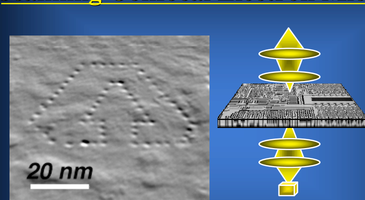
Scanning Confocal Electron Microscopy

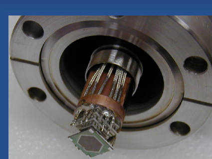


Nestor J. Zaluzec

Office of Science - BES Program
US Department of Energy

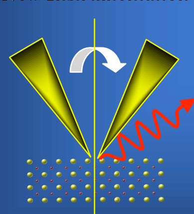
Electron Microscopy Center
Materials Science,Division, Argonne National Laboratory

The Evolving Frontiers of Materials Characterization

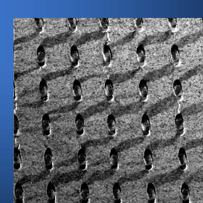


New Instrumentation

TelePresence Collaboratories
Scientific Workplaces of the Future



New Methodologies
For Characterization
at the "Bleeding Edge"



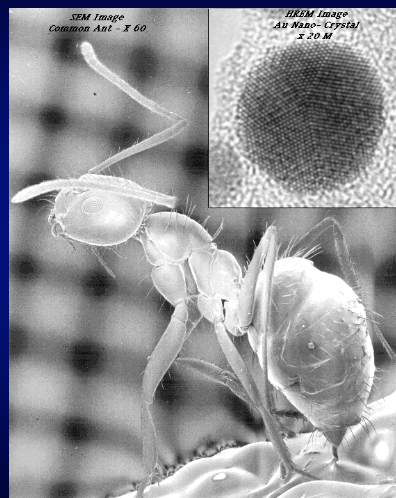
Materials Science/Physics

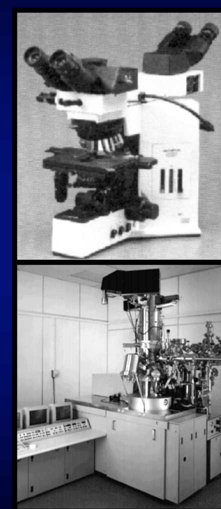
From Ants to Atoms

Microscopy is one of the few methodologies applied to nearly every field of science and technology in use today.

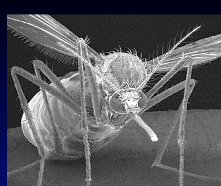
A microscope can be as simple as a hand held optical device or as complex as a multi-million dollar research tool.

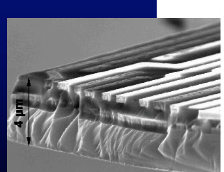
Using these instruments, both scientists and students can explore the synergistic relationships of structure and properties of a wide variety materials in both the Physical and the Life Sciences.

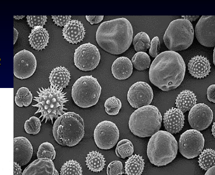


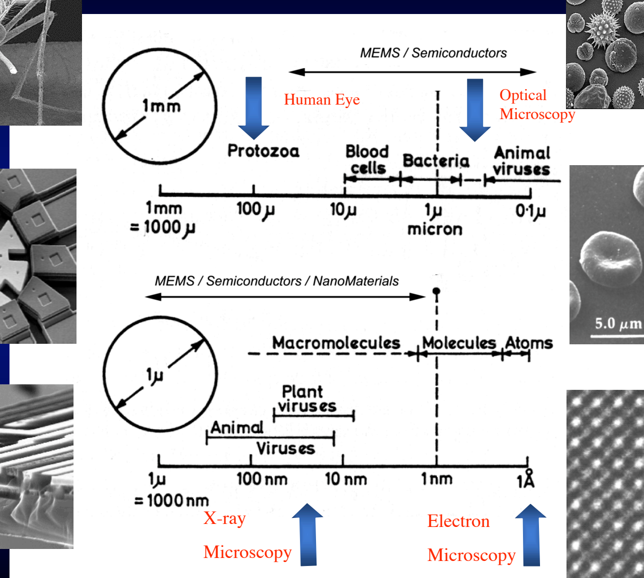


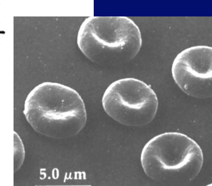
From Ants to Atoms
Microscopy is needed nearly everywhere

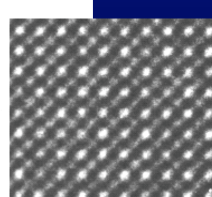


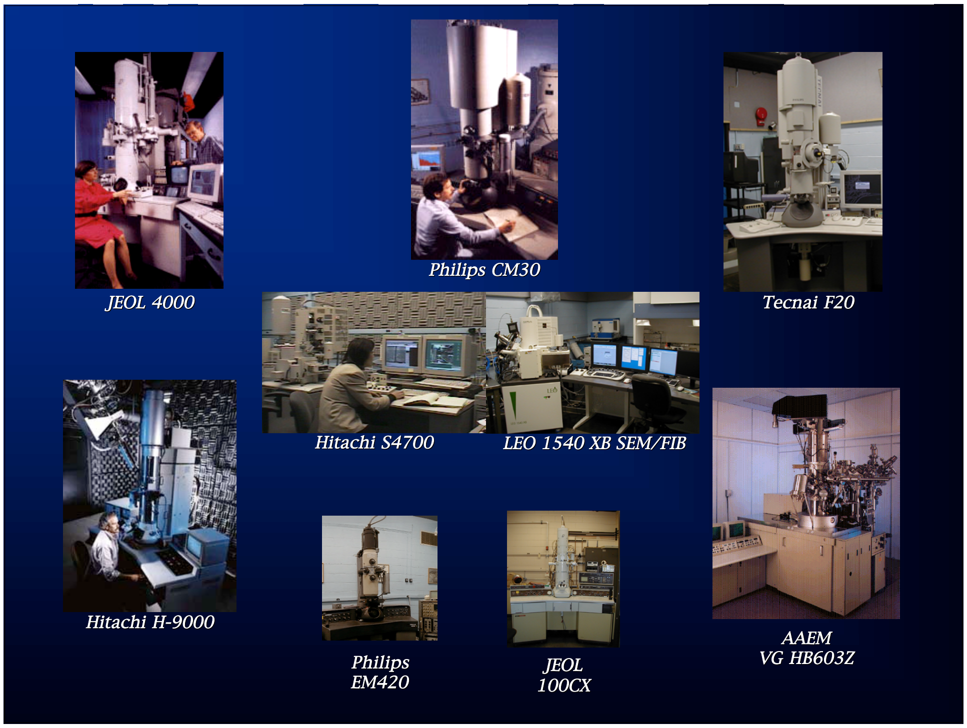




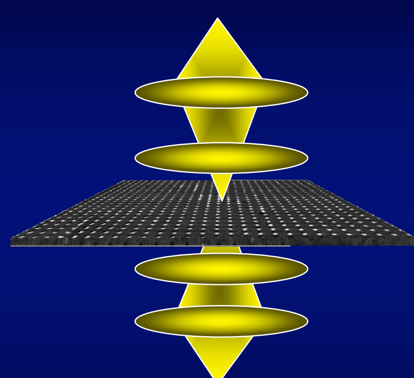






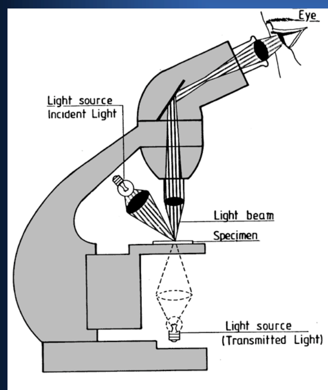


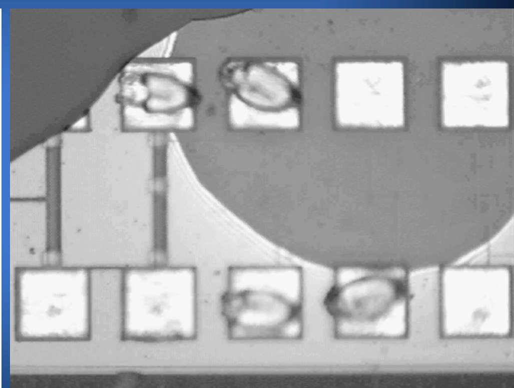
Scanning (Electron) Probe Microscopy
Deals Mainly with Near Surface Region



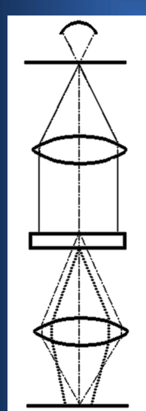
Transmission (Electron) Probe Microscopy
Deals Mainly with Internal Structure

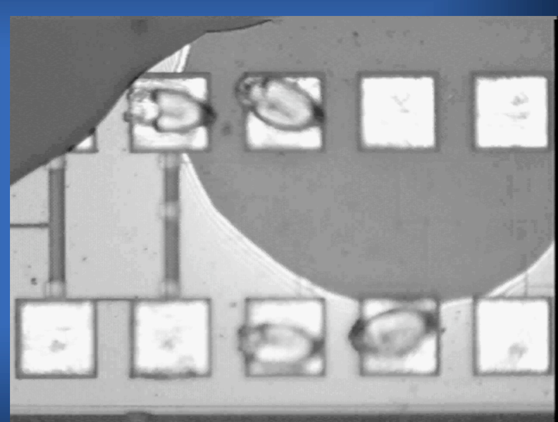
Optical Microscopy



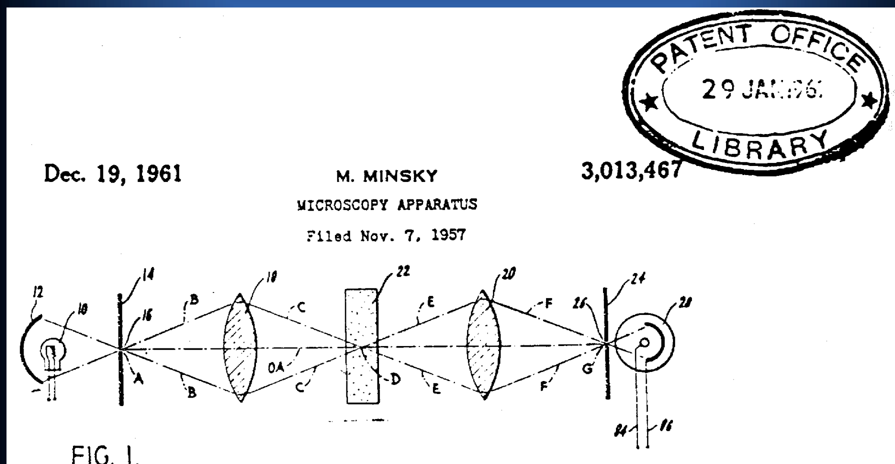


Optical Microscopy

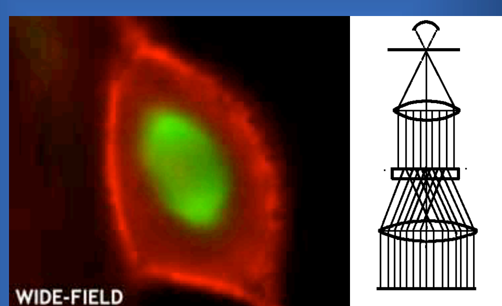




Scanning Confocal Optical Microscopy

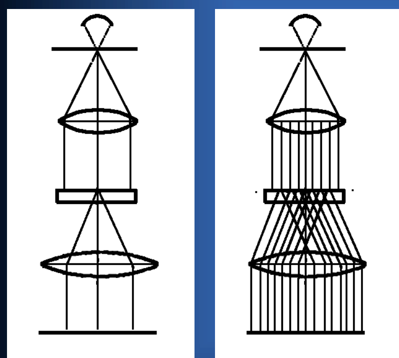


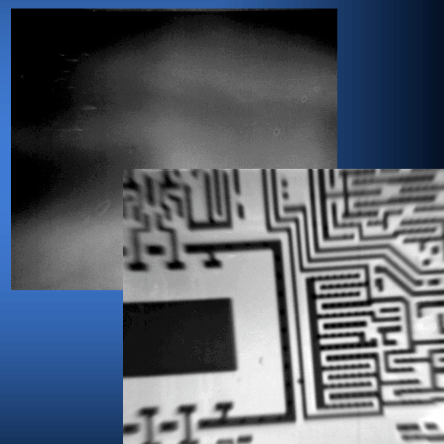
Optical Microscopy



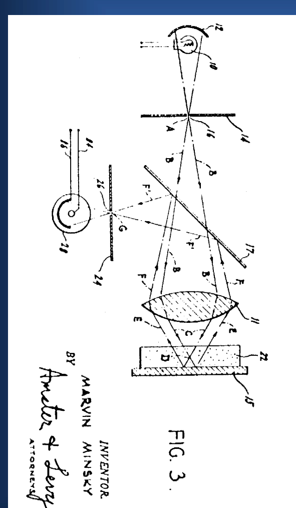
http://www.itg.uiuc.edu/technology/atlas/microscopy/confocal.htm

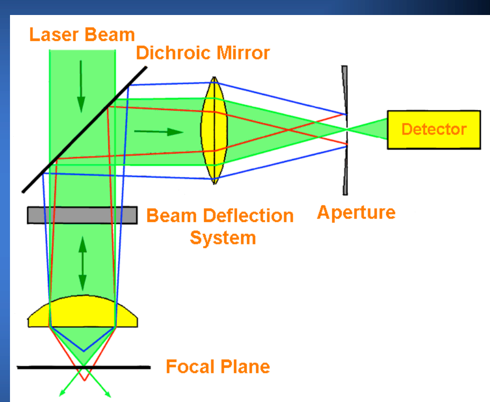
Transmission Microscopy



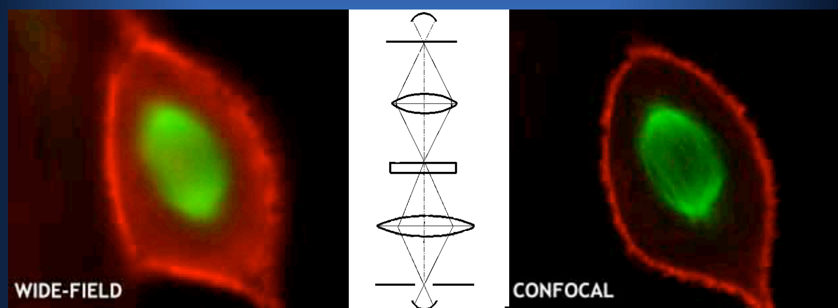


Scanning Confocal Optical Microscopy



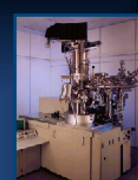


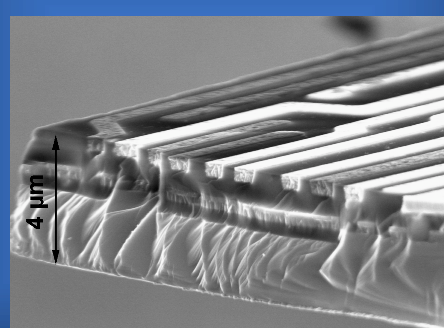
Scanning Confocal Optical Microscopy



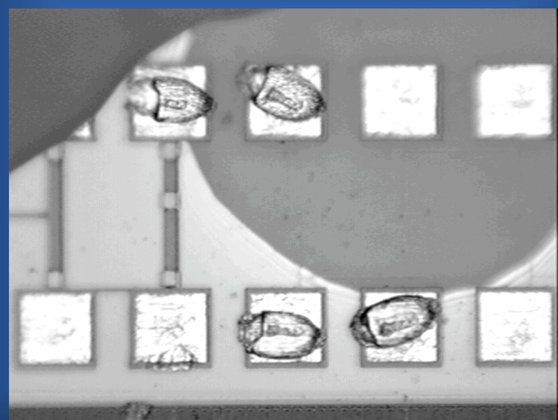
http://www.itg.uiuc.edu/technology/atlas/microscopy/confocal.htm

Scanning Confocal Microscopy
In Materials Research



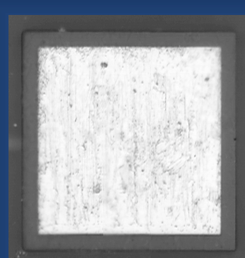


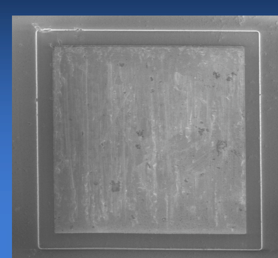
Enter the Challenge!



Comparison of Modes

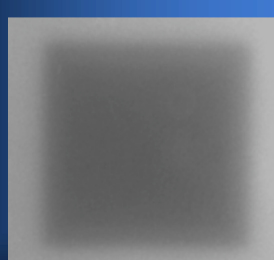
OM





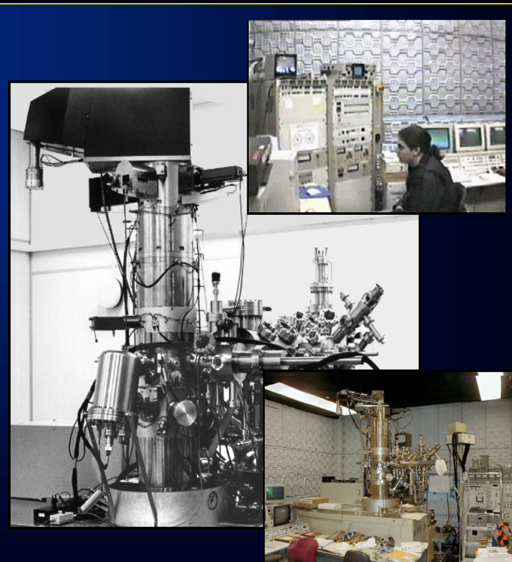
SEM

TEM

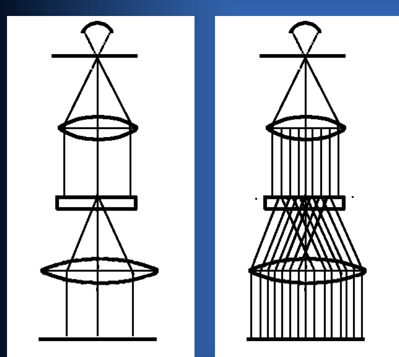


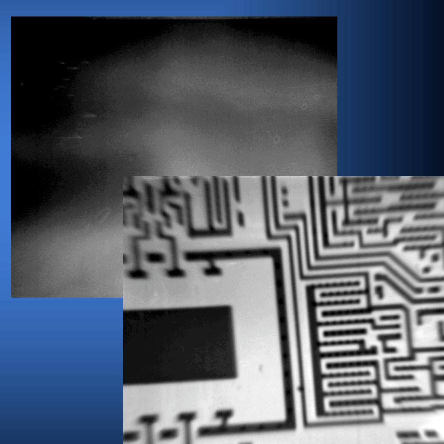
ANL - Advanced Analytical Electron Microscope

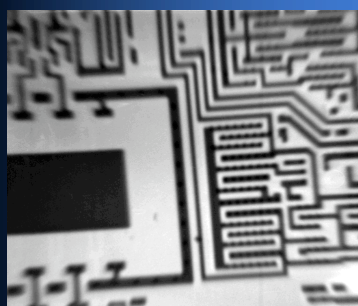
- ***Cold Field Emission Electron Source***
  - Vo: 50 - 300 kV
- ***Ultrahigh vacuum (UHV) environment***
  - ~ 1 x 10^{-11} torr - Gun, < 2 x 10^{-10} torr - Column
  - < 5 x 10^{-10} torr - Specimen Preparation Chamber
- ***Electron Optics capable of :***
  - STEM / SEM:
  - TEM:
  - CBED/SAED:
  - Other Modes: TSEM, TSED, RHEED
- ***Side Entry Goniometer Stages***
  - RT Double Tilt Beryllium:
  - LN2 Cooled Double Tilt Be Stage:
  - Single Tilt Heating Stage:
- ***Analytical SubSystems on the E/O Column***
  - XEDS , EELS, AES
- ***Specimen Preparation Chamber***
  - High Pressure/Temperature Gas Reaction Cell
  - Thin Film Evaporation Chamber
  - Mini-SIMS system - Gallium LMIS, Quad Mass Analyzer
  - RV LEED & Vacuum Transfer Vessel
  - ANL MultiPort Station for development work.
- ***Computer Control***
- ***Special Objective Lens Port Configuration***
  - 7 Experimental Ports on Objective Lens for Analytical Equipment
  - 3 Additional Ports for Electrical Feedthrus etc...

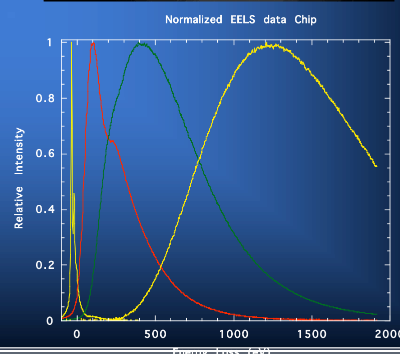


Transmission Microscopy

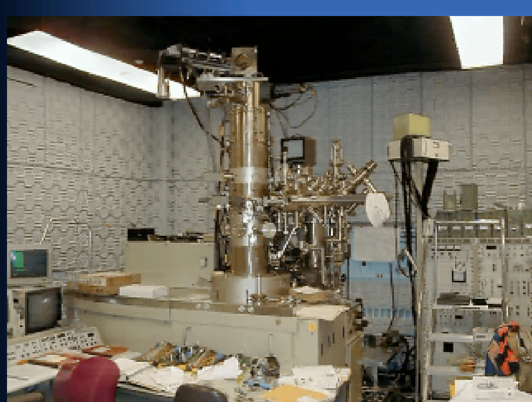


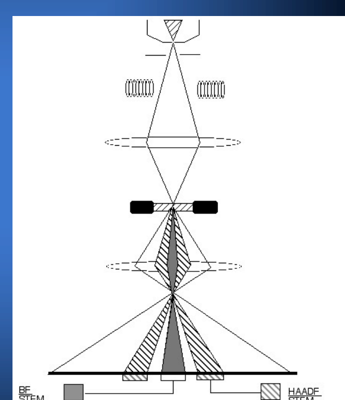




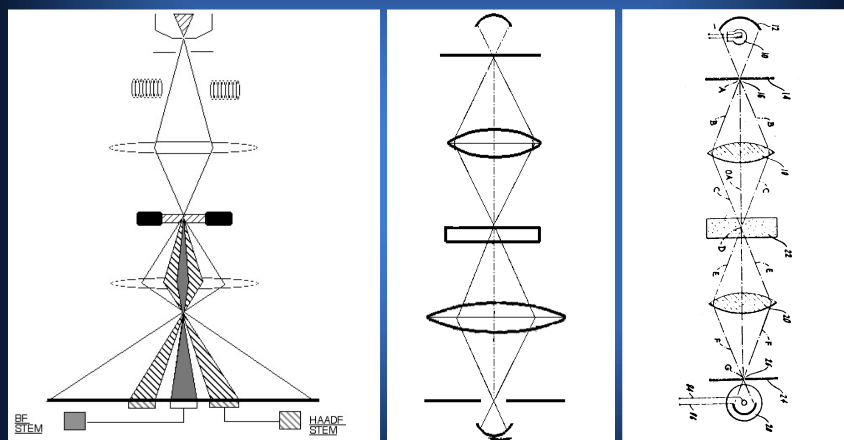


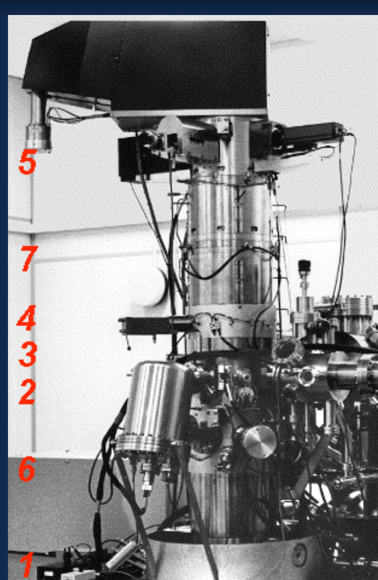
ANL AAEM
An Electron Optical Bench

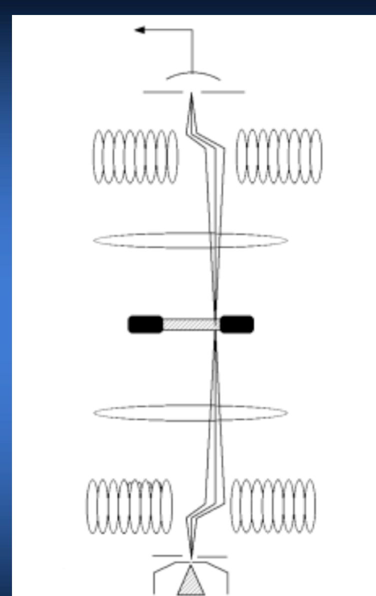




Scanning Confocal Electron Microscopy

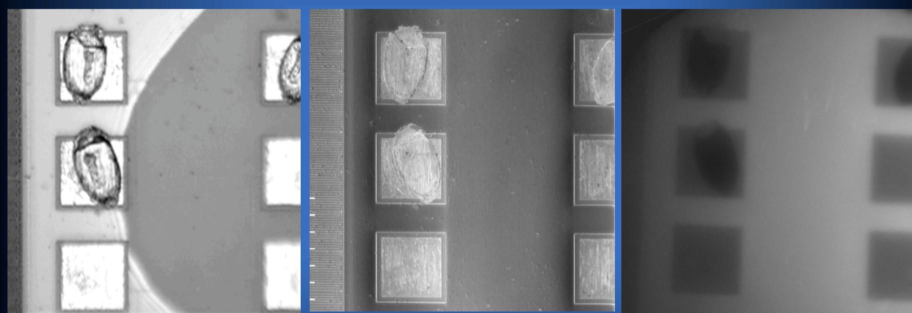






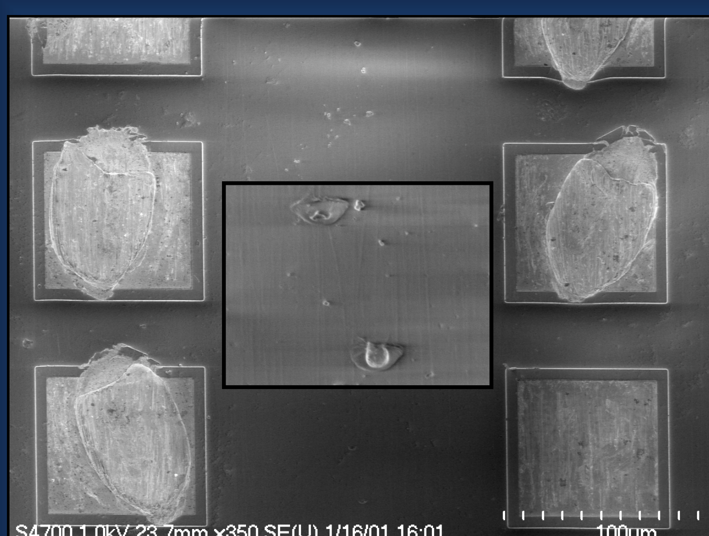
US Patent # 6,548,810

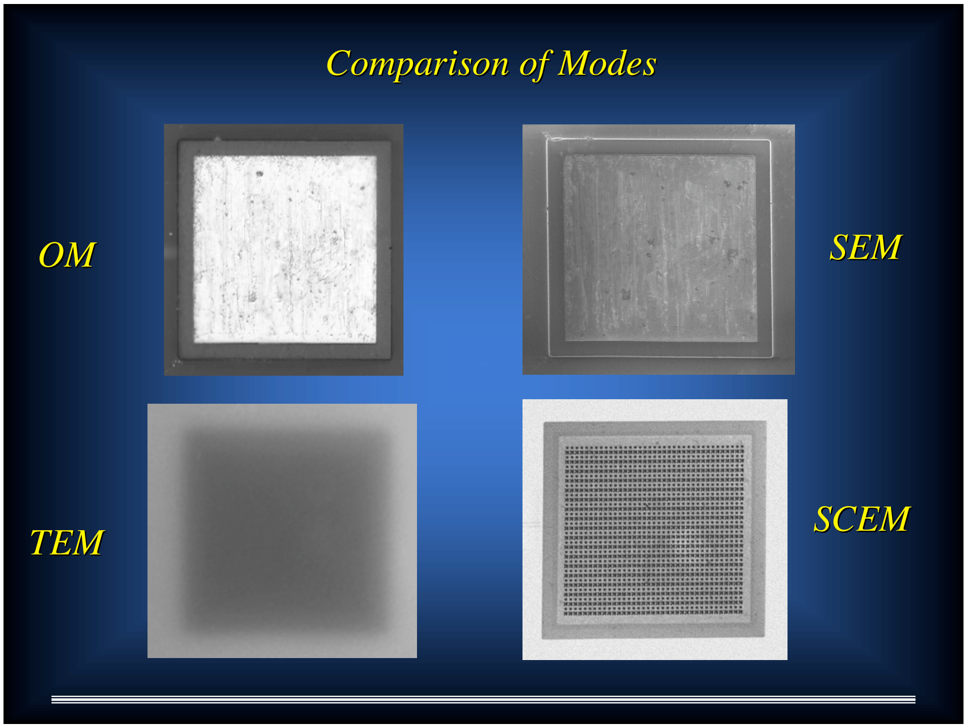
Optical / SEM / TEM Comparison

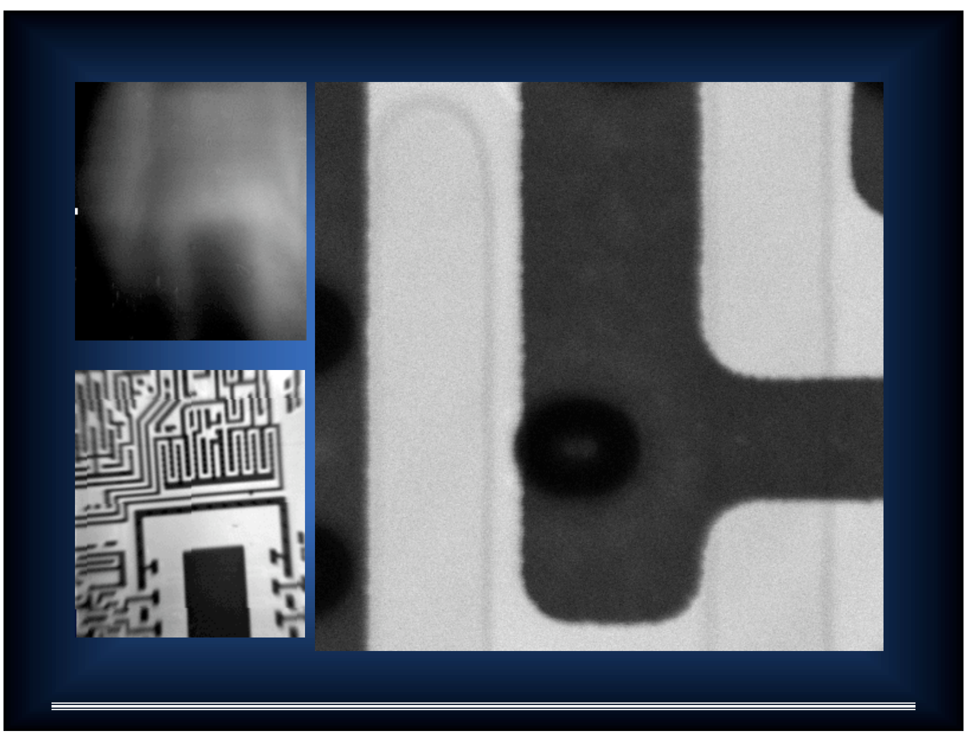


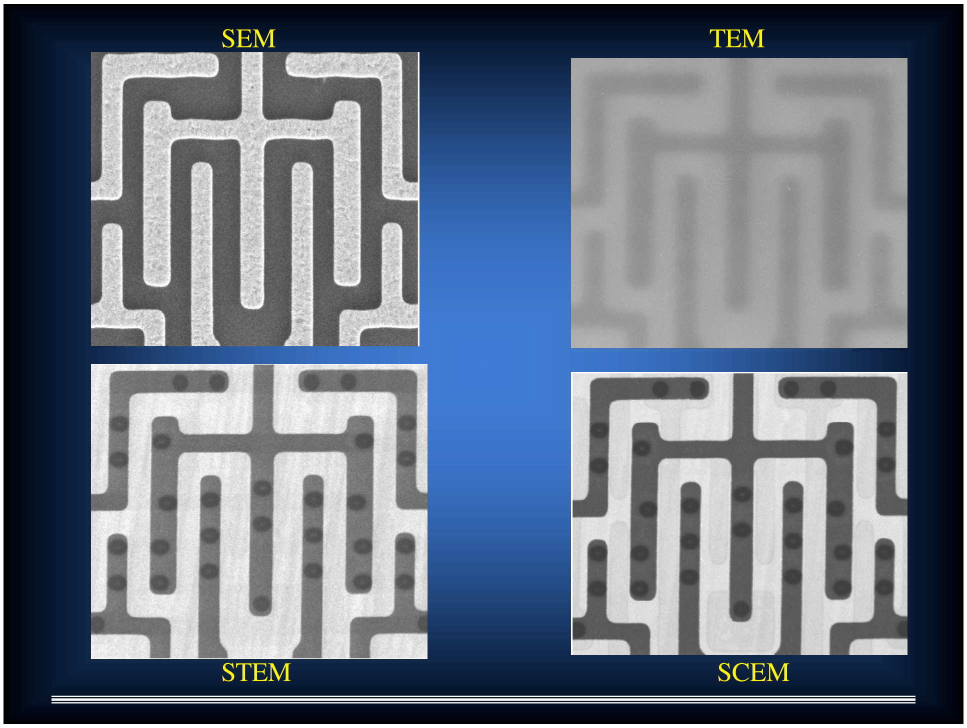
80 μm

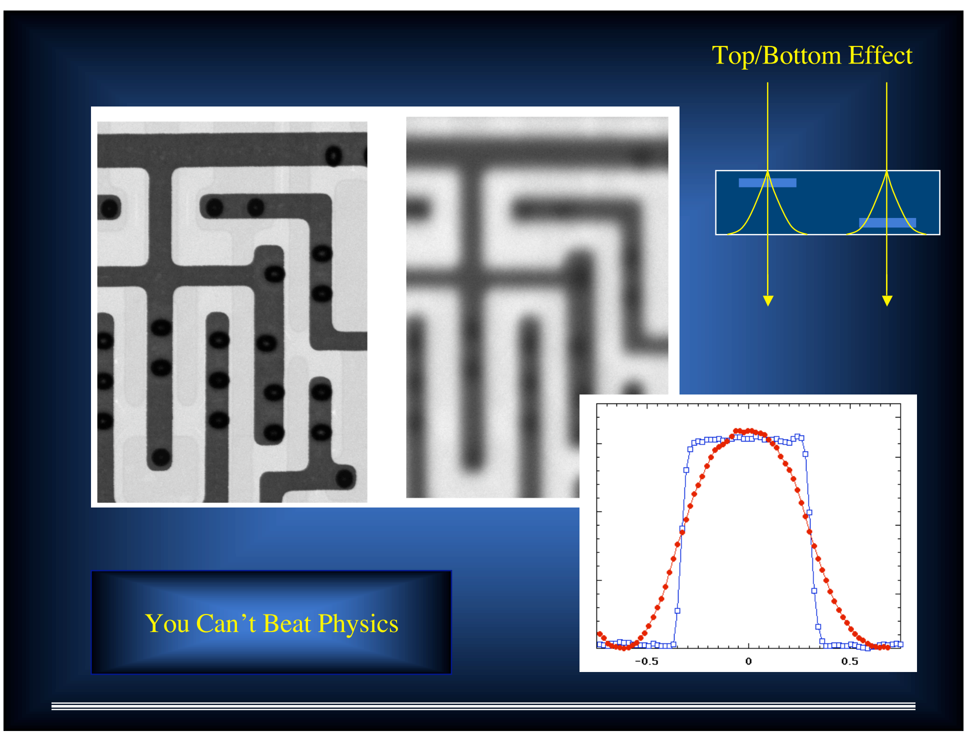
Scanning Electron Microscopy

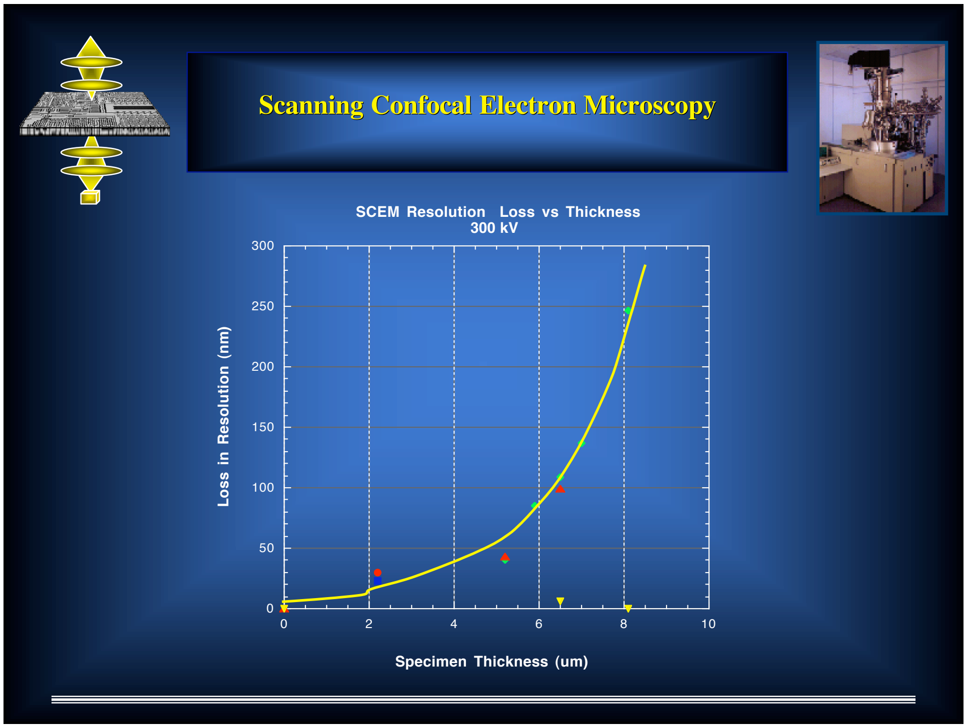


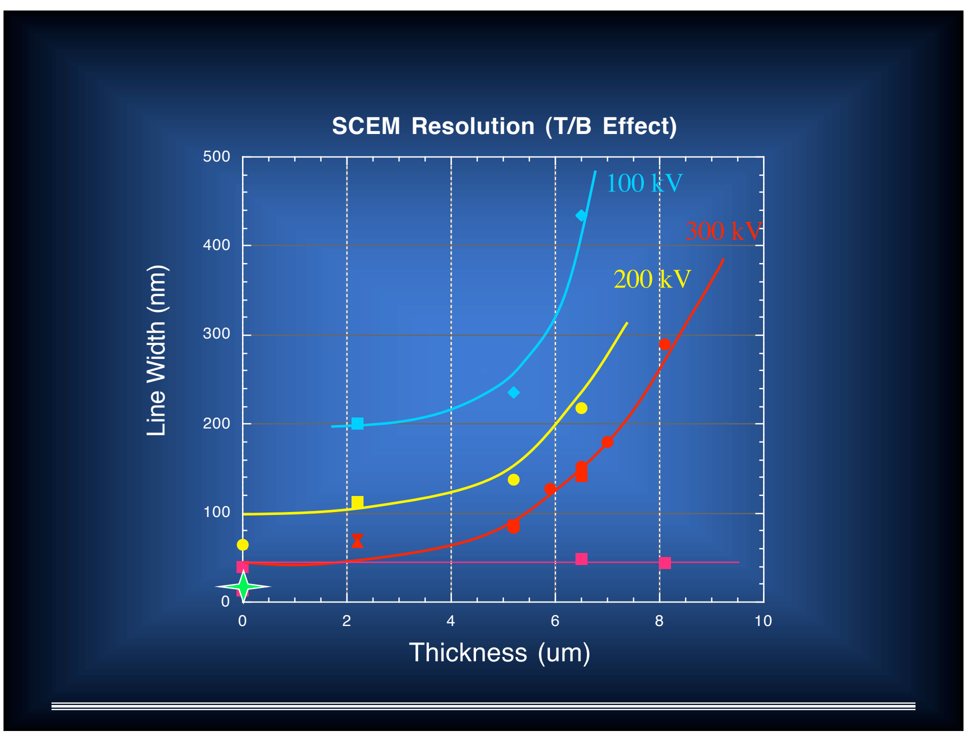

Comparison of Modes
OM
SEM
TEM
SCEM



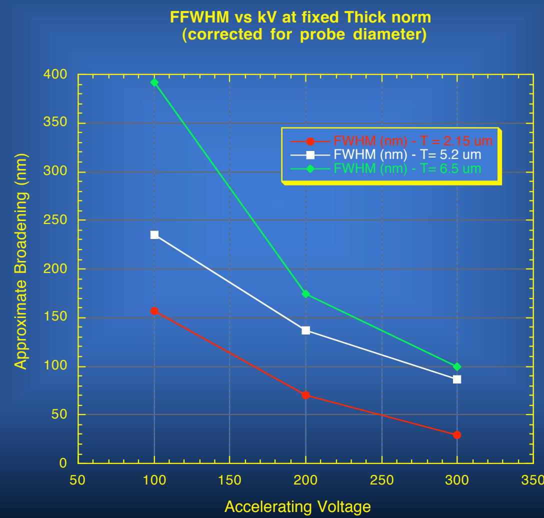

SEM
TEM
STEM
SCEM


Top/Bottom Effect
You Can't Beat Physics
-0.5
0
0.5

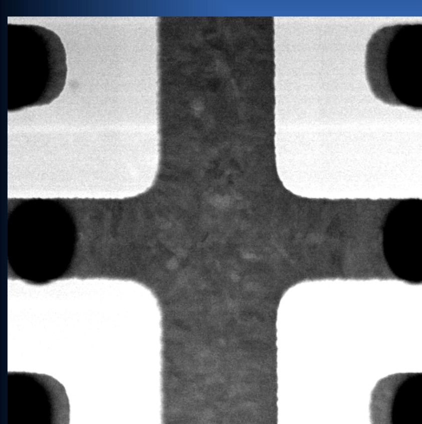

Scanning Confocal Electron Microscopy
SCEM Resolution Loss vs Thickness
300 kV
Loss in Resolution (nm)
Specimen Thickness (um)
0
50
100
150
200
250
300
0
2
4
6
8
10

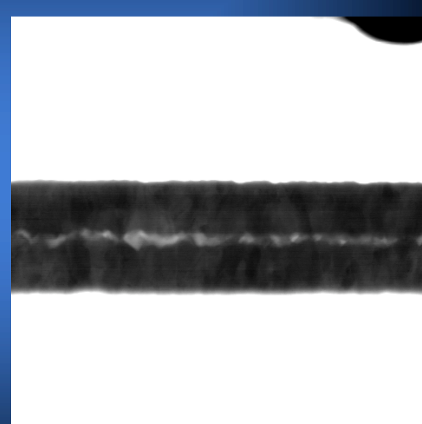

SCEM Resolution (T/B Effect)
100 kV
300 kV
200 kV
Line Width (nm)
Thickness (um)
0
100
200
300
400
500
0
2
4
6
8
10

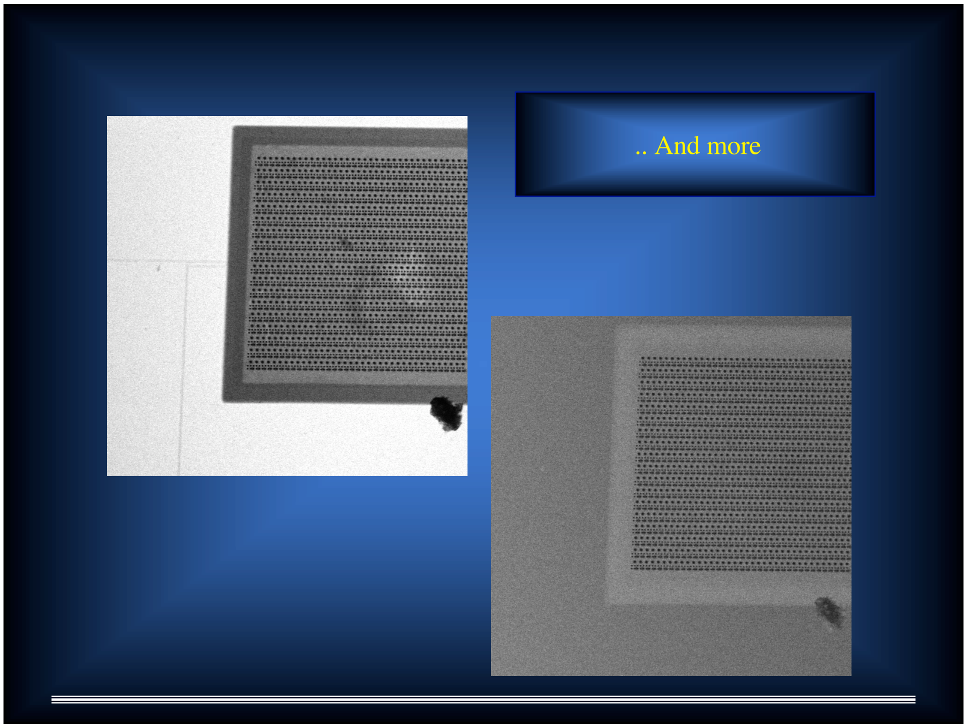
Scanning Confocal Electron Microscopy

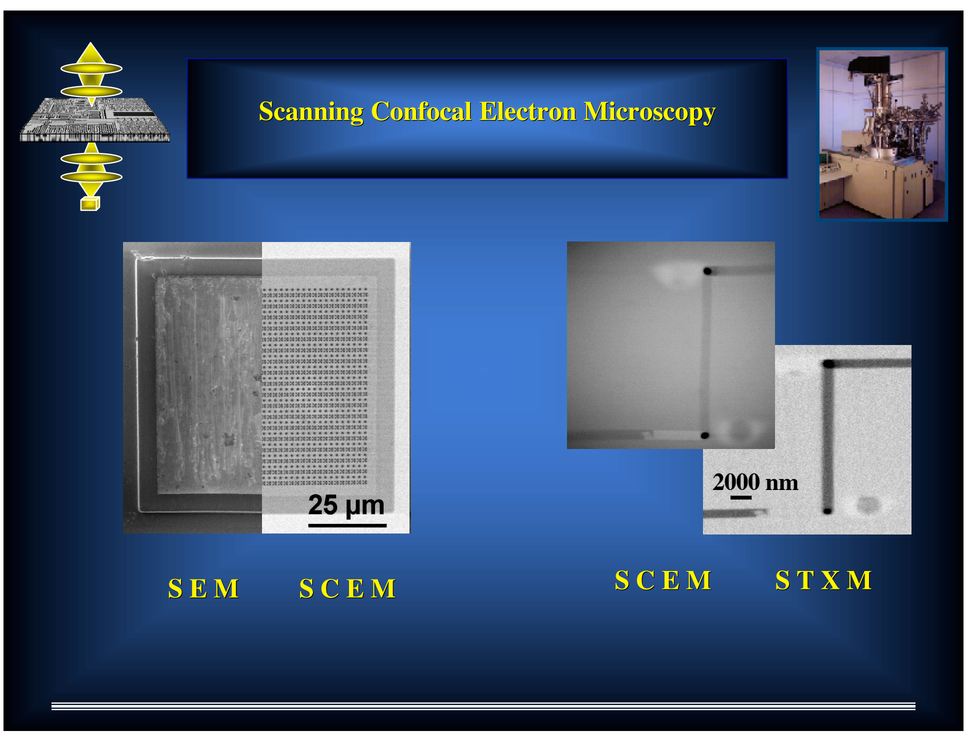


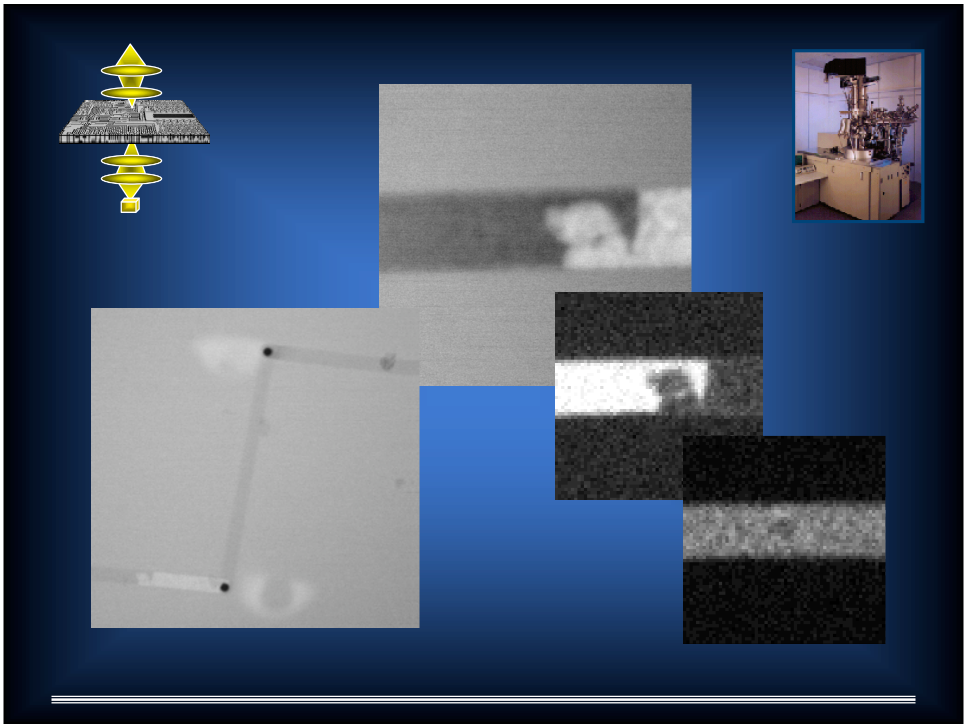
There is still more you can do



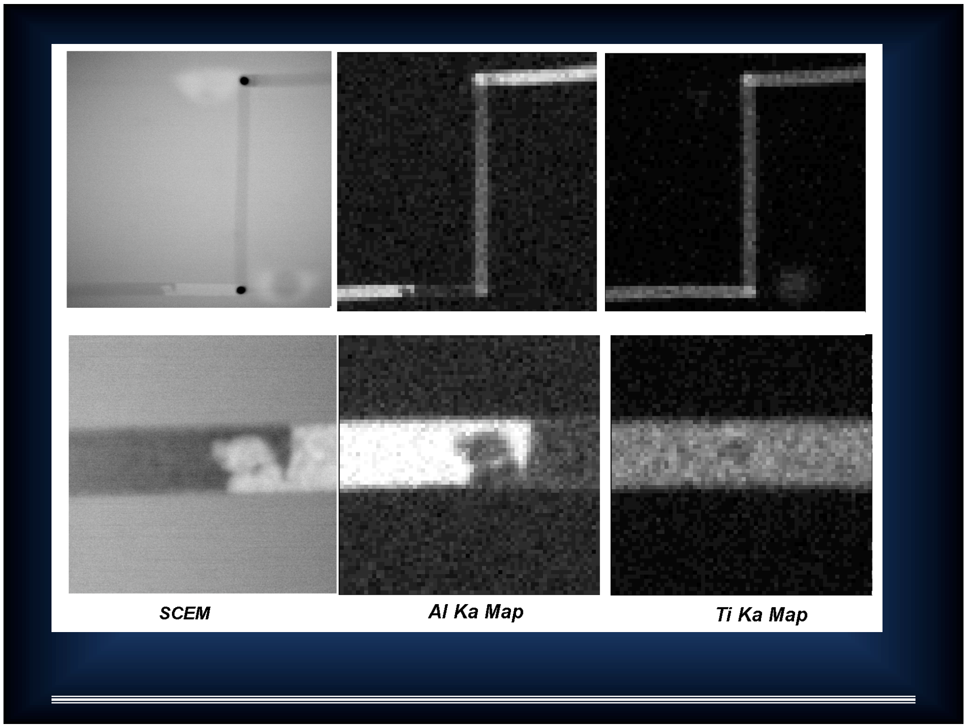


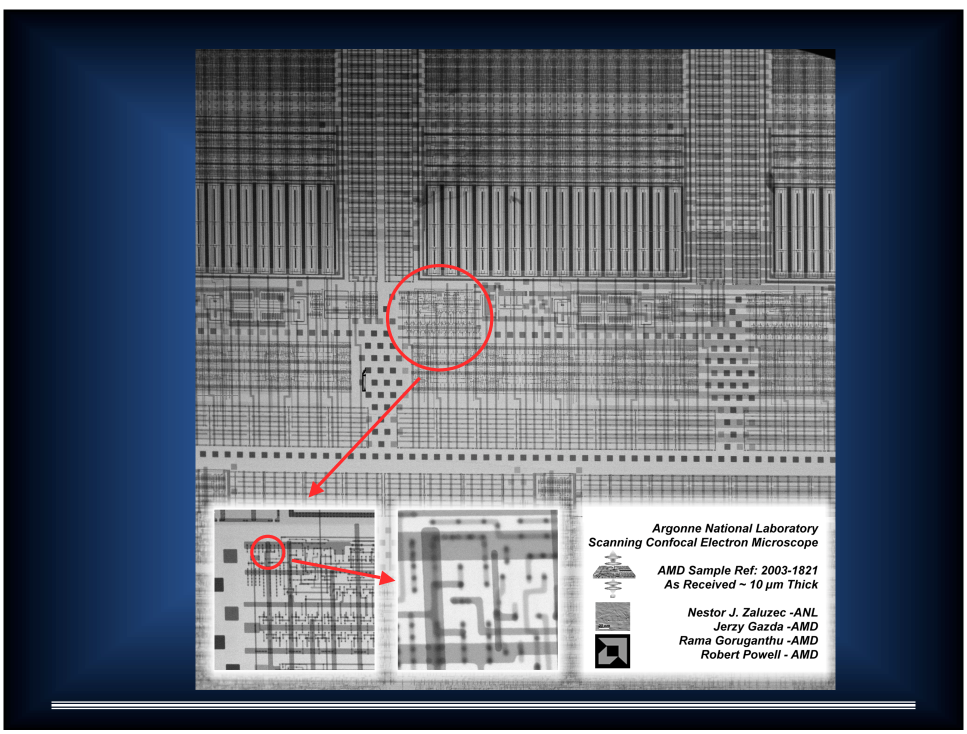

.. And more

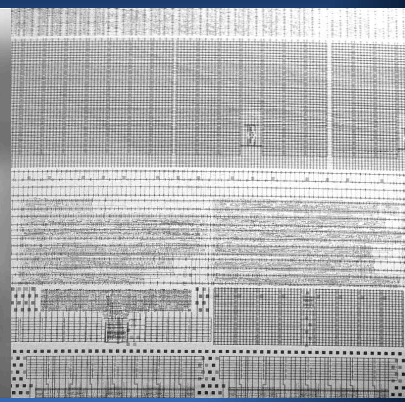

Scanning Confocal Electron Microscopy
25 µm
2000 nm
SEM
SCEM
SCEM
STXM



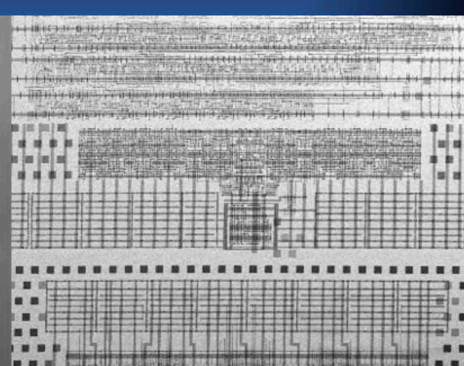

Scanning Confocal Electron Microscopy


SCEM
Al Ka Map
Ti Ka Map


Argonne National Laboratory
Scanning Confocal Electron Microscope
AMD Sample Ref: 2003-1821
As Received ~ 10 µm Thick
Nestor J. Zaluzec -ANL
Jerzy Gazda -AMD
Rama Goruganthu -AMD
Robert Powell - AMD

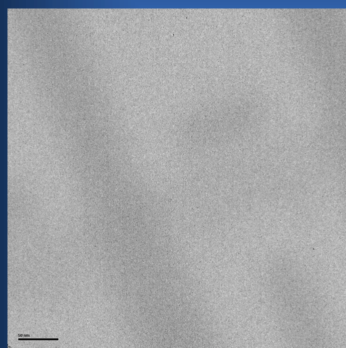



TEM SCEM

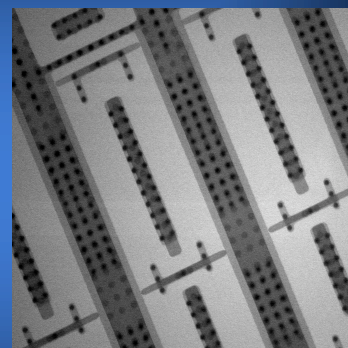



TEM SCEM

Real World Application of SCEM
Semiconductor Device Failure

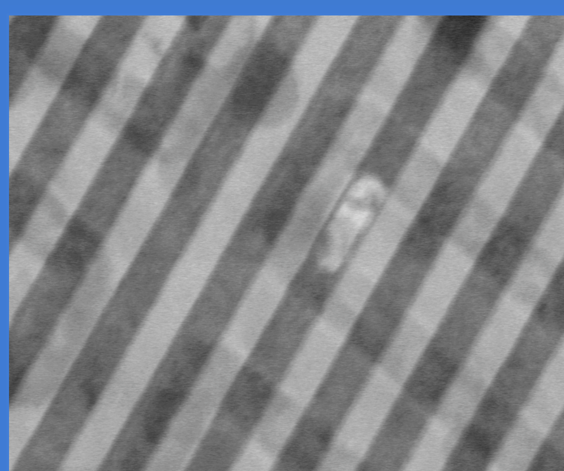


TEM



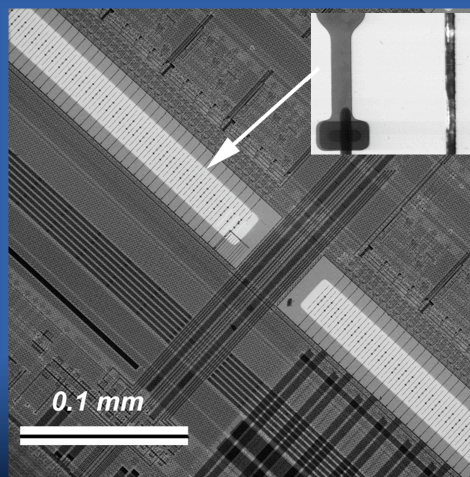
SCEM

Electromigration Voids in"Real Devices"



Collaboration with J. Gazda of AMD

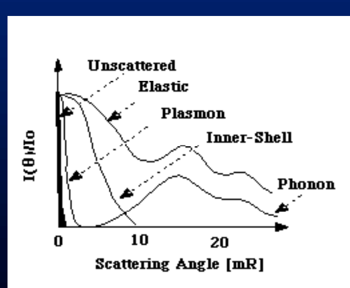
Scanning Confocal Electron Microscopy

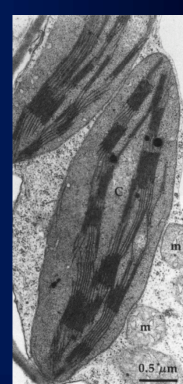


Amorphous Solids:

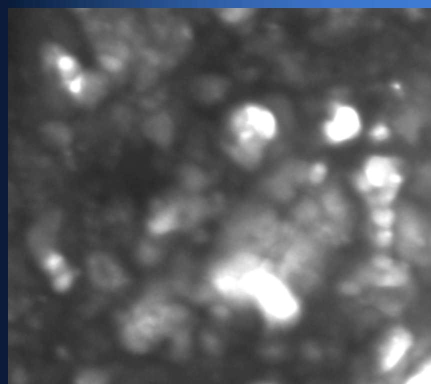
$$I(\theta) \sim |f(\theta)|^2 \bullet \left| 1 + \frac{\sin(kR)}{kR} \right|$$

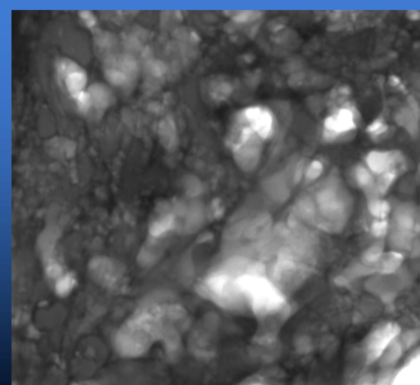
$$k = \frac{4\pi}{\lambda} \sin(\theta)$$



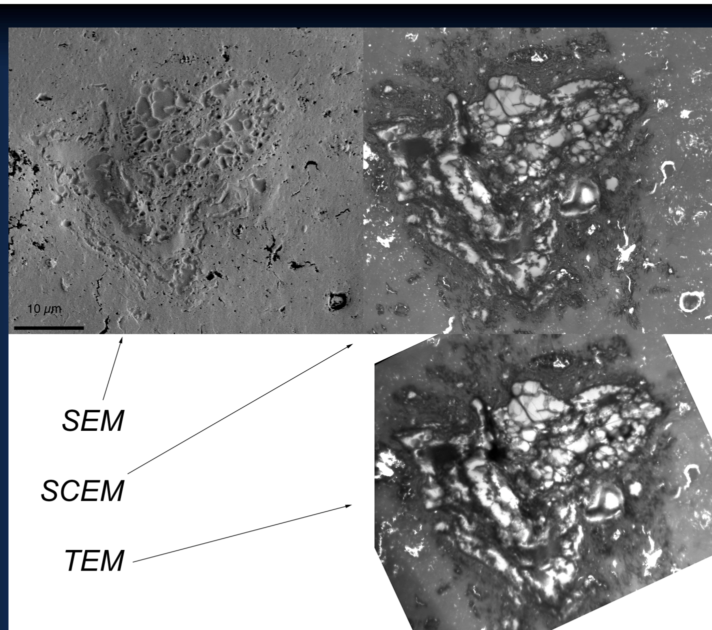


Biological Applications
5 μm thick TEM section

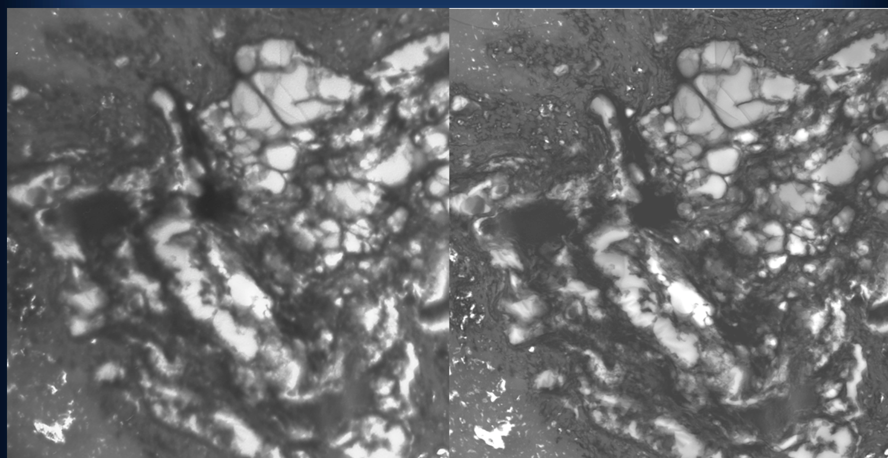




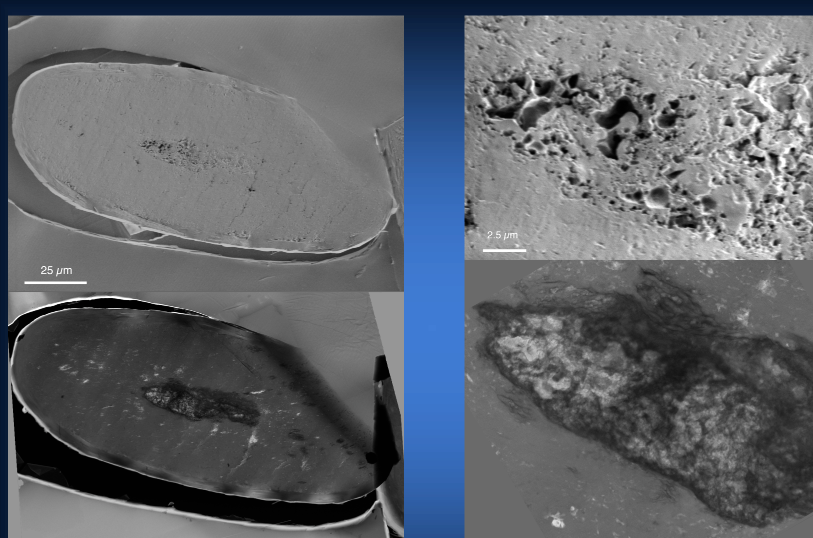
Collaboration with P. Hallegot L'Oreal Research Institute



1 μm thick sections

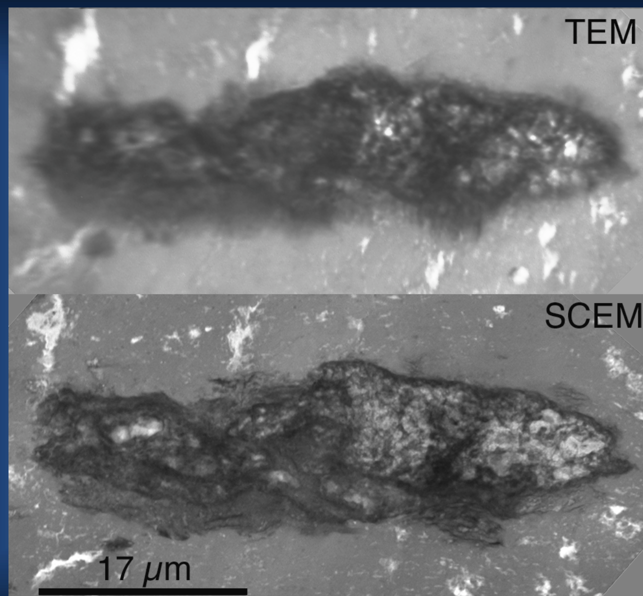


TEM SCEM
1 μm Thick

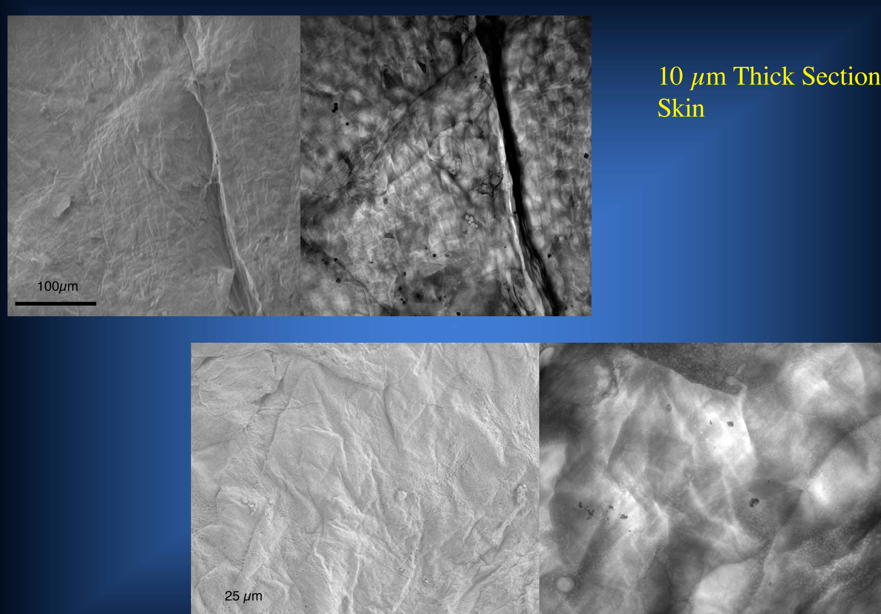


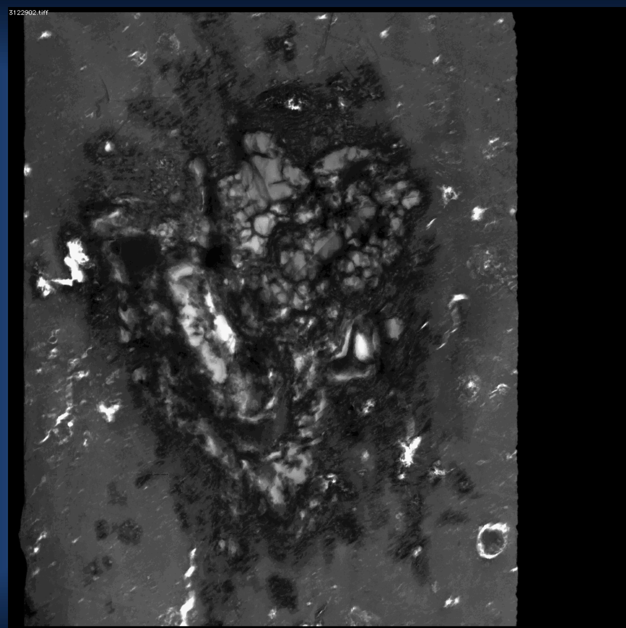
SEM, SCEM

5 μm Thick



10 μm Thick Sections
Skin





What is Next?

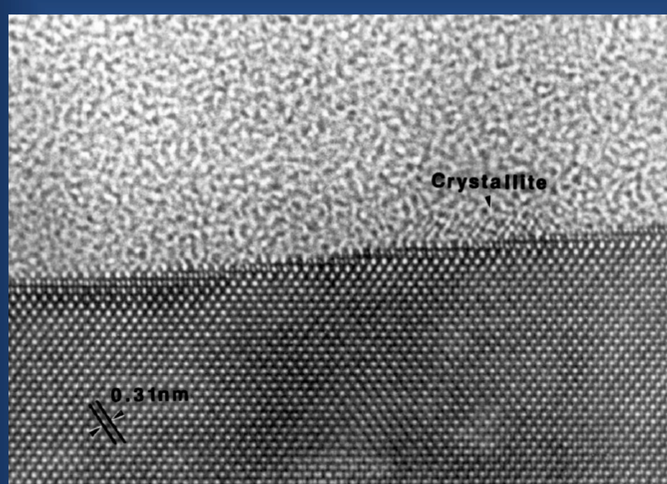
Feynman's Challenge

"It would be very easy to make an analysis of any complicated chemical substance; all one would have to do would be to look at it and see where the atoms are. The only trouble is that the electron microscope is one hundred times too poor … I put this out as a challenge: Is there no way to make the electron microscope more powerful?"

– Richard P. Feynman, 1959,
"There's Plenty of Room at the Bottom"

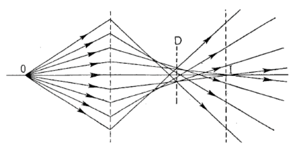
Atomic-scale imaging plays a unique role in discovering how nanostructures work.

High Spatial Resolution Imaging



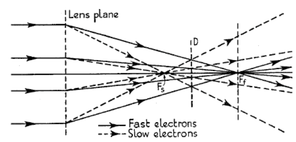
Aberrations

- Spherical



$$r_{sph} = C_s \beta^3$$

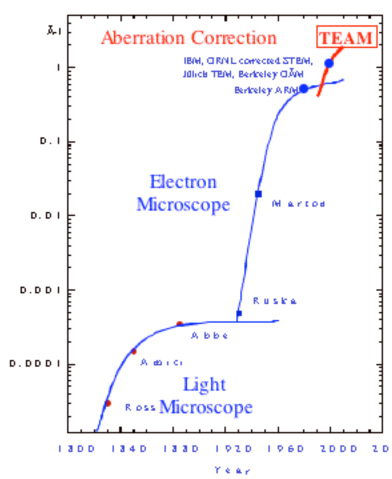
- Chromatic



$$r_{chr} = C_c \frac{\Delta E}{E} \beta$$

Perspective - why now?

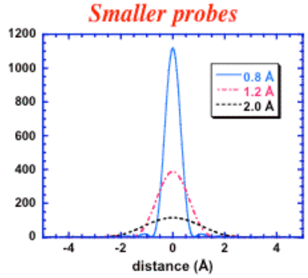
- A breakthrough in electron optics removes the barrier that has limited the performance of the electron microscope since its invention.
- Simultaneous advances in electronics, detectors, MEMS and computers.
- Nanoscience and technology centers need atomic-level characterization.
- Important opportunity for DOE investment in development of electron optical instrumentation.



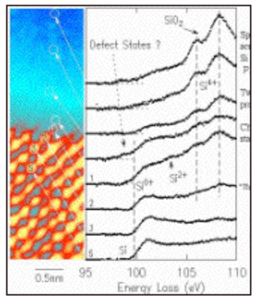
Adapted from Rose

What does aberration correction buy us?

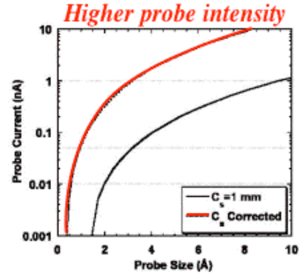
Smaller probes



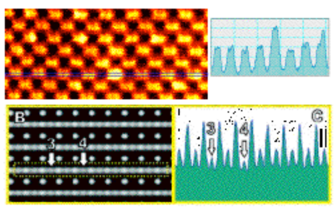
More signal



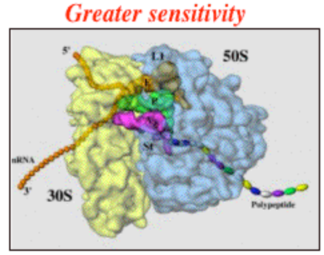
Higher probe intensity



Greater contrast



Greater sensitivity



Extraordinary scientific opportunities

by direct observation of individual nanostructures

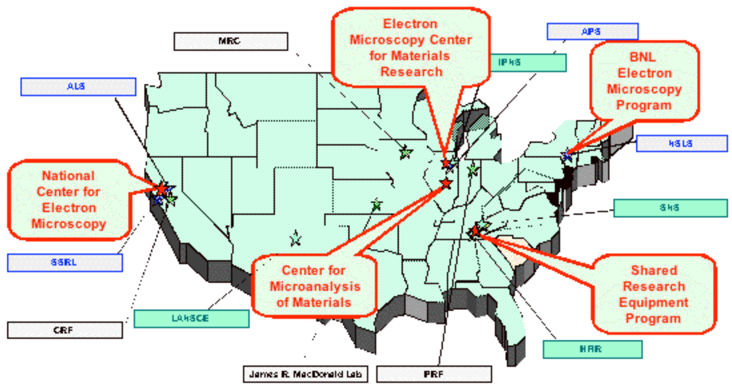
- three-dimensional atomic-scale structure, shape, and defect distribution
- spectroscopic identification and location of individual dopant atoms
- direct imaging of the atomic-scale structure of glasses
- electronic structure of individual point defects
- non-spherical charge density and valence electron distribution
- in-situ synthesis of novel nanoscale structures
  - e.g., electron-beam lithography with sub-Angstrom beams
- in-situ observation of properties and response to external variables
  - temperature, stress, chemical activity, and applied electric and magnetic fields…

… all with unprecedented spatial, spectral & temporal resolution

A collaborative project

coordinated proposal by BES efforts in e-beam microcharacterization

- TEAM goals: capitalize on major recent advances in electron optics to design, build, and operate the next-generation electron microscope capable of atomic resolution imaging in real time and 3-D with single atom sensitivity.
- Redesign electron microscope around aberration corrected optics to serve as platform for suite of new instruments with unparalleled performance.

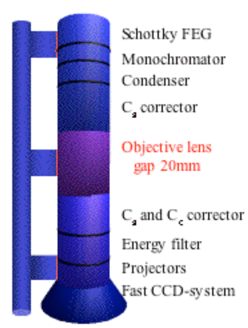


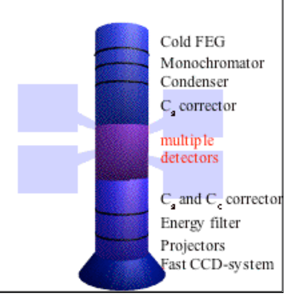
Beyond the TEAM development project

Based on TEAM platform, develop suite of aberration-corrected instruments for

- In-situ synthesis
- UHV environment
- Ultimate microanalysis
- Fast dynamics
- Lorentz imaging, …

- Stimulate revitalization of electron optics in US.
- Instruments to be remotely accessible as a "distributed facility".
- Proximity to nation's synchrotron light and neutron sources and Nanoscience Centers.
- Well established user programs with missions that are aligned with BES science goals and provide national coverage - geography, scientific expertise, user community, instrumentation.



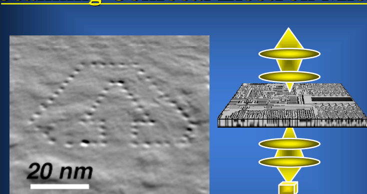


TEAM - a unique tool for scientific discovery

- Driven by scientific needs - powered by electron optics revolution.
- Unique in providing 3D atomic-scale structure and dynamics of individual nanostructures.
  - From 2D to ***3D***
  - From atomic columns to ***atoms***
  - From static to ***dynamic***
- New opportunities for materials discovery through combined atomic-scale characterization and in-situ measurement.

- *Meeting Feynman's challenge:* atomic-scale characterization defining the quantum mechanical boundary conditions for the electronic structure calculations necessary to determine how nanostructures work.

Scanning Confocal Electron Microscopy



Nestor J. Zaluzec