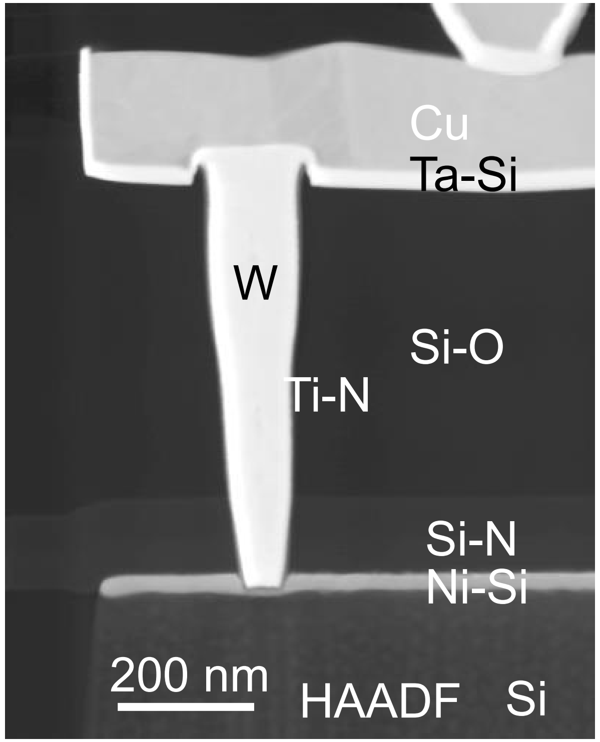
SAND2021-13457PE

Introduction to TEM: A Powerful Tool for Microelectronics FA

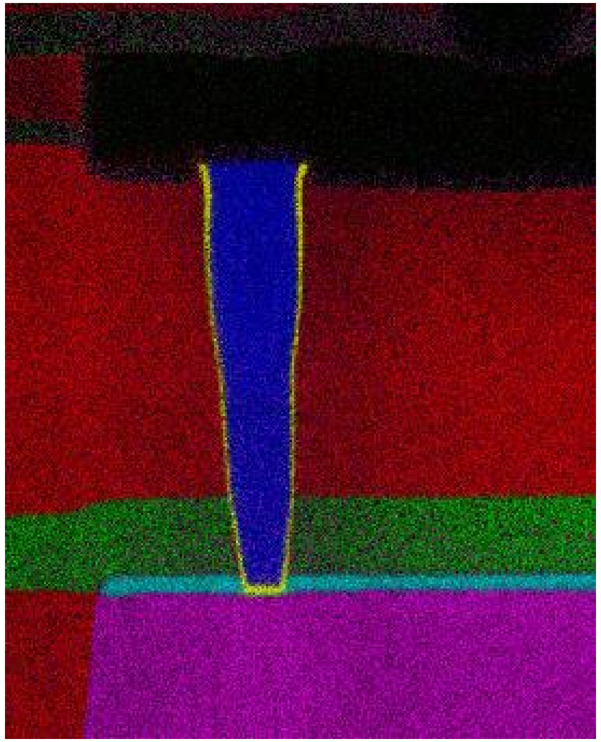


Paul G. Kotula

Sandia National Laboratories, Albuquerque, NM, USA

Thanks to Barry Carter for some of his slides!

Paul.Kotula@sandia.gov



About me: Characterization answering mostly materials science questions

- I started doing TEM in 1991 in grad school
  - Solid state chemistry in epitaxial oxide thin film reaction couples
- Post doc at LANL
  - Analysis of oxides, intermetallics, and other things…with TEM
- Staff at Sandia
  - I look at almost all classes of materials with TEM/STEM
  - Technique development in hyperspectral imaging and data analysis
  - Most interesting project: Analysis of Anthrax attack materials as part of the FBI’s Amerithrax investigation (2001-2008)

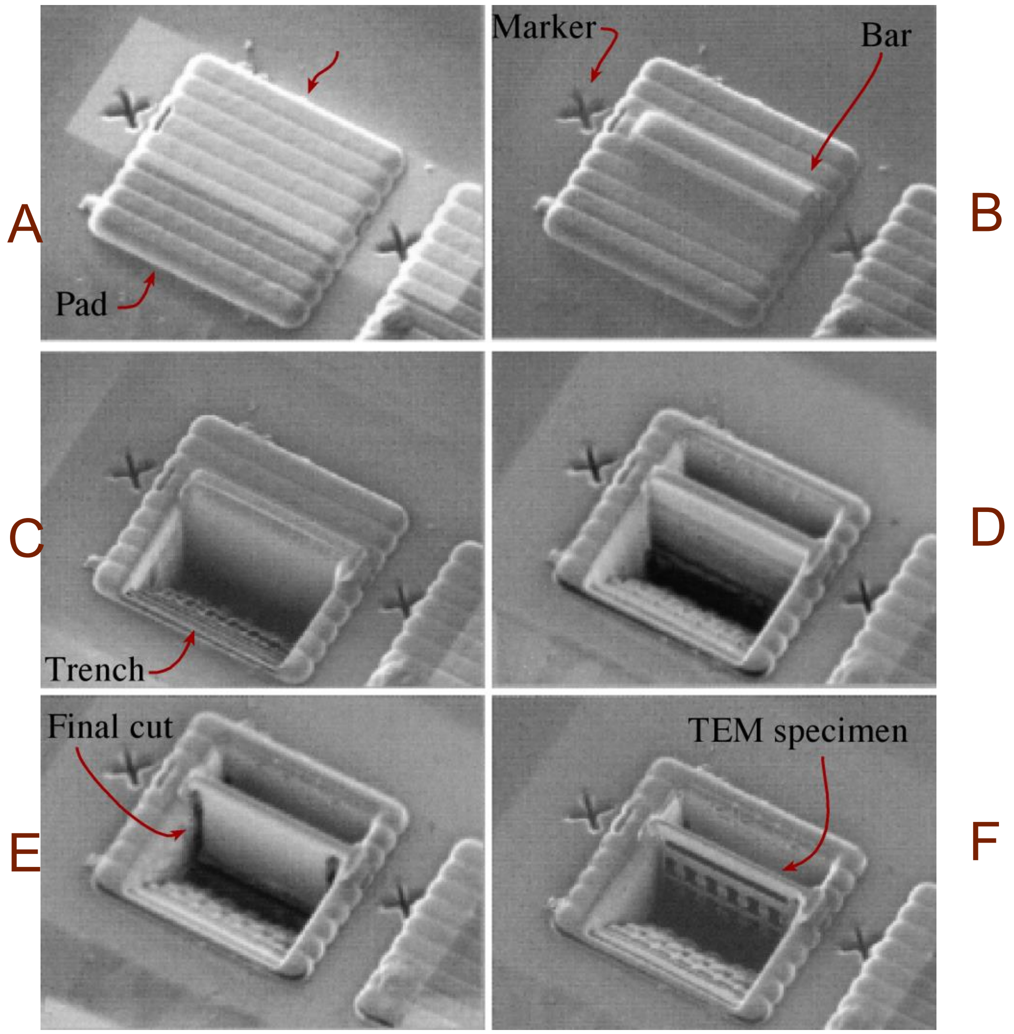
What is TEM?

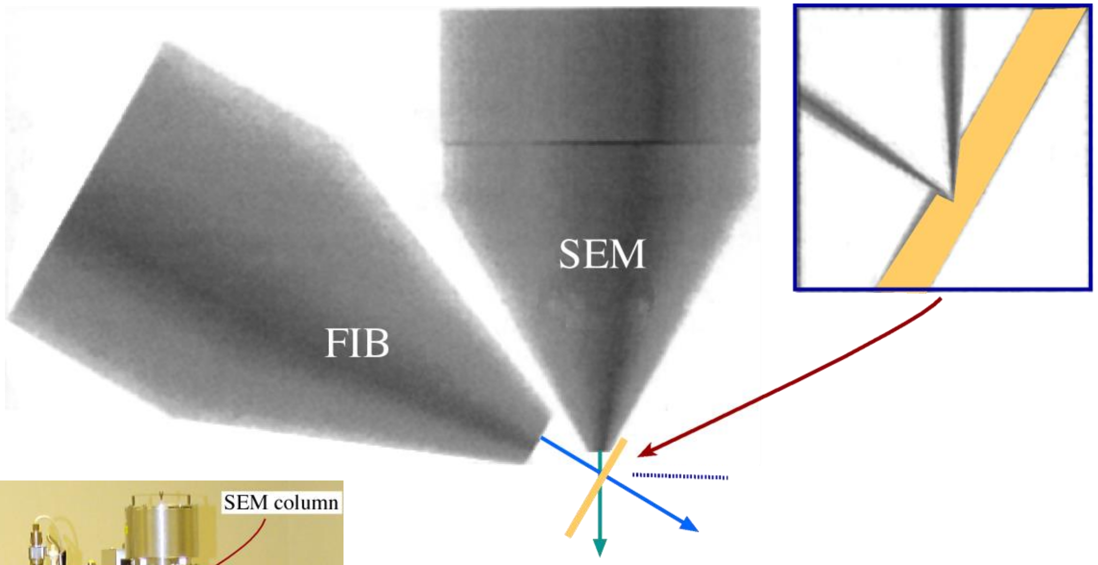
- High-energy (100 kV-300kV) electrons pass through a sample.
  - Relativistic light particles with wavelengths of 0.037Å-0.0197Å
  - Interesting fact: There’s only one electron passing through the sample at a time. Wave? Particle? Yes both!
- The sample must be thin since electrons interact strongly with matter. Electrons are easy to steer and we can do imaging and diffraction in the same tool.
- For FA, FIB allows us to make site-specific sample with little effort. Other FA techniques are typically used to identify defects and mark locations prior to FIB.

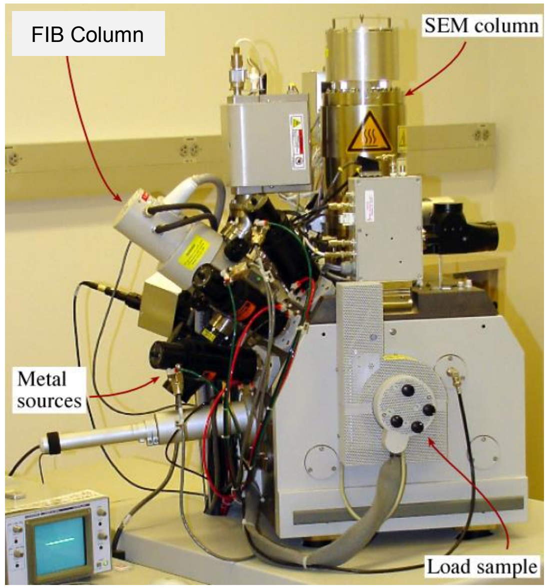
What information does TEM give us?

- Metrology: e.g., gate oxide thickness
- Defects: e.g., dislocations, mask issues, etc.
- Chemistry
  - In STEM mode, scanning TEM analogous to SEM but in transmission
  - The fast electrons can eject core and bonding electrons from the atoms in the sample. Relaxation results in:
    - Characteristic X-rays (EDXS)
    - Energy-loss electrons resulting from X-ray and Auger transitions

Sample Preparation





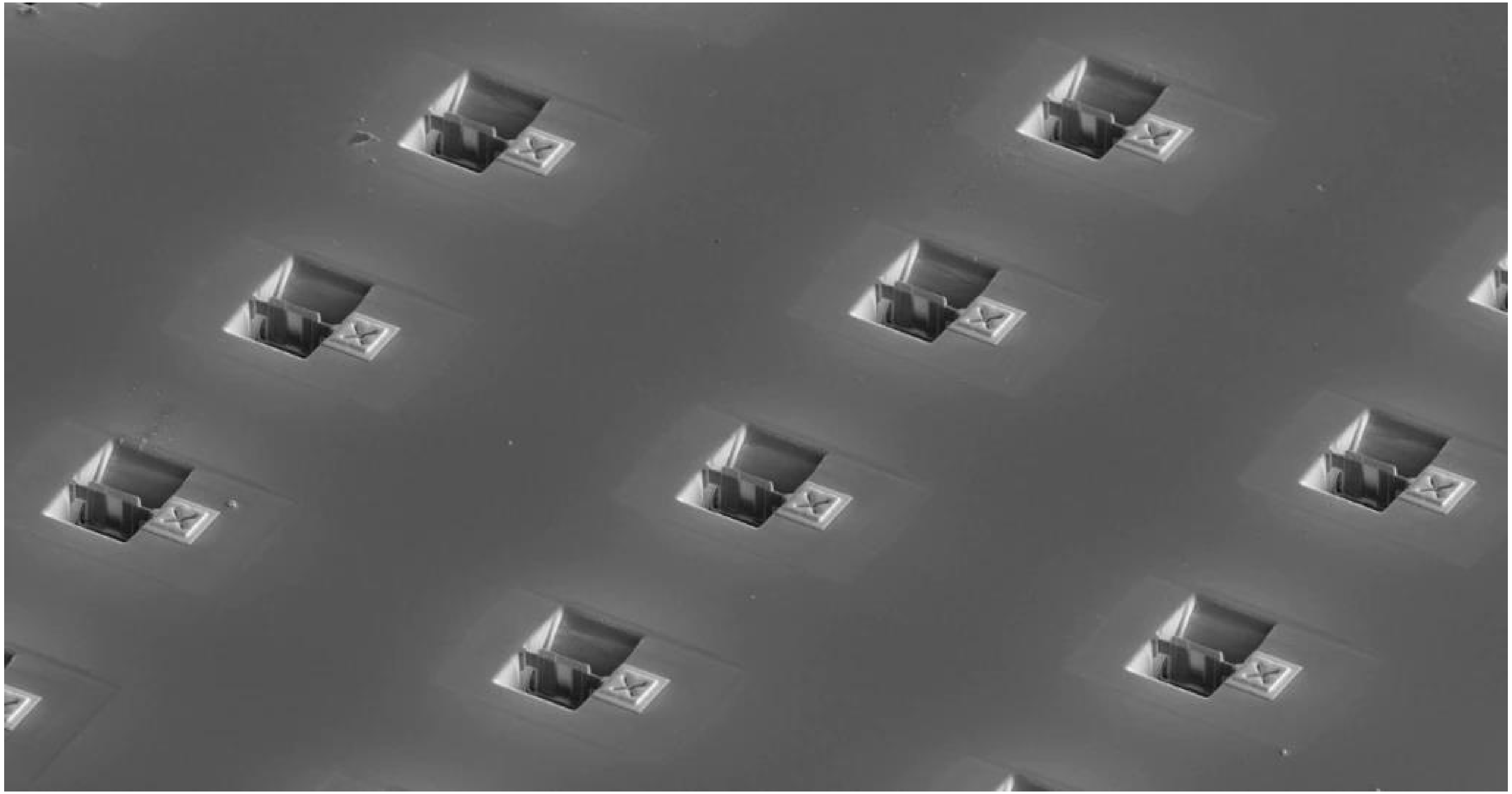


Most FIBs use Ga LMIS but Xe plasma FIBs are now here

Goal: Sample 50 nm -100 nm thick, suitable for TEM analysis

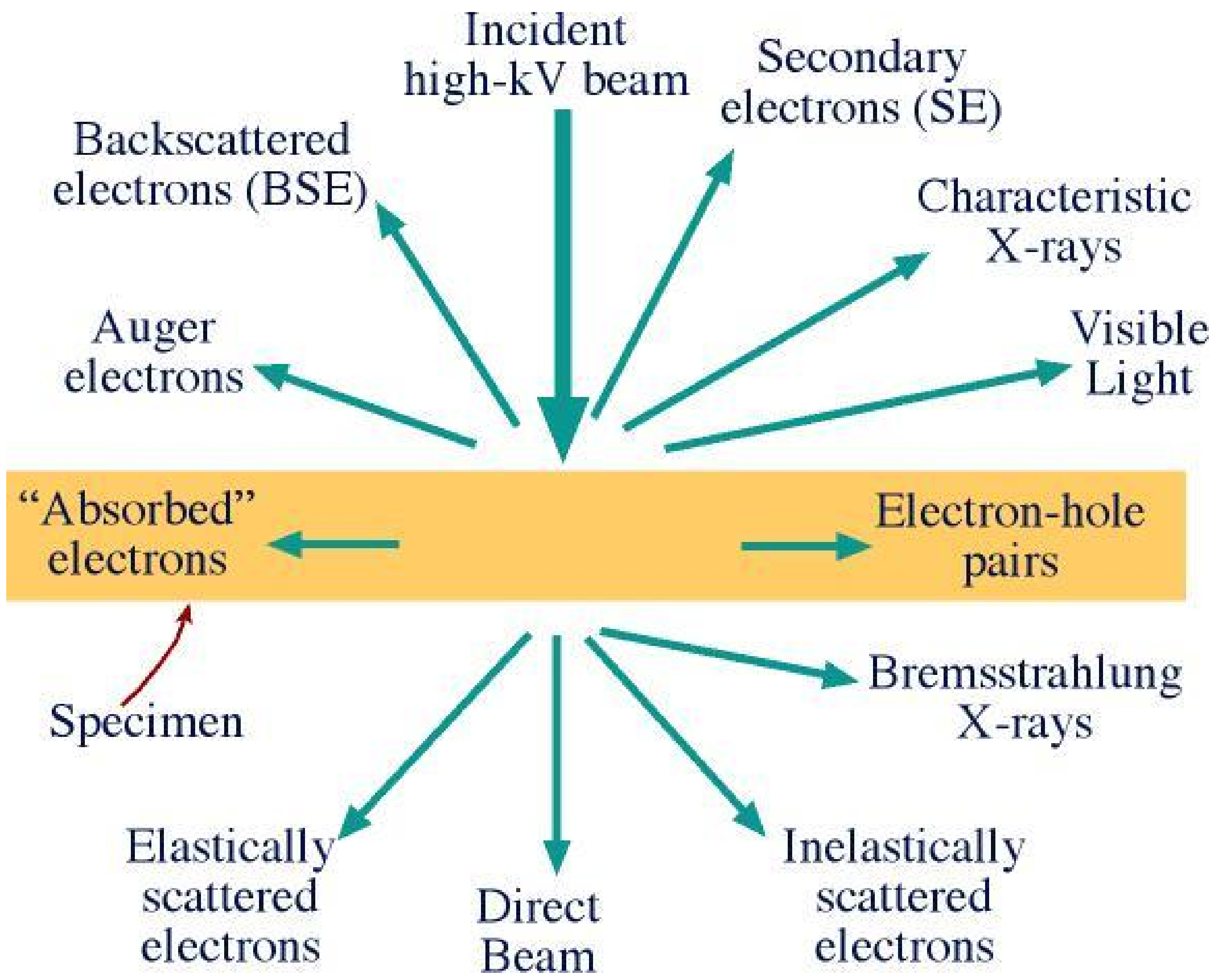
High-throughput FIB sample preparation via automation

Fortunately this is not the type of stuff Sandia does



Courtesy Thermo Fisher Scientific

Basics of TEM/STEM



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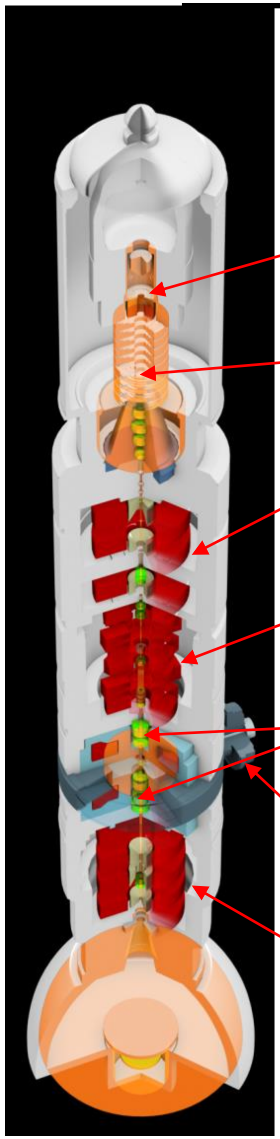
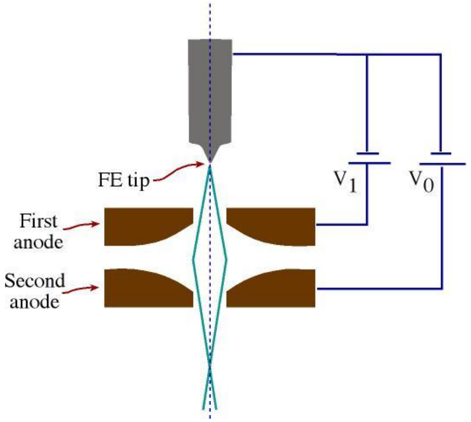
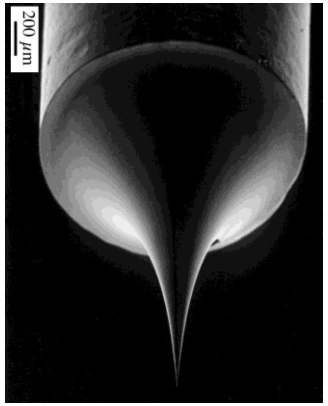
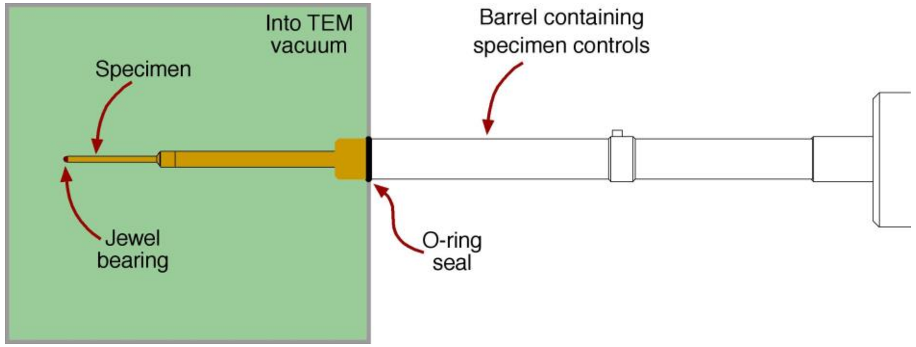
TEM/STEM basics: Parts of the microscope



- High-voltage generator
- Electron Source (typically FEG)
- Accelerator
- Condenser Lenses
- Spherical aberration corrector*
- Objective Lens (Immersion)
- Sample goniometer
- Projector Lenses
- Detectors (electrons/photons)







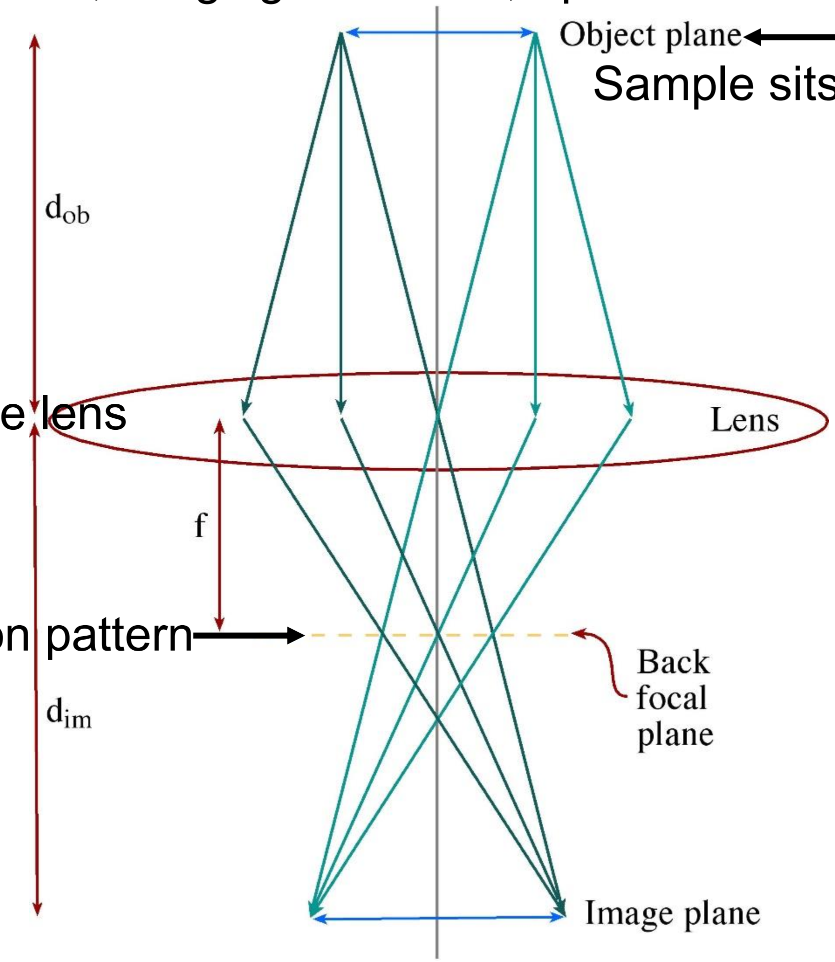
TEM specimen grids are 3 mm in diameter

*optional

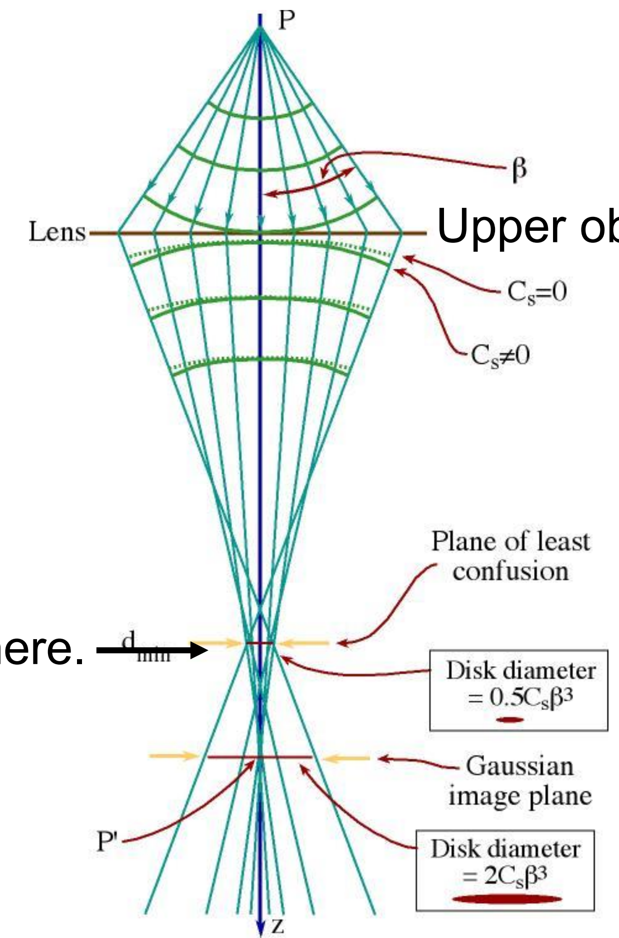
Basics of TEM/STEM

Our sample sits inside the objective lens which has an upper and lower part

TEM mode, imaging/diffraction, ~planar illumination



STEM mode, diffracted electrons imaged on detectors



Like an SEM, for each beam position we record a signal

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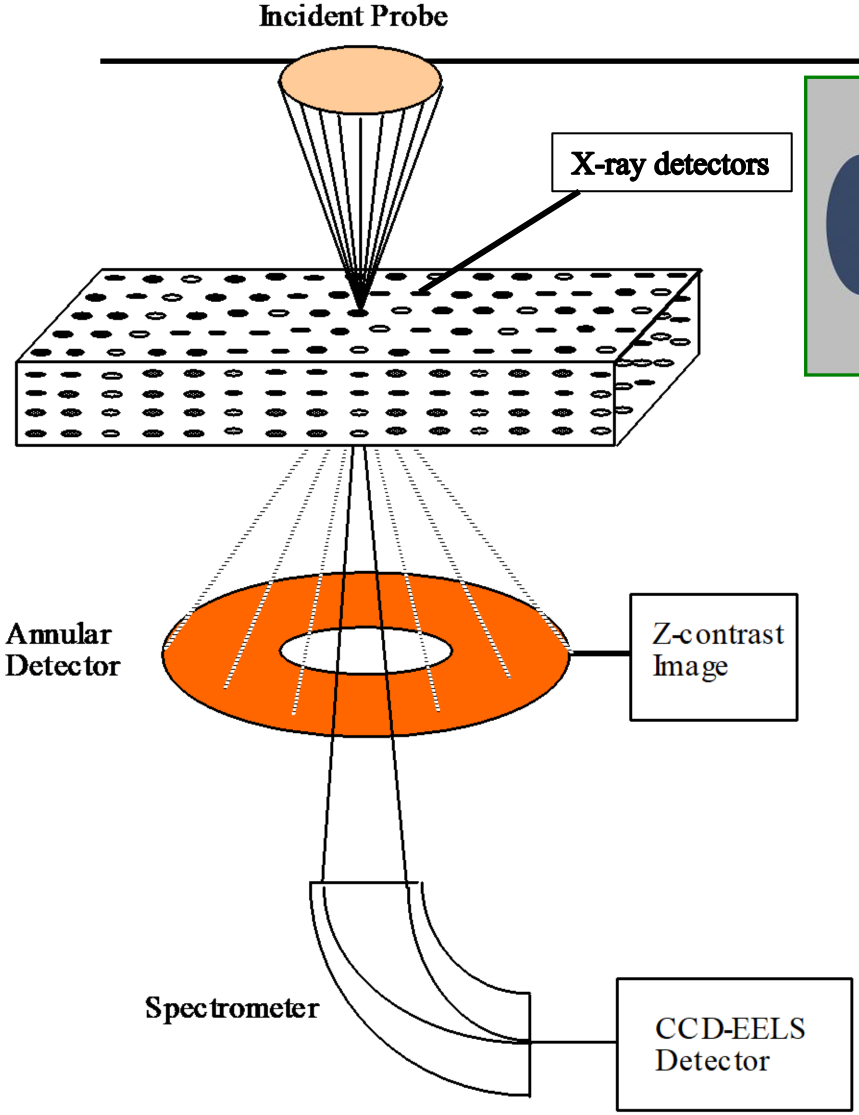
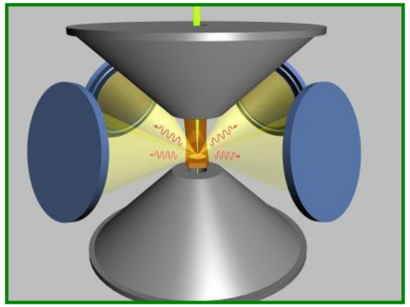
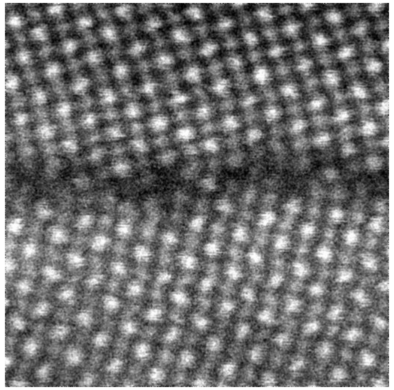
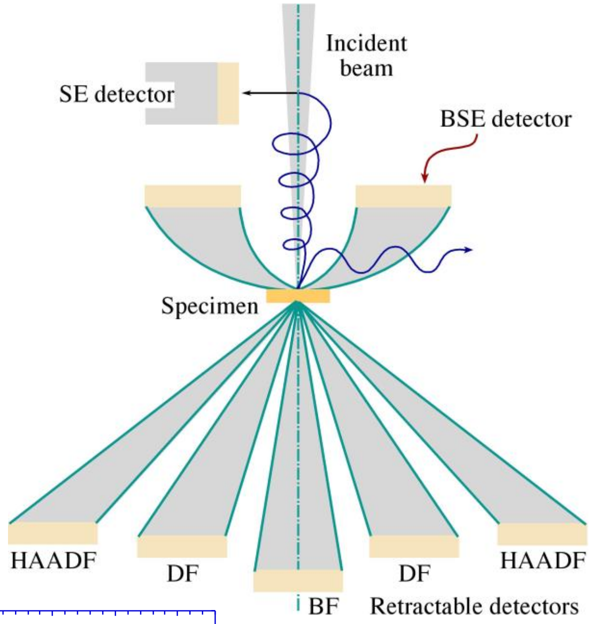
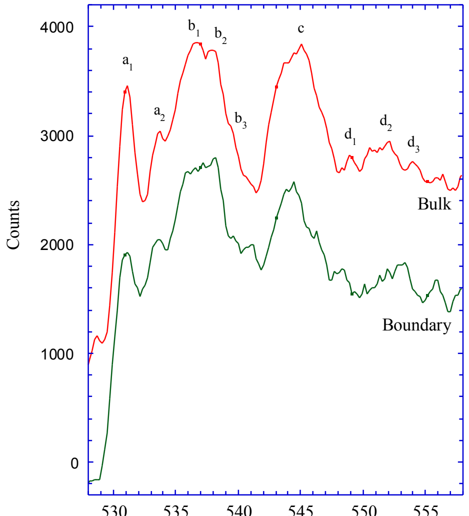
For most microelectronic FA work, STEM is used











Parts courtesy Nigel Browning

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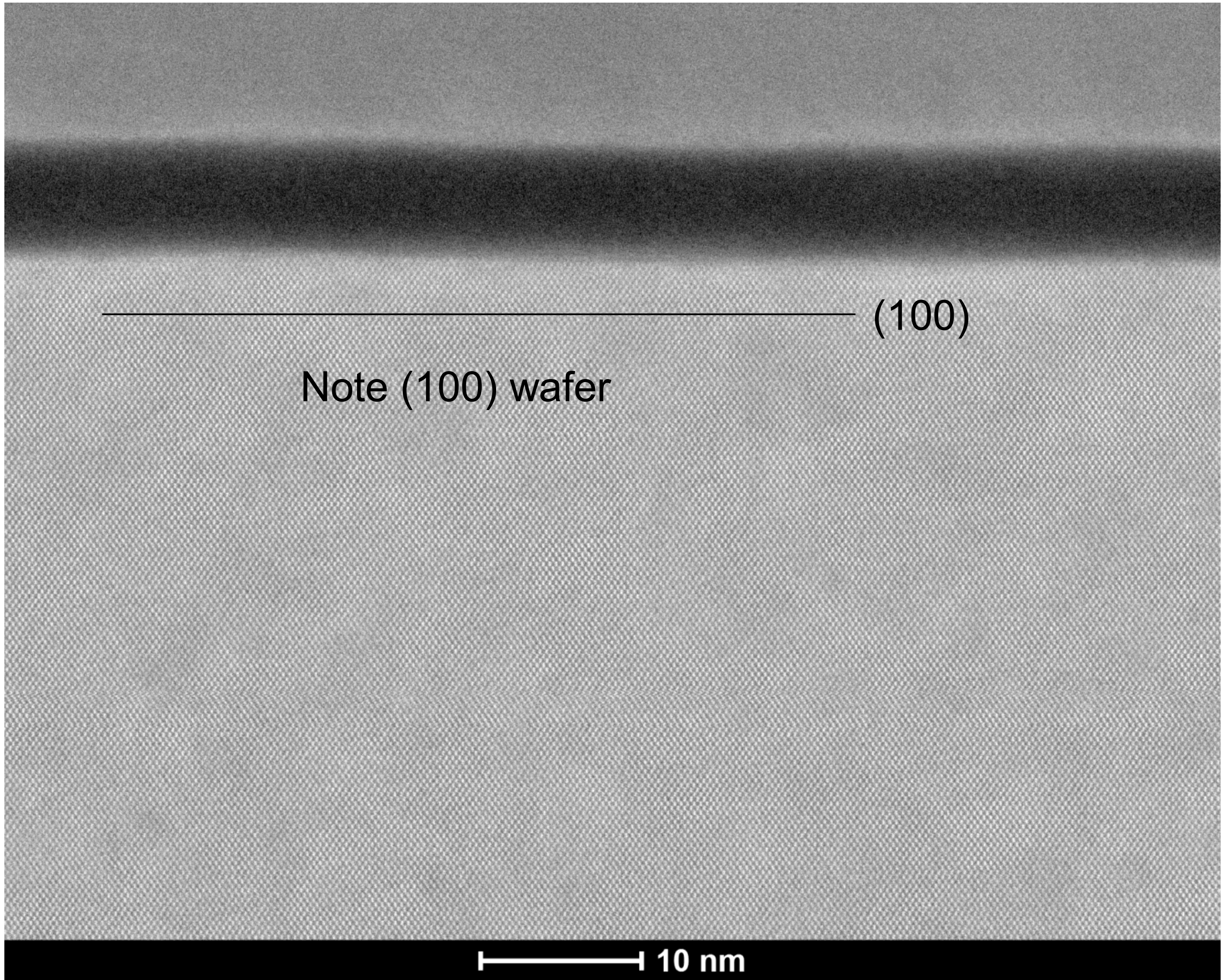
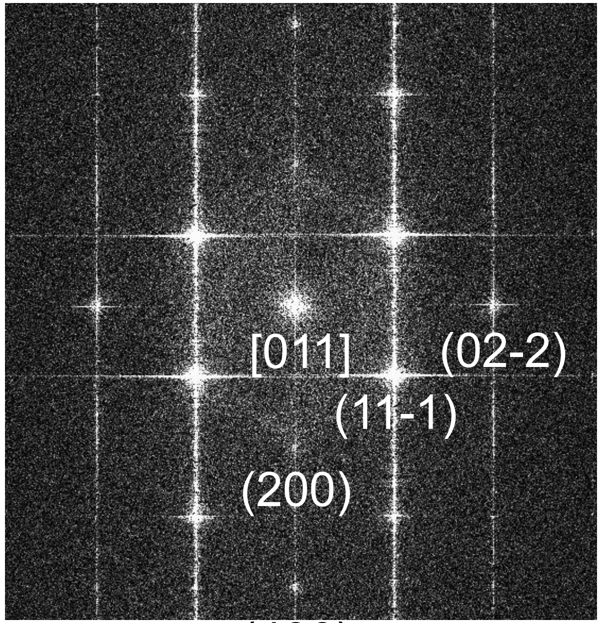
Metrology



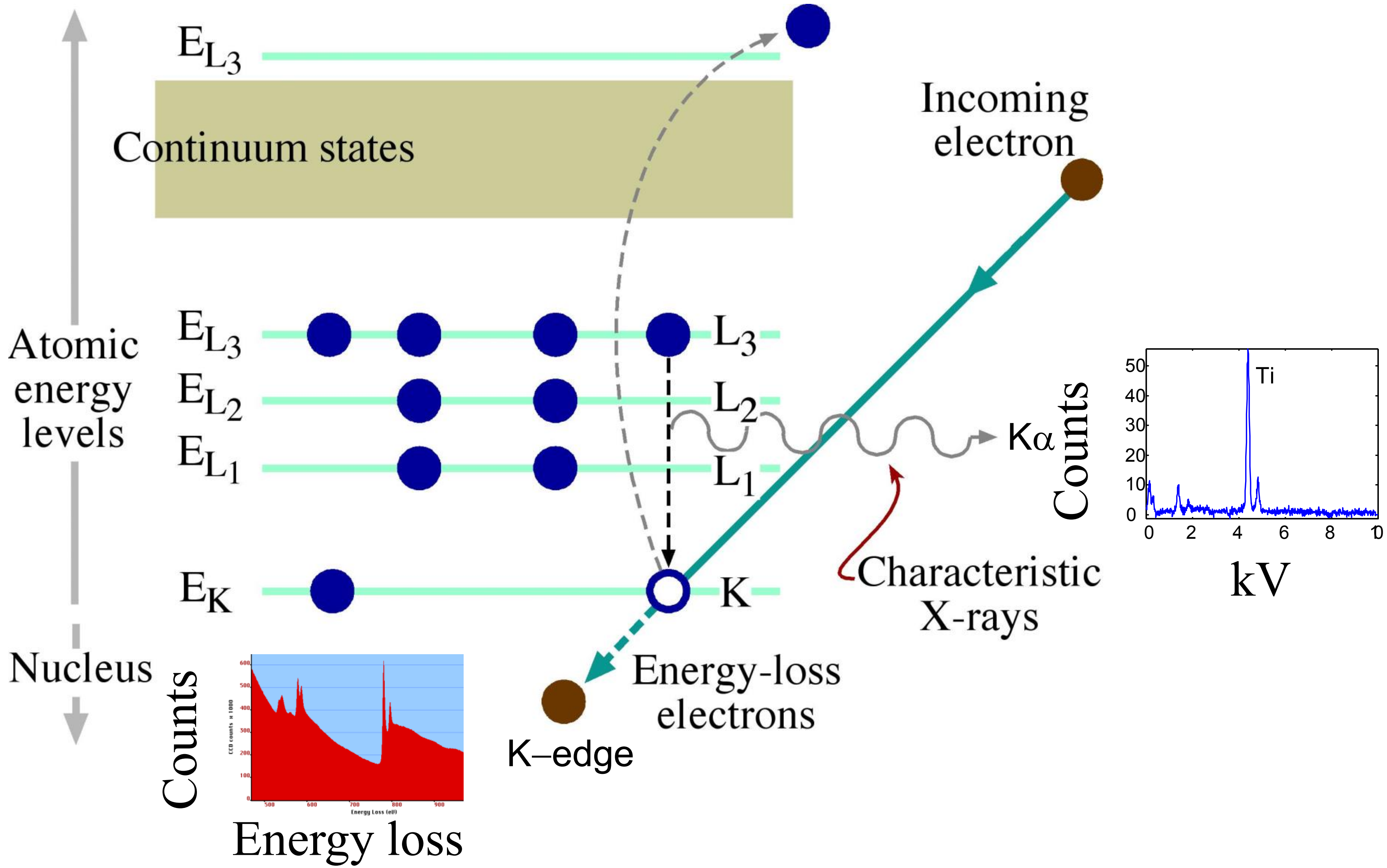
Critical dimensions can me measured, like an oxide layer thickness by using the Si lattice (bottom) observed along the [110] direction. NIST considers the spacings of the Si lattice as standard dimensions no matter how it's doped.



(400)

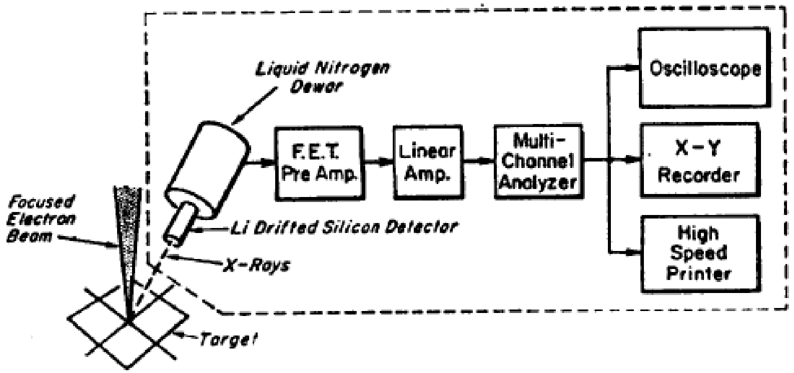
FFT of the lower Si

STEM-EDXS/EELS analytical signals



X-ray Detectors

Lithium-drifted Si reigned from 1968 until the early 2000's!



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NUCLEAR INSTRUMENTS & METHODS IN PHYSICS RESEARCH Section A

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Optimized readout methods of silicon drift detectors for high-resolution X-ray spectroscopy

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[b] MPI Halbleiterlabor, Otto-Hahn-Ring 6, 81739 Munich, Germany
[c] Max-Planck-Institut für Physik, 80805 Munich, Germany
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[e] Politecnico di Milano, Dipartimento di Elettronica e Informazione, Via Golgi 40, 20133 Milano, Italy

Available online 7 July 2006

The silicon drift detector…1984

SEMICONDUCTOR DRIFT CHAMBER – AN APPLICATION OF A NOVEL CHARGE TRANSPORT SCHEME

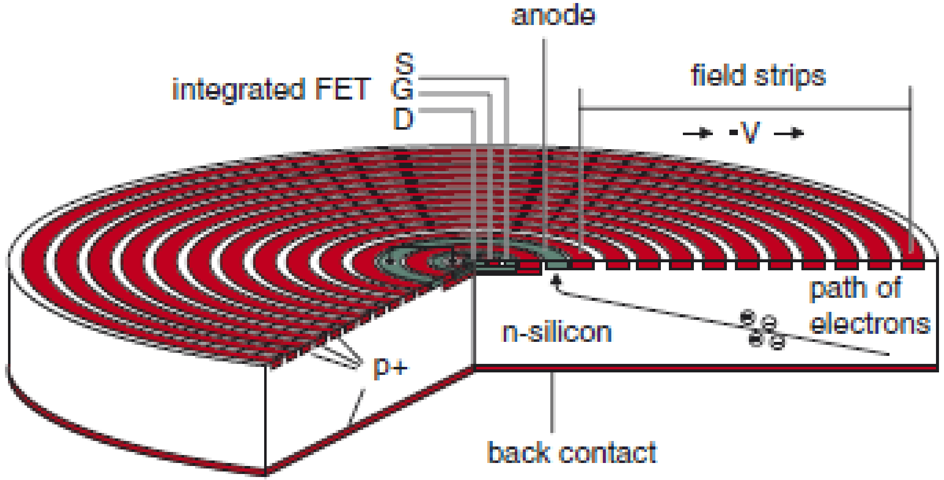
Emilio GATTI [1)] and Pavel REHAK

Brookhaven National Laboratory, Upton, New York 11973, USA

The purpose of this paper is to describe a novel charge transport scheme in semiconductors, in which the field responsible for the charge transport is independent of the depletion field. The application of the novel charge transport scheme leads to the following new semiconductor detectors:

1) Semiconductor drift chamber;
2) Ultralow capacitance – large area semiconductor X-ray spectrometers and photodiodes;
3) Fully depleted thick CCD.

Special attention is paid to the concept of the semiconductor drift chamber as a position sensing detector for high energy charged particles. Position resolution limiting factors are considered and the values of the resolutions are given.



Finally perfected in 2006!

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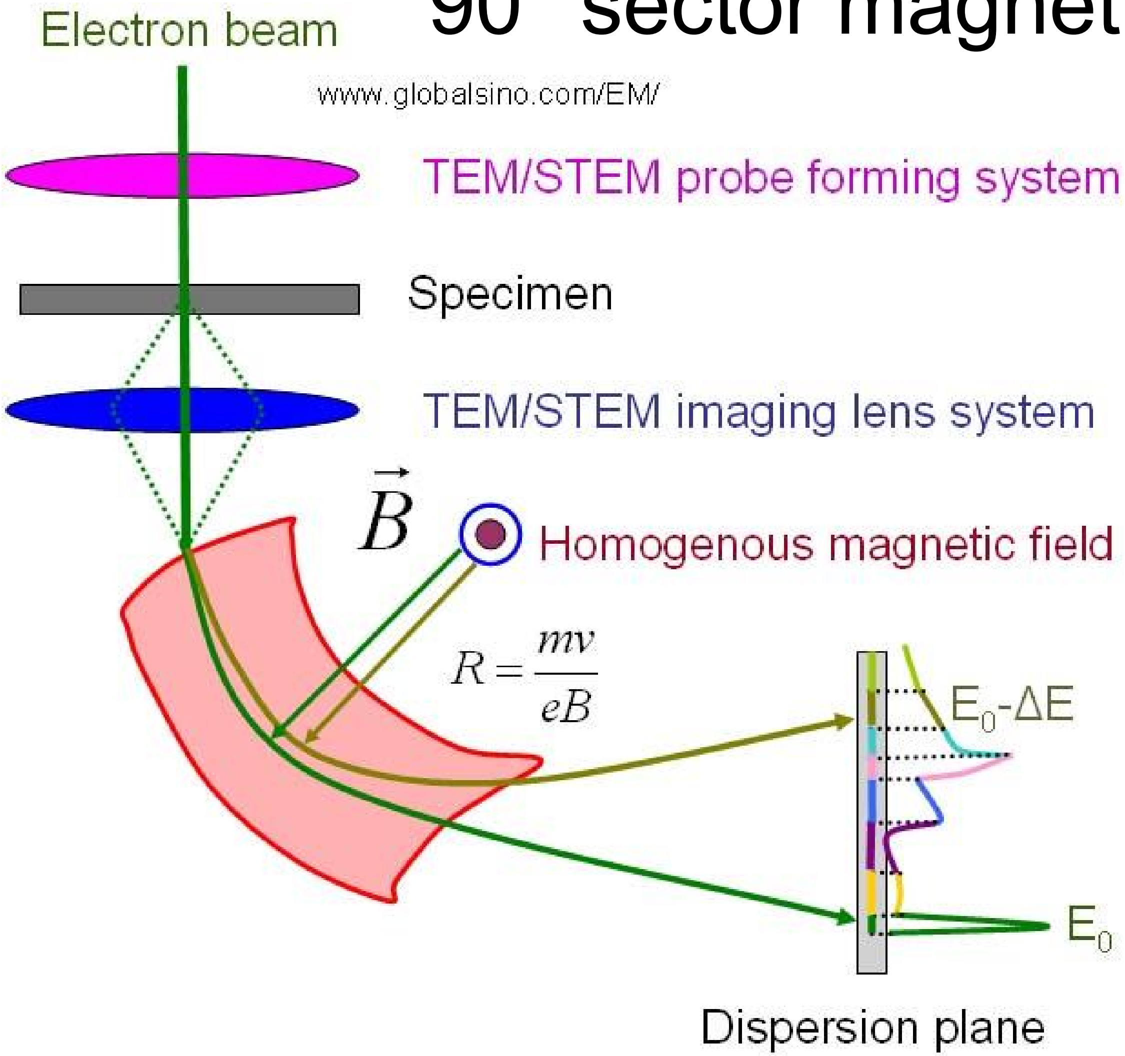
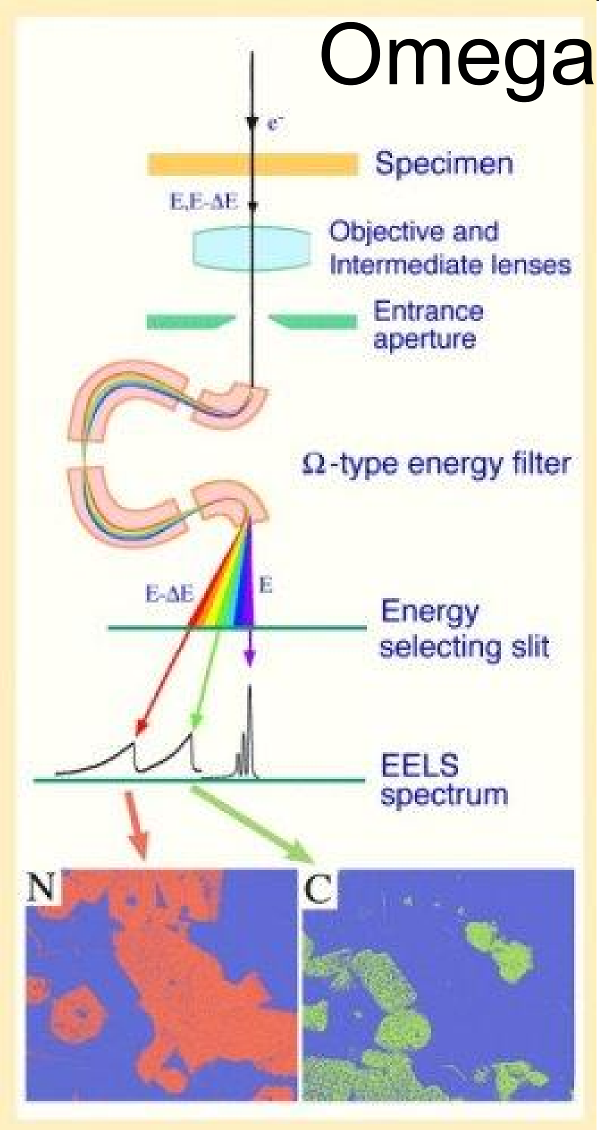
EELS Spectrometers

90° sector magnet



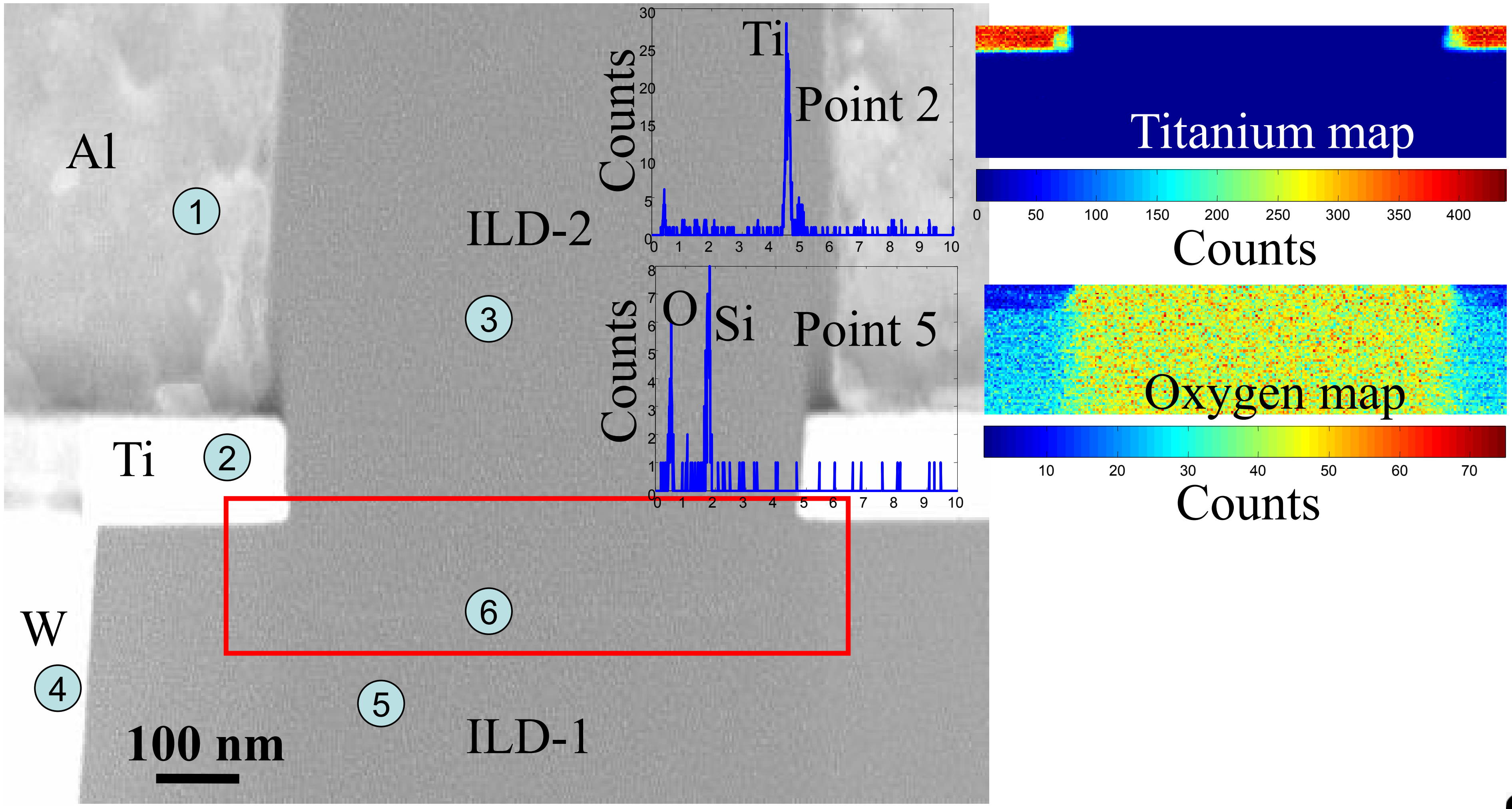
Omega



EDXS versus EELS

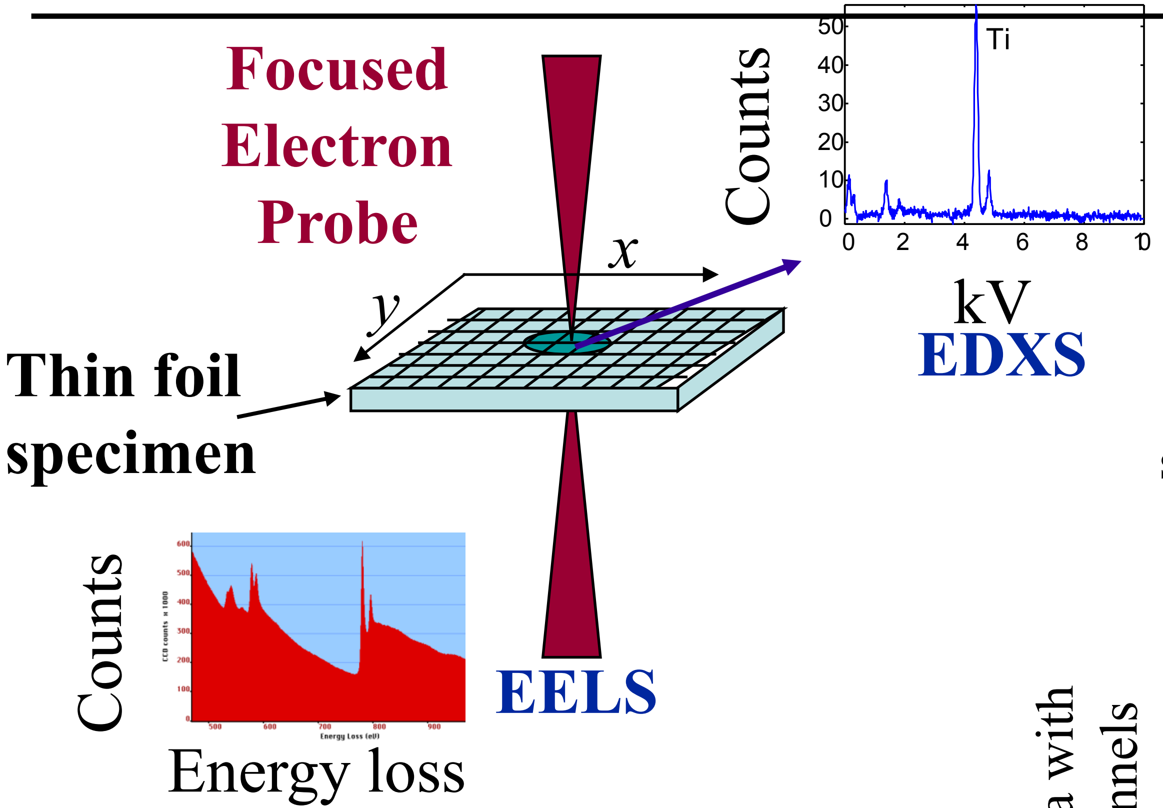
- EDXS covers most of the periodic table (B and above but B, C, N, O, F K-X rays are strongly absorbed in even thin samples). The signals for harder X-rays are readily quantified.
- EELS wins for light elements. The edge energy and ELNES, energy-loss near-edge structure, are sensitive to local chemical environment. The low-loss spectrum can be used (with care) to measure band gaps.

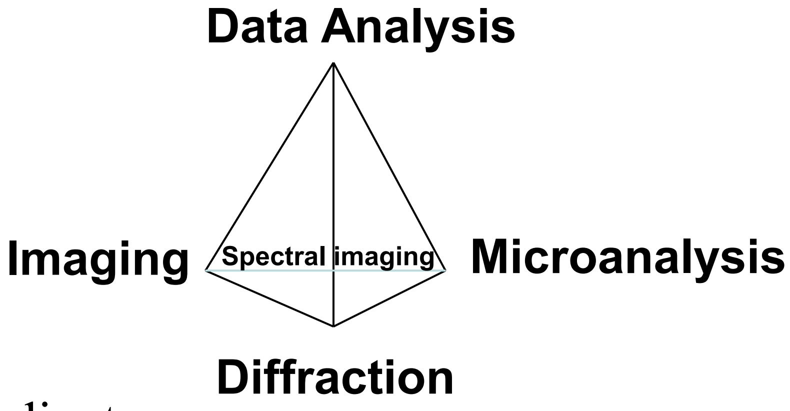
STEM-EDS sampling: Points & Maps

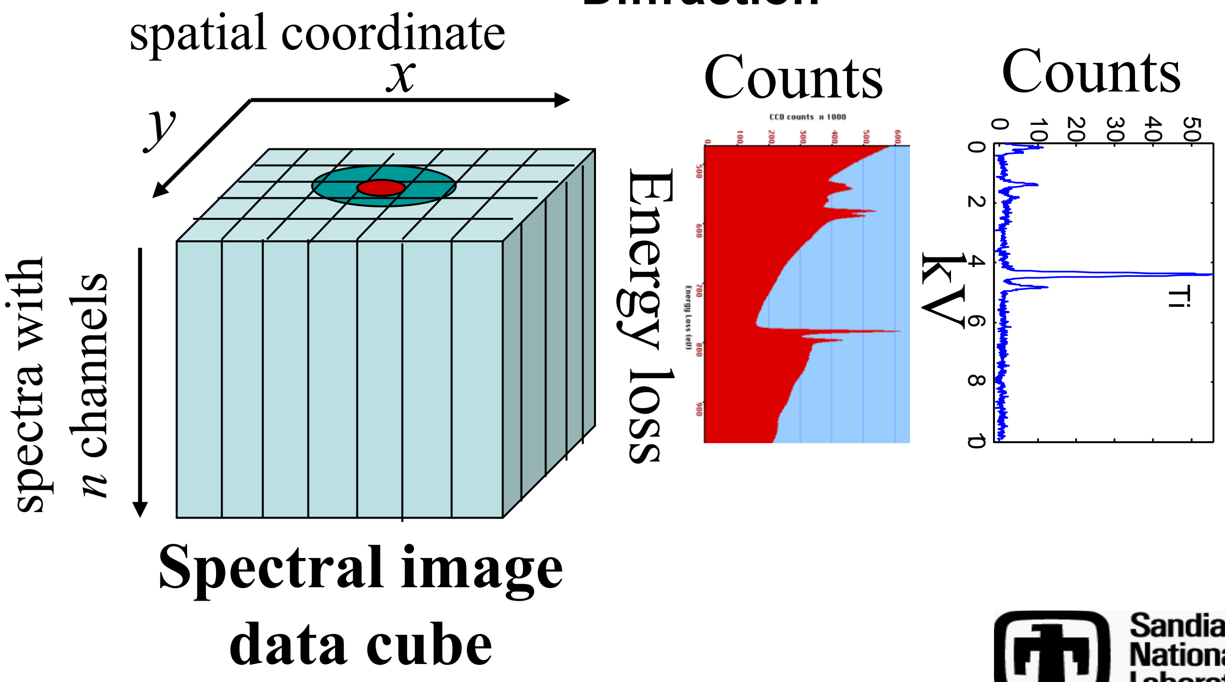


P.G. Kotula, et al. *Microsc. Microanal.* **12** [6] 538-544.

AEM Spectral Imaging



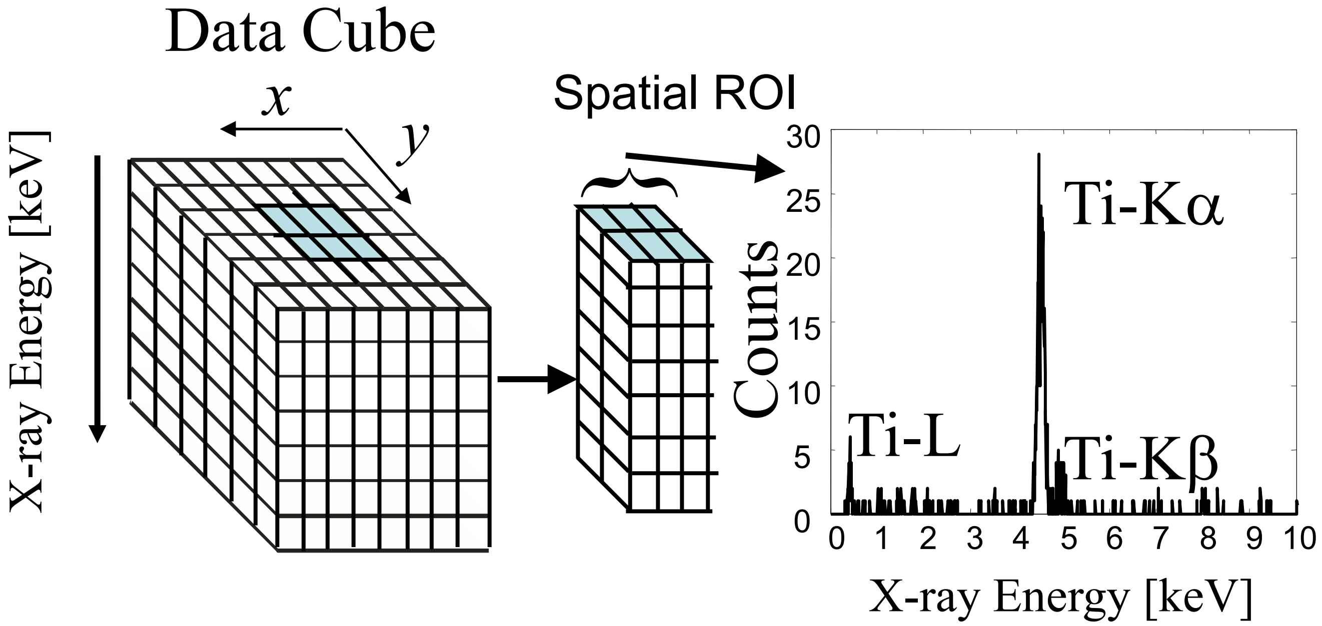


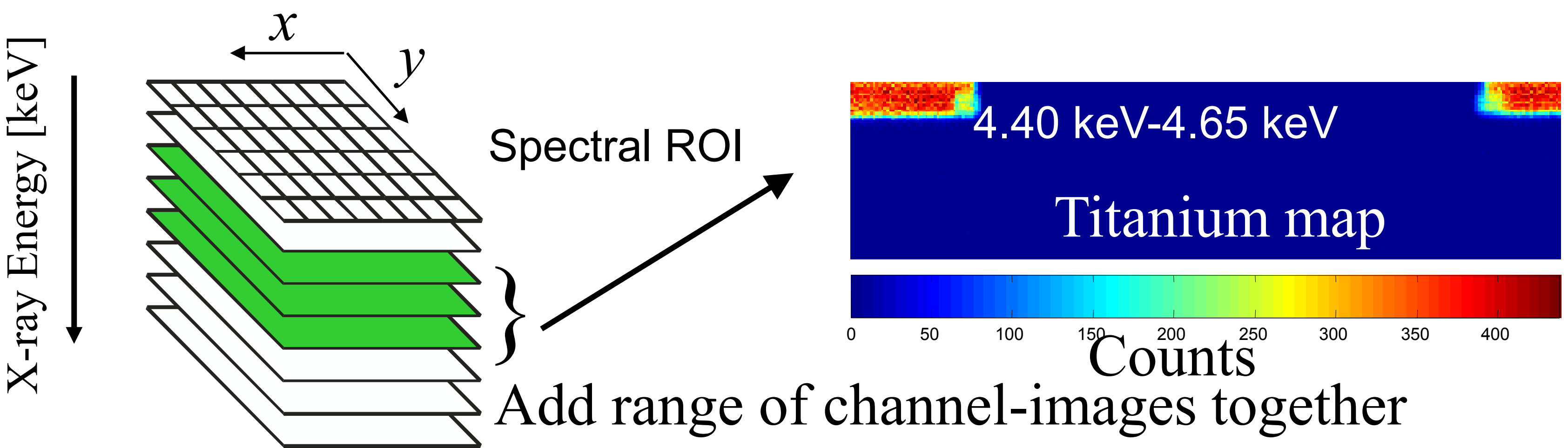


AEM is analytical electron microscopy, basically STEM-EDS

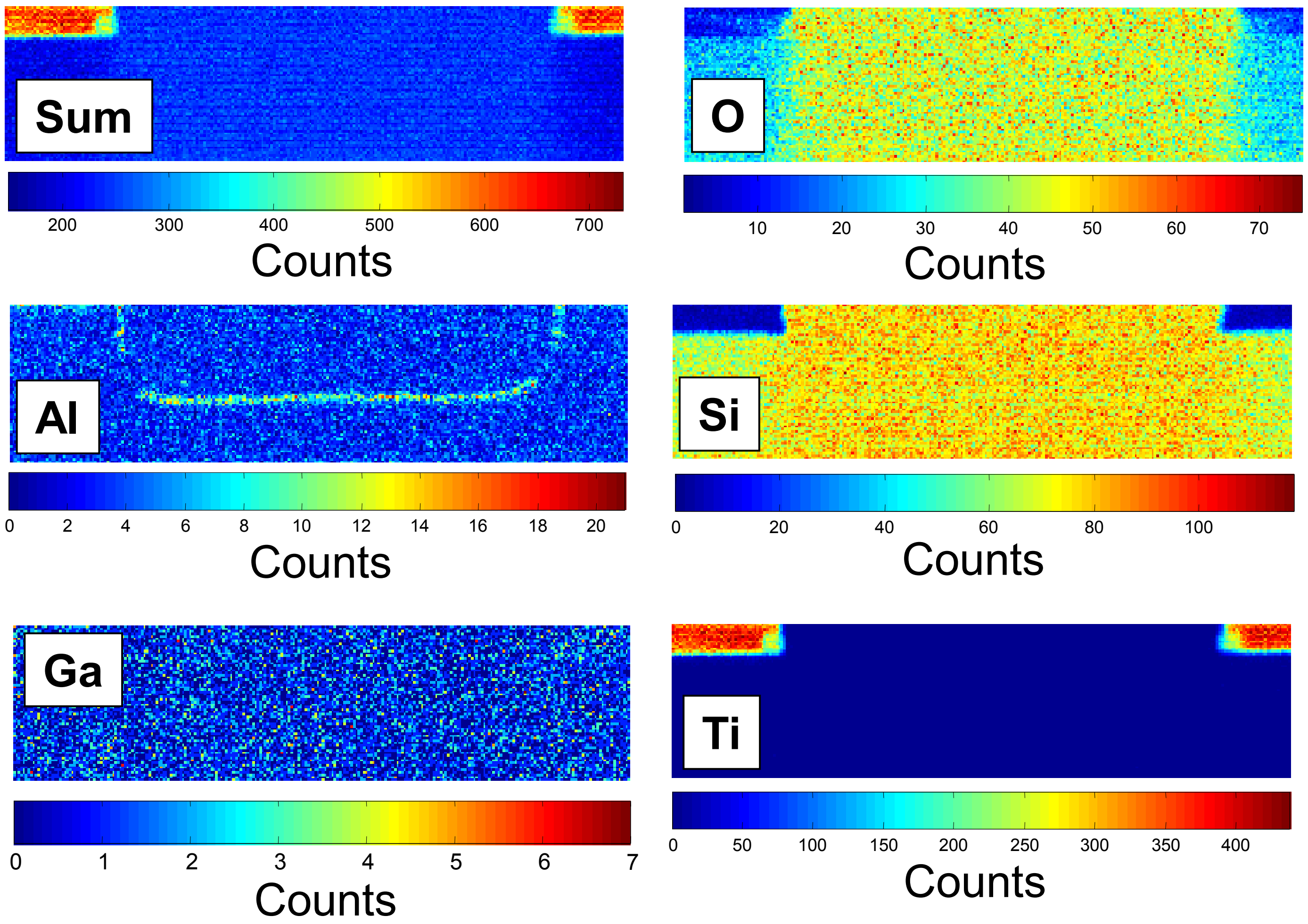
P.G. Kotula, et al. *Microsc. Microanal.* **12** [6] 538-544.

Conventional Data Analysis





Example of Conventional Analysis



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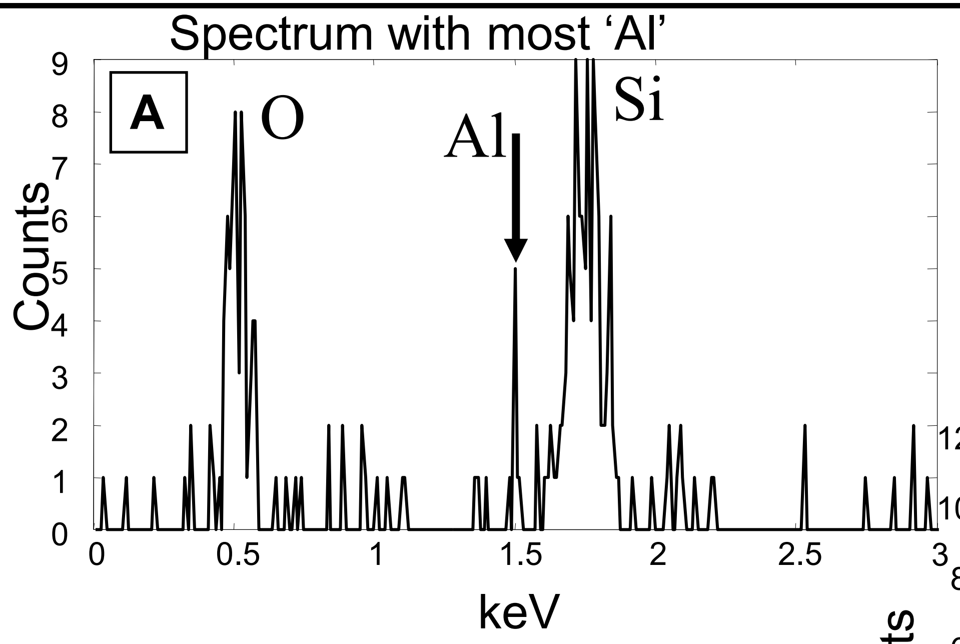
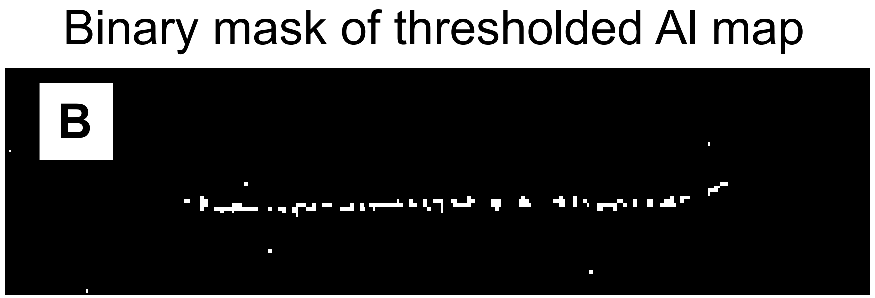
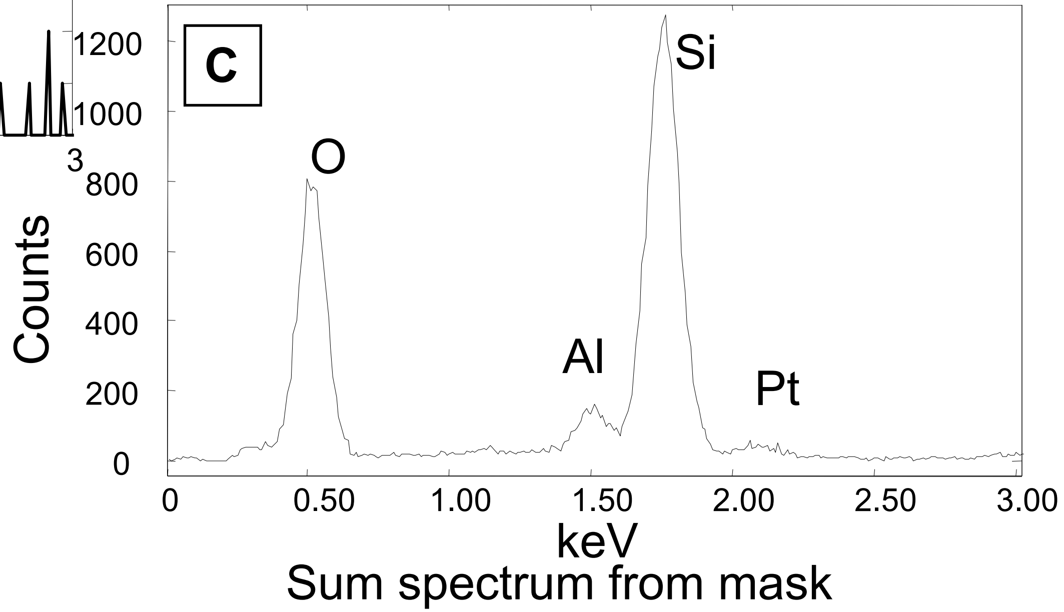
Example of Conventional Analysis







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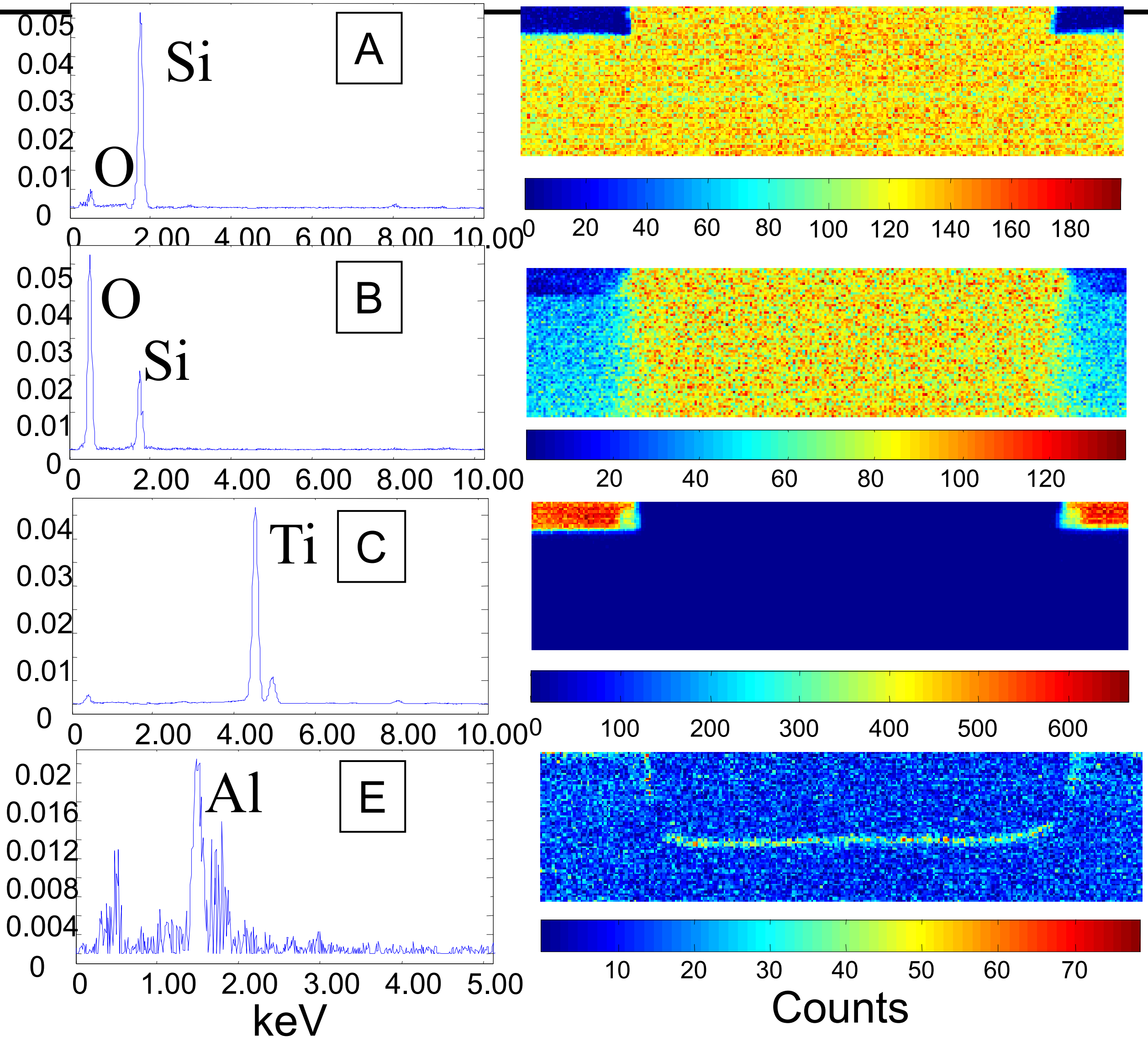
Multivariate statistical analysis of the same data



A few seconds of computation

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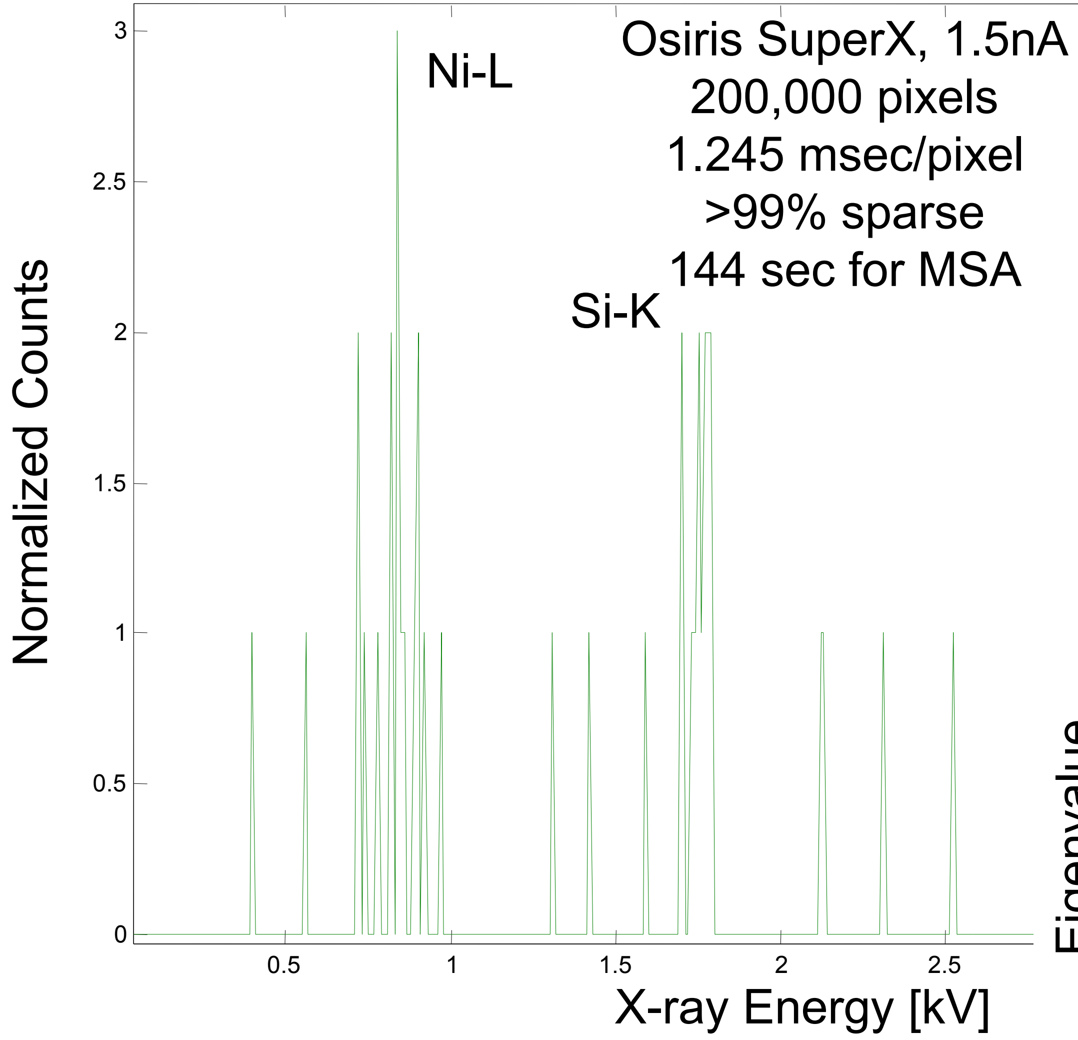
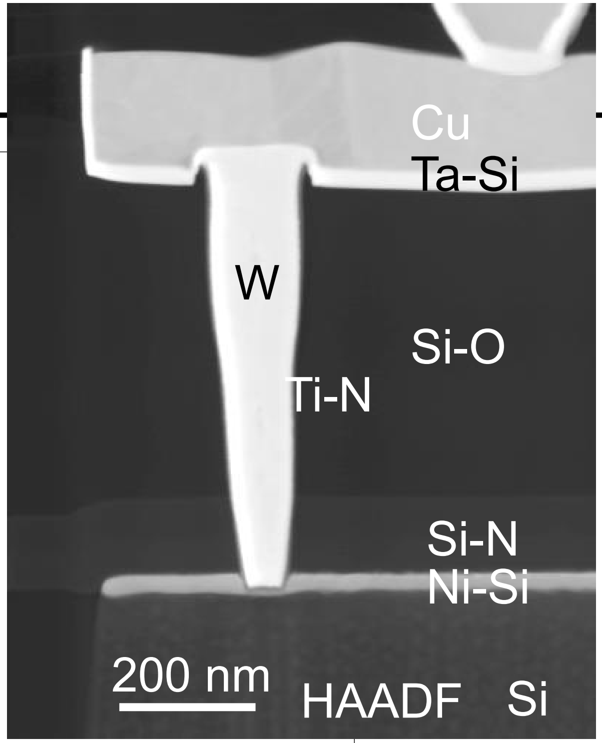
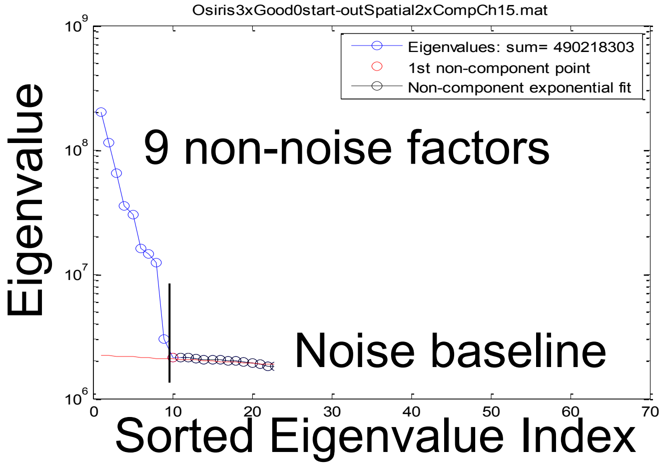
The Al was found at the interface between two of the dielectric layers, providing a current leakage path.

Raw spectrum from a CMOS spectral image

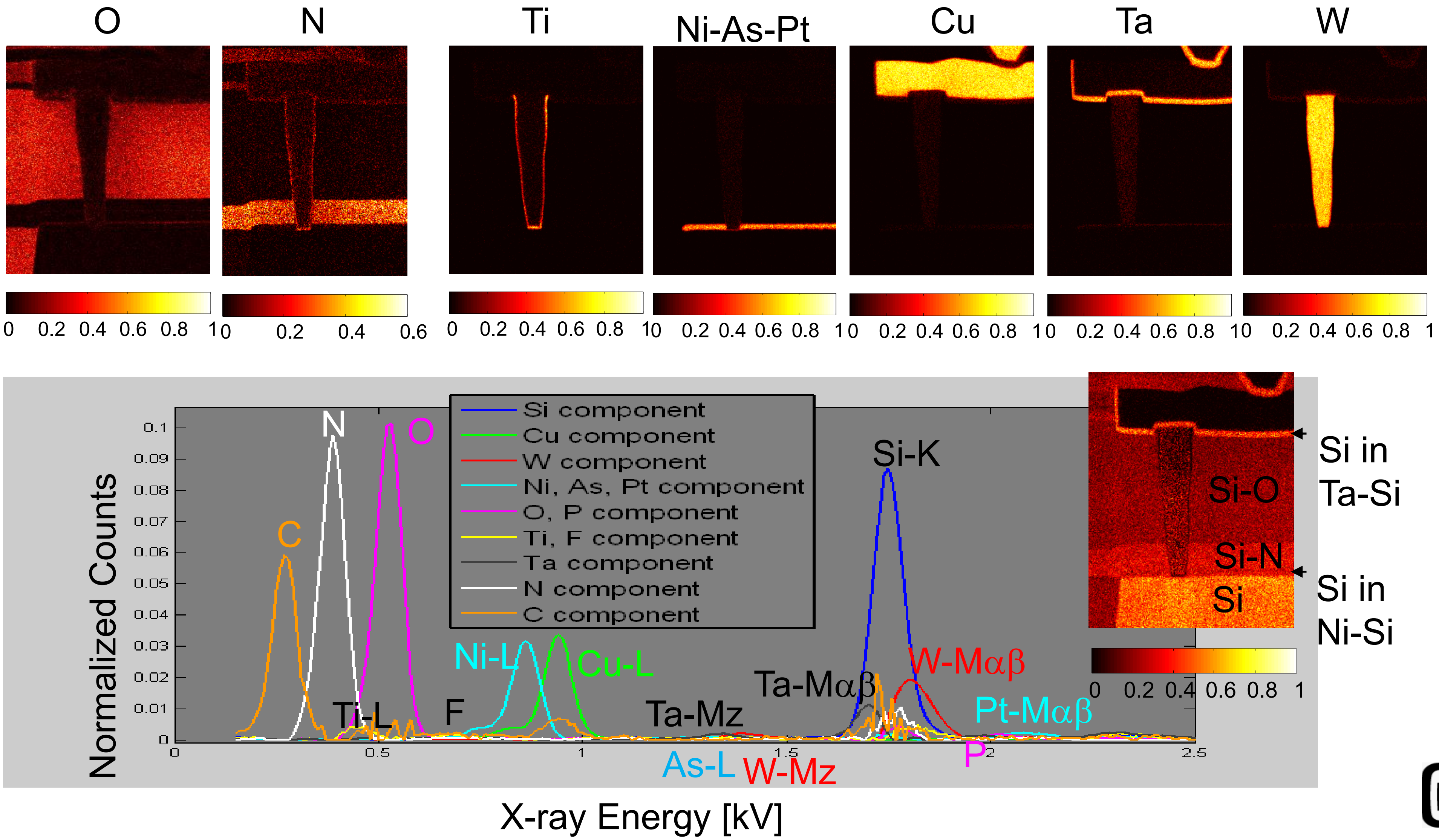






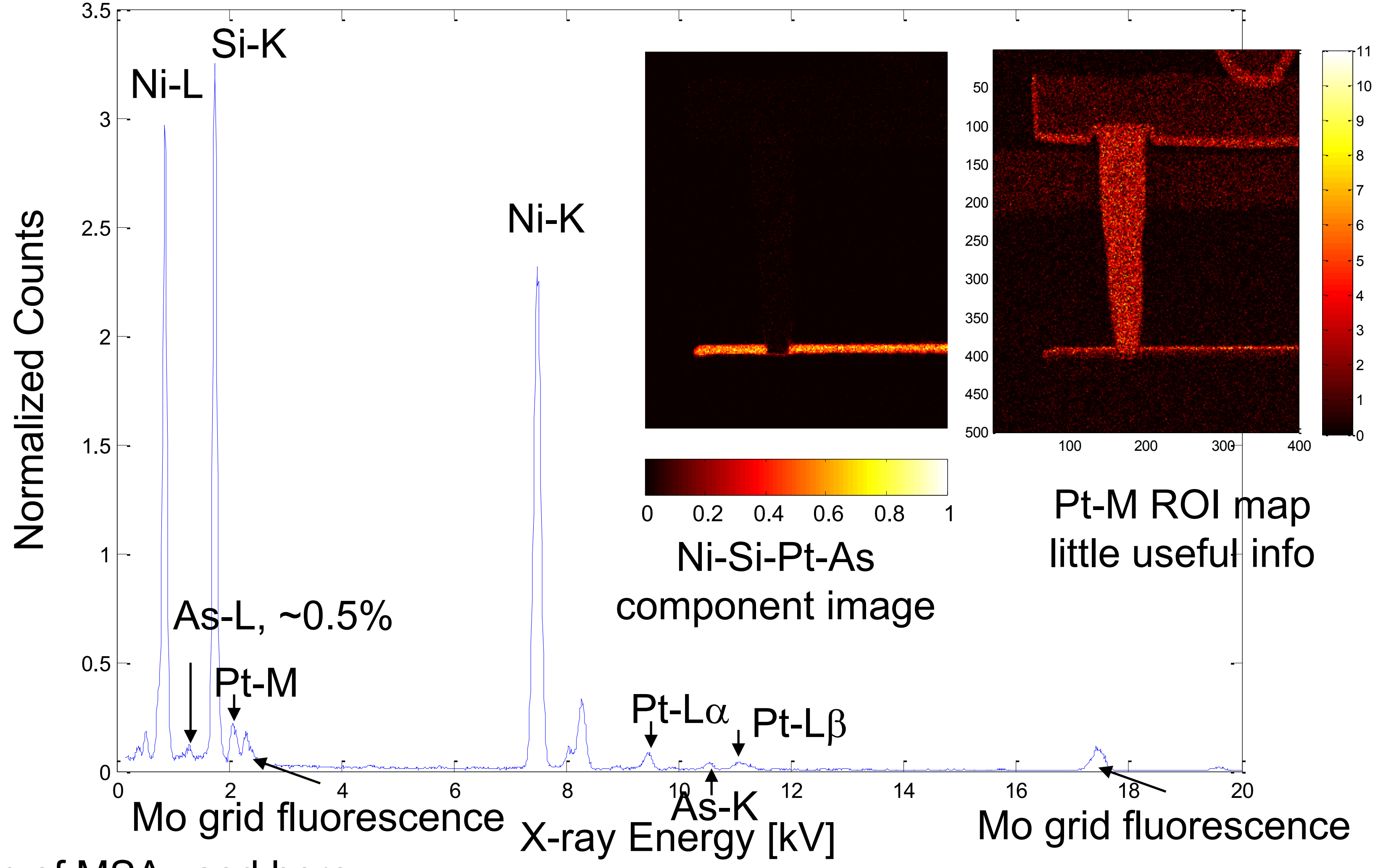
Data courtesy Dmitri Klenov, FEI on sample provide by SNL

Results of MSA on this noisy spectral image



MSA shows minor elements

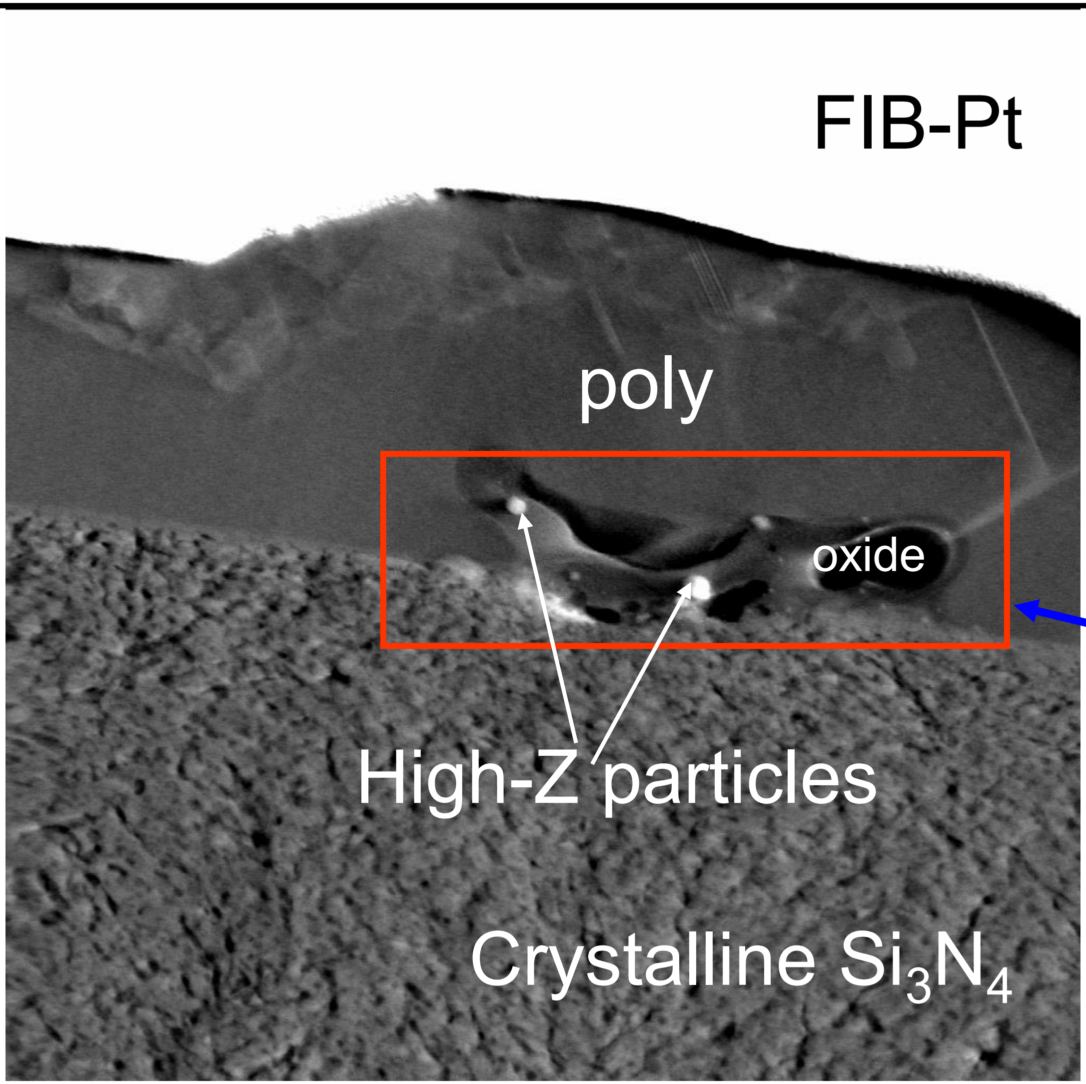
Note Pt is added to stabilize the $NiSi_2$ while As comes from the underlying doping of the Si



A different variation of MSA used here

Microelectronics process problem: Crystallizing Si-N

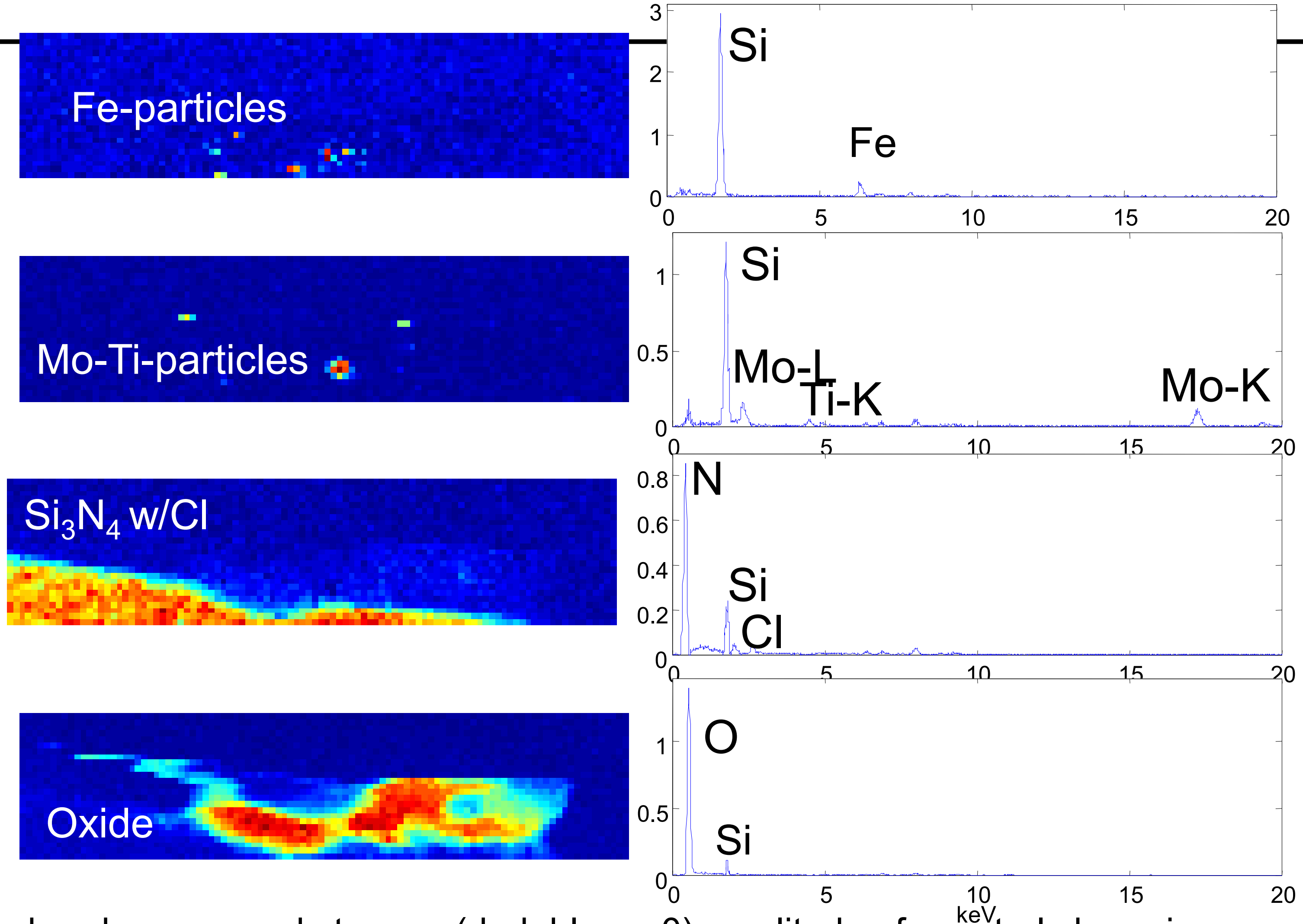
HAADF STEM image of the nucleation site



Region of SI
100 x 400 nm

Microelectronics process problem: crystallizing Si-N

Component Images and Spectra from MSA



Dark red corresponds to max (dark blue = 0) amplitude of spectral shape in pure component images. Spectral intensities are normalized units.

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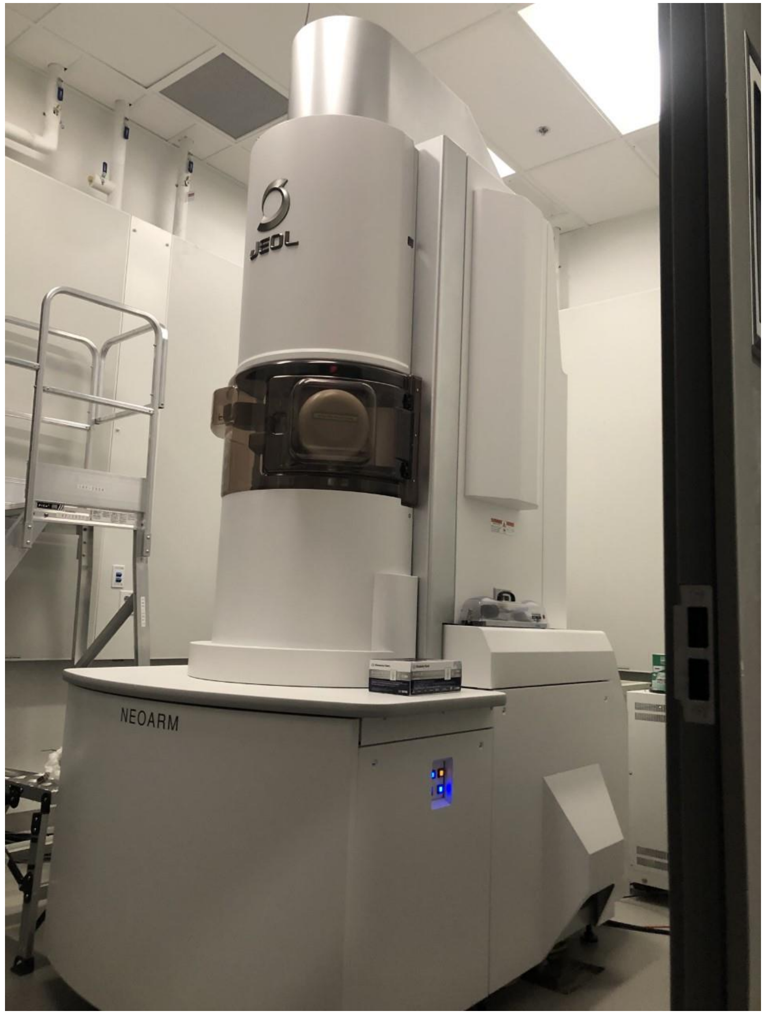
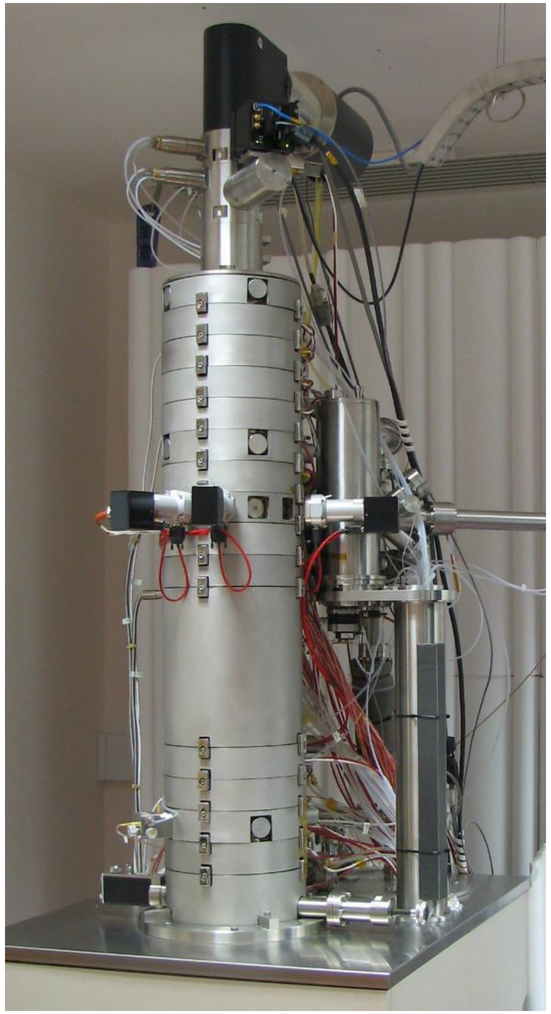
Different Flavors of Corrected STEMs



JEOL NEOARM at UNM

FEI Titan at Sandia



NION STEM

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Spherical Aberration Correction

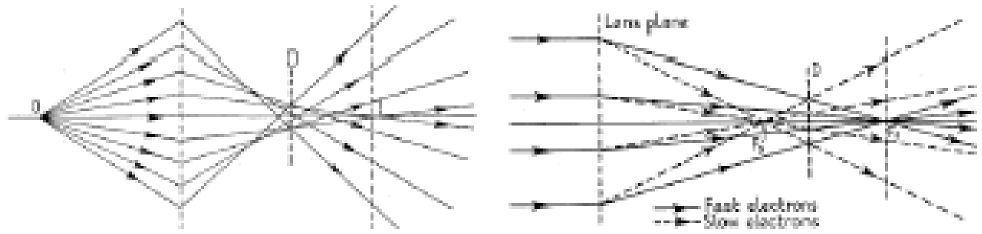
Sherzer's Theorum

O. Scherzer, *Z. Phys.* **101** (1936) 593

Round electromagnetic lenses suffer from spherical and chromatic aberration

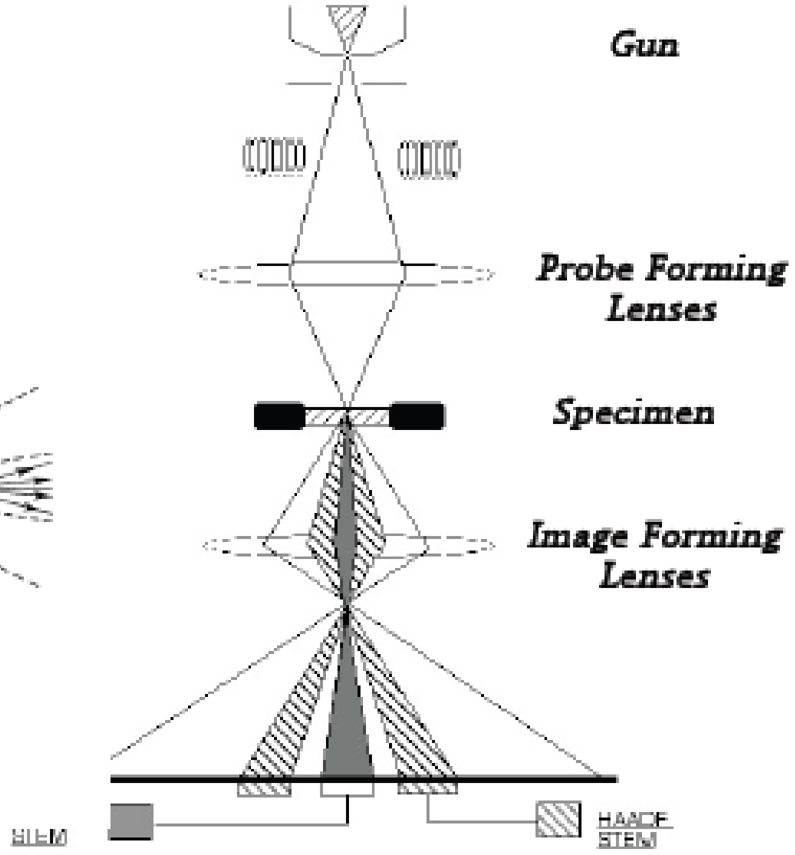
Aberrations

- Spherical
- Chromatic



$$r_{sph} = C_s \beta^3 \qquad r_{chr} = C_c \frac{\Delta E}{E} \beta$$

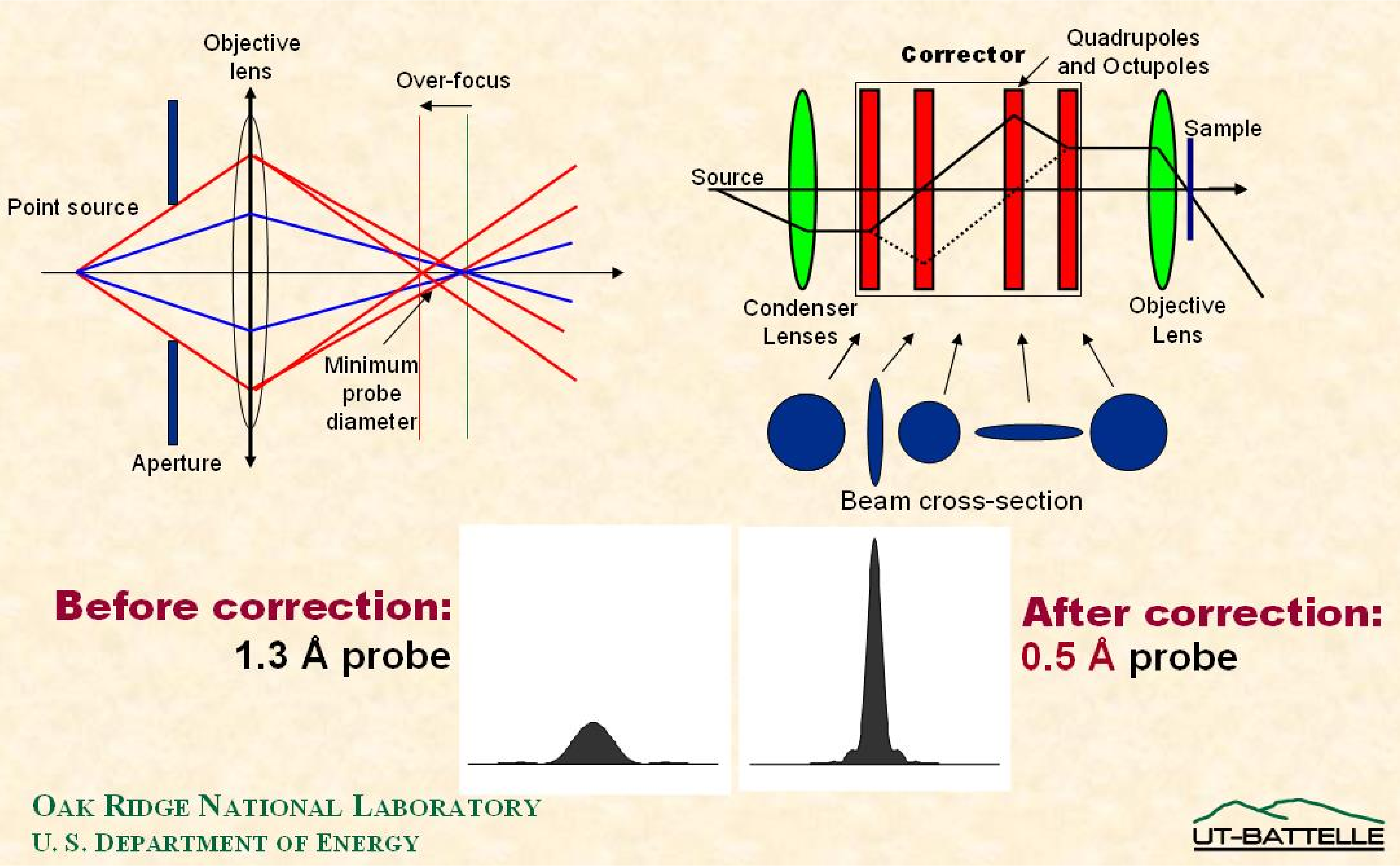
Courtesy Nestor Zaluzec, ANL



At high voltages, like 200kV, ΔE/E is small so chromatic aberration isn't limiting.
At 40 kV-60kV that's not true any more which is why monochromation helps here, also chromatic aberration correctors

Spherical aberration correction: Break round symmetry

http://www.ornl.gov/~grg/BES_sym/talks/Pennycook.ppt#386,9,2002: NION builds successful STEM aberration corrector



NION uses quadrupoles and octupoles. CEOS uses hexapoles and makes correctors for Thermo and Jeol.

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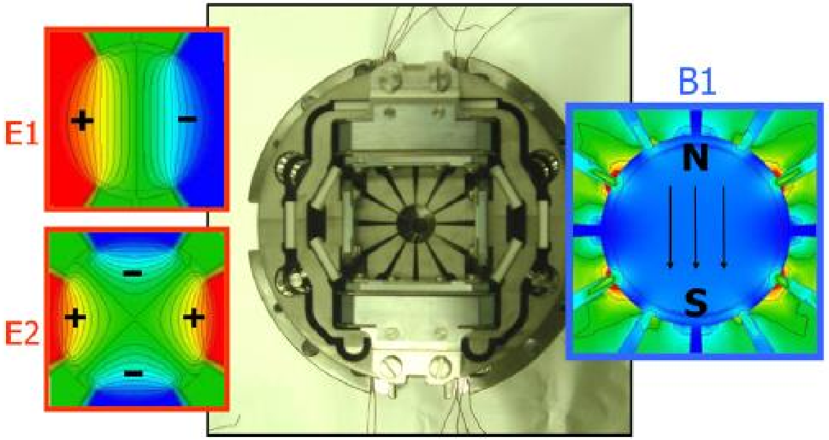
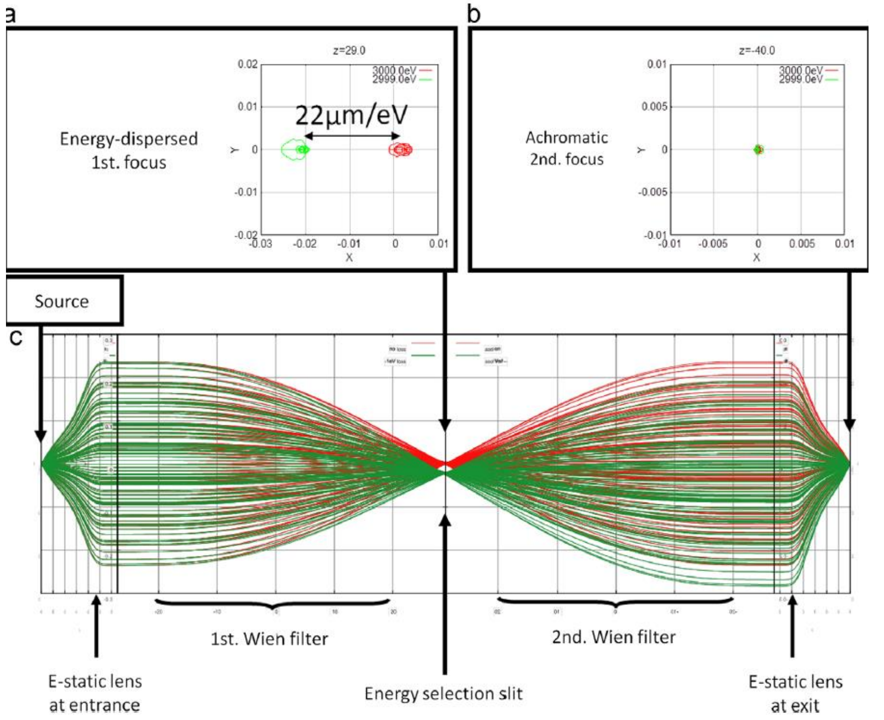
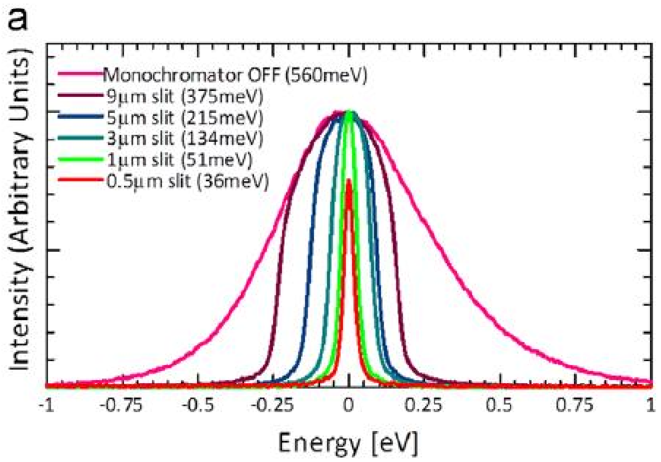
Monochromation

- The Wien filter is uses orthogonal electric and magnetic fields, so that particles with the correct speed will be unaffected while other particles will be deflected



The monochromator design used in the UNM NEOARM

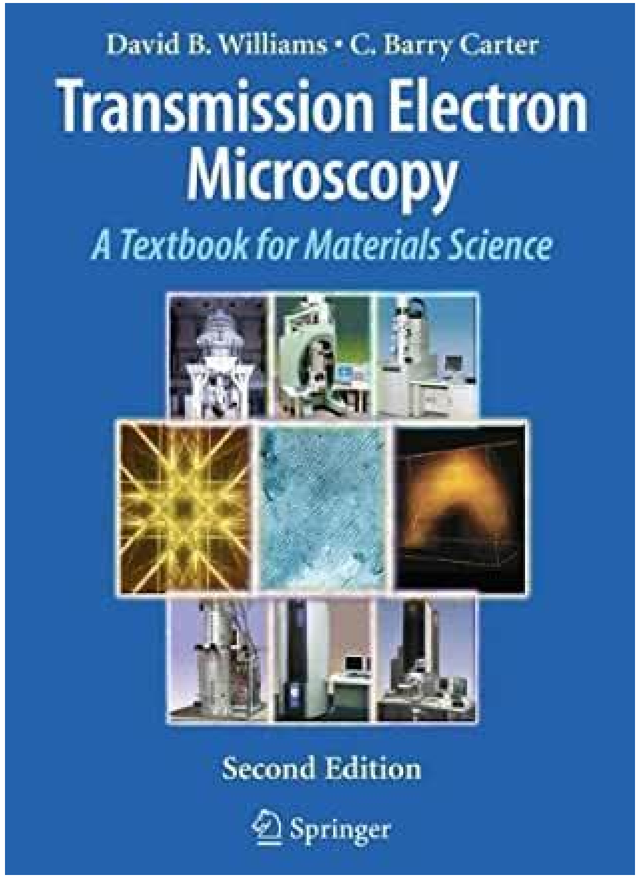




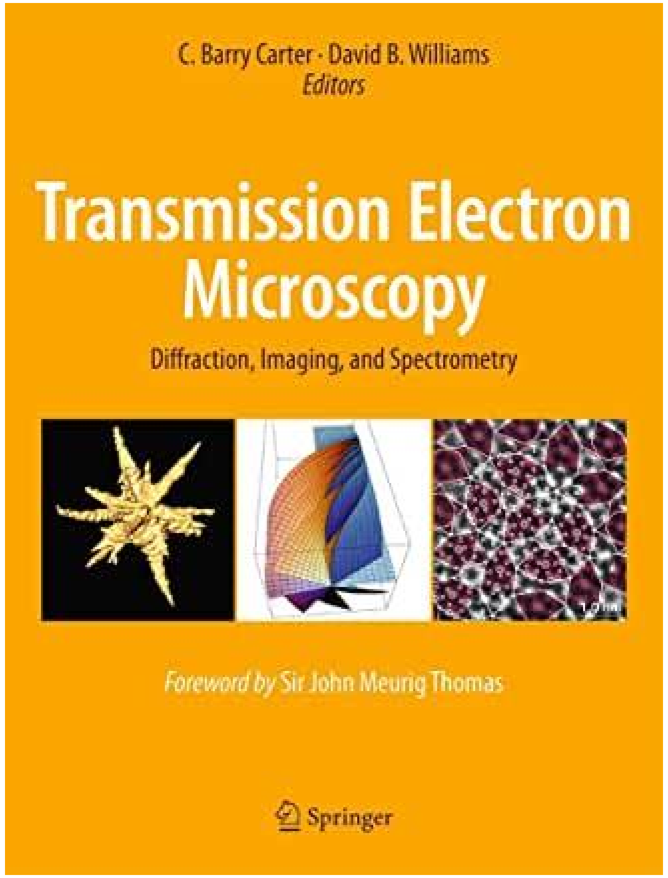
M. Mukai et al. / Ultramicroscopy 140 (2014) 37–43

Resources

- Williams and Carter/ Carter and Williams

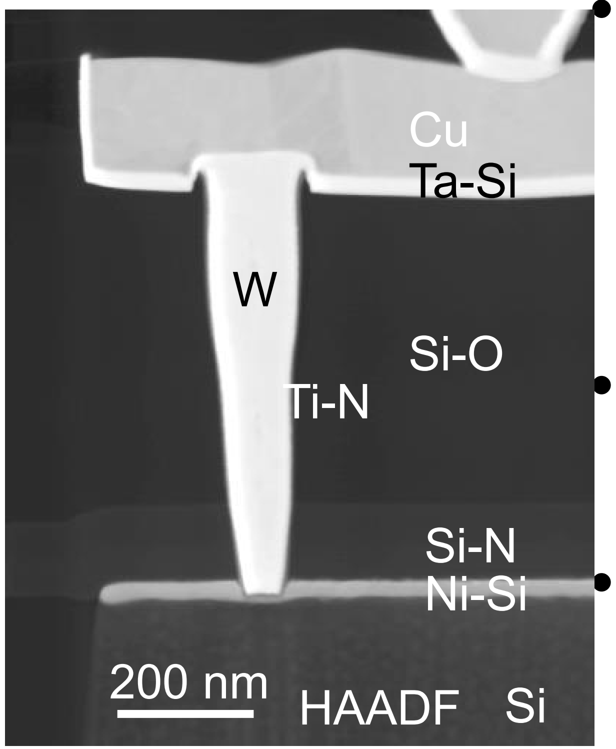


All the background and basics you'll need.

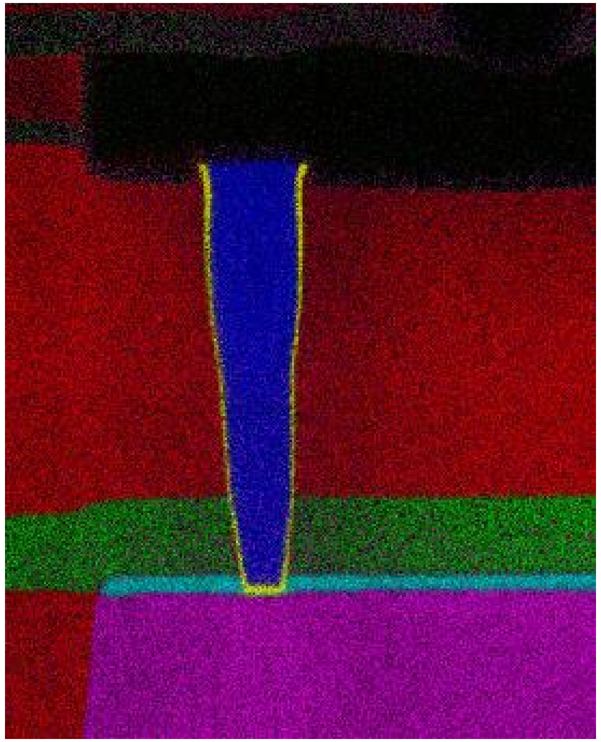


Advanced techniques and practical instructions.
e.g., Chapter 16: X-ray and EELS Imaging, written by me…

Conclusions



- TEM/STEM are powerful and versatile tools for materials characterization including failure analysis covering microns to sub-Å
- Automation and data analytics are the present and future
- Sandia has had MSA-based data analytics for over 20 years that are still used commercially and in research
- Future UNM side to my research
- Paul.Kotula@sandia.gov



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