Cs+ SIMS using a Low Temperature Ion Source (LoTIS)

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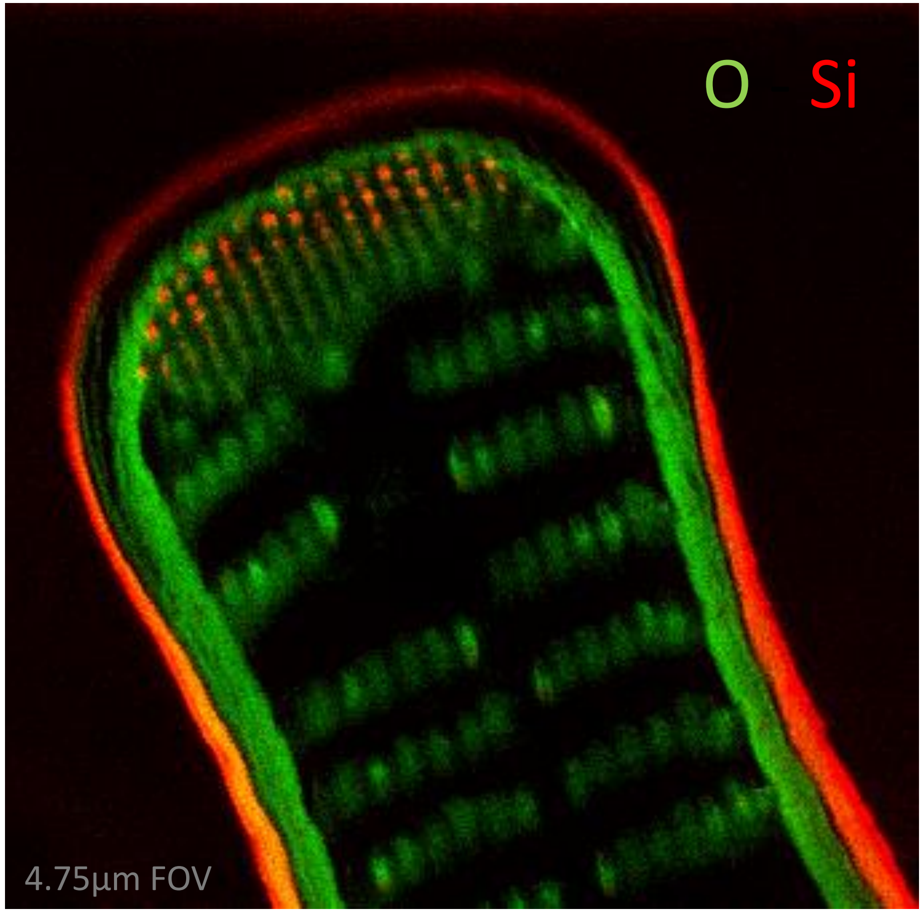


J-N Audinot, O de Castro; acquired using SIMS:ZERO at zeroK

What can LoTIS do?

Energy (kV)	Current (pA)	Spot Size (1-σ nm)
16	1.5	<2.0
8	3	2.5
5	3.5	7
2	3.7	20
16	100	30
8	100	55
16	5500	580
8	2600	510
Spotsize conversions	$d_{35-65} = \sigma/1.3$ $d_{16-84} = 2\sigma$ $d_{50} = 2.2\ \sigma$	

LoTIS produces Cs+ ion beams that can be focused very tightly

- 2-16 keV
- Spotsize <2nm at low current (pA)
- Good spotsizes even at low beam energy
- Up to 5nA beam current

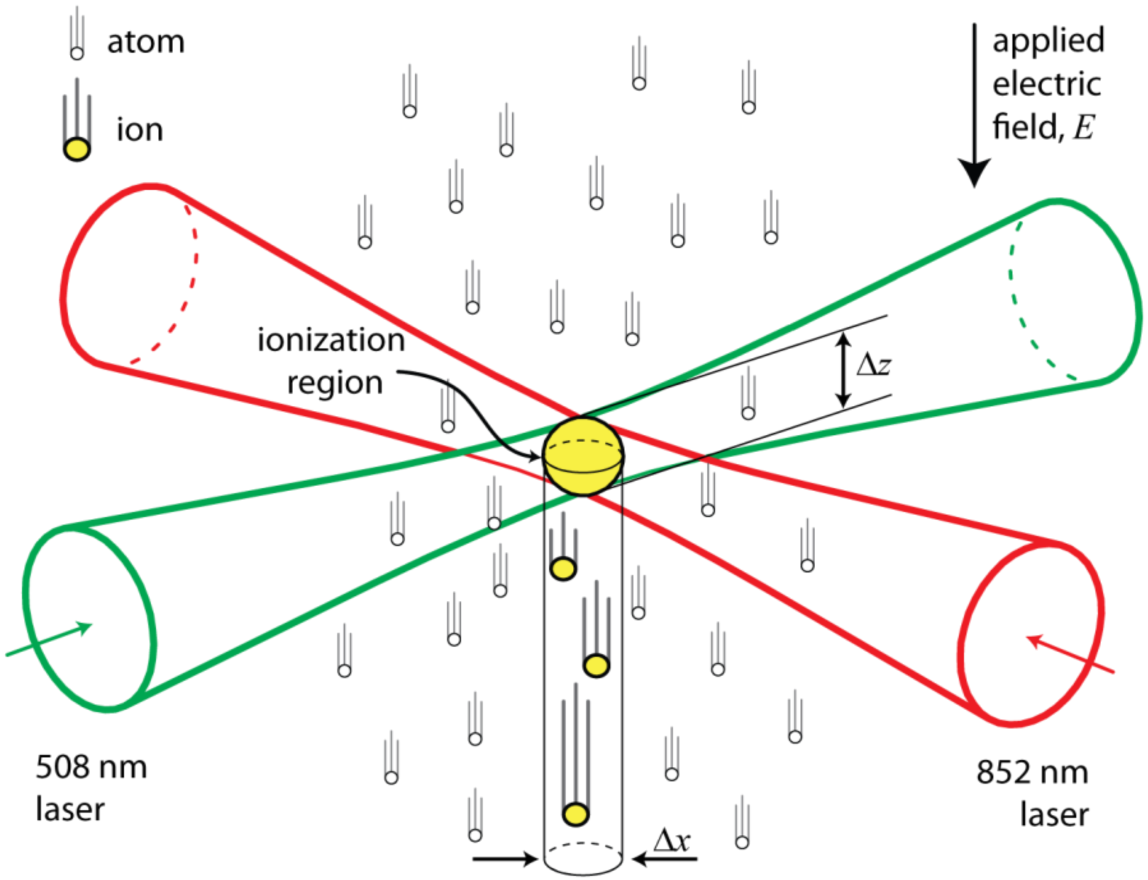
Good for

- High spatial resolution microscopy (SIMS and SE)
- FIB Sample preparation
- Nanofabrication

How does LoTIS work?

Ions are created in a laser-cooled atomic beam as it flows through the intersection of photoionizing laser beams

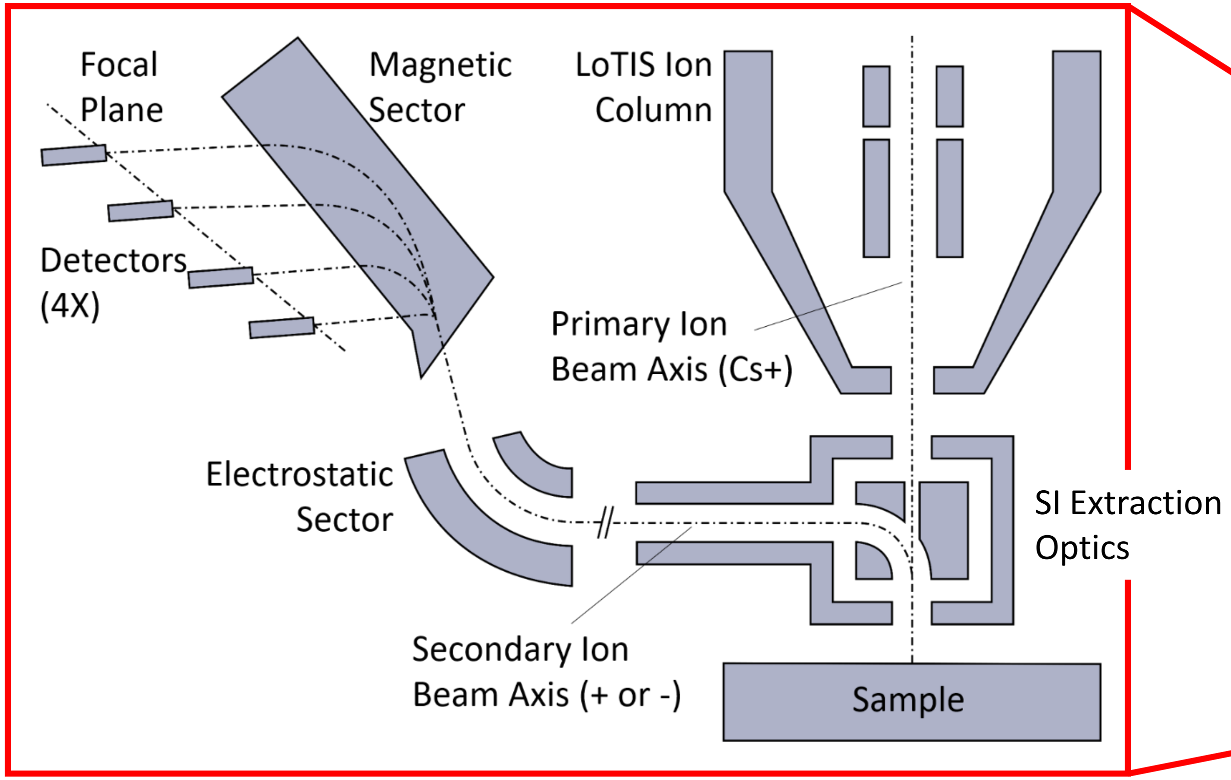
The cold temperature (~10 µK) is the key to achieving finely focused beams

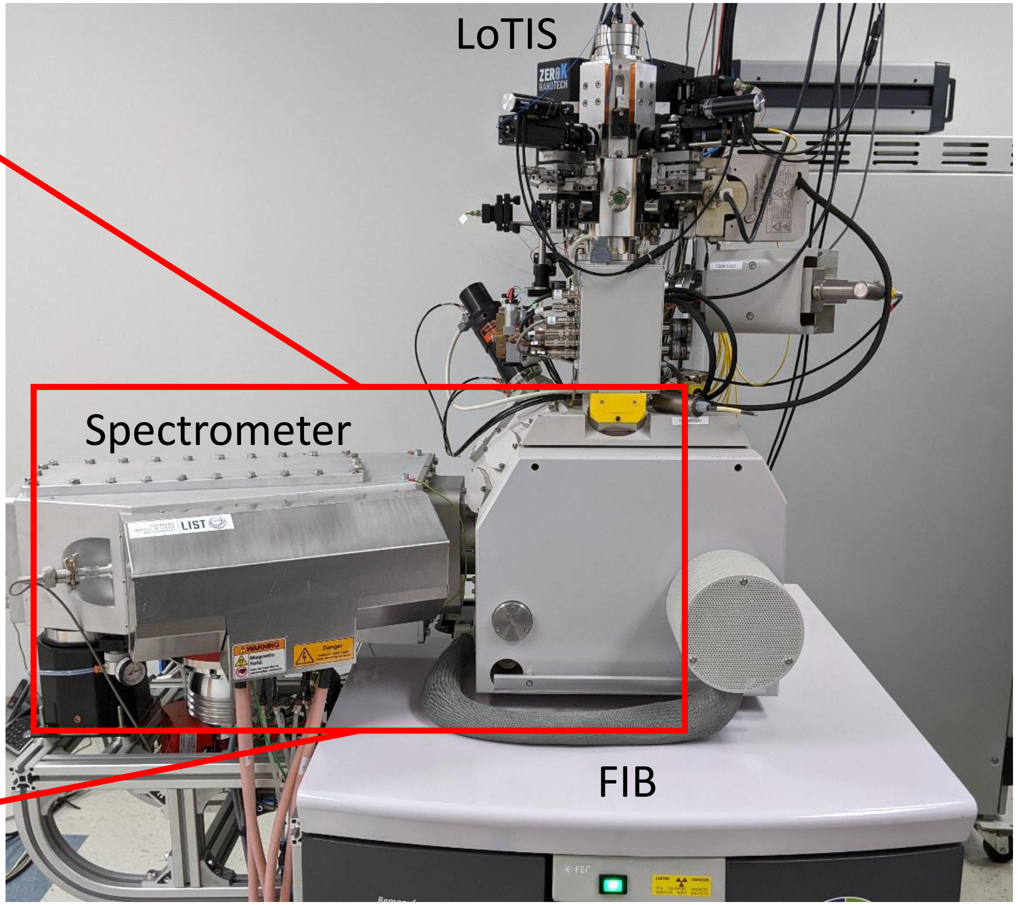


SIMS:ZERO

Instrument Overview

Cs+ FIB:ZERO (zeroK) and SIMS spectrometer (LIST: Luxembourg Institute of Science and Technology) on a 600 series FIB (FEI)



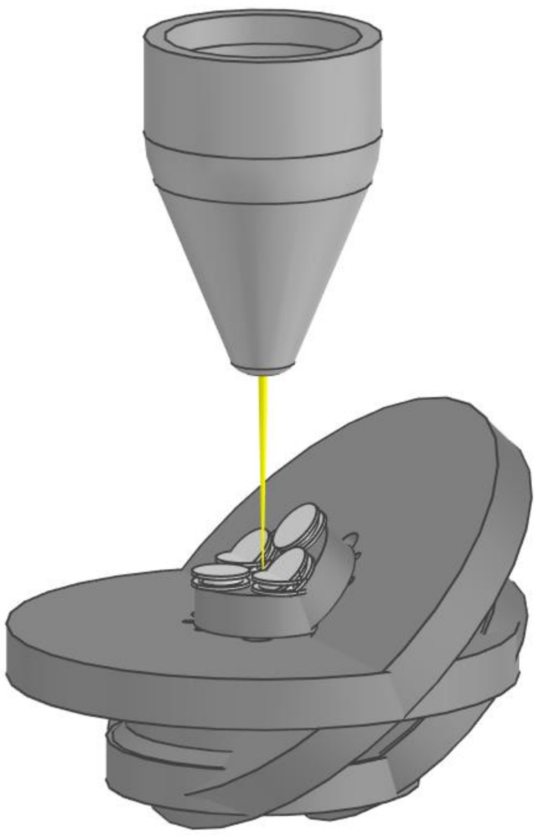


- FIB online 6/2020
- SIMS online 5/2021

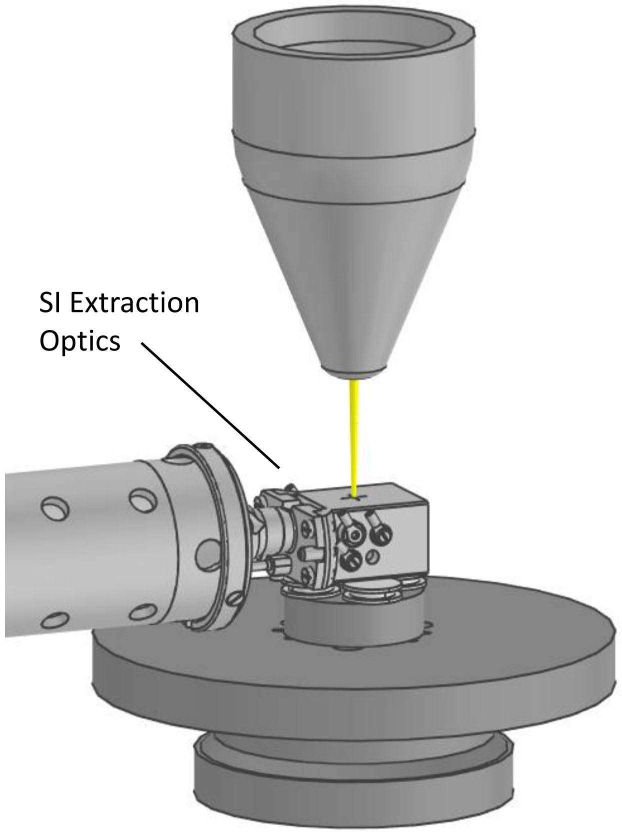
FIB / SIMS Combination

Sample Prep / SIMS Analysis, Nanofabrication / SIMS Process Control

FIB Mode



SIMS Mode



LoTIS capabilities

- 2-16 keV Cs+ beam
- Up to 5nA beam current
- Spotsize <2nm at low current
- Good spotsizes even at low beam energy

FIB Mode (SIMS Extraction Optics Retracted)

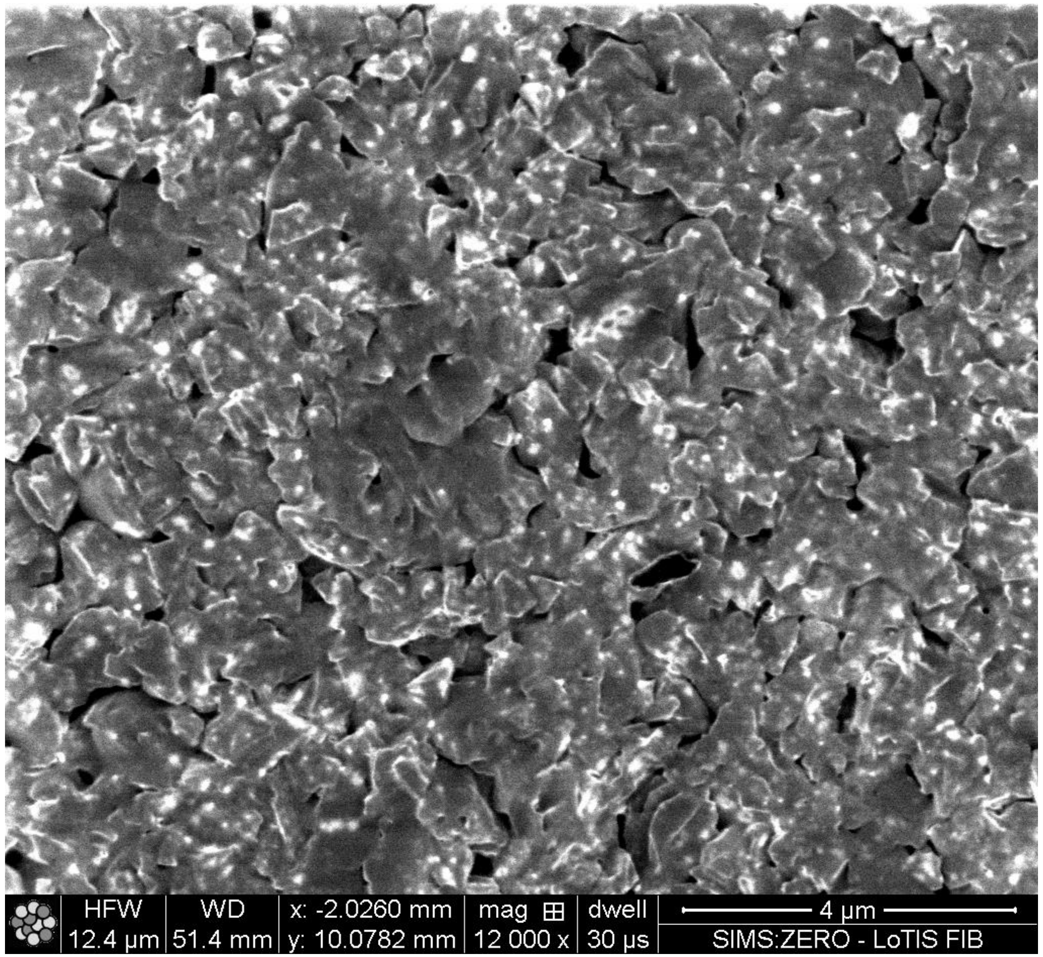
- Milling
- Sample Preparation (eg Sectioning, Polishing)
- Nanofabrication
- Gas-assisted processes (eg Platinum Deposition)
- Tilt stage

SIMS Mode (SIMS Extraction Optics Inserted)

- Highest spatial resolution SIMS imaging
  - $\sigma = 6$ nm demonstrated
- Mass resolution $M/\Delta M = 400$
- Mass range up to 300 amu
- High secondary ion throughput (~40% simulated)
- 4-Channel Detector Standard (Continuous Focal Plane Detector available)

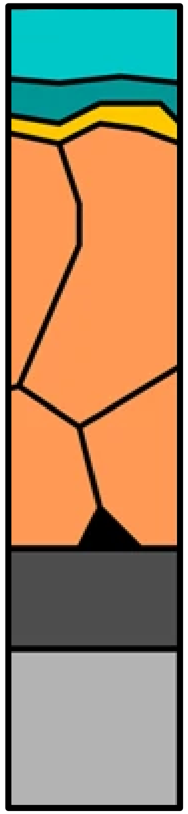
SIMS Analysis Example

CIGS $Cu(In,Ga)Se_2$ – Rb doped



SE Image Cs+, 16keV, 10pA, 51.6mm WD

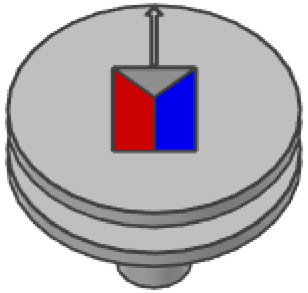
- CIGS is a solar cell absorber material
- Multilayer sample a few micron thick with pronounced grain structure
- Section view technique provides superior SIMS + SE imaging data



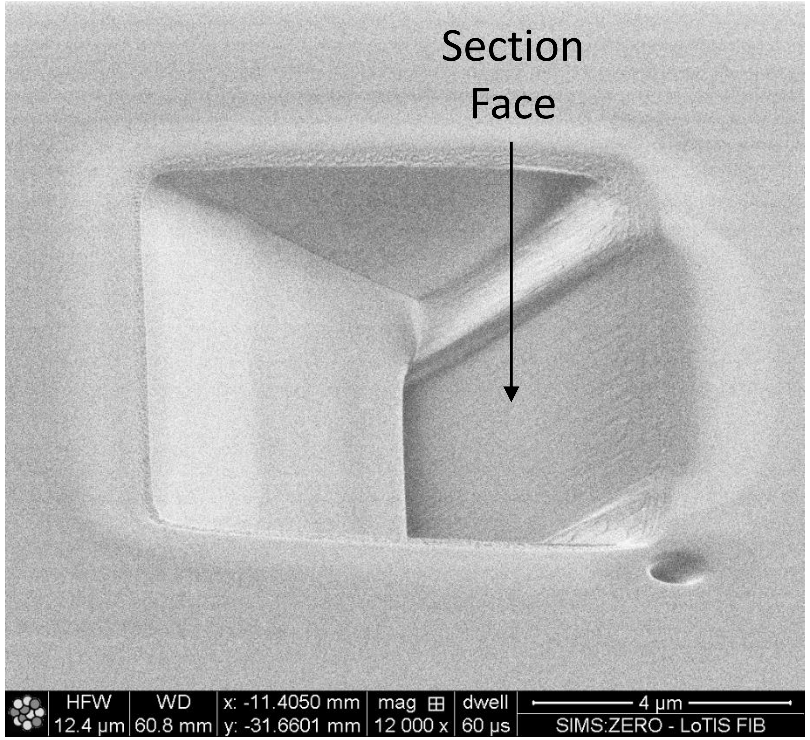
Werner, et al. *Scientific Reports* **volume 10**, 7530 (2020)

SIMS-Compatible Section View

45° Angle Cut - Example



View with
Sample Tilted at 45°
& Rotated 90°



For many samples, working with a section view is a sensible choice

1. Reveal sub-surface structure
2. Obtain depth profile information without accumulated topography from uneven sputtering
3. Polish rough samples to isolate elemental from topographical contrast
4. Build 3D tomographic reconstructions through serial sectioning/polishing

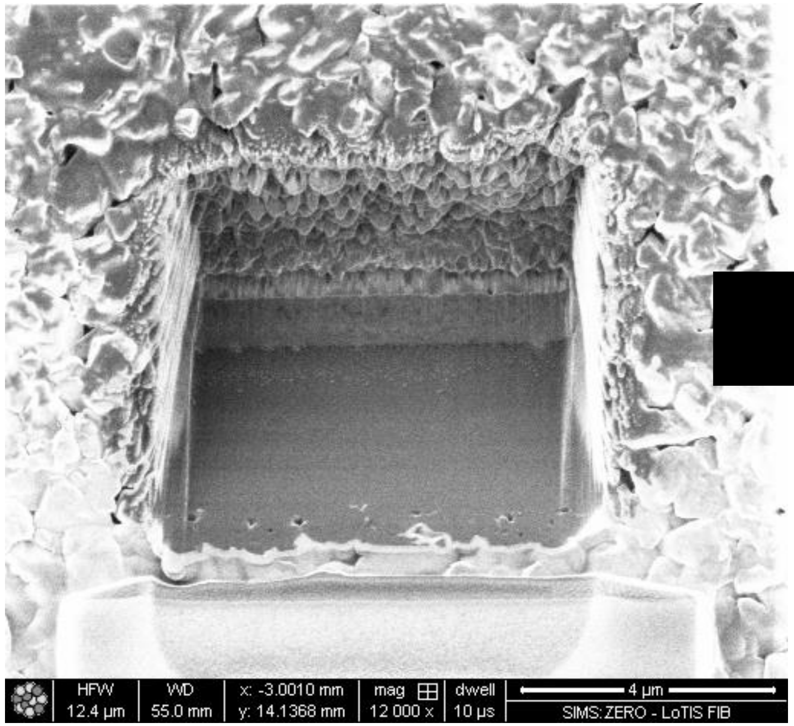
In SIMS:ZERO, sample must be normal to ion beam in SIMS Mode, so section face is cut at 45° to sample surface

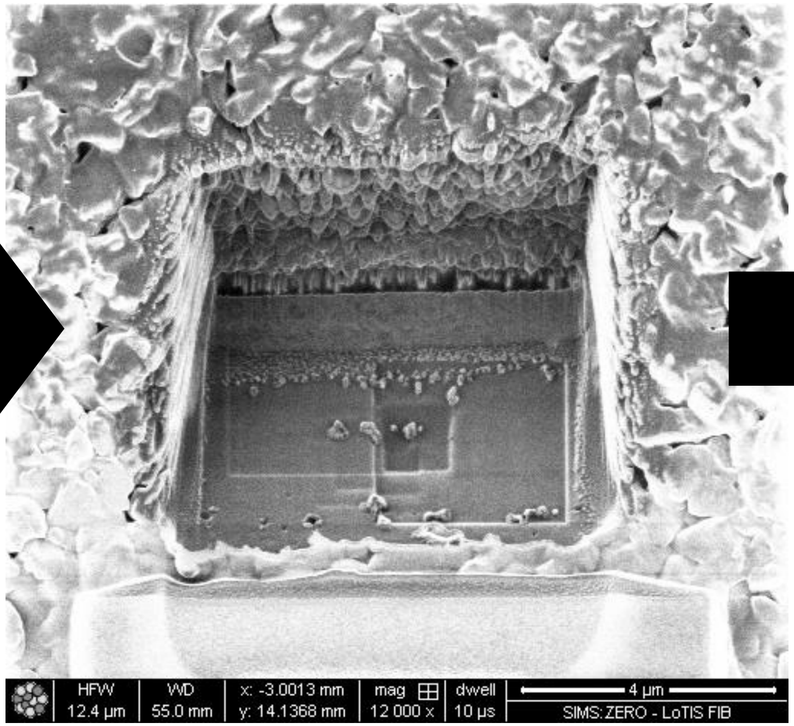
This sectioning is done in FIB mode. Switching between FIB and SIMS mode takes about 2 min

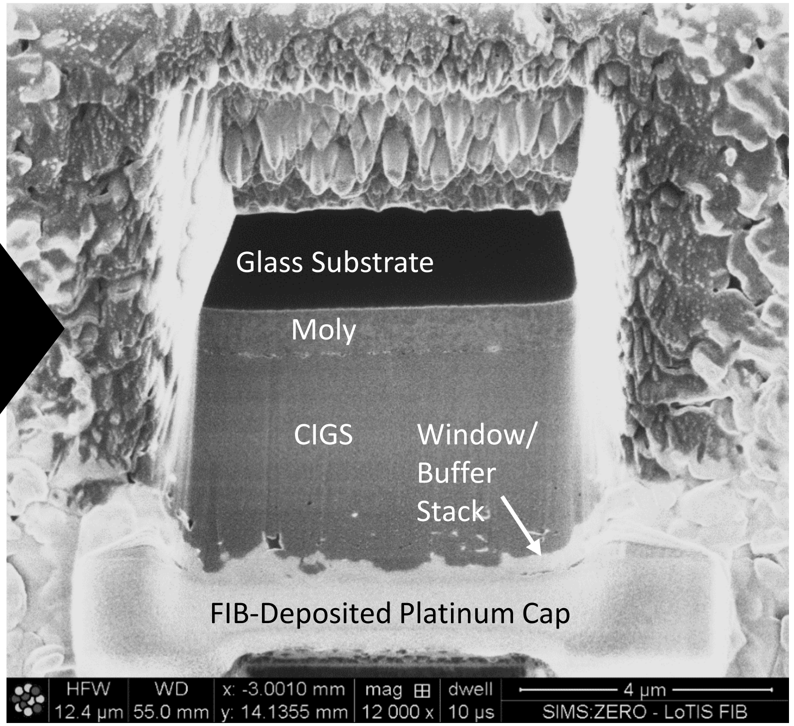
CIGS $Cu(In,Ga)Se_2$ – Rb doped

Serial Sectioning / Imaging / Polishing Work-Flow

SE Images







SIMS section, prepared with low surface topography, reveals layer structure (glass, moly, CIGS, Window/Buffer Stack)

After SIMS Imaging, section face develops topography which obscures elemental contrast / distribution information

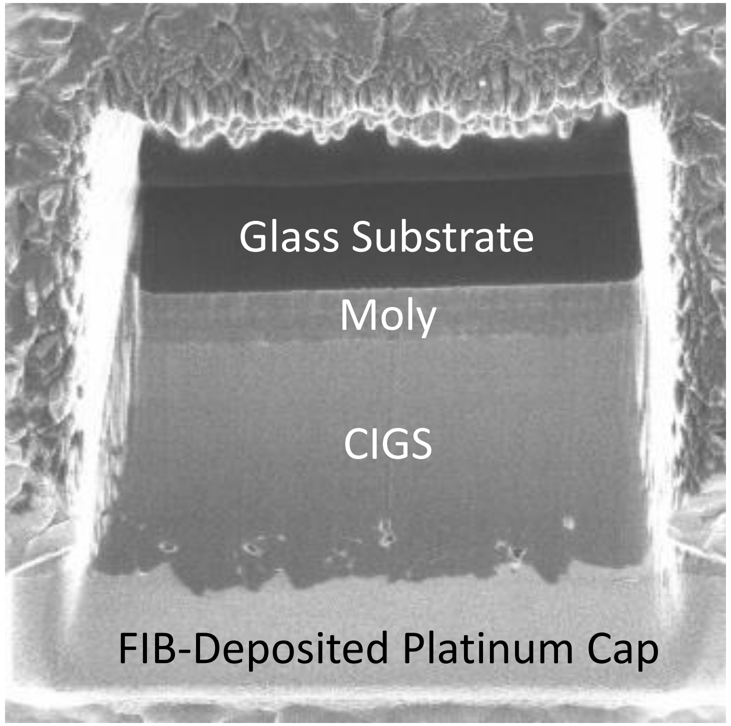
Section face after cleanup mill. Ready for SIMS on next layer

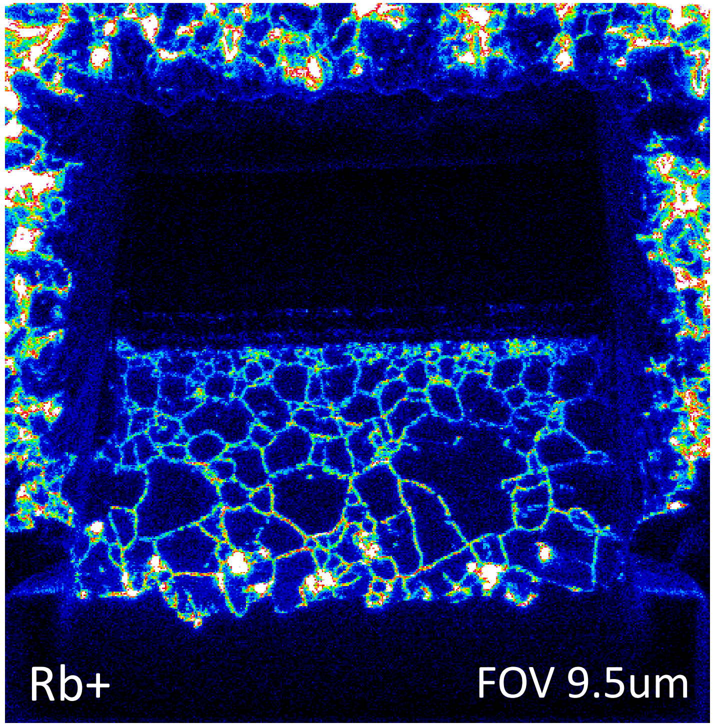
Cs+, 16keV, 10pA, 51.6mm WD

CIGS $Cu(In,Ga)Se_2$ – Rb doped

Section View – Positive Ions

SE Image – Pre-SIMS



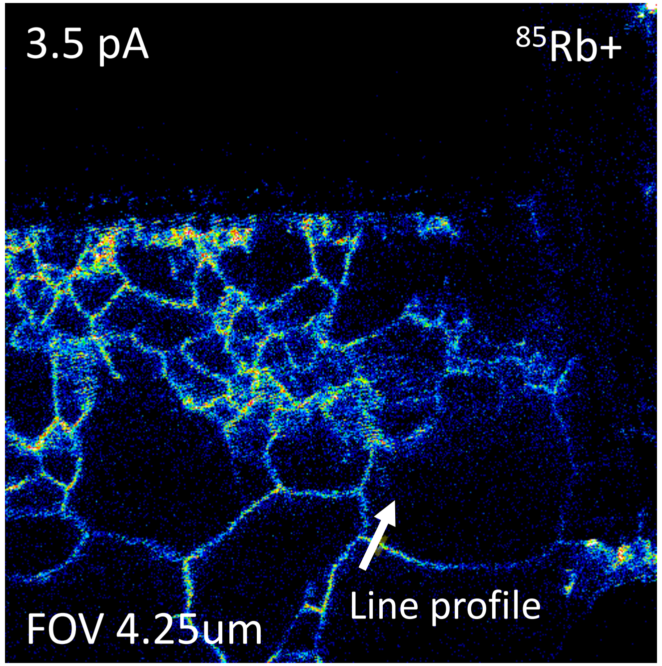


- Rb confined to grain boundaries
- Grains are smaller near the interfaces
- Bilayer structure in the Moly layer

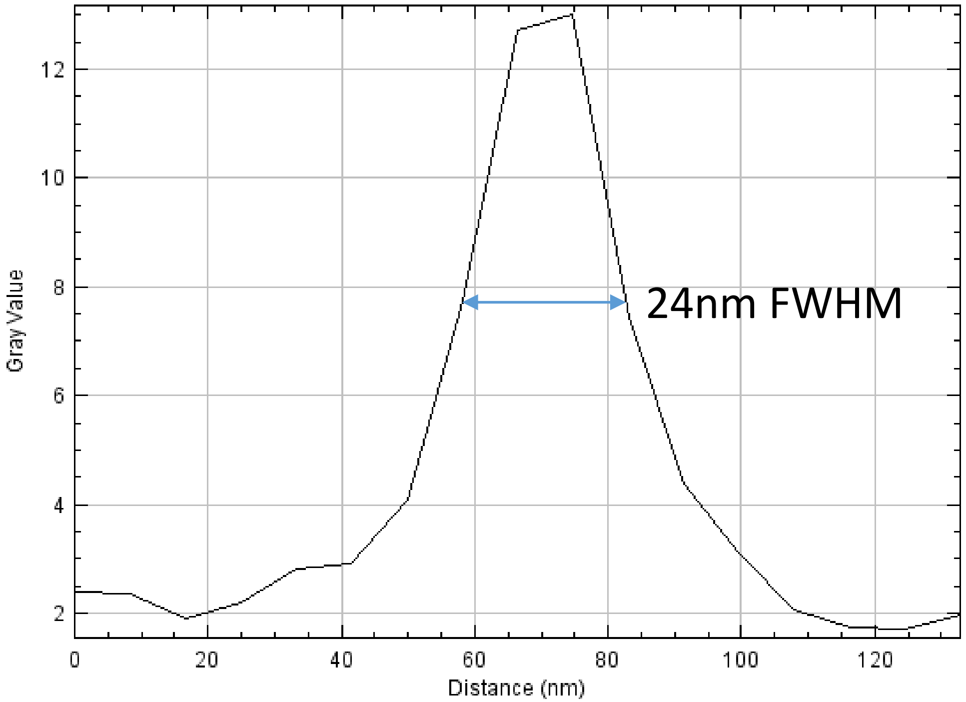
Cs+, 16keV, 3.5pA, 51.6mm WD
CIGS_Pos_2107161606287.csv
CIGS_Pos_2107161613425.csv

CIGS $Cu(In,Ga)Se_2$ – Rb doped

Section View – Positive Ions



Apparent width of Rubidium signal between grains



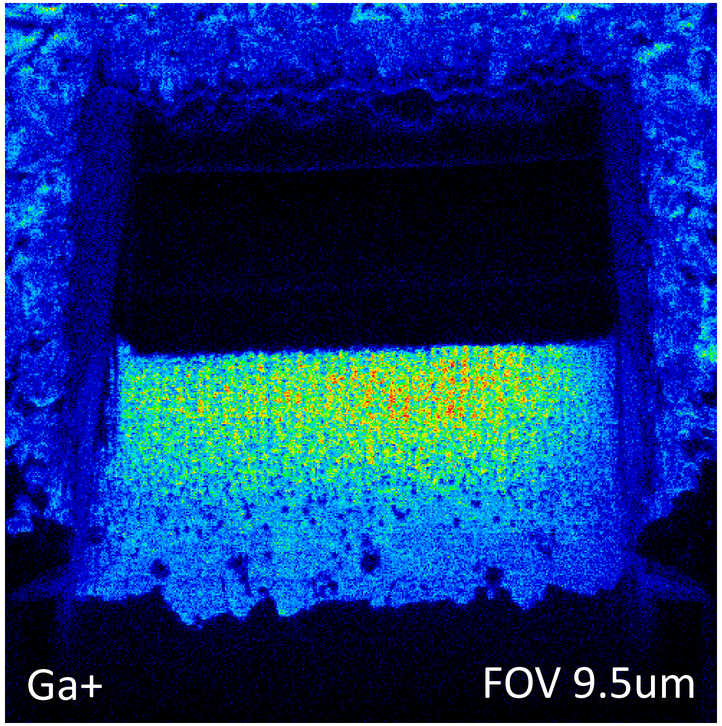
Cs+, 16keV, 3.5pA, 51.6mm WD

CIGS_Pos_2107151409368.csv

CIGS $Cu(In,Ga)Se_2$ – Rb doped

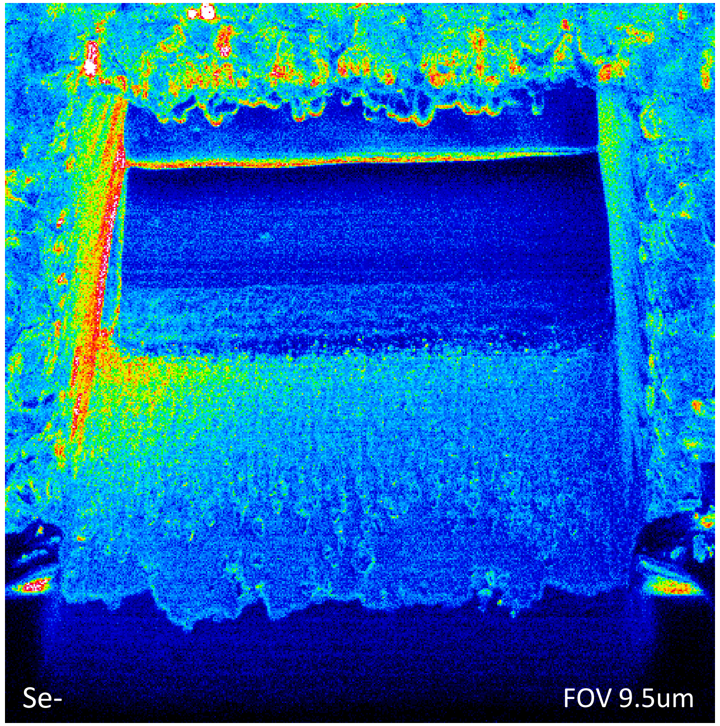
Section View – Positive Ions / Negative Ions / SE

Positive Mode



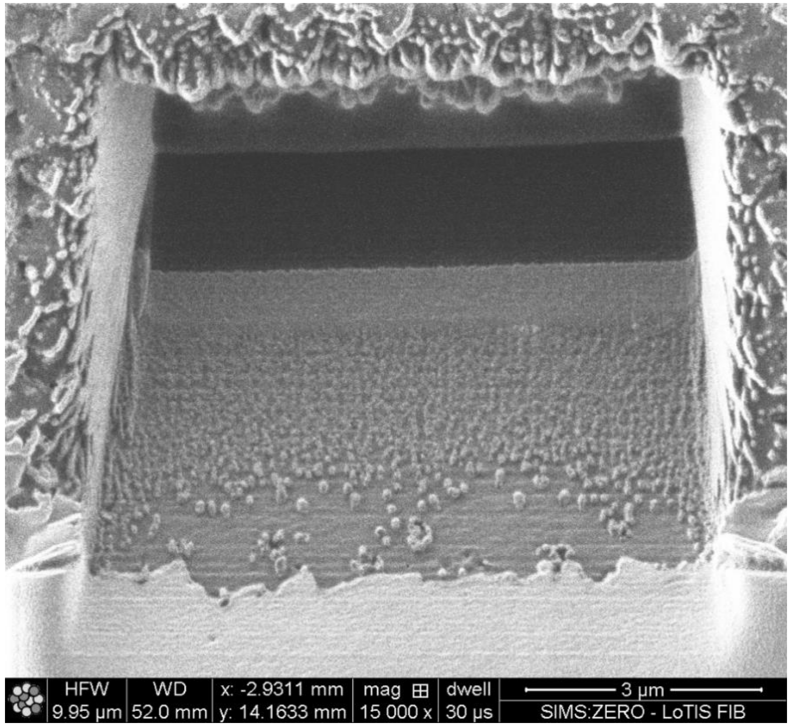
- Ga concentration gradient ↑
- Dark spots appear on image

Negative Mode



- Spherical drops containing Se develop on CIGS region after repeated imaging

SE Image – Post SIMS



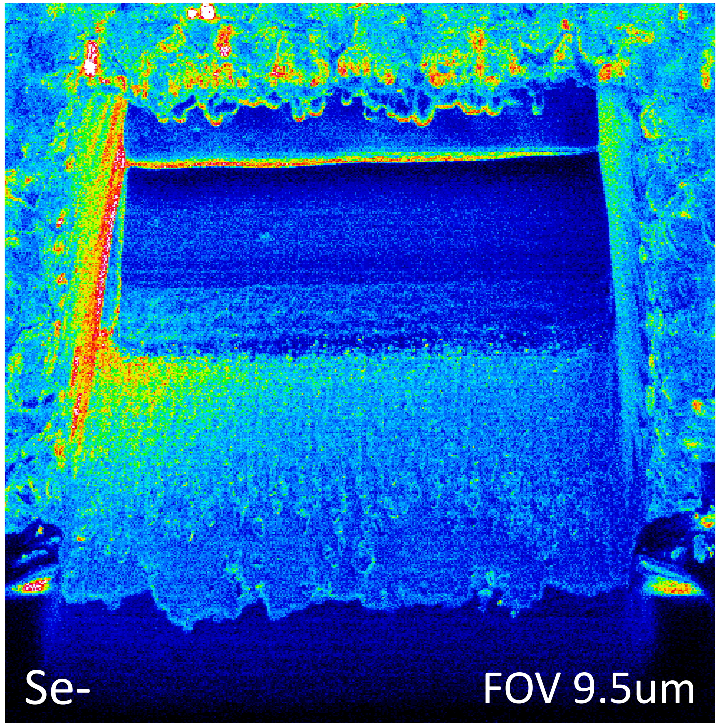
- Unwanted topography can be cleaned up by polishing in FIB mode

Cs+, 16keV, 3.5pA, 51.6mm WD
CIGS_Pos_2107161613425.csv
CIGS_Neg_2107161719423.csv

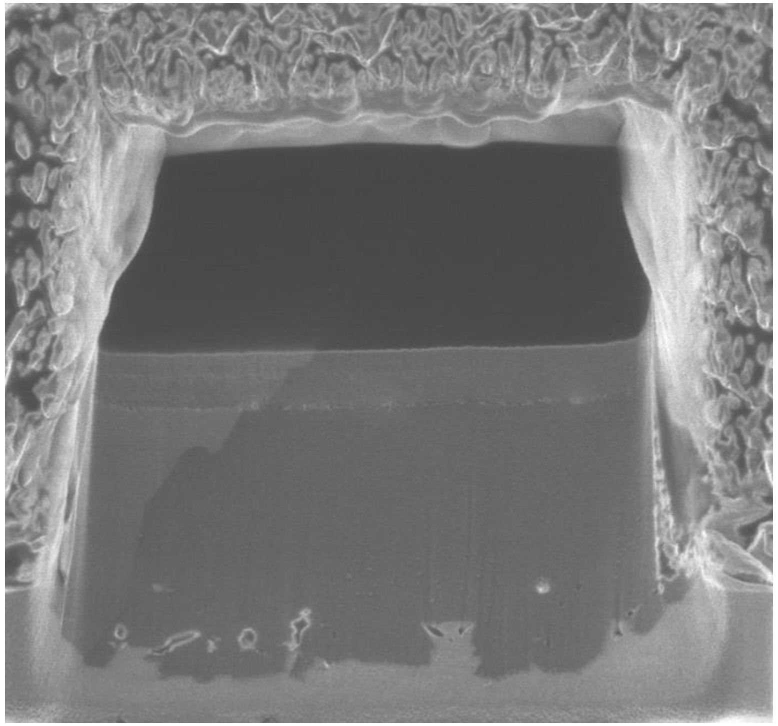
CIGS $Cu(In,Ga)Se_2$ – Rb doped

Section View – Negative Ions – Post 2nd Polish

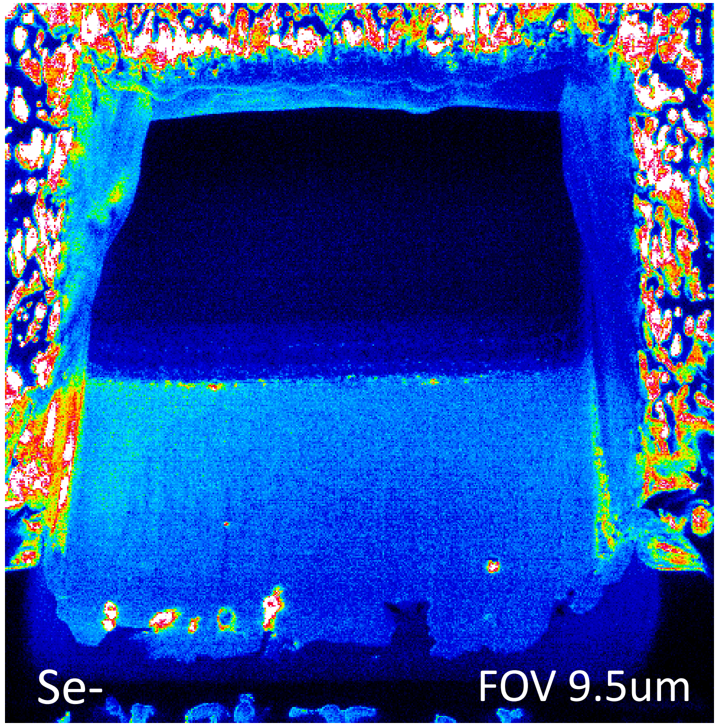
Cs+, 16keV, 10pA, 51.6mm WD
CIGS_Neg_2107201513310.csv



Spherical drops exhibiting Se signal develop on CIGS region after repeated imaging



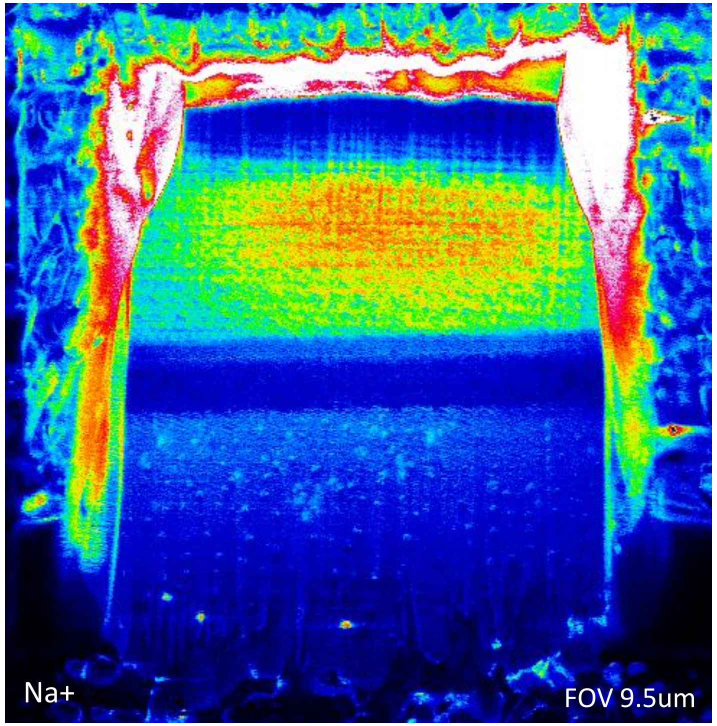
SE Image – Post Polish
Low topography restored



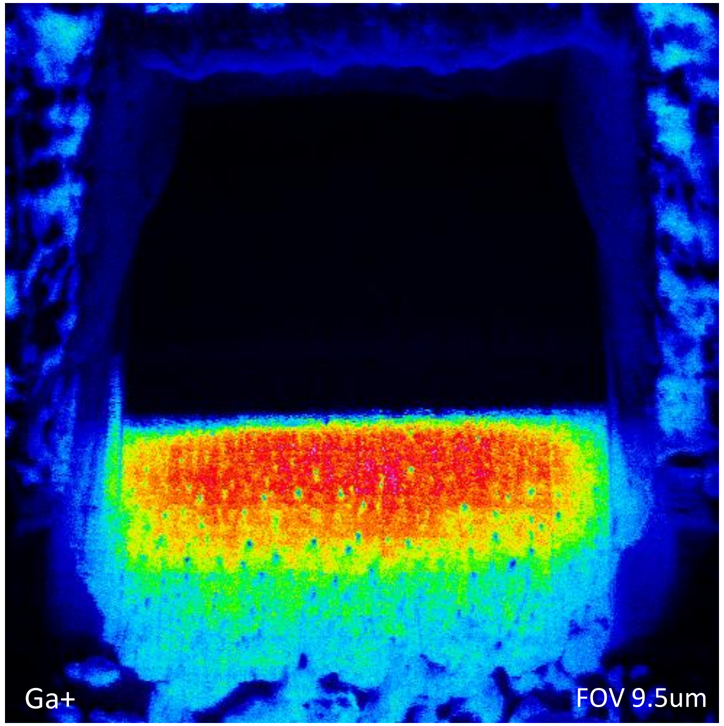
Se is more uniformly distributed in CIGS layer; droplets at moly interface, a few inclusion near surface

CIGS $Cu(In,Ga)Se_2$ – Rb doped

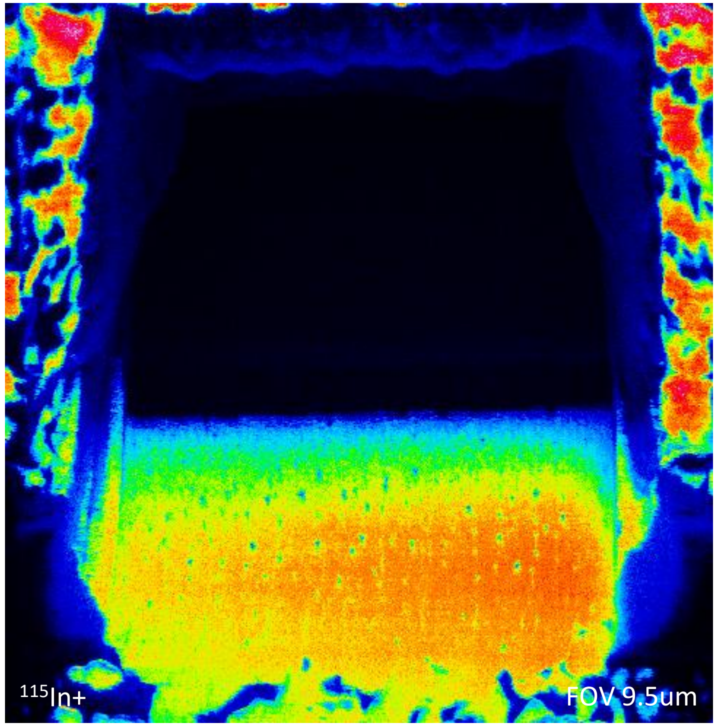
Section View – Positive Ions – Post 3rd Polish



Na – Soda Lime Glass



Ga concentration gradient ↑

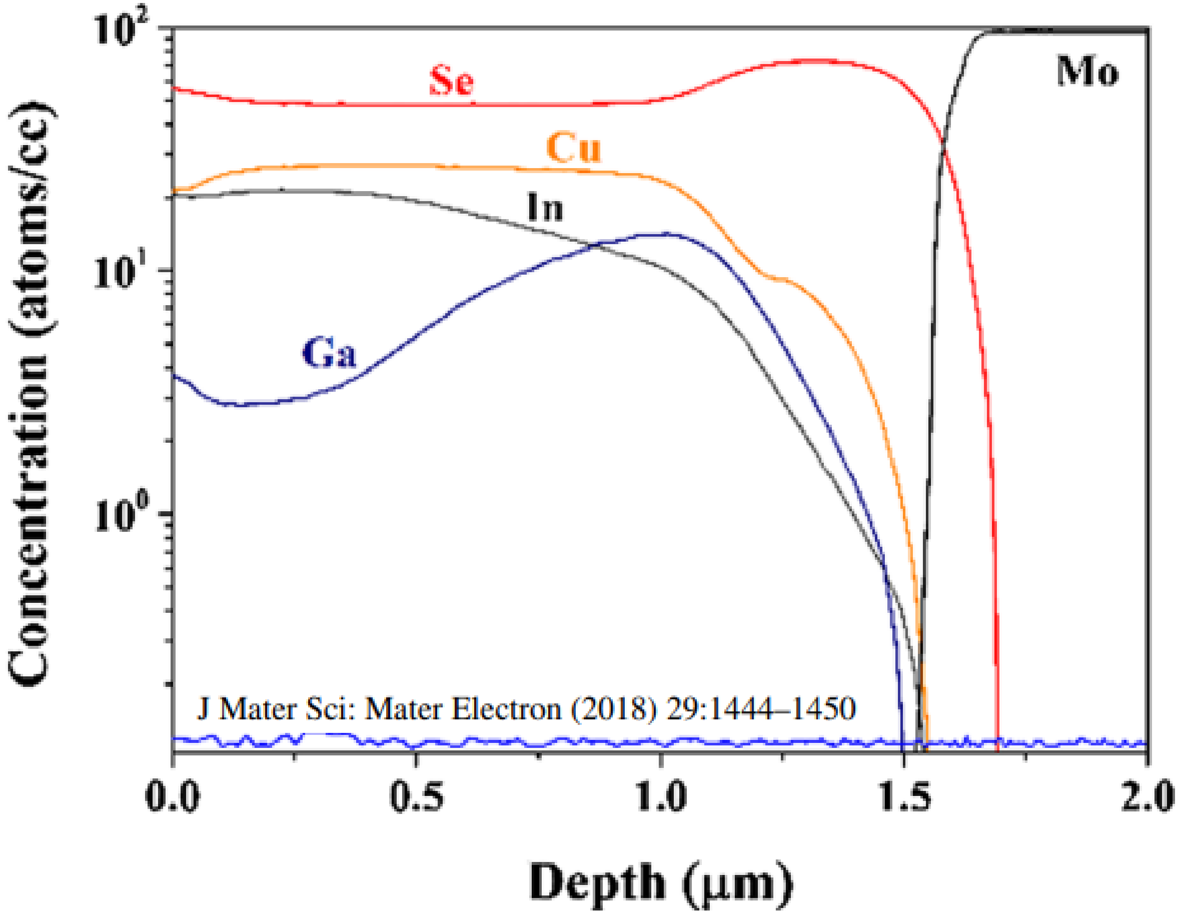


In concentration gradient ↓

Cs+, 16keV, 10pA, 51.6mm WD
CIGS_Pos_2107201626359.csv

CIGS $Cu(In,Ga)Se_2$

SIMS Depth Profile From Literature

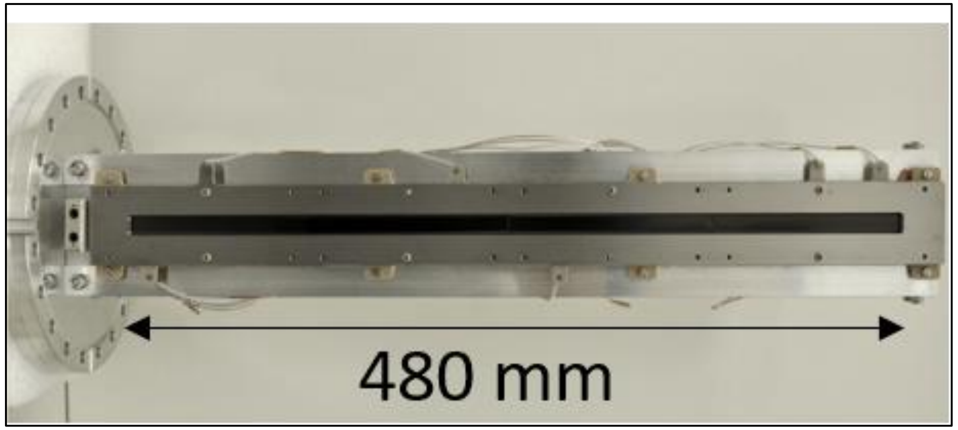


SIMS + SE imaging data contains Depth Profile information and more…

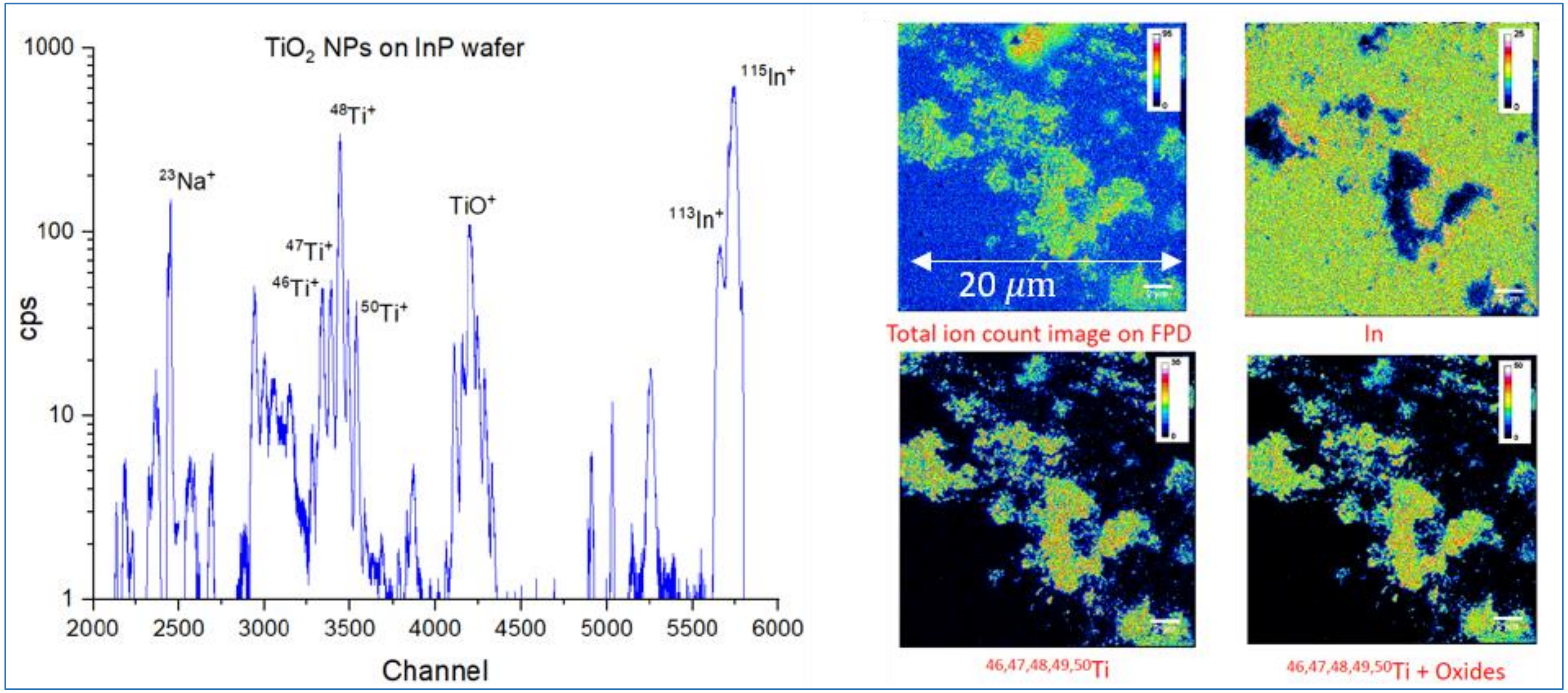
- Selenium localization in nodes on Moly/CIGS boundary and within the Moly bilayer
- Grain/Layer structure in the Moly layer
- Rb reveals intricate grain structure with grain size gradients
- Images contain concentration gradient information for Ga and In
- High resolution SE data provides info
  - Film structure
  - Layer thickness
  - Presence of topographical features

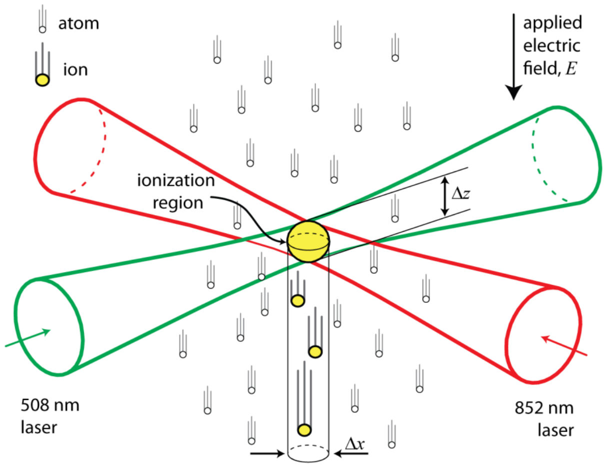
Continuous Detector

A SIMS:ZERO Option



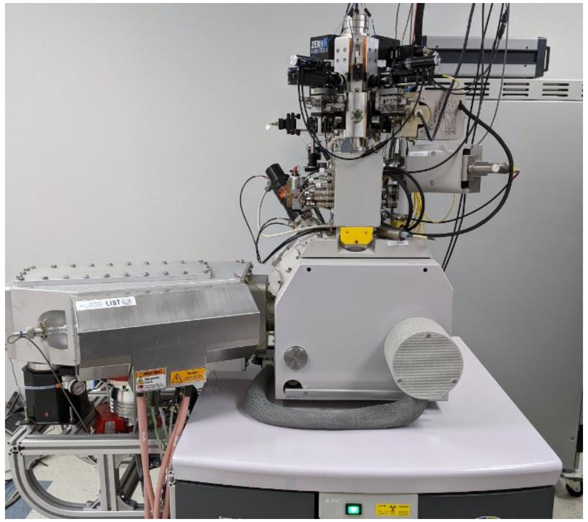
- Replace discrete detectors with continuous focal plane detector
- Sample the entire mass spectrum at once
- Reduce loss of information caused be discrete detector array
- Combine related signals (eg Ti, TiO, etc) to increase signal intensity / SNR
- Short acquisition times compared to TOF

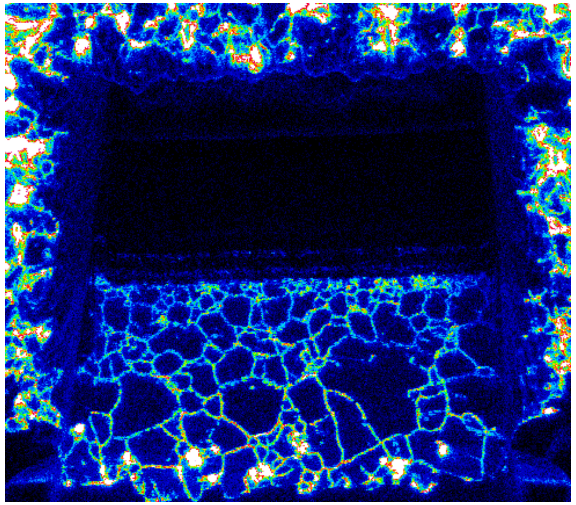




LoTIS is a new Cs+ ion source providing both high resolution SIMS and FIB machining capability, compatible with a range of ion optical systems

SIMS:ZERO offers Cs+ LoTIS for
SIMS + Sample Prep + Nanofabrication
in a single instrument

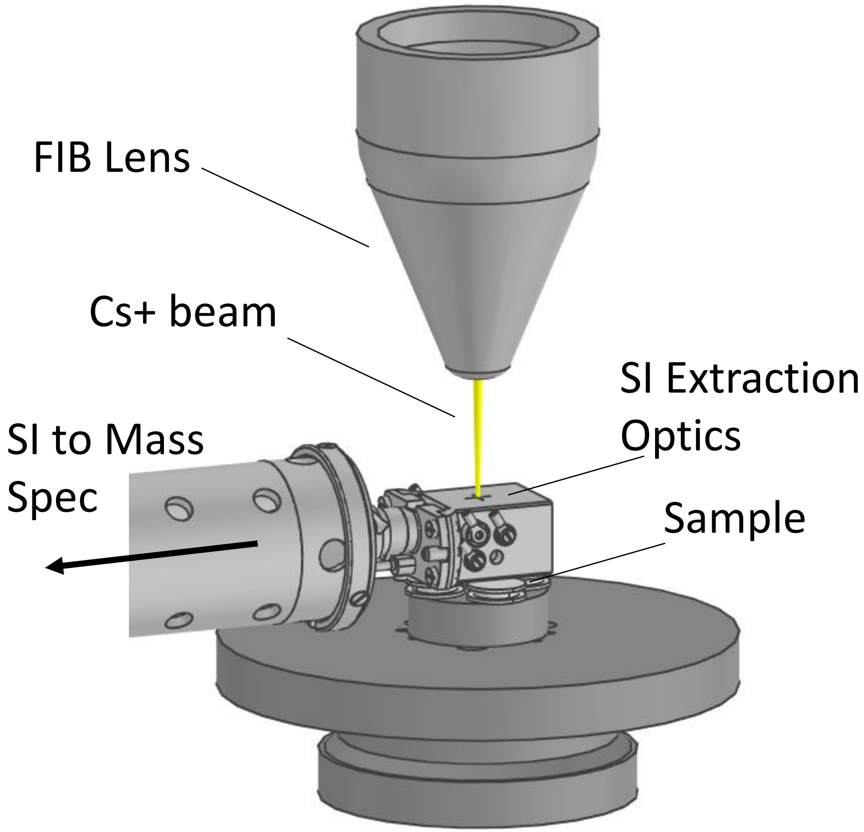




SIMS analysis with LoTIS provides insight into the structure and composition materials at previously unachievable resolution

Nanofabrication with SIMS:ZERO

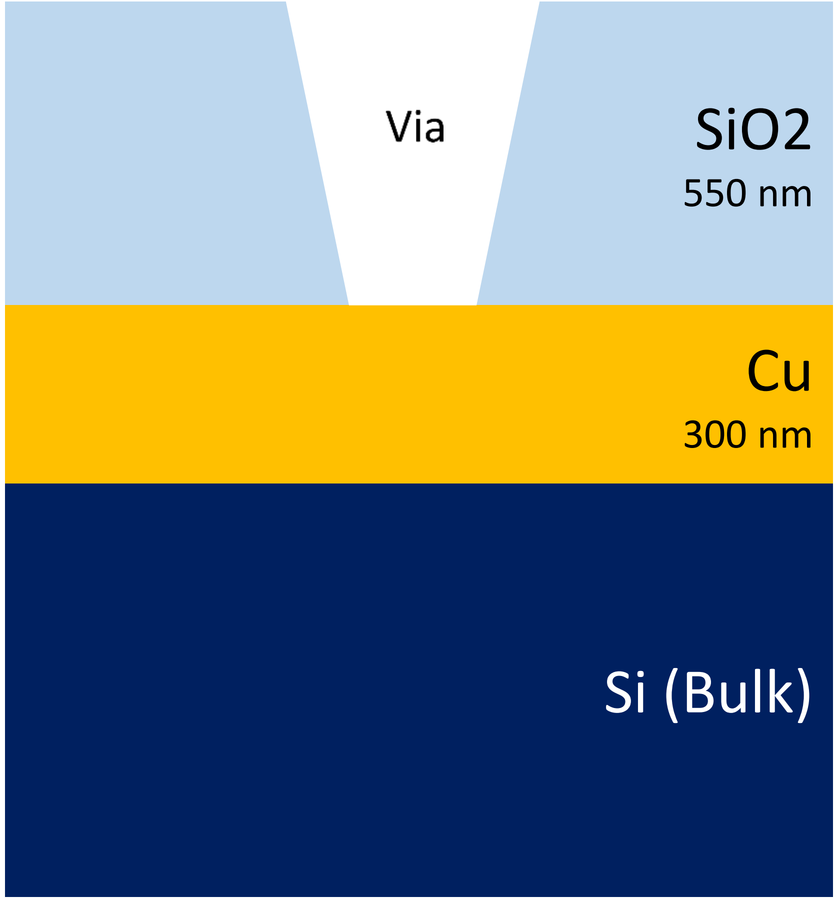
FIB machining process control using SIMS



- To collect SIMs data while machining
  - Secondary Ion (SI) extraction optics are inserted between FIB lens and sample
  - SIs extracted while machining are transferred to a mass spectrometer to analyze the composition of machined material in real time
- FIB machining acuity is minimally impacted by the transit through the extraction optics
- Gas delivery can be integrated into the SI Extraction Optics assembly

Endpointing Example

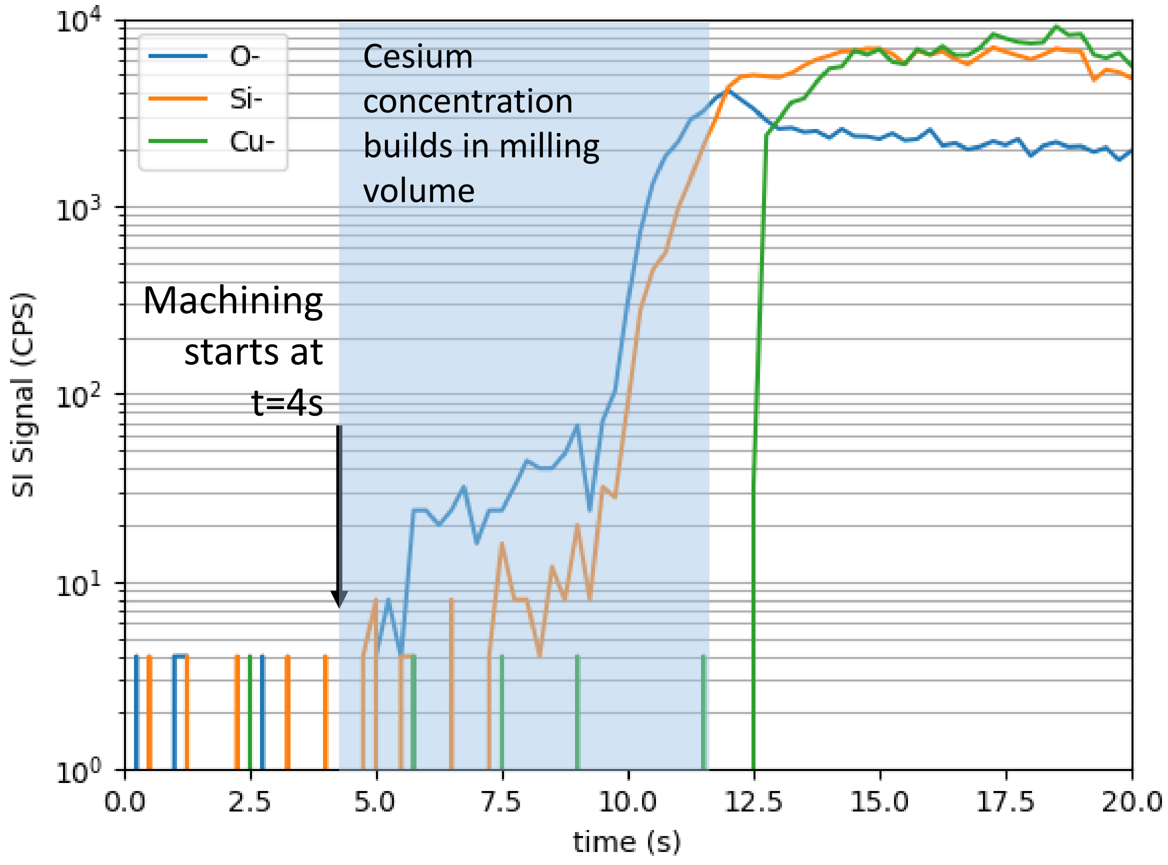
Test sample SiO_2 on Cu



- Objective is to mill via through SiO_2 and stop when Cu is reached without over-milling
- Typically done by monitoring for a change in SE yield, but SE signal can be difficult to interpret
  - SE yield can change due to topography (sidewall), grounding (voltage), material contrast, etc
  - SNR, Contrast is very low for high aspect ratio vias
- Monitoring the Secondary Ion Signal on one or more elemental channels provides
  - Multiple signal channels for analysis
  - More definitive information, ie "Cu is Cu", "Si is Si", etc
  - High SNR, Contrast signals

SIMS Signal while Machining

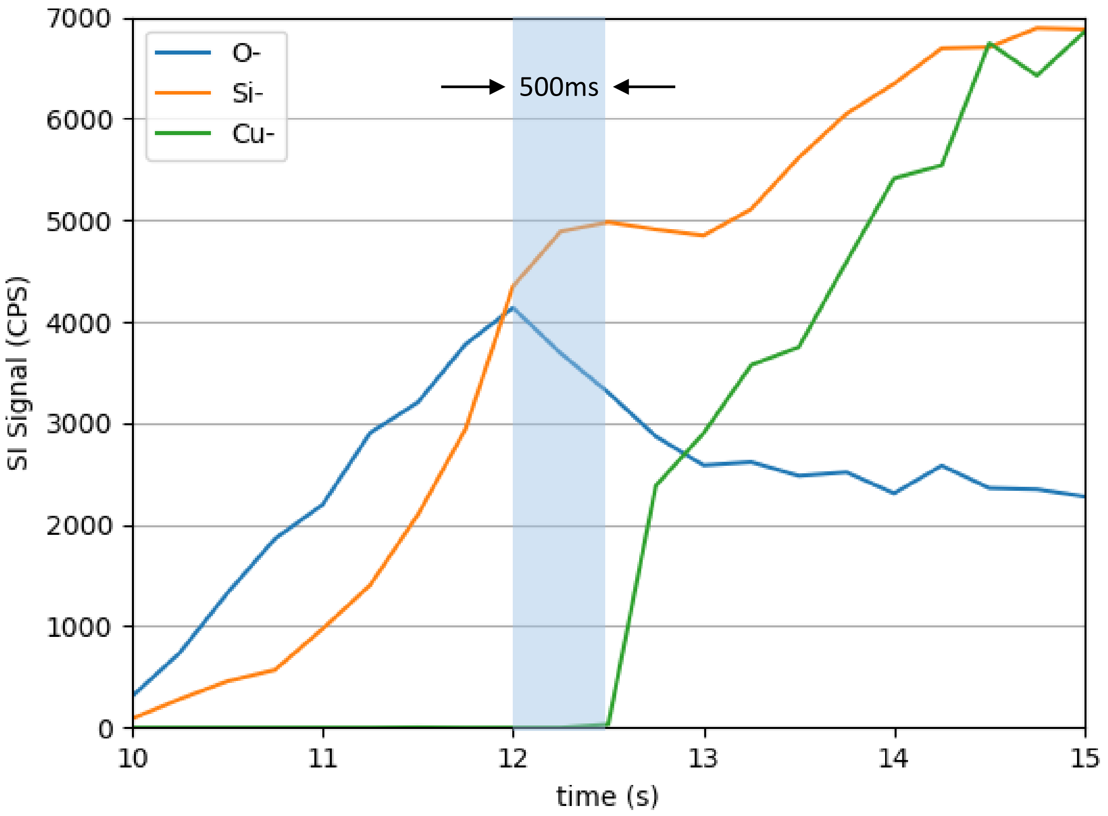
100nm Square Mill Box, 5 pA, 16 kV, 54 mm WD, Negative SIs



- Initial signal levels rise as cesium concentration builds to enhance the SI yield by ~3 orders of magnitude
- All 3 channels show abrupt changes when crossing the SiO2-Cu interface
- Si- and O- exhibit clear features just prior to Cu- appearance
  - Could be used as advance "predictors" of the endpoint, eg indicate when to change milling parameters like dose rate to optimize machining at the endpoint, eliminate reaction time error
- Cu- signal
  - Exhibits extremely high contrast between off and on
  - Changes from 0 to 2500 CPS over 250ms == minimum time step from integration

Zoom on SIMS Signal at interface

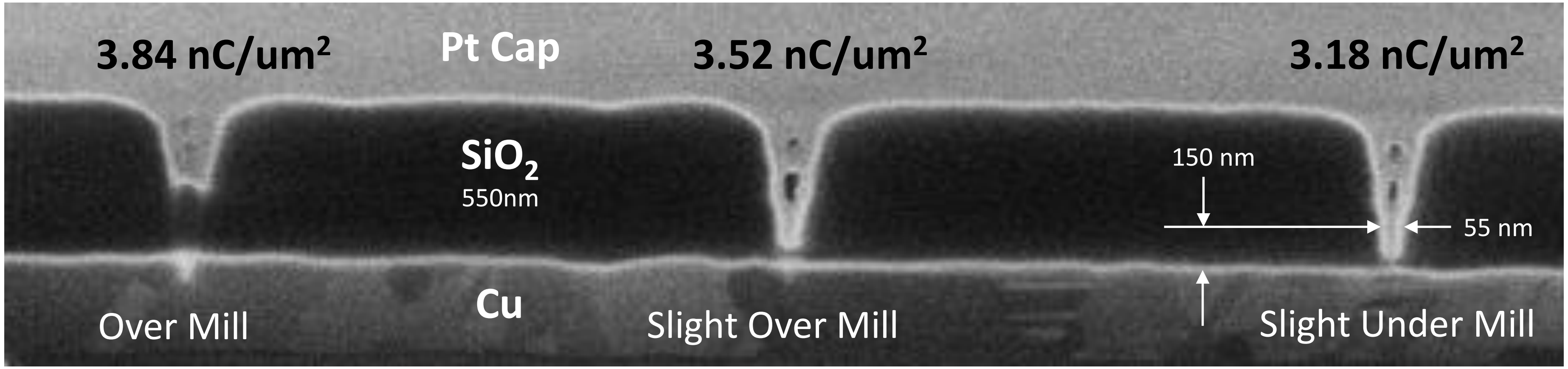
100nm Square Mill Box, 5 pA, 16 kV, 54 mm WD, Negative SIs



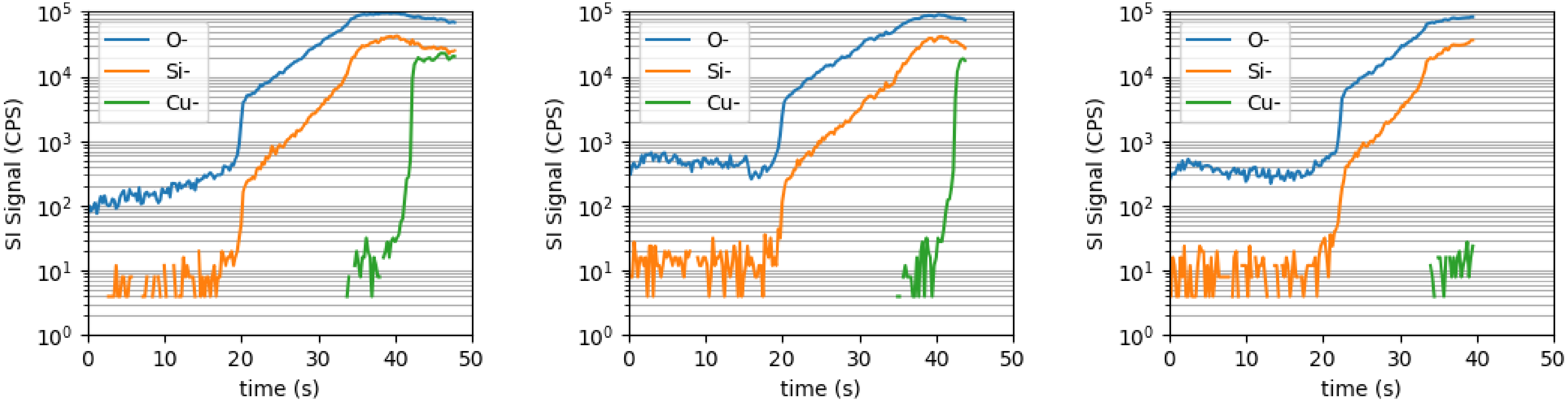
- Si- and O- signals as endpoint predictors
  - O- signal abruptly peaks and drops to 80% of peak value in 500ms prior to Cu- signal
  - Si- signal levels off (second derivative changes sign) over the same interval
- Cu- signal
  - Exhibits extremely high contrast between off and on
  - Rises from ~0 to 2500 CPS over 250ms == minimum time step from integration

Section View of 50nm Rectangular Vias

50nm x 500nm Mill box, 2.0 pA, 16 kV, 54 mm WD



SIMS signals Predictive of Milling Results

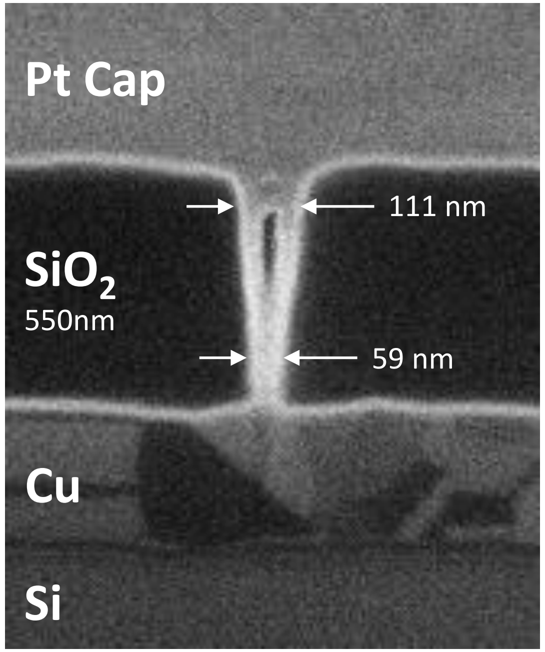


Signal Level Remains High Despite Higher Aspect Ratio

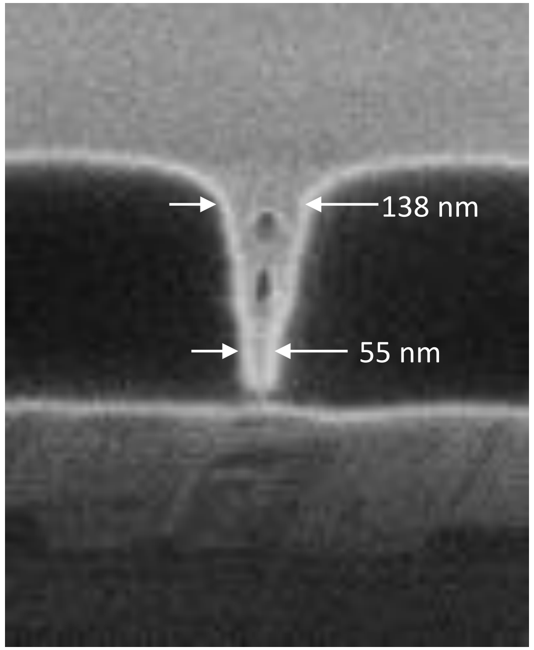
Section View of 50nm Rectangular Vias

50nm x 500nm Mill Box, 2.0 pA, 16 kV

Milled at Short Working Distance (17 mm)

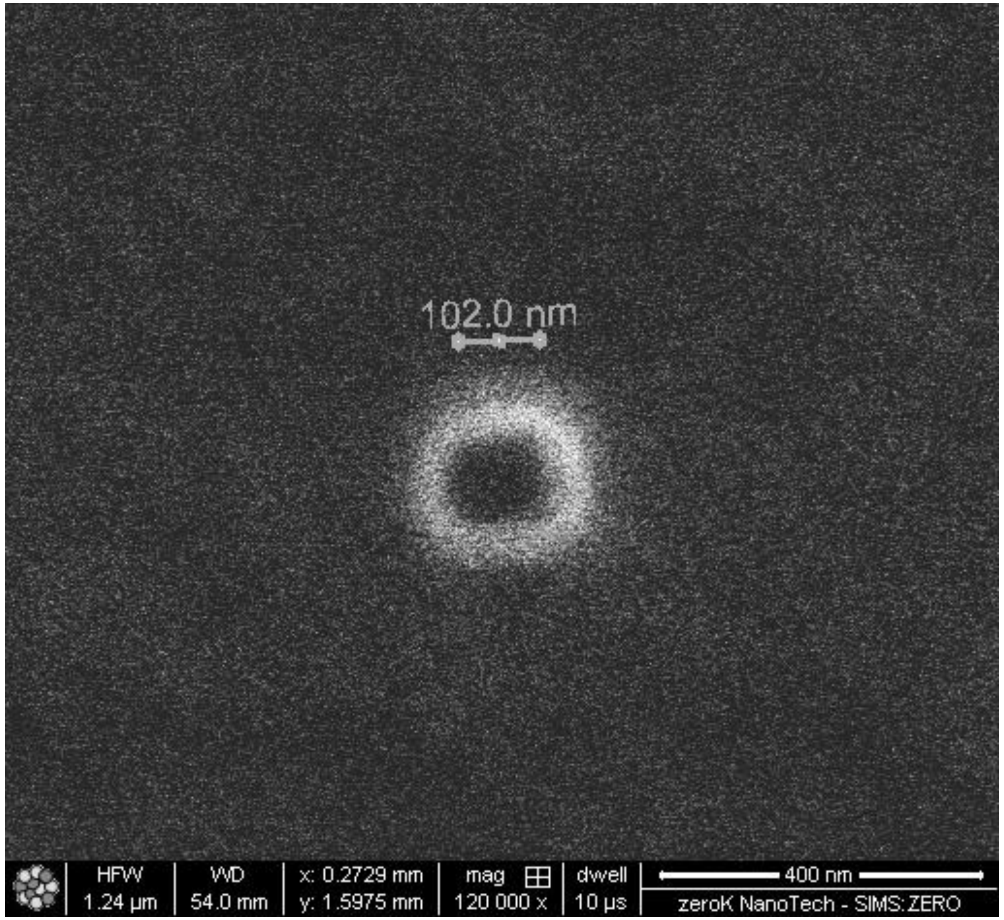


Milled at Long Working Distance(54 mm)



SE image of 100nm Via

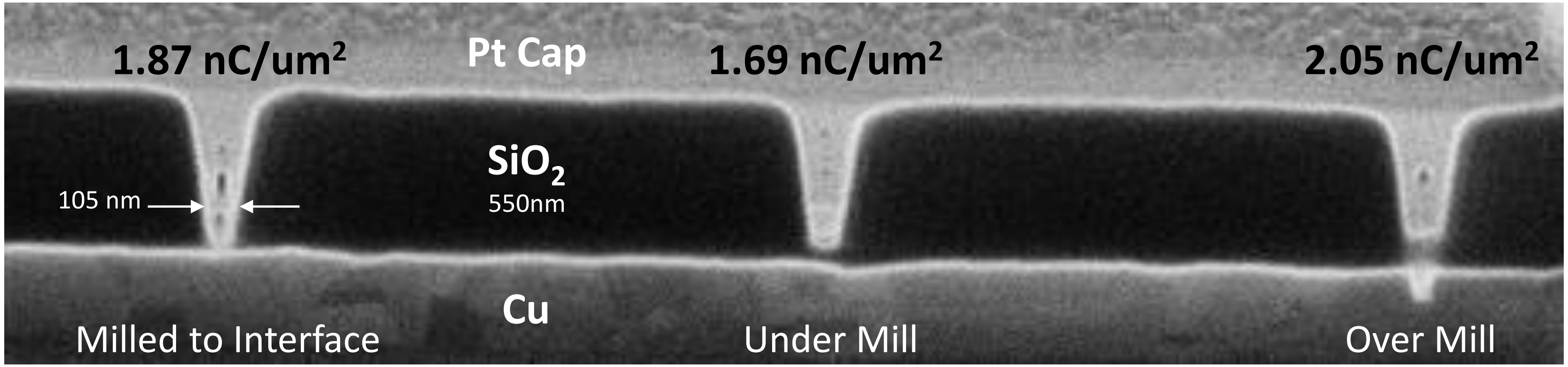
100nm Square Mill Box, 5 pA, 16 kV, 54 mm WD



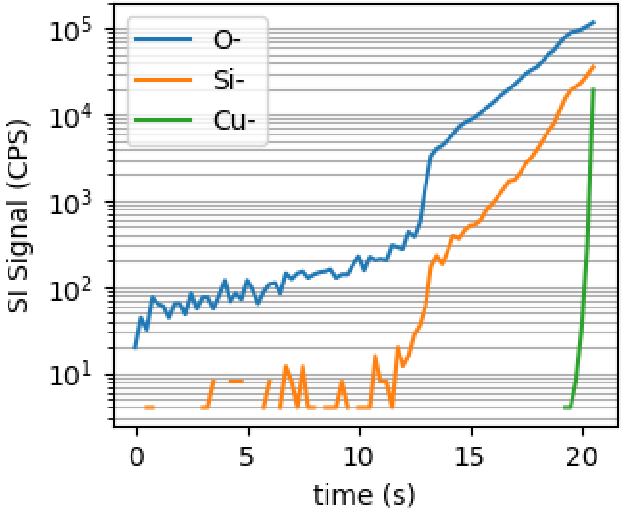
- 100 nm box with reasonable fidelity
- Imaged after machining via with SIMS analysis
- Working Distance = 54mm is long
- Optimized system configuration with 20mm WD possible

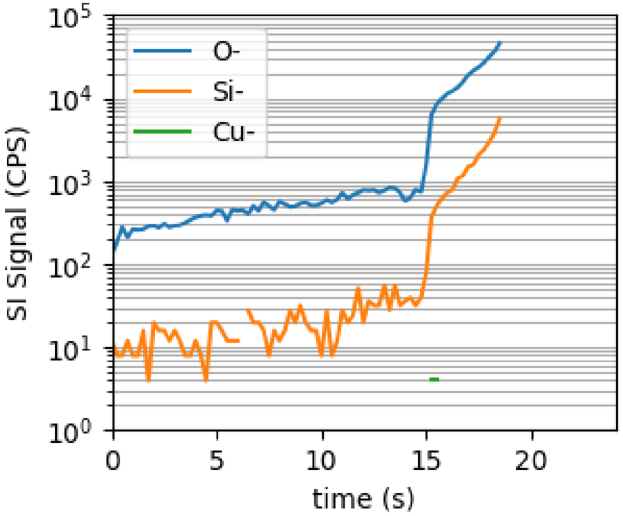
Section View of 100nm Rectangular Vias

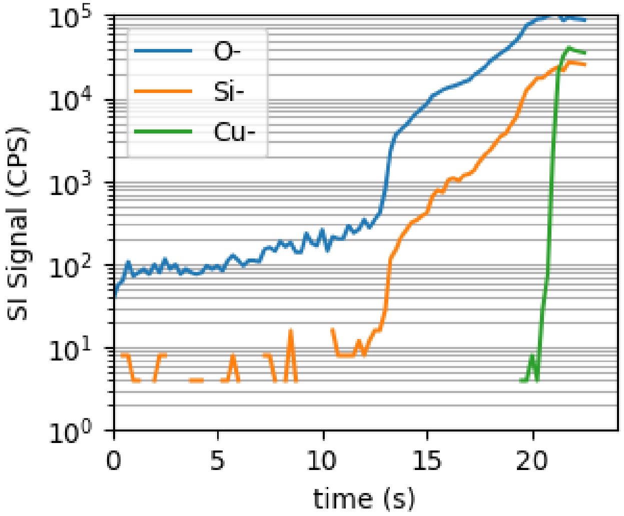
100nm x 500nm Mill Box, 4.5 pA, 16 kV, 54 mm WD



SIMS signals Predictive of Milling Results







High SIMS Signal Levels