curt Richter (curt.richter@nist.gov)

Goals

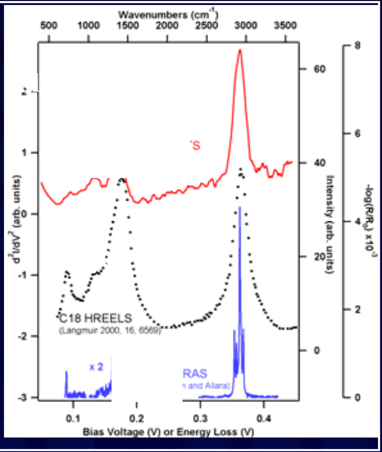
- *In situ* characterization of molecules within molecular electronic devices.

Approach

- Utilize advanced electrical measurement and analysis techniques to determine fundamental electronic properties of molecular junctions

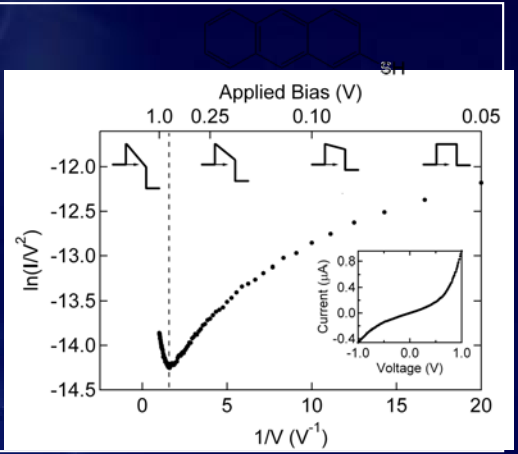
Inelastic Electron Tunneling Spectroscopy

W. Wang et al., *NanoLetters,* 2004 (4) 643.
J. Kushmerick et al. *NanoLetters* 2004 (4) 639.



Transition Spectroscopy

JM Beebe, et al. Phys. Rev. Lett. 2006.



Impact/Importance

- Provide unambiguous proof that molecules are in current path.
- Spectroscopically probes the molecules within the junction.
- Map the pathways of electrons through molecules.
- Extract fundamental electronic barrier heights.

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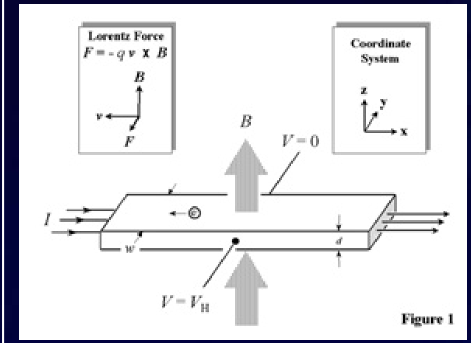
Hall Effect

W. Robert Thurber (w.thurber@nist.gov)

Principle/Goals

- Principle
  - Lorentz force on moving charge carrier produces transverse voltage
- Goals
  - Key material characterization technique
    - Carrier density and type
    - Carrier mobility
    - Activation energy
  - Magnetic field sensors

Experimental/Equipment



Results

- Photo Hall effect uses light instead of temperature to determine activation energy, density, and mobility of carriers
- Multicarrier analysis, e.g., gives carrier densities and mobilities in channel for HEMT structures
- Hall sensors fabricated with 100 nm dimensions for scanning Hall probe microscopy (Candini et al., Nanotechnology, **2006**, 17, 2105)

Impact/Importance

- Characterization tool for semiconductor materials including 2D multilayered structures and inversion/accumulation layers
- Applications such as measurement of magnetic field, contactless switching, position sensing, and plasma thrusters
- Popular Website describing technique and calculations (receives over 100,000 hits per year) (www.eeel.nist.gov/812/hall.html)

Defect Characterization of nanostructures and CMOS with high k FET devices using noise and RTS analysis

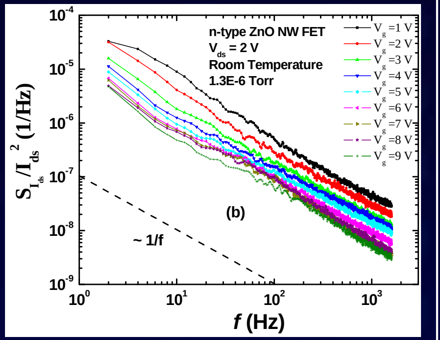
John Suehle (john.suehle@nist.gov)

Goals/Principle/Method

- Noise and Random Telegraph Signals (RTS) can be used as probes to characterize electrical defects in nano-scale electronic devices.
- The bias-voltage/temperature dependence of the capture and emission times allow one to determine the type, location, and barrier energy of the defects.

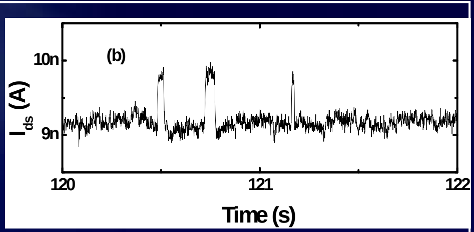
Experimental/Equipment

- Nano-FET drain current is conditioned with a trans-impedance amplifier and characterized with a dynamic signal analyzer and digital oscilloscope.



Data/Results

- Three level RTS observed in ZnO nano-wire FETs indicate two discrete trap states.
- Spatial distribution of defects (H. Xiong et al., Microelectronic Engineering, 24 (2007), 2230



H. Xiong, et al, Appl. Phy. Lett, 91, 2007, 53107

Impact/Importance

- Single electron traps can produce large fluctuations in nano-scale devices.
- Sensitivity to single active trap state.
- Trap states can be profiled spatially and energetically.

Ultra-fast electrical measurements for transient trapping characteristics in emerging technologies

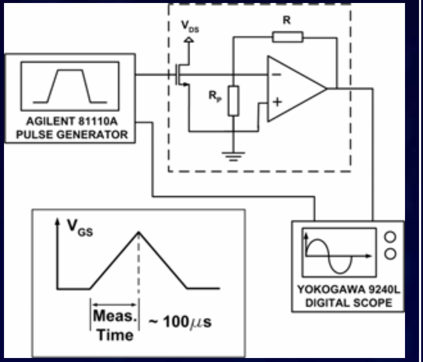
John Suehle (john.suehle@nist.gov)

Goals/Principle/Method

- Charge trapping due to voltage stress can be severely underestimated using standard DC measurements due to relaxation effects.
- Ultra-fast electrical characterization techniques are required to avoid recovery.
- Accurate reliability projections

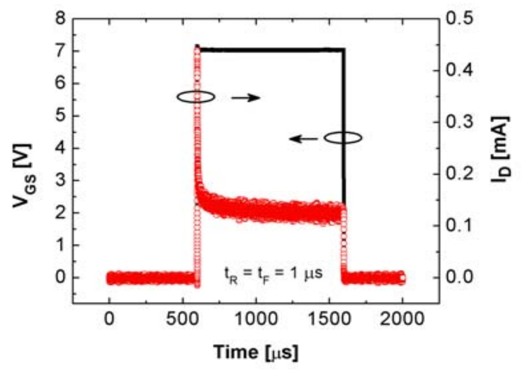
Experimental/Equipment

- A current amplifier is placed at the probe tip to reduce noise and increase frequency response. A digitizing oscilloscope captures the transient.



Data/Results

- Very fast trapping events can be observed and studied in nanoelectronics with new materials systems.
- Data on HfOSi, see M. Gurfinkel et al., IEDM 2006, p. 755



M. Gurfinkel, et al, IRPS . 45, 2007

Impact/Importance

- Accurate reliability projection requires measurement of parameter drift without recovery.
- Transient characterization can determine real physical nature of defects in nano-scale devices that are comprised primarily of interfaces.

Optical Techniques and Applications

Technique/Acronym/References	Typical Applications
Ellipsometry (Spectroscopic) **SE**	Thickness and optical constants of thin films, composition
Fourier Transform Infrared Spectroscopy **FTIR**	Organic contamination, and Low level impurities and dopants
Interferometry	Wafer flatness, surface texture
Optical Microscopy **OM**	Examination of device and material defects, and structural surface details larger than 0.2 mm
Photoluminescence **PL**	Low level impurities and dopants, and Determination of energy bandgap in photonic materials
Photoreflectance **PR**	Quantum well widths, barrier heights, electric fields, strain in layered systems, and doping density
Raman Spectroscopy **Raman**	Organic contaminants, crystallinity and orientation, local stress, temperature
Reflectometry	Rapid determination of single and multilayer film thickness
Scatterometry	Surface and sub-surface defects and topography

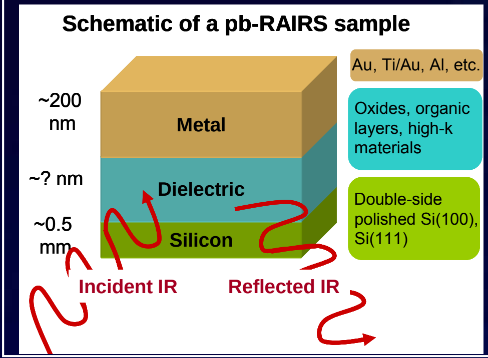
P-polarized Reflection Absorption Spectroscopy (pb-RAIRS or Backside FTIR)

Christina Hacker (christina.hacker@nist.gov)

Goals/Principle/Method

- Vibrational spectroscopy gives chemical (composition) and conformational information (atomic arrangements) at the buried interface
- Study bonding of metal with molecular monolayers and high-K dielectrics.

Experimental/Equipment

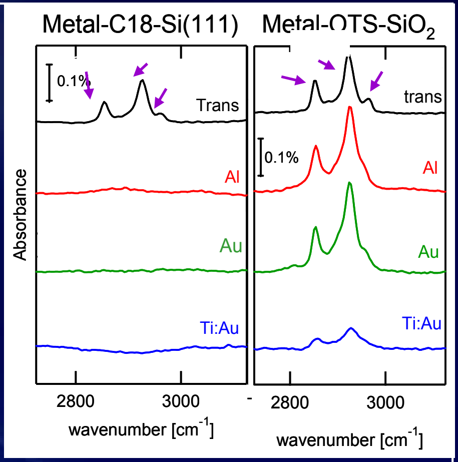


Spectroscopic characterization of buried metal/dielectric interfaces through IR-transparent substrates and thin films.

Data/Results

Observe interaction of metal with molecular layer on silicon molecular electronic structures

C.A. Hacker, et al, *J. Phys. Chem. C*, 2007, *<u>111</u>*, 9384-9392; C.A. Richter, et al, *J. Phys. Chem. B*, 2005, *109*, 21836.



Impact/Importance

- Characterize dielectrics under full metallization
- No special substrate shapes
- Frequency range is not restricted
- Identical samples for FTIR and electrical characterization

Internal Photoemission (IPE) Spectroscopy

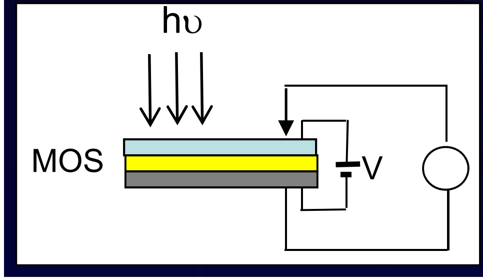
Nhan V. Nguyen (nhan.nguyen@nist.gov)

Goals/Principle/Method

- Determine band offsets of advanced metal oxide semiconductor (MOS) structures.
- IPE generates photo-excitation of electrons and holes over the energy barriers at the interface of metal/oxide and semiconductor/oxide.
- Barrier heights are determined from the quantum yield of IPE signals. Other electronic information can also be obtained including field dependence of the barrier, carrier trapping and scattering.

Experimental/Equipment

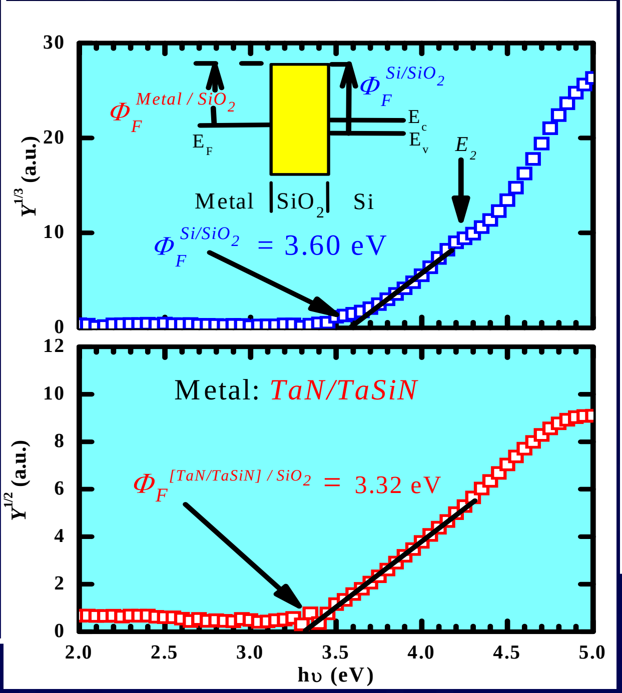
NIST custom-built IPE system is capable of measuring barrier heights of MOS interfaces accurately.



At each applied bias V, the photocurrent (I) is measured as a function of incidence photon energy (hυ) Photoemission yield Y(hυ) is obtained from measured current I(hυ) per incident photon.

Data/Results

- Recent collaboration with Sematech to investigate the band offsets of TaCN and TaSiN metal on SiO_2 and HfO_2/SiO_2 dielectric stacks.
- Barrier heights FF at the interfaces of the metal / [HfO_2/SiO_2] stack / Si and metal / SiO_2 / Si were determined from IPE photoemission yields
- N. V. Nguyen et al., Frontiers of Characterization and Metrology for Nanoelectronics, pp. 308-314 (30-SEP-2007)
- "Internal Photoemission Spectroscopy of [TaN/TaSiN] and [TaN/TaCN] Metal Stacks On SiO_2 and [HfO_2 / SiO_2] Dielectric Stack", N. V. Nguyen et al , submitted to APL



Impact/Importance

- New metals and high-*k* dielectrics are needed for the next advanced IC generation It is critical to know accurately the band offsets at the MOS device interfaces.
- Electronic interface property changes for nanometer film stacks can be monitored and characterized by IPE.

Vacuum-Ultraviolet Spectroscopic Ellipsometry (VUV-SE)

Nhan V. Nguyen (nhan.nguyen@nist.gov)

Goals/Principle/Method

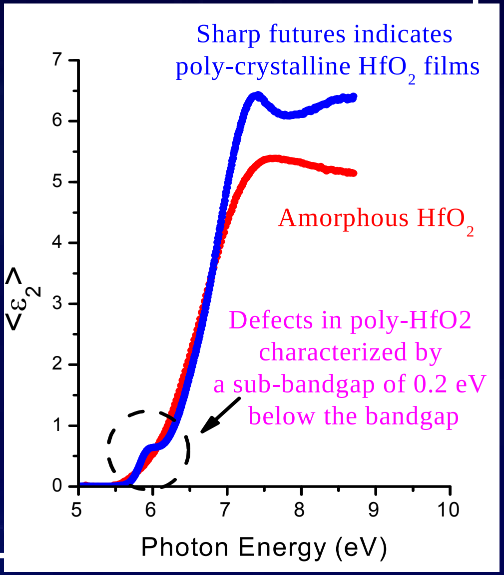
- Optical characterization of materials and thin films relevant to IC industry <u>with sensitivity to subnanometer thicknesses.</u>
- Non-destructive, non-contact, indirect optical technique that measures light polarization changes by multilayer thin films.
- Information extracted by modeling includes thickness, refractive index, absorption coefficient, optical band-gap, composition, density, thin film crystallinity, surface roughness, growth rate, morphology.

Experimental/Equipment

- Two instruments available:
  - Vacuum-ultraviolet spectroscopic ellipsometer (1.0 eV – 10 eV)
  - Fast-scanning mapping spectroscopic ellipsometer (1.0 eV – 5 eV).
- Capabilities: multiple angle of incidence, reflection and transmission ellipsometry, anisotropic material
- Extensive, sophisticated modeling software available to public

Data/Results

- **Current activities: characterizing high-k dielectric thin films on Si and III-V semiconductors**
- **Recent collaboration with IBM to investigate optical properties of ALD HfO_2 by VUV-SE, combined with FTIR and XRD**
- **Please see: "*Sub-bandgap defect states in polycrystalline HfO_2 and their suppression by admixture of Si*", N. V. Nguyen et al., APL 87, p. 192903 (2005)**



Impact/Importance

- Indispensable tool for thickness and optical properties of film stacks.
- Determination of structural film qualities, bandgap, band structure critical points, possible defects.
- Focusing on thin film structures that are technologically important for IC industry such as metal gate and high-k dielectric gate stacks.
- <u>Sensitivity to submonolayer thicknesses and to size effects which are critical to nanotechnology</u>.

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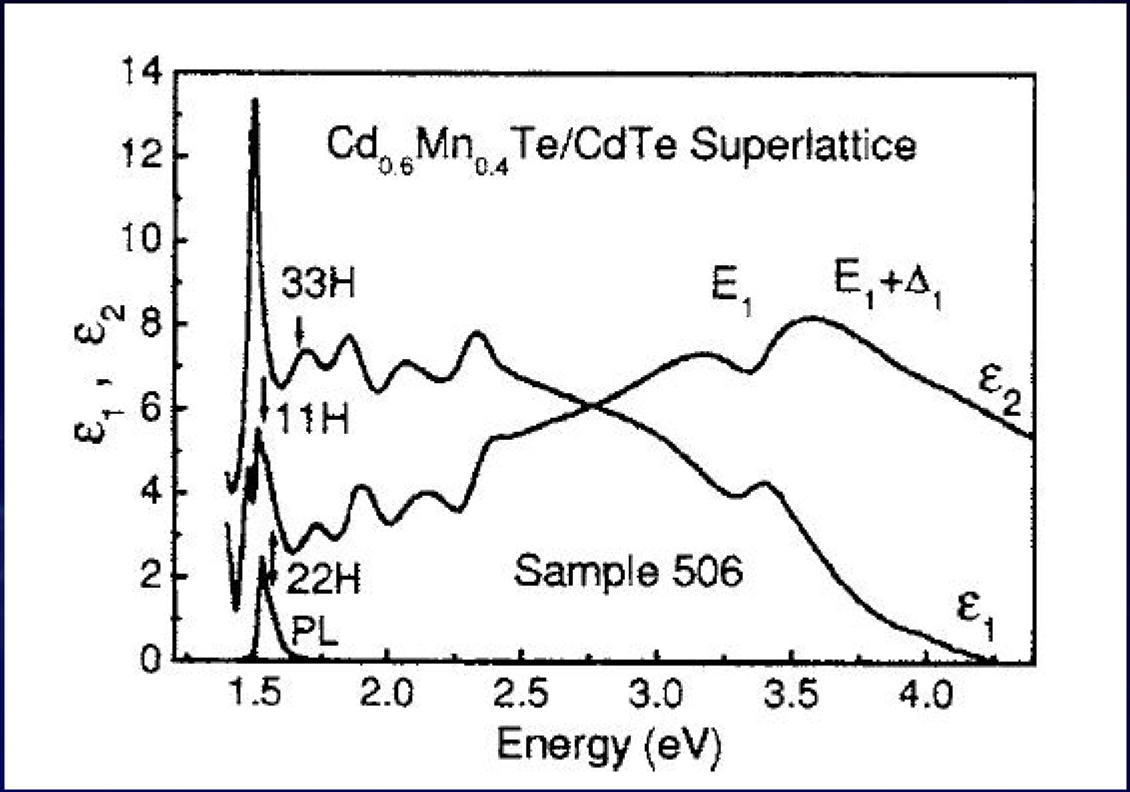
Supporting Data



Dielectric function e1 and e2 and PL spectra obtained for Cd0.6Mn0.4Te/CdTe Superlattice (Vittorio Bellani et al., JAP 98 (2005)

Raman Spectroscopy for Nanocharacterization

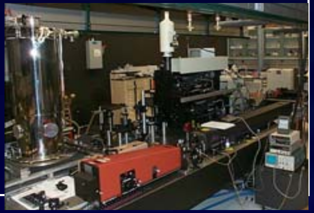
A.R. Hight Walker (angela.hightwalker@nist.gov)

Goals/Principle/Method

- Characterize vibrational modes, lattice modes, torsional modes, chemical composition, molecular structure, crystal structure, alignment, fluorescence, energy transfer, and purity
- Sample preparation is not required and the sample is not destroyed during examination.

Experimental/Equipment

- Unique NIST Facility
  - Spectrometers: triple-grating & micro-Raman
  - Lasers: Multiple! NIR-UV laser excitation
    - Argon ion, Ti:sapphire, HeNe, and Dye
  - Temperature: 4K – 400K cryostat
  - H-field: 8T super-conducting magnet
  - Imaging: Microscope & nano-positioning



Data/Results

- Determine the diameter, purity, alignment of single-walled carbon nanotubes
- Quantify the number of layers of graphene
- Identify magnetic nanoparticle composition: ex. Iron oxide chemical formula, change overtime and magnetic properties.
- *JACS* 29 (34), pp. 10607 -10612, *PRL **98,** 147402 (2007)*

Impact/Importance

- Multiple tools are needed for nano characterization. Raman is one critical component.
- Determine composition and physical properties at the nanoscale
- Water is not a problem, so bio-compatible studies are possible.

The Harnessing of Young's Modulus Characterization for Multi-layered Thin Film Structures

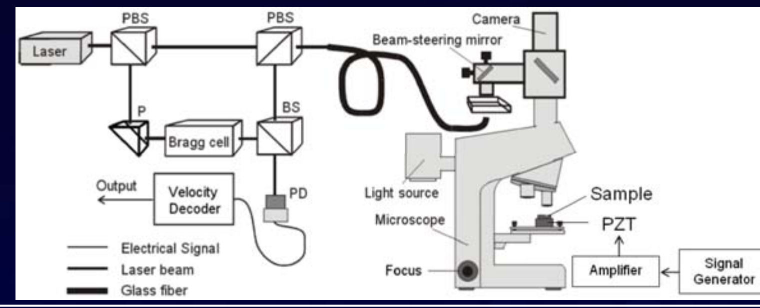
Janet Marshall (janet.marshall@nist.gov)

Goal

- To determine the Young's modulus values of all the layers in a multi-layered thin film structure
- To accurately predict the dynamic behavior, for example the resonant frequency, of a MEMS/NEMS device

Equipment

laser Doppler vibrometer



Results

- Young's modulus values found for multiple layers:
- E_{gl} **= 54.2 GPa ± 1.5 GPa**
- E_{m1} **= 62.1 GPa ± 4.3 GPa**
- E_{de} **= 74.6 GPa ± 4.1 GPa**
- E_{p1} **= 172.3 GPa ± 11.1 GPa**
- E_{fo} **= 63.5 GPa ± 4.3 GPa**

- Marshall, Herman, Vernier, DeVoe, and Gaitan, "Young's Modulus Measurements in Standard IC CMOS Processes Using MEMS Test Structures" Electron Device Letters, <u>28</u>, 2007

Impact

- Young's modulus and dimensional metrology stand out as the most significant test structure needs
- <u>To improve the yield in CMOS processes by reducing the residual stress</u>
  - Given Young's modulus values (for all the layers as described in EDL article) and residual strain values (using ASTM E 2245)
- Web-based data sheet (under construction) will allow quick calculations
  - http://www.eeel.nist.gov/812/test-structures/MEMSCalculator.htm

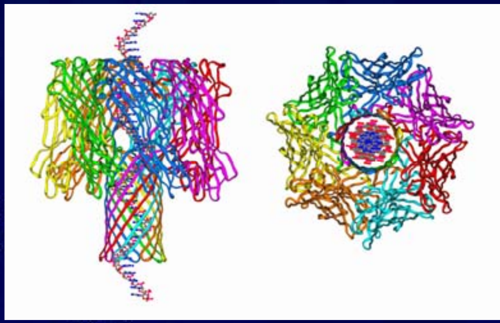
Nanometrology via Nanopores

New Tool for Biological & Chemical Analysis

Enables real-time electronic detection & characterization of:

- single ions and molecules,
- distributions of molecules,
- intra- and inter-molecular interactions,

… without the need for radioactive or fluorescent labels!

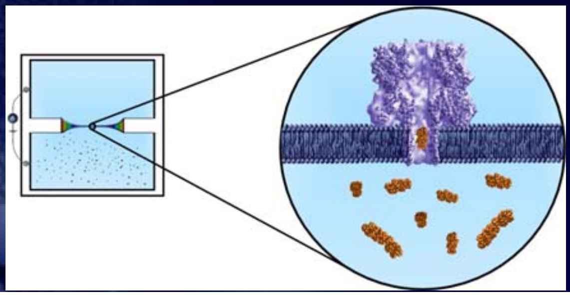


Aqueous Single-Molecule Mass Spectrometry via Electronic Measurement

John Kasianowicz (john.kasianowicz@nist.gov)

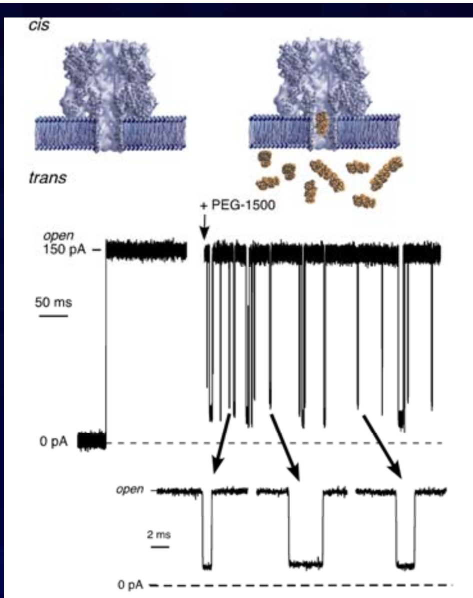
Goals/Principles/Method

Nanoscale Coulter-counter methods are being developed as sensitive molecule-sizing devices. Ionic current through a nanochannel is monitored and molecules partially block the current when they are in the pore. The magnitude of the current blockade can be used to accurately size molecules in aqueous solution.



Experimental/Equipment

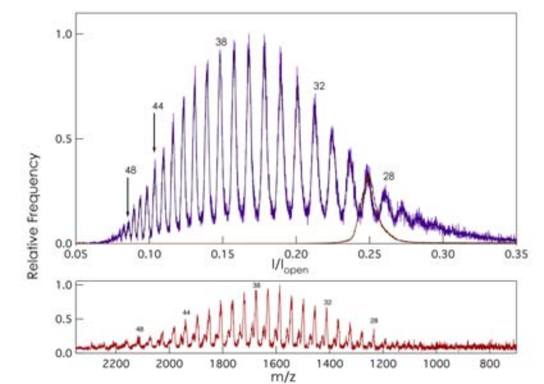
- **Single biological nanopore embedded in a 4 nm thick membrane separating two aqueous solutions.**
- **Voltage applied (& current measured) via Ag-Ag/Cl electrodes. High resolution/low noise current amplifier provides good S/N.**
- **Without polymer sample, ionic current is quiescent.**
- **Adding polydisperse polymer causes transient current blockades.**



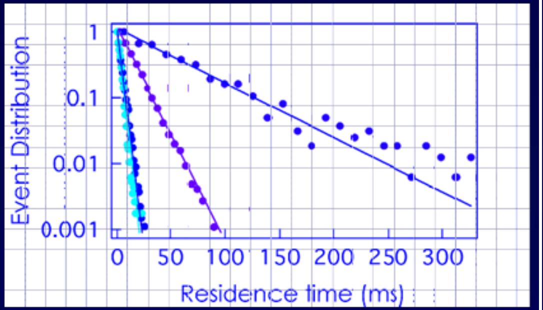
Data/Results

Separation of large mixtures of polymer molecules in aqueous solution obtained from the magnitude of the resistive-pulses and the residence time distributions for each size polymer in the nanopore.

Polymers decrease nanopore current in proportion to their volume (blue); MALDI-TOF control (below)



Residence time of several different polymer sizes in the mixture: 2nd estimate of mass



JWF Robertson et al., Proceedings of the National Academy of Sciences 104 (20): 8207, May 2007.

Impact/Importance

Electronic estimate of molecule mass spectrometry of aqueous samples: complimentary method to traditional mass spectrometry in gas phase.

Technique should prove useful for *in situ* health care monitoring, rapid drug screening , etc.

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technology

Electronic Detection and Characterization of Lethal Bacterial Toxins: Anthrax

John Kasianowicz (john.kasianowicz@nist.gov)

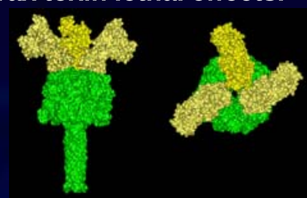
Collaboration with USAMRIID and NCI (Fort Detrick, MD)

Goals/Principles/Method

Bacillus anthracis **(anthrax) secretes 3 lethal proteins: Protective Antigen (PA), Edema Factor (EF), and Lethal Factor (LF). The activate form of PA (PA63) forms a nanopore to which EF and LF bind and presumably gain access into host cells.**

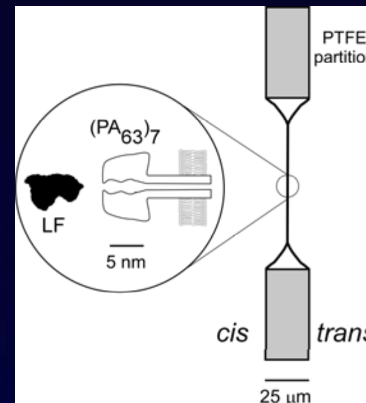
Use high-resolution electronic methods to detect LF and EF, and develop a rapid High Throughput Screening method for therapeutic agents against anthrax toxin lethal effects.

Bacillus anthracis

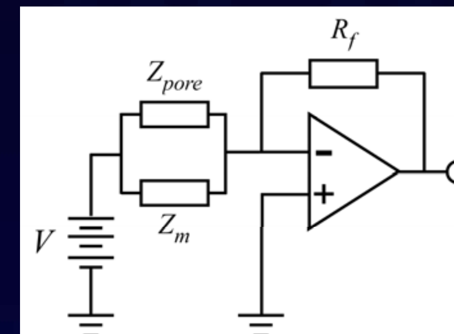


Model for LF (*yellow*) binding to a PA63 nanopore (*green*). Halverson, et al. *J.Biol.Chem.* 2005

Experimental/Equipment

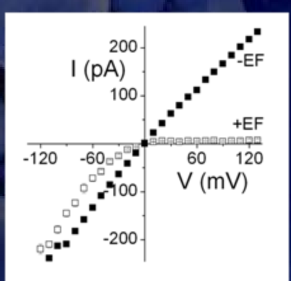


Anthrax PA63 nanopores embedded in artificial lipid membrane formed on an insulating Teflon partition.

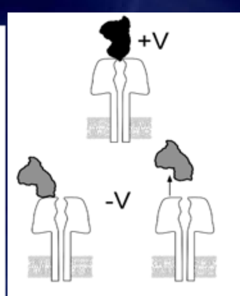


High-impedance amplifier to measure ionic current through membrane and anthrax PA63 nanopore.

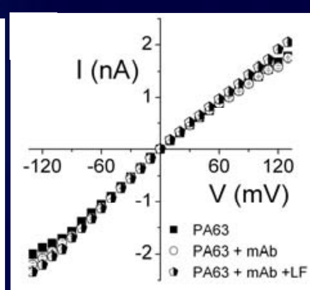
Data/Results



I-V relationship of PA63 nanopores in the absence and presence of EF. EF blocks the pore conductance for + V. Similar results were obtained with LF.



Model for interaction between EF (or LF) & PA63 nanopores: +V forces EF or LF into the pore; - V forces EF out of the pore (either by rotation or expulsion).



An inhibitor of anthrax lethal toxin prevents LF from blocking the PA63 nanopore conductance. This electronic method could be used to rapidly screen a large number of potential therapeutic agents.

Impact/Importance

The ability to rapidly quantitate LF and EF binding to the PA63 nanopore:

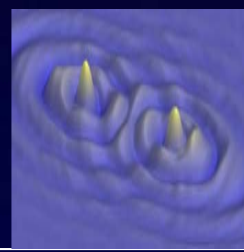
- **Better understanding of how EF, LF and PA63 manifest disease**
- **Electronic method for rapidly screening for therapeutic agents against anthrax infection.**
- **Similar technique used to rapidly detect anthrax toxin complex in infected animal (minutes, not many hours)**
- **Potential tool to combat bioterrorism agents of bacterial origin (e.g., *Yersinia pestis* and multi drug resistant *Mycobacterium tuberculosis*)**

Metrology for Spin Electronics

Bill Rippard (rippard@boulder.nist.gov)

Goals/Principle/Method

- Spintronics uses the quantum-mechanical spin of the electron instead of its charge
- Advantage is low power dissipation and high frequency
- Spintronic devices are enabled only at the nanoscale
- Can combine logic and memory



Experimental/Equipment

Spin-transfer microwave nano-oscillators ...

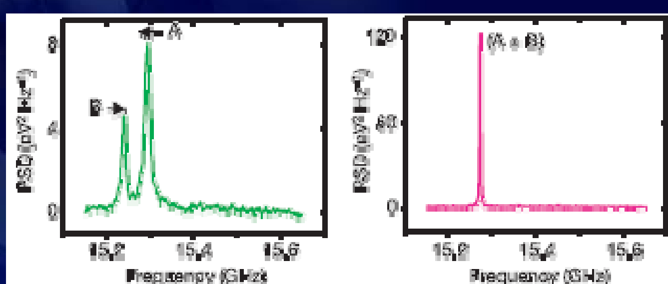
• Electron spin induces coherent precession of magnetization • Tunable from 5 to > 40 GHz with dc current and magnetic field • Diameter 50 nm
• Very narrow spectral linewidths

Requires high frequency measurement of ...

• Spin lifetimes & diffusion lengths • Spin waves

Data/Results

Phase-locking of spin-transfer oscillators …



***Nature* 437, 389-392 (2005), *Phys. Rev. Lett.* 97, 087206 (2006).**

Impact/Importance

Future "beyond-CMOS" devices, such as ...

• spin oscillators • spin batteries • spin transistors
• spin mixers • spin-wave interconnects

With applications in ...

• nanoscale clocks • spectrum analyzers • high-frequency signal processors • magnetic random access memory

Instrumentation and Metrology

- **R&D**
  - Resolution
    - molecular to atomic spatial scales
    - short temporal scales
  - Sensitivity and Specificity
    - molecular or atomic level sensitivity and specificity with simultaneous imaging and identification
    - simultaneous multiple probes (spectroscopies, etc.) for chemical and physical properties
  - 3-D characterization capability, atom by atom, or molecule by molecule, over many thousands of atoms.
  - Increase knowledge of the fundamental physical understanding of current instruments
  - Signal-to-noise ratio vs. speed
  - Supporting models, methods, standards, data, uncertainty analyses
  - Interdisciplinary research teams
- **Nanomanufacturing**
  - All the above and…..
  - What is needed for nanomanufacturing and production?
  - When will it be needed?
  - Roadmap?
  - Who will pay?

nanotechnology

Metrology, Nanocharacterization and Instrumentation for Emerging Nanotechnology and Nanoelectronics – *Particle Beam and Scanned Probe Instrumentation*

Dr. Michael T. Postek, Chief

Precision Engineering Division

Manufacturing Engineering Laboratory

National Institute of Standards and Technology

IEDM 2007
Washington D.C.
December 2007

NIST
National Institute of Standards and Technology
Technology Administration, U.S. Department of Commerce

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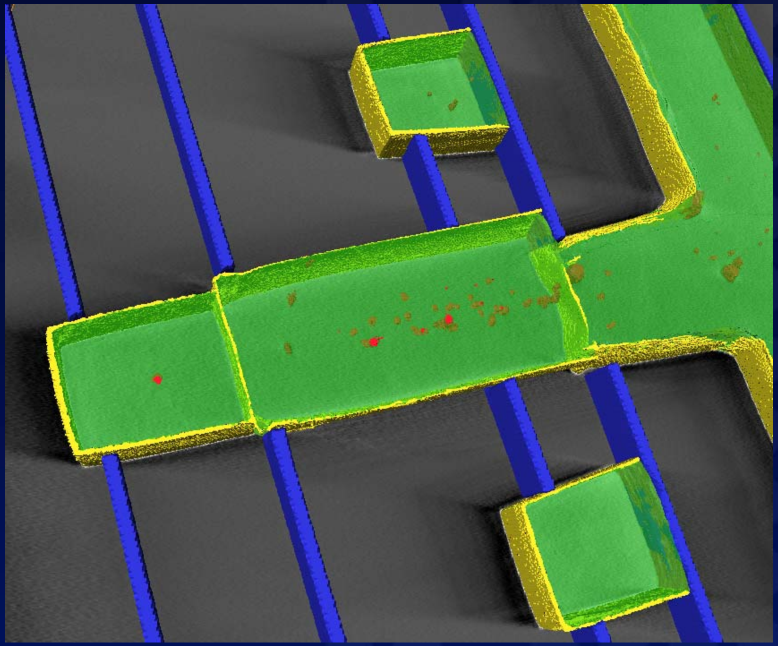
Nanotechnology/Nanoelectronics

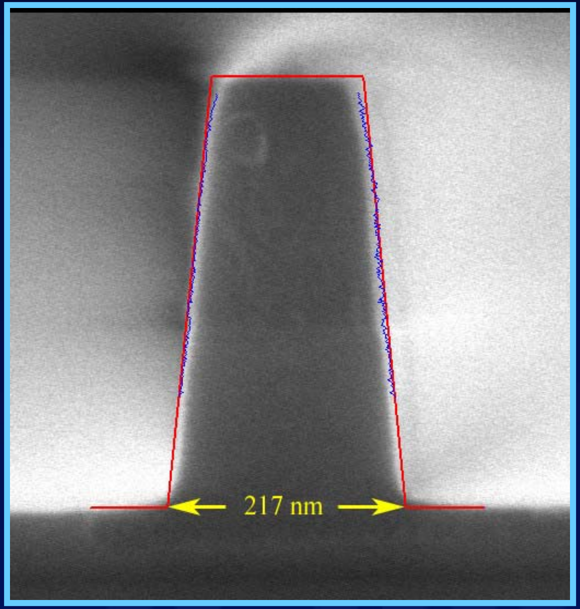
- Technology that is the convergence of the state of the art in materials science, chemical science and biological science to make better products for the future.
- Potentially revolutionize semiconductor industry and yield many new high-tech products
  - Link between scientific discovery and commercial products:
    - Nanomanufacturing



MEL Innovation & productivity

Imaging and Measurements

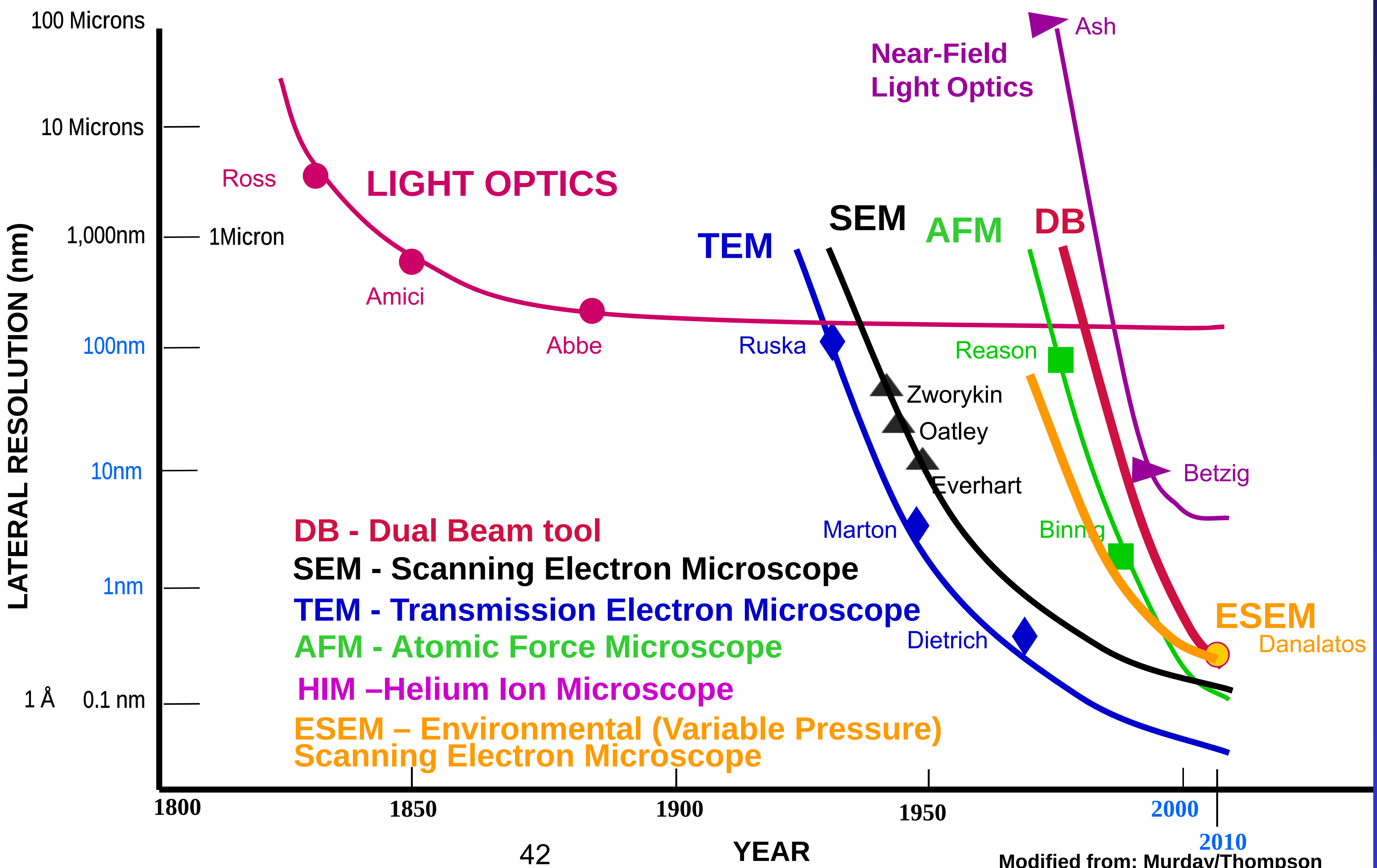
- Both nanoelectronics and nanomanufacturing require:
  - Atomic level measurement accuracy and repeatability
  - Ability to measure desired performance attributes
  - Commercially viable production costs
  - Accurate high throughput measurements
- Advanced measurement technology is needed by nanoelectronics and nanomanufacturing more than for any other prior technology
  - Avoid past technological problems
    - Asbestos



Nanometrology Challenge

- Much of the measurement infrastructure currently available for nanoelectronics and nanomanufacturing is only evolutionary
  - Optics
  - Transmission Electron Microscope
  - Scanning Electron Microscope
  - Scanning Probe Microscope
- Automated, operator-independent instrumentation adapted to nanomanufacturing must be developed and is indispensable
  - This was one of the key conclusions at the NNI Grand Challenge Workshop on Instrumentation and Metrology and the recent NIST Nanomanufacturing Workshop
- New, potentially revolutionary metrology is needed for many applications
  - Helium Ion Microscope

Nanoelectronics Imaging and metrology



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  - This was one of the key conclusions at the NNI Grand Challenge Workshop on Instrumentation and Metrology and the recent NIST Nanomanufacturing Workshop
  - *Will the semiconductor industry remain the strong driver?*
- New, potentially revolutionary metrology is needed for many applications
  - Helium Ion Microscope

Nanometrology Challenge

- Much of the measurement infrastructure currently available for nanotechnology and nanomanufacturing is only evolutionary
  - Optics
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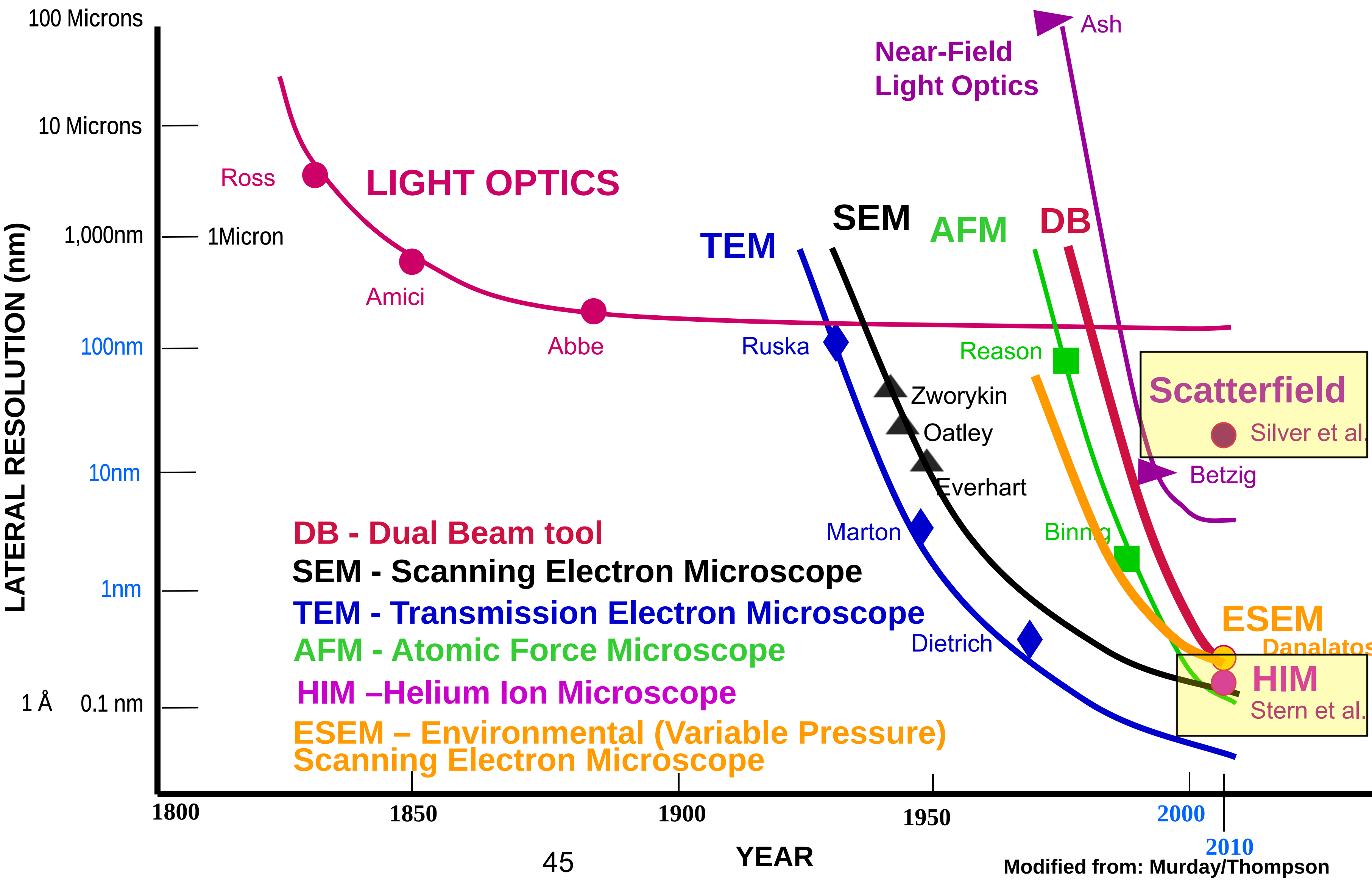
- New, potentially revolutionary instrumentation is needed for many applications
  - Helium Ion Microscope
  - Scatterfield Microscopy

Nanoelectronics Imaging and Metrology



Typical Particle Beam Instrument Resolution

Field Emission Scanning Electron Microscope	~8-9 Å (high kV)
Critical Dimension Scanning Electron Microscope	~18-20 Å (low kV)
In-Lens Field Emission Scanning Electron Microscope	~4 Å
Environmental Scanning Electron Microscope	~8-9 Å
Dual Beam Focused Ion Beam	~8-9 Å (SEM Column) ~30-50 Å (Ion Column)
Atomic Force Microscope	~1-5 Å
Aberration Corrected Transmission Electron Microscope	~0.5 Å
Helium Ion Microscope	~2.5-3.0 Å

Nanoelectronics Manufacturing Instrument Requirements

- Variable depending upon the phase of the process:
  - Research - the business is providing data/information
  - Development - the business is providing proof of product or process
  - Manufacturing - the business is establishing, maintaining or improving economic yield
    - Achieving the economy of scale

Optical Microscopes (over 300 years)

- Optical microscopes in the fab from the beginning
- Serious measurement scrutiny began around 1973 (Diana Nyyssonen) with photomask metrology
  - Standard photomask reference materials SRM 473, 474 and 475
  - Continued today with SRM 2059 photomask standard
- Evolution of optics continues today as workers try to push optics as far as possible because of the advantages
  - Non destructive
  - Fast
  - No vacuum

Scatterfield Optical Microscopy

Rick Silver (silver@nist.gov)

Goals/Principle/Method

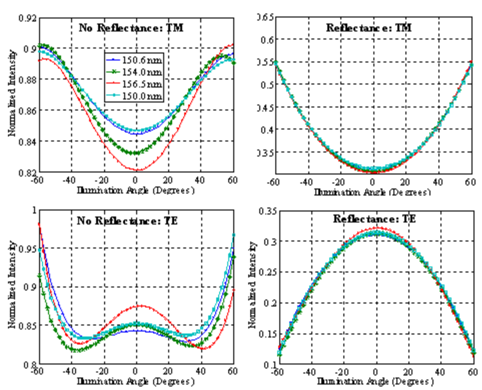
- High throughput optical techniques for metrology and process control with nanometer sensitivity.
- Adapting structured illumination in bright field optical tools enables sensitivity to sub-nanometer dimensional changes.
- Zero-order imaging techniques are extensible beyond the 22 nm node.

Experimental/Equipment

- The scatterfield configuration enables scanning plane waves of illumination.
- Scanned illumination with high magnification optics allows microscopic angle resolved intensity scans in parallel over a large area.
- Angle resolved measurements substantially enhance access to information content.

Data/Results

Sensitivity to nanometer changes in linewidth using scanned and structured illumination.



R.M. Silver, et al, Applied Optics, **2007**

Impact/Importance

- The technique can be applied to dense positioned features smaller than 20 nm CD.
- Applications include angle resolved illumination for defect metrology.
- Super-resolution for overlay metrology.

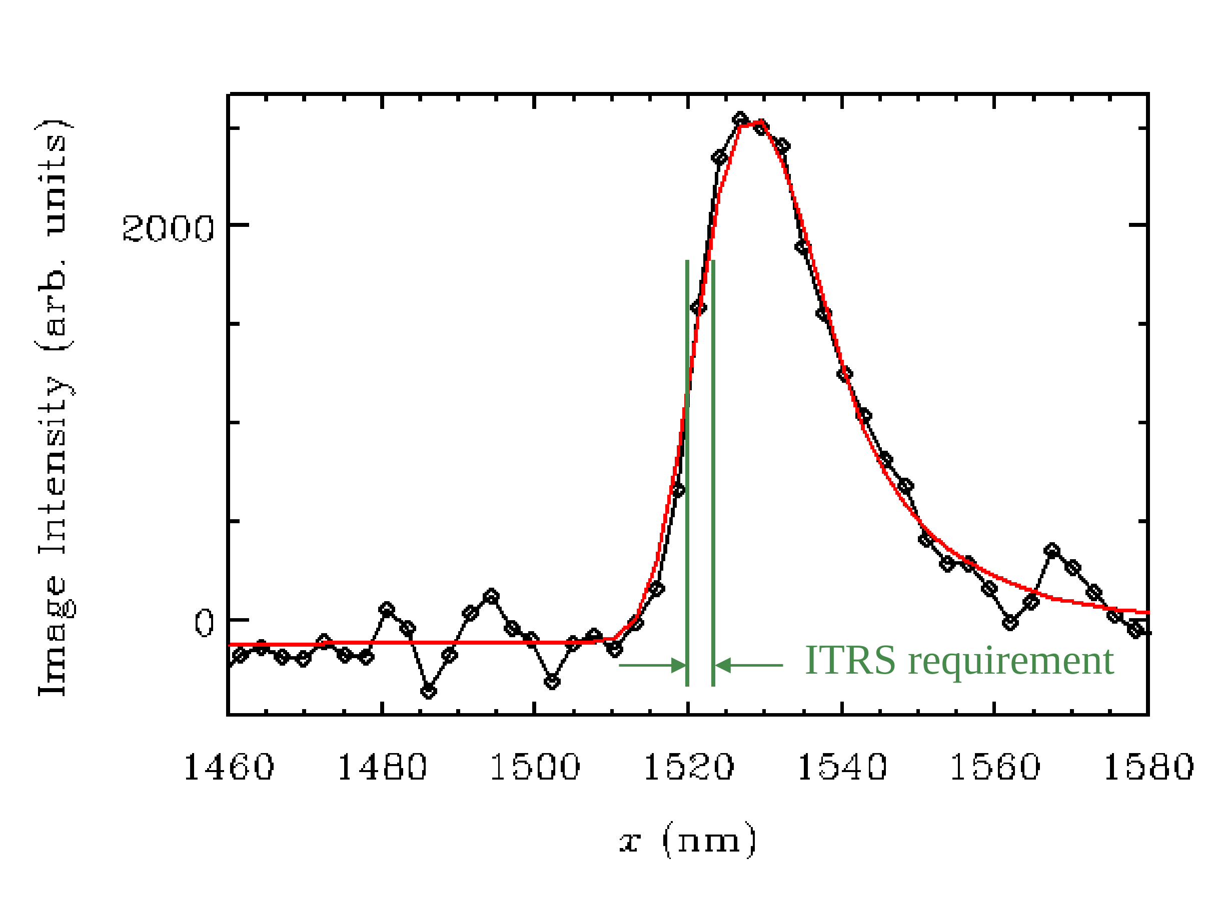
Scanning Electron Microscopes (~1980)

- **SEMs began to seriously evolve in electronics manufacturing about 1980**
- **Evolved from modified laboratory tools used on the production lines and mask houses to dedicated, fully automated production tools**

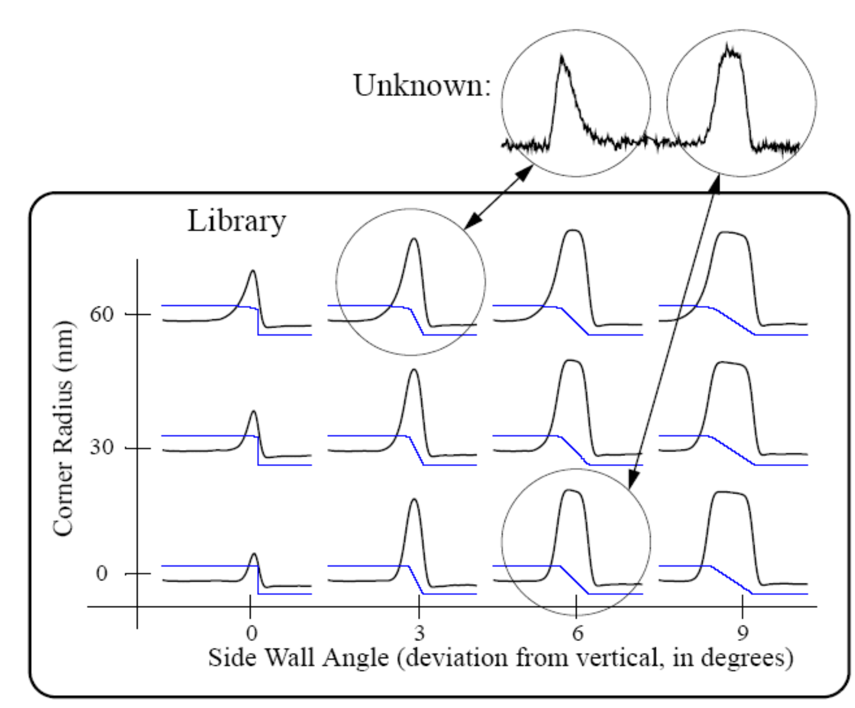
Courtesy of Hitachi

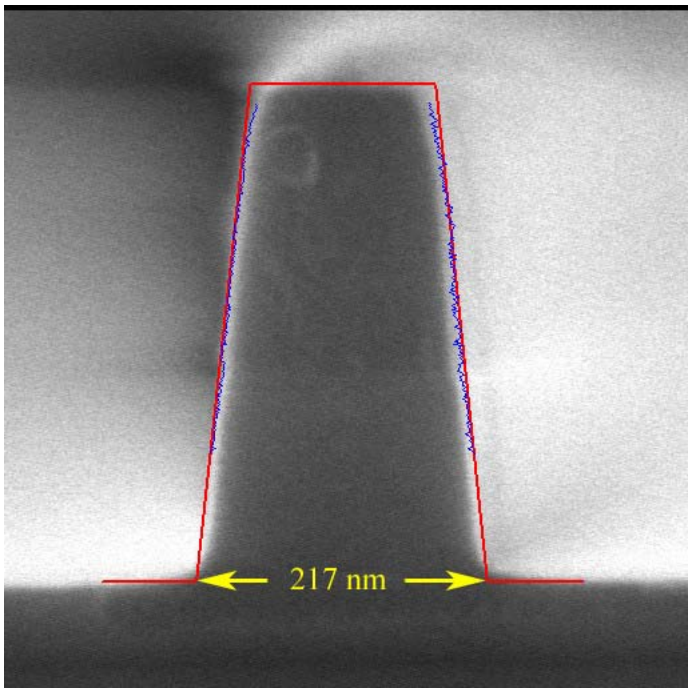
Shape-Sensitive Linewidth Measurement for Accurate Nanometrology

- **With narrower lines and tighter tolerances, bloom widths are no longer insignificant.**
- **A more quantitative imaging model is needed**
- **Adjust unknown instrument and sample parameters for best fit.**



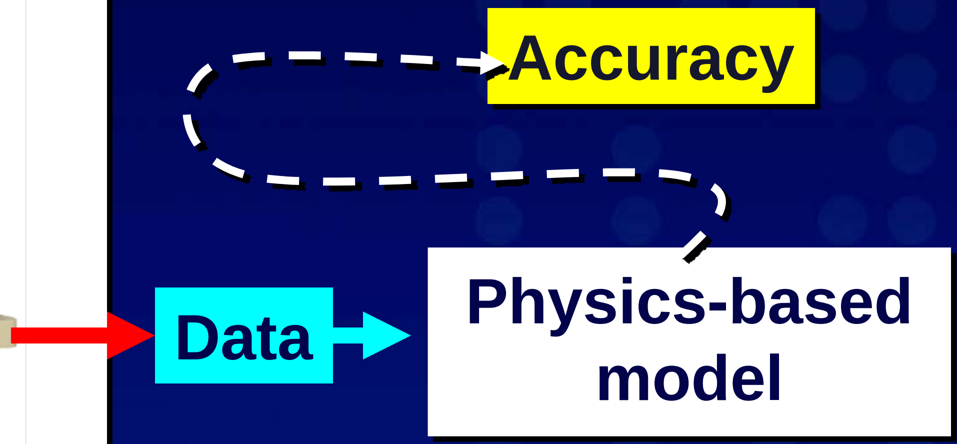
- Time to match an isolated line 1s/linescan





Model-based Metrology for Accurate Measurements

- **Because Physics is implemented in measurement algorithm MBL demonstrated a 3x precision improvement over current measurement methods.**
  - Competitive advantage
  - HHTA sent Ms Maki Tanaka, an engineer from Japan to work at NIST as a guest researcher to learn this technology.



SEM, Ion Beam & HIM Dimensional Nano-Metrology

Andras E. Vladar SEM Project Leader (andras@nist.gov)
John Villarrubia, Modeling Project Leader (John.Villarrubia@nist.gov)

Goals/Principle/Method

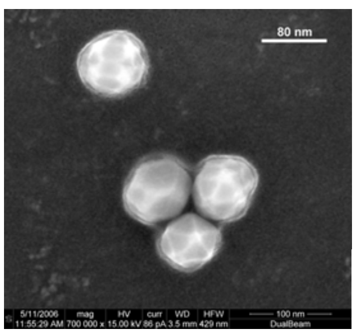
- Accurate, 3-dimensional size, shape and composition metrology with scanned electron and ion beams with 1 nm or better resolution.
- Non-destructive technique used for variety of research, development and process control applications in semiconductor and nano- and bio technology.

Experimental/Equipment

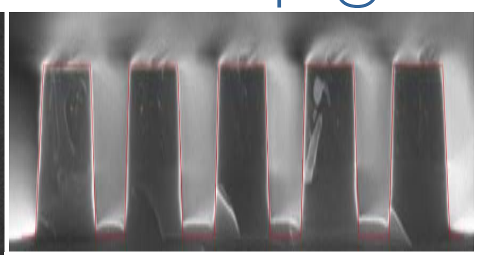
- Can image and measure conductive and non-conductive solids and liquids (in frozen state) in the size range of 5 cm to 5 nm.
- Secondary, transmitted, backscattered electron signals, material deposition and ion milling, etc.
- State-of-the-art scanning electron and scanning Ga and He ion microscopy

Data/Results

Size and shape @ nm



80 nm colloidal RM gold particles



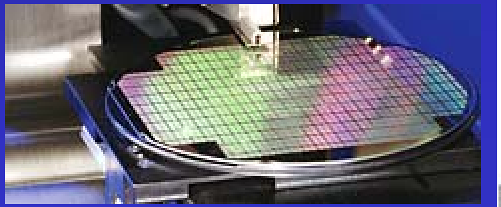
Amorphous Si integrated circuit structures and model-based determination of their size and shape (red line)

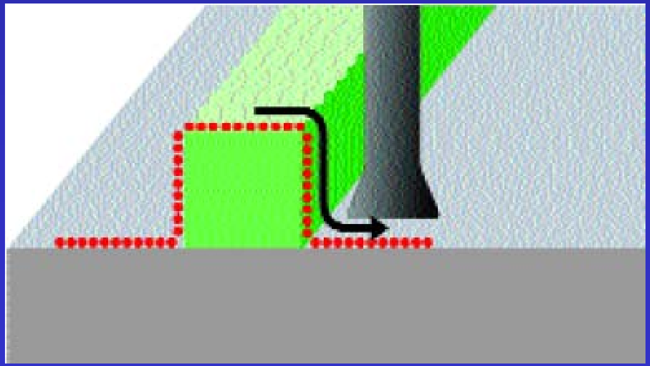
Impact/Importance

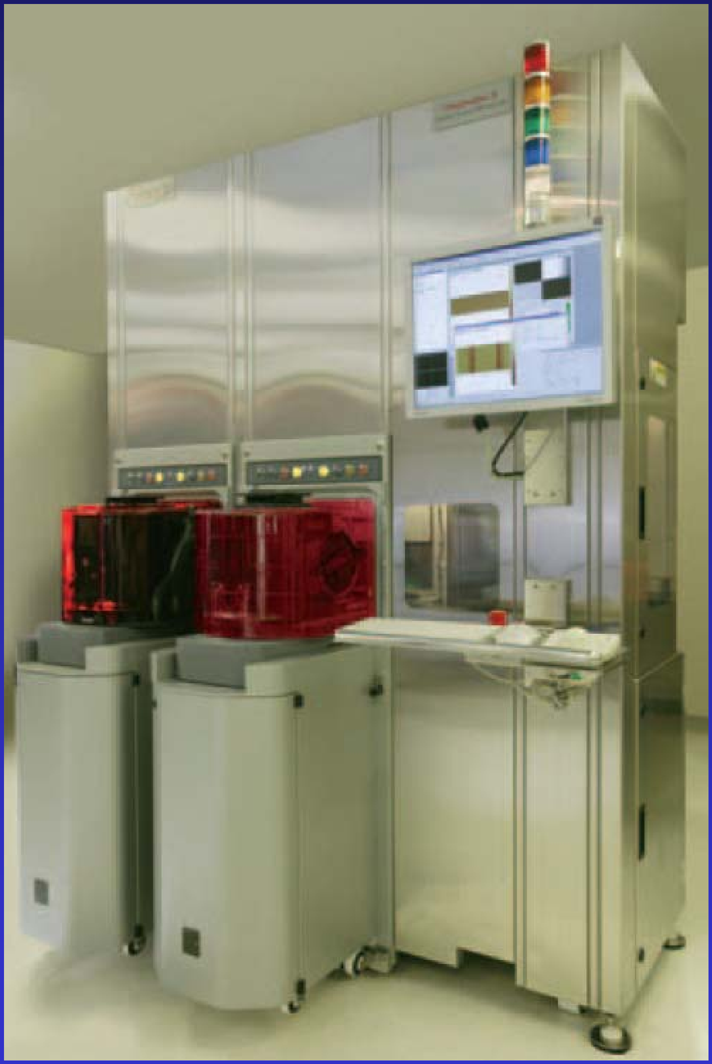
- One of the most widely used, versatile techniques for a variety of scientific and industrial fields and applications
- 1 nm equals to 1 billion dollars in the semiconductor industry
- Key imaging and measurement technique for the emerging nanotechnology

Atomic Force Microscopes (~1986)

- AFMs began as a laboratory instrument around 1986 and commercial instruments began to appear soon thereafter
- Automated AFM evolved from laboratory tools used for dimensional measurements on the production lines and in mask houses







Courtesy of Veeco

Traceable Atomic Force Microscope (AFM)

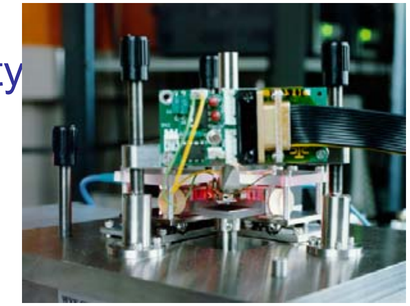
Ronald Dixson (ronald.dixson@nist.gov)
George Orji (ndubuisi.orji@nist.gov)

Goals/Principle/Method

- AFM surface sensing combined with traceable displacement metrology offers excellent reference metrology.
- AFM is a non-destructive technique.
- Non-topographic contrast is negligible in many circumstances, allowing dimensional measurements with competitive uncertainties.

Experimental/Equipment

- The NIST Calibrated AFM (C-AFM) has Intrinsic SI traceability in all three axes – through use of displacement interferometry.



Data/Results

- Major C-AFM pitch measurements:

Measured Pitch	Expanded Uncertainty (k=2)
199.85 nm	0.90 nm
1999.5 nm	3.1 nm
287.01 nm	0.72 nm
700.1 nm	2.0 nm

Impact/Importance

- C-AFM provides NIST-internal and some external reference measurements. It also has been used to participate in three international comparisons of pitch and step height measurements.

Reference Measurement System (RMS)

George Orji (ndubuisi.orji@nist.gov)
Ronald Dixson (ronald.dixson@nist.gov)

Goals/Principle/Method

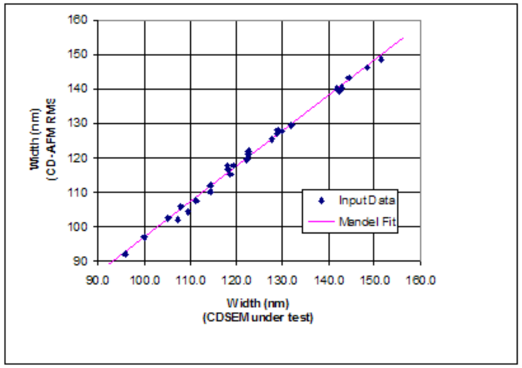
- Critical dimension atomic force microscopy (CD-AFM) can sense the near-vertical sidewalls of semiconductor structures.
- Current generation CD-AFMs have production-compatible throughput.
- If scanner displacements and tip width are calibrated, CD-AFM is capable of linewidth measurements with expanded uncertainty of about 1.6 nm (k = 2).

Experimental/Equipment

- Starting in 2001, NIST assumed metrological responsibility for a CD-AFM at SEMATECH and implemented it as a reference measurement system (RMS).
- The RMS provides a source of traceable reference metrology for both SEMATECH internal projects and direct support for member companies.

Data/Results

- The RMS has been used as a source of accuracy for SEMs at SEMATECH



Impact/Importance

- The CD-AFM based RMS at SEMATECH has supported several generations of SEM and OCD tool evaluation and benchmarking.
- It has also strengthened general NIST-SEMATECH cooperation in metrology.

Dual Beam (1998)

- **Development of dual beam tools transformed electron microscopy from a passive data collection instrument to a tool which can both image and modify matter to the nanoscale.**

+

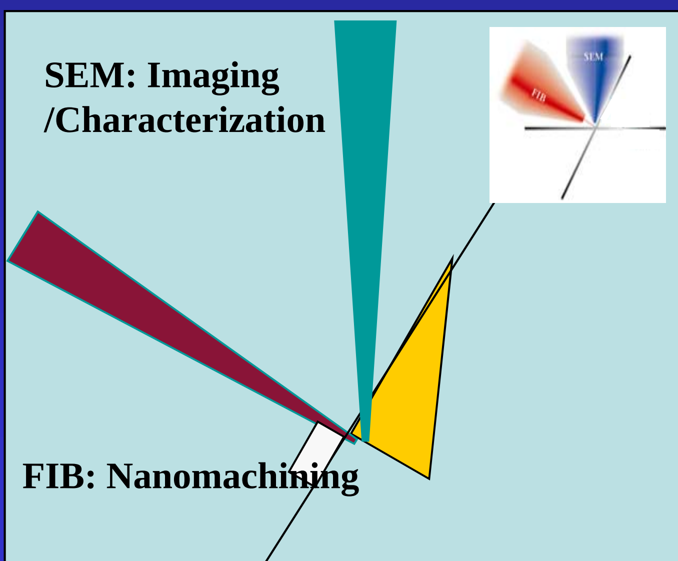
=

Scanning Electron Microscope + Ion column = Dual Beam tool

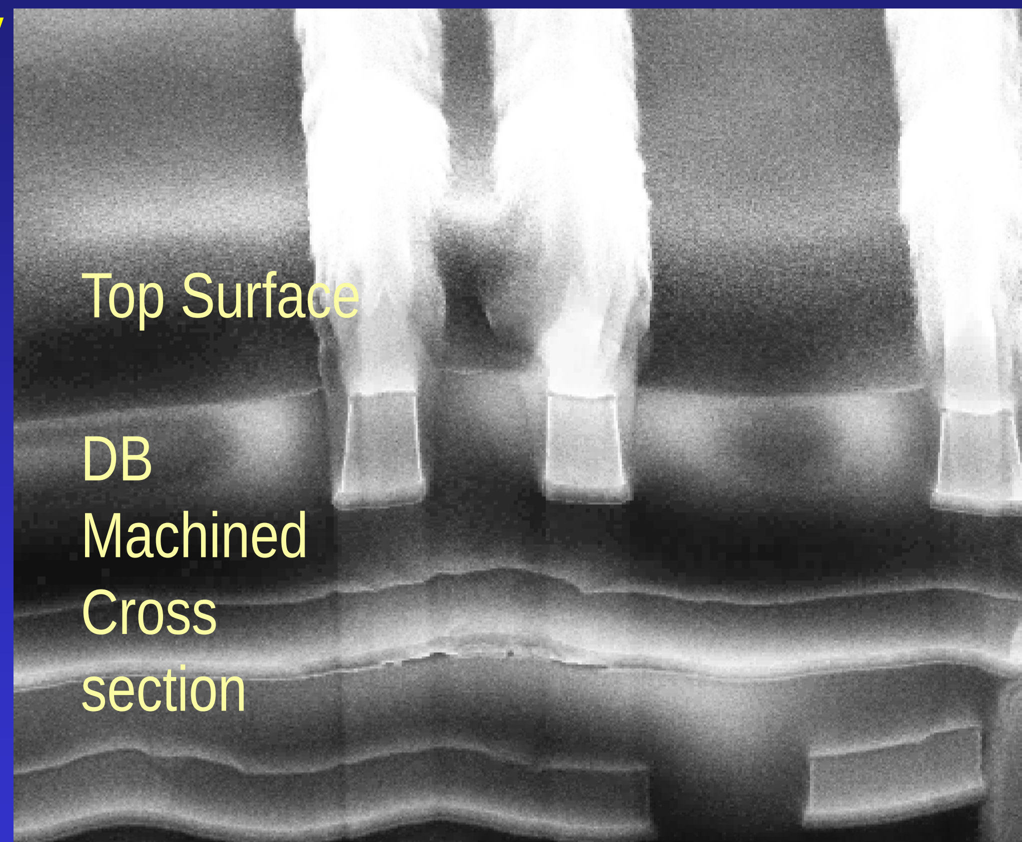
Courtesy of FEI

The Dual Beam tool: Micro/Nano Machining

Technique.
The SEM electron beam images the object, the ion beam machines away material. Gas chemistry is used to remove the sputtered material in gaseous form

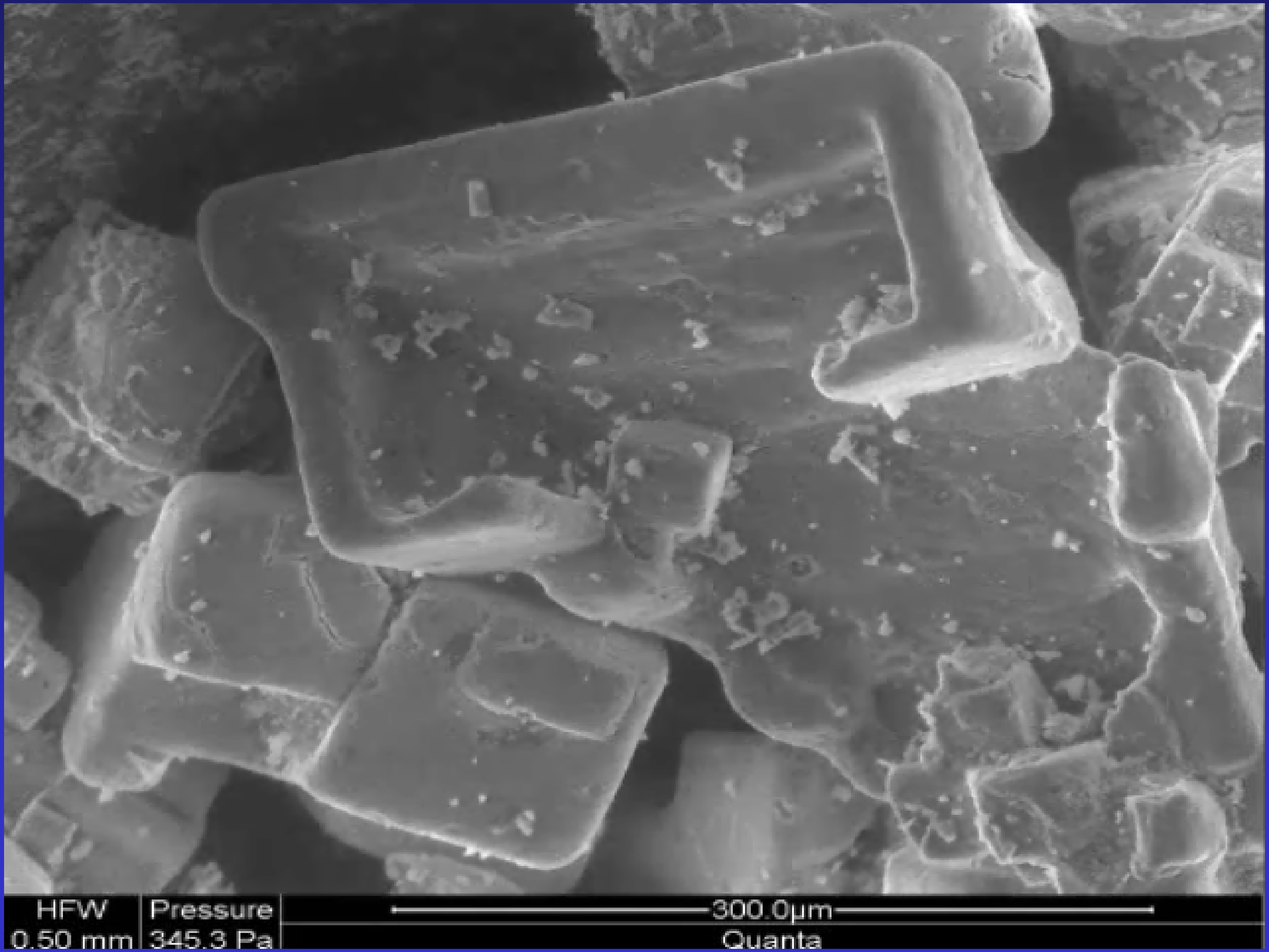


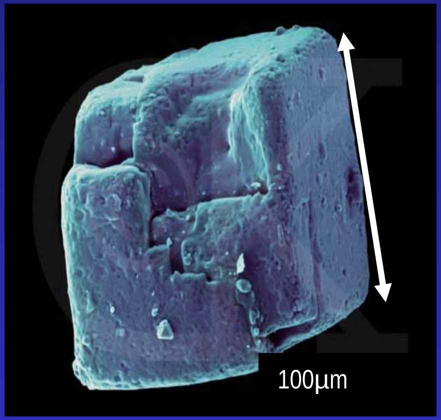
Cross-sectioning



Courtesy of FEI

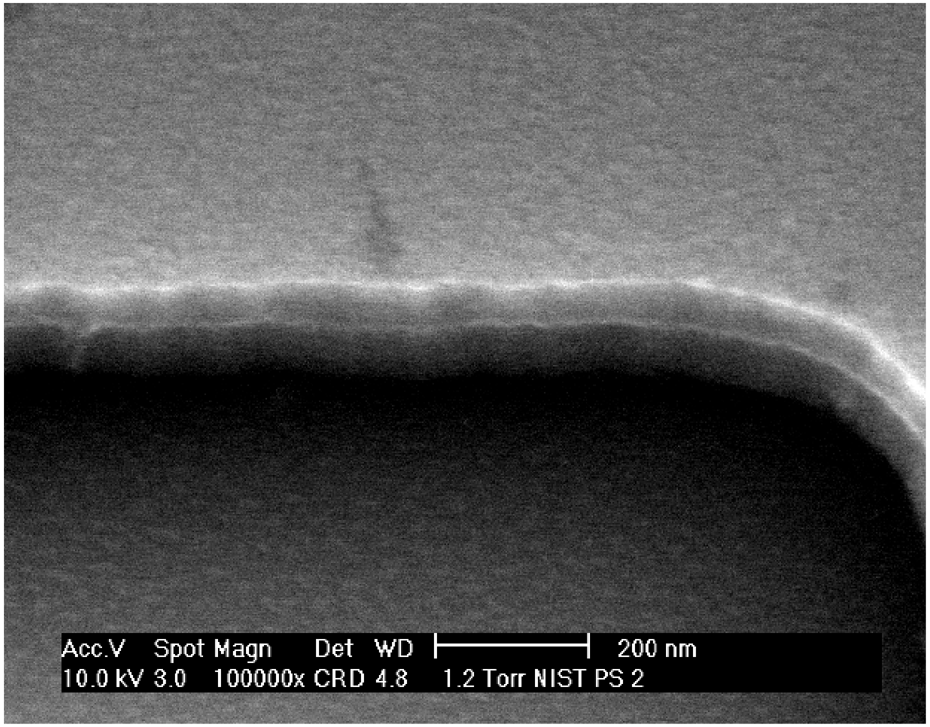
ESEM: Observe Dynamic Processes and Achieve Charge Reduction

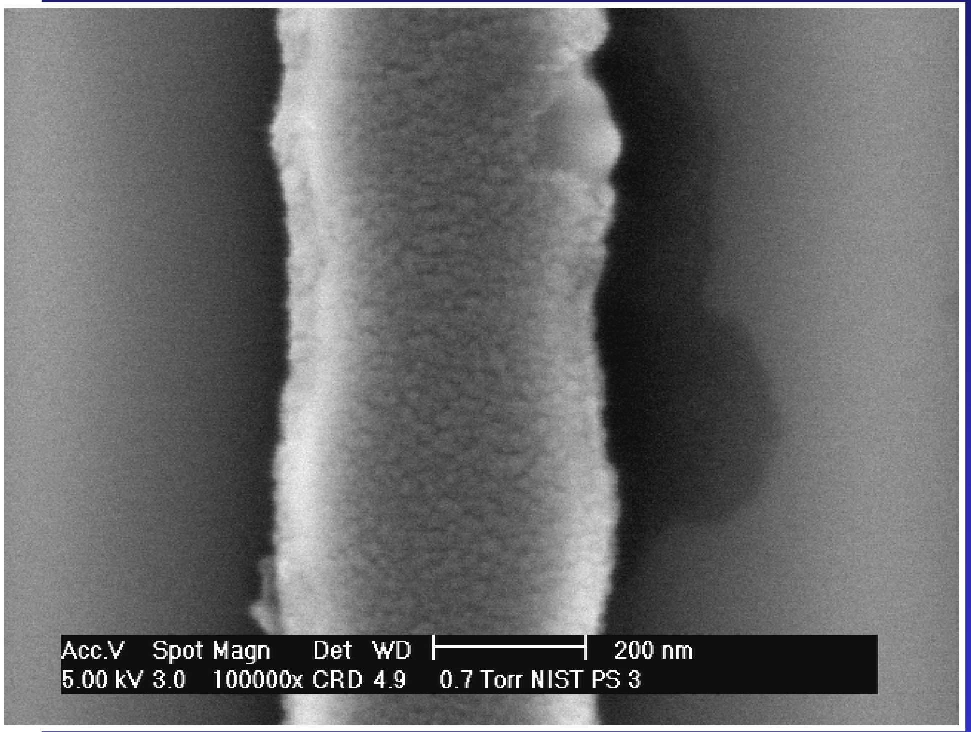




Environmental SEM imaging of NaCl Dissolution & recrystallisation

ESEM: Achieving Charge Reduction and High Resolution for Photomask and Resist Imaging and Metrology





Tilt

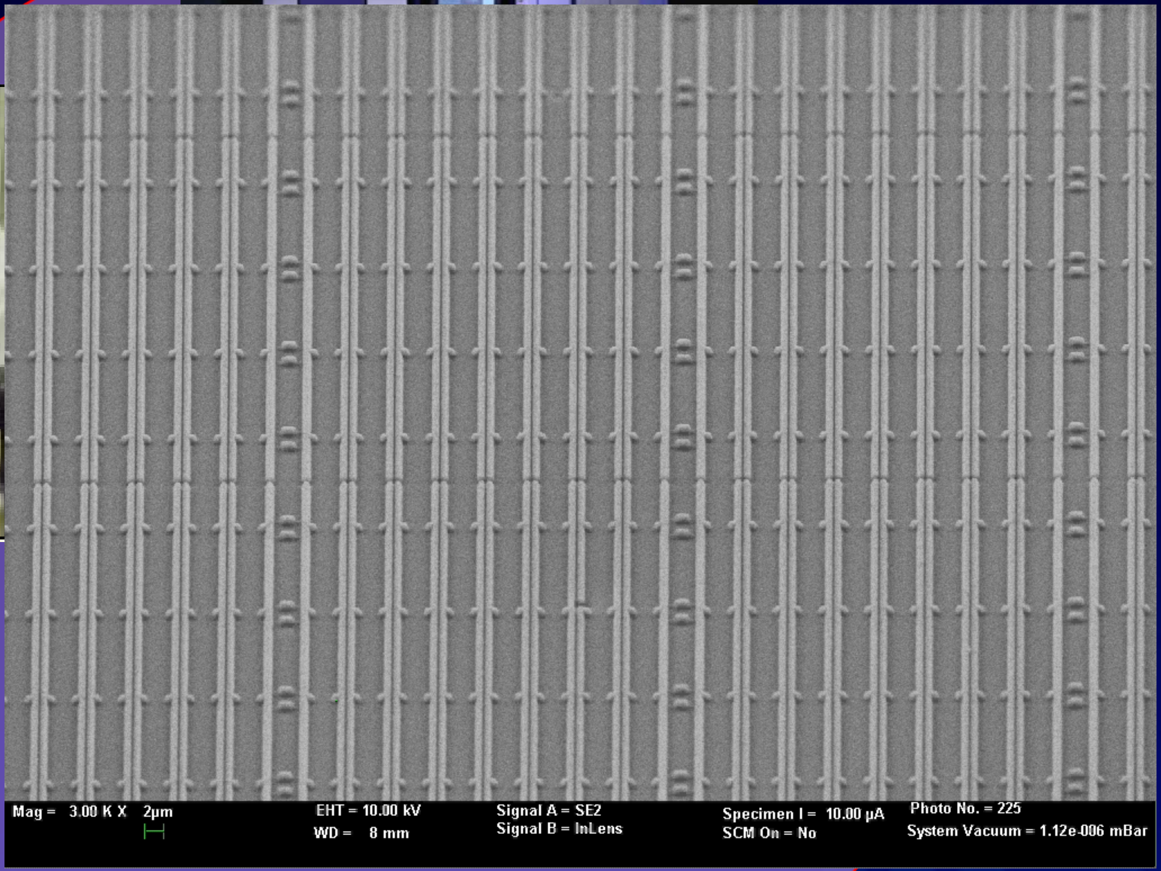
1.2 Torr
10 kV
100,000 x

Top Down

0.7 Torr
5 kV
100,000 x

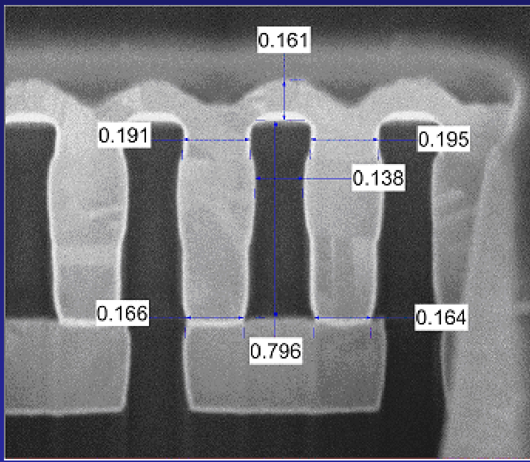
Wafers to Atoms

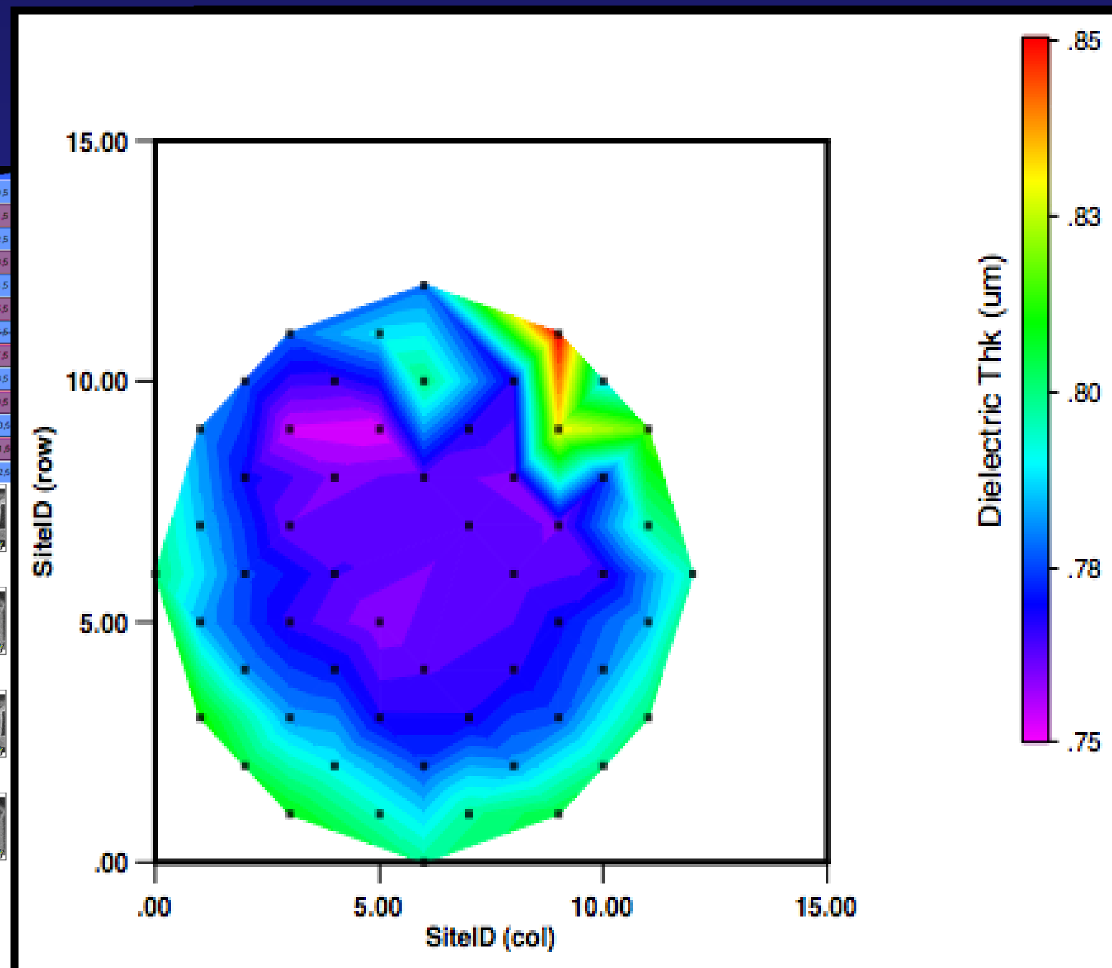
12 inch wafer



Courtesy of Zeiss

Metrology: Dual Damascene Via Chain Thickness

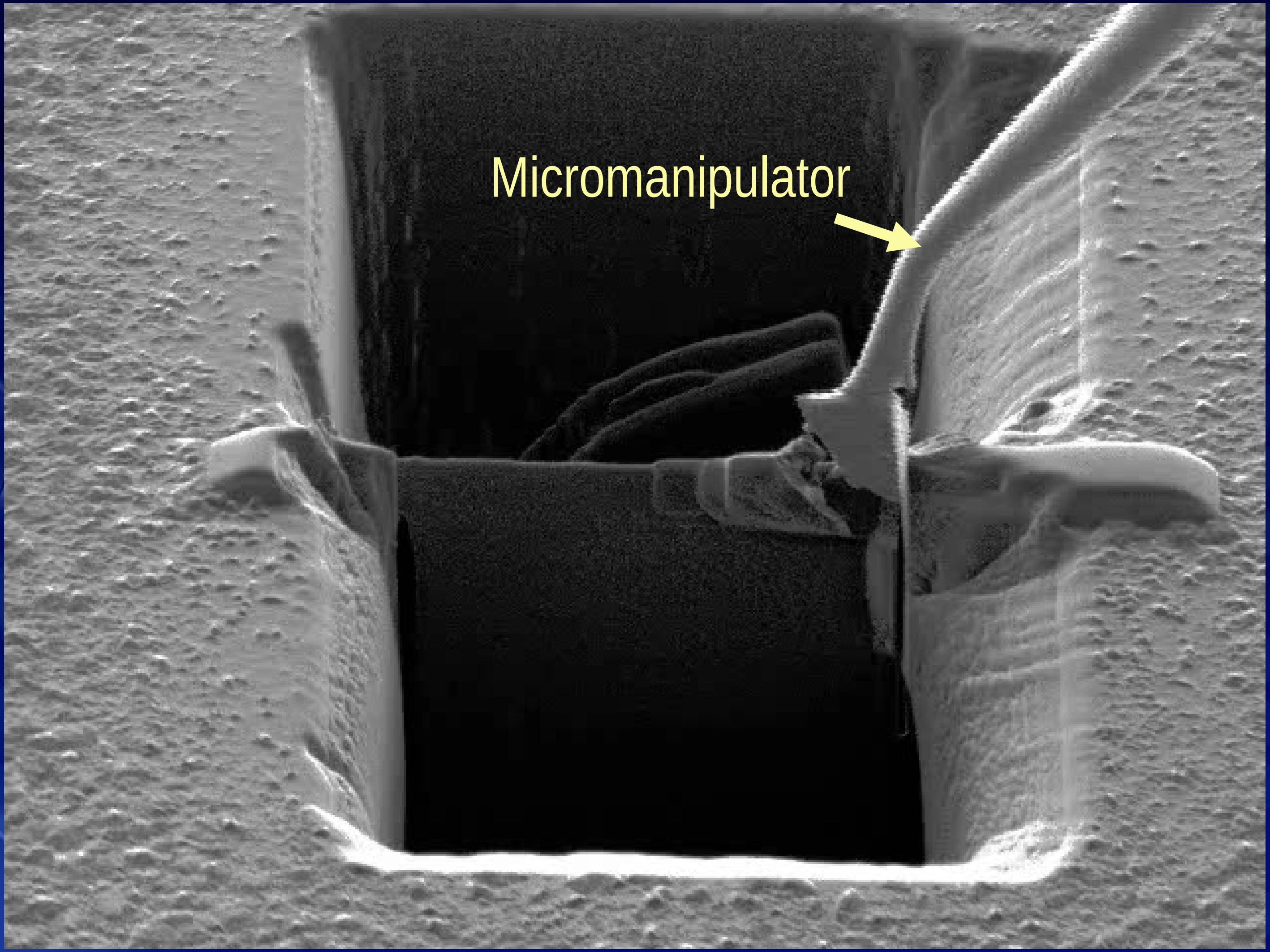




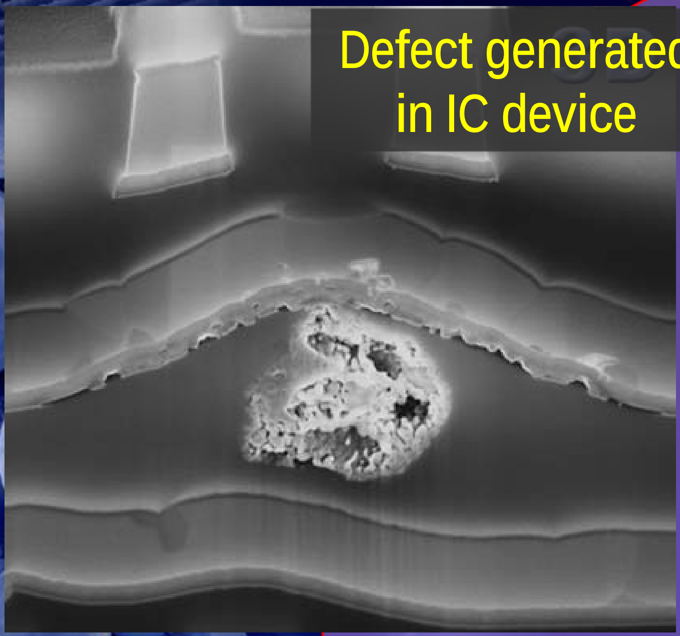
- 155sec per Site Cross Section Data Showed ~10% Center to Edge Thickness Variation

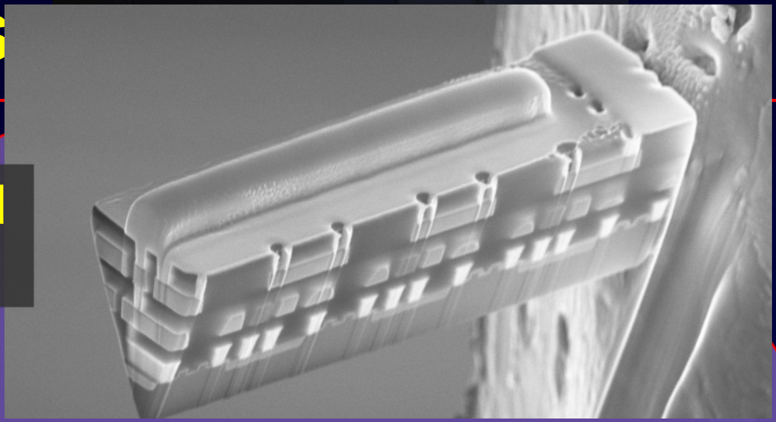
Courtesy of FEI

The Dual Beam tool: Micro/Nano Manipulation

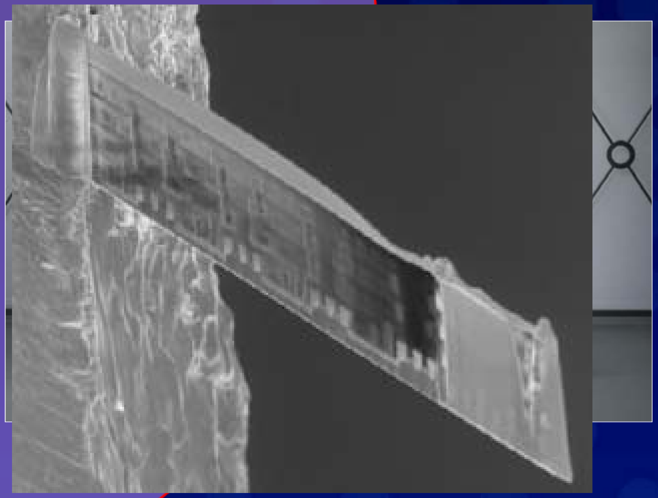


Wafers to Atoms





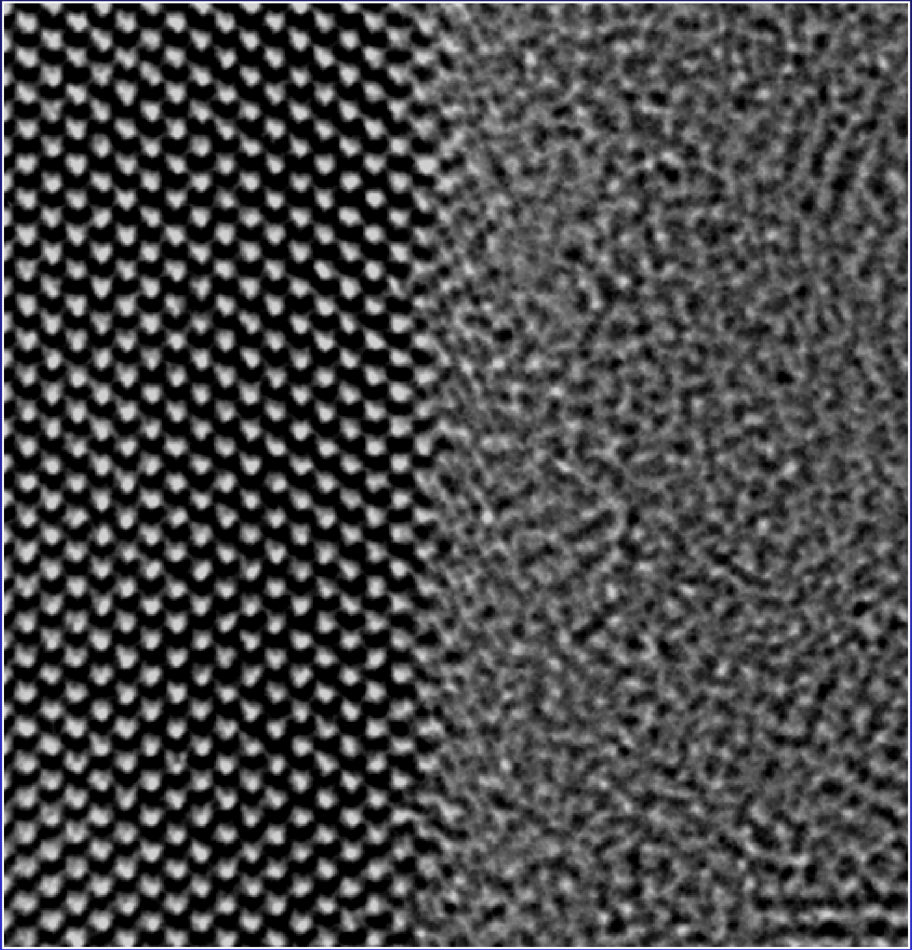
Automated Lift-out



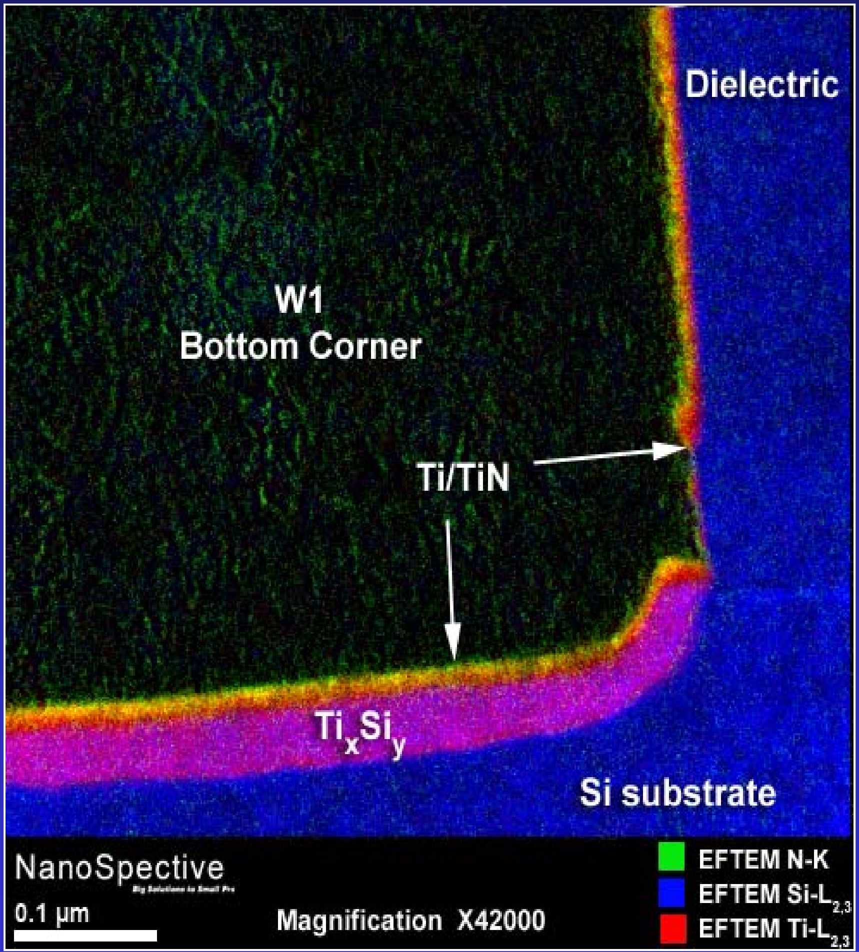
Sample Thinning

innovation & productivity

TEM: Atomic Imaging and Chemical Analysis of Devices



Crystalline Amorphous



Energy Filtered chemical Mapping

Courtesy of FEI

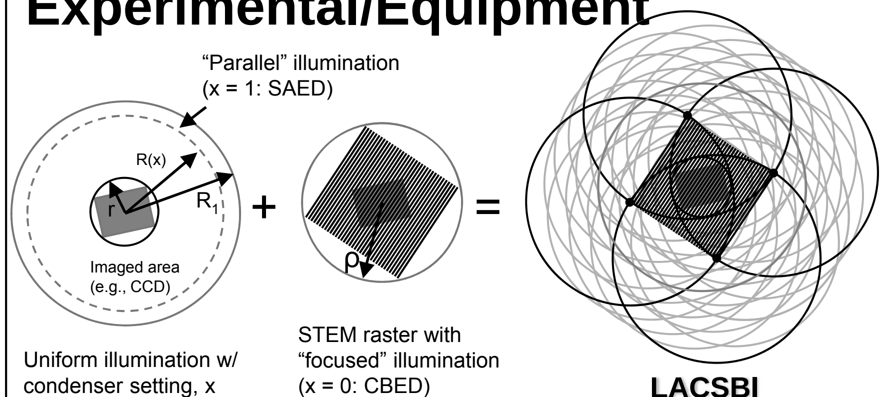
Large Angular Convergence Scanned Beam Illumination (LACSBI): Incoherent Imaging for Conventional TEM

Ian M. Anderson (ian.anderson@nist.gov)
John Small (john.small@nist.gov)

Goals/Principle/Method

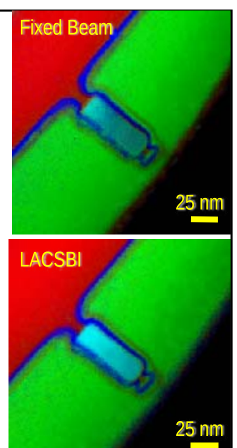
- CTEM provides a significant advantage in data collection efficiency relative to STEM, since pixels are acquired in parallel rather than in series.
- Quantitative CTEM imaging of crystalline specimens is inhibited by diffraction effects that can lead to errors in quantitative compositional analysis of a factor of 2 or more.
- The LACSBI method involves CTEM imaging while using the STEM raster to damp down coherent diffraction effects.

Experimental/Equipment



Data/Results

- EFTEM spectral image shows next-generation transistor with single-crystal Si, amorphous SiO_2, Si_3N_4, and HfO_2 phases
- Complex layered dielectric structure visible
- Intensity variation due to diffraction effects in Si fin (fixed beam) removed by LACSBI with no visible loss of spatial resolution



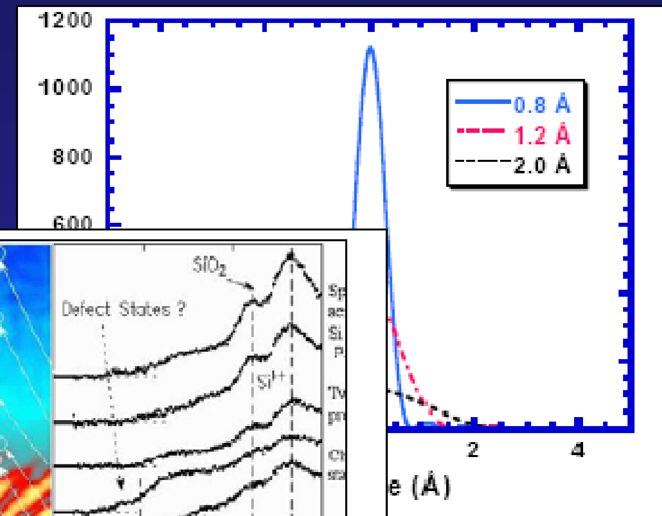
Collaboration with Brendan Foran, Aerospace Corp

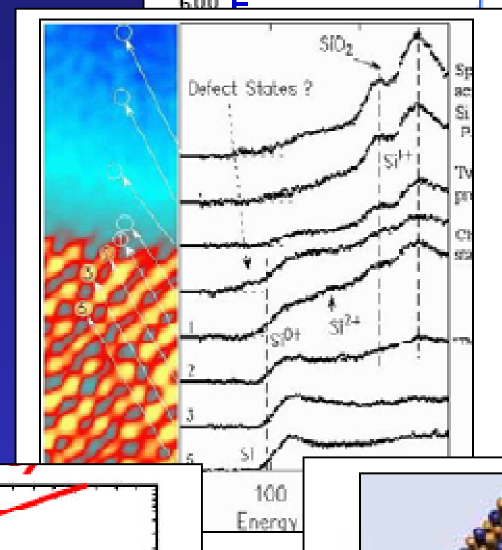
Impact/Importance

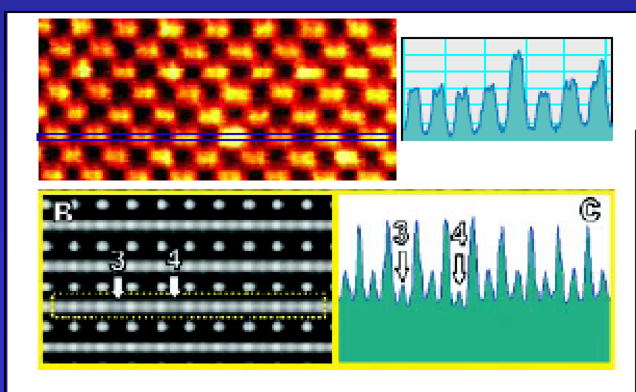
- The LACSBI method damps coherent contrast mechanisms in CTEM imaging, thus removing "show stopping" obstacle to efficient quantitative chemical imaging at nanometer resolution.

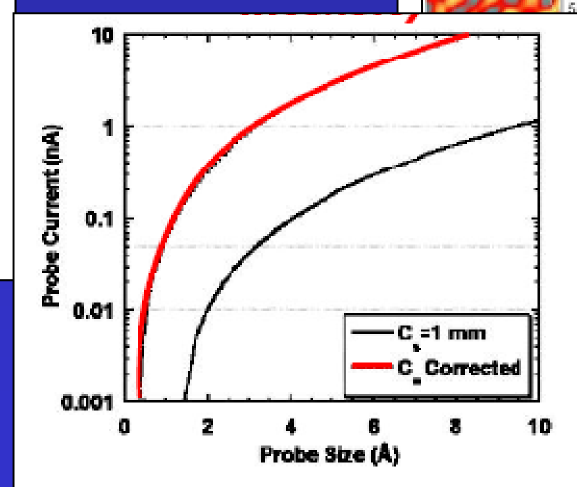
Aberration Corrected TEM

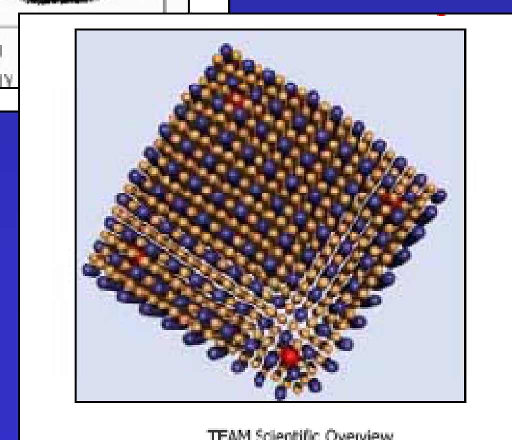
- Smaller Probe
- Increased Signal
- Improved Contrast
- Higher Probe Intensity
- Greater Sensitivity



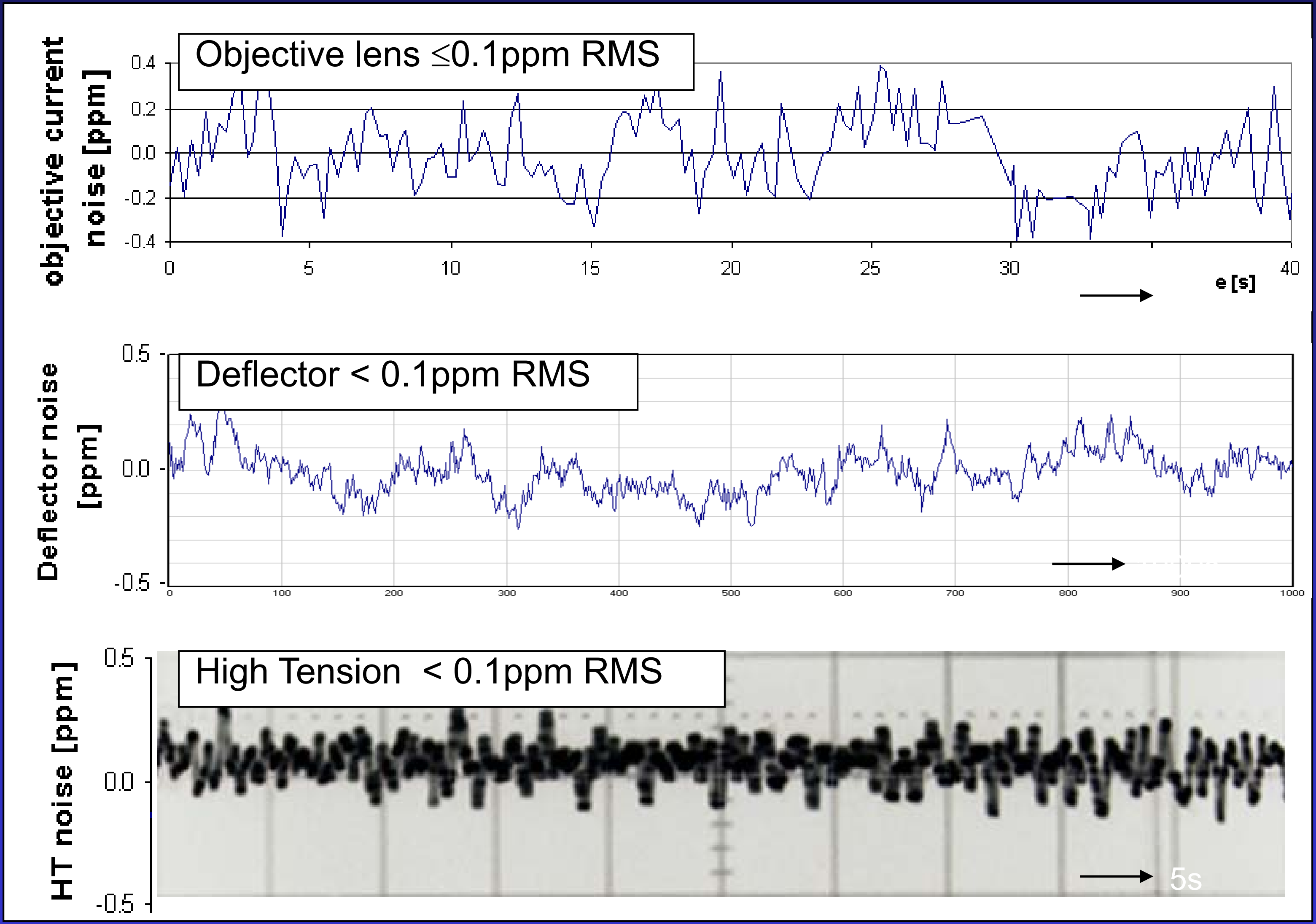








Trade-off: TEM Electronic Correctors need 0.1 ppm stability



Helium Ion Microscope (HIM)

- **First of this new type of instrument has recently been installed at NIST within the Manufacturing Engineering Laboratory, Precision Engineering Division**
- **Helium ions (He^+) are used to irradiate the sample**
- **HIM resolution is expected to be 0.25 nm or 4 times better than the best current large sample SEMs**
  - **Detail obscuring specimen coating is eliminated**
  - **Surface detail information is enhanced due to physics of signal formation**
- **Application of NIST expertise to the study of the understanding of the physics of this instrument will facilitate more accurate measurements and standards development.**
- ***Will this be the next production CD tool?***

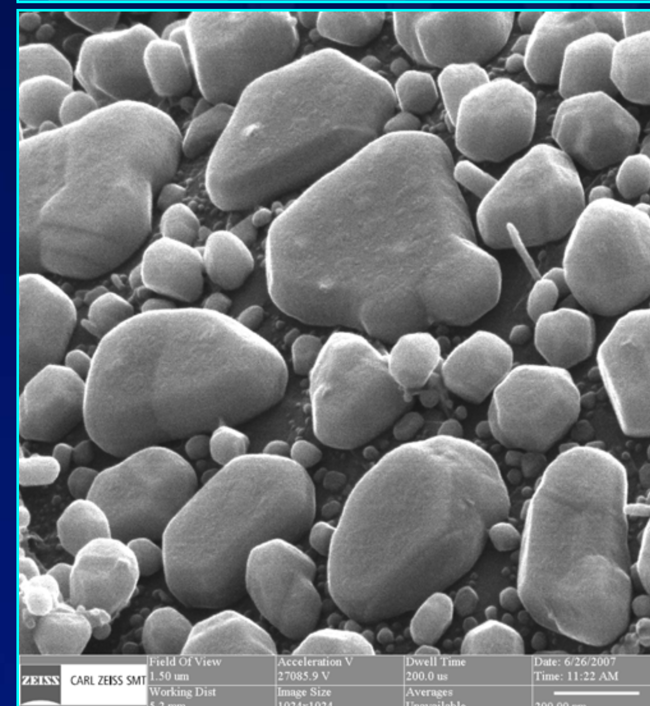
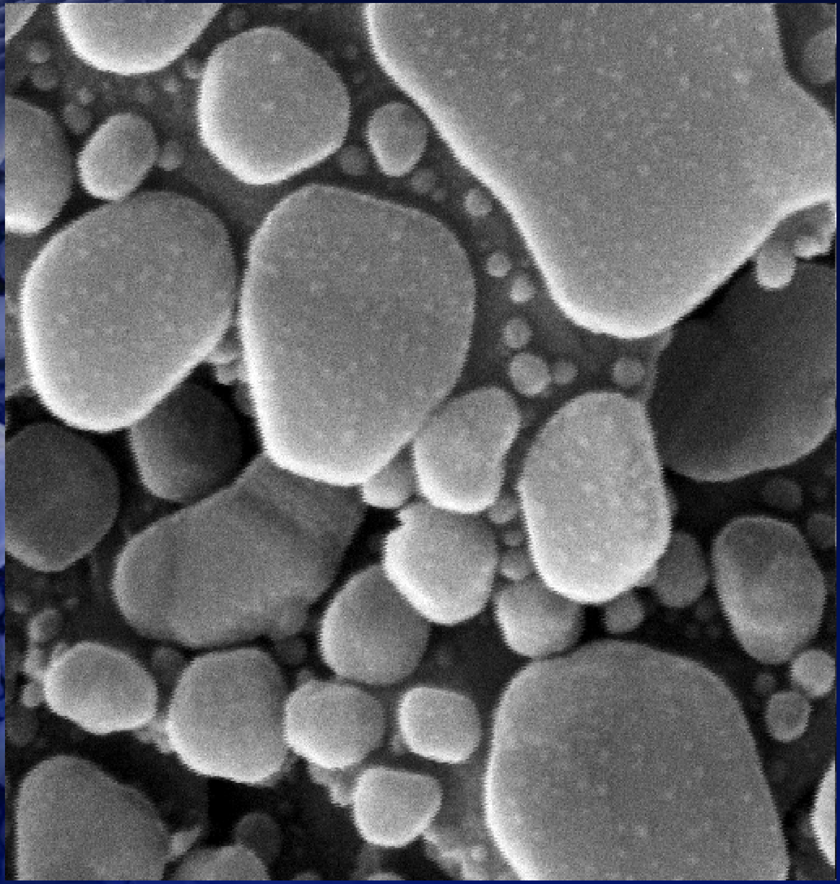
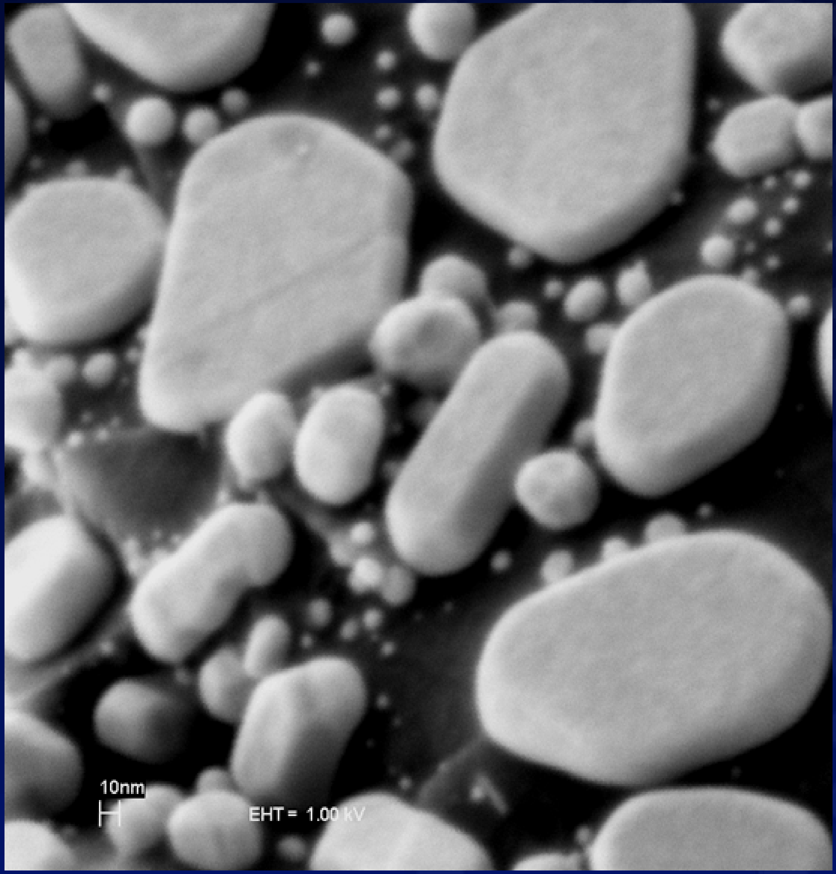


Image Comparison



HIM

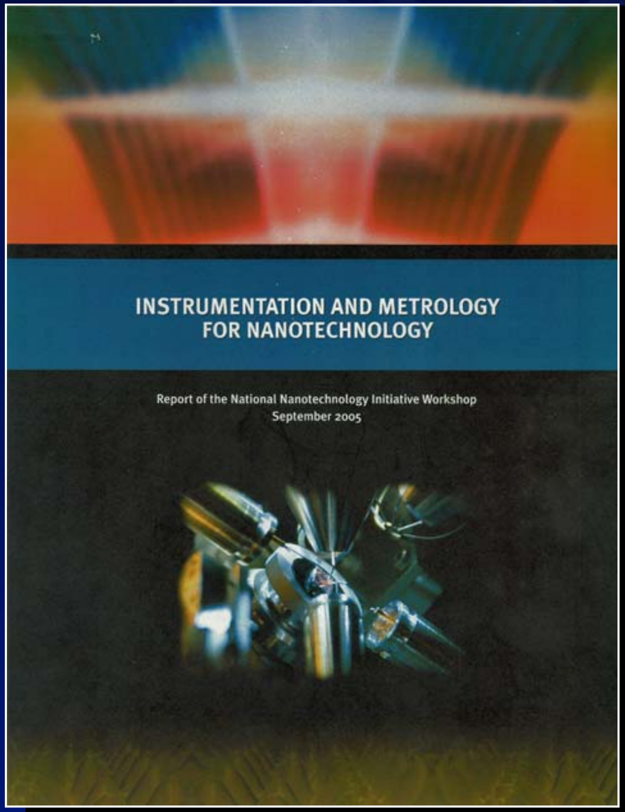


SEM

Gold-on-carbon sample. 1 μm field-of-view images, 1 pA beam current at 30 keV (HIM) and 20 pA beam current at 1 keV (SEM) landing energies.

Grand Challenge Workshop:
Instrumentation and Metrology for Nanotechnology

- The NNI Interagency Workshop on Instrumentation and Metrology for Nanotechnology Grand Challenge Workshop hosted at the National Institute of Standards and Technology campus in Gaithersburg, Maryland
- Composition: ~1/3 Industry, 1/3 Academia. 1/3 Government
- Over 250 attendees
- Report is completed and available
  - www.nano.gov



MEL
Innovation & productivity

IWG Workshop:

Instrumentation, Metrology, and Standards for Nanomanufacturing

- **Workshop of the National Science and Technology Council (NSTC) Interagency Working Group (IWG) on Manufacturing Research and Development (R&D)**

Sponsored by:

- **The National Institute of Standards and Technology (NIST), the National Science Foundation (NSF) and the Office of Naval Research (ONR)**

http://www.mel.nist.gov/nano.htm

NIST and Nanotechnology

- **Across NIST there were approximately 120 nanotechnology related projects that reported notable accomplishments for FY 2004-2005.**
  - **Printed report is available**
    - **CD**
    - **Hardcopy**

Nanoelectronics Metrology

- Issues for the future:
  - Improved signal-to-noise ratio
  - Reduced or no sample damage
  - Elimination of surface contamination or alteration
  - Sample preparation problems
    - Serious complication in every single possible measurement method currently in use.
- All these issues present formidable but, very exciting challenges.
- Some solutions are already known, but for others the work has just begun
  - Leading to even more revolutionary metrology and characterization instrumentation
  - This is not a cheap process – but the rewards are great

Acknowledgements

- The presenters thank and acknowledge:
  - Office of Microelectronics Programs of NIST for partial funding of ongoing work in nanoelectronics metrology
  - Veeco, Hitachi, FEI, and Zeiss for providing images and instrument information
  - John Small, Andras Vladar, Ron Dixson for providing slides
  - Charles Cheung, Curt Richter, Erik Secula, and many other NIST staff members