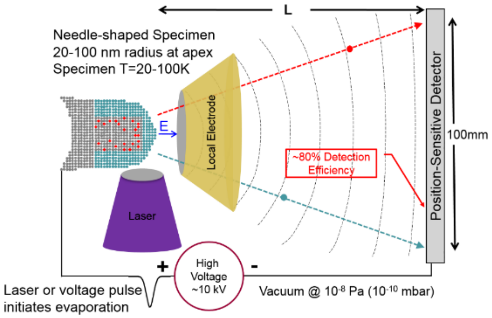
Advances in Atom Probe Metrology

Karen T. Henry – Intel Corporation

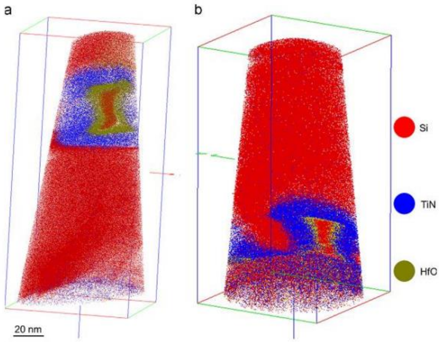
Thomas F. Kelly – CAMECA Instruments, Inc.

Introduction

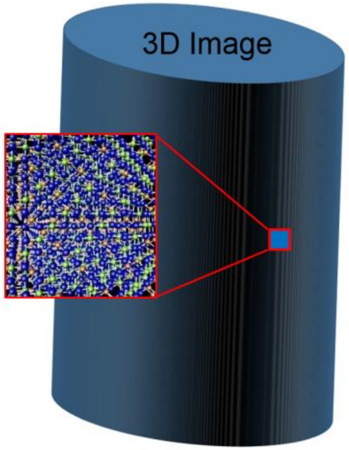


Current Status

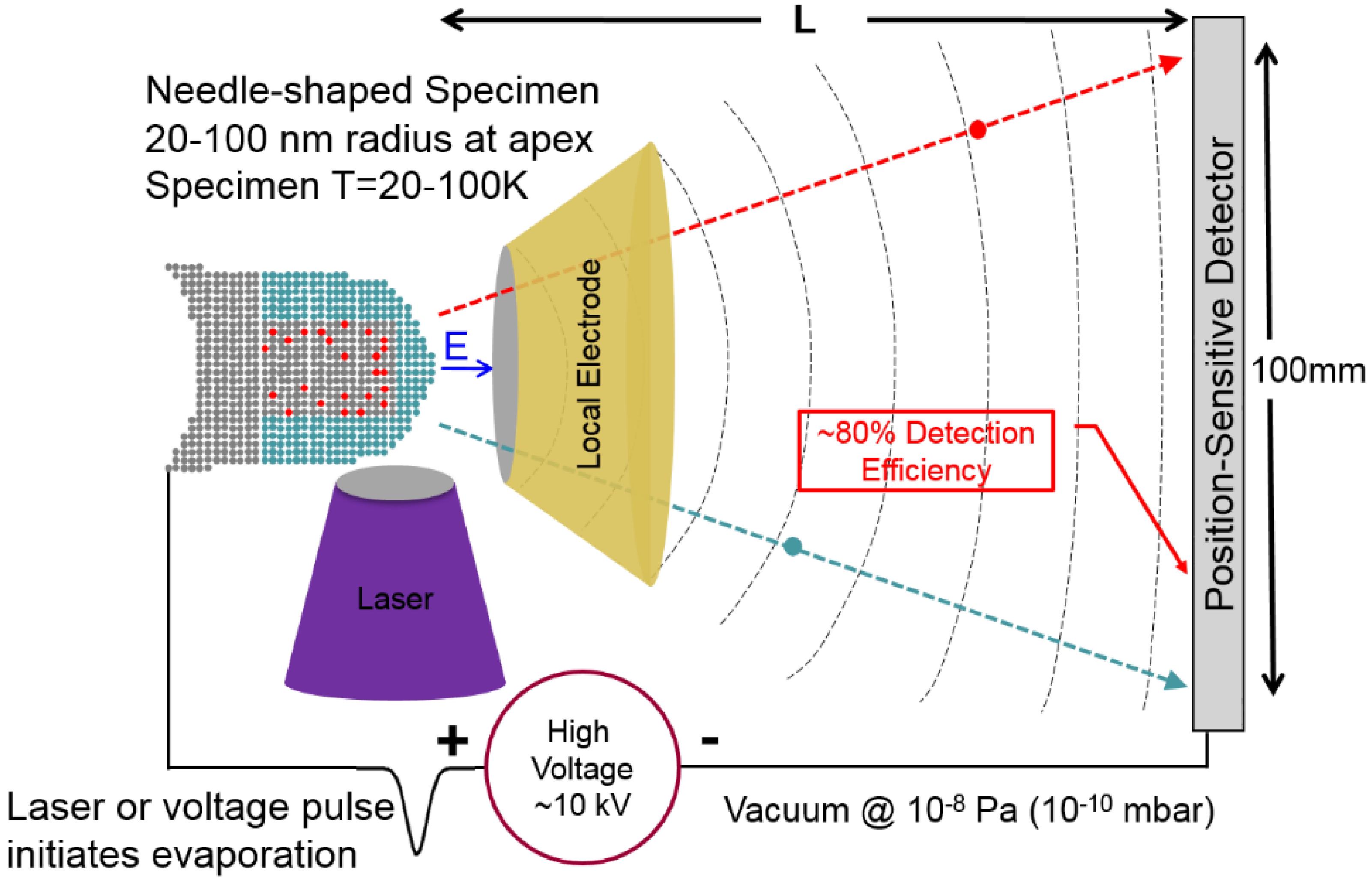
Current Applications



Roadmap for Improvement

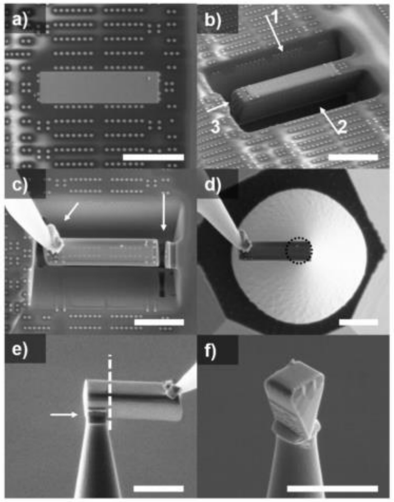


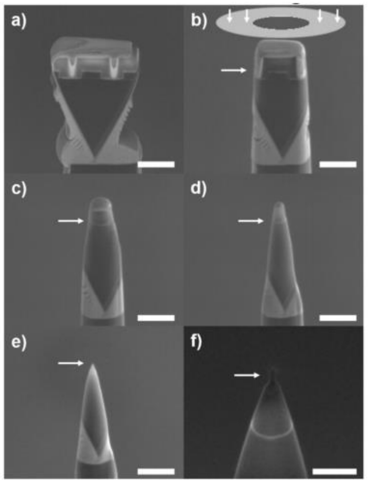
Essentials of How It Works



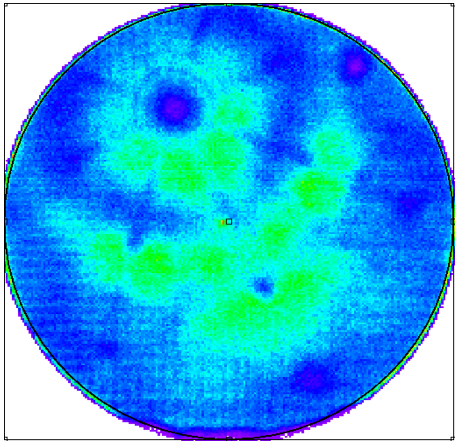
Typical Application Flow

Sample Preparation

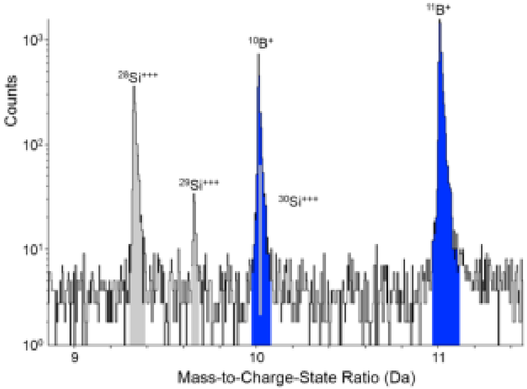


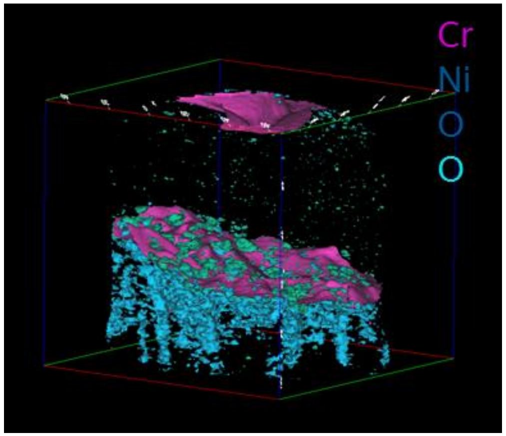


Data Acquisition



Data Interpretation





Current Status of APT

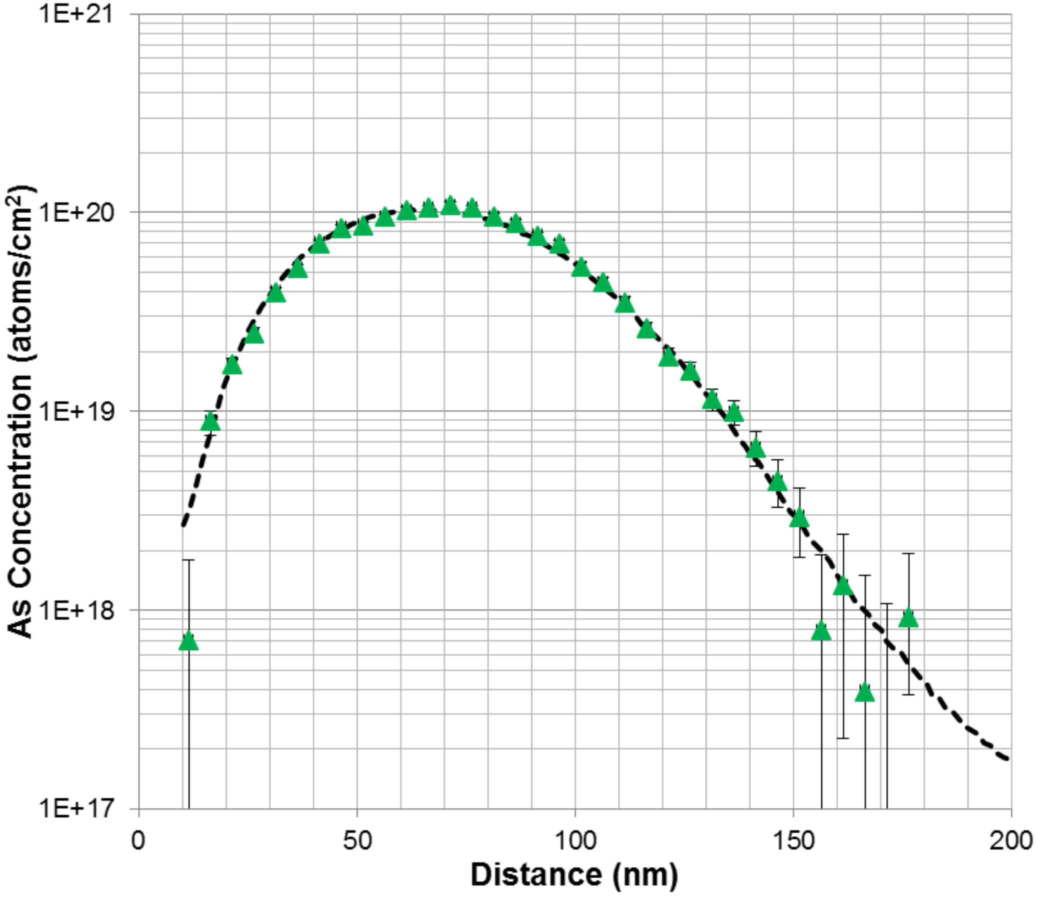
Strengths of APT	Limitations of APT
Discrete 3-dimensional image	Not all materials will run well
High analytical spatial resolution (0.3 nm)	Limited field of view (<300 nm)
High analytical sensitivity (<10 appm)	Reconstruction distortions for heterogeneous materials
Specimen preparation is similar to TEM	High detection efficiency ~80% (but not 100%)
All atoms detected with equal efficiency	Crystallographic information is limited
High detection efficiency (>80%)	No chemical bonding information
	Time to data

- 3D Imaging
- Local composition, spatial relationships and dimensions
- Quantification of low Z materials such as boron & carbon

Current Applications

Dopant Profiling - Precision/Accuracy

NIST SRM 2134

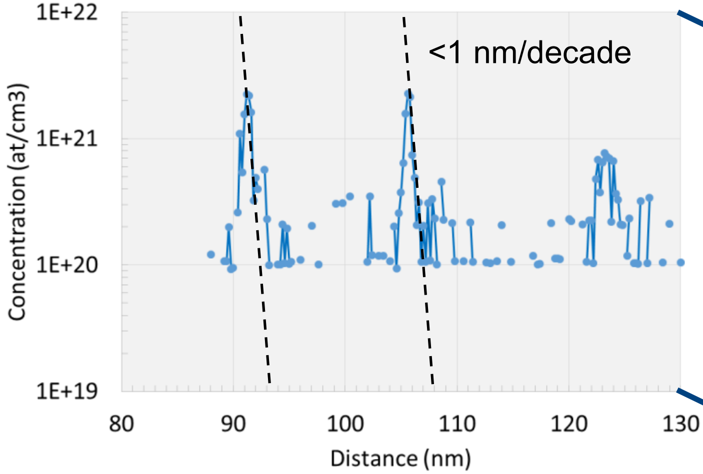


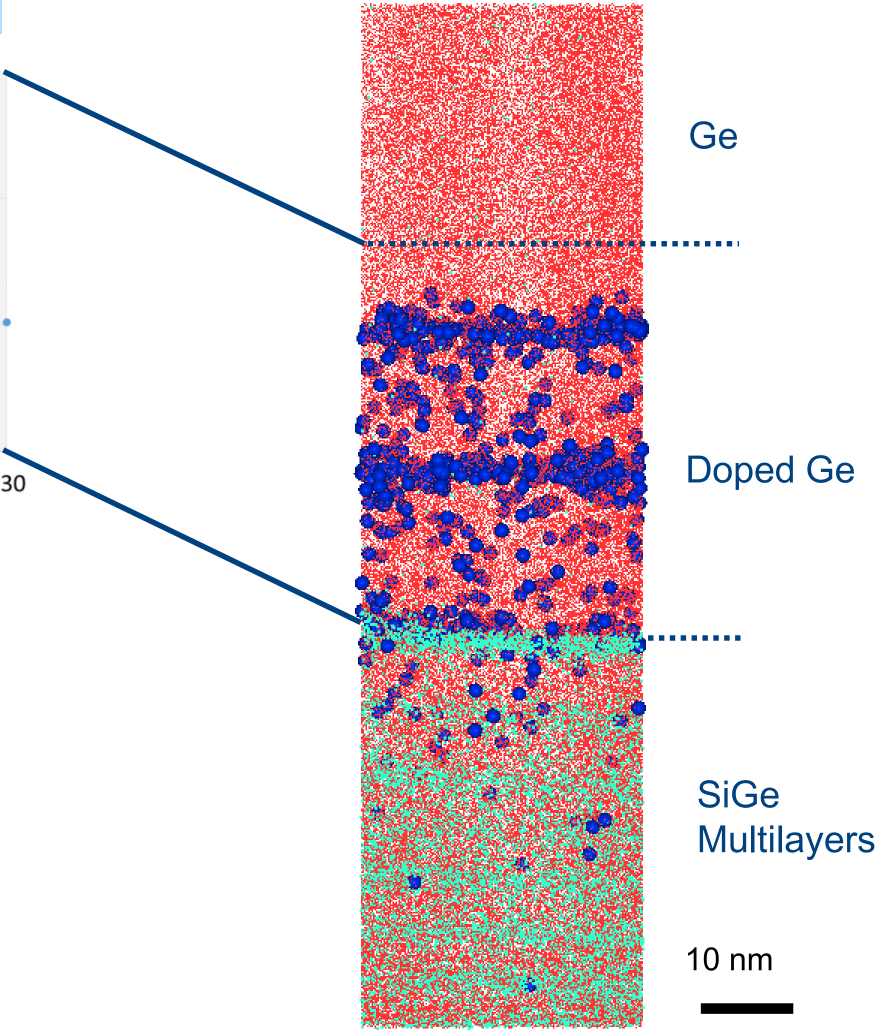
- Characterization of individual dopant atoms is critical as device dimensions continue to scale
- APT can provide both accurate and precise measurements of dose
- Specimen shape and instrumental factors must be taken into consideration for high precision measurements

Prosa et al., *Ultramicroscopy* **132** (2013)

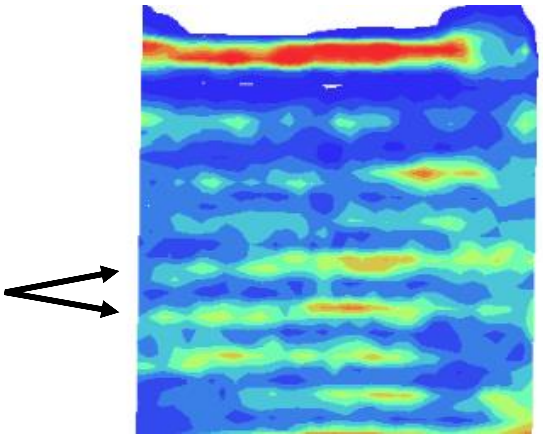
Dopant Profiling and Elemental Mapping

Depth Profile



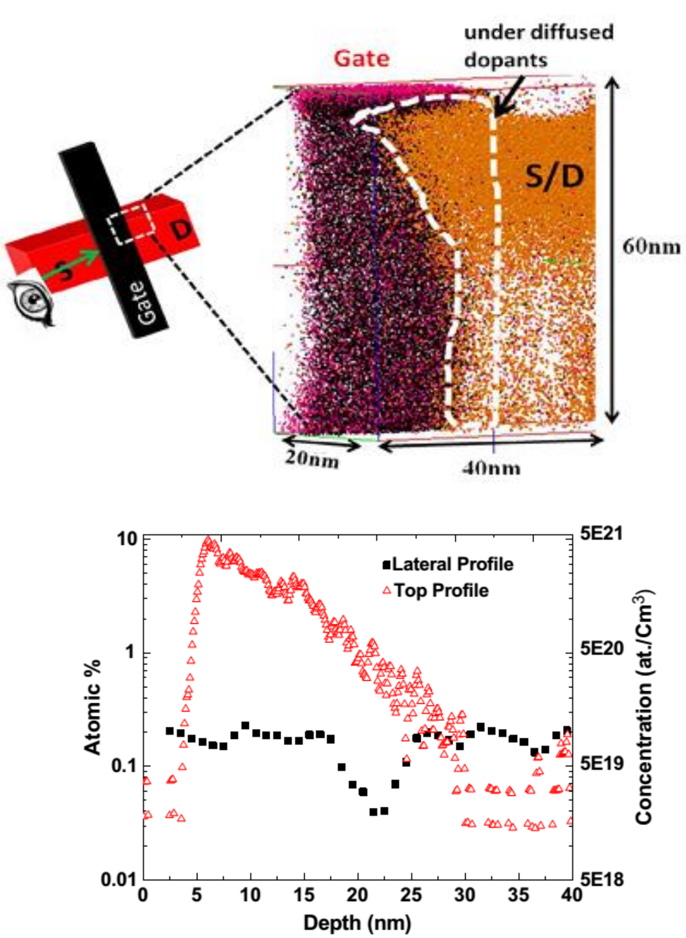


2D Elemental Mapping

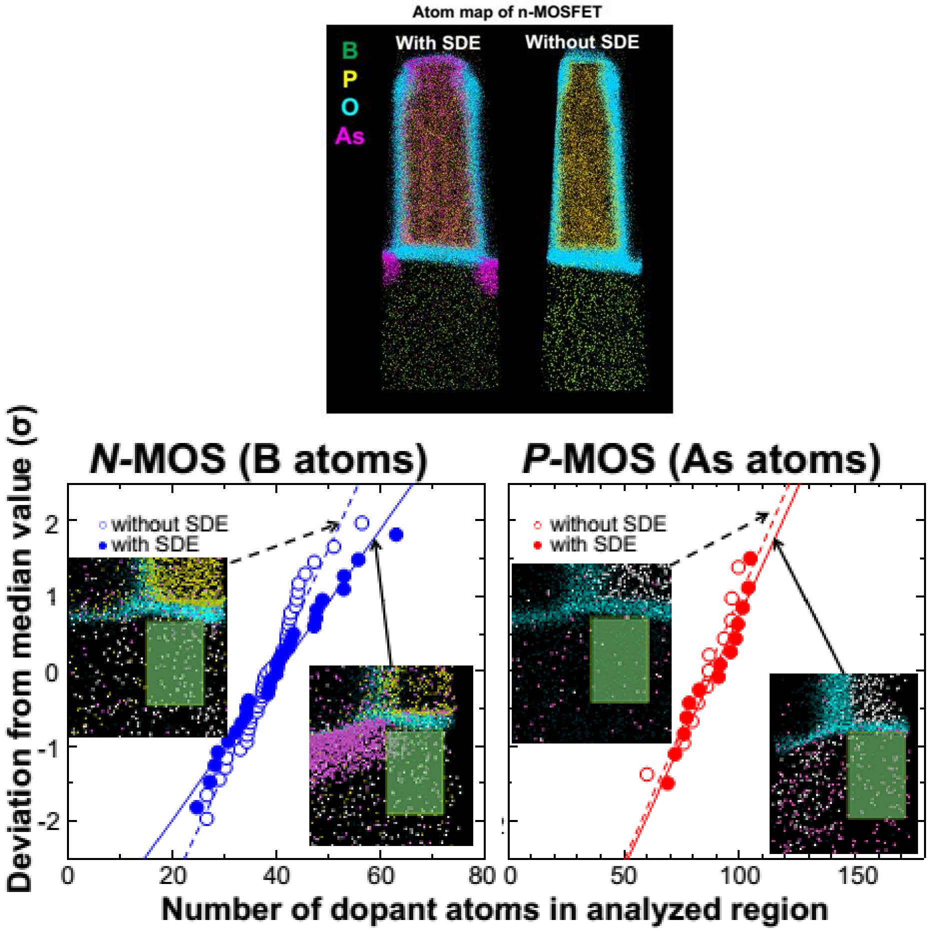


- ✓ High depth resolution
- ✓ High sensitivity for low Z elements

Dopant Analysis - Confined Volumes



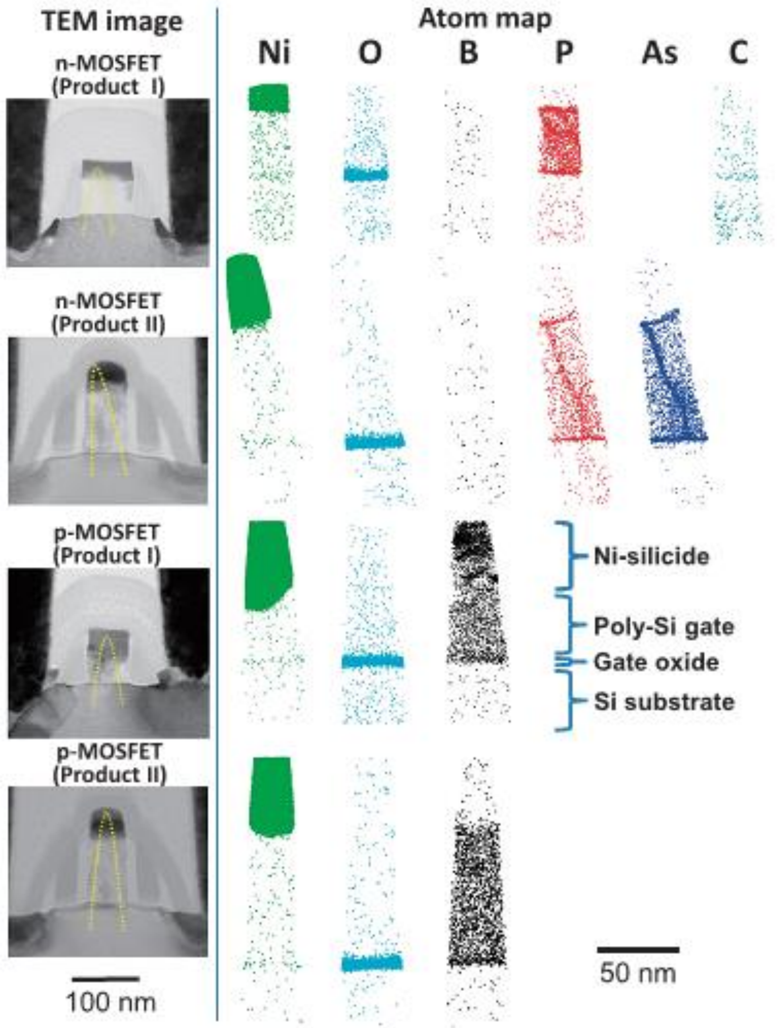
Kambham et al., *Ultramicroscopy* **132** (2013)



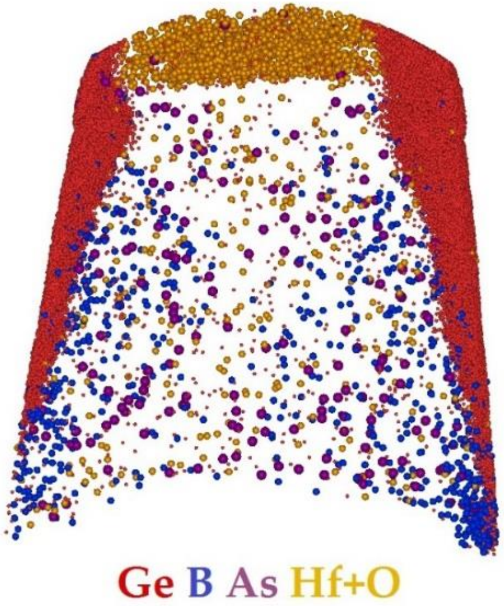
Takamizawa et al., *APL* **99** (2011)

- 3D imaging of dopant atoms within the channel
- Profiles and dopant distributions within confined volumes possible

Dopant Analysis – Fully Processed Devices



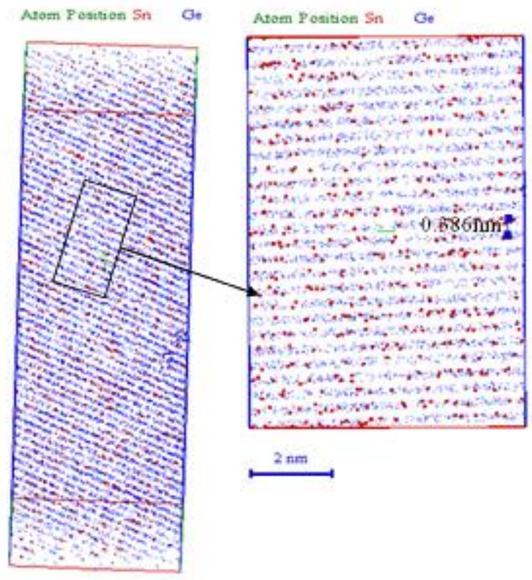
Inoue et al., *APE* **6** (2013)



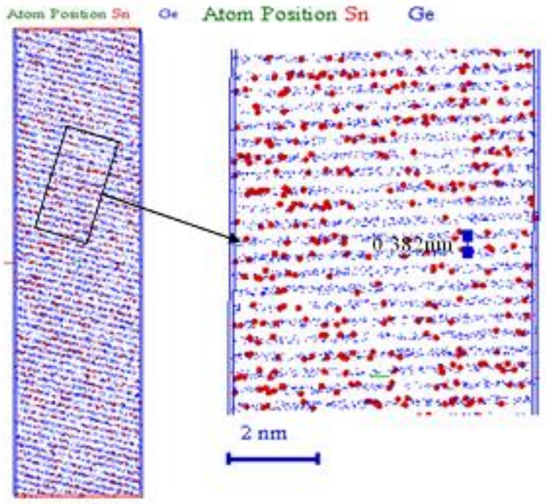
Larson et al., *JOP:Conference Series* **326** (2011)

- Characterization of dopant distributions in commercially available products
- Elemental mapping within gate region of a fully processed device

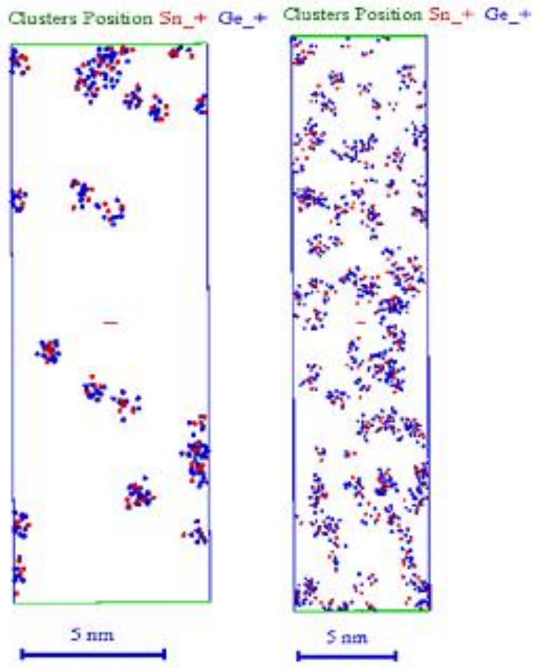
Every Atom Counts



Sample A



Sample B

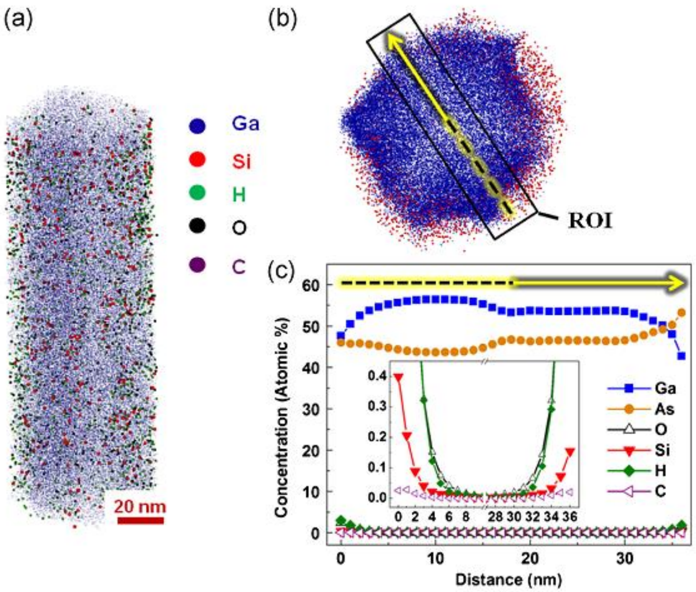


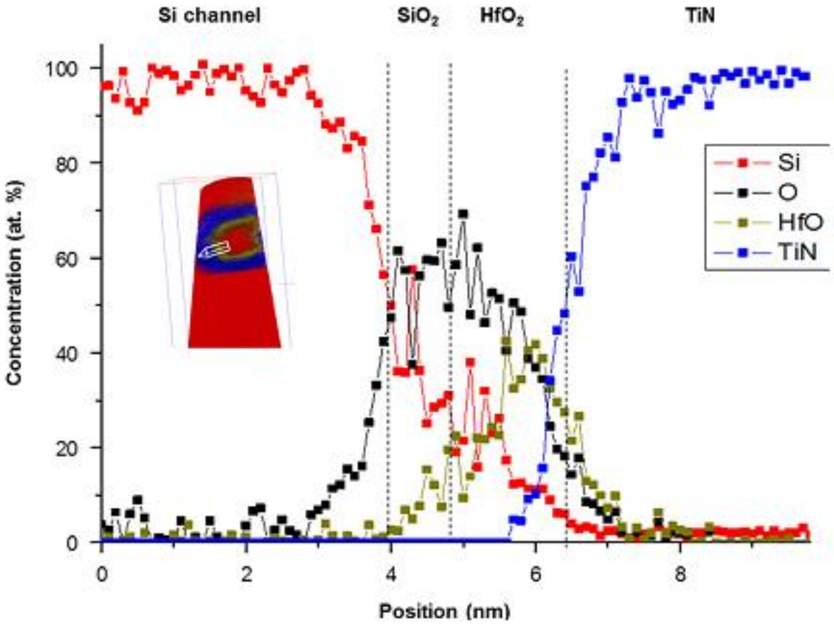
Sample A Sample B

- APT provides atom by atom analysis
- Precipitation and clustering can be investigated

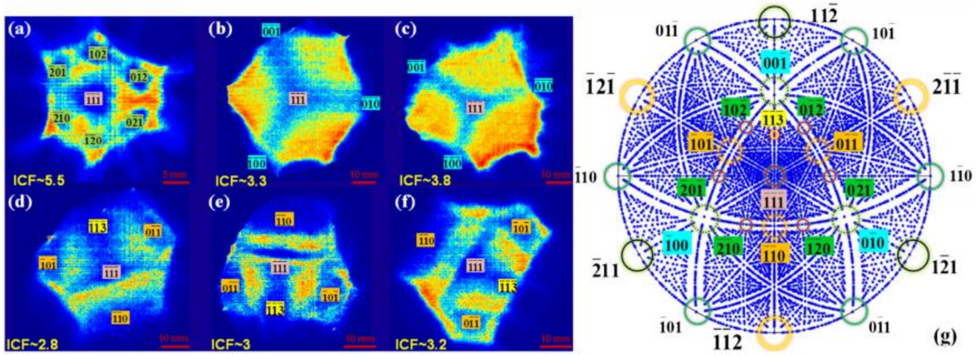
Kumar et al., *Ultramicroscopy* **132** (2013)

Nanowire Characterization



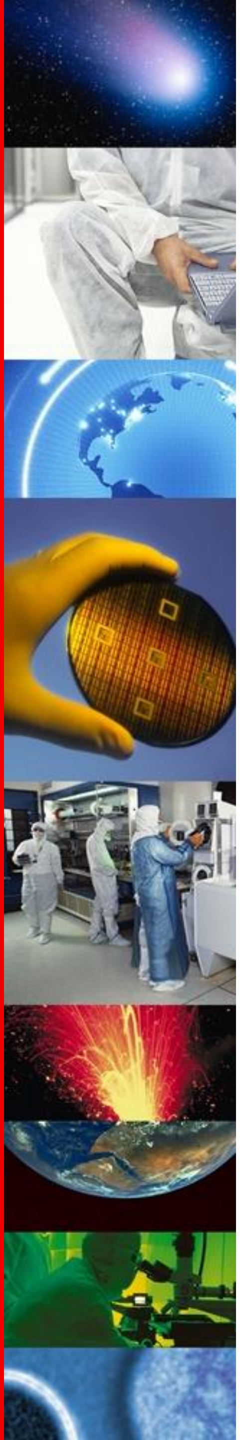


Grenier et al., *Ultramicroscopy* **136** (2014)



Du et al., *Ultramicroscopy* **132** (2013)

- Standalone NW or Embedded NW can be analyzed
- Elemental mapping and profiling with high spatial resolution is possible
- Crystallography information can be obtained



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SCIENCE & METROLOGY SOLUTIONS

Roadmap for Atom Probe Tomography

Thomas F. Kelly

FCMN 2015

April 15, 2015

www.cameca.com

AMETEK®
MATERIALS ANALYSIS DIVISION

Limitations of APT

- Lack of General Applicability
  - Not all materials will run well
  - Low Specimen Yield in Some Cases
- Hydrogen mapping not yet reliable

Gas Handling

- Projection aberrations limit spatial resolution
- Crystallographic information is limited
- No chemical information

APT-TEM

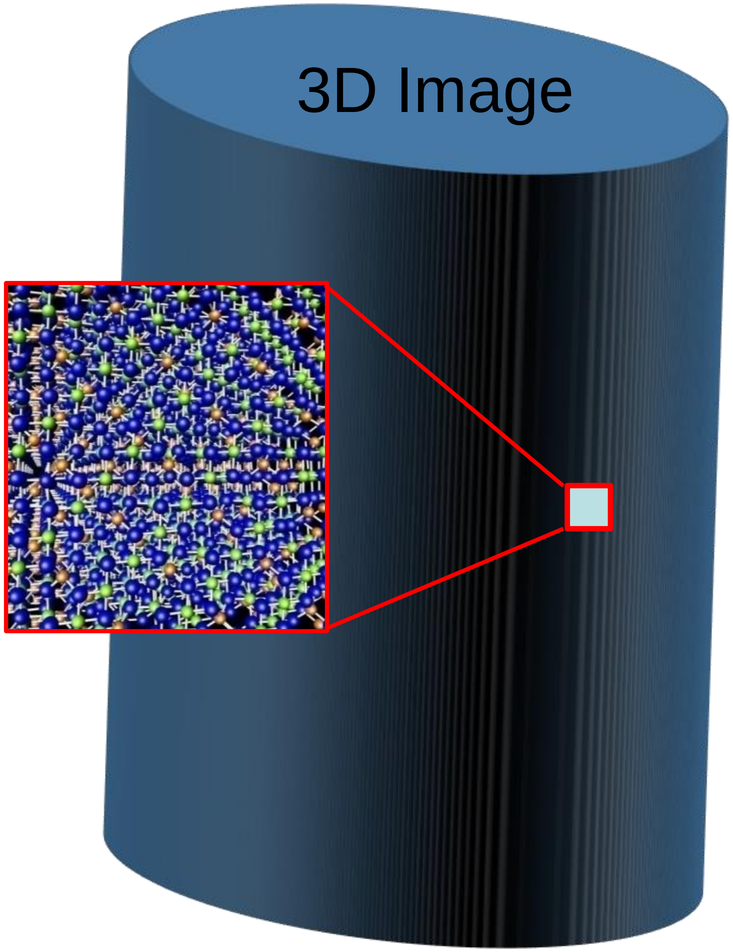
- Detection efficiency is high ~80% (but not 100%)
- Compositional Accuracy
  - Limits of mass interferences
  - Finite multihit resolution of detector
- Maximum Field of View ~200 nm diameter
- Time to knowledge could be reduced

Detector

Gas Handling

- Lower fields are required in presence of gas
  - Reduce fractures by operating at elevated pressure (10^{-8} mbar)
  - Must eliminate resultant noise at detector
  - See later discussion of superconducting detectors
- Working on elimination of hydrogen in images
  - Goal: Reliable hydrogen mapping with atom probe tomography
  - Cryogenic specimen transport/insertion developing to freeze hydrogen in place

Atomic-Scale Tomography



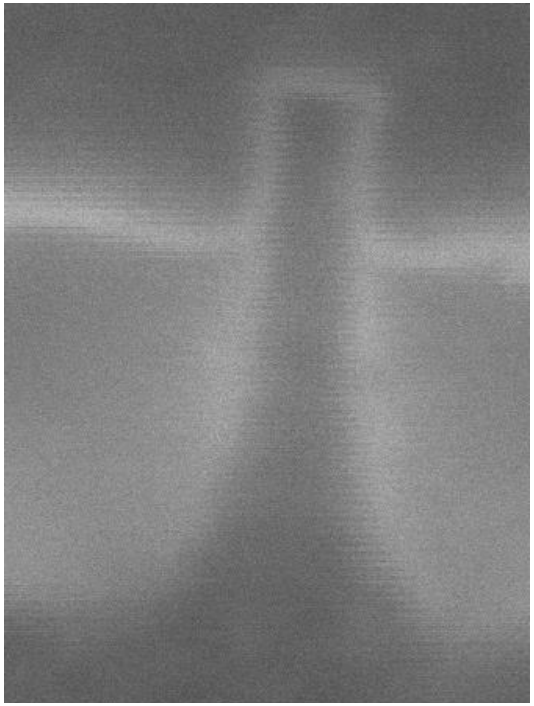
APT → AST

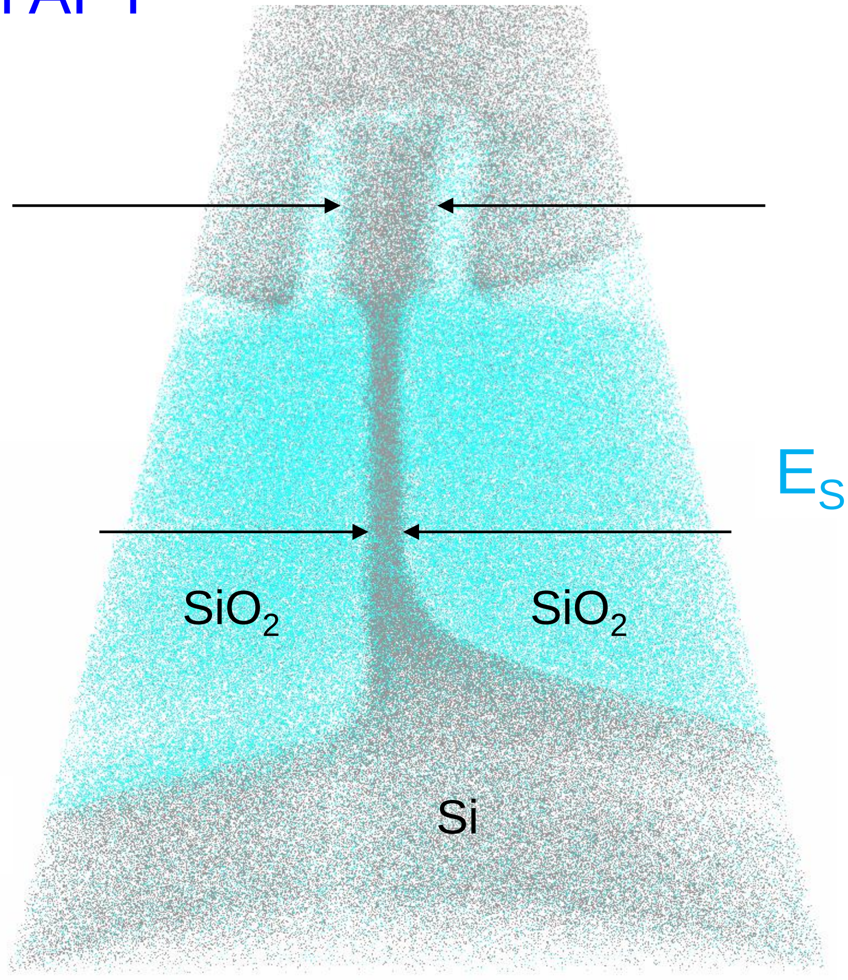
- Definition
  1. Atoms positioned with high precision
  2. 100% of atoms detected
     - Isotopic identity valuable at times
  3. Atoms identified with high precision
  4. Discrete 3-D image for a large volume (500x500x500 nm^3, i.e., billion atoms)

Kelly, Miller, Rajan and Ringer, *Microscopy and Microanalysis* (2013) **19(03)**, pp 652 – 664. DOI: http://dx.doi.org/10.1017/S1431927613000494.

Polyphase Distortions in APT

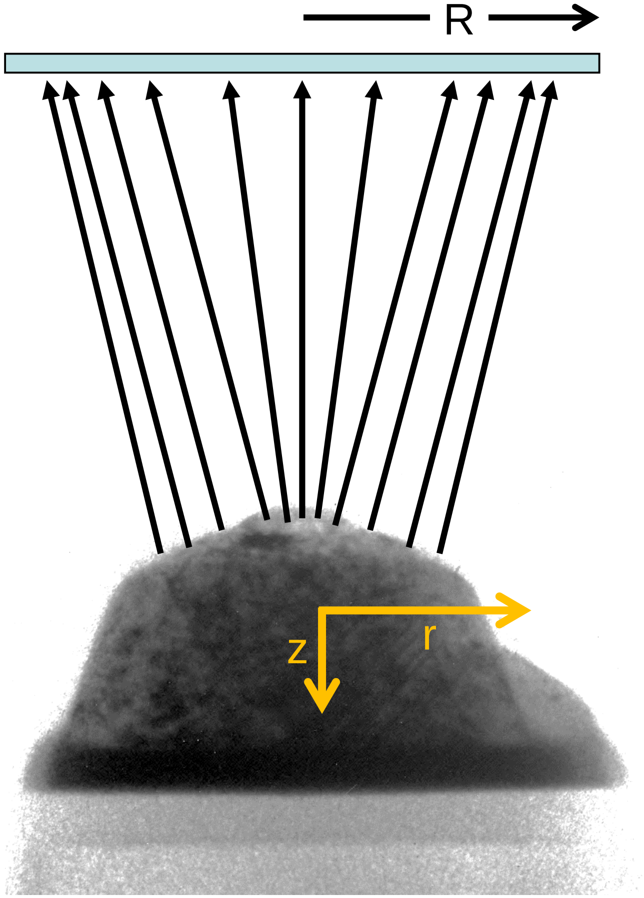
STEM/TEM information has been used to correct reconstruction in APT





$E_{SiO2} > E_{Si}$

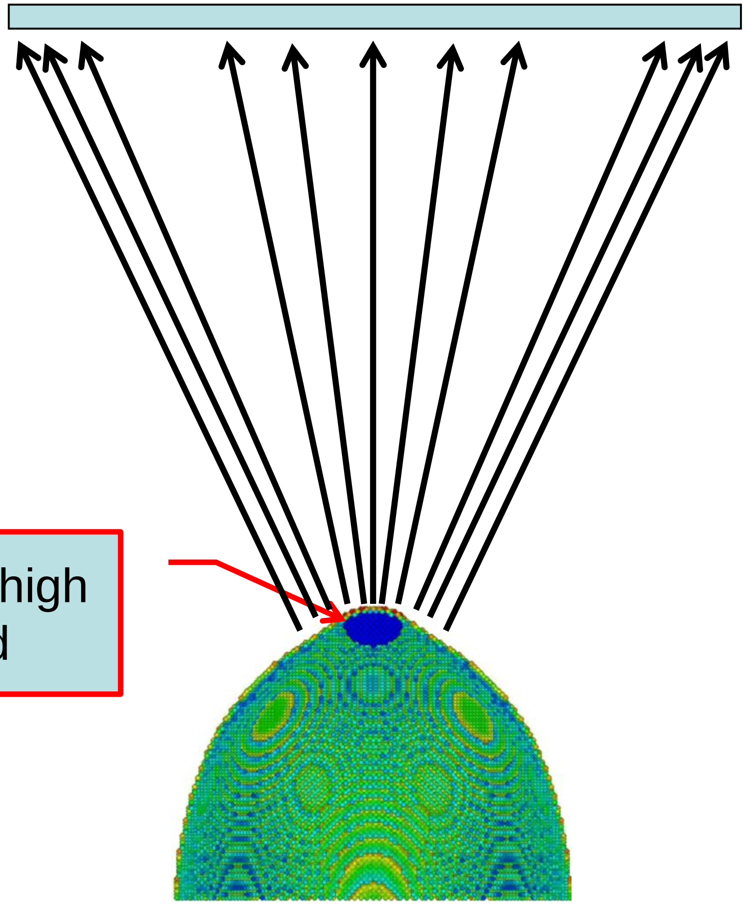
Specimen Apex Shape is the Key



- Real specimen shapes can be complicated
- Image angular magnification functions vary in (r, ϕ, z)
- Knowledge of the apex shape will result in improved atom probe data reconstruction

D. J. Larson et al., *J. Microscopy* 243 (2011) 15

Polyphase Distortions

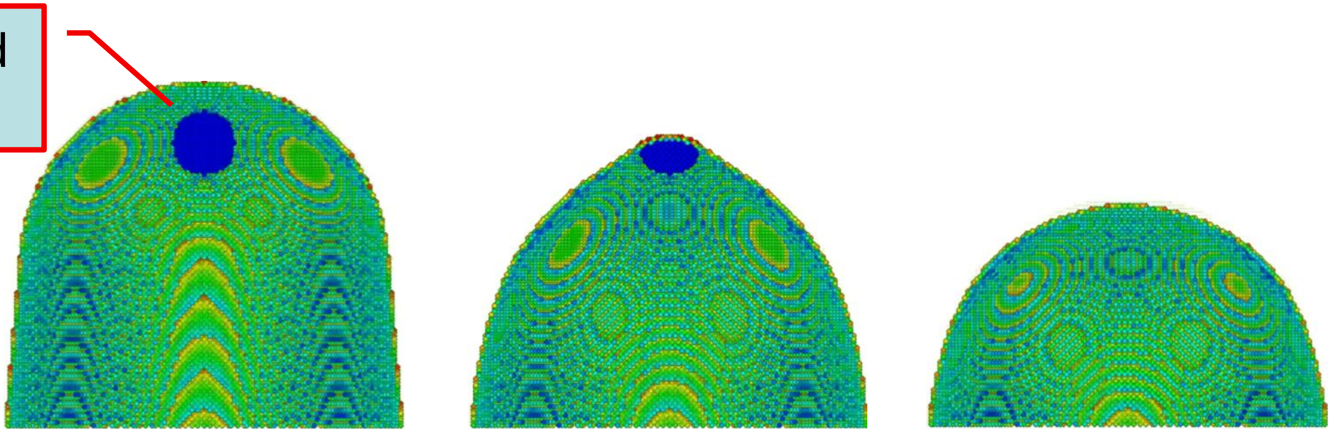


Second phase with high evaporation field

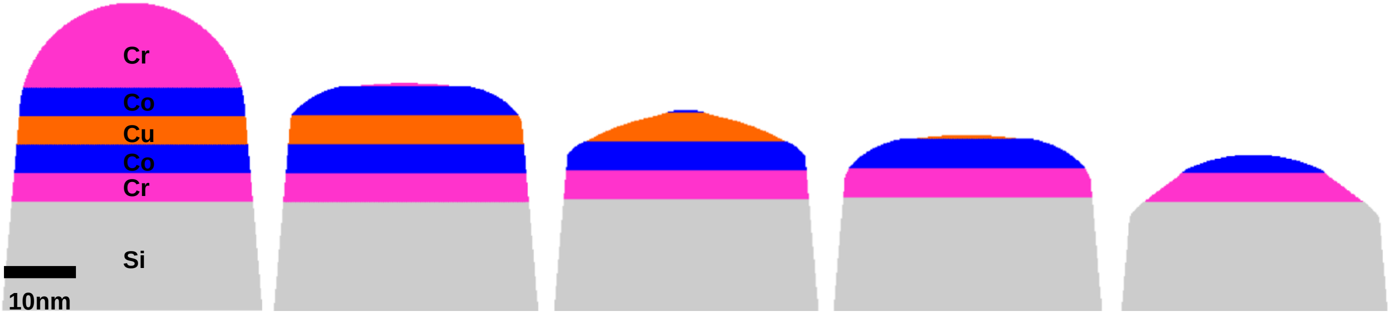
Second Phases Cause Non-Spherical Endforms

Apex Shape (Projection Law) changes during run

We must know the apex shape during entire run


High-field phase

O. Dimond, *Part II Thesis* (University of Oxford) (1999)



D. J. Larson et al., *Ultramicroscopy* 111 (2011) 506

Determination of Specimen Apex Shape

- Microscopy
  - Electron microscopy
    - TEM/STEM
    - SEM
    - …
  - Scanning probe microscopy
  - Field ion microscopy
- Simulation
  - Can simulation of field evaporation be good enough?
  - Iteration with actual data
  - Today’s algorithms are not sufficient
  - Need 100X increase in computing power or algorithm speed

Collaborators: The ATOM Project

Atomic-Scale Tomography = ATOM Project

- Simon P. Ringer
  - University of Sydney
- Michael K. Miller
  - Oak Ridge National Laboratory
- Krishna Rajan
  - Iowa State University
- Ondrej Krivanek, Niklas Dellby
  - Nion Instruments

CAMECA

- Brian P. Geiser
- David J. Larson
- Ed Oltman
- Ty Prosa
- Jeff Shepard

References

"Atomic-Scale Tomography: A 2020 Vision"
Thomas F. Kelly, Michael K. Miller, Krishna Rajan, and Simon P. Ringer, *Microscopy and Microanalysis*, Invited Review, vol. 19 (2013) pp. 652 – 664.

"Visions of Atomic-Scale Tomography"
Thomas F. Kelly, Michael K. Miller, Krishna Rajan, and Simon P. Ringer, *Microscopy Today*, May 2012, pp. 12-16.

Collaborators: The ATOM Project

Atomic-Scale Tomography = ATOM Project

- Dierk Raabe
  - Max Planck Institute Dusseldorf
- Rafal Dunin-Borkowski, Joachim Mayer
  - Forschungzentrum Jülich
- Max Haider
  - CEOS
- Brian Gorman, David Dierks
  - Colorado School of Mines
- Christoph Koch
  - Universität Ulm

References

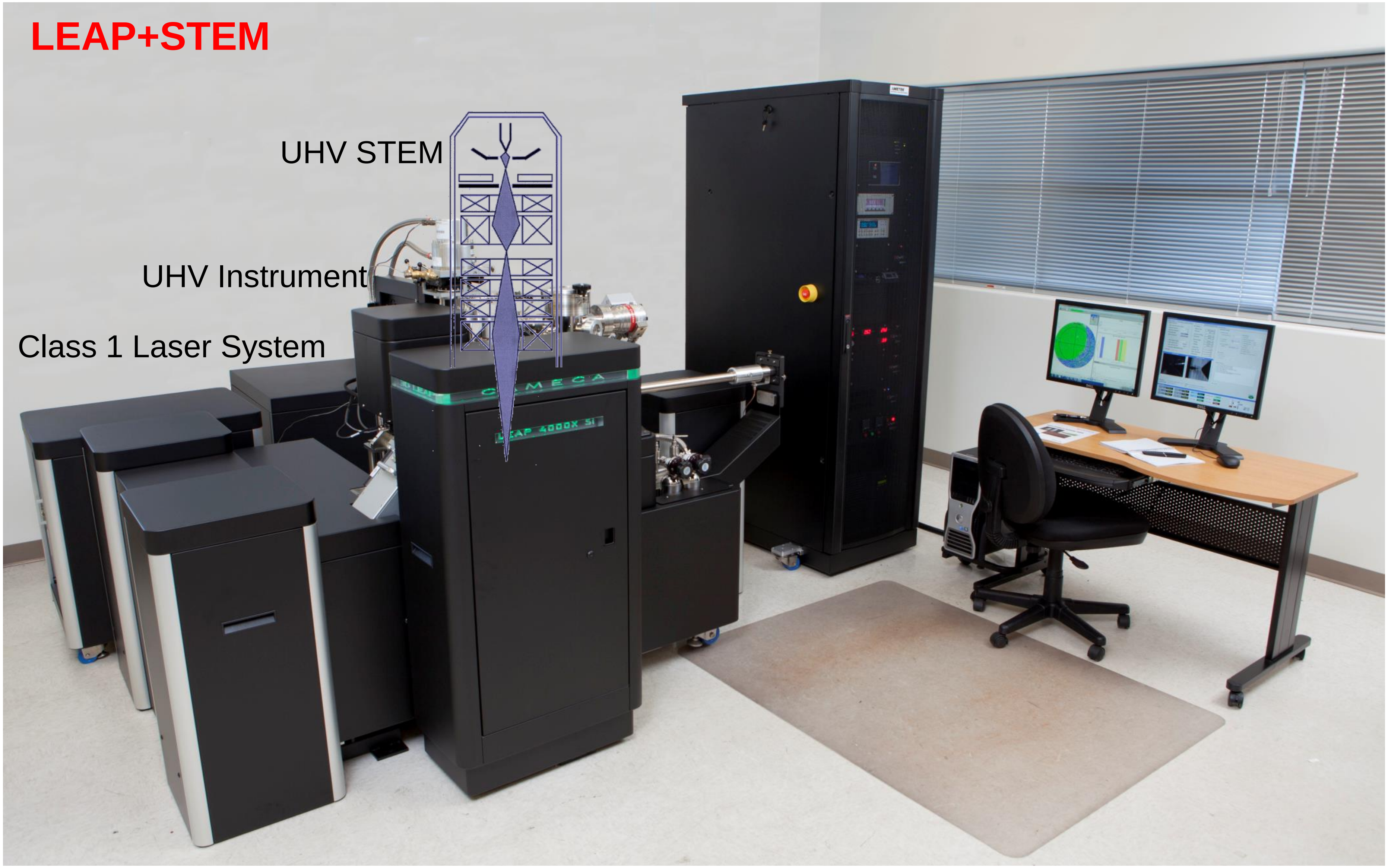
“Atomic-Scale Tomography: A 2020 Vision”

Thomas F. Kelly, Michael K. Miller, Krishna Rajan, and Simon P. Ringer, *Microscopy and Microanalysis*, Invited Review, vol. 19 (2013) pp. 652 – 664.

“Visions of Atomic-Scale Tomography”

Thomas F. Kelly, Michael K. Miller, Krishna Rajan, and Simon P. Ringer, *Microscopy Today*, May 2012, pp. 12-16.

The ATOM Project: LEAP STEM



LEAP+STEM

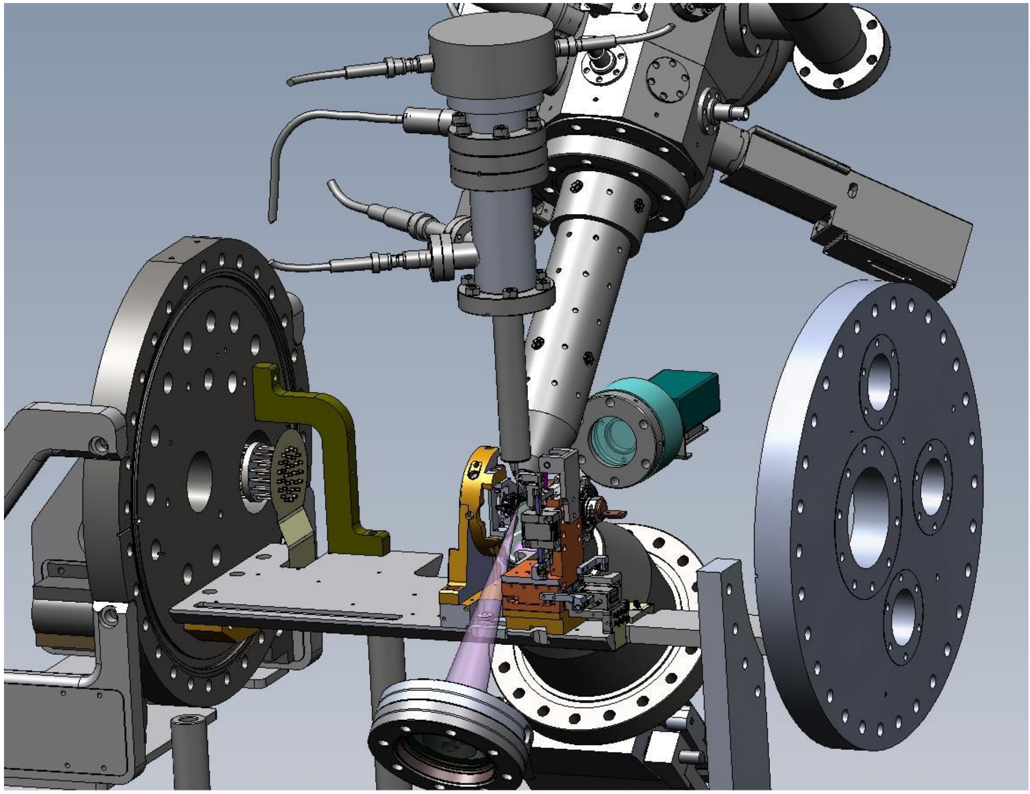
UHV STEM

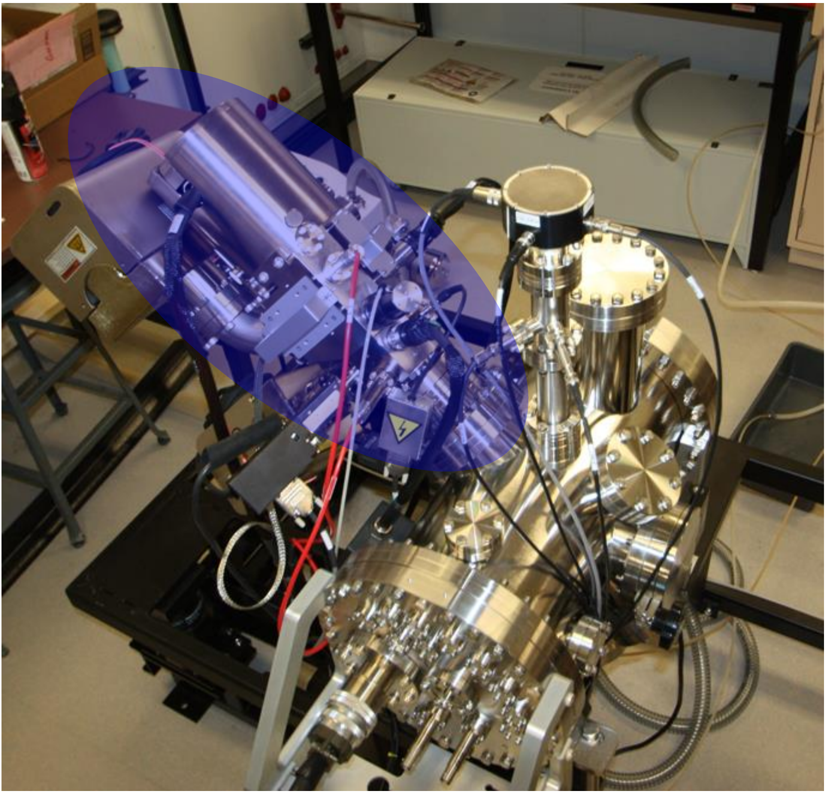
UHV Instrument

Class 1 Laser System

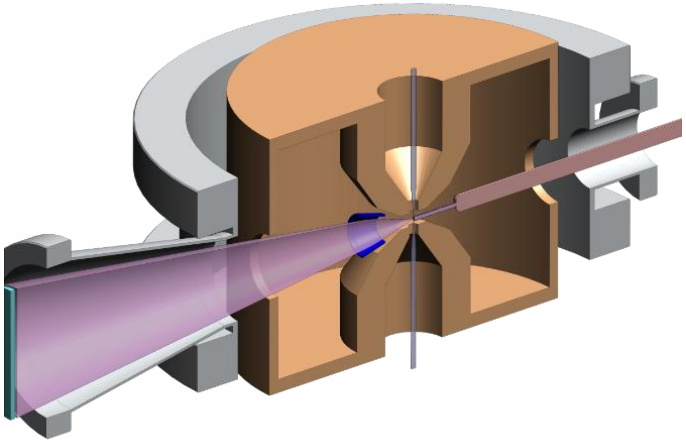
LEAP+STEM Proof of Concept

With Brian Gorman, David Dierks, Colorado School of Mines





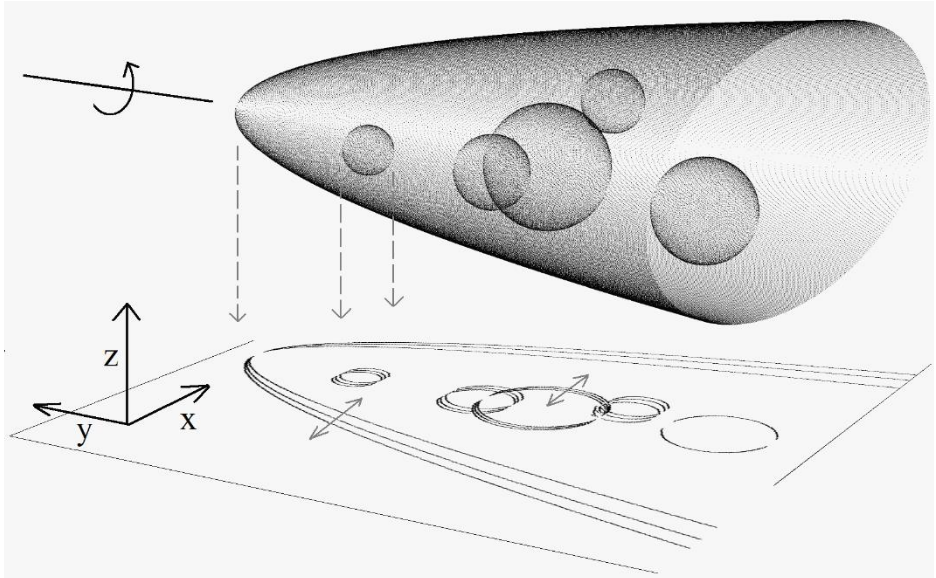
The ATOM Project: TEM LEAP



Build objective lens assembly with atom probe inside

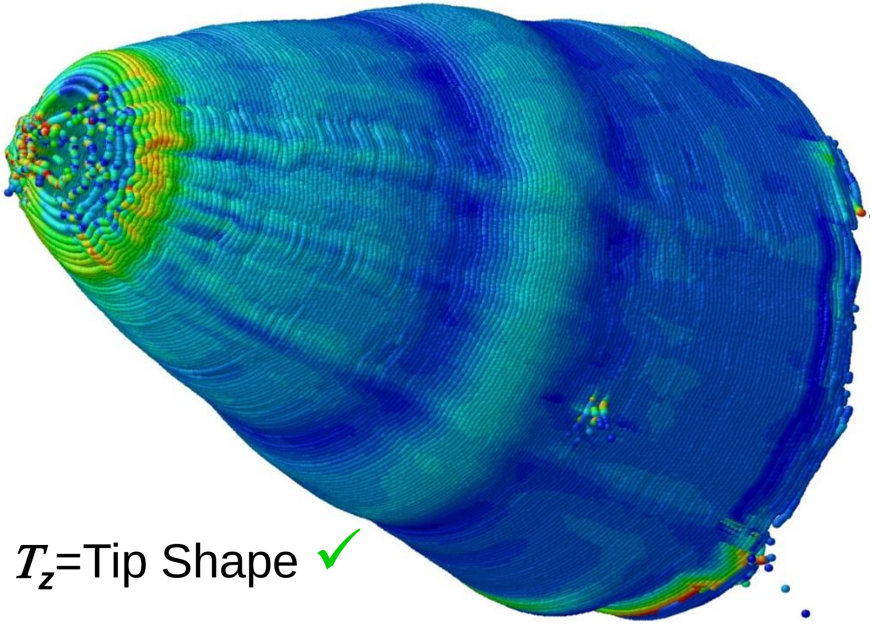
With Dirk Raabe, Rafal Dunin-Borkowski, Joachim Mayer

Tip Shape from Surface Tangent Algorithm



Petersen & Ringer, *Journal of Applied Physics*, **105**, 103518 (2009)
Petersen & Ringer, *Computer Physics Comms*, **181**, 676, (2010)

- Track the smooth movement of interest points in a tilt series.
- Need 10 images to determine surface shape.



T_z=Tip Shape ✓

- STA point cloud showing
  - Tip Shape
  - Mean Curvature (color scale)

High Resolution Imaging with Ptychography

HUMPHRY, M.J., KRAUS, B., HURST, A.C., MAIDEN, A.M. & RODENBURG, J.M. (2011). Ptychographic electron microscopy using high-angle dark-field scattering for sub-nanometre resolution imaging. Nature Communications (2012) Mar 6;3:730. doi: 10.1038/ncomms1733.

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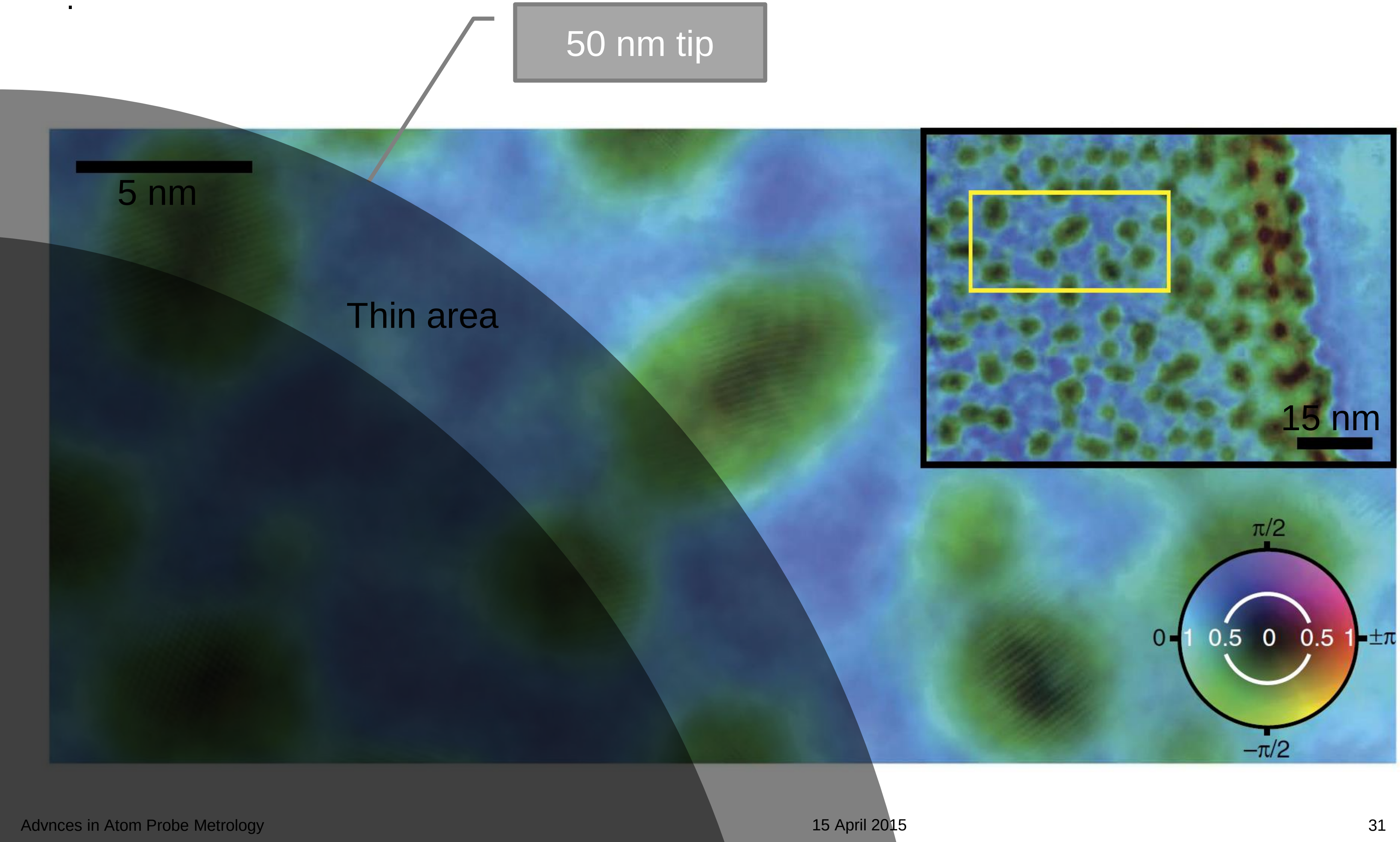
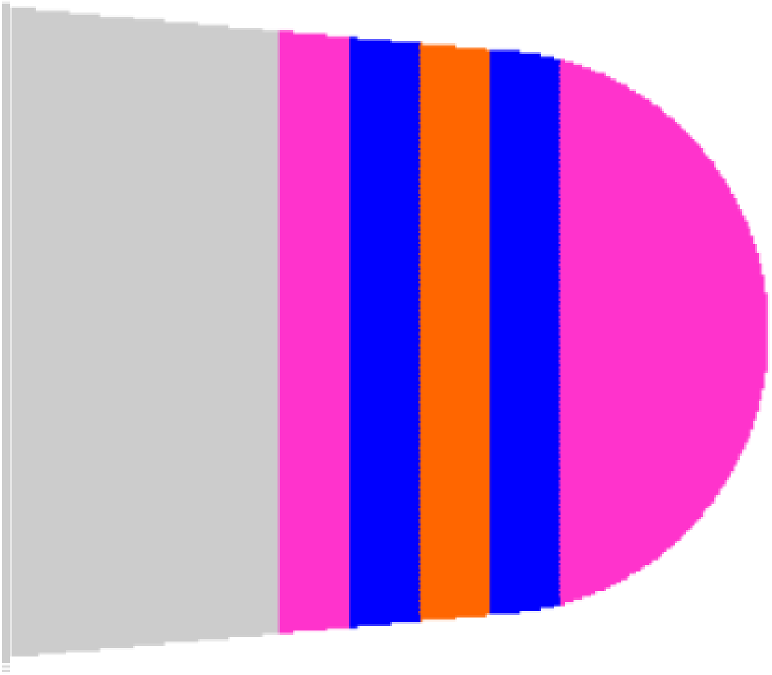


Specimen Evolution Models



D. J. Larson et al., *Ultramicroscopy* 111 (2011) 506

- Experimental specimen apex shapes are expensive
- Simulated specimen apex shapes are expensive
- Interpolation of the apex shape between snapshots could solve this challenge
- Recent work is promising:
  - D. Haley, M.P. Moody, G.D.W. Smith, Microsc. Microanal., 19(06) (2013) 1709-1717. doi: 10.1017/S1431927613013299
  - D. Haley, T. Petersen, S.P. Ringer, G.D.W. Smith, J. Microscopy 244 (2011): 170–80. doi:10.1111/j.1365-2818.2011.03522.x.

Superconducting Detectors

Collaborators

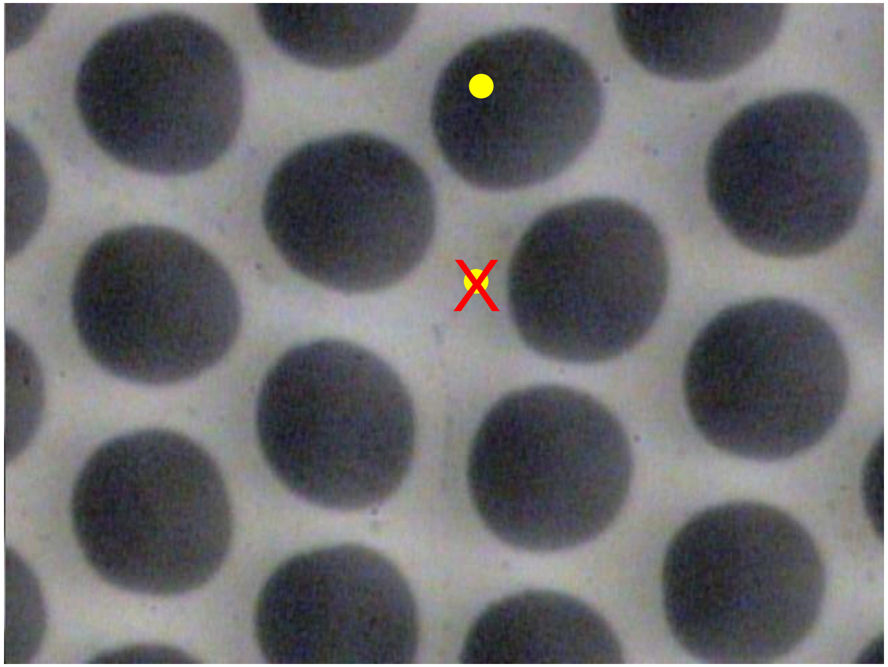
Robert McDermott, Joseph Suttle

- University of Wisconsin - Madison

- Develop new detector technology
  - 100% detection efficiency
  - Solve peak overlap in ToF spectra
  - Improve multihit resolution

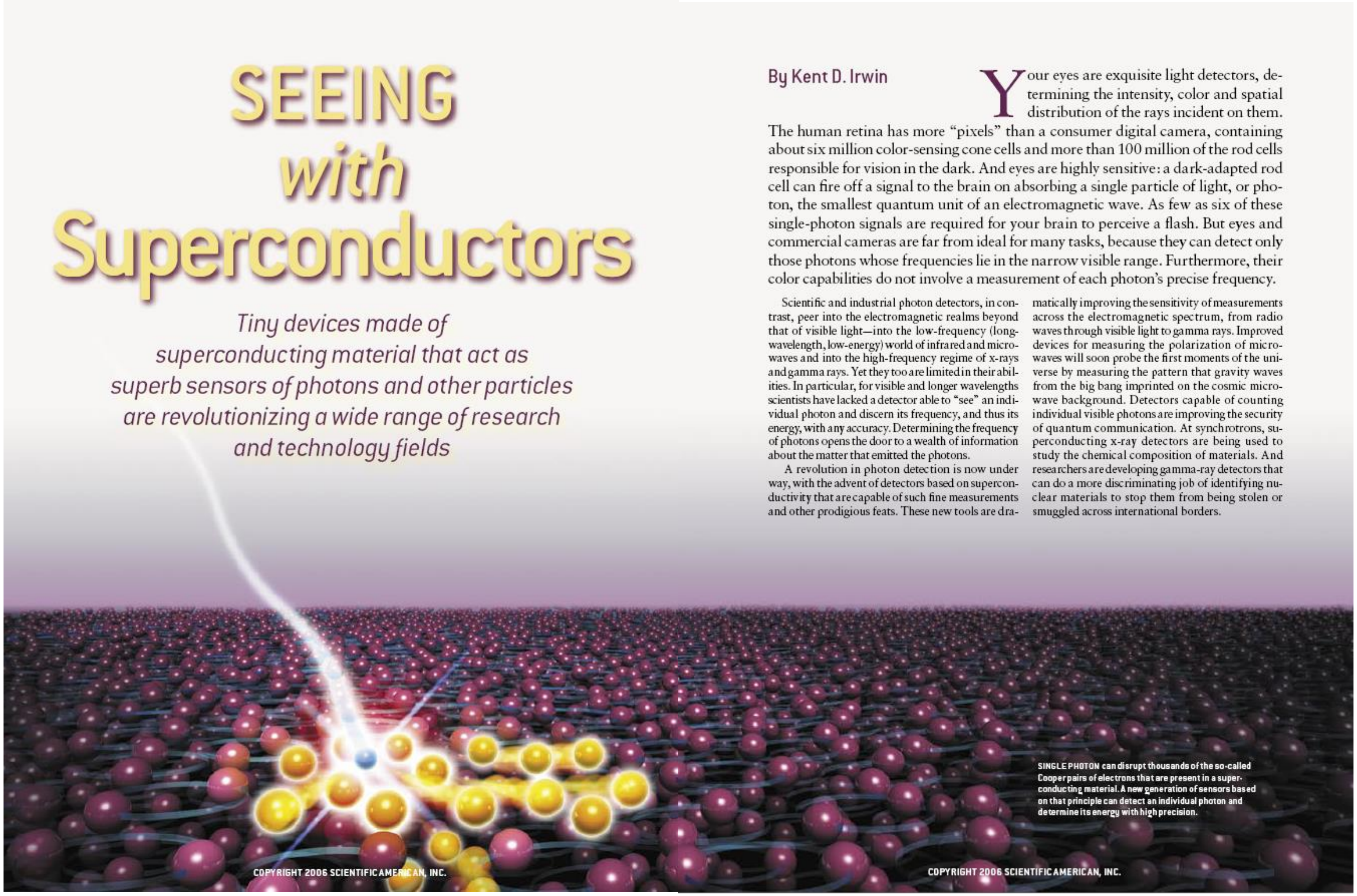
MCP Construction and Performance

Conventional Pb-glass MCP



- Ions hitting in channel mostly get amplified
- Ions hitting on flat face mostly do not get amplified
- Sub 100 ps timing resolution possible
- High gain
- “No” variation in detection efficiency with atomic number

Superconducting Detectors



SEEING *with* Superconductors

Tiny devices made of superconducting material that act as superb sensors of photons and other particles are revolutionizing a wide range of research and technology fields

By Kent D. Irwin

Your eyes are exquisite light detectors, determining the intensity, color and spatial distribution of the rays incident on them. The human retina has more "pixels" than a consumer digital camera, containing about six million color-sensing cone cells and more than 100 million of the rod cells responsible for vision in the dark. And eyes are highly sensitive: a dark-adapted rod cell can fire off a signal to the brain on absorbing a single particle of light, or photon, the smallest quantum unit of an electromagnetic wave. As few as six of these single-photon signals are required for your brain to perceive a flash. But eyes and commercial cameras are far from ideal for many tasks, because they can detect only those photons whose frequencies lie in the narrow visible range. Furthermore, their color capabilities do not involve a measurement of each photon's precise frequency.

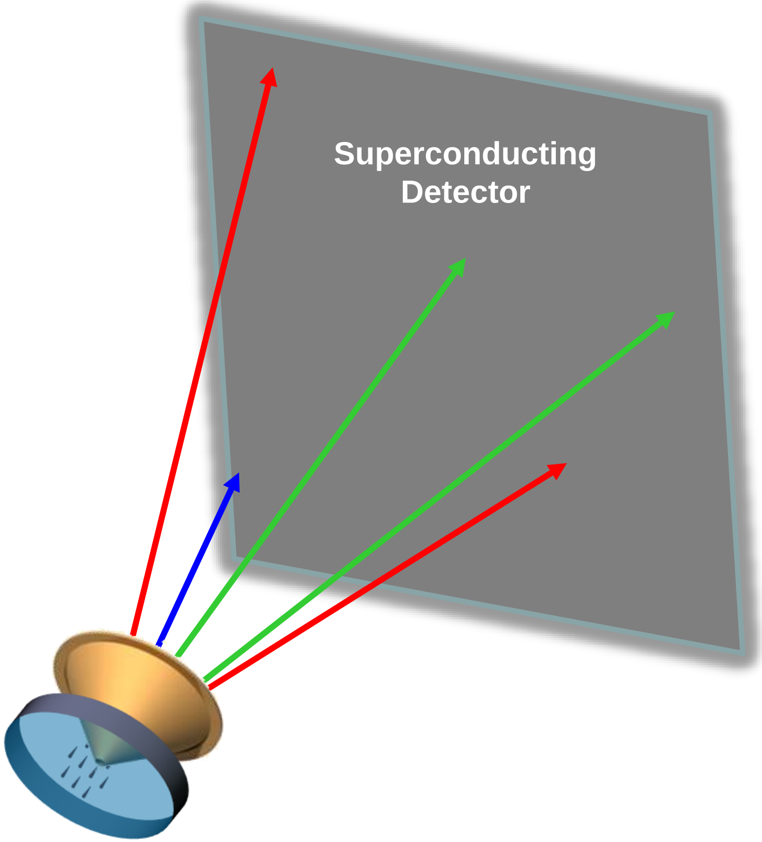
Scientific and industrial photon detectors, in contrast, peer into the electromagnetic realms beyond that of visible light—into the low-frequency (long-wavelength, low-energy) world of infrared and microwaves and into the high-frequency regime of x-rays and gamma rays. Yet they too are limited in their abilities. In particular, for visible and longer wavelengths scientists have lacked a detector able to "see" an individual photon and discern its frequency, and thus its energy, with any accuracy. Determining the frequency of photons opens the door to a wealth of information about the matter that emitted the photons.

A revolution in photon detection is now under way, with the advent of detectors based on superconductivity that are capable of such fine measurements and other prodigious feats. These new tools are dramatically improving the sensitivity of measurements across the electromagnetic spectrum, from radio waves through visible light to gamma rays. Improved devices for measuring the polarization of microwaves will soon probe the first moments of the universe by measuring the pattern that gravity waves from the big bang imprinted on the cosmic microwave background. Detectors capable of counting individual visible photons are improving the security of quantum communication. At synchrotrons, superconducting x-ray detectors are being used to study the chemical composition of materials. And researchers are developing gamma-ray detectors that can do a more discriminating job of identifying nuclear materials to stop them from being stolen or smuggled across international borders.

SINGLE PHOTON can disrupt thousands of the so-called Cooper pairs of electrons that are present in a superconducting material. A new generation of sensors based on that principle can detect an individual photon and determine its energy with high precision.

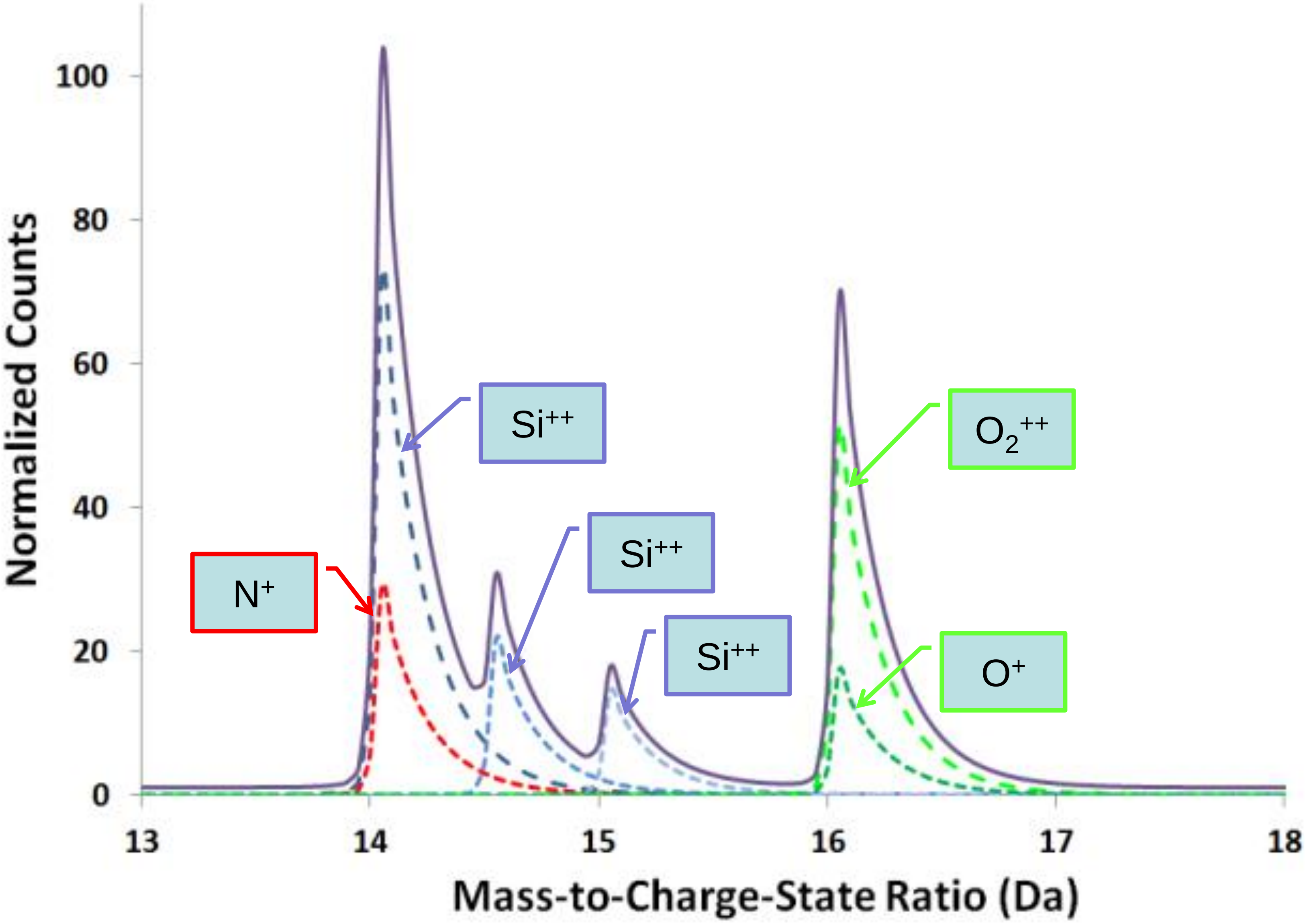
Kent D. Irwin, "Seeing with Superconductors," Scientific American, Nov. 2006, pp. 86-94.

Superconducting Detector Technology for Atom Probe Tomography

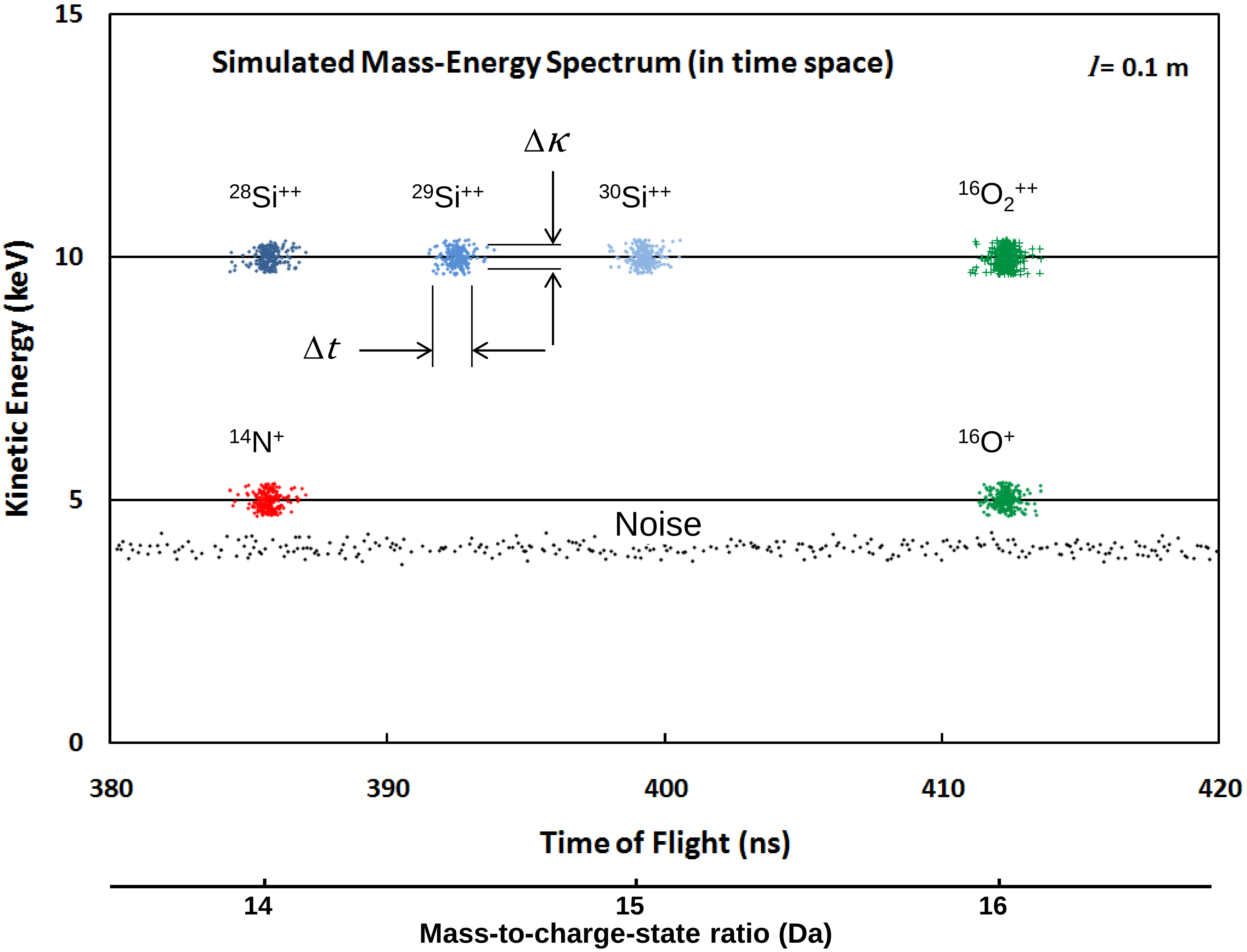


- Potential for 100% Detection
- Potential for kinetic-energy information
- Want ≥ 1k x 1k pixels
- Need ≤ 100 ps timing precision
- Detector size limited by design performance
  - ~100 mm diameter challenging
- Higher data collection rates likely

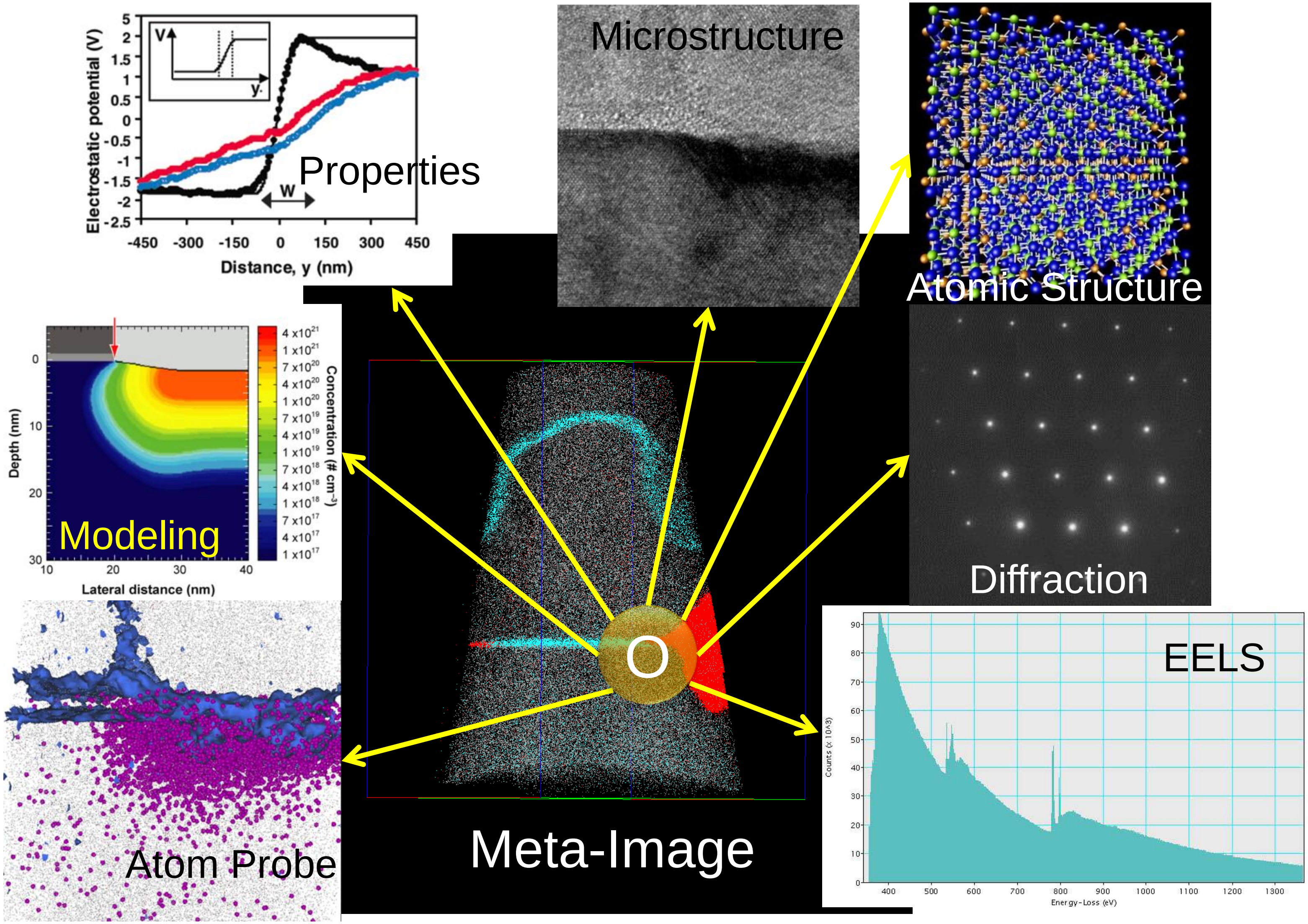
Mass Spectrum with Interferences

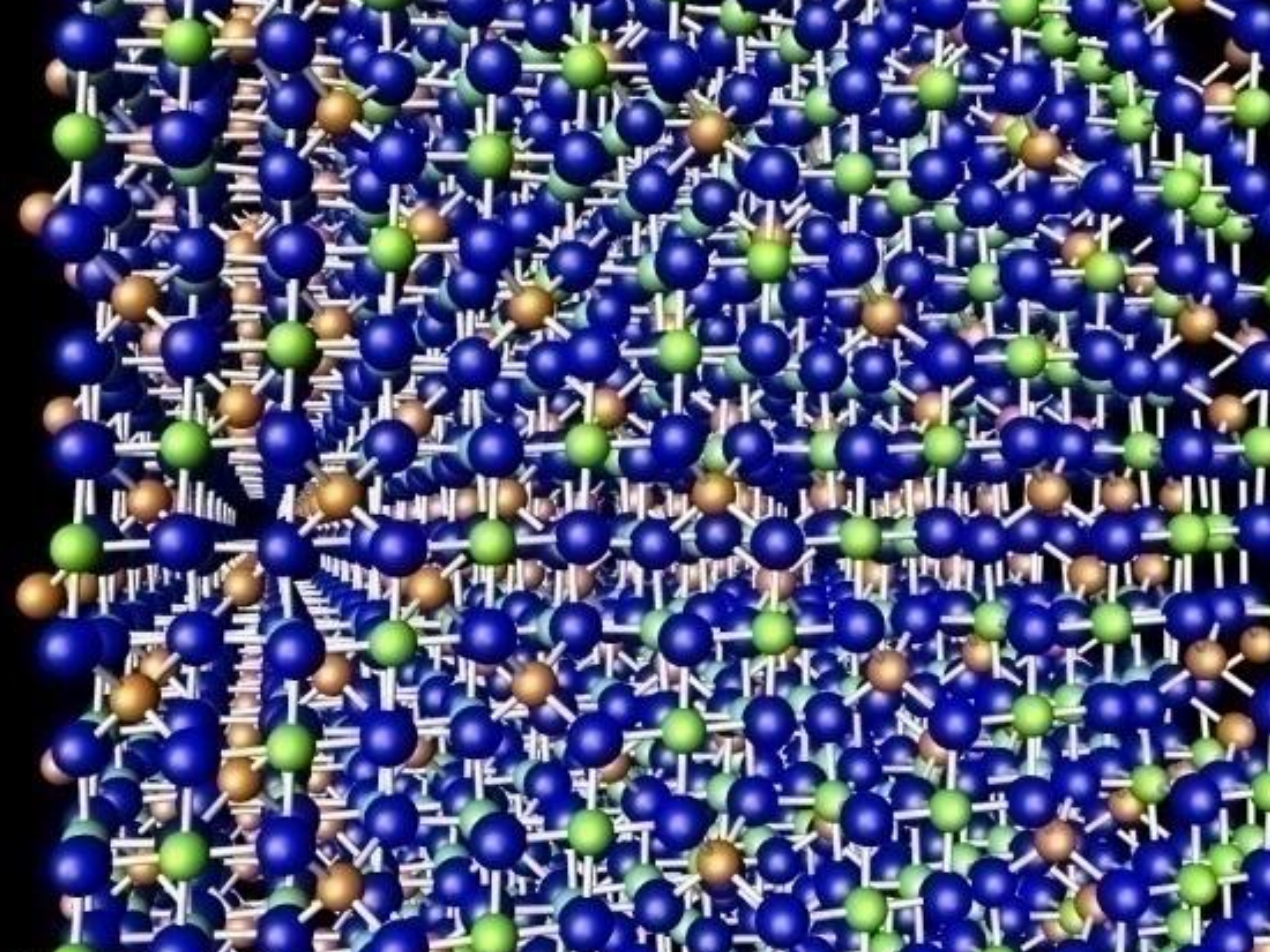


Mass-Energy Spectrum Would Improve Discrimination



Putting It All Together





THE END