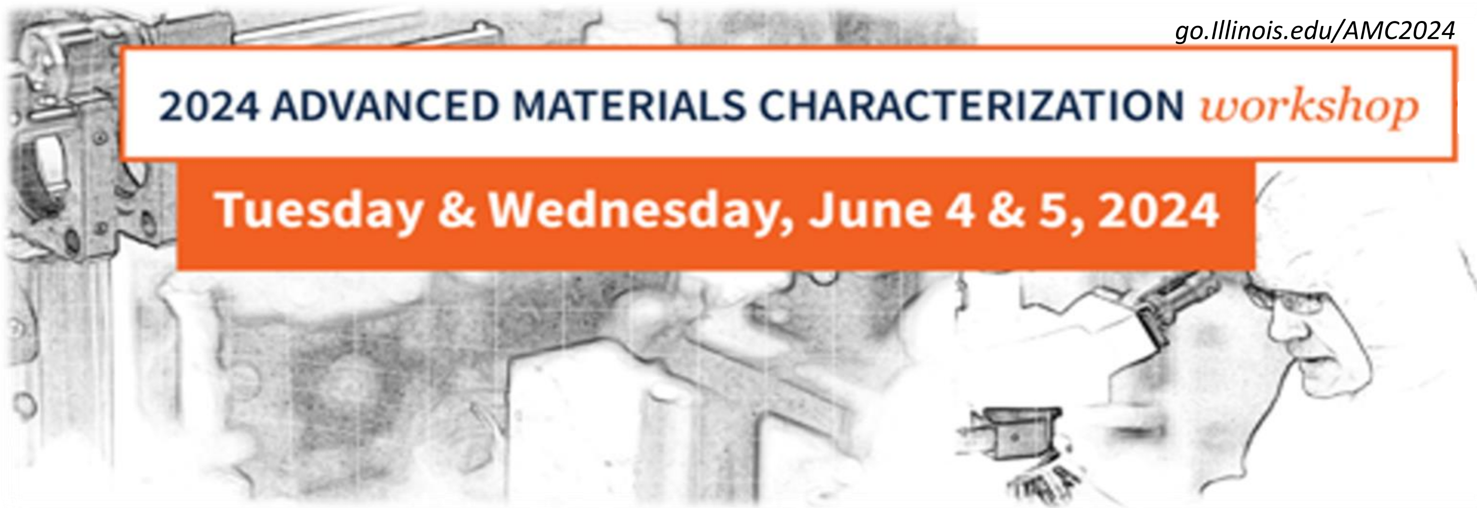


Atom Probe Tomography

Timothy P. Spila, Ph.D.
Sr. Research Scientist

Materials Research Laboratory
MRL.Illinois.edu
University of Illinois at Urbana-Champaign

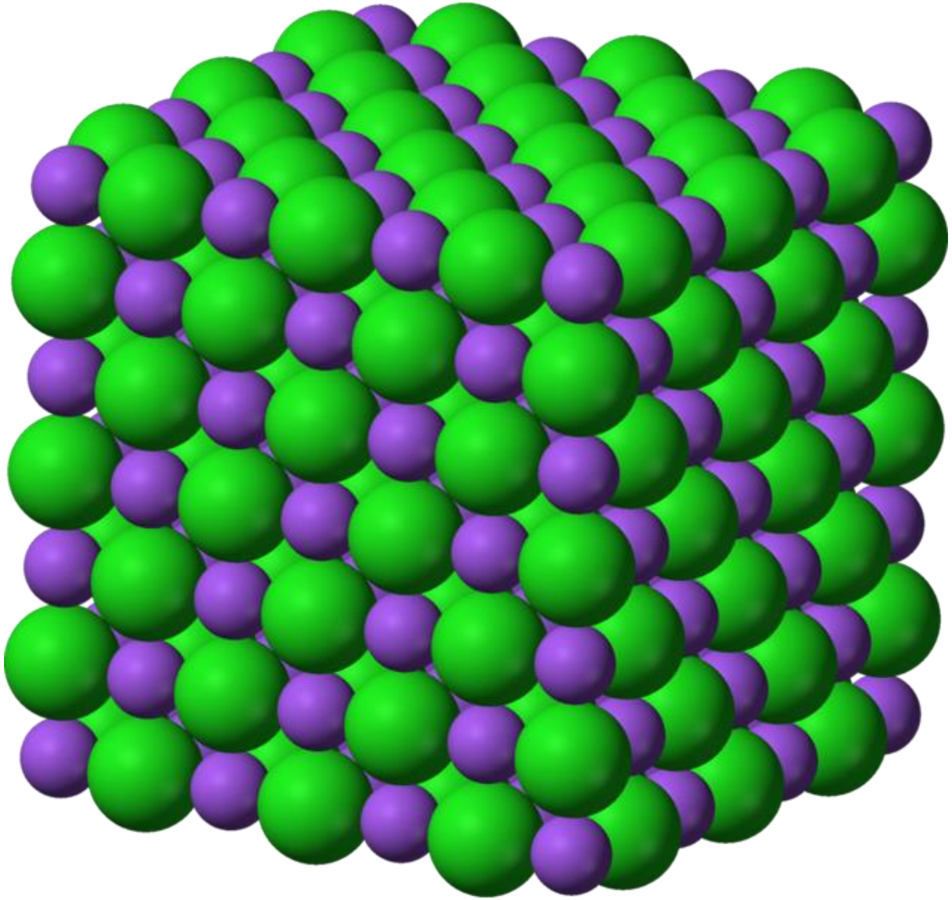
Atomistic Structure of Matter

Atom: derived from the ancient Greek word *atomos*, which means "uncuttable" (4th Century BC).

Modern view of the structure of an atom was derived by Rutherford (1913) and Bohr (1913).

Experiments on atomic nature of materials (packing of atoms in crystals) was deduced by Bragg and Bragg (1913).

Adapted from D.J. Larson et al., Local Electrode APT, book 2013



https://commons.wikimedia.org/wiki/File:Sodium-chloride-3D-ionic.png

Atom Probe Tomography

Spatial Resolution

0.1 – 0.3 nm in depth

0.3 – 0.5 nm laterally

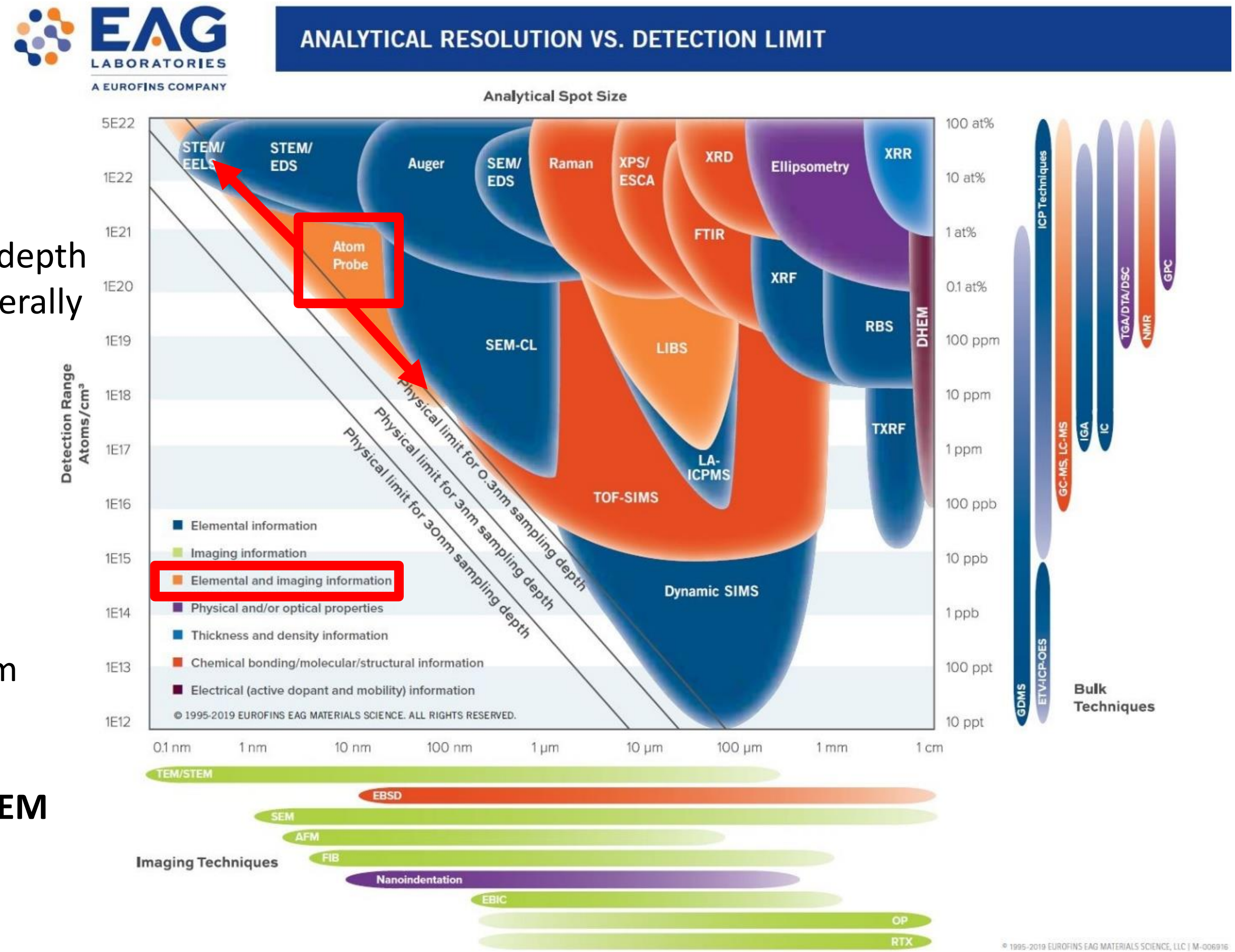
Field of View

100 – 200 nm laterally

Time-of-Flight Mass Analysis

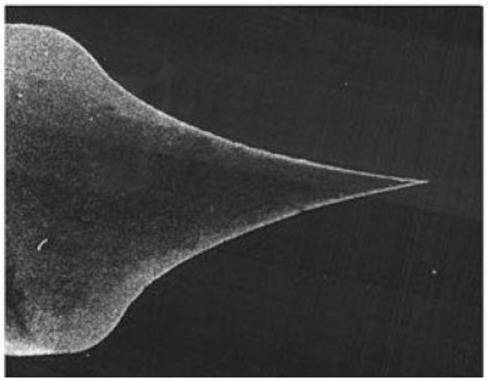
Mass range from 1 – 600 amu

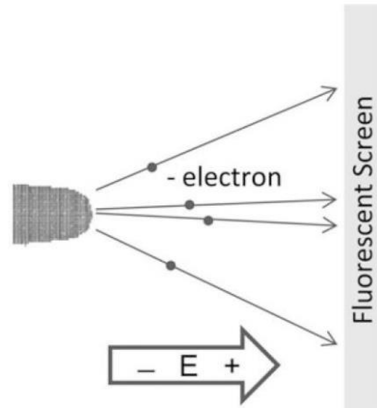
Complementary to TEM

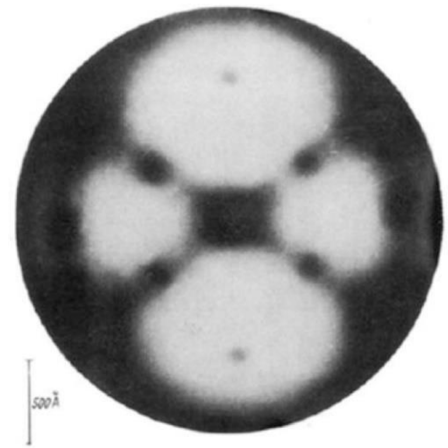


Field Electron Emission Microscope (FEEM)

Erwin Wilhelm Müller - 1935







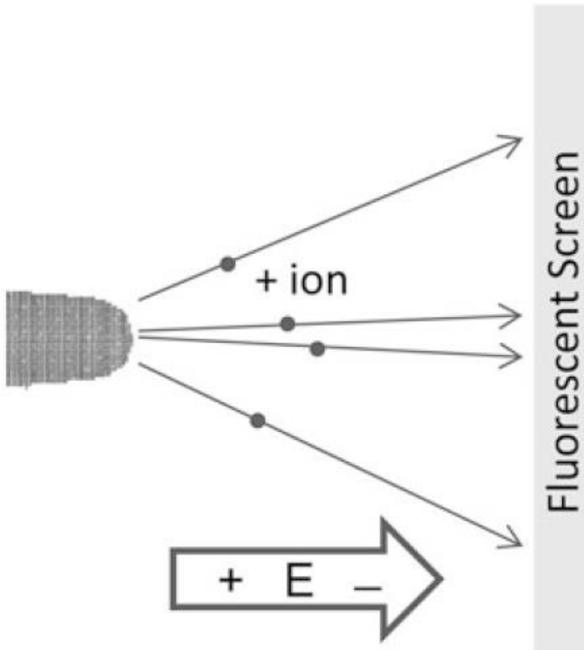
- 10^9 V/m needed to strip an electron from an atom
- Sharp point produces enhanced electric field
- 10,000 V with 1 um tip radius => 10^9 V/m

Adapted from D.J. Larson et al., Local Electrode APT, book 2013

Field Ion Microscope (FIM)

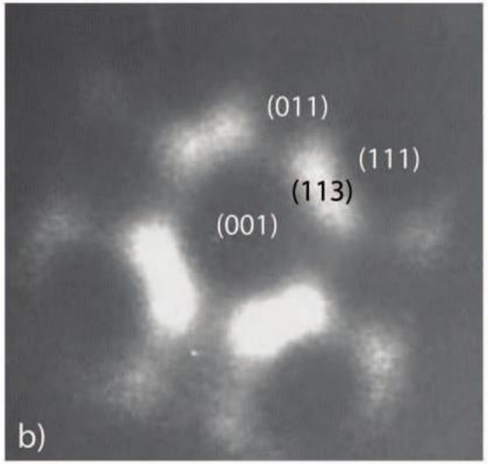
Kanwar Bahadur and Erwin W. Müller - 1955

10^{-3} Pa He imaging gas, Tip cooled to 20-80K, Very sharp tip: 80 nm or less



W(011)

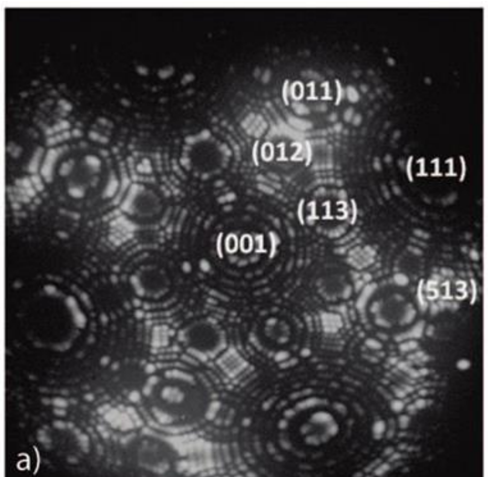
Adapted from D.J. Larson et al., Local Electrode APT, book 2013

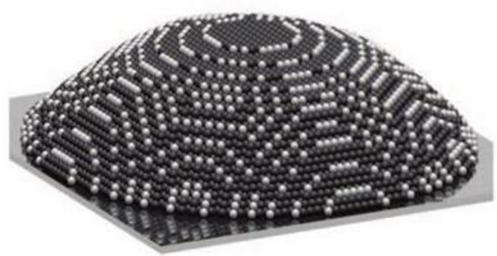


FEM (thermal form), Ni(001)

M. Moors et al., ACS-Nano **3** (2009) 511

(Ni: cubic close packed)

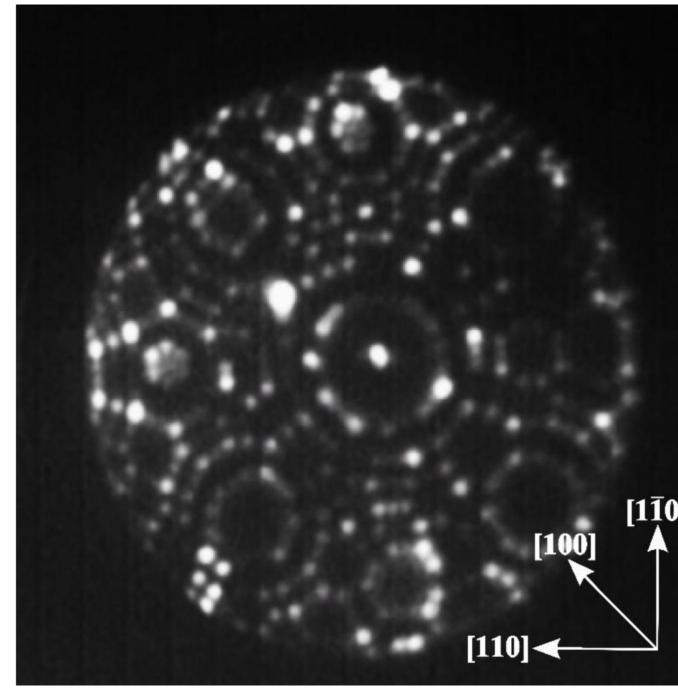




FIM (field form), Ni(001)

M. Moors et al., ACS-Nano **3** (2009) 511

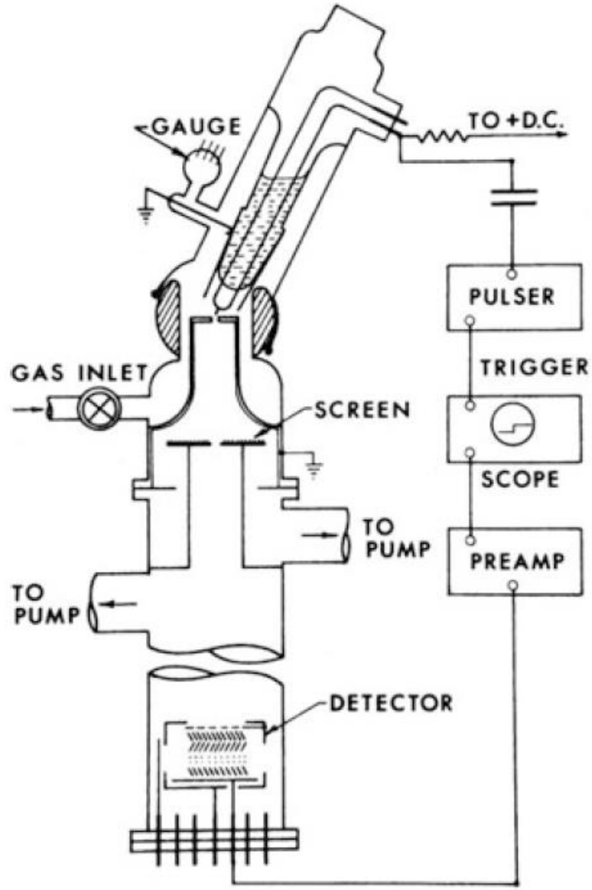
(W: BCC)



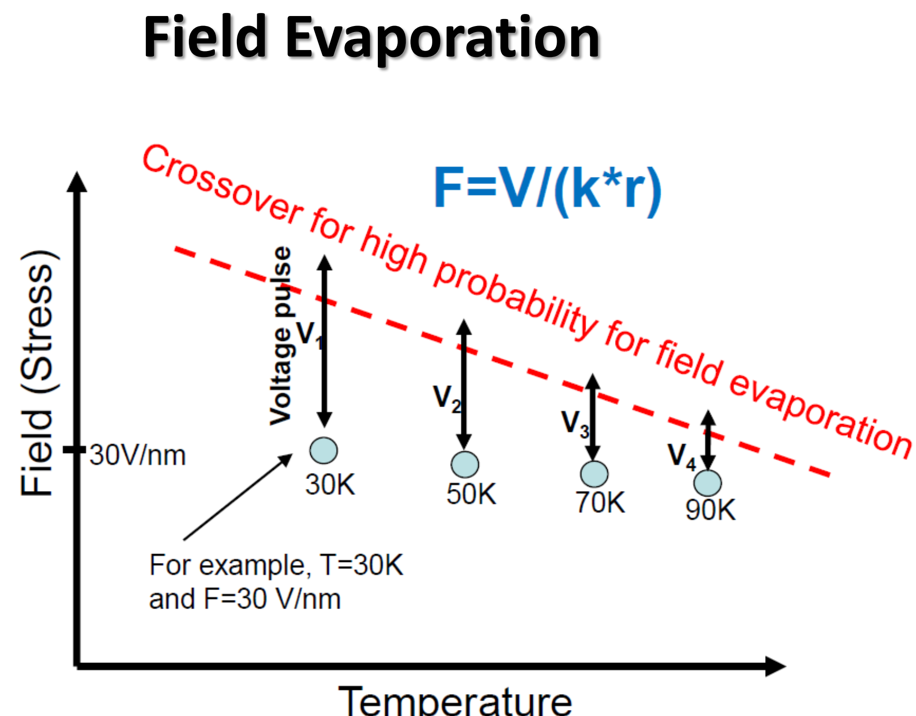
FIM (field form), W(001)

T. Olewicz et al., PRB **89** (2014) 235408

Atom Probe Field Ion Microscope – Voltage Mode

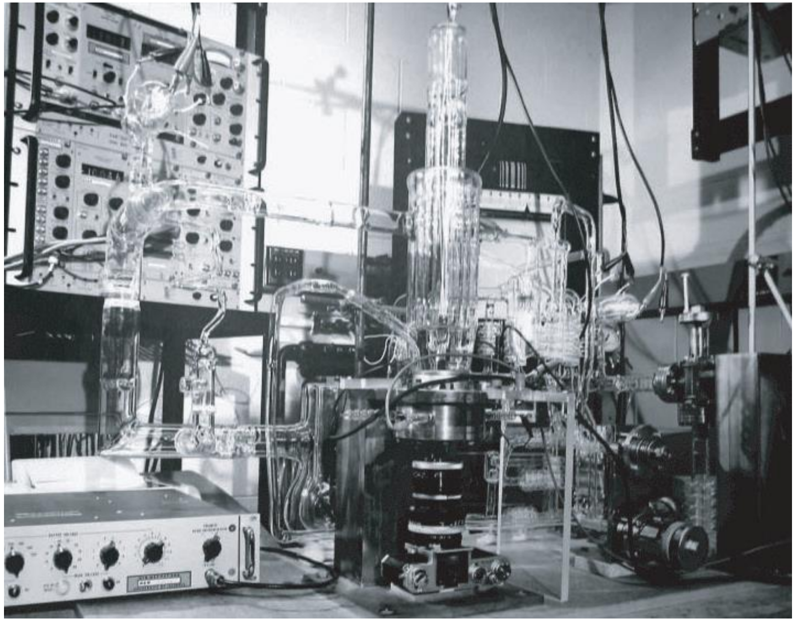


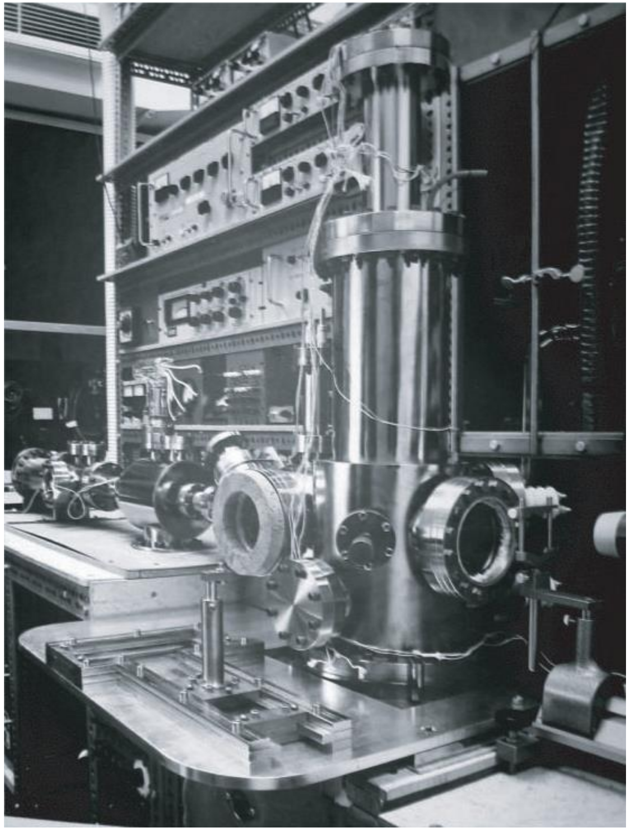
E. W. Müller, J. A. Panitz and S. B. McLane, *The atom-probe field ion microscope*. Rev. Sci. Instrum. **39** (1968) 83–86.



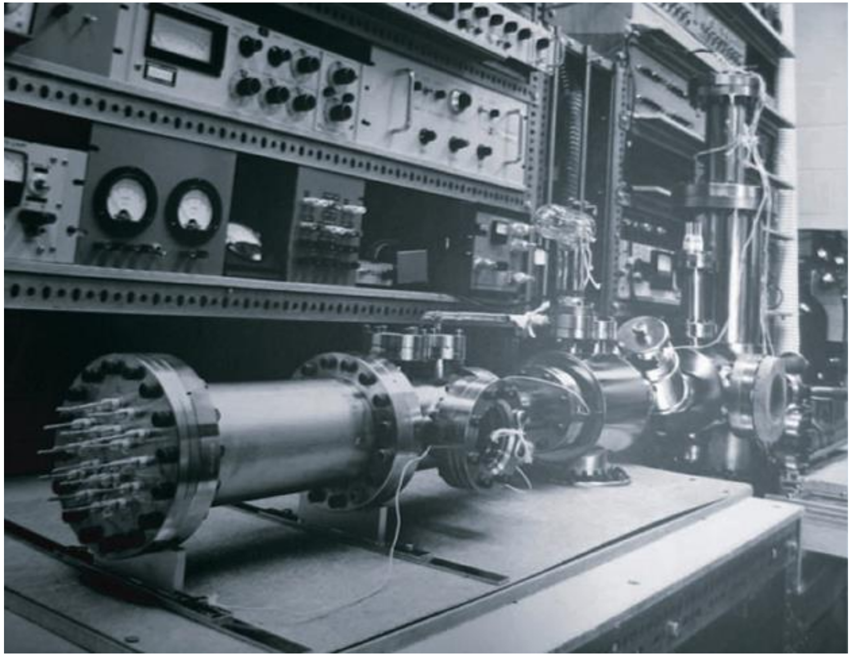
Adapted from: CAMECA, 2019

Early AP-FIM Development at Univ. of Illinois



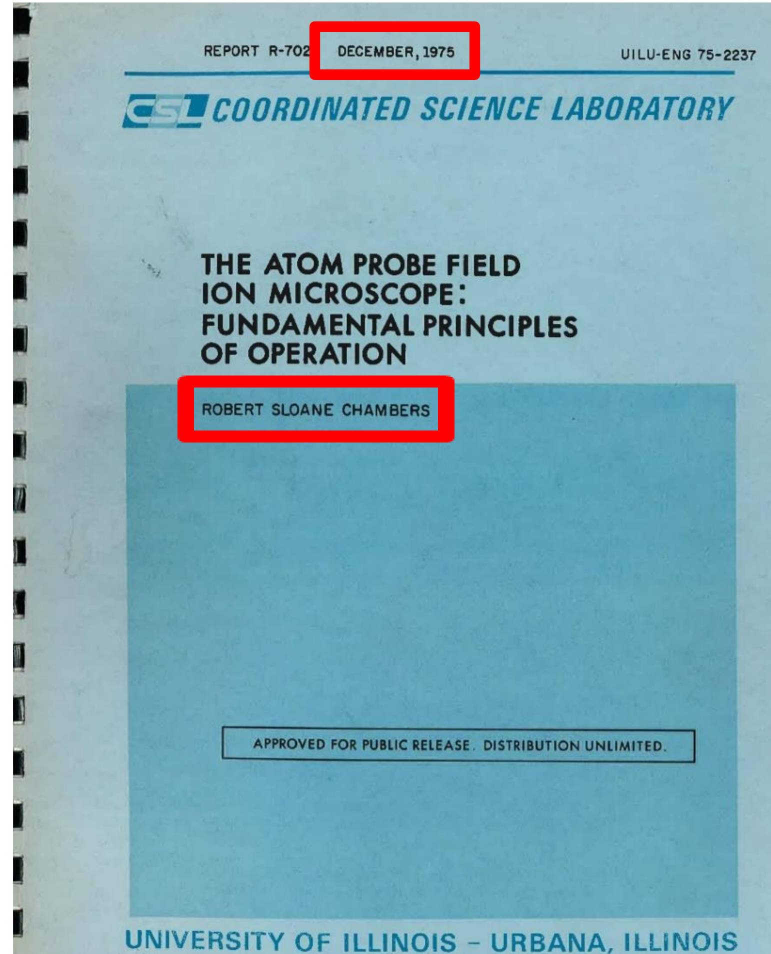


1968-1976: Prof. Gert Ehrlich and Rob Chambers;
FIM stand alone (glass) – left,
FIM side of FIM-AP (all metal) – right.



1968-1976: Prof. Gert Ehrlich and Rob Chambers; AP side of FIM-AP (all metal).

Gert Ehrlich
1926-2012



REPORT R-702 DECEMBER, 1975 UILU-ENG 75-2237

CSL COORDINATED SCIENCE LABORATORY

THE ATOM PROBE FIELD ION MICROSCOPE: FUNDAMENTAL PRINCIPLES OF OPERATION

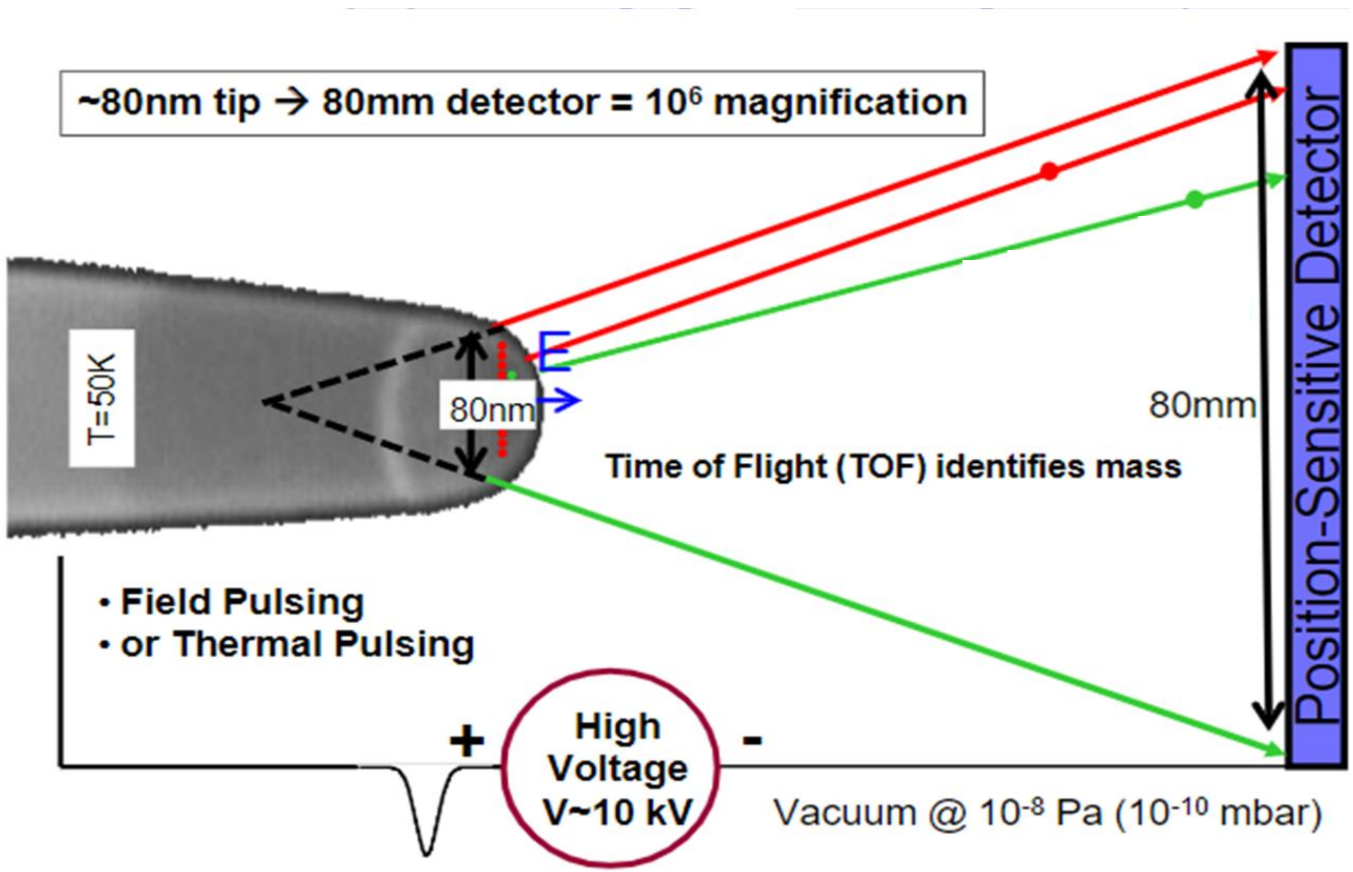
ROBERT SLOANE CHAMBERS

APPROVED FOR PUBLIC RELEASE. DISTRIBUTION UNLIMITED.

UNIVERSITY OF ILLINOIS – URBANA, ILLINOIS

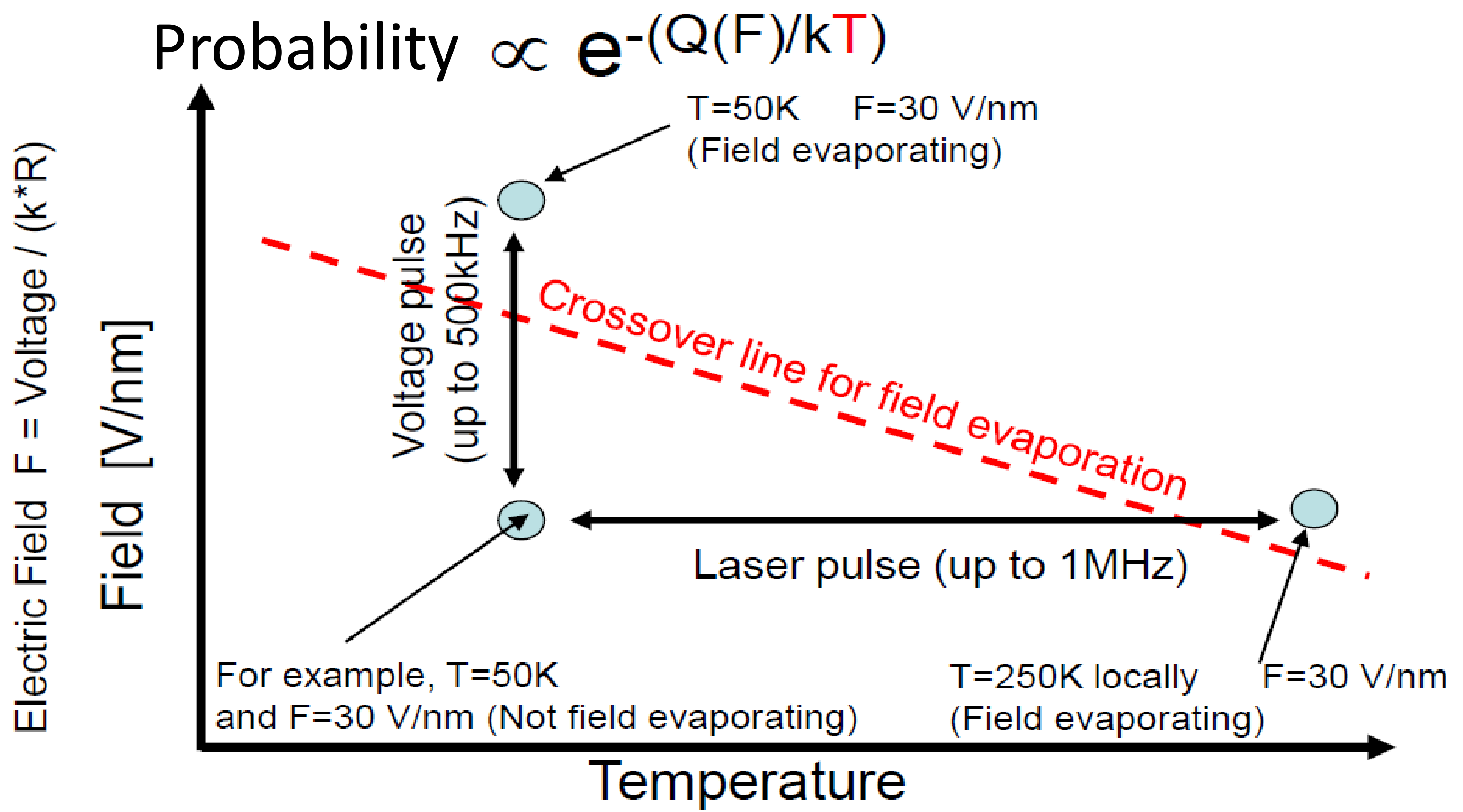
R.S. Chambers and G. Ehrlich, J. Vac. Sci. Technol., **13** (1), 273, 1976.

Atom Probe projection system



Adapted from: CAMECA, 2019

Atom Probe Ion Emission



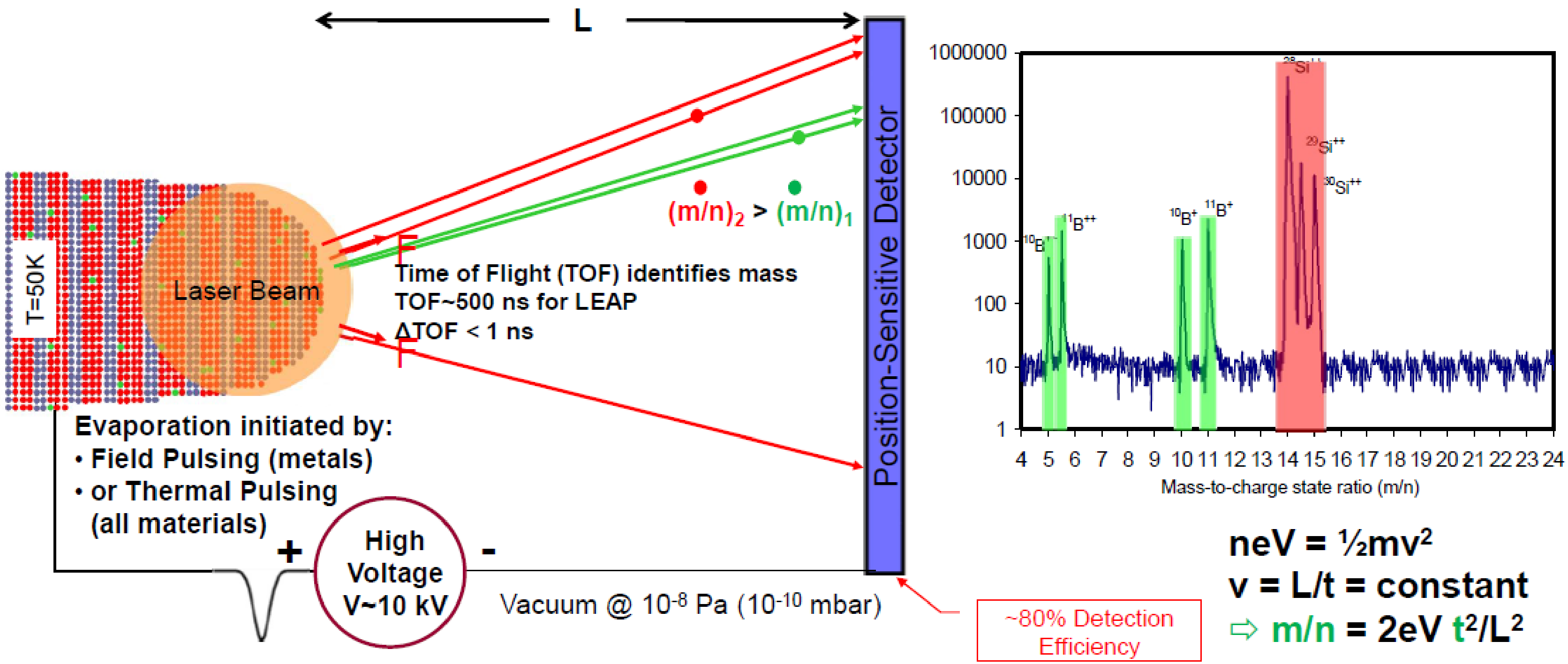
Adapted from: CAMECA, 2019

Field Evaporation of Ions – Mass Spectrometry

Description of Atom-Probe Operation

CAMECA
SCIENCE & METROLOGY SOLUTIONS

Atom Probe = projection imaging with time-of-flight mass spectrometer



$$neV = \tfrac{1}{2}mv^2$$
$$v = L/t = \text{constant}$$
$$\Rightarrow m/n = 2eV\, t^2/L^2$$

AMETEK
MATERIALS ANALYSIS DIVISION

Adapted from: R.M. Ulfig (CAMECA, 2020)

Local Electrode Atom Probe

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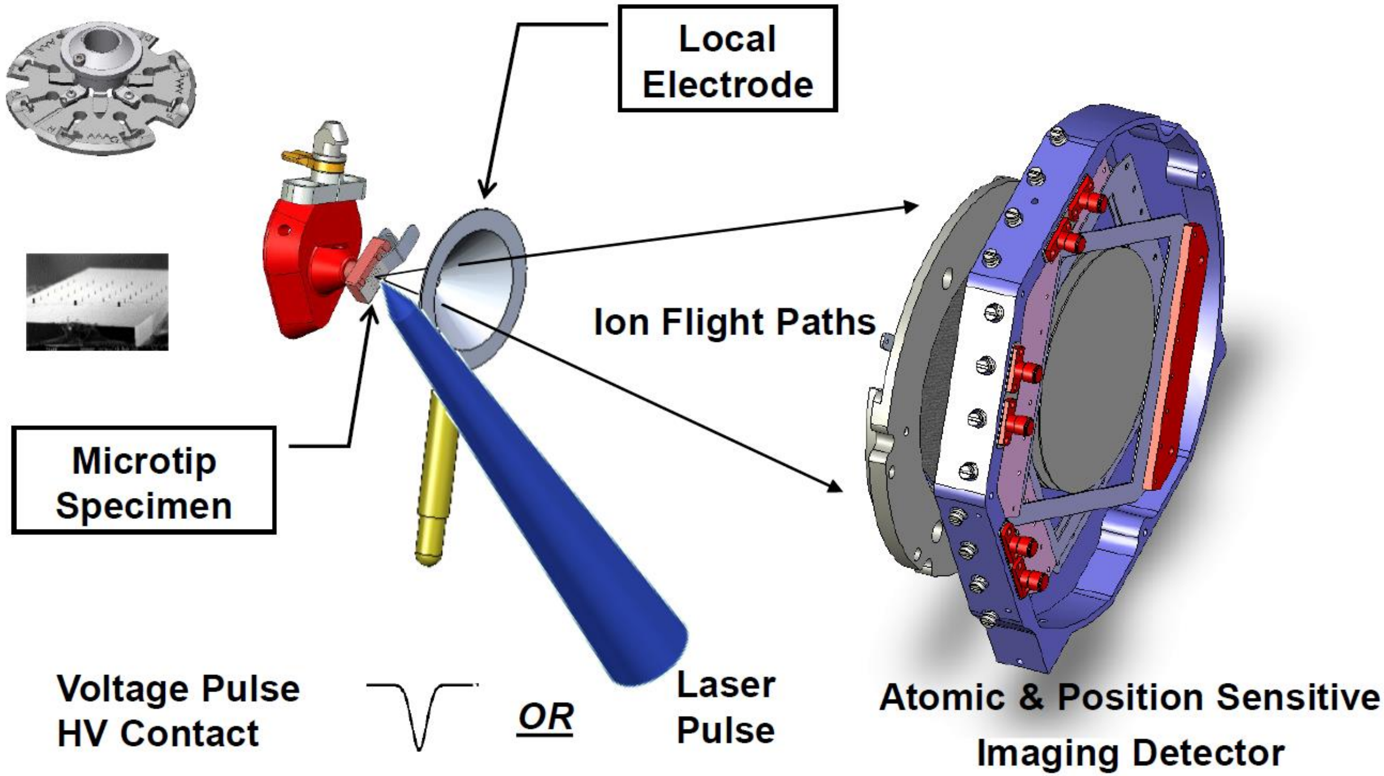
SCIENCE & METROLOGY SOLUTIONS

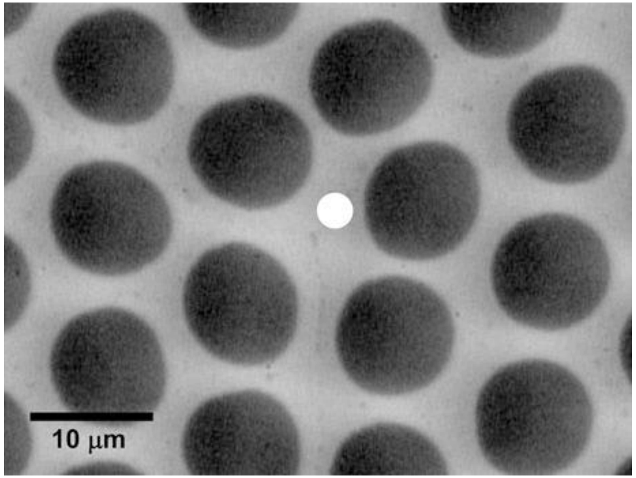
The LEAP® Microscope Geometry



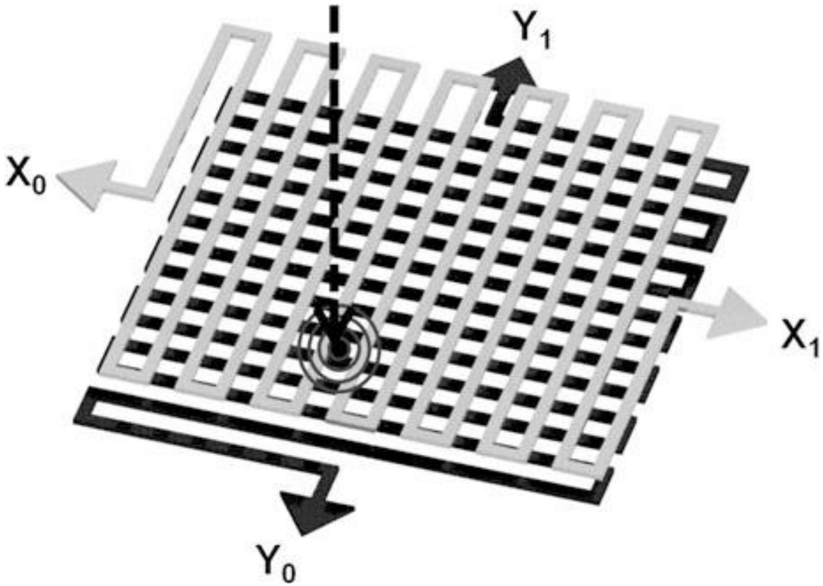
Straight Flight Path 'DT-200' Shown

Position Sensitive Detector

Microchannel Plate (MCP)

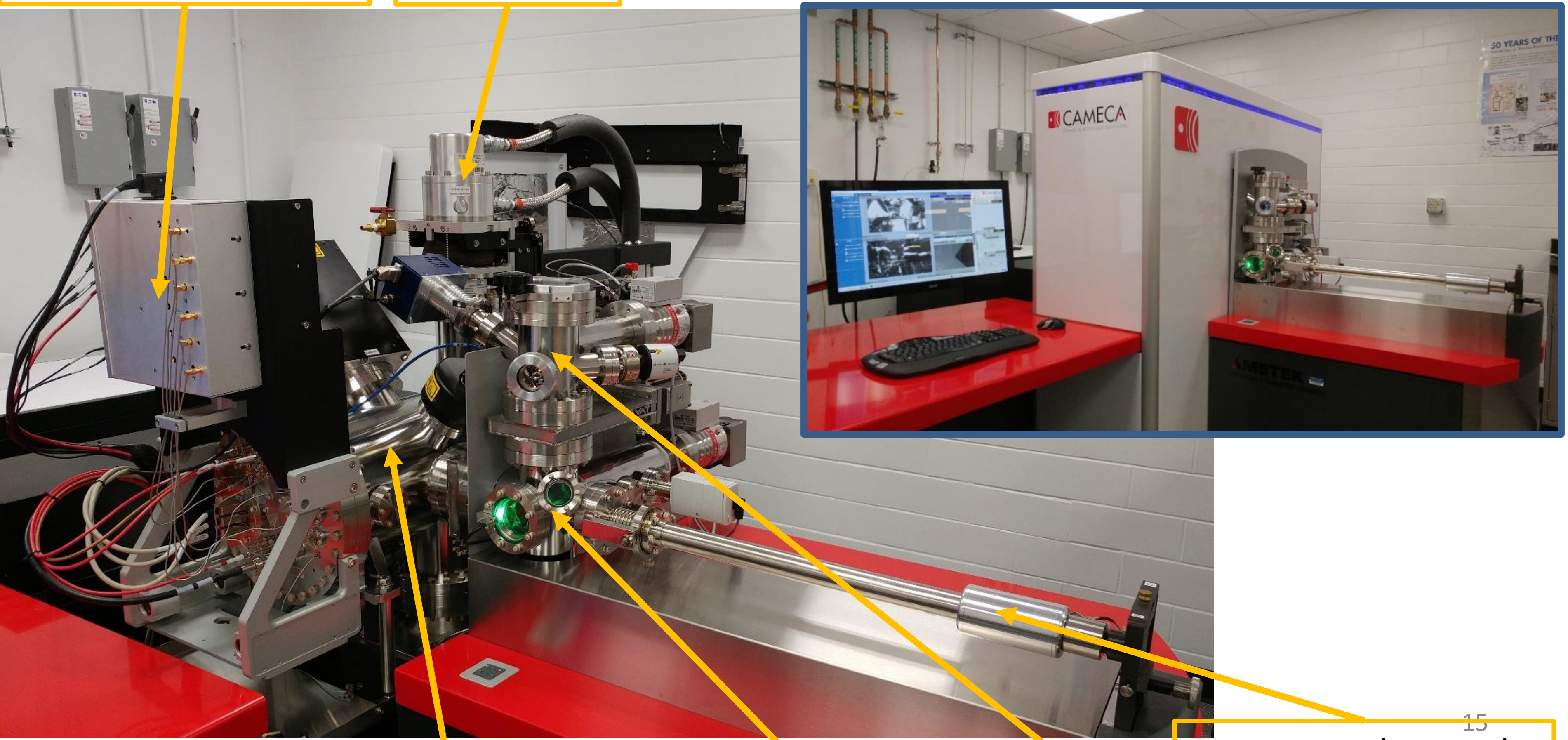


Delay Line Anode (DLA)

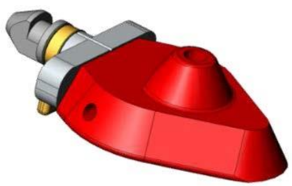


Adapted from D.J. Larson et al., Local Electrode APT, book 2013

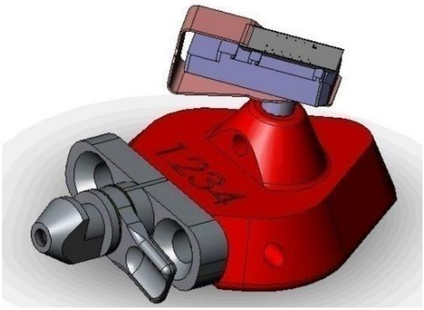
LEAP 5000 XS / CAMECA (AMETEK)



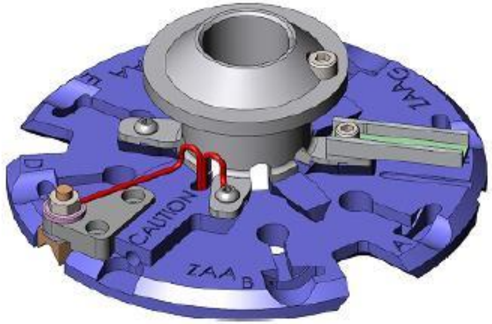
Sample Puck



Puck with Sample Coupon

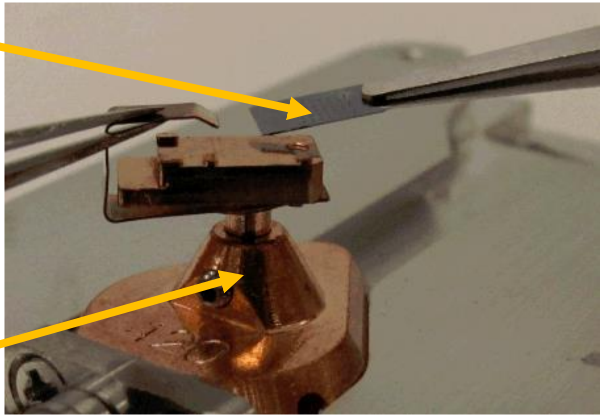


Sample or Local Electrode Storage Carousel

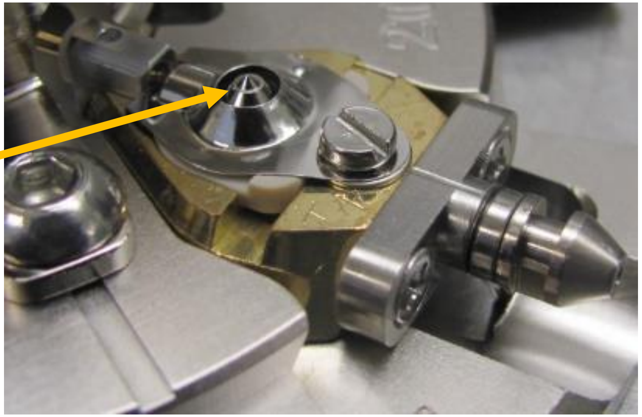


Coupon

Sample Puck

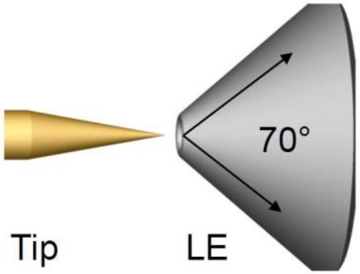


Local Electrode

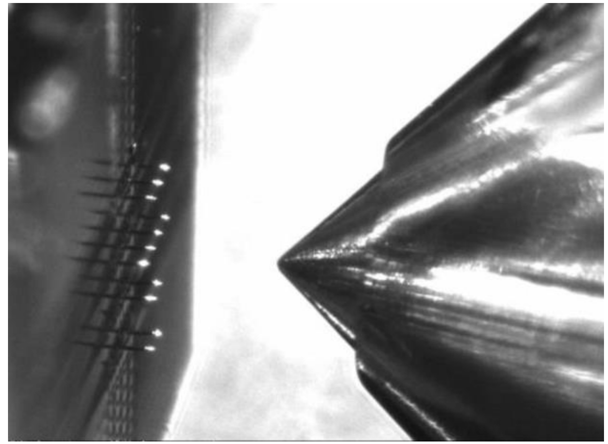


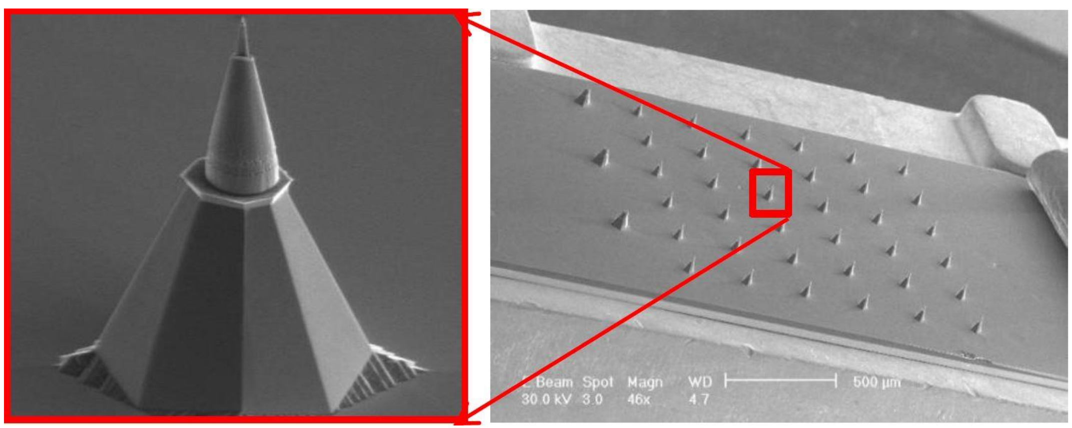
https://www.atomprobe.com/keyaptlinks/options-accessories-consumables

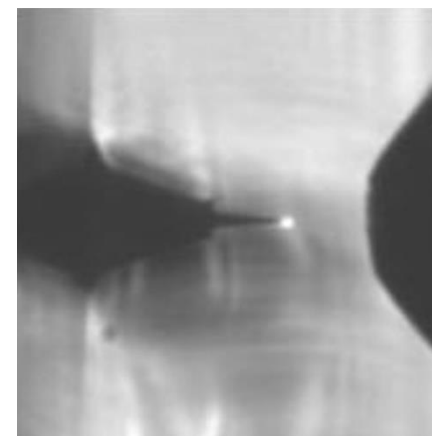
Local Electrode Atom Probe



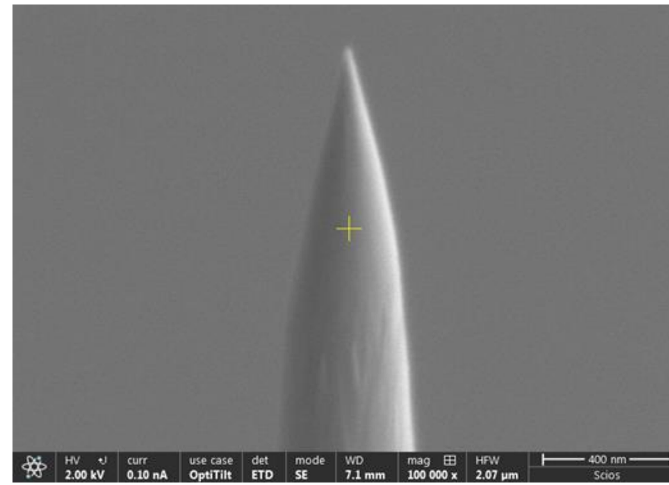
Model Parameters
- 50 nm tip radius
- 10° shank angle
- 50 micron wire
- 1 mm long
- Specimen-to-local-electrode distance is that required to permit a 70° geometric FOV



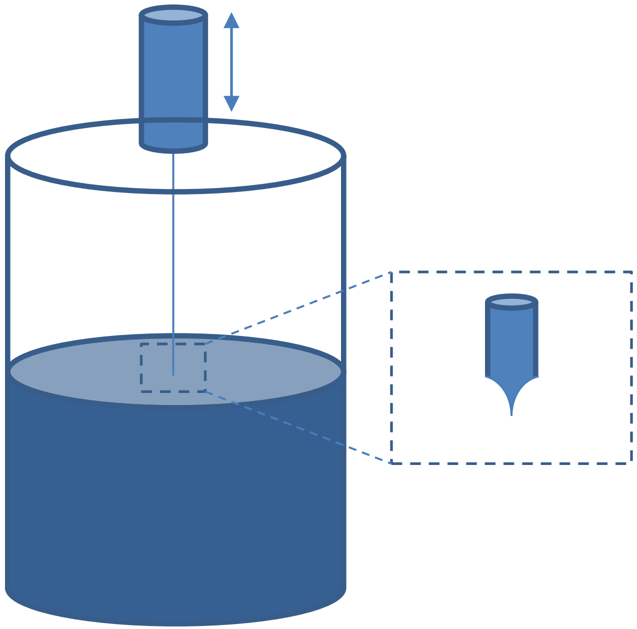




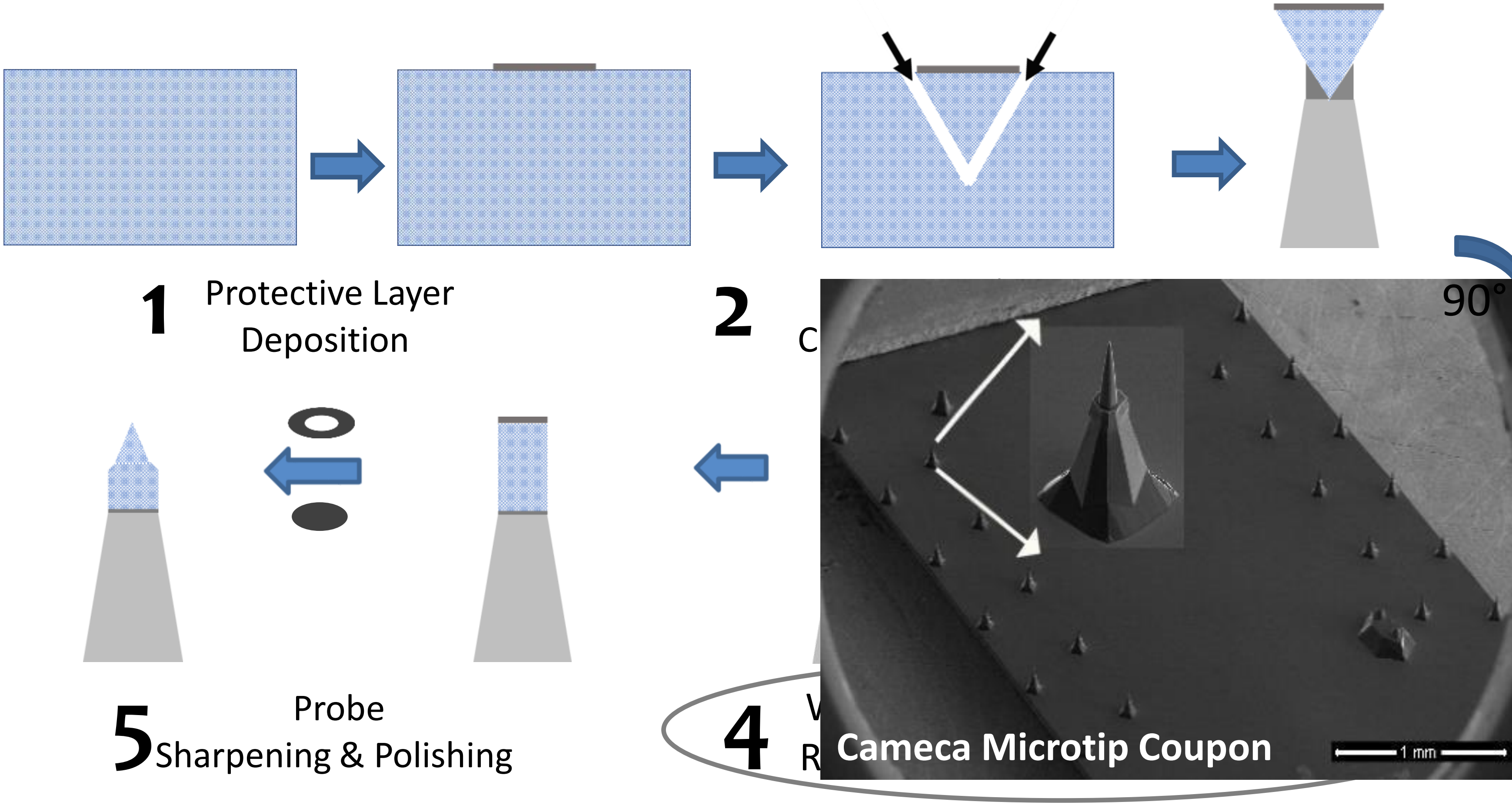
- Requirements for APT samples
  - Specimens must be sharp with a radius of curvature of ~100 nm or less
  - Feature of interest within 50 to 150 nm of specimen apex



- Electrochemical process where material is removed leaving a sharp tip
- Polishing done using meniscus of electrolyte
- Electrolyte chosen based upon material being polished



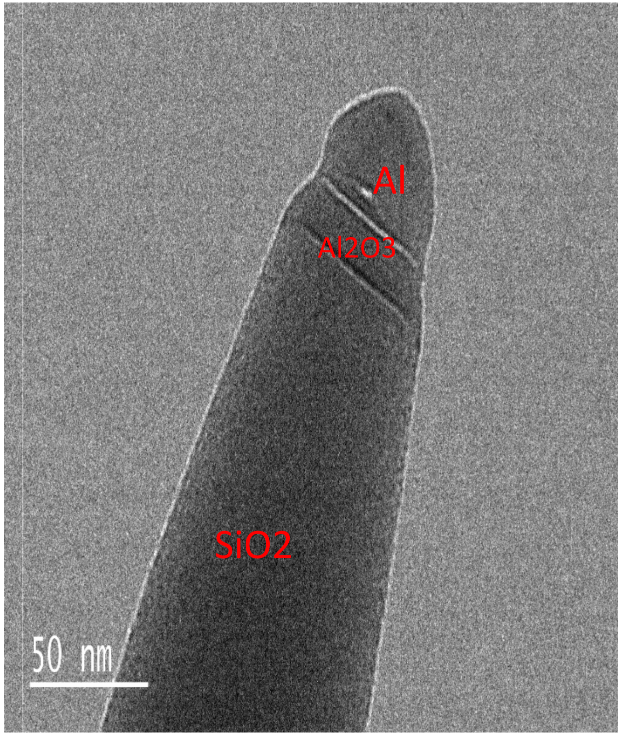
Schematic of APT-tip FIB Preparation



Analysis of Dielectric Layer of Quantum Dot Devices

APT analysis of semiconductor quantum dot devices to investigate the effects of impurities and roughness at the interfaces around the Al_2O_3 dielectric layer.

Goal: To determine the relationship between transport properties of the quantum dot and interface imperfections.



Sputtered Al (100 nm)

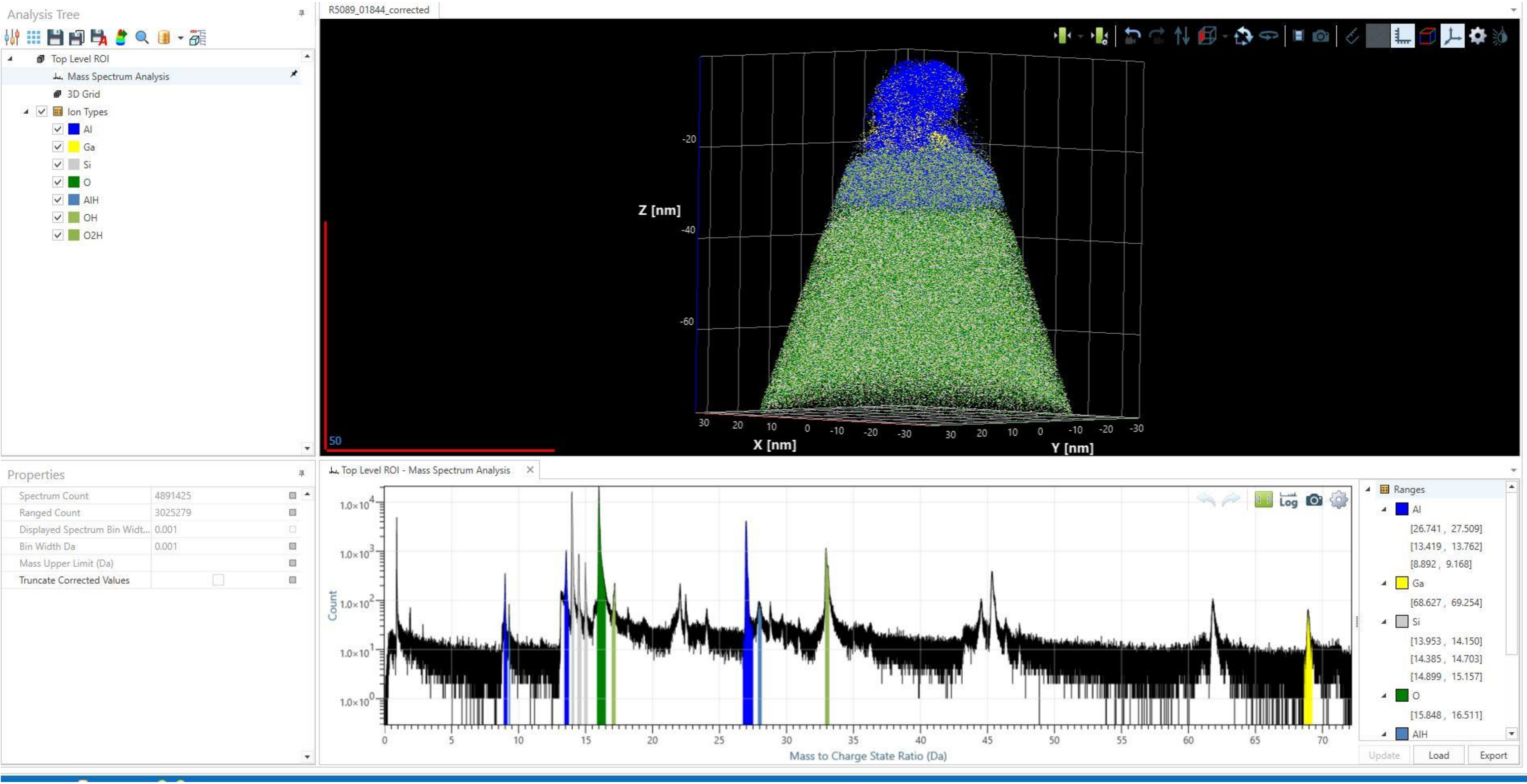
ALD Al_2O_3 (10-15 nm)

SiO_2 (1000 nm)

Si substrate

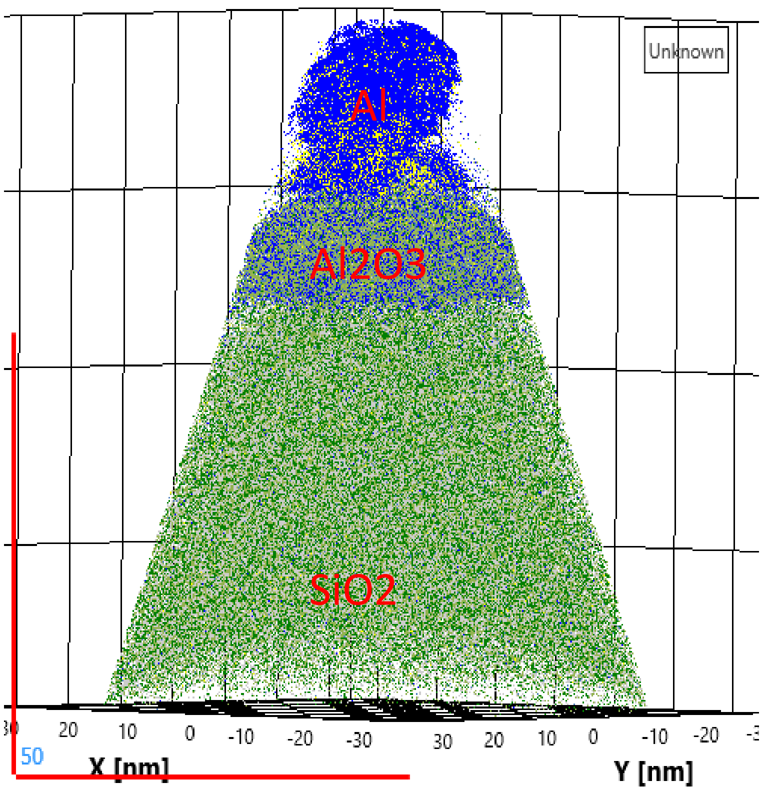
Courtesy: J. Huang and N. Mason, University of Illinois

Analysis of Dielectric Layer of Quantum Dot Devices

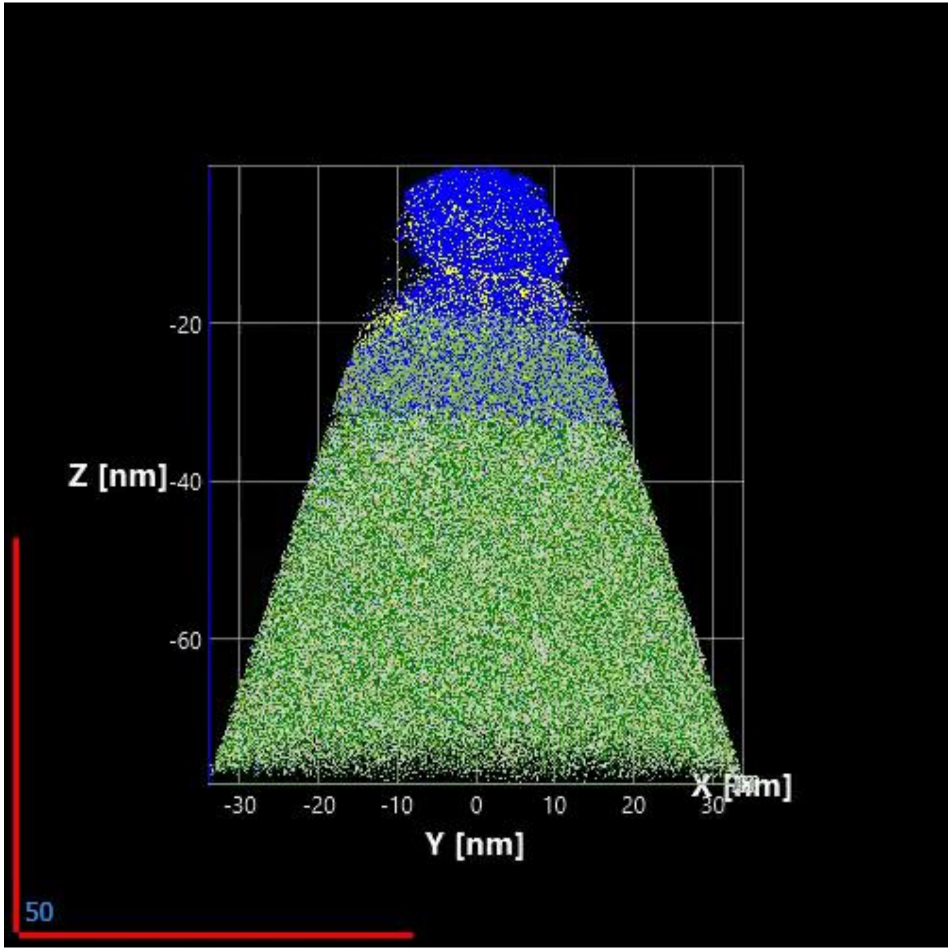


APT Reconstruction: Al in blue, O in green, Si in gray, Ga in yellow.

Analysis of Dielectric Layer of Quantum Dot Devices



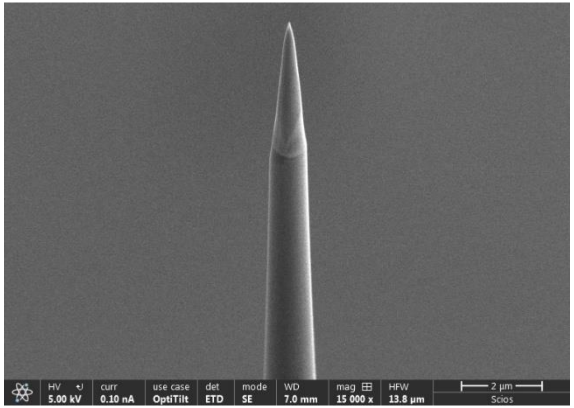
Al in blue, O in green, Si in gray

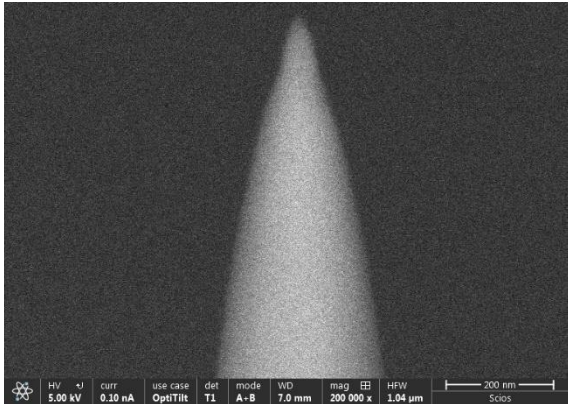


APT Analysis of Proton Irradiated Mixed Phase 308L Stainless Steel

- FIB of needle specimen
- Approximately 80M hits in laser pulse mode
- Standard analysis using IVAS:
  - 1-D concentration profile
  - Cluster Analysis
  - Nearest Neighbor Analysis
    - Local Concentration
    - Cluster composition analysis for two separate regions
  - Cluster Size Analysis
  - Cluster Composition Analysis
  - Iso-surfaces
    - Si/Ni
  - Frequency Distribution Analysis

Samples courtesy: B. Heuser, University of Illinois
FIB preparation: H. Zhou, University of Illinois
APT measurement: W. Swiech, University of Illinois



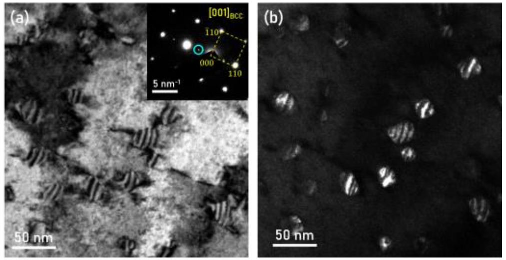


SEM Images

APT Analysis of Proton Irradiated Mixed Phase 308L Stainless Steel

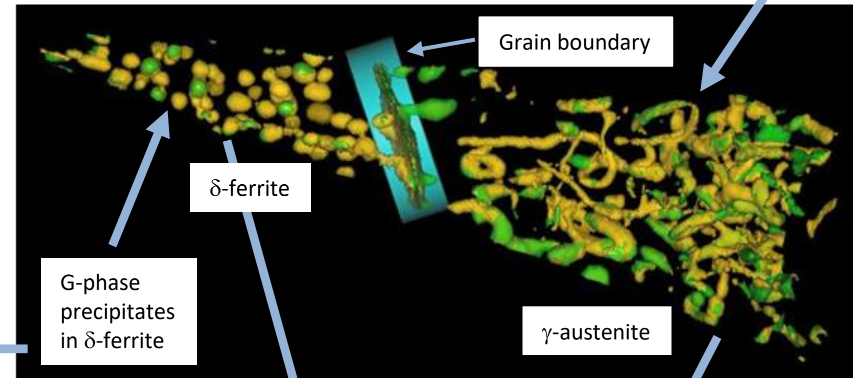
Mixed phase: δ-ferrite and γ-austenite

TEM dark field of G-phase particles



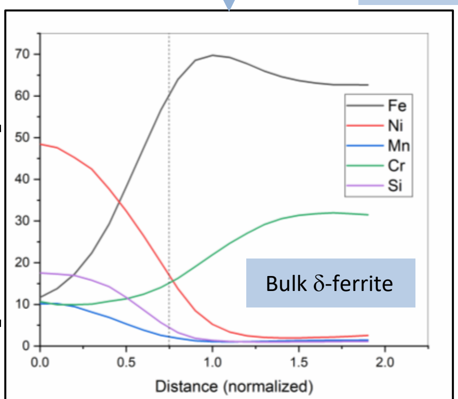
2:10% Si:Ni iso-surface

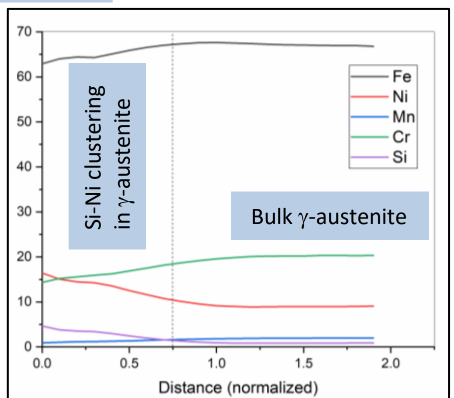
Si-Ni clustering in γ-austenite



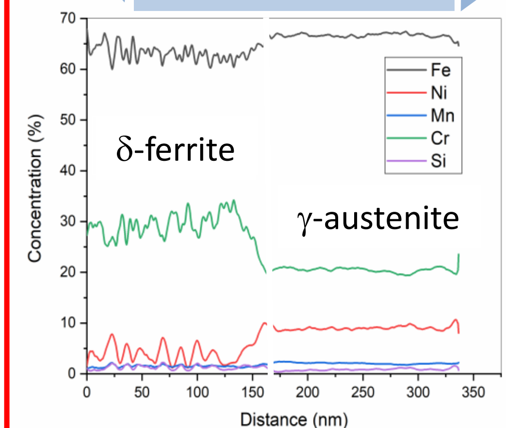
G-phase

Cluster Analysis





1-D composition profiles

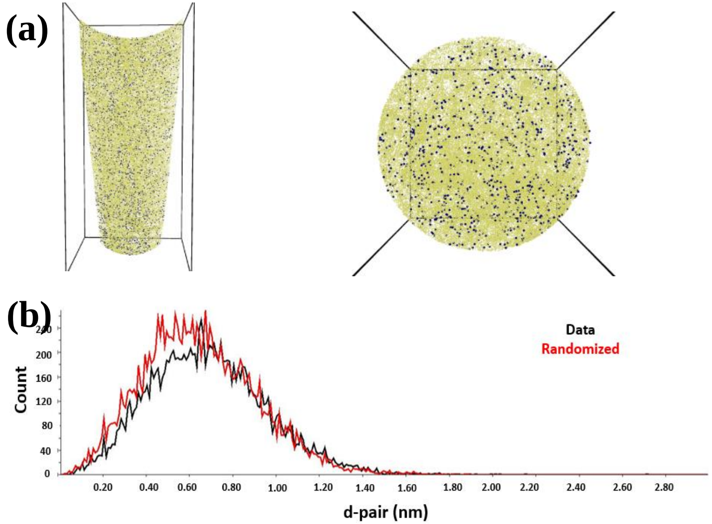


Courtesy: B. Heuser, University of Illinois

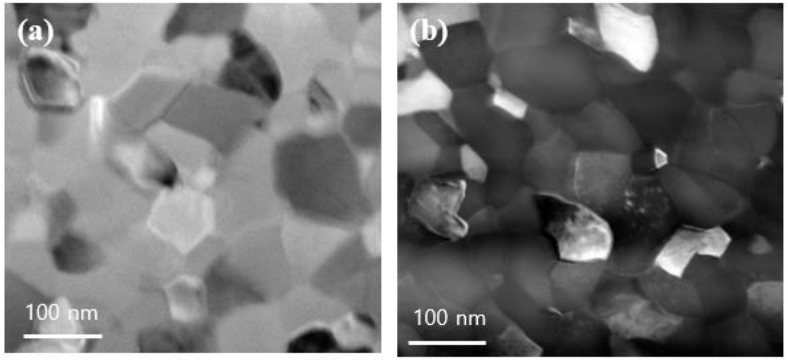
Evolution of Dilute Al-Sc Alloys During Annealing

APT analysis

Atom	Al	Sc	Ga	O	C	Pt	H
Composition (at.%)	98.17	1.37	0.1	0.01	0.01	0.04	0.32



STEM analysis

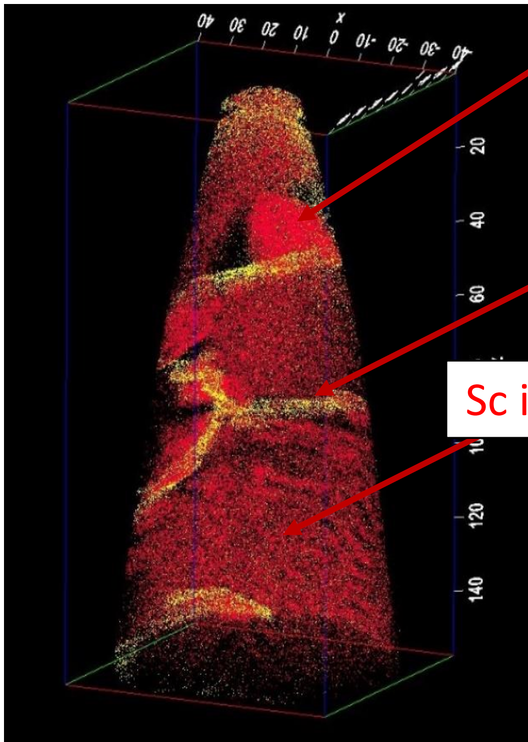


As-grown

Annealed for 1 hour at 150 °C

Courtesy: S. Jana, S. Kim, R.S. Averback, P. Bellon, University of Illinois

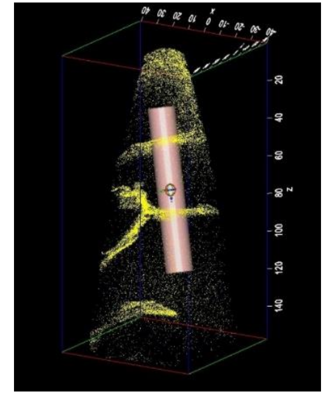
Grain Boundary Depletion: Annealing at 180 °C



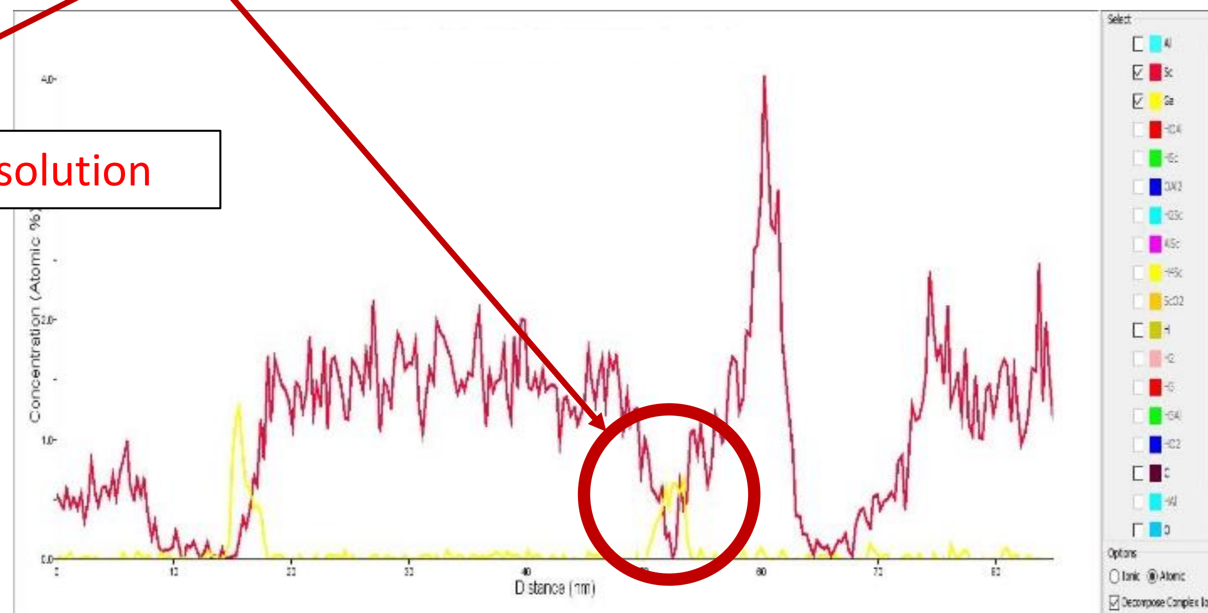
Sc-rich precipitate

Sc-depleted GB

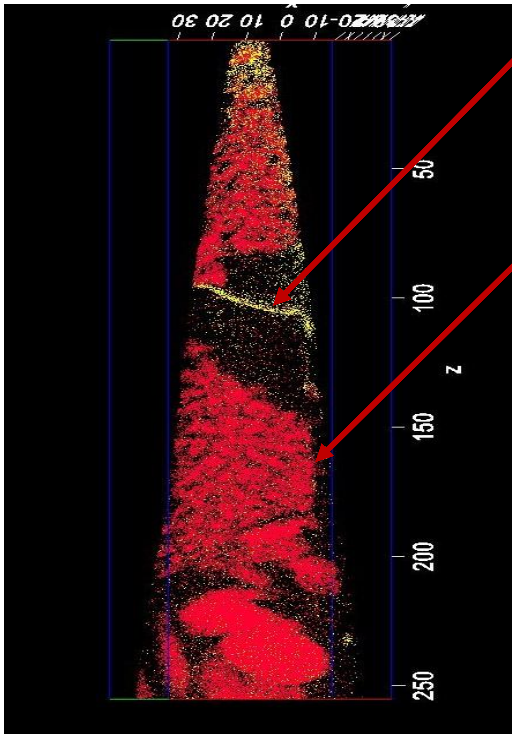
Sc in solution



1D concentration profile along cylinder

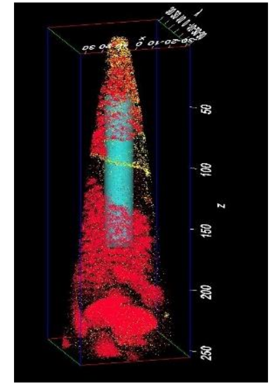


Grain Boundary Depletion: Annealing at 300 °C

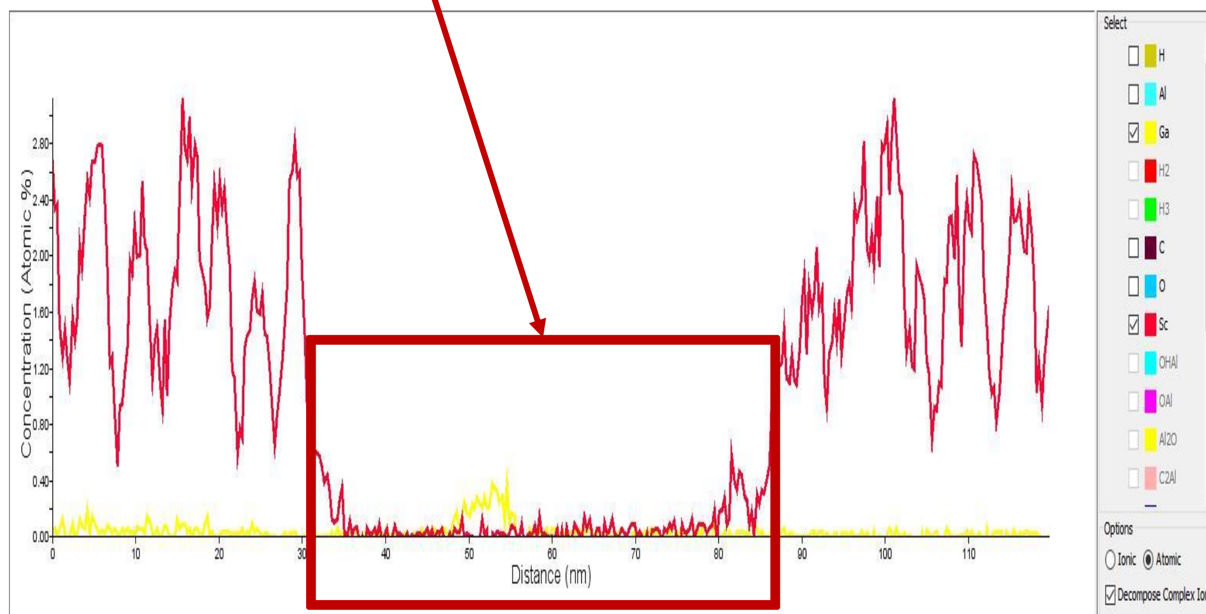


Sc-depleted GB

Sc-rich nanoclusters



1D concentration profile along cylinder



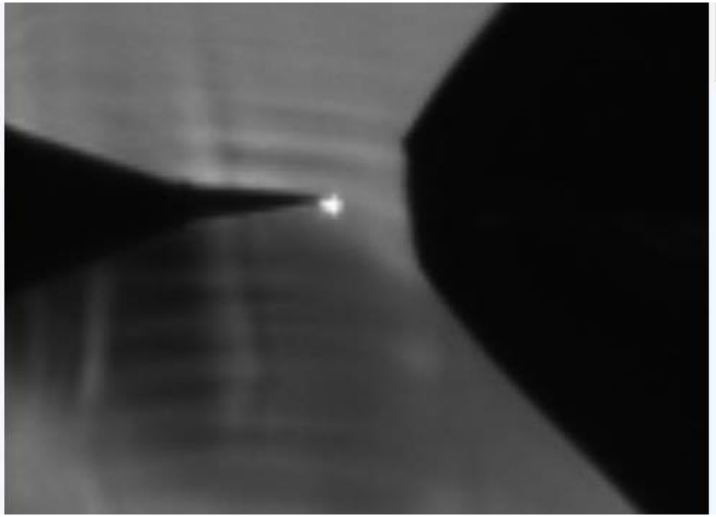
Atom Probe Tomography

Spatial Resolution
- 0.1 – 0.3 nm in depth
- 0.3 – 0.5 nm laterally

Field of View
- 100 – 200 nm laterally

Time-of-flight mass analysis
- Mass range from 1 – 600 amu

Compositional analysis
- Near 100% ionization of emitted atoms
- Up to 80% of all atoms analyzed
- Sensitivity ~ ppm



Well suited for analysis of
- Precipitates
- Grain boundaries
- Isotopic variations

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