Frontiers of Atom Probe Microscopy

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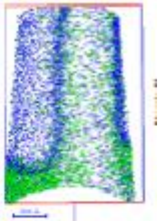
Outline

- Introduction
  - The atom probe basics
  - Brief description of analysis capabilities
- Sample preparation techniques
- Applications
  - Dopant profiles
  - 3-D structures
  - III-V multi-layers

Technology Timeline



Kambham et al.

IAP
Panitz

PoSAP
Cerezo et al.

LEAP
Kelly et al.

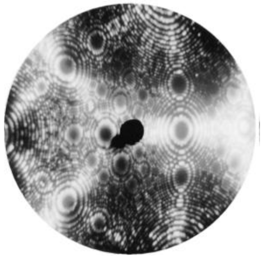
1^{st} commercial
PLAP

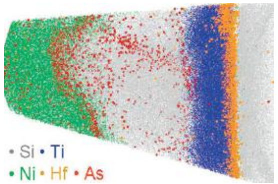
APFIM
Muller et al.

PLAP
Kellog and Tsong

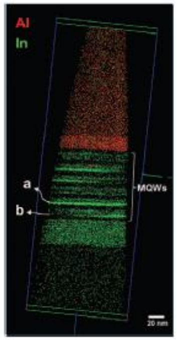
SAP
Nishikawa et al.

Groups
reexamine PLAP





Panciera et al.



Choi et al.

1960 1970 1980 1990 2000 2010 2020

State of the art in APT

Mass Resolving Power (MRP): 1500

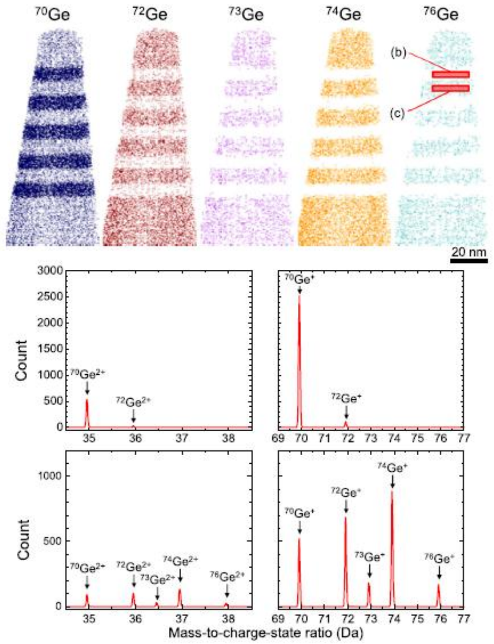
Data Collection Rate (ions/min): > 5M

Field of View (nm): > 200

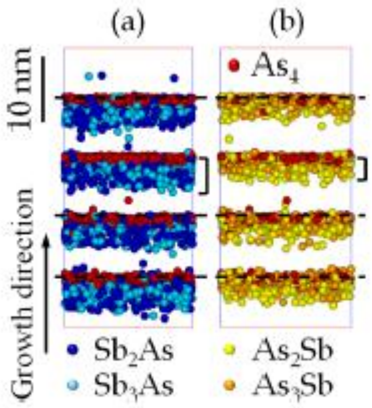
Lateral Resolution (nm): 0.2

Depth Resolution (nm): 0.05

- Sample preparation is a critical component of the technique
- APT is becoming more versatile technique with the ability to analyze non-metallic samples with the assistance of laser pulsing



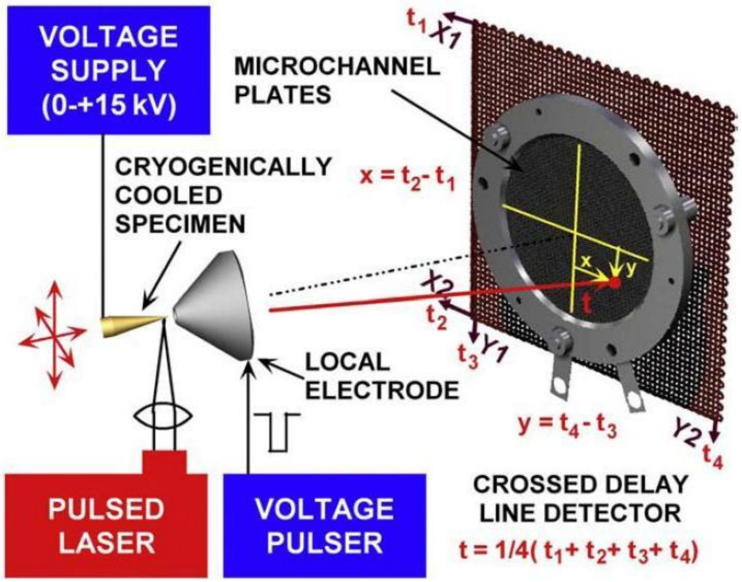
Shimizu et al., JAP 2013

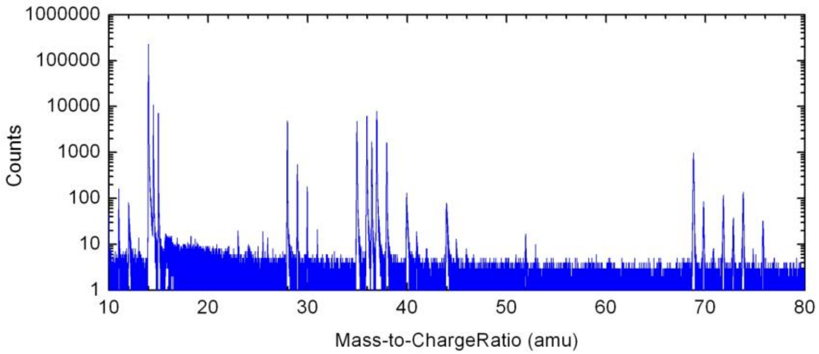


Muller et al., APL 2012

Atom Probe Tomography Primer

Field Ion Projection Microscopy + Time-of-Flight Mass Spectrometry





- Cryogenically cooled needle-shaped specimen
- Pulsed voltage or laser initiates the evaporation process
- 2D position-sensitive detector determines x,y coordinates of atoms
- Evaporation sequence determines z coordinate in 3D reconstruction
- Time-of-flight mass spectrometry determines the ion identity

Data Reconstruction Basics

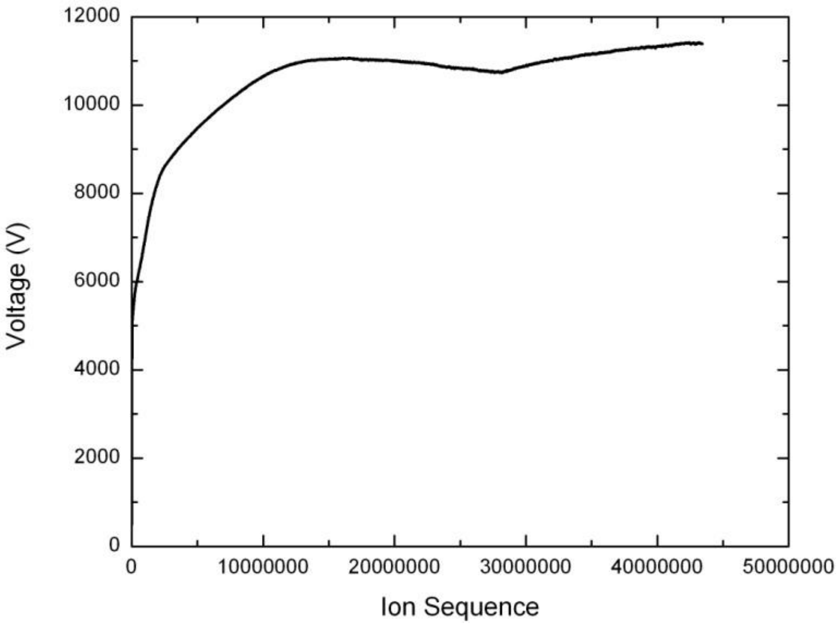
- A portion of the voltage history is selected from the raw data to analysis
- The radius of curvature of the end form is related to the voltage at the apex of the tip

$$R \propto \frac{V}{F_e}$$

R –radius of curvature

V – apex voltage

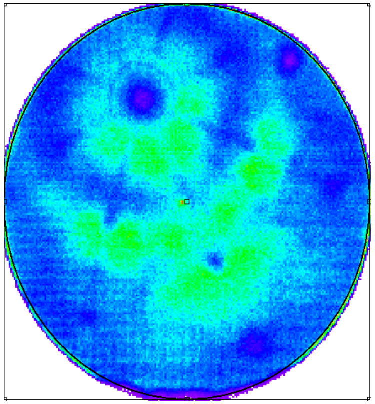
F_e – evaporation field



Data Reconstruction Basics

- XY data is converted from the detector
- A point-projection model is used to reconstruct the raw data
- The magnification of the image on the detector is given by

$$M = \frac{d}{\xi R}$$



Event histogram

ξ- the image compression factor (ICF)

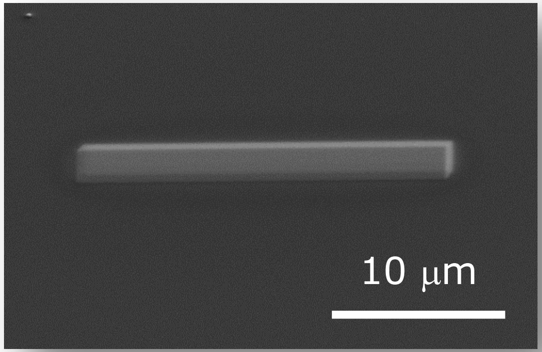
d - specimen to atom detector distance

R - radius of curvature of the specimen

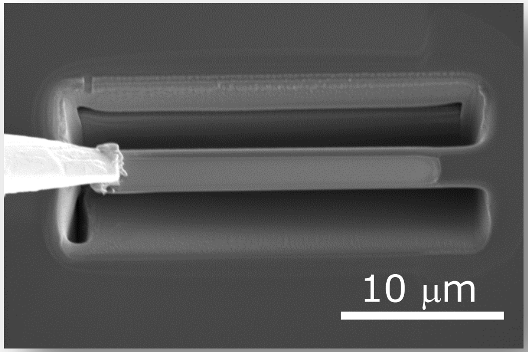
- The *z* position is determined from the order in which the ions strike the detector

Sample Preparation

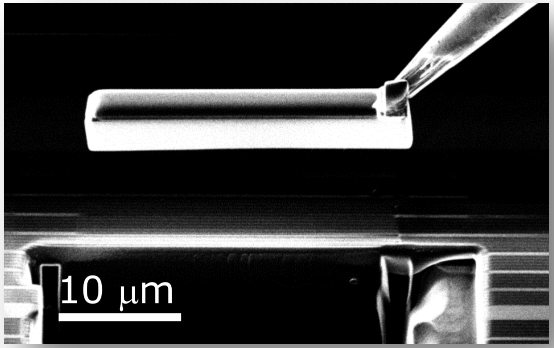
The dual beam focused ion beam (FIB) is used to cut away a wedge of material from a specific region of interest (ROI)



Protective Pt capping layer

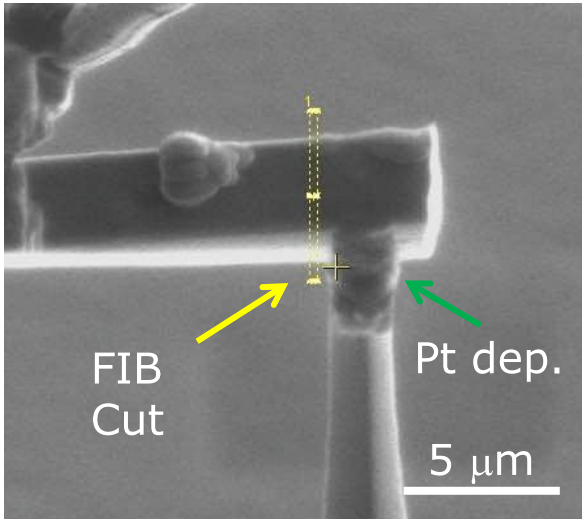


Attachment of micromanipulator to wedge

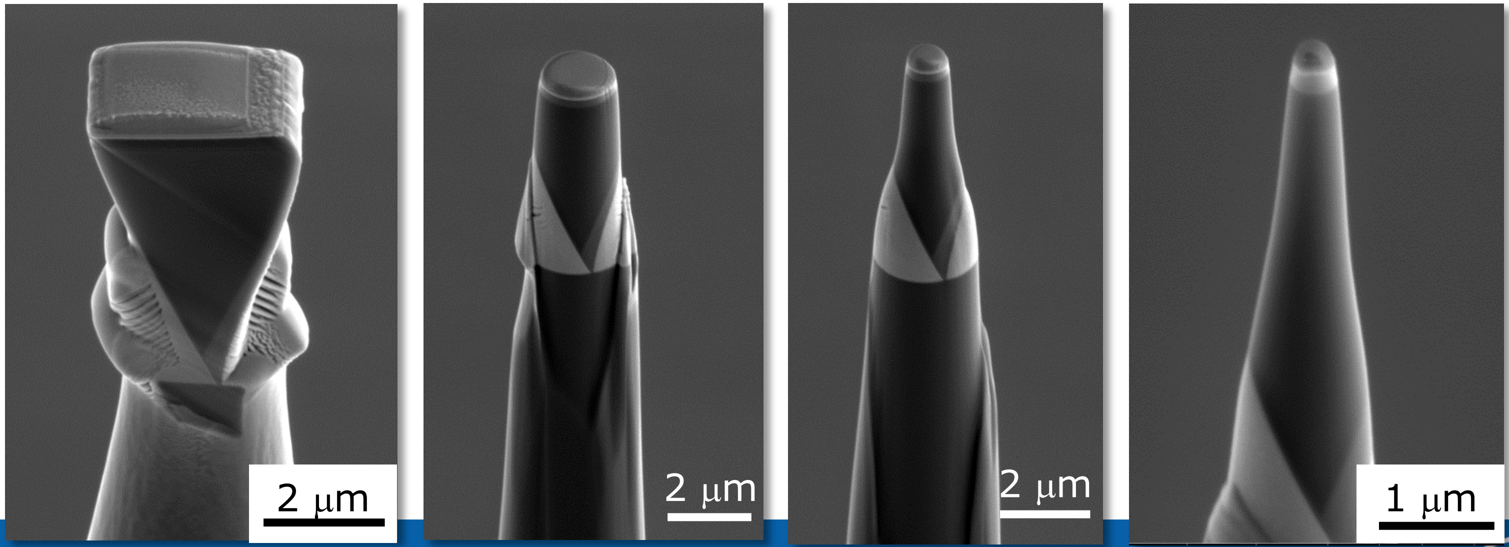


Removal of wedge from bulk sample

Sample Preparation



- An array of micropillars can be used as substrates for the wedge attachment
- Propagation of wedge provides several samples from a single ROI
- Annular milling used to form the needle-like specimen



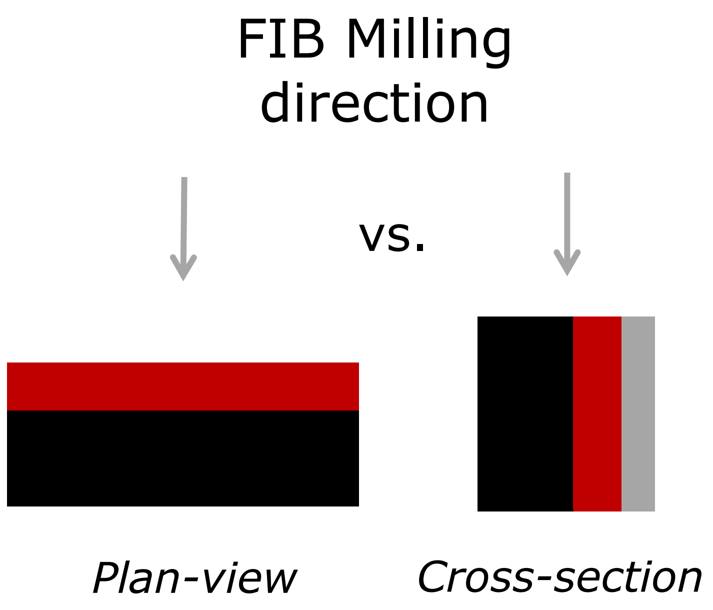
Sample Preparation

Top-down or Plan-view Method

X Region of interest is the thin film thickness or device height
X Protective coating required
X Specimen fracture at interfaces

Cross-section Method

✓ Increased region of interest
  - Thin films
  - Nano-electronic devices
✓ Minimized Ga damage during sample preparation
✓ Layers of differing evaporation fields removed simultaneously

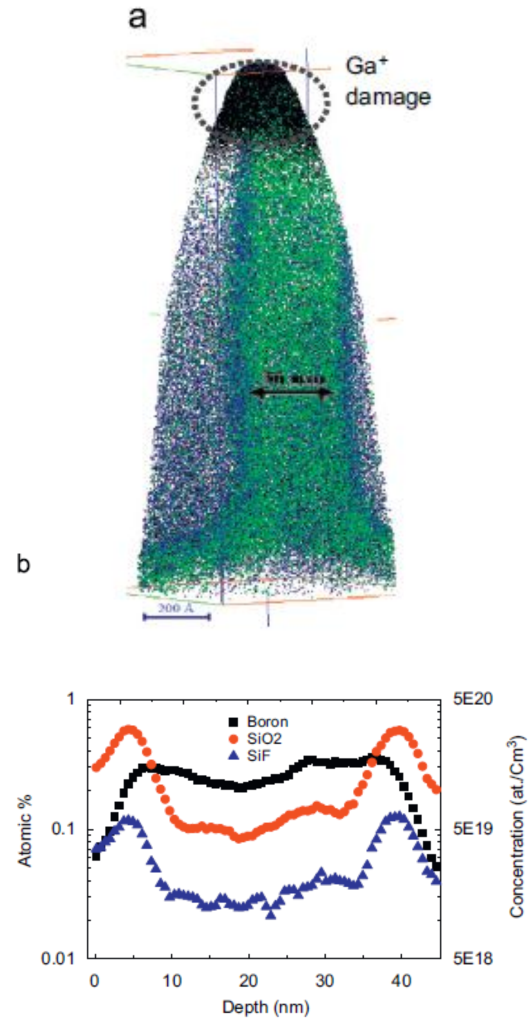


Considerations

- Reproducible geometries
- Capping layer ROI matching
- Automated sample preparation

Dopant profiles

- Secondary ion mass spectrometry (SIMS) is heavily relied upon in the industry to measure dopant profiles.
- The analyzed sample volume required to achieve maximum sensitivity is not constrained, with analyzed areas often several hundred square microns
- Mapping dopant distributions with high spatial resolution has been recognized as an important part of the roadmap for the design of future transistors
- Sub-nanometer metrology techniques are needed to characterize 3D dopant/carrier distributions in these devices to optimize the performance
- APT is ideally suited to investigate dopant distributions of device-level volumes



Kambham et al., *Ultramicroscopy* 2011

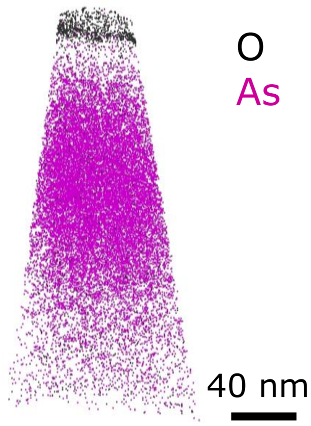
SRM – 2134 As Dopant Profile

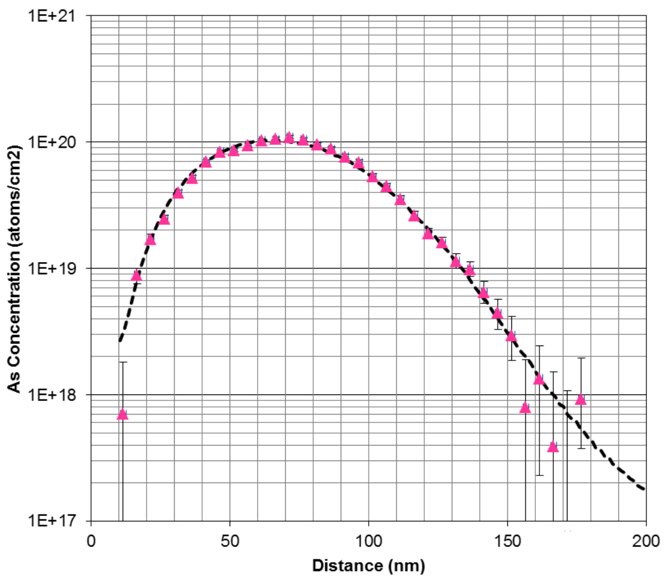
- APT measurements can provide accurate and precise measurements of dose when best practices for analysis are followed

- Consideration must be given to how specimen shape and instrumental factors affect reconstruction accuracy given available reconstruction algorithms

- The silicon planes can be resolved along the <100> pole except near the peak of the implant profile

- This internal standard allows one to evaluate the reconstruction based on the known silicon lattice spacing of 0.54307 nm





Prosa et al., M&M 2012

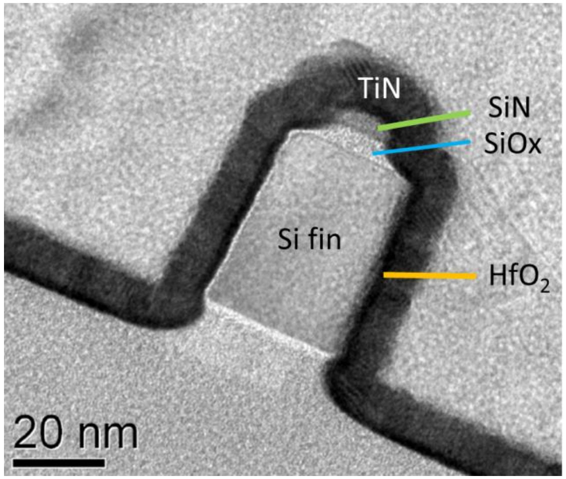
APT of 3-D Nanostructures

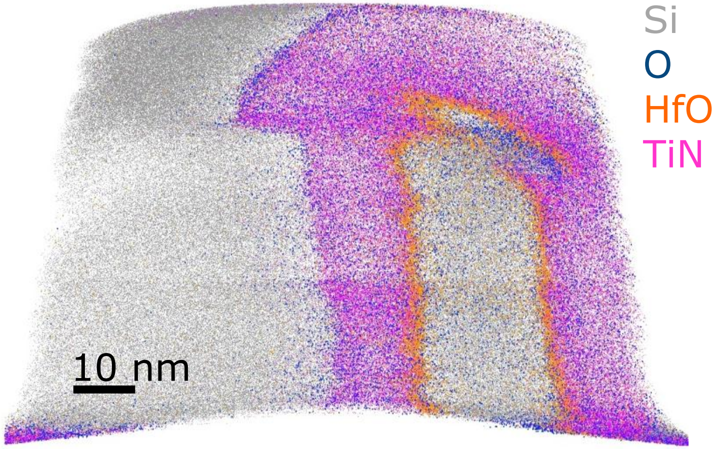
- Complex 3-D geometries comprised of several materials are used to achieve desired performance in today's CMOS technologies

- Slight process variations can affect the structural and material properties in such highly engineered devices

- APT is used to provide 3D chemical imaging of device-level volumes

- Potential challenges may occur during data acquisition or reconstruction



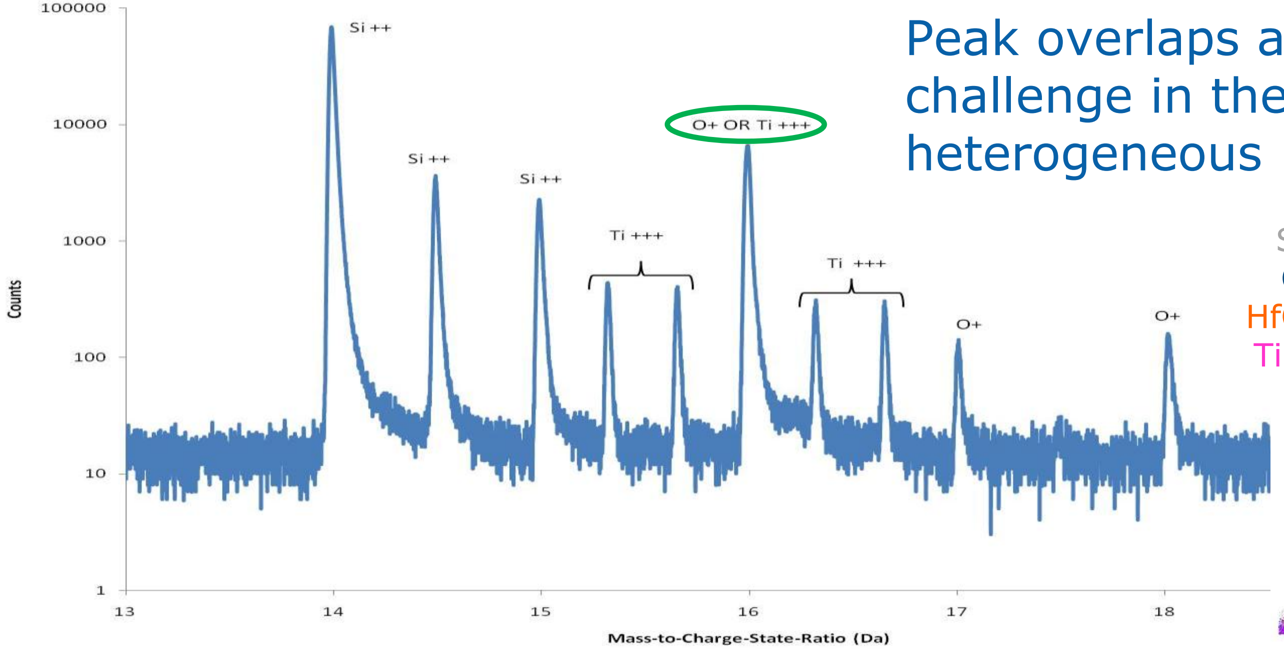


TEM image provided by K. Akarvardar

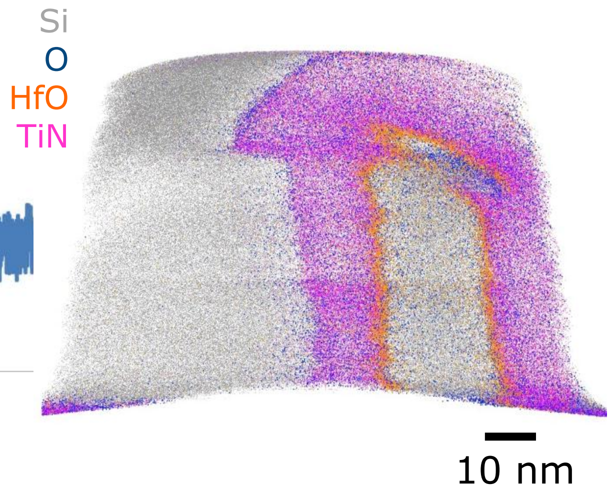
APT of 3-D Nanostructures

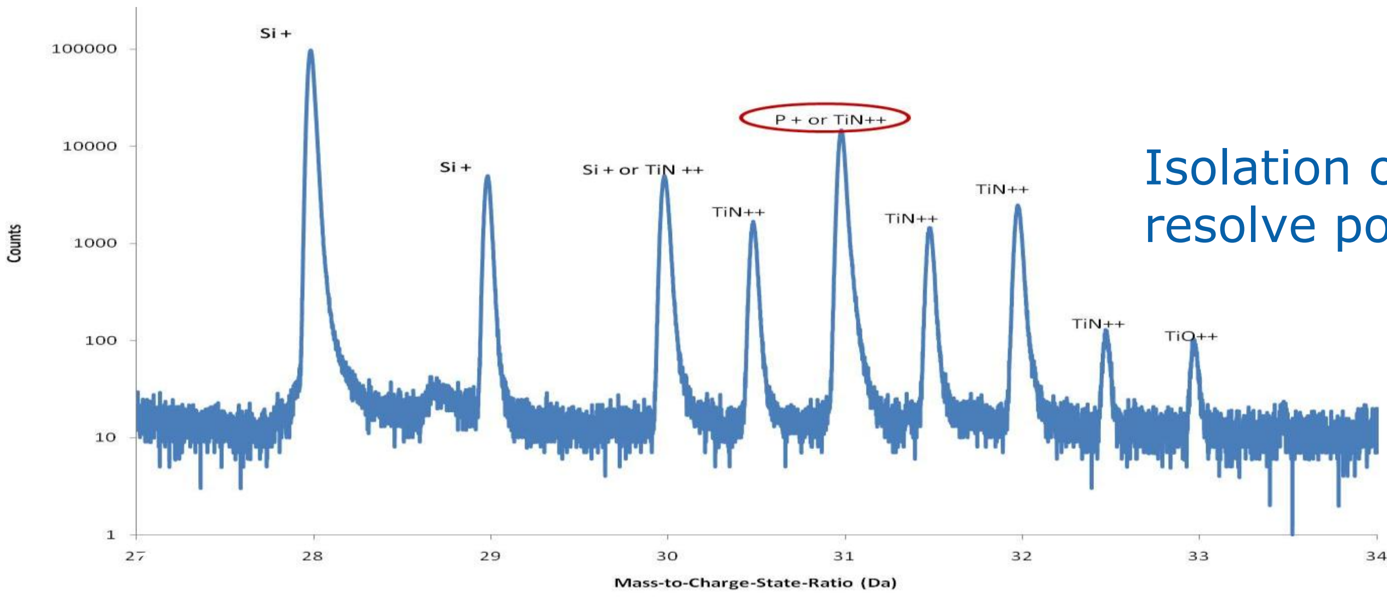
- Fracture failures increase for engineered nanostructures containing dissimilar materials
- As the specimen field evaporates through an interface, the dissimilar evaporation fields create a tip surface that is non-uniform
- Reconstruction software can be used to minimize aberrations originating from the evolving non-spherical tip shapes
- TEM images of the structures are needed to improve accuracy of the reconstructions
- Analyzing devices with different orientation within the atom probe tip can improve the reconstruction

Mass Spectrum Analysis



Peak overlaps are an additional challenge in the reconstruction of heterogeneous nanostructures

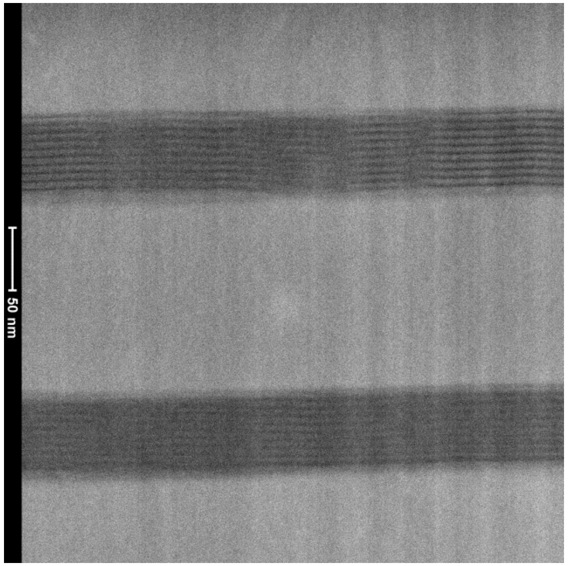


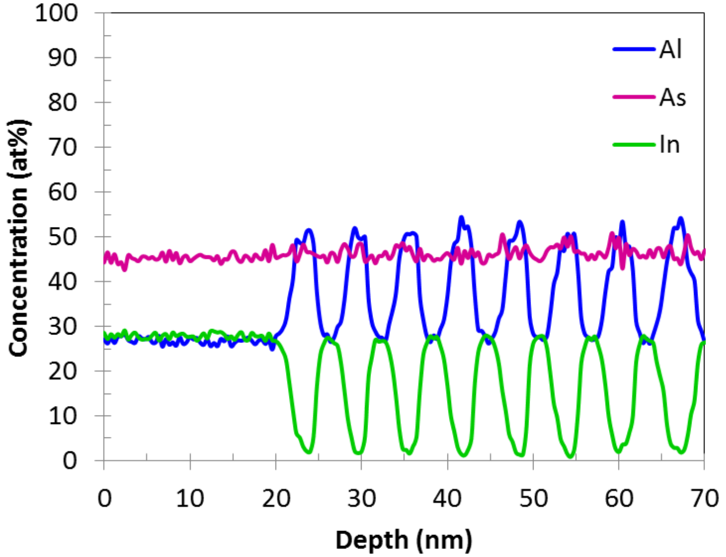


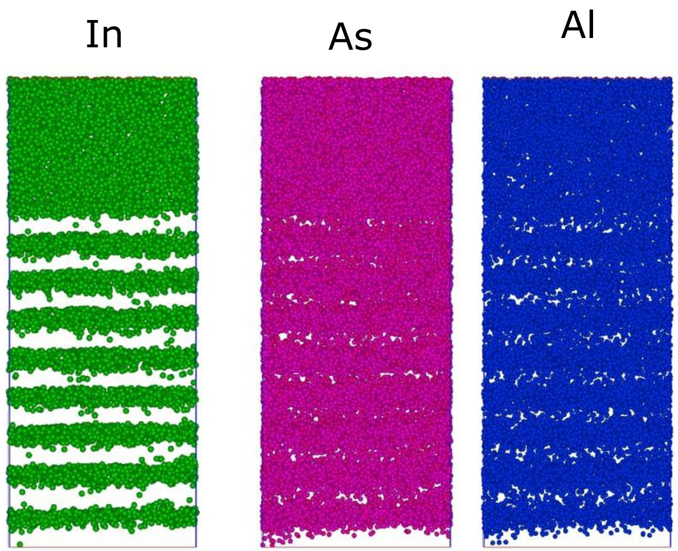
Isolation of the ROI can help resolve potential overlaps

III-V Multi-layers

- Compositional analysis of III-V materials may be necessary for future technologies
- STEM imaging reveals multilayer (ML) structures in 2 regions
- APT results reveal alternating Al-rich and In-rich layers







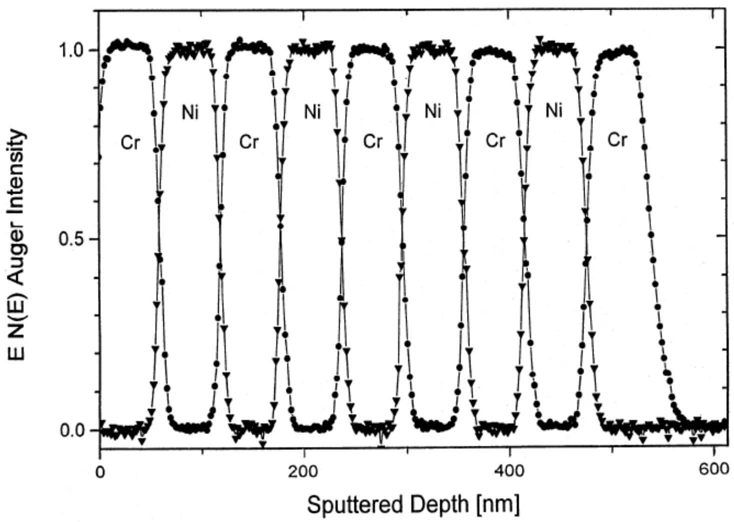
$30 \times 30 \times 70\ nm^3$

SRM 2135c - Ni/Cr Thin Film Depth Profile

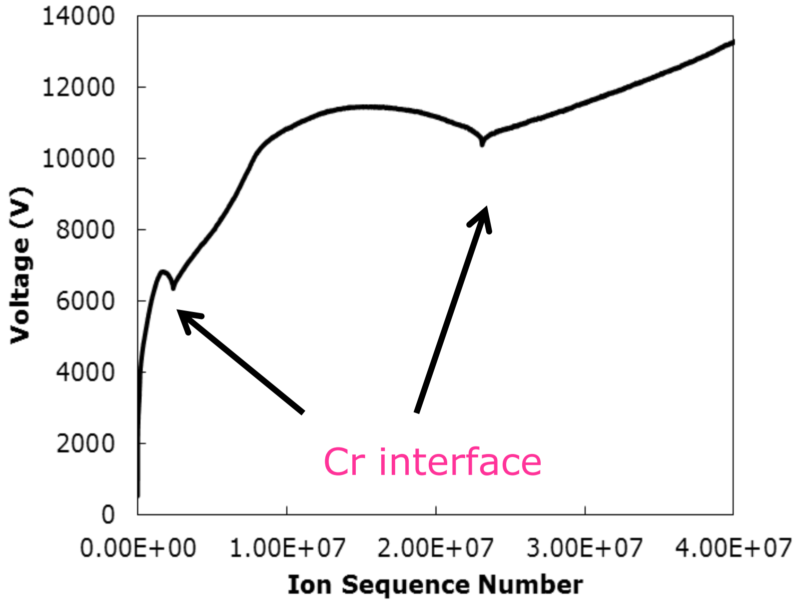
- Voltage curves illustrate the evaporation field differences in Ni/Cr layers

  Theoretical evaporation field values
  - Cr ~ 27 (V/nm)
  - Ni ~ 35 (V/nm)

- Magnification can vary over the surface of the specimen

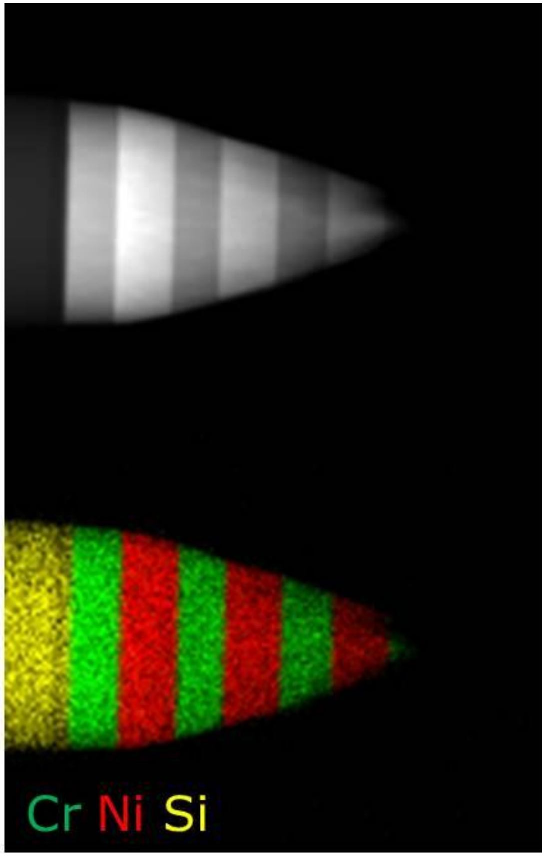


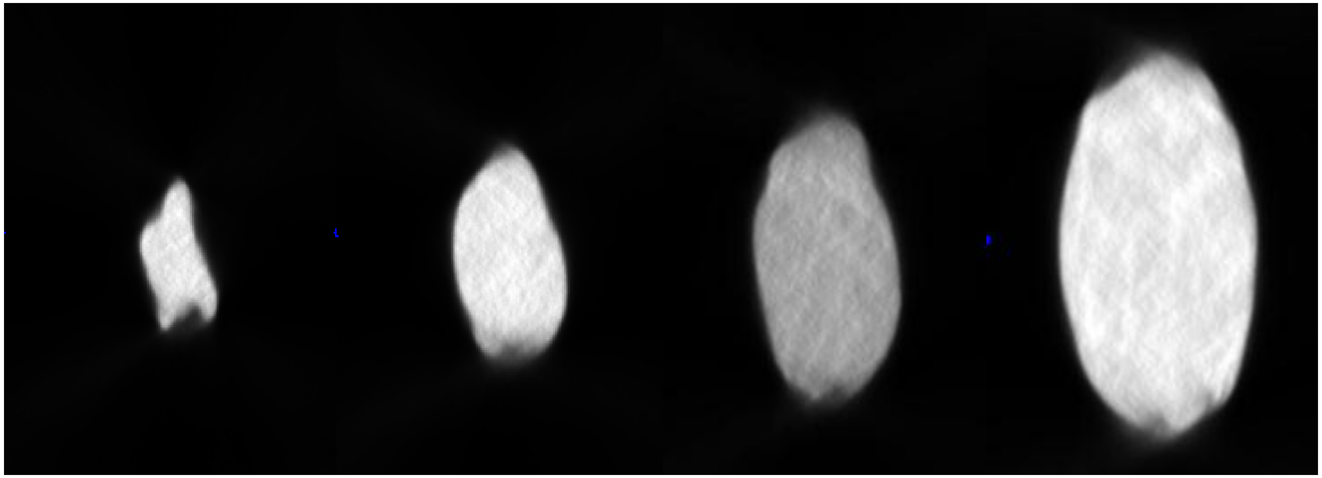
Taken from the SRM 2135c Certificate



TEM Tomography

- TEM tomograms of Ni/Cr samples indicate a non-spherical tip shape
- Differing ion sputter yields of Ni and Cr result in ledges at the Ni/Cr interfaces during sample preparation
- Tomograms provide additional information for the APT reconstruction process

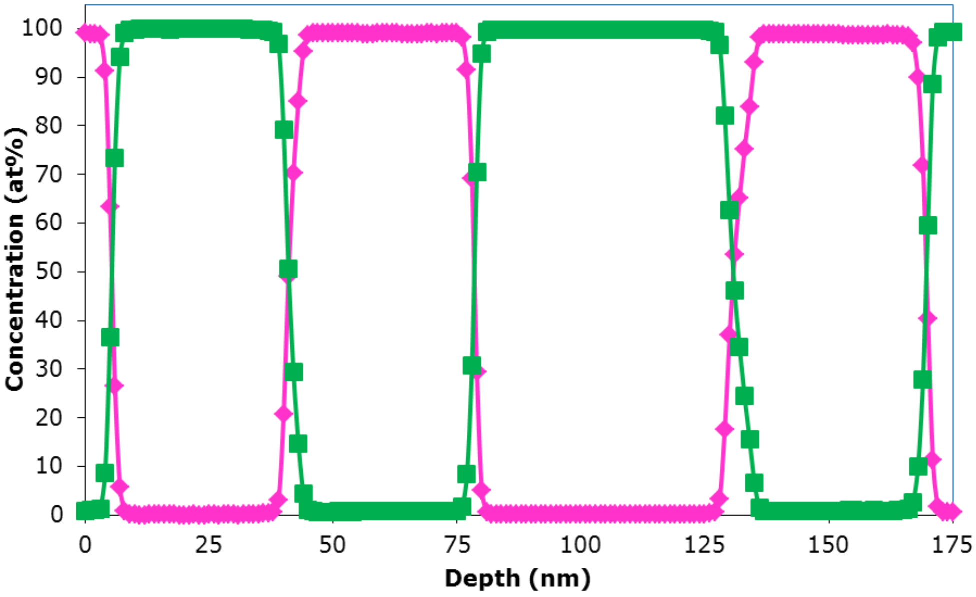


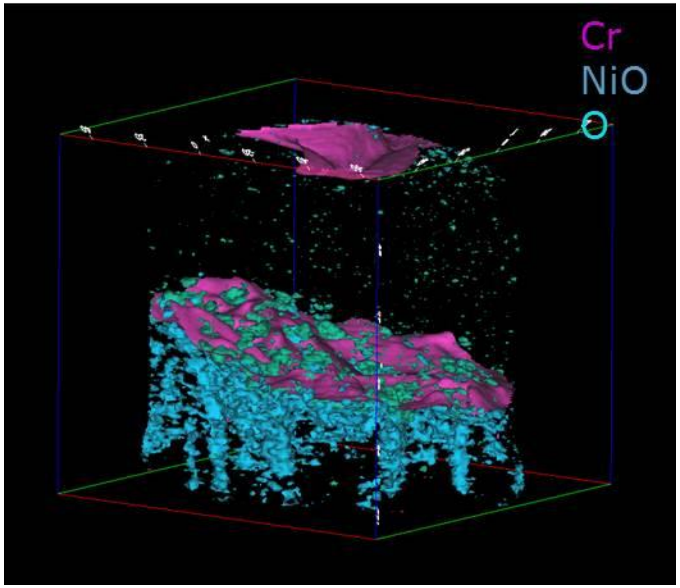


TEM imaging provided by A. Herzing, NIST

SRM 2135c Ni/Cr Results

- APT analysis revealed oxygen in the Cr layers and at the Ni/Cr interfaces
  - Oxygen not observed in TEM EDS spectrum analysis
- Individual mass spectrum analysis can be performed on the oxide nodules isolated by iso-concentration surfaces





Looking Forward

As engineered nanostructures continue to shrink, the ability to measure the position and composition of individual atoms becomes more critical

- APT is one of the few methods available to image and analyze solid materials at the atomic scale in 3D

- For continued advancements, the development of APT methods to improve yield and reconstructions of manufactured nanostructures are necessary

- Complexity of 3D structures poses a significant challenge for the APT technique
    - Metals
    - Semiconductors
    - Di-electrics

- Materials with grossly dissimilar properties lead to a number of challenges
    - Sample preparation
    - Data acquisition
    - 3D volume rendering

Standards and Reference Materials

- Reference materials for instrumentation evaluations
  - Mass resolving power
  - Spatial resolution
  - Field of View
- Standard methodologies for reconstruction
  - Mass spectrum analysis
  - Reconstruction parameter selection
- Standard datasets to evaluate new reconstruction algorithms

- Simple standard reference materials used to evaluate reconstruction methodologies
  - Dopant profile – As SRM
  - Metallic thin film structure – Ni/Cr thin films

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- Collaborators at Cameca Instruments, Inc.