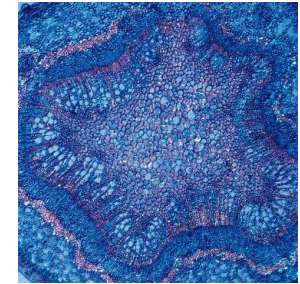
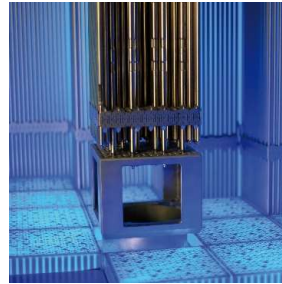
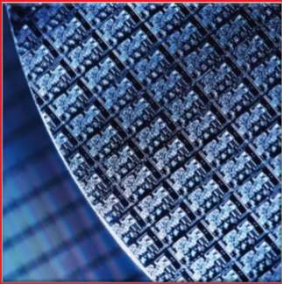


Atom Probe Tomography Anna Bui

Dec 3 2019

Introduction to Atom Probe Tomography (APT)

- ▀ Introduction to AMETEK and CAMECA Worldwide
- ▀ A Review of Atom Probe Tomography Technology
- ▀ Sample preparation
- ▀ 2 APT versions: LEAP5000 and EIKOS UV
- ▀ Cryo transfer: why and how
- ▀ Discussion Q&A



Brief Introduction to CAMECA/AMETEK



Annual sales of approximately \$5B,
15,000 employees in 30 countries

Electromechanical Group

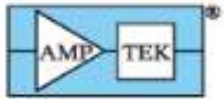
Electronic Instruments Group

Our newest addition - 2019

AMETEK[®]
MATERIALS ANALYSIS DIVISION

SIMS
EPMA

Advanced Measurement Technology



VISION
RESEARCH

SPECTRO

EDAX

CAMECA Instruments, Inc.
US Headquarters, Madison, WI

Atom Probe Tomography (APT)

World Leader in Elemental & Isotopic Microanalysis

SXFiveTACTIS



**IMS 7f-Auto |
7f-GEO**



**IMS 1300-HR³ |
KLEORA**



NanoSIMS 50L



LEAP[®] 5000



EX-300



IMS Wf | SC Ultra



AKONIS



SIMS 4550



EIKOS[™] UV



Electron Probe
Microanalyzers
(EPMA)
&
LEXES*
metrology tools

Secondary Ion Mass Spectrometers (SIMS)

3D Atom
Probes
(APT)

World Leader in Elemental & Isotopic Microanalysis

SXFiveTACTIS



**IMS 7f-Auto |
7f-GEO**



**IMS 1300-HR³ |
KLEORA**



NanoSIMS 50L



LEAP[®] 5000



EX-300



IMS Wf | SC Ultra



AKONIS



SIMS 4550



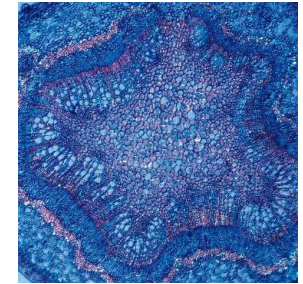
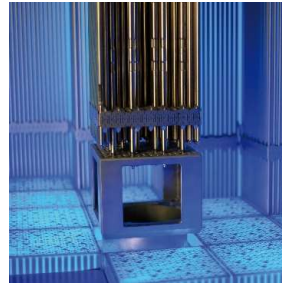
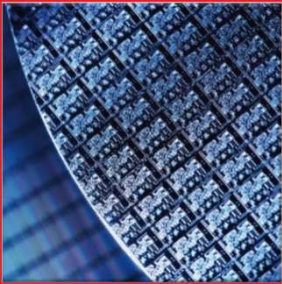
EIKOS[™] UV



Electron Probe
Microanalyzers
(EPMA)
&
LEXES*
metrology tools

Secondary Ion Mass Spectrometers (SIMS)

3D Atom
Probes
(APT)



Atom Probe Tomography Technology Introduction – 2019

Atom Probe Tomography – In 90 Seconds!



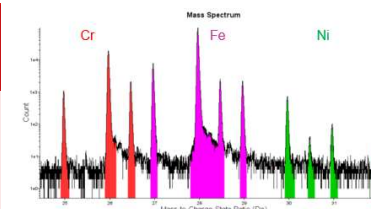
The Strengths of Atom Probe Tomography (APT)

3D Spatial Resolution	0.4 nm Laterally, ~ 0.2 nm Depth (single lattice plane)
Sensitivity	Better Than 5 appm with up to 80% of Atoms Identified

Compositional Analysis

Time-Of-Flight Mass Spectrum

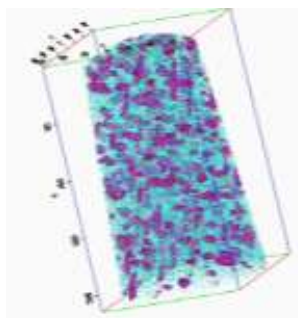
- Identify elements and their location in the specimen
- Phase and composition identification
- Statistical methods can be used for detection of fine-scale ordering, phase decomposition etc.



Compositional Imaging

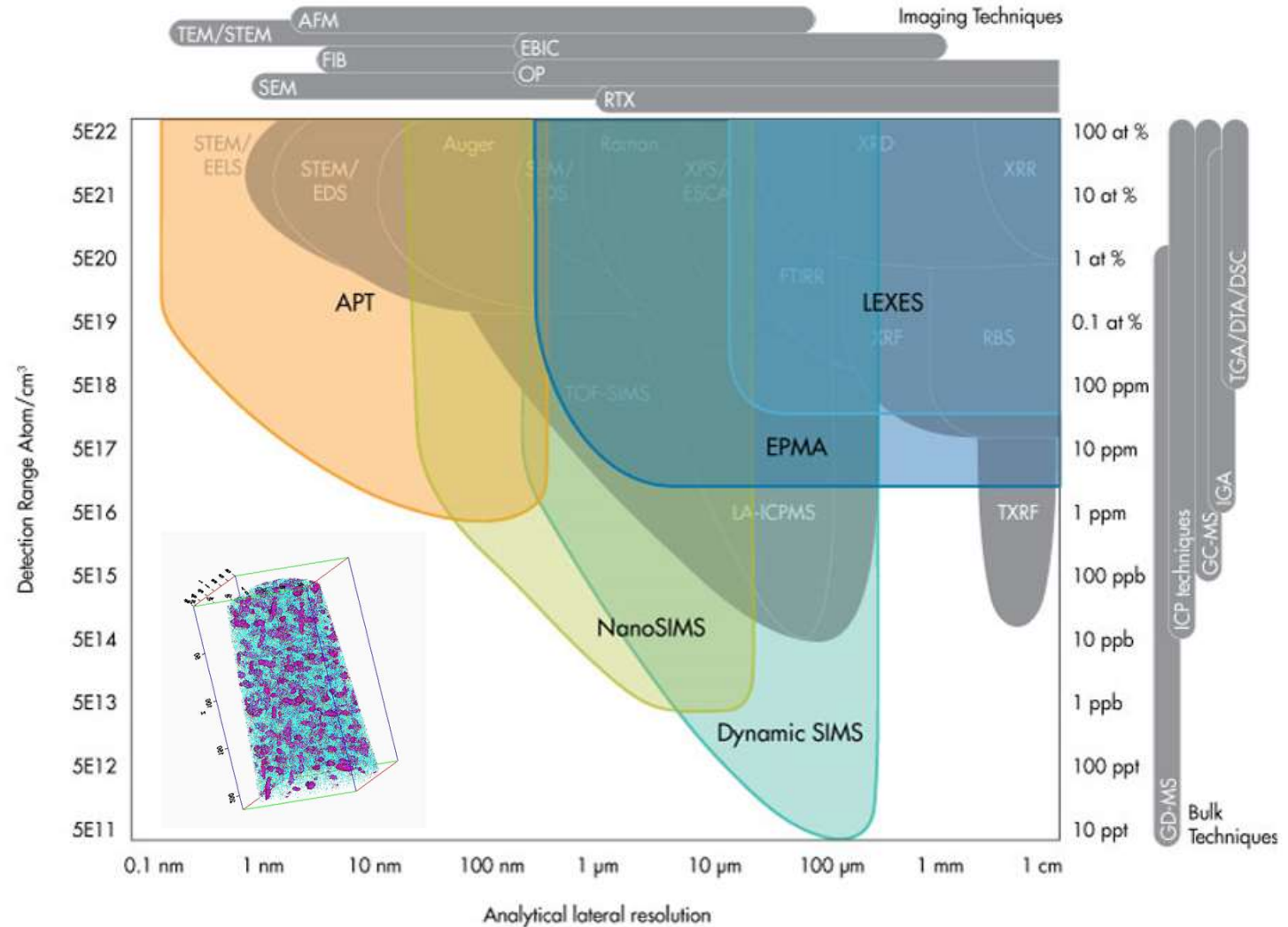
3D Image – Nanoscale Point Cloud

- Buried interfaces, phase boundaries, grain boundaries, dislocations and defects
- **Structural Imaging**
 - Number densities, feature morphology/ roughness, size distributions etc



CAMECA Products Analytical Resolution

- CAMECA's core technologies now span the entire achievable analytical capabilities
- SIMS/NanoSIMS
- EPMA/LEXES
- APT
 - Subnanometer 3D
 - Isotopic/Elemental information
 - Highest sensitivity analytical technique
 - TOF – sees all
 - Equally sensitive to all



Erwin Wilhelm Müller

- 1911: Born June 13.
- 1936: Degree with Gustav Hertz.
- 1938: Invented the FEEM.
- 1941: Discovered field-desorption.
- 1951: Invented the FIM.
- 1952: Joined Penn State Faculty.
- 1956: First observation of atoms.
- 1966: Developed the Atom-Probe.
- 1975: Elected, National Academies of Science and Engineering.
- 1977: Received the National Medal of Science.
- 1977: Considered for the Nobel Prize in Physics.
- 1977: Died May 17.



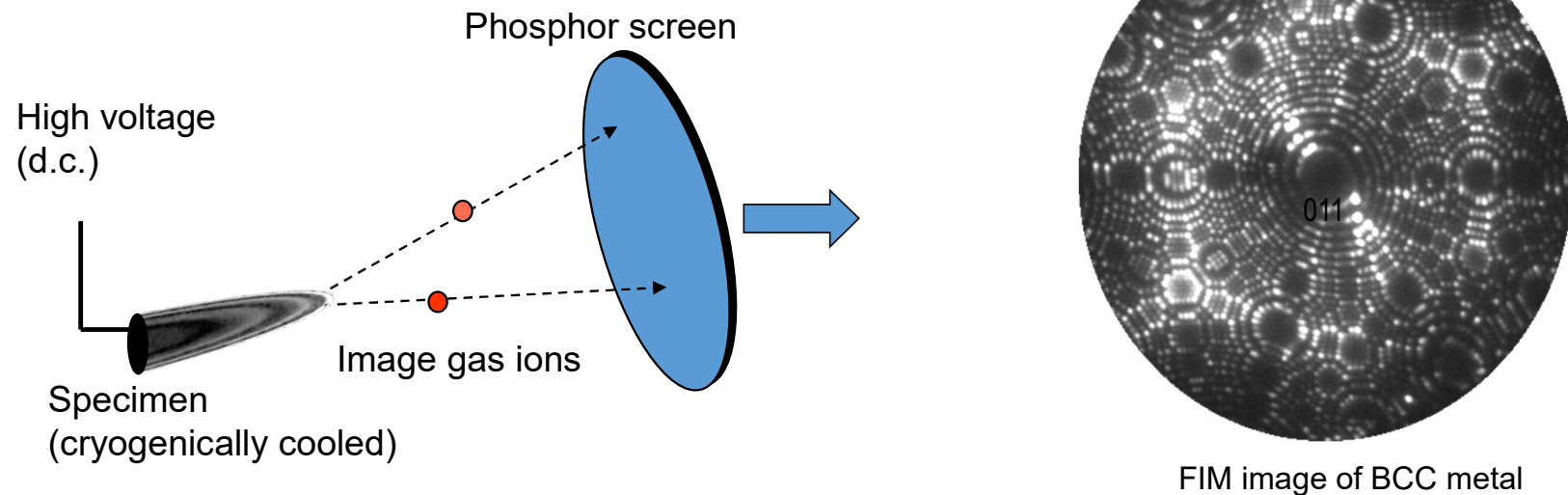
Professor Erwin W. Müller (1911-1977):
Father of High Field Nanoscience

Holding a hand-made glass UVH FIM system that would be immersed in liquid nitrogen

‘Ball Model’ of a cubic lattice Cut by a hemisphere – showing ‘poles’

APT is based on the Field-ion Effect

- *Field-ion Effect first observed in the Field Ion Microscope (FIM)*
- *At high applied fields ($\sim V/nm$) surface ion-removal occurs.*
- *Needle-shaped specimens allow high fields to be achieved at reasonable ($<20kV$) applied voltages*



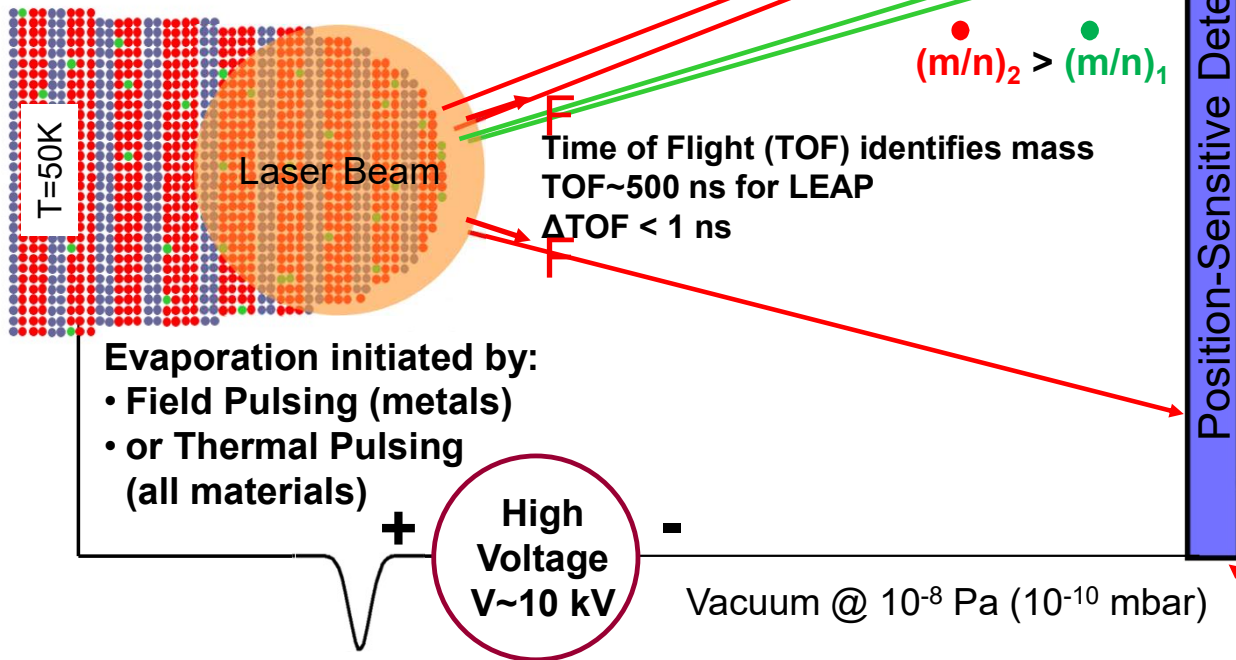
- FIM was the first technique to achieve imaging at atomic resolution (1956)!

Description of Atom-Probe Operation

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

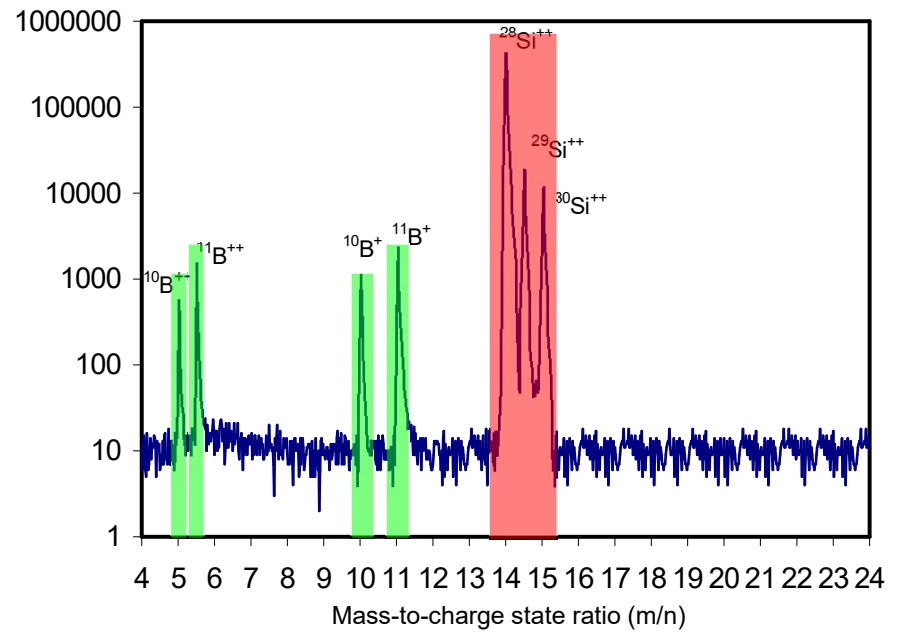


~80nm tip → 80mm detector = 10^6 magnification



Time of Flight (TOF) identifies mass
TOF~500 ns for LEAP
 Δ TOF < 1 ns

$(m/n)_2 > (m/n)_1$

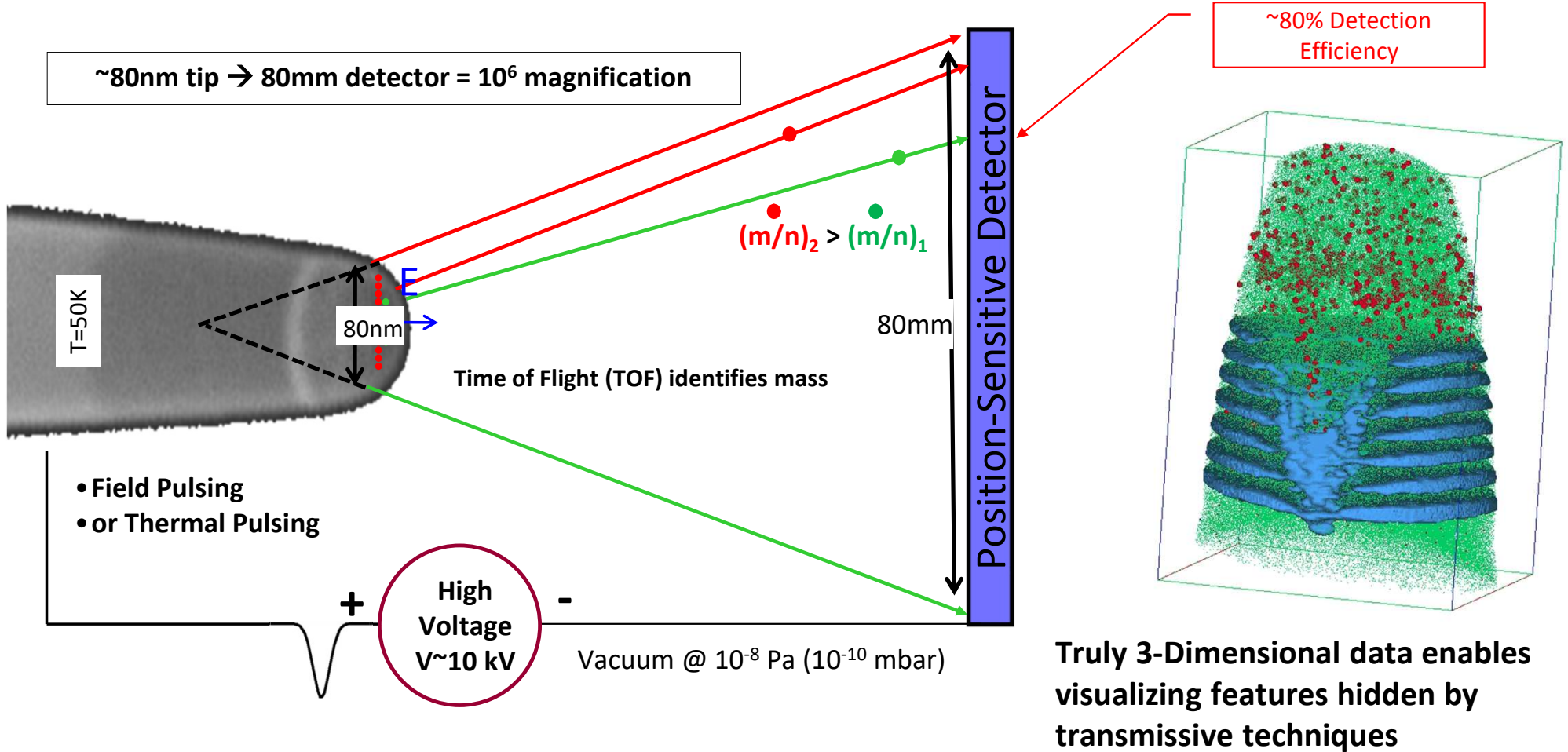


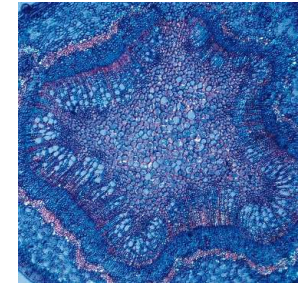
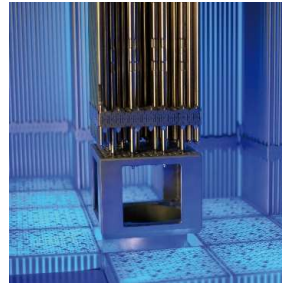
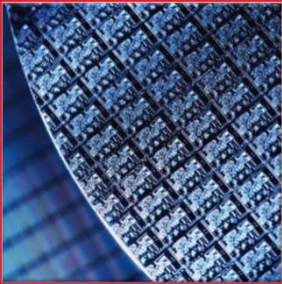
~80% Detection Efficiency

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

Description of Atom Probe Operation

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

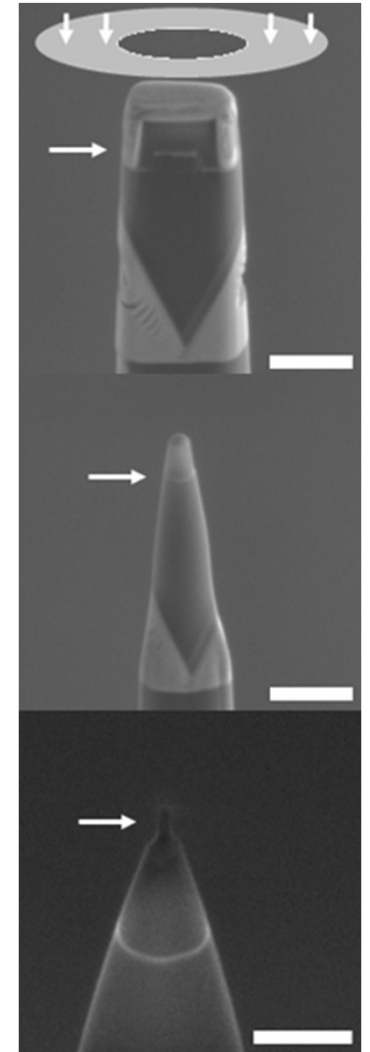
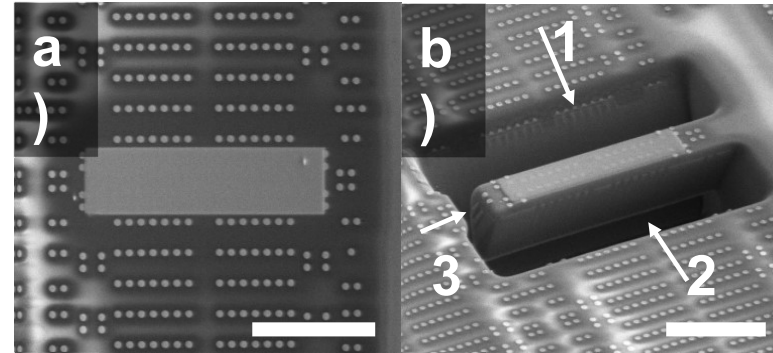




Atom Probe Tomography Sample Preparation– 2019

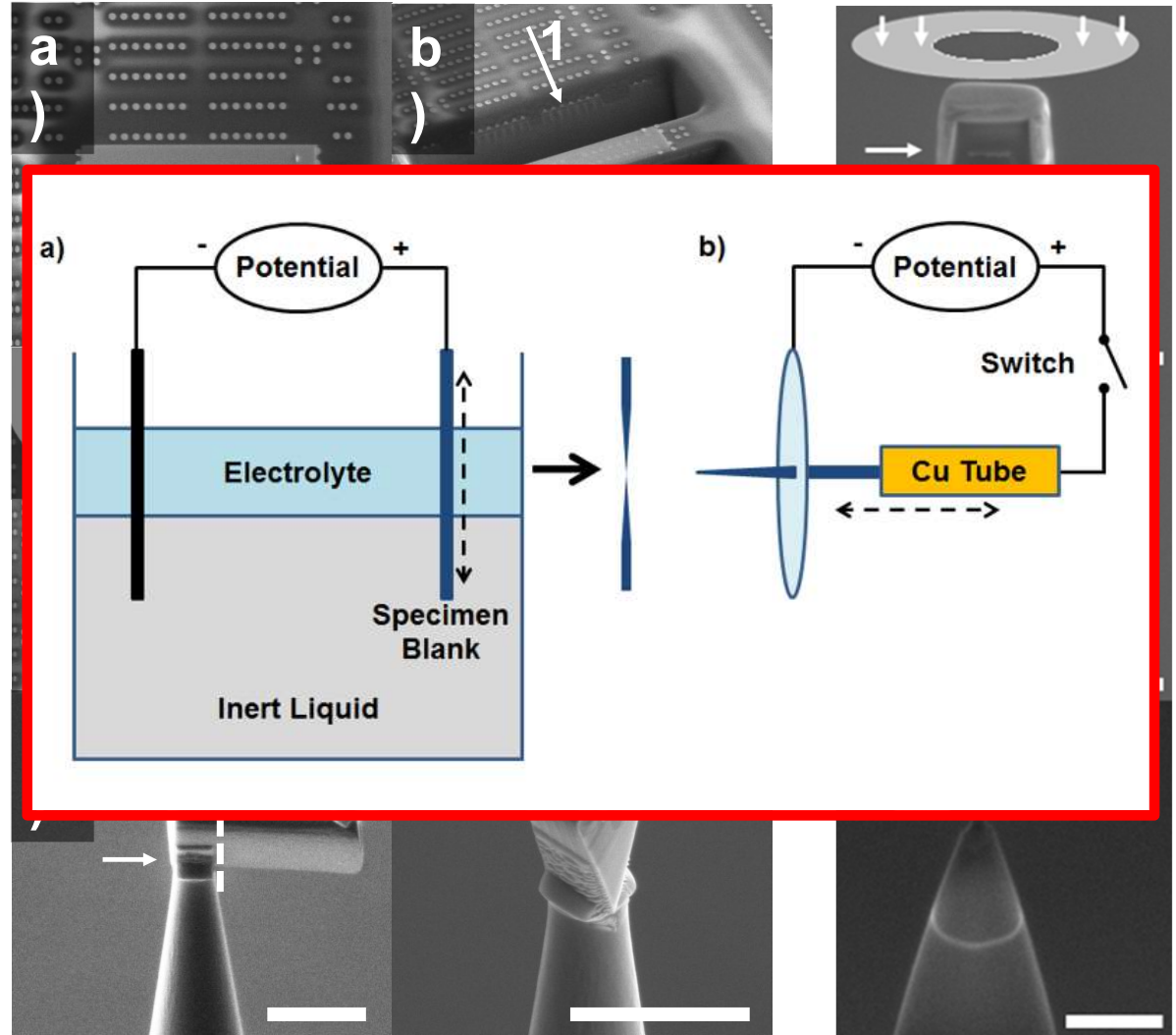
Specimen Preparation: General FIB Liftout Method

- a) Protective strip (Pt) is deposited over the region of interest (ROI)
- b) Material is removed around three sides of the region (arrows) as well as underneath to produce a long wedge of material containing the ROI
- c) The wedge is removed by first attaching a micromanipulator to one end of the wedge (left arrow) and then cutting the wedge free from the substrate (right arrow)
- d) The wedge is transferred to a carrier microtip (black dashed circle indicates location of flat 2 μm tip) and attached with FIB-deposited Pt
- e) Wedge is cut free of the of the carrier tip (dashed line) for transfer to additional tips
- f) The final mounted wedge-section is shown with FIB-deposited Pt at each wedge-tip interface.
- g) Then the sharpening process with an annular mill pattern

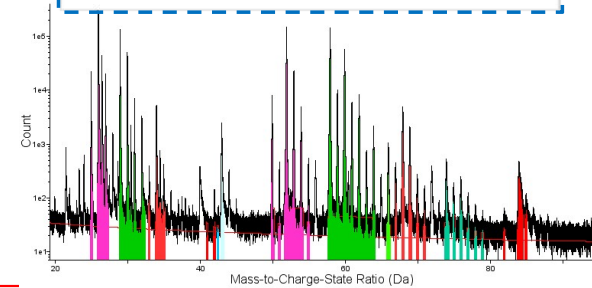
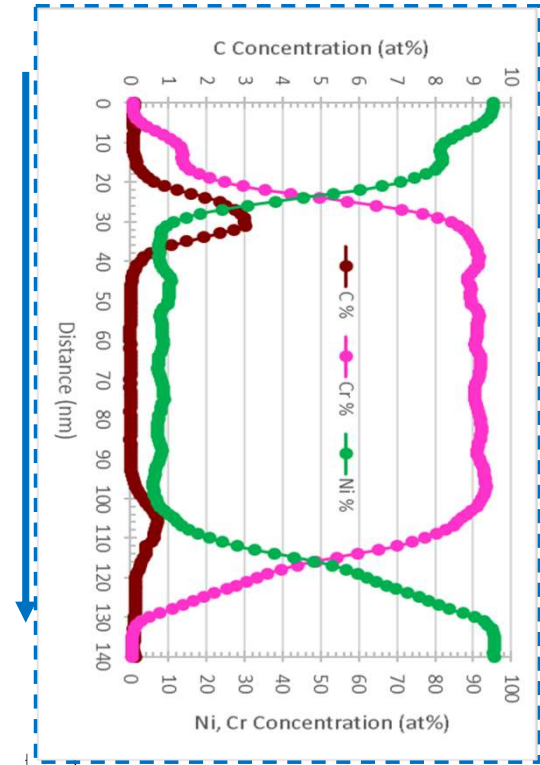
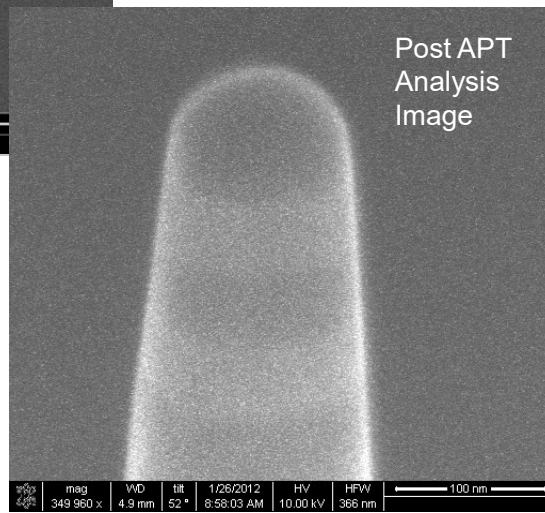
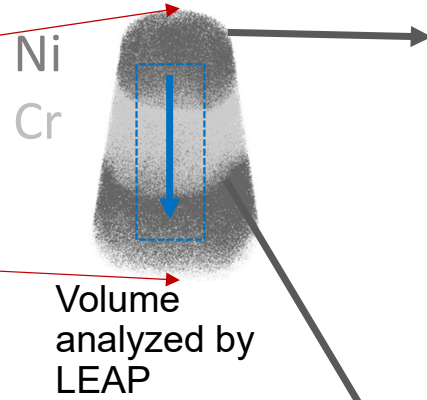
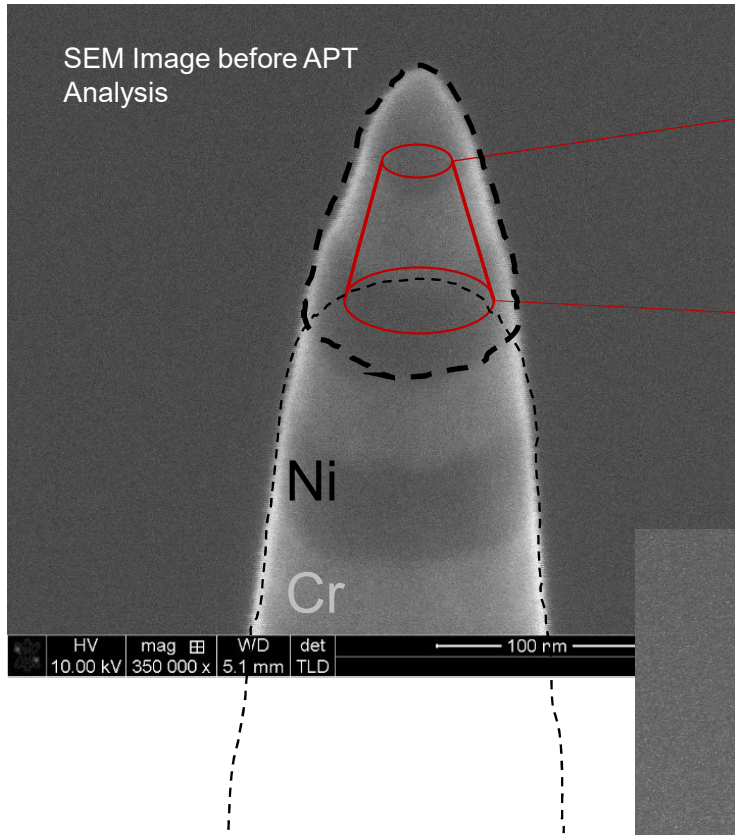


Focused Ion Beam: Liftout Method

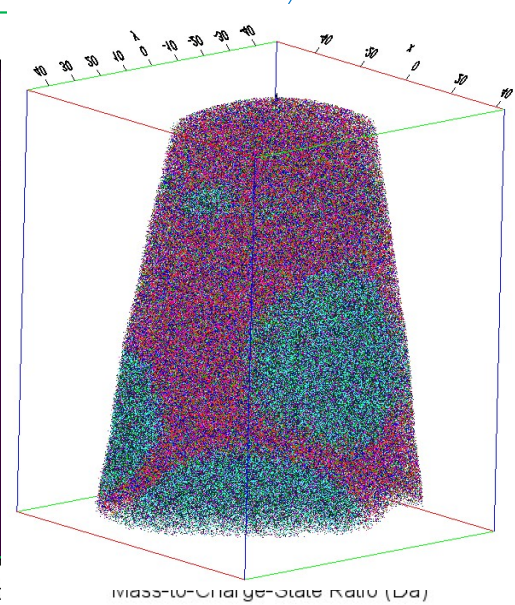
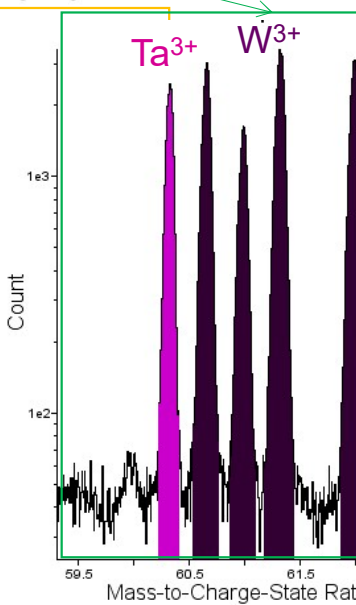
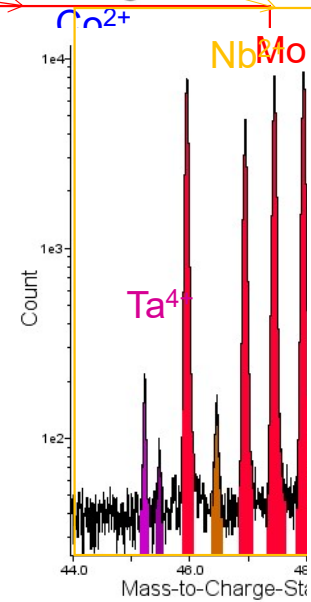
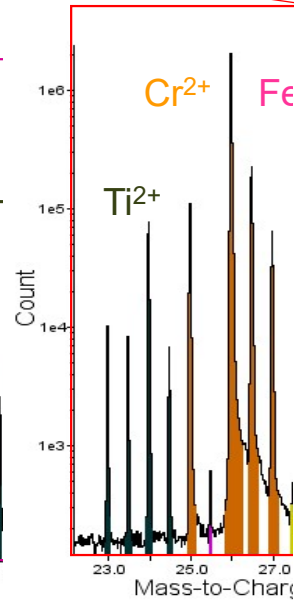
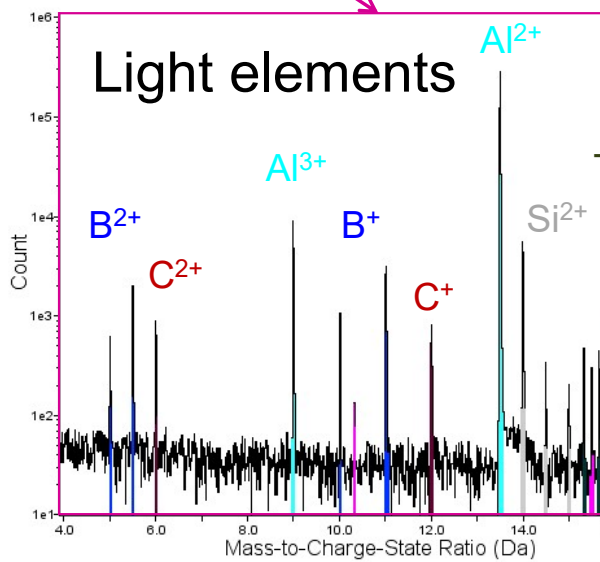
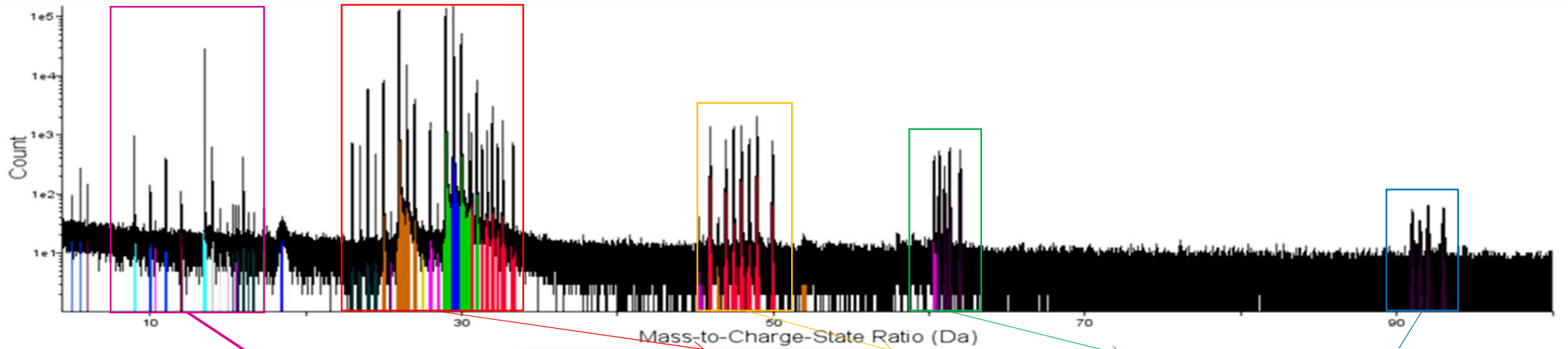
- a) Protective strip (Pt) is deposited over the region of interest (ROI)
- b) Material is removed around three sides of the region (arrows) as well as underneath to produce a long wedge of material containing the ROI
- c) The wedge is removed by first attaching a micromanipulator to one end of the wedge (left arrow) and then cutting the wedge free from the substrate (right arrow)
- d) The wedge is transferred to a carrier microtip (black dashed circle indicates location of flat 2 μm tip) and attached with FIB-deposited Pt
- e) Wedge is cut free of the of the carrier tip (dashed line) for transfer to additional tips
- f) The final mounted wedge-section is shown with FIB-deposited Pt at each wedge-tip interface.
- g) Then the sharpening process with an annular mill pattern
- h) **Also electropolishing for metals (cheaper and faster – not site specific)**



Thin Film Specimen Before and After APT



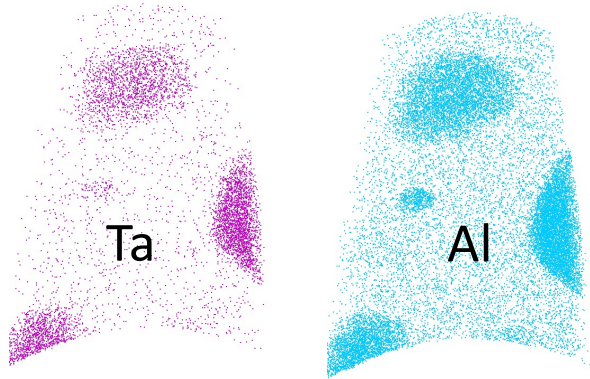
TOF Mass Spectrum (Ni-based superalloy)



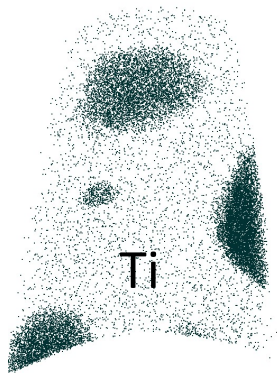
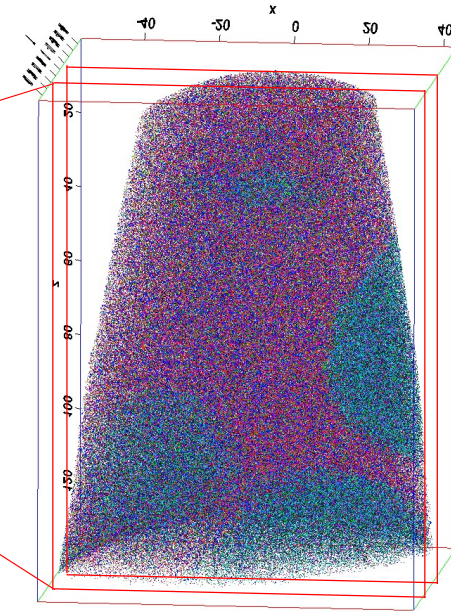
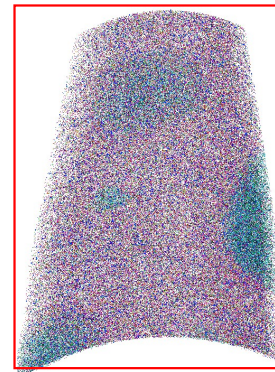
(100x100x250 nm) 20

Compositional Imaging

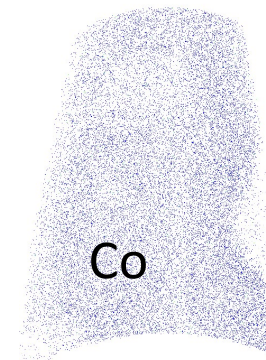
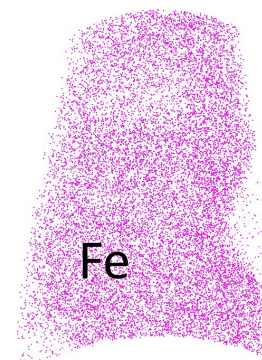
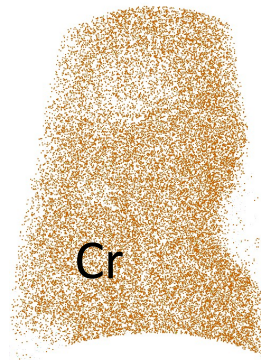
Al-Ti-Cr rich phase



20 nm slice



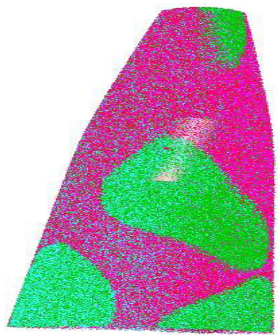
Co-Cr rich phase



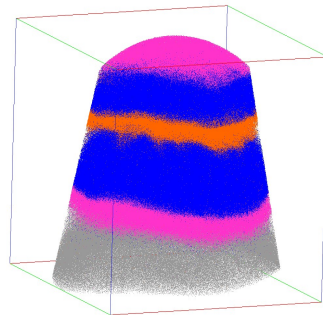
Virtual TEM lamella – with compositional information

APT – A Fast Growing Set of Applications

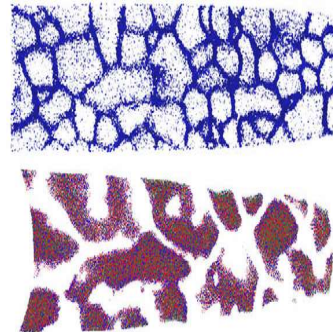
With the introduction of the Local Electrode Atom Probe (LEAP) in 2003, APT has been transformed from a useful but limited metallurgical technique to the standard material science method for the ultimate in 3D nanoscale compositional characterization.



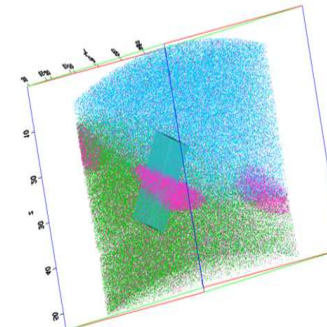
Advanced Alloys



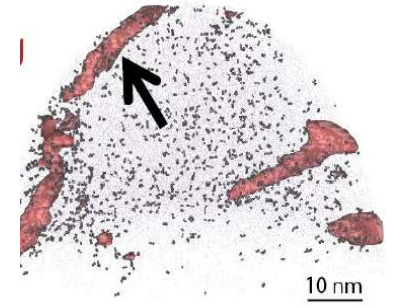
Thin Films



Silicides



Oxide growth



Biominerals

Worldwide Atom Probe Microscopes

APT Microscopes Today:

- Oxford Nano Science 3DAP (5)
- CAMECA – TAP Products (~10)
- Imago LEAP 3000 (~10)
- CAMECA LEAP 4000/5000 and EIKOS (~85)



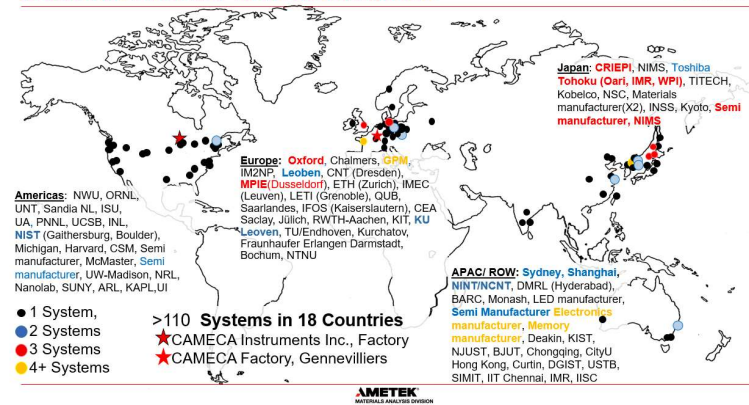
LEAP 5000



EIKOS

Over 110 Systems Worldwide

Worldwide Atom Probe Installations

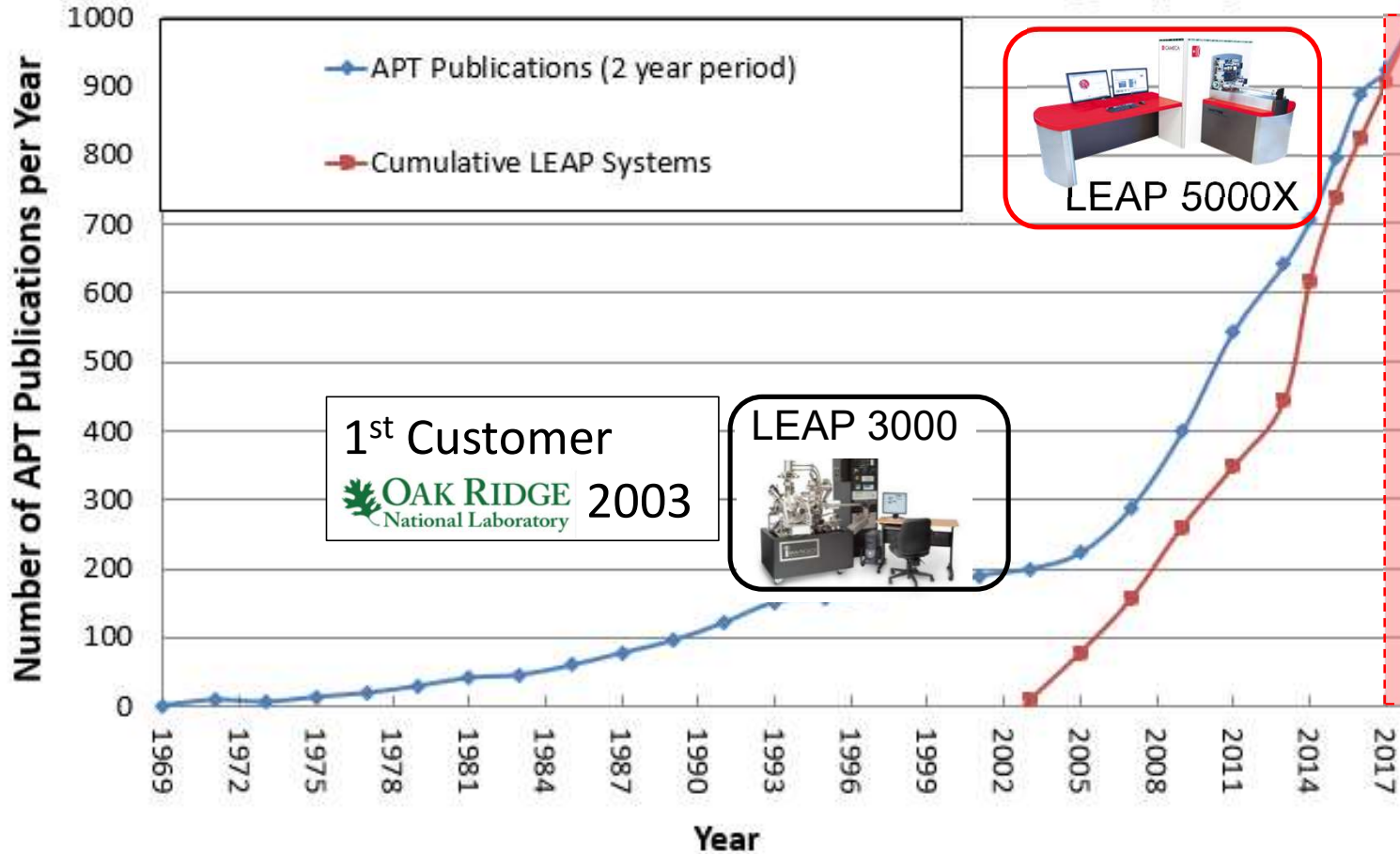


Leading Edge Atom Probers . . .



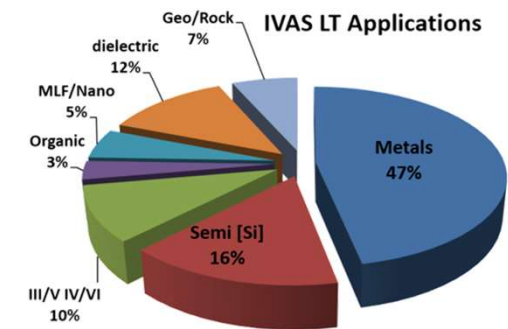
The Growth of Atom Probe Tomography

Growth of Atom Probe Tomography



Continued innovation in APT has lead to:

- Rapid growth of installed base
- Rapid increase in applications
- Rapid increase in publications in traditional and new applications
- Over 110 systems worldwide in 18 countries



LEAP 5000 Product Range

LEAP 5000 R

- ▀ Voltage mode instrument
- ▀ Ultimate performance for voltage-mode applications

R = Reflectron

S = Straight flight path

X = Laser mode option

LEAP 5000 XR

- ▀ Combines both voltage and laser mode for broadest range of applications
- ▀ Workhorse material science tool

LEAP 5000 XS

- ▀ Flagship laser mode instrument offering fastest throughput and highest detection efficiency
- ▀ Targeted for non-metal applications where sample throughput is critical

Introducing



A new APT instrument released in August 2019

New for 2019

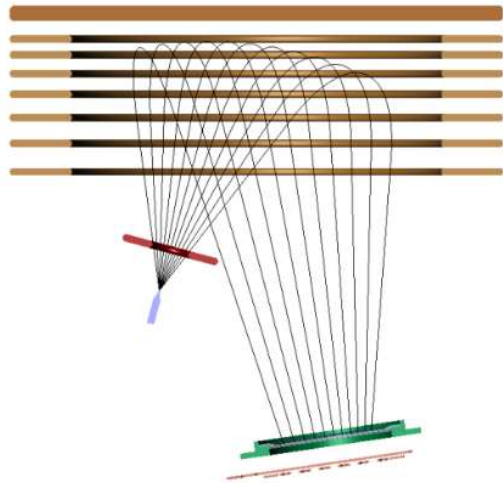
EIKOS-UV design delivers:

- All the benefits of the APT technique
 - *Single atom detection with outstanding efficiency*
 - *3D tomographic data with near-atomic spatial resolution*
 - *Equal sensitivity to all elements and isotopes*
 - *Quantitative composition measurements (from sub-nm to μm)*
- Wide range of applications – metals, coatings, thin films, ceramics, minerals and functional materials – enabled by UV laser
- In an easy-to-use and affordable package

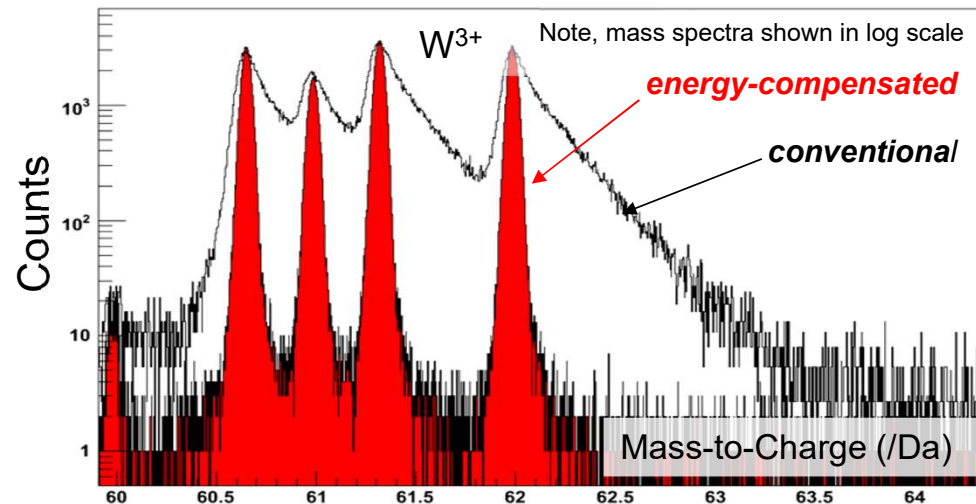


EIKOS Technology

- The EIKOS platform combines proven flight path and detector technologies
 - An energy-compensated (Reflectron) flight path provides excellent Mass Resolution for improved compositional accuracy
 - **In voltage mode** energy deficits are removed to produce narrow, Gaussian peaks
 - In **UV laser mode** the extended flight path length provides enhanced peak shapes



Energy-compensated reflectron ion flightpath



Example shown is comparison between **energy-compensated** and **conventional** for W^{3+} . The **reflectron** provides narrow Gaussian peaks shapes.

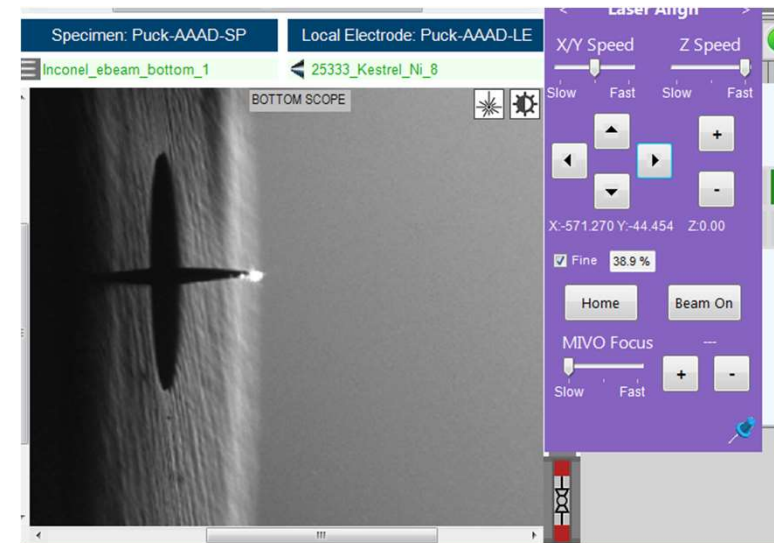
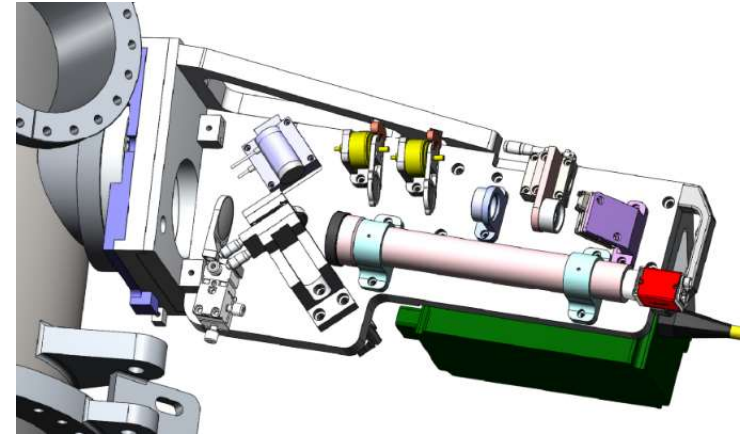
EIKOS-UV Technology – UV Laser Module

■ UV Laser Module Design

- *Focused spot, 355 nm (UV) design for proven performance with the widest range of applications*
 - Metals, coatings, & thin films
 - Ceramics, mineral and functional materials
- *Automated alignment protocols for ease of operation and consistent data quality*
- *Ultra-wide pulse energy dynamic range (1Mx) for optimized analysis of widest range of specimen materials*

■ Robust platform design

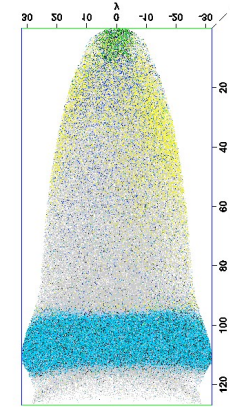
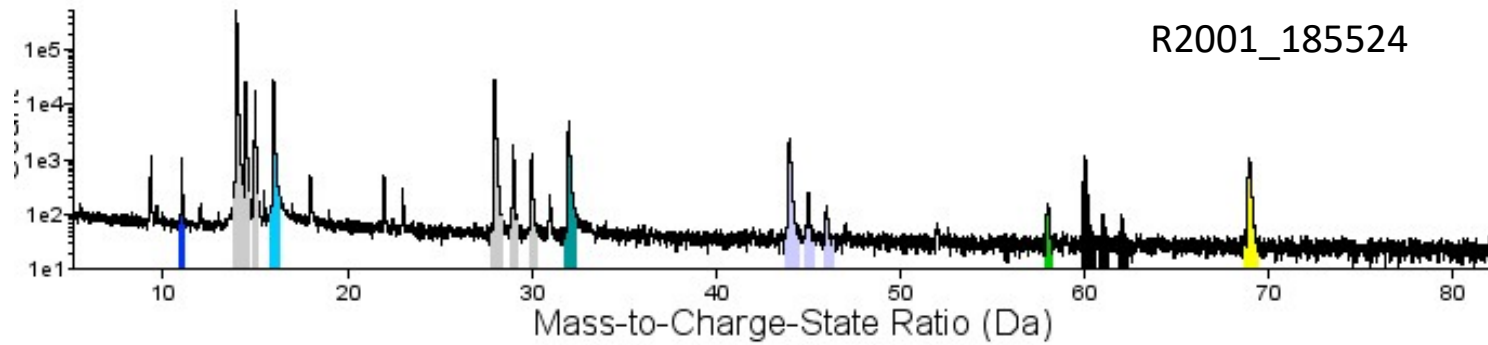
- *Highly industrialized laser provides excellent stability and reliability*
- *Elegant design for ease of use*



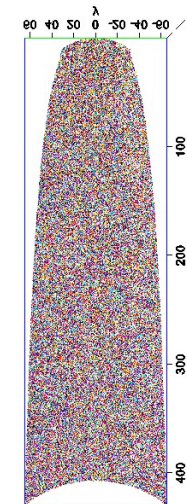
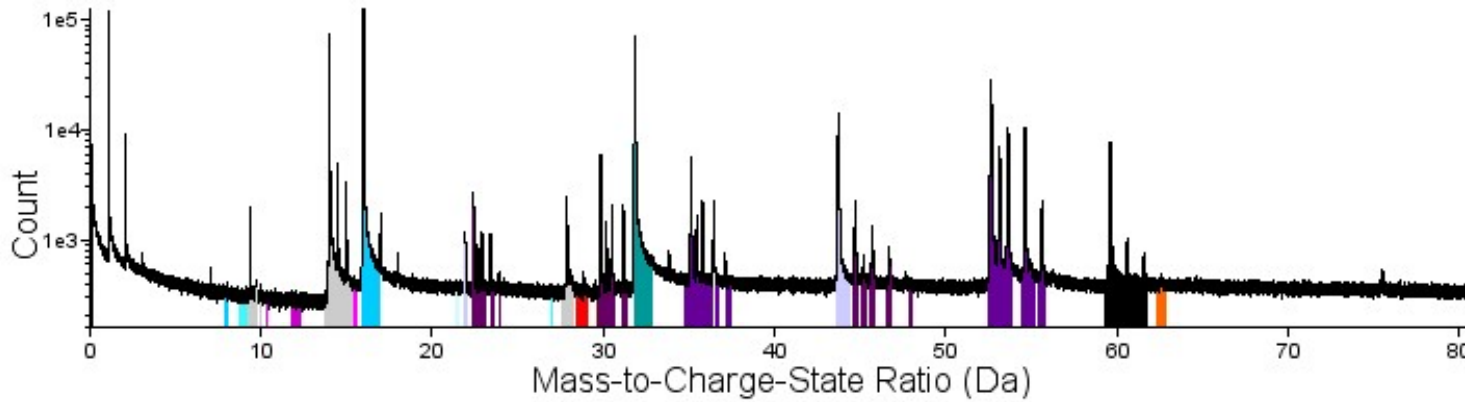
Examples of UV-enabled Applications

Buried Oxide 'Yield' Sample

R2001_185524



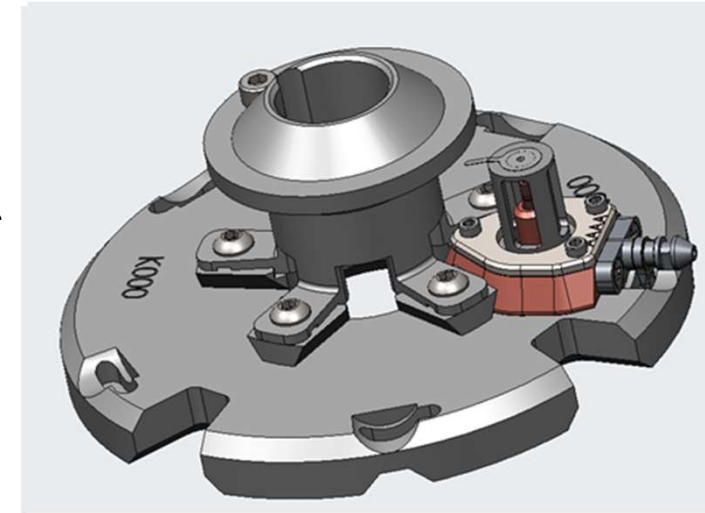
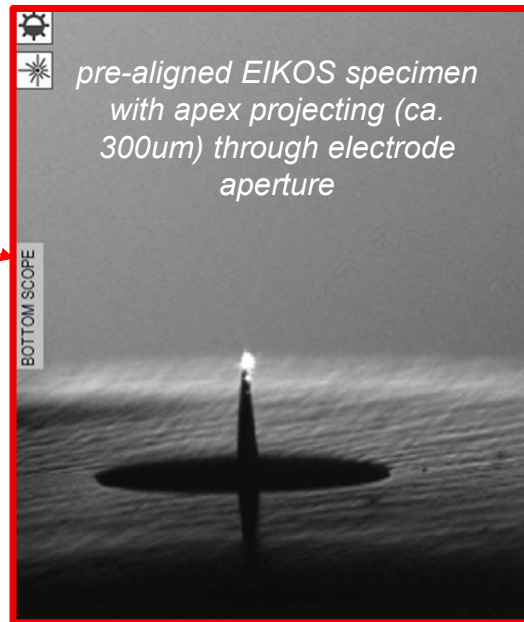
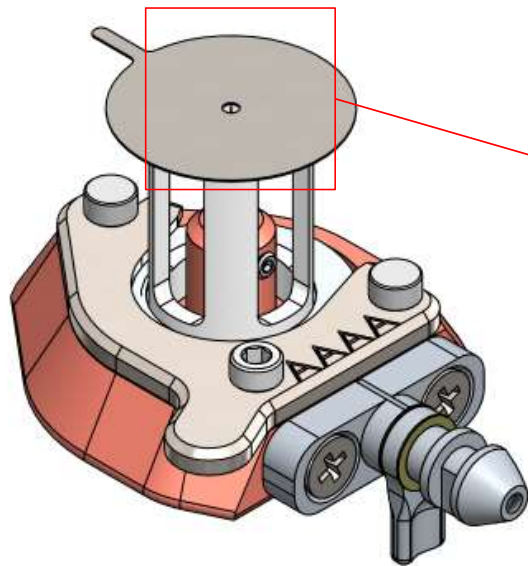
Mineral Sample



EIKOS Advanced Design Features

■ Integrated Counter Electrode (ICE)

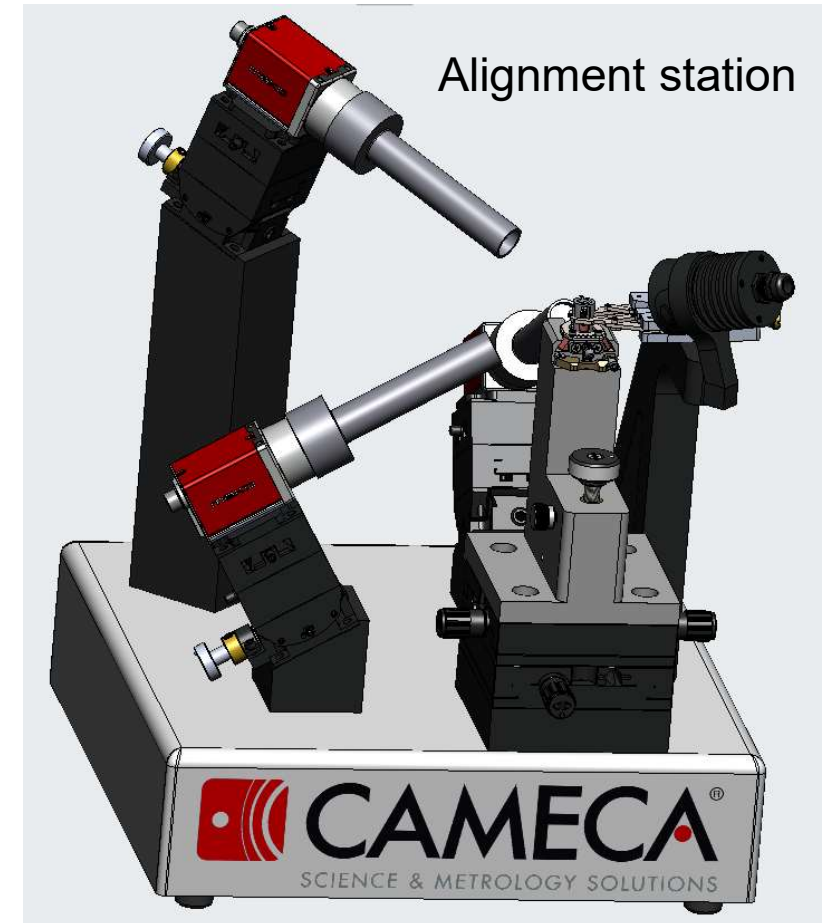
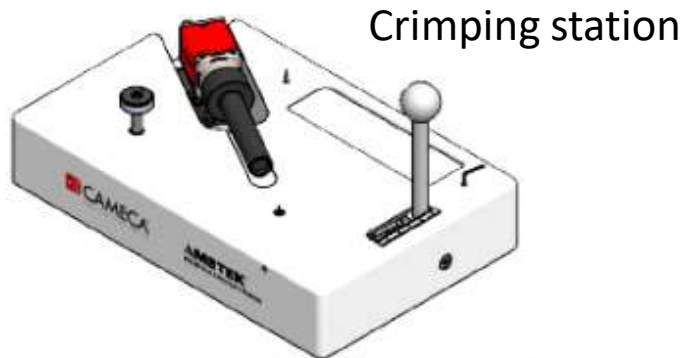
- *Pre-aligned electrode eliminates need for in-vacuum specimen alignment**
- *Configured for needle specimens and reduces/ eliminates common APT consumables*
- *Utilizes proven puck/ carousel design*

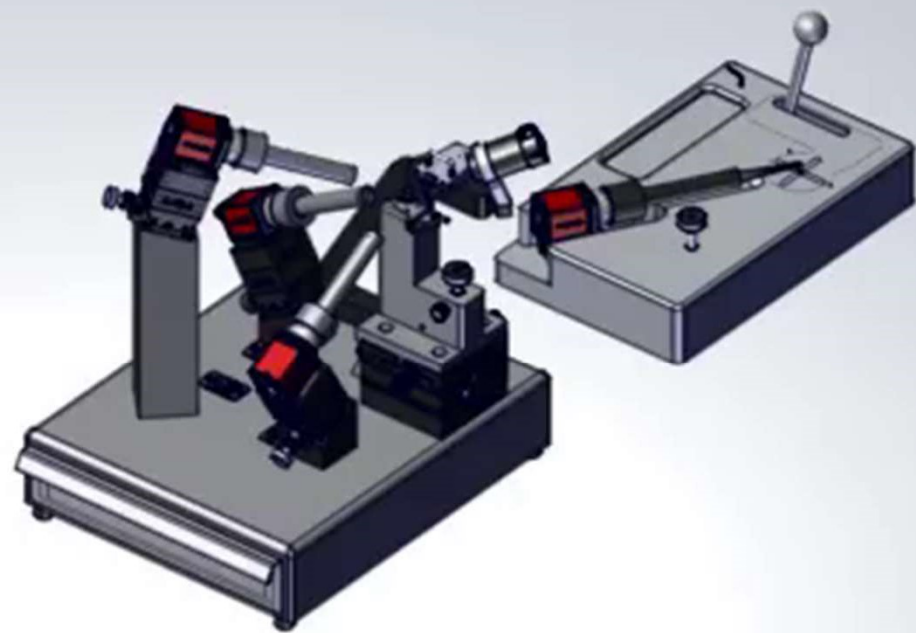


*Removing the need to align specimens within the instrument allows elimination of internal moving parts. This enables the simplicity and efficiency of the EIKOS-UV design.

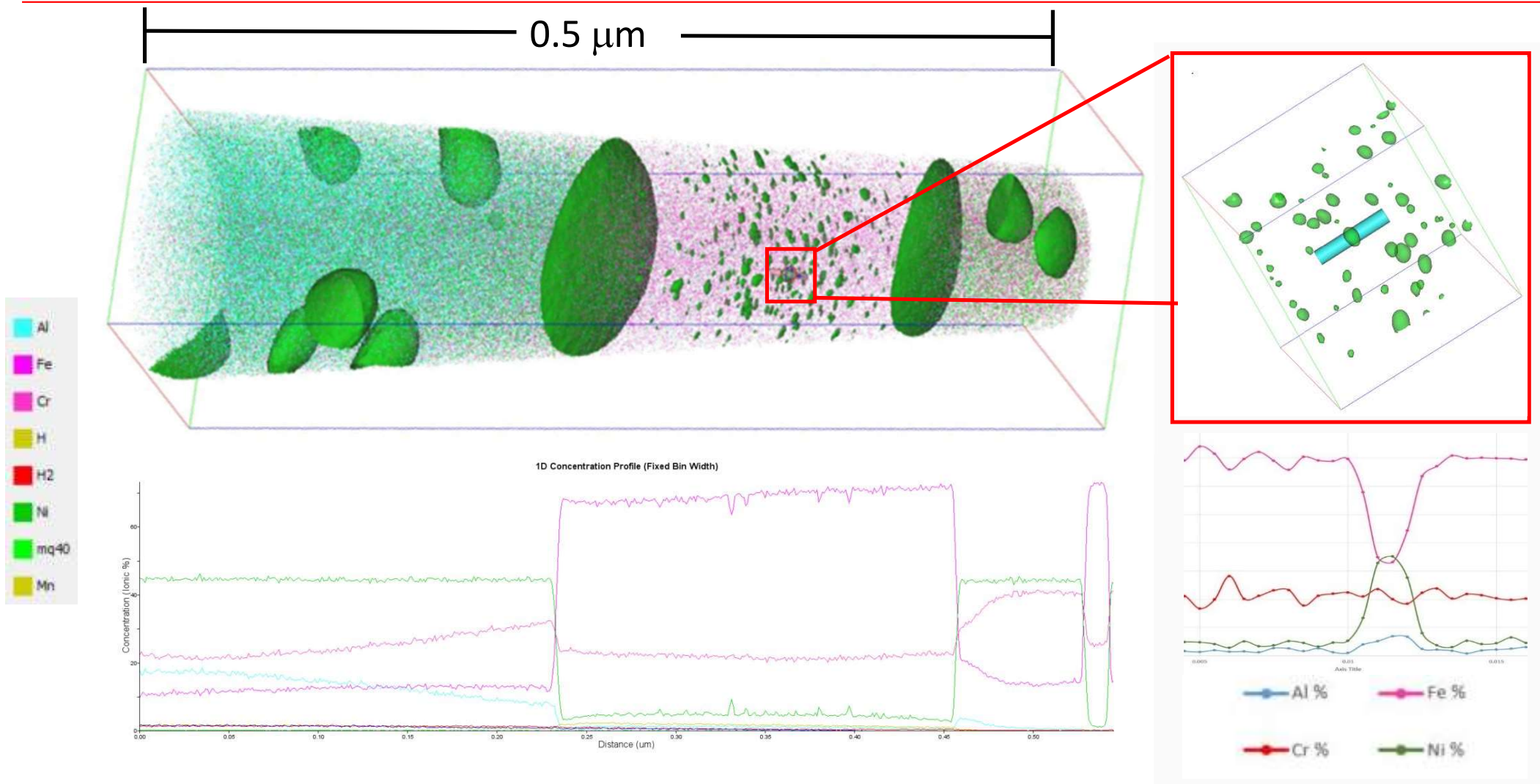
The ICE Ex-situ Specimen Alignment Procedure

- Hardware is provided to allow easy installation of the ICE during specimen mounting
 - Crimping station*
 - Alignment station*
- Crimping station eliminates misaligned wires/needles
- Alignment station includes integrated computer/video system for ILE mounting and alignment
 - Simply and easy to use*
 - Integrated into the specimen database*





EIKOS Data: Iron Based Super alloy

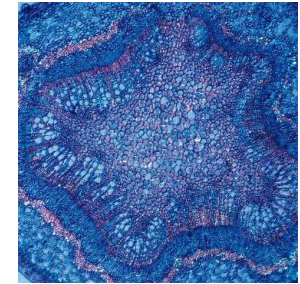
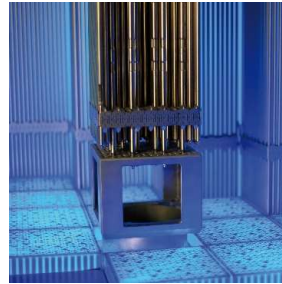
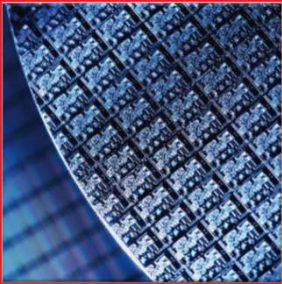


Summary

EIKOS-UV design delivers:

- All the benefits of the APT technique
 - *Single atom detection with outstanding efficiency*
 - *3D tomographic data with near-atomic spatial resolution*
 - *Equal sensitivity to all elements and isotopes*
 - *Quantitative composition measurements (from sub-nm to μm)*
- Wide range of applications – metals, coatings, thin films, ceramics, minerals and functional materials – enabled by UV laser
- In an easy-to-use and affordable package

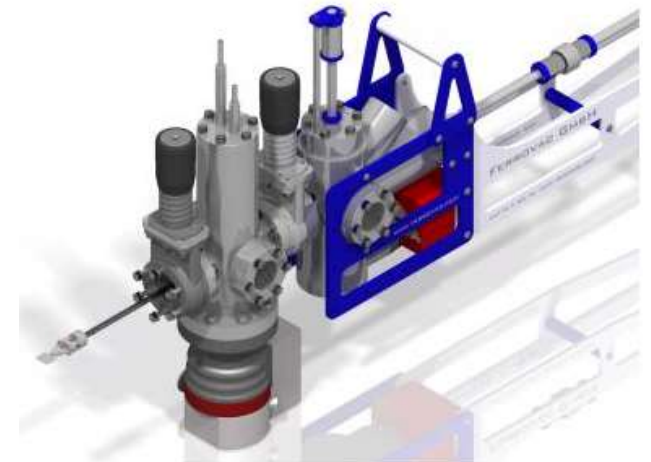




Vacuum Cryo Transfer Module: A Suitcase Solution

Why Cryo? Why now?

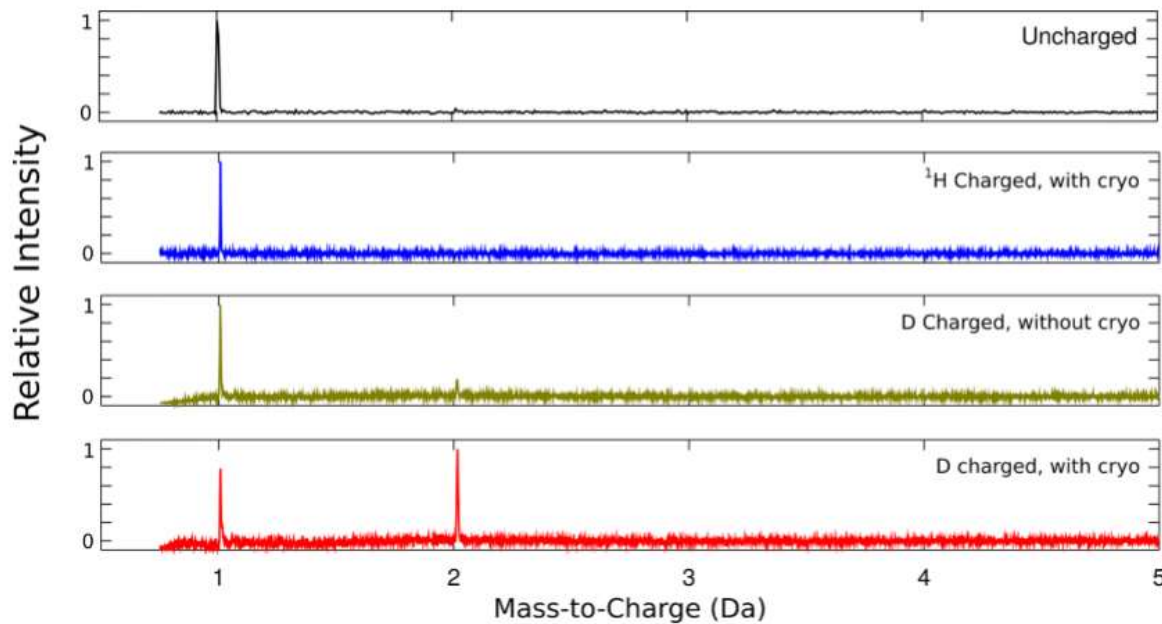
- A number of both prospective and current LEAP customers have requested the ability to transfer samples in and out of the LEAP microscope under either ultra-high vacuum (UHV) or cryogenically cooled conditions. Samples could be transferred to/from SEM, TEM, FIB, coating tools, etc.
 - **Transport between various analysis modalities (e.g. FIB, TEM) under vacuum conditions – e.g. Radioactive materials/ contamination control**
 - **Rapid oxidation studies (e.g. uranium, lithium, aluminum)**
 - **Catalyst/reaction chamber studies of surface contamination**
 - **Characterization of hydrogen embrittlement of metals**
 - **Analysis of “soft” (i.e. biological) materials**
- Since ~2010 we have delivered prototype solutions to customers at ETH and University of Michigan based on a transfer module manufactured by Leica (VCT 100/500).
- APT users have been developing their own solutions independently
- Growing interest and partnerships have encouraged a standard offering



Why Cryo Transfer? – Hydrogen in Steel Example

▀ Cryo transfer enables/ improves the ability to collect atom probe data for difficult applications

- *Rapidly oxidizing materials (uranium, plutonium etc)*
- *Detection of hydrogen in alloys (Zr and steels)*
- *Characterization of H embrittlement of steel*
- *Transport between various analysis modalities (e.g. FIB, TEM, glovebox) under vacuum conditions*

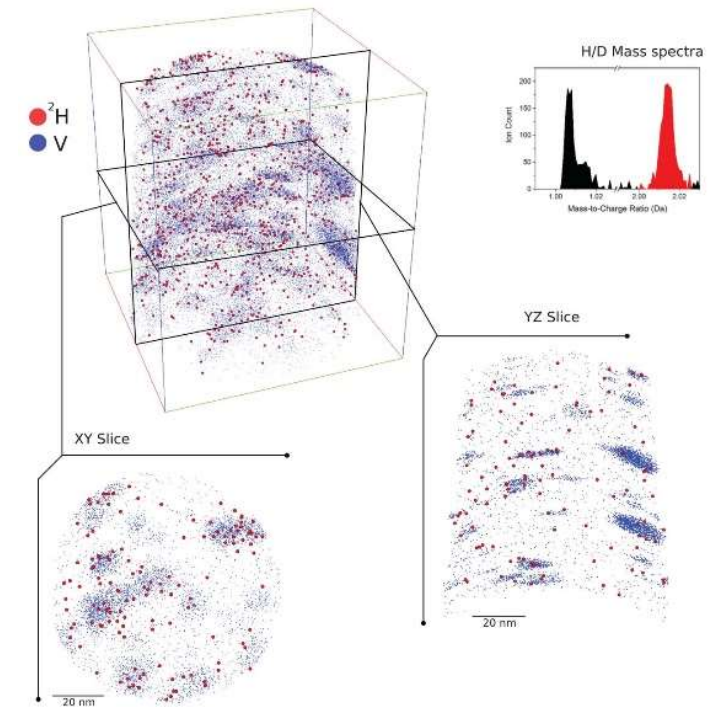


Direct observation of individual hydrogen atoms at trapping sites in a ferritic steel

Y.-S. Chen, D. Haley,* S. S. A. Gerstl, A. J. London, F. Sweeney, R. A. Wepf, W. M. Rainforth, P. A. J. Bagot, M. P. Moody

*Corresponding author. Email: daniel.haley@materials.ox.ac.uk

Published 17 March 2017, *Science* **355**, 1198 (2017)
DOI: 10.1126/science.aal2418



User Options for Cryo Transfer



Leica VCT 500

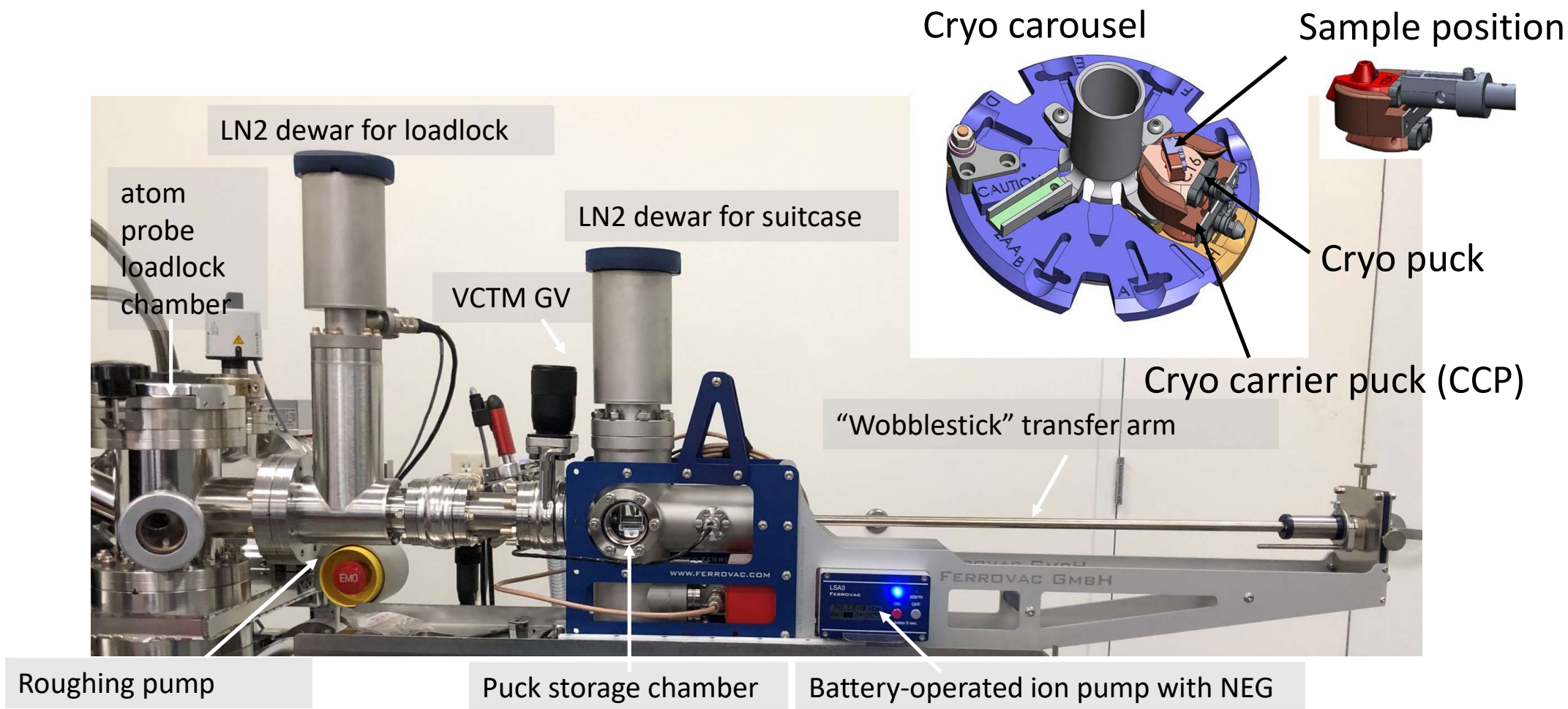


Quorum PP3006

- Leica and Quorum chambers not actively pumped, may compromise vacuum quality



Cryogenic Transfer Components



Transfer Procedure

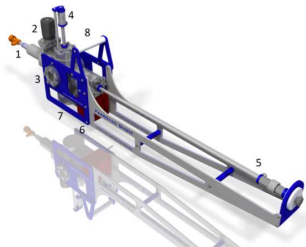
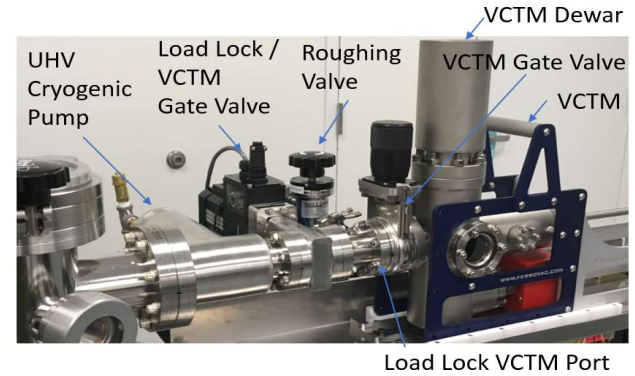


Figure 3: Overview of the NexGeneration UHV suitcase and its parts.

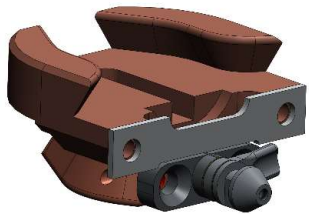


Sample prep in Cryo FIB/glovebox/reaction chamber runs

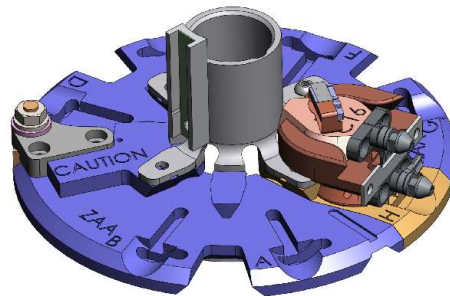
Suitcase attached to FIB/glovebox, sample transferred to cold stage. (3-5 min)

Suitcase travels to LEAP

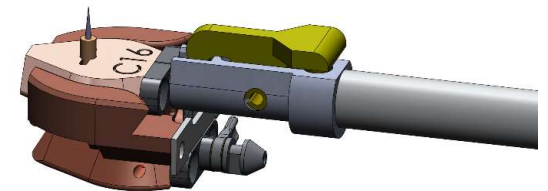
Suitcase attached to LEAP loadlock chamber (5 min)



Carrier puck pre-cooled in LEAP analysis chamber at UHV (1 hour)



Carrier puck transferred to LEAP loadlock, specimen puck is attached and lowered to buffer chamber (30 seconds)



Specimen puck transferred to the analysis chamber (30 seconds)

For your memory

- ▀ APT = 3D analysis at atomic scale.
- ▀ There are 2 methods to prepare samples : electrolytic (conductive samples) and FIB (mostly non-conductive samples)
- ▀ There are 2 commercialized version of the APT: LEAP5000 (versatile and high T-put) and EIKOS (nearly full automation + easy to use).
- ▀ LEAP5000 has 2 versions : straight (Si \geq 77% of transmission) vs reflection (R = no more than 50% of transmission).
- ▀ EIKOS has only the R version
- ▀ All APTs can be equipped with laser (UV) to analyse non-conductive materials.
- ▀ Cryo transfer is only available with LEAP5000.

Thank you for your intention!

