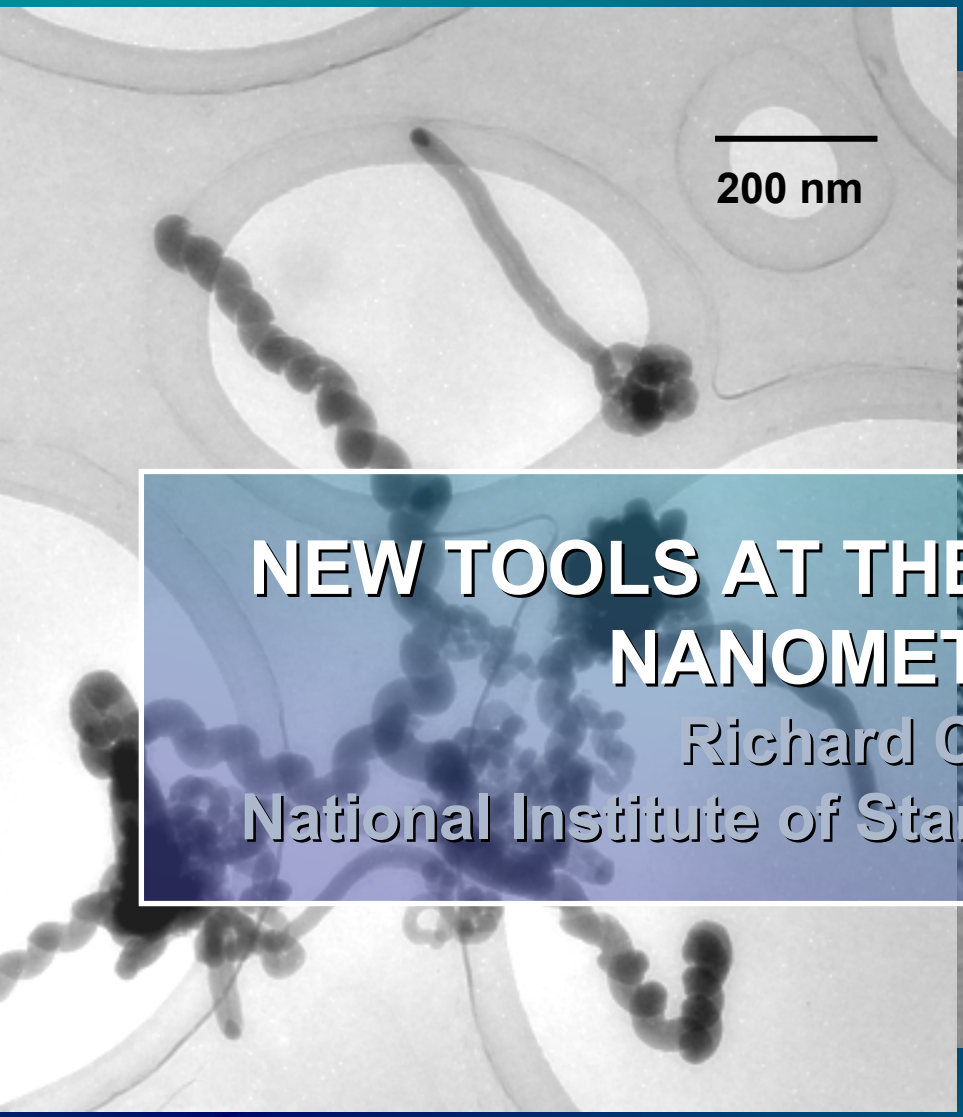
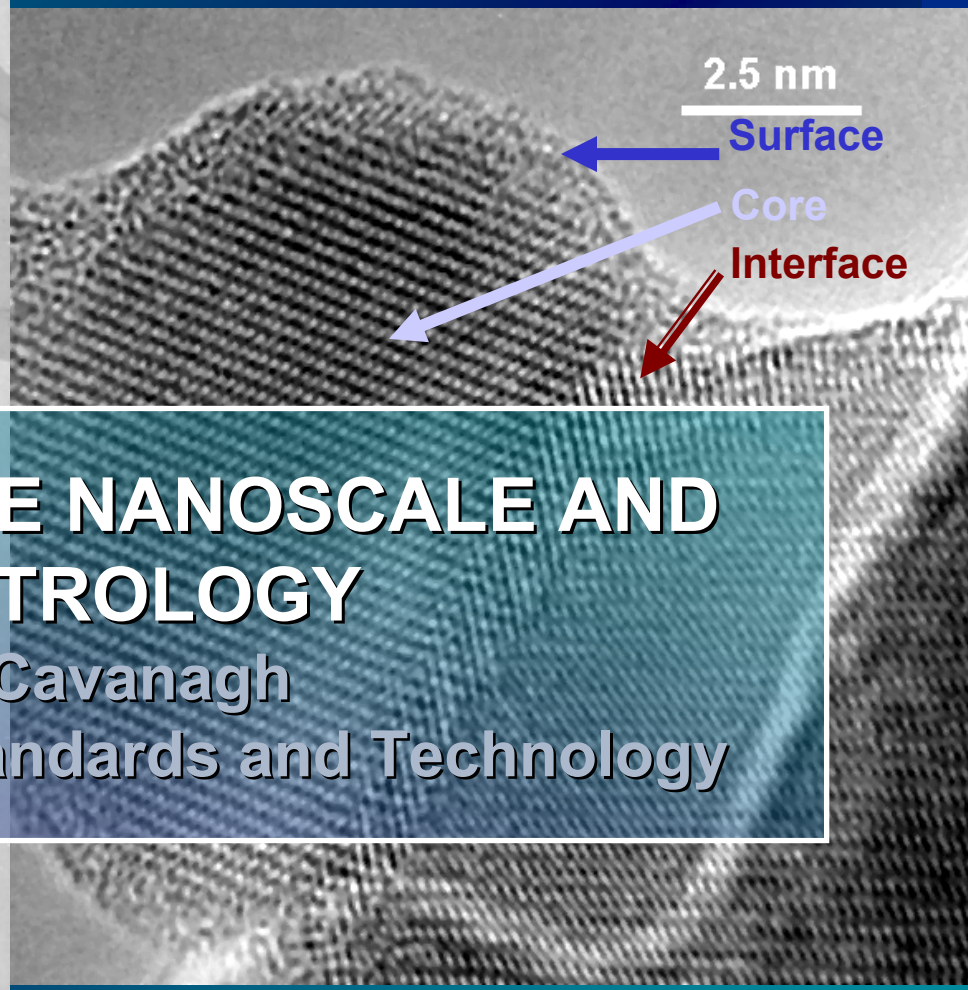




NEW TOOLS AT THE NANOSCALE AND NANOMETROLOGY

Richard Cavanagh
National Institute of Standards and Technology

Carbon Nanotubes courtesy of Richard Cavicchi and Shirley Turner, NIST

Yttria particles courtesy Nanophase Technologies Corporation. Image courtesy of John Henry Scott, NIST

Major Facilities and Select Metrology Tools

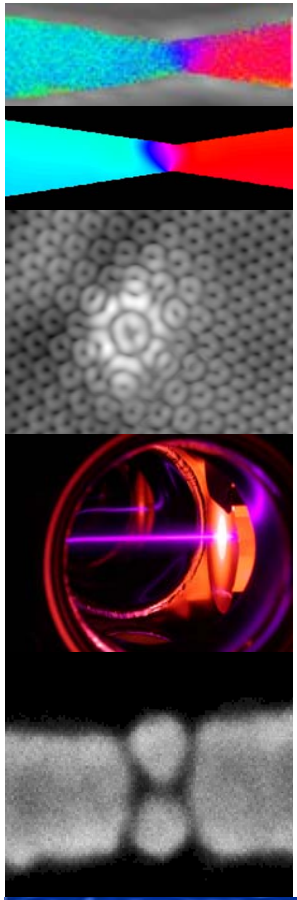
- Facilities
  - CNST
  - NCNR
- NanoMetrology Tools
  - Electron Microscopy
  - Helium Ion Microscopy
  - Super Resolution Optical Microscopy
  - Standards for the Nanoscale

CNST – NIST's Center for Nanoscale Science and Technology

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

- Provides **measurement methods**, standards and technology to support all phases of nanotechnology development from discovery to production,
- develops and maintains a national shared use facility, **the Nanofab**, with state-of-the-art, nanoscale measurement and fabrication capabilities in Gaithersburg, MD
- applies a **multidisciplinary** approach to problem solving that involves partnering with industry, academia, and other government agencies, and
- helps to **educate** the next generation of nanotechnologist.

Research Program

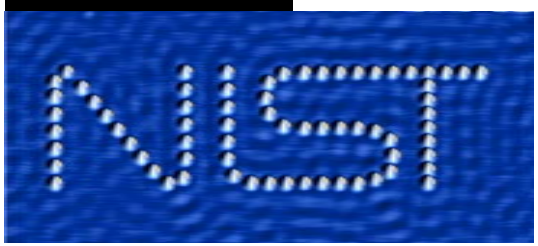


Developing measurement capabilities for:

- **Future Electronics**
  - Devices, architectures, interconnects
- **Nanomanufacturing and Nanofabrication**
  - Top-down and bottom-up fabrication and assembly
- **Energy**
  - Conversion, storage, and transport

Initial areas of expertise:

- Nanofabrication, nanophotonics, nanoplasmonics, transport, modeling and simulation, force microscopy, tunneling microscopy, nanomagnetics, optical nanomanipulation, nanofluidics

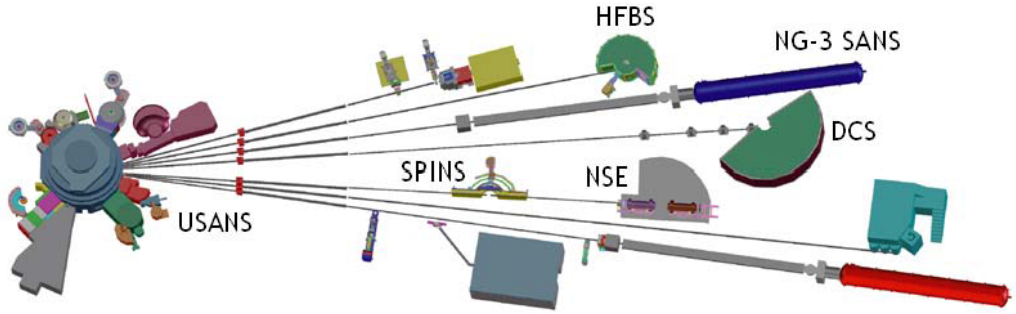


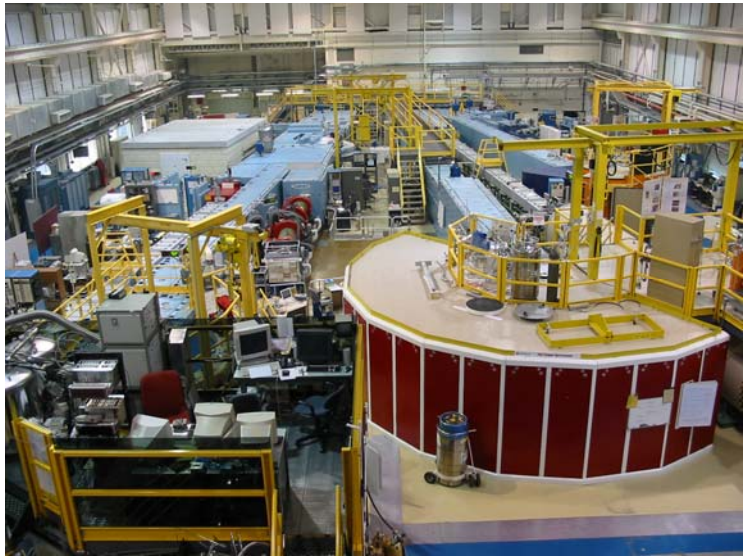
CNST's Nanofab

- A national, state-of-the-art, shared use facility for the fabrication and measurement of nanostructures
  - Measurement and fabrication equipment located both within and outside of cleanroom
  - 19,000 sq ft cleanroom (8,000 sq ft class 100)
  - Advanced lithography and microscopy
  - Experienced staff to train users or operate the tools
  - Open to all users
  - Shared use - based on NSF model, e.g. similar to Cornell Nanoscale Facility

NIST Center for Neutron Research

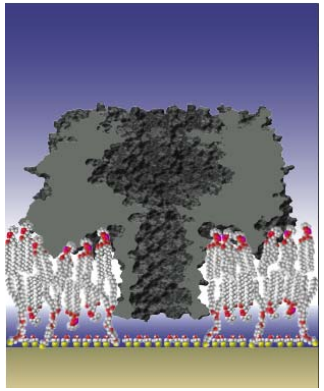
NCNR

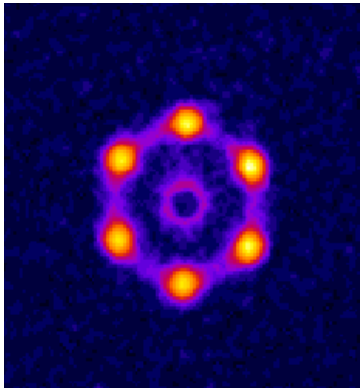


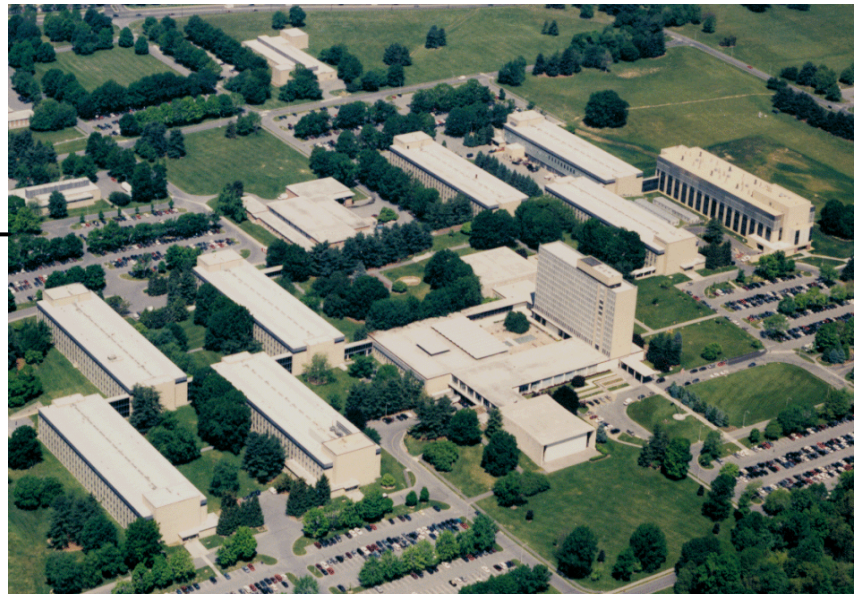


- User facility serving researchers from industry, academia, and government with calls for proposals twice/year
- www.ncnr.nist.gov
- Exploration of nanoscale structure and dynamics in diverse systems such as soft matter and magnetic systems

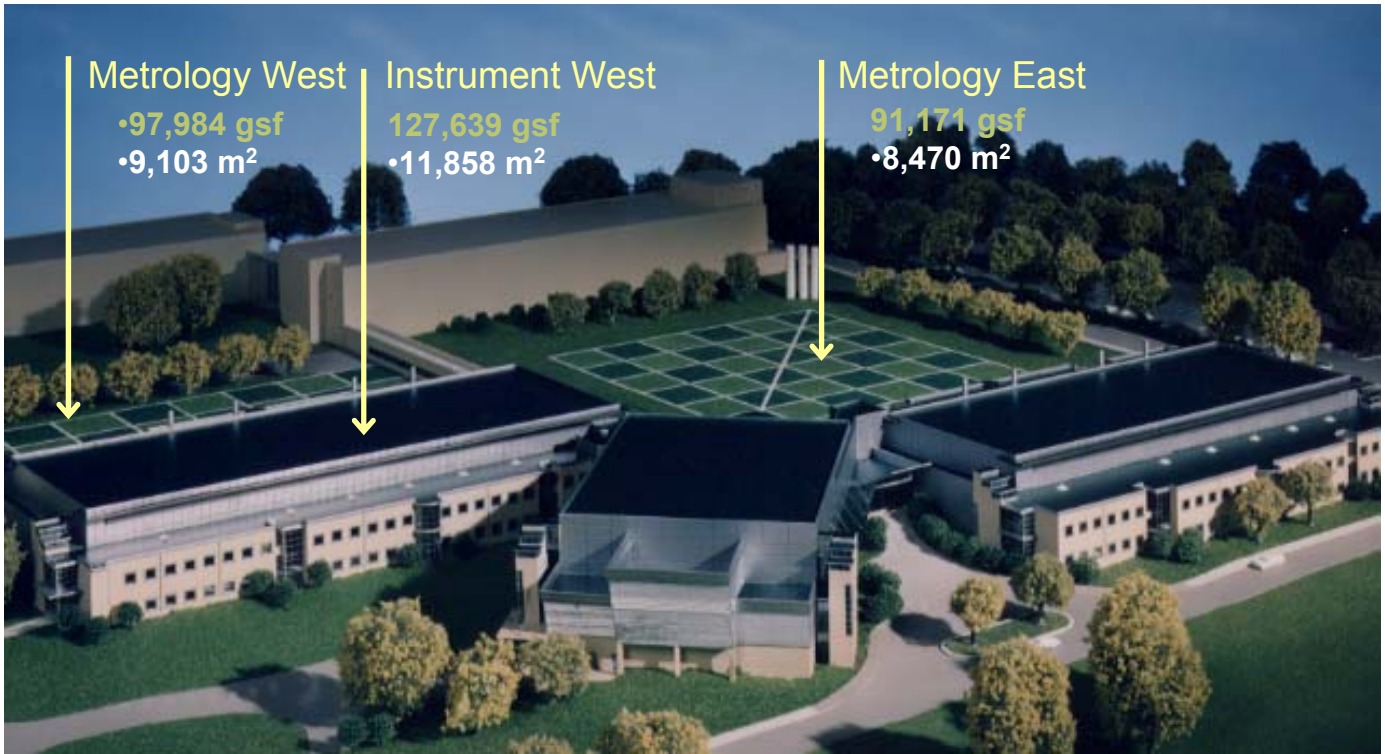
NSF sponsors CHRNS at the NCNR, the Center for High Resolution Neutron Scattering, serving the most users of any neutron facility in North America







Chemistry
Physics
Materials
Electronics
Manufacturing



NIST AML Critical Criteria

	Current NIST Facilities	NIST AML Criteria	Nano Benefit
Temperature Control +/- 0.01 Degrees Celsius	+/- 2	+/- 0.01 to 0.25	Thermal expansion
Humidity Control +/- 1 %	+/- 20%	+/- 1% to 5%	Stable refractive index
Air Quality Class 100 - M3.5	2-4 million	100 to 1000	Sample contamination
Vibration Control 0.2 - 3 Micrometers/second	3 to 20	0.2 to 3	Stable optical columns Stable specimen mounts

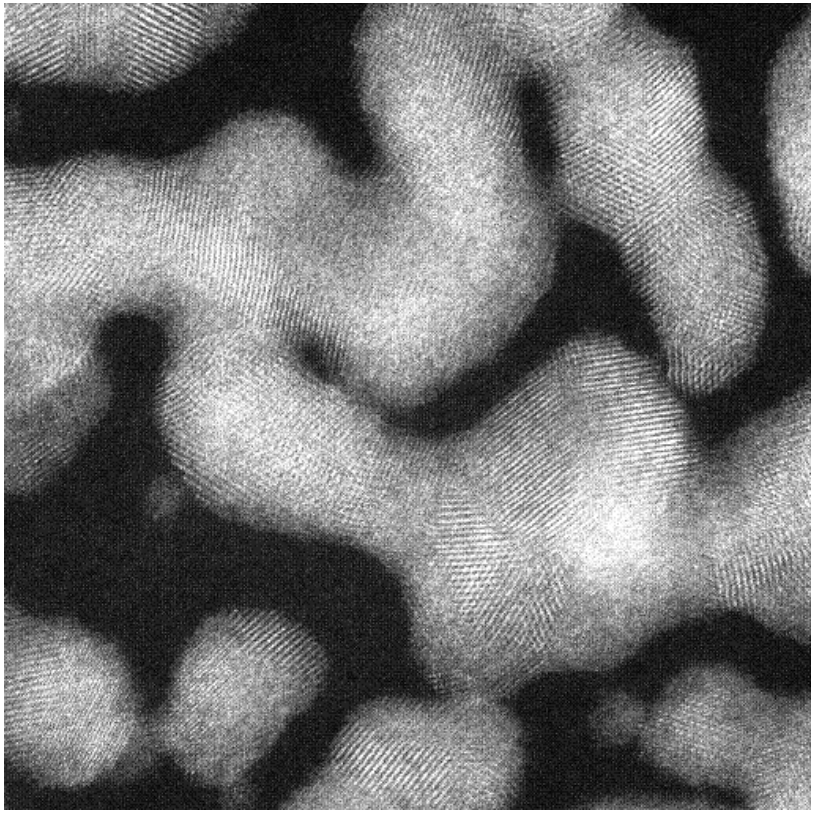
NanoMetrology

Aberration-corrected monochromated AEM

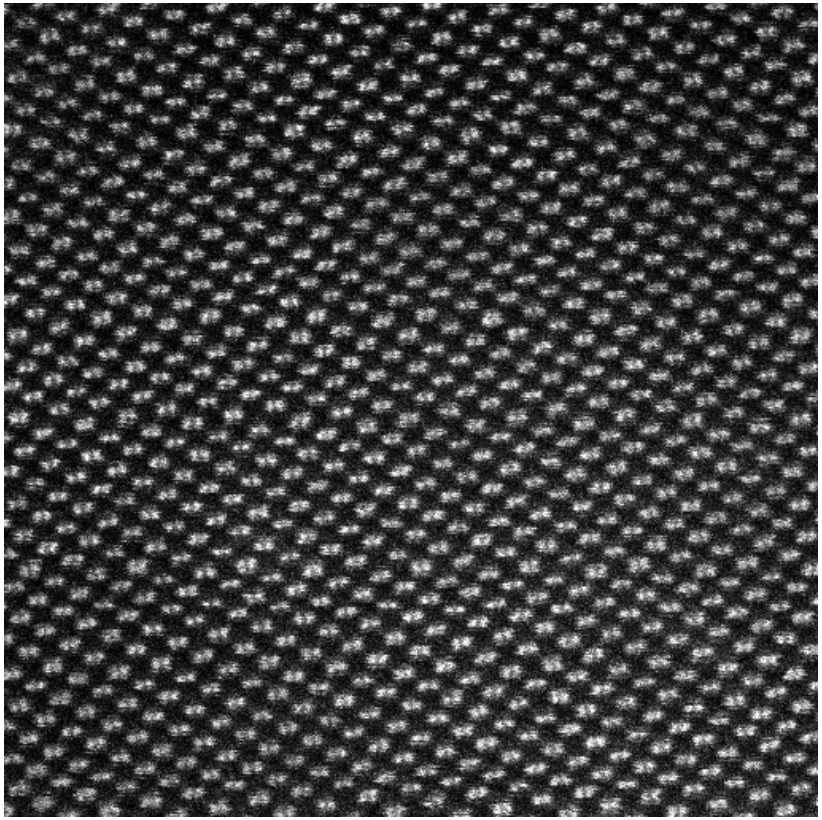
Delivered to NIST March 2006

- Proven capable of sub-Angstrom imaging in both TEM and STEM modes
- Demonstrated information limit of less than 0.09 nm (0.07 nm anticipated) will allow <0.1 nm imaging in TEM mode with through-focal series reconstruction
- Monochromator to provide < 0.2 eV EELS spectral resolution with < 0.2 nm probe
- Planned technique development includes 3D spectroscopic identification of coordinates and elemental identities of all atoms comprising a small (~100-1000 atom) nanostructure

NIST Titan Acceptance Tests



5.6 nm



3 nm

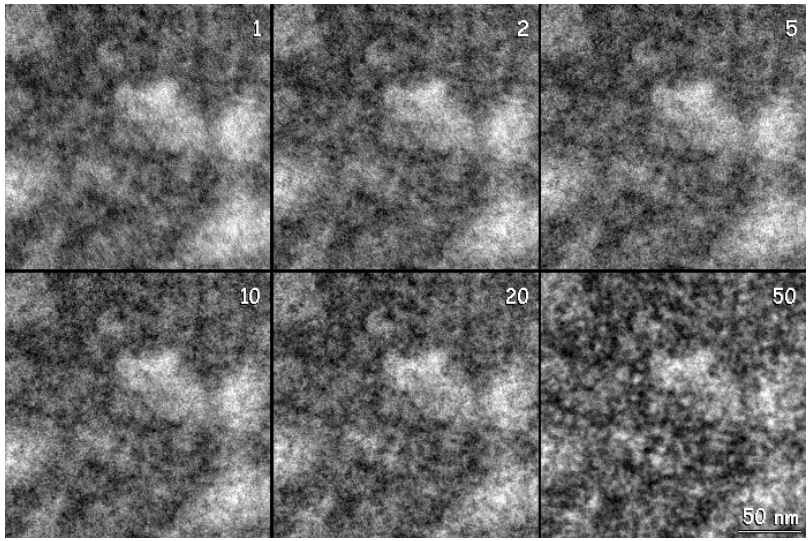
- 80 keV to 300 keV
- < 0.10 nm probe
- ~100 meV energy spread
- HRTEM information limit < 0.1 nm

0.136 nm Si “dumbbells” as seen in STEM

NCI/NCL

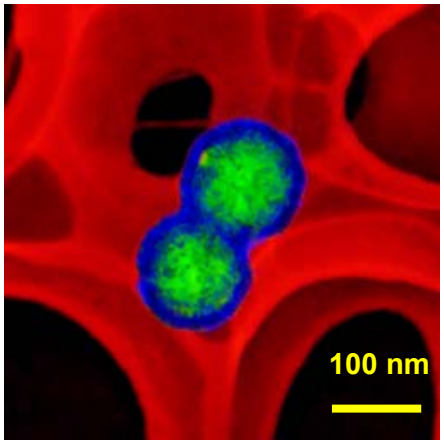
- Division 837 - working with the NCL program since 2005.
- Work has involved primarily TEM & AEM investigations of Dendrimers and Gold Particles
- Additional investigations have been done with FEGSEM, Scanning Auger Microscopy and TOF SIMS to determine surface functionalization.

A series of images showing the effects of increased electron dose on Dendrimer material. Upper left image represents the first exposure or lowest dose. This series of images show increased contrast, but alteration of structure.

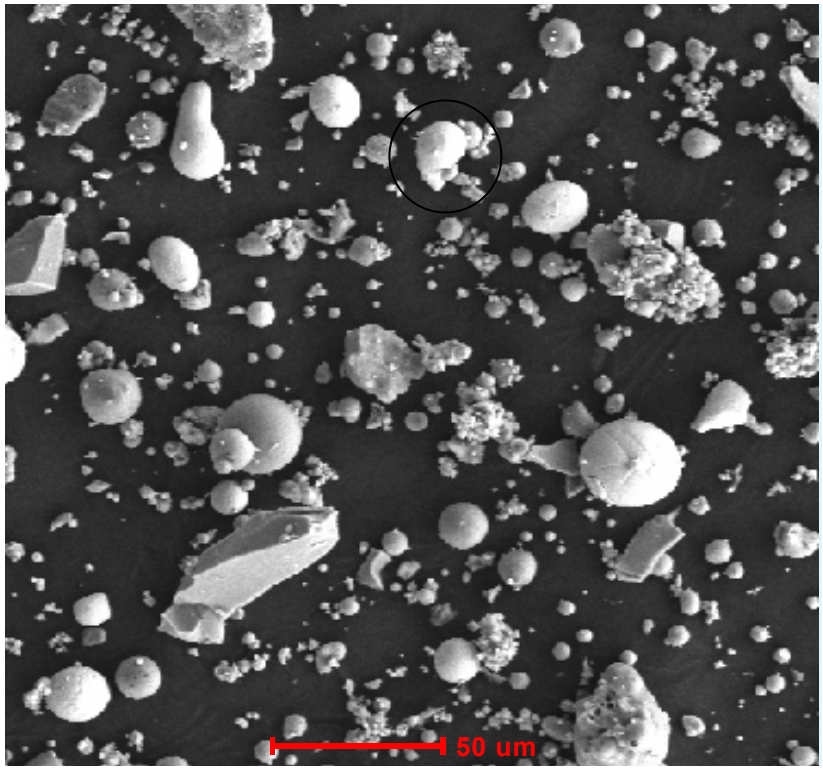


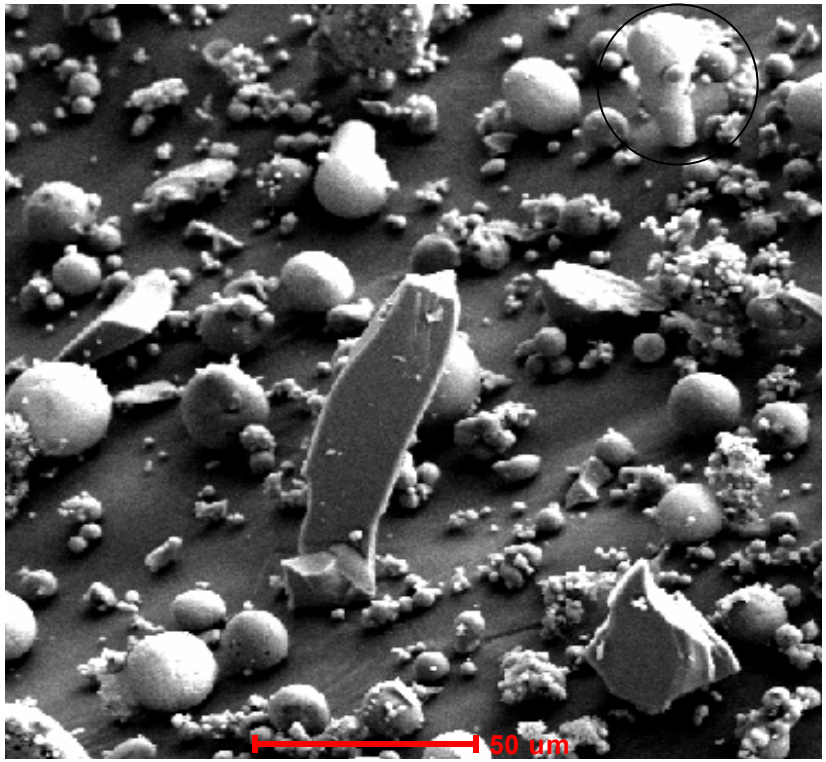
AEM analysis of sample for particle size, agglomeration, and coating continuity.

- Diameter of 138 ± 52 nm (2σ / 95th percentile)
- Nanoparticle shape is round, with few exceptions
- Thickness of coating is uniformly 13 to 14 nm, where intact
- Holes in coating of ≈10 nm diameter observed in most particles



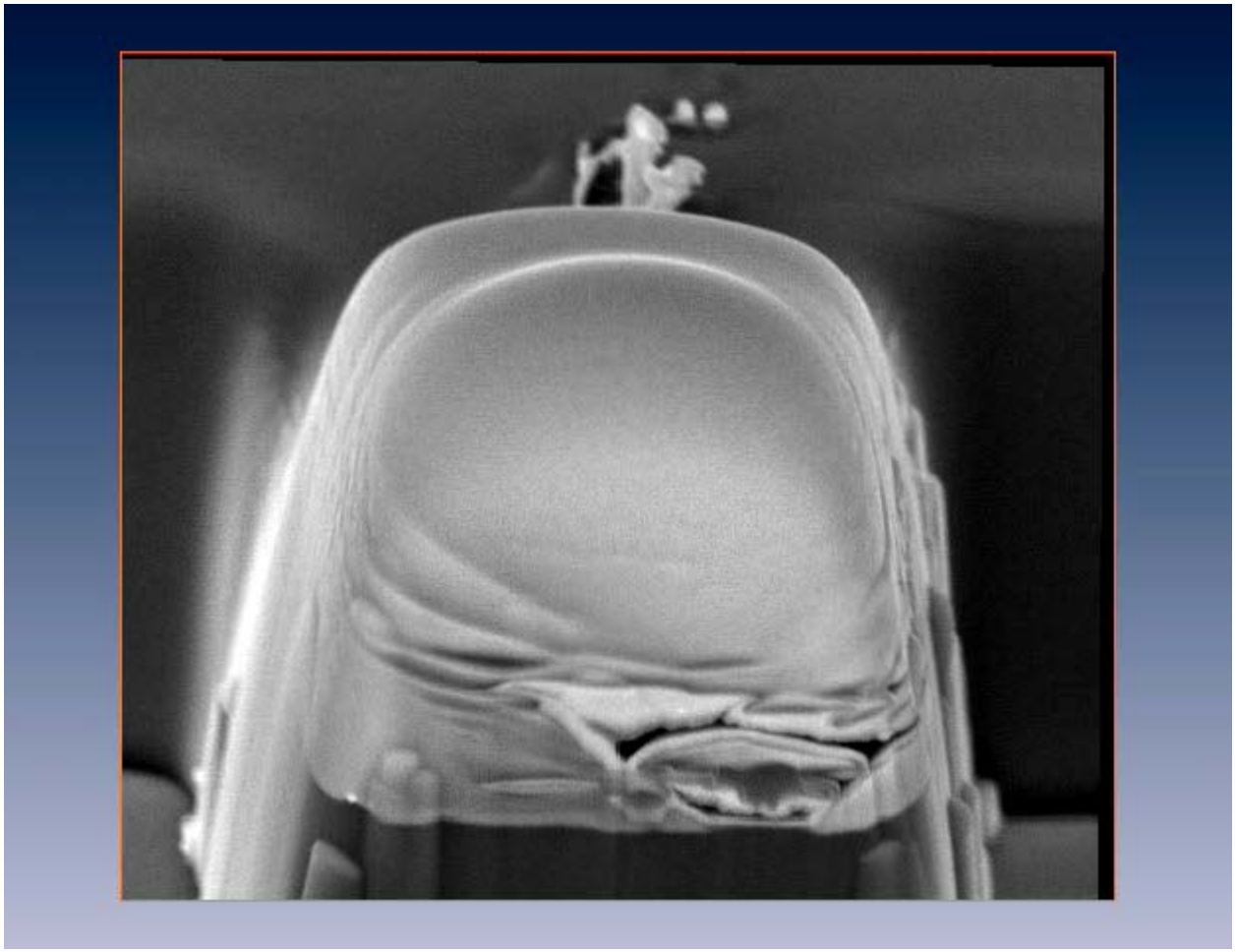
What about the third dimension?



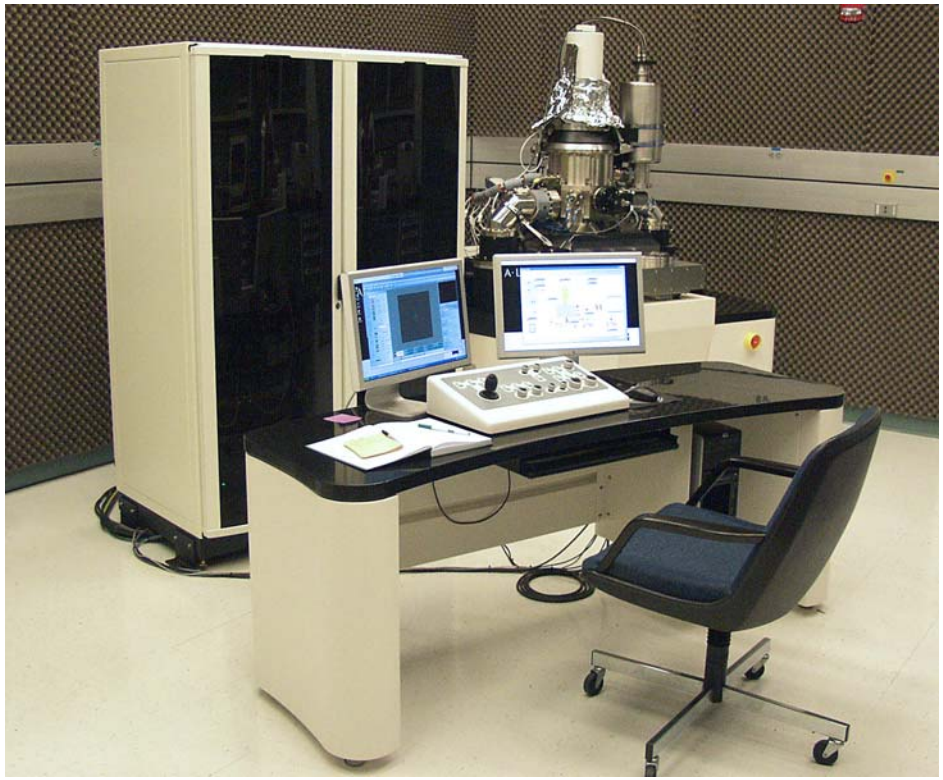


Slice and View Movie

unattended milling and SEM imaging 200 slices 30 nm/slice in Z direction



Helium Ion Microscopy:
A technology for nanometrology



Helium Ion Microscope (HIM)

- First of this new type of instrument has recently been installed at NIST
- Helium ions (He^+) are generated and used to irradiate the sample
- HIM resolution is theoretically 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 (and others) expertise to the study of the understanding of the physics of this instrument will facilitate more accurate measurements and standards development.

Stable Helium Ion Emission – Key Element

- The ion source is cryogenically cooled with liquid N_2 in a low-pressure helium gas environment.
- Helium ionization takes place at the atomically sharp tip.
- The arriving helium atoms adhere everywhere to the tip
- Only at the very tip of the pyramid is the electric field high enough for the atoms to ionize and fly away.
- Emitter control and manipulation literally is at the level of a *single* atom.

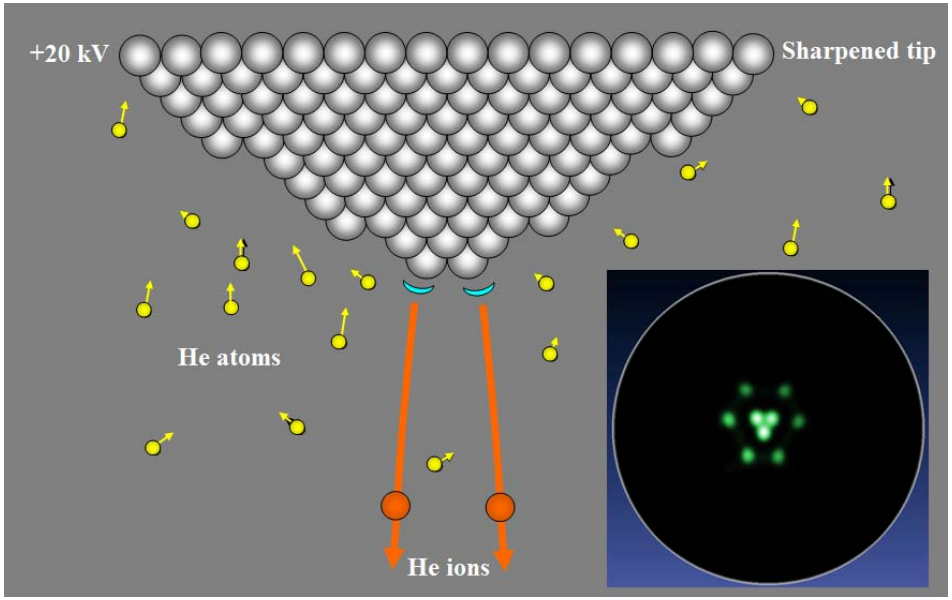
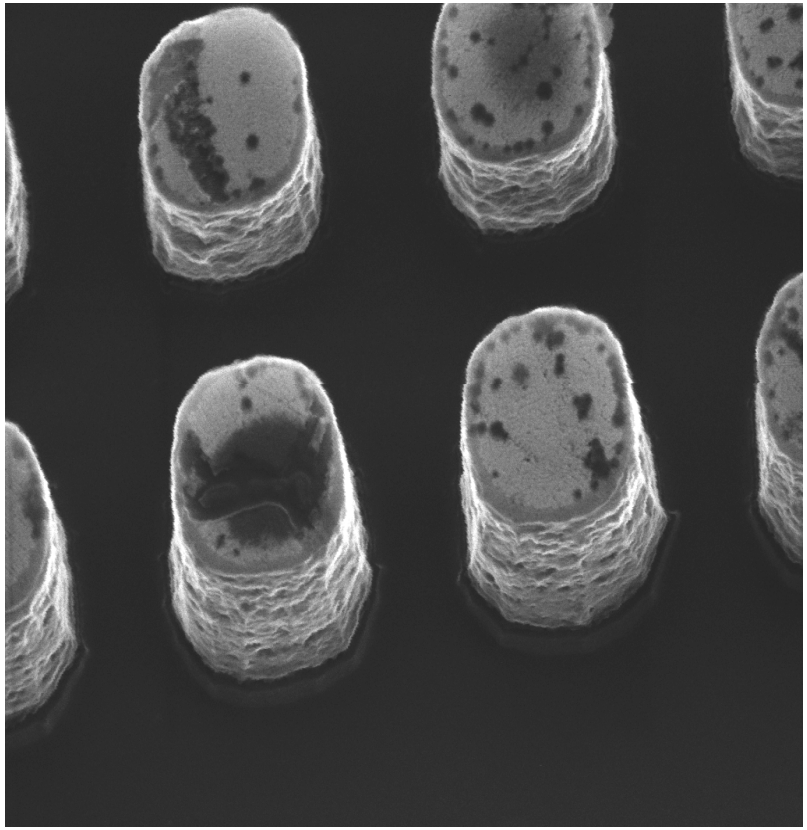
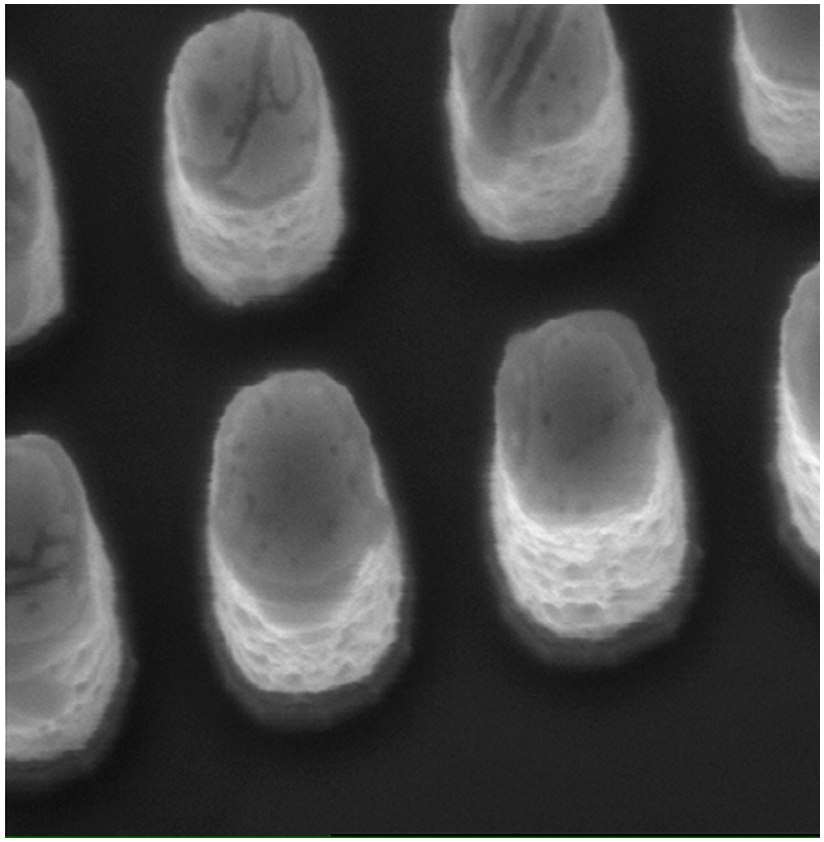


Image Comparison



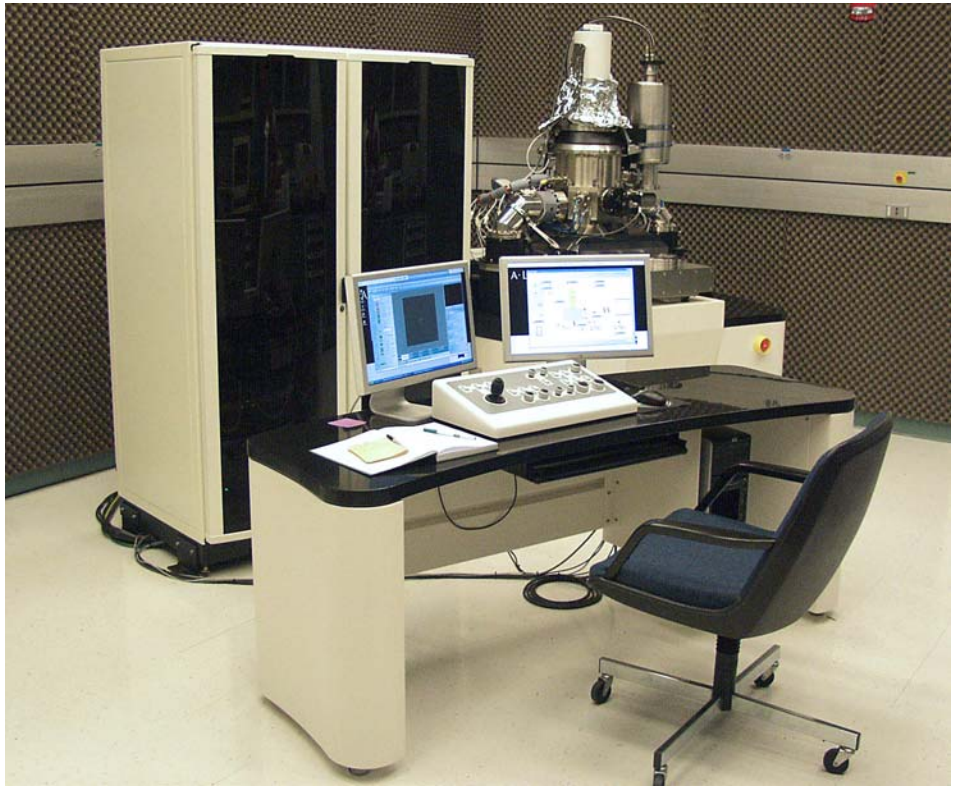


HIM **SEM**

Aluminum posts on silicon sample.
3 µm field-of-view images.

HIM vs SEM

- SEM and HIM
  - Have some overlapping territory
  - Remain complimentary
- HIM is forging new scientific territory for nanotechnology and nanomanufacturing

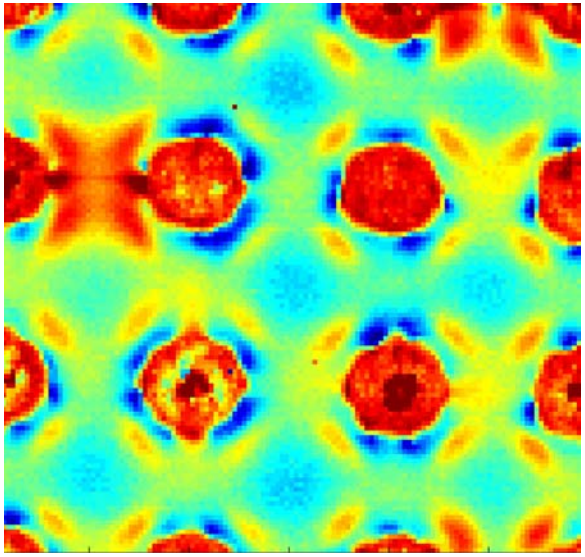


- HIM - an emerging technology, which once optimized will develop new science and contribute to the progress in nanotechnology

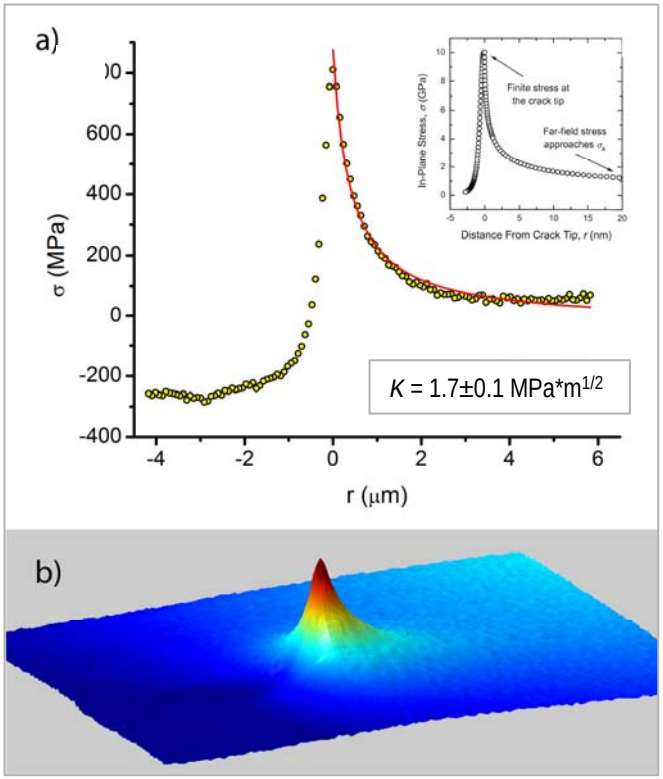
Nanoscale Materials Analysis: Toughness

Key Collaborations: Ford Make Every Day Exciting

Approach: Use SotA Raman microscopy to probe nanoscale mechanical properties (stress and toughness) that ultimately determine product reliability.



$$\sigma(r) = \frac{K}{(2\pi r)^{\frac{1}{2}}}$$

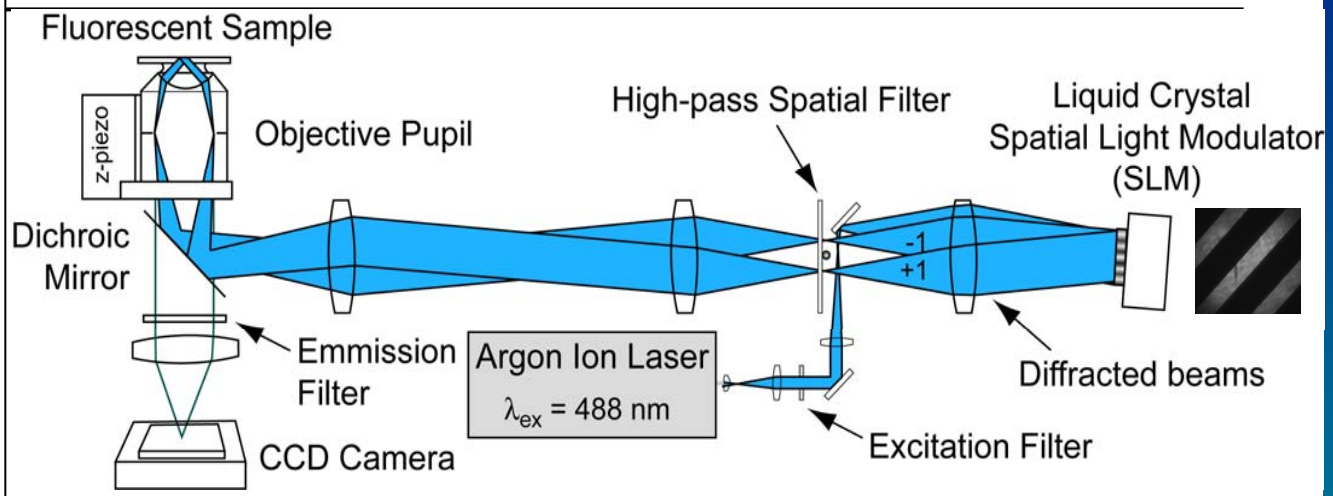


- **ABOVE: 30 mm field of view of a series of nano-indentations in Si result in a complex stress field – spanning 7 cm^{-1} Raman shift.**
- **RIGHT: Analysis of a fracture feature in Si can be used to determine the materials toughness or suitability for a given application, e.g. MEMS or NEMS.**

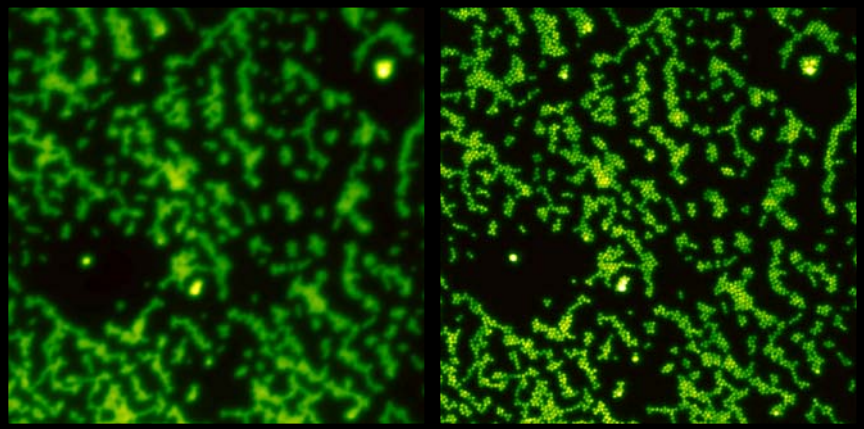
Super Resolution Microscopy

The structured illumination microscope is based on a epi-fluorescence microscope.

A Spatial Light Modulator projects a fine pitched interference pattern onto the sample. This pattern mixes and down converts the spatial frequencies into the microscopes bandwidth

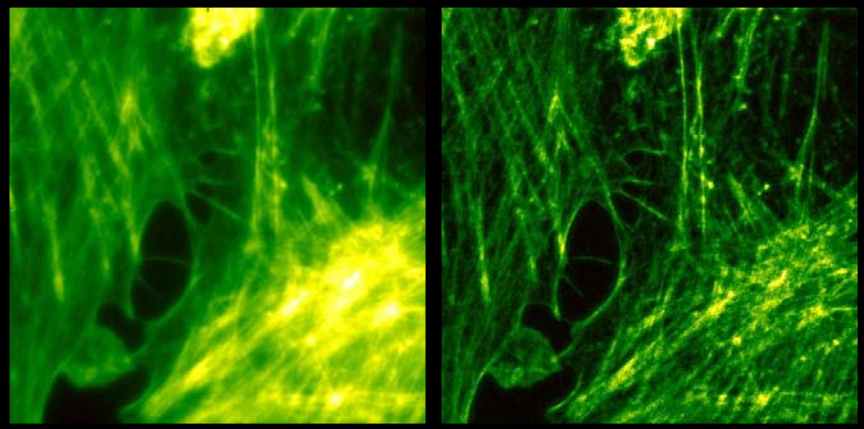


Conventional | Super Resolution



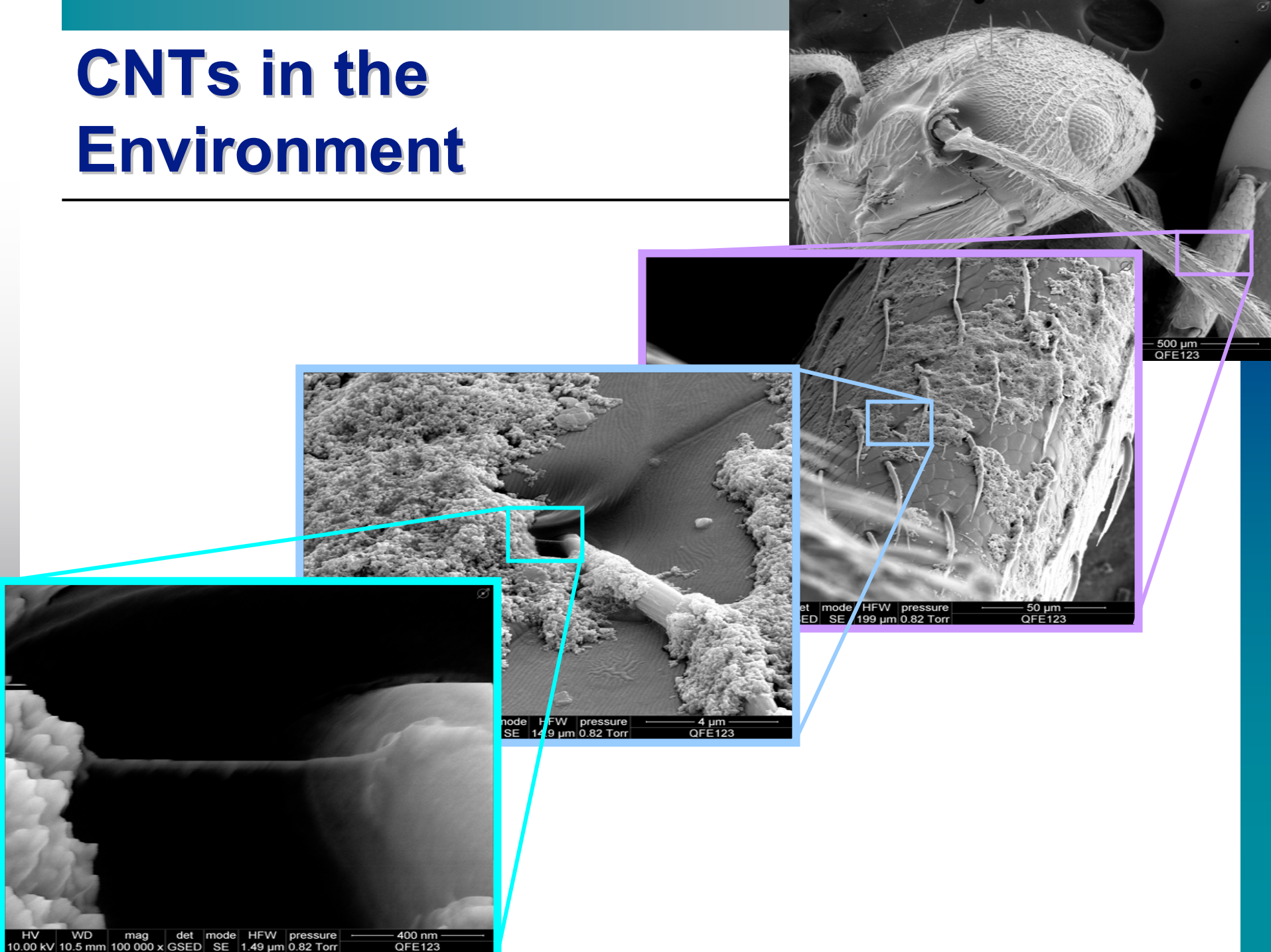
100 nm fluorescent beads

Conventional | Super Resolution



Bovine pulmonary artery endothelial cells.

CNTs in the Environment



Molecular Scale Imaging of BioSystems

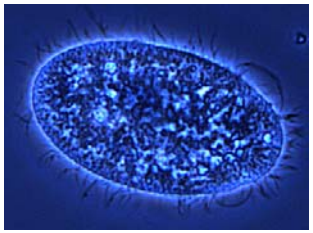
- Demonstrated that nanomaterials (surface functionalized quantum dots) can be bioconcentrated and biomagnified in a simplified food web under laboratory conditions.*

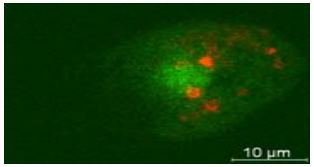
- One of the first studies to demonstrate that engineered nanomaterials discharged into the environment may enter and remain in the food chain.

QD

H_2O

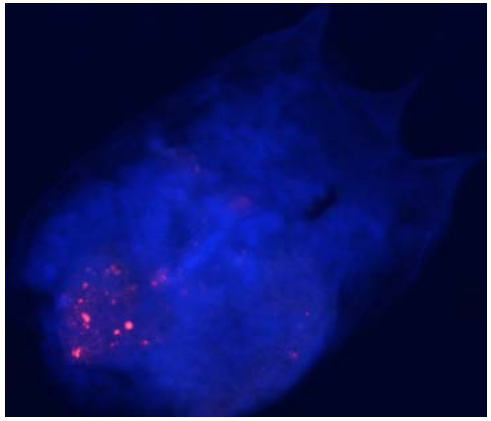
Tetra

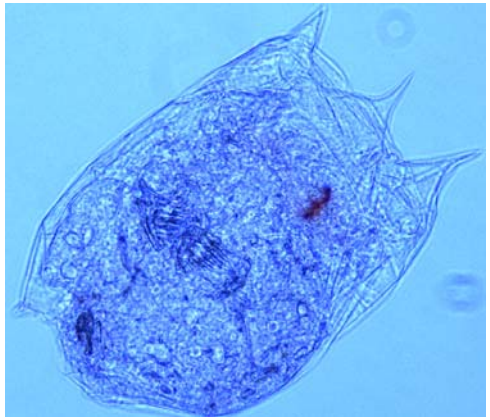




Length = 40 – 60 µm
Width = 20 – 30 µm

Rotifer





Length = 180 – 300 µm

***Mechanisms of Toxicity, Nanomaterials: Environmental Fate, Effects, LCA, and Risk Assessment, Presented at SETAC Europe 2007, Porto, Portugal May 24, 2007**

Workshop on Standards for EHS Research Needs for Engineered Nanoscale Materials

September 12 –13, 2007

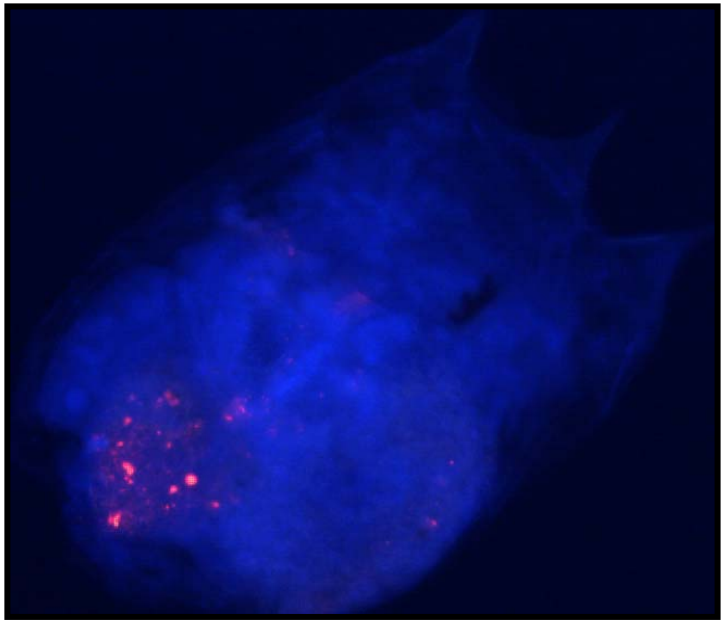


Image courtesy of David Holbrook, NIST

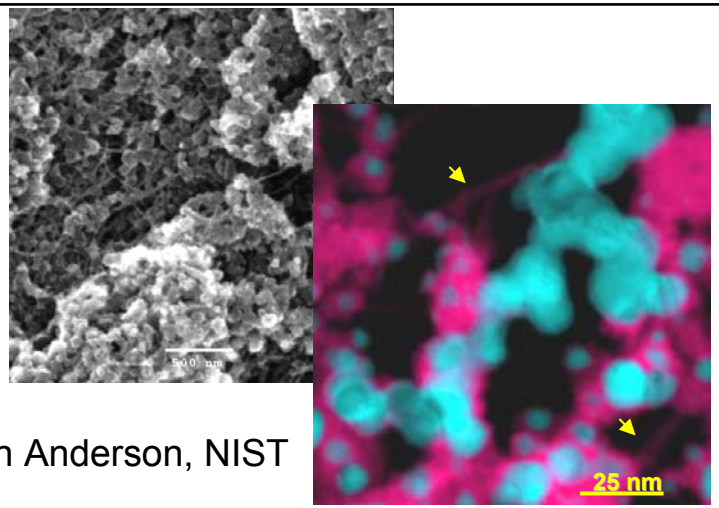
Sponsored by National Nanotechnology Initiative
Hosted by NIST

www.cstl.nist.gov/div837/Division/meetings/NanoWorkshop/ENMworkshop.htm

NIST & Nanotechnology:

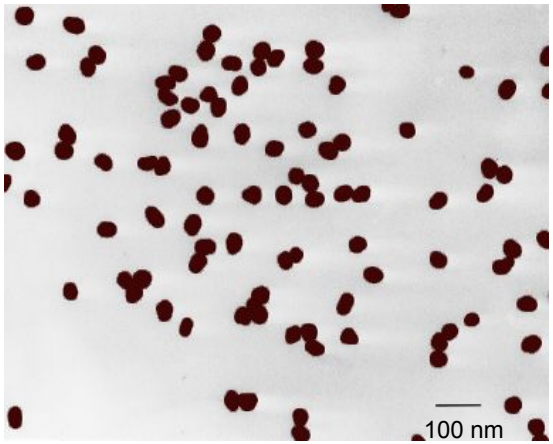
Standards On the Horizon

- Article of commerce Proposed RM 8475 CNT:
- RM 8281 SWNT long and short length in solution.



Ian Anderson, NIST

- Au nanoparticle RM 8011-8013 in conjunction with NIH/NCL: 10 nm, 30 nm, and 60 nm mean particle size.



www.huck.psu.edu

Nano Metrology Efforts at NIST

- Facilities
  - Center for Nanoscale Science and Technology – Robert.Celotta@NIST.GOV
  - NIST Center for Neutron Research – Patrick.Gallagher@NIST.GOV
- NanoMetrology Tools and Standards
  - Electron Microscopy – John.Small@NIST.GOV
  - Helium Ion Microscopy – Michael.Postek@NIST.GOV
  - Super Resolution Optical – Stephan.Stranick@NIST.GOV
  - Ref Materials for EH&S Nano – Laurie.Locascio@NIST.GOV