Ion Microscopy, Machining, and Elemental Analysis with the Cesium Low Temperature Ion Source (LoTIS)

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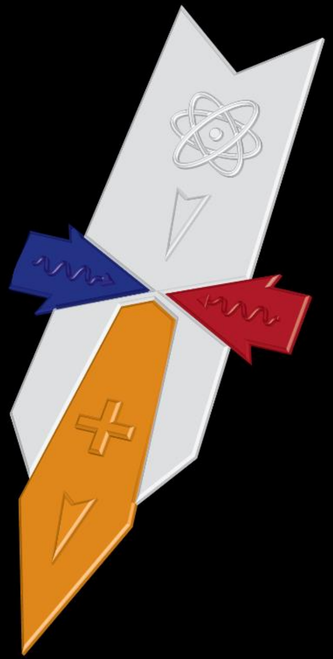
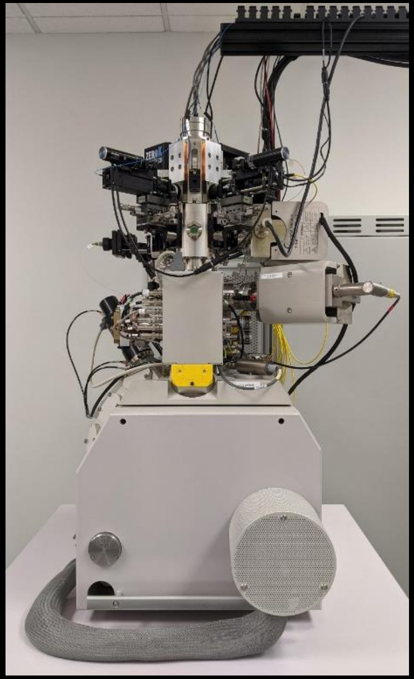
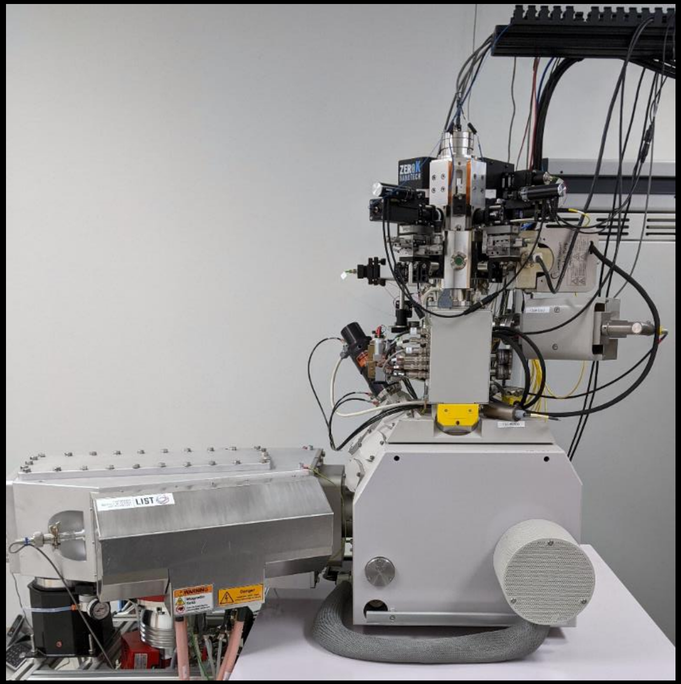
Technology and Applications

ZER0K







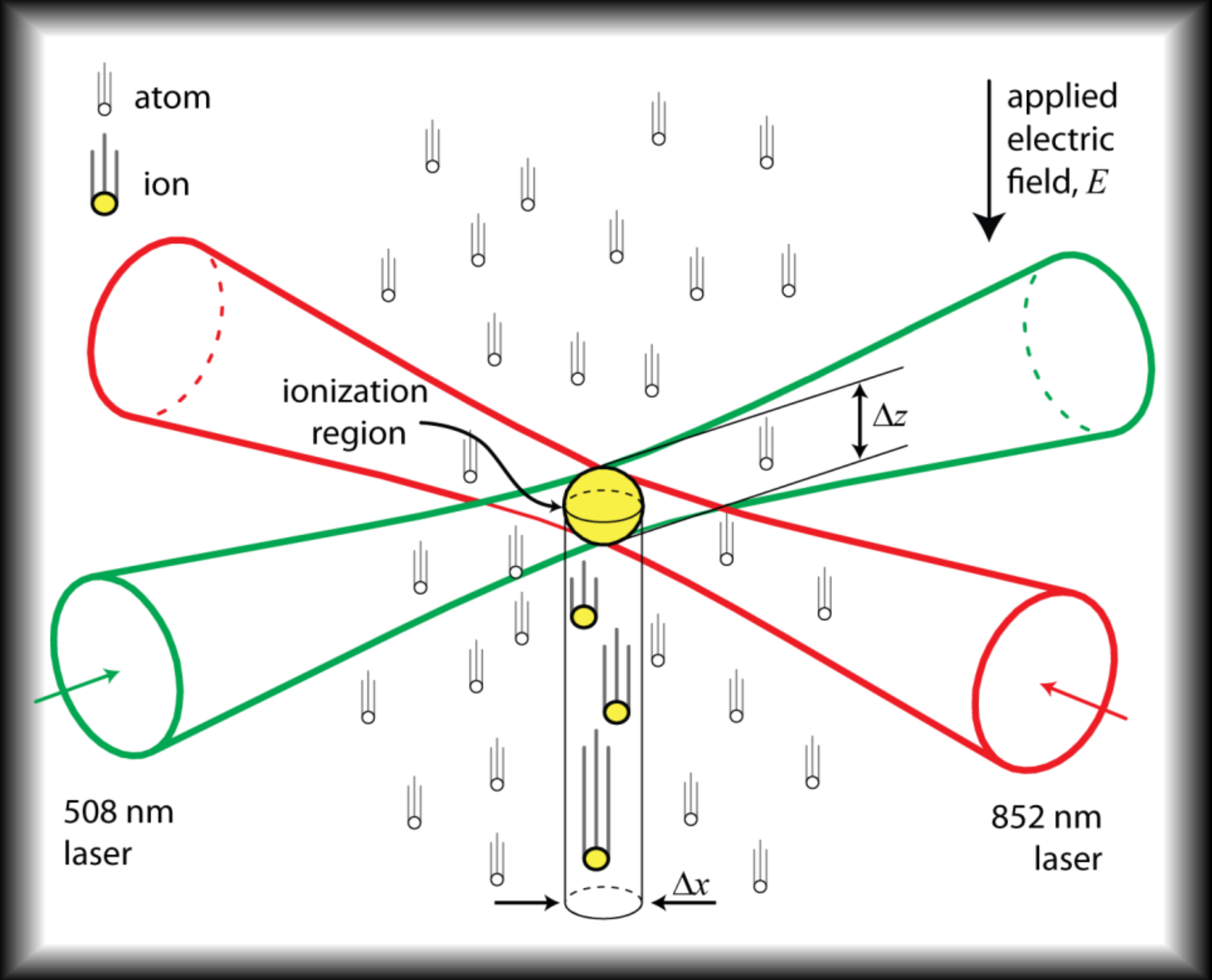
Cs$^+$ LoTIS	FIB:ZERO	SIMS:ZERO

• Low Temperature Ion Source
  • Laser-cooling + Photoionization
• Heavy ion nanomachining
• Small spot sizes
• Excellent resolution at low energy (~2 nm resolution at 1 pA, 16 kV)
• 1 pA - 10 nA
	• LoTIS + FIB
	• Comparable to standard Ga$^+$ FIB, with 2x higher resolution at low beam currents
	• Compatible with normal peripherals, gas chemistries etc..
		• FIB:ZERO with SIMS
		  • Analysis of secondary ions in a mass spectrometer
		• Best for elemental-compositional analysis
		• Collab. with Luxembourg Institute of Science and Technology (LIST)

How does LoTIS work?

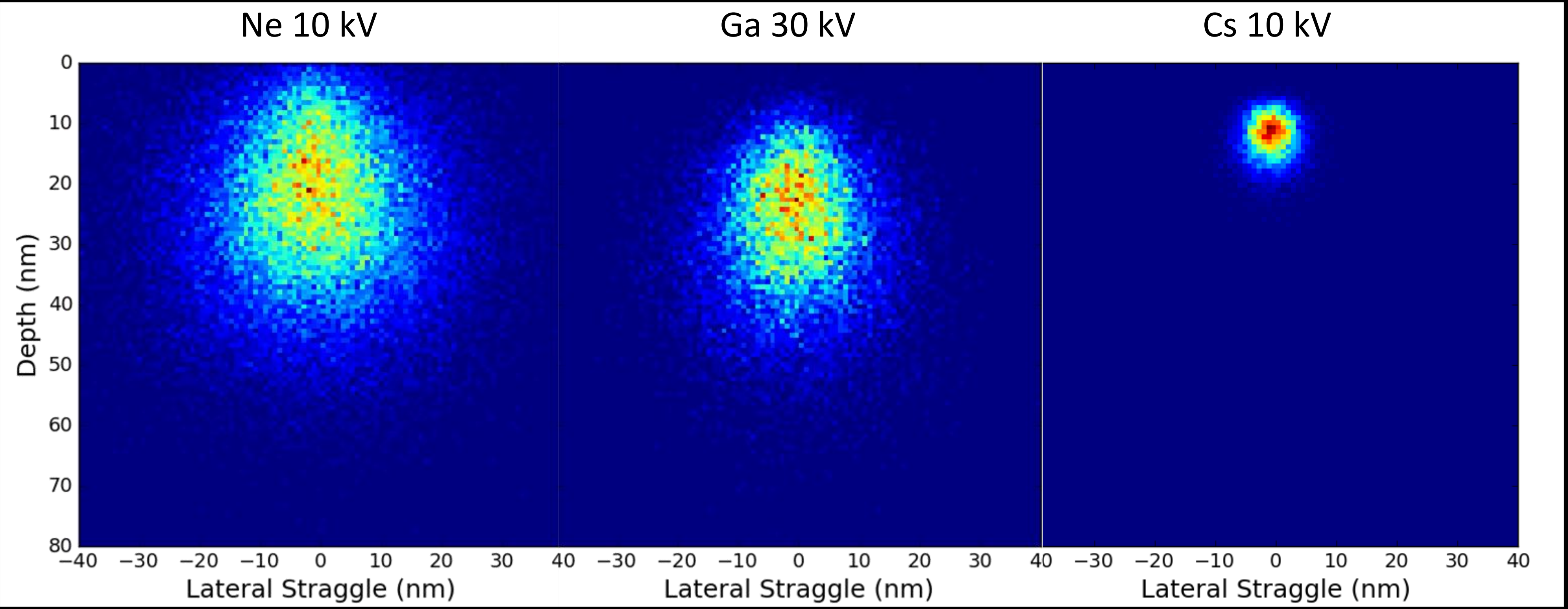
Ions are created in a laser-cooled atomic beam as it flows through the intersection of photoionizing laser beams

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



Implant Depth Comparisons (SRIM simulation)

- Comparison of three scenarios where spot size might be ‘good enough’
- Cs has significantly reduced straggle and implant depth

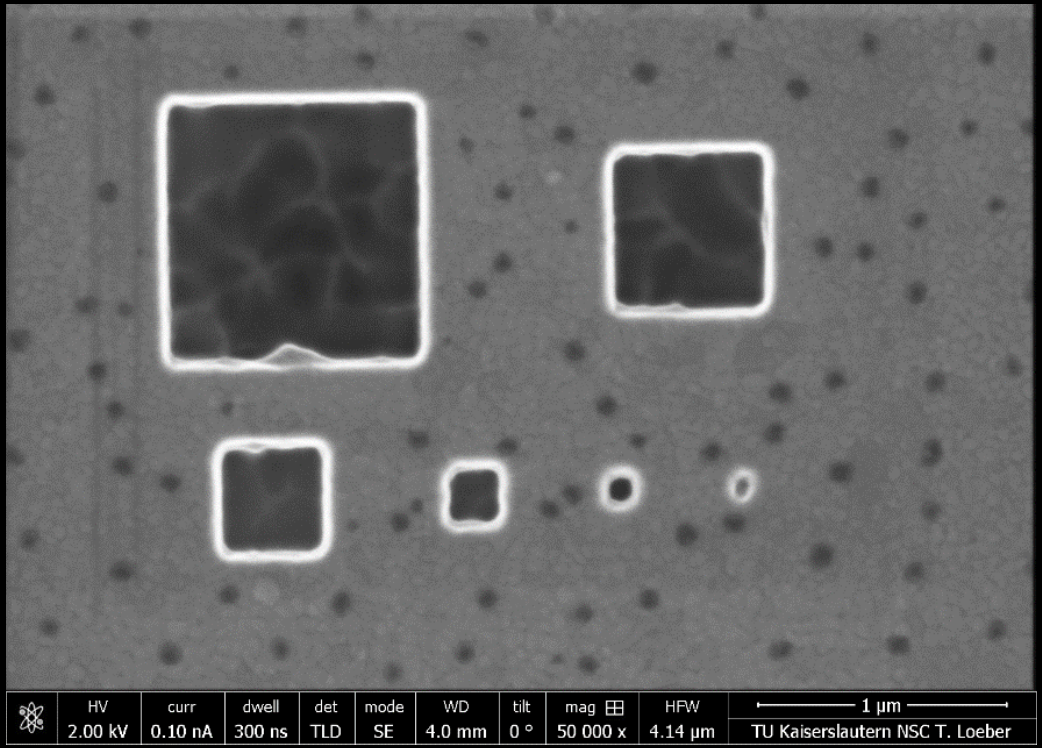


FIB:ZERO

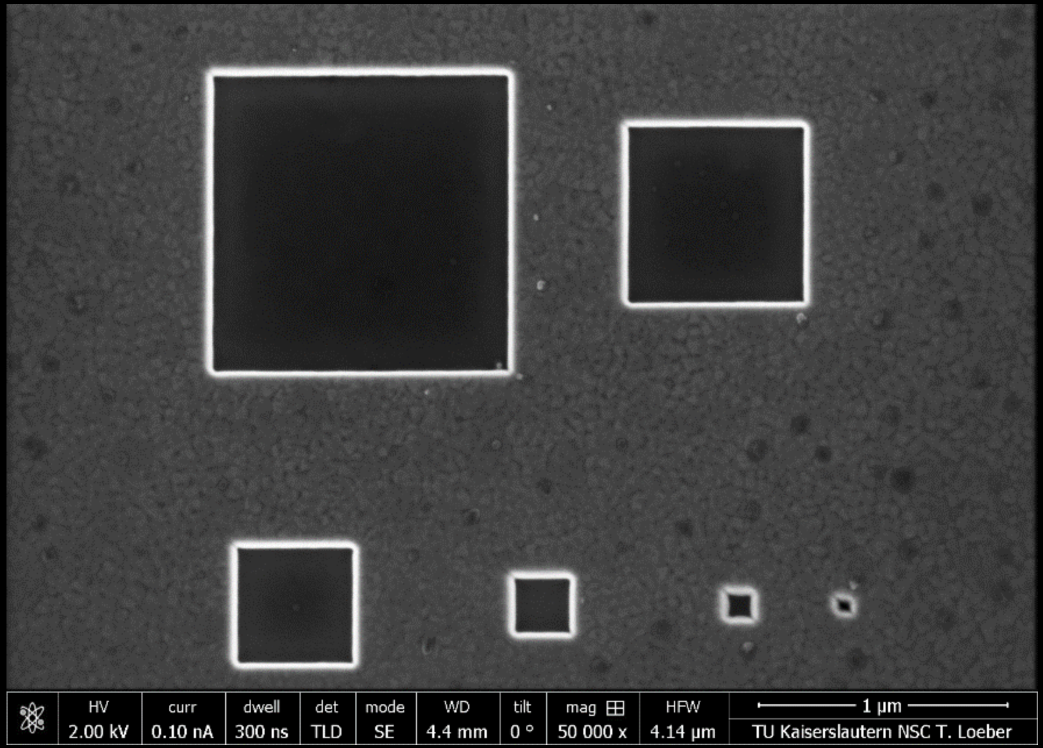
Milling Accuracy: 110 nm Au on Si

→ LoTIS provides clean mill boxes with sharp corners

Milled with Ga^+ LMIS



Milled with Cs^+ LoTIS



- squares with 1, 0.6, 0.4, 0.2, 0.1 and 0.05 µm length
- milled through the Au layer
- milling time Ga and Cs almost the same

FIB:ZERO Milling Rates

ZEROK

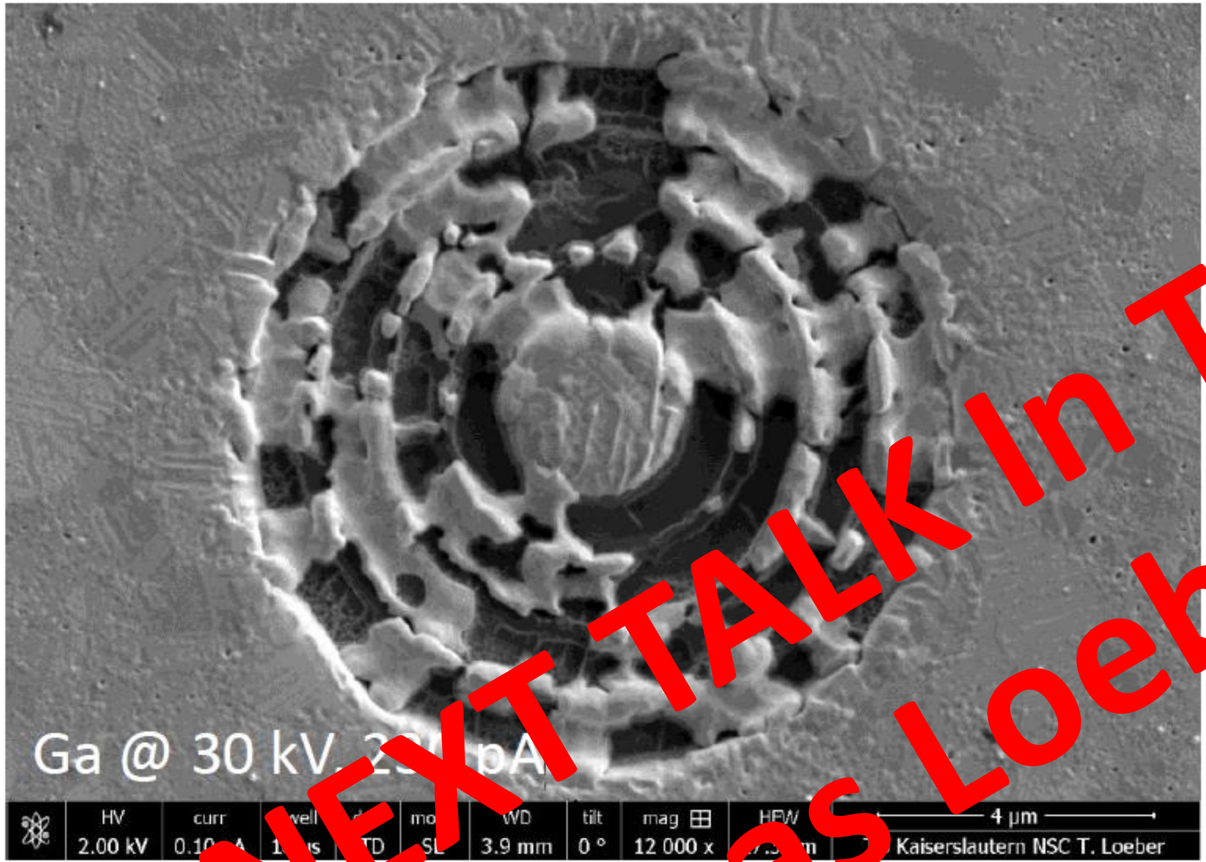
Milling rate of 10 kV Cs^+ FIB:ZERO about 15% lower than 30 kV Ga^+ for Si

Cs^+ LoTIS milling rates 90% higher than Ne^+ (and **much** higher than He^+)

Ne 10 kV	Ga 30 kV	Cs 10 kV
1.00-1.38 at/ion	2.20-2.40 at/ion	1.90-2.15 at/ion

Milling in silver

NEXT TALK In THIS SESSION:
Thomas Loeber



- plasmonic structures
- Ga: inhomogeneous milling in polycrystalline silver
- Cs: significant better rings

Existing Elemental Analysis Techniques and a New Solution

EDX/EELS

- Long sample-prep times
- 3D analysis infeasible
- Low-Z elements challenging

Site-Specific SIMS

- Resolution limited to ~50 nm with high yield (CAMECA NanoSIMS), or
- Can get a high resolution FIB (Ga, He, Ne) with a time-of-flight SIMS analyzer. But low secondary ion yields from these beams usually results in poor lateral resolution. Additionally, time-of-flight analyzers necessitate **long** acquisition times.

These points are addressable by

SIMS:ZERO

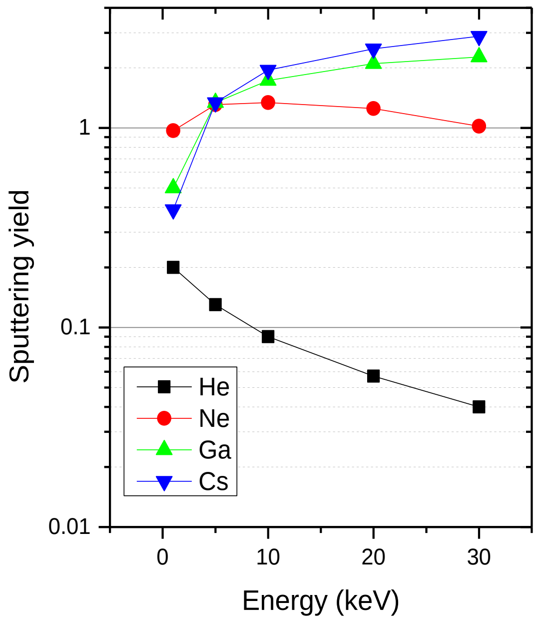
- Few-nanometer resolution (slide 21)
- High secondary ion yield (slides 23,24)
- Integrated sample-prep and analysis capability (slides 25-31)

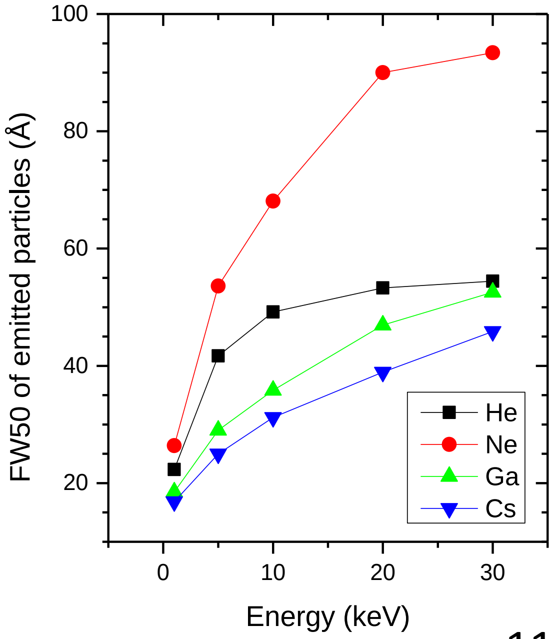
Primary Ion Species Matters

Differing Sputter Rates
→ Analysis Time

Differing interaction Volumes
→ Resolution

Differing Yields
→ Sensitivity Floor, SNR

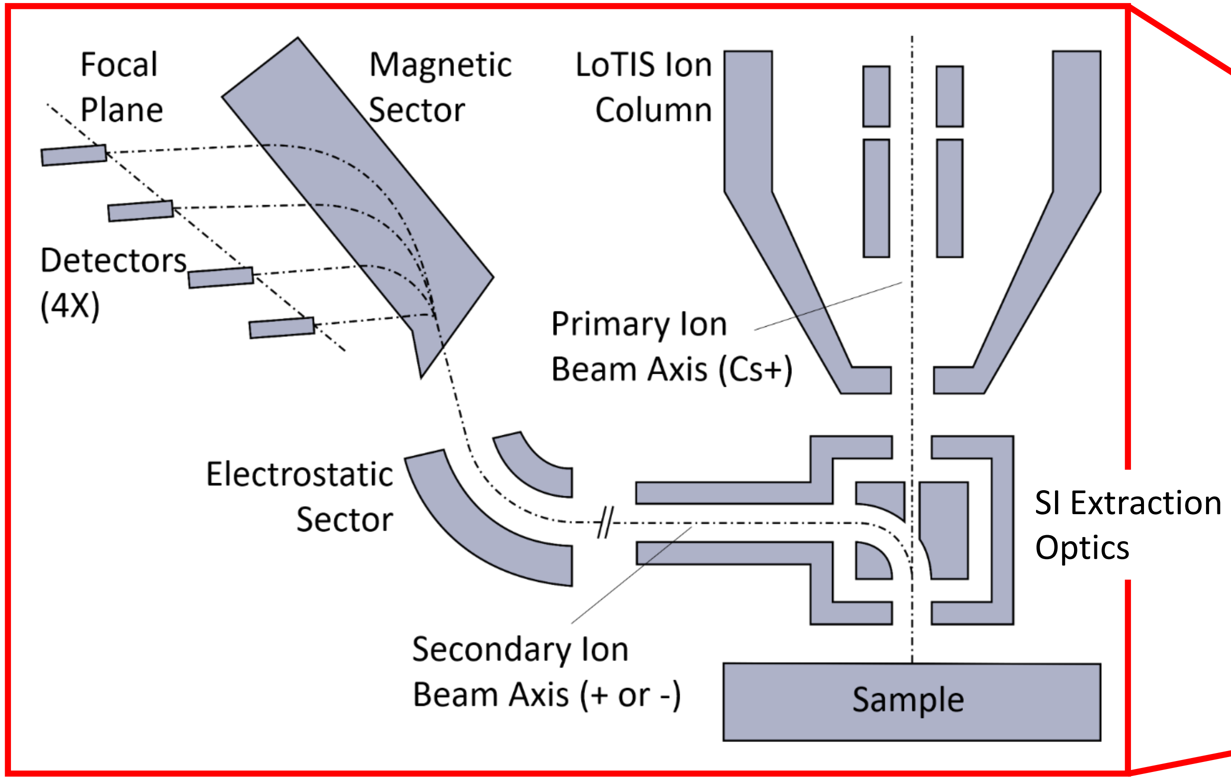


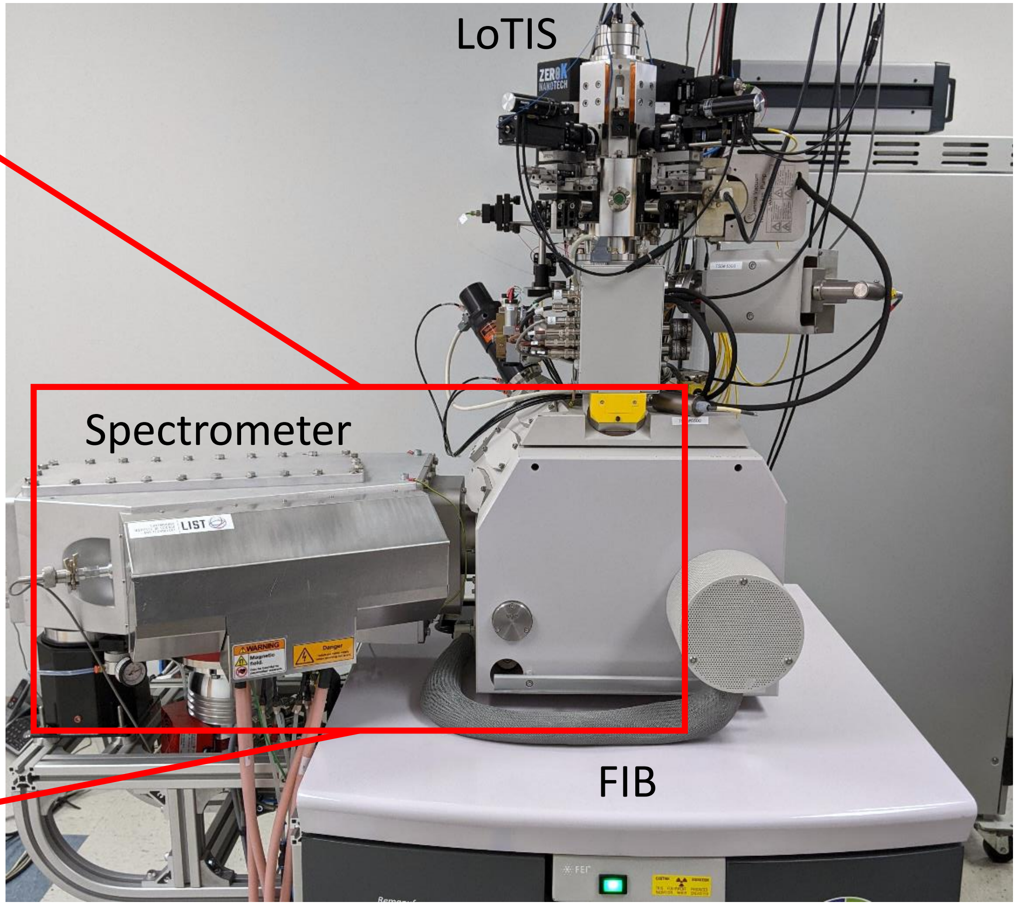


SIMS:ZERO

Instrument Overview

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



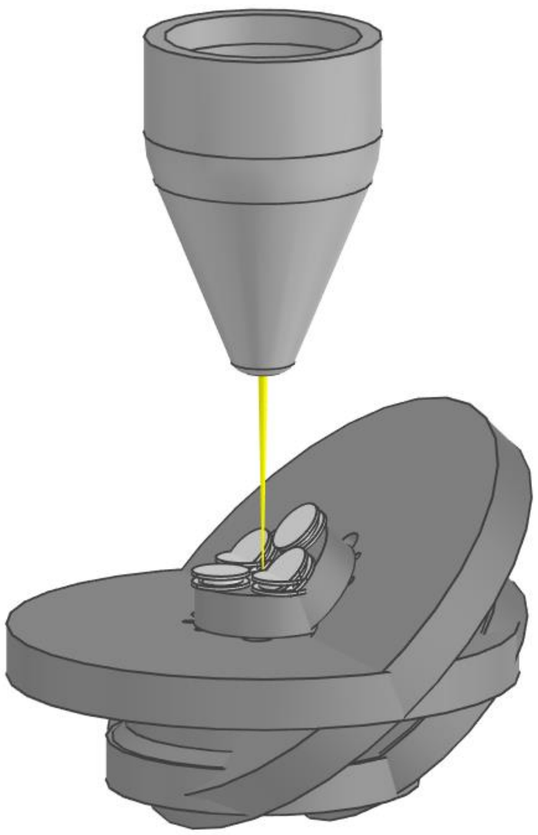


- FIB online 6/2020
- SIMS online 5/2021

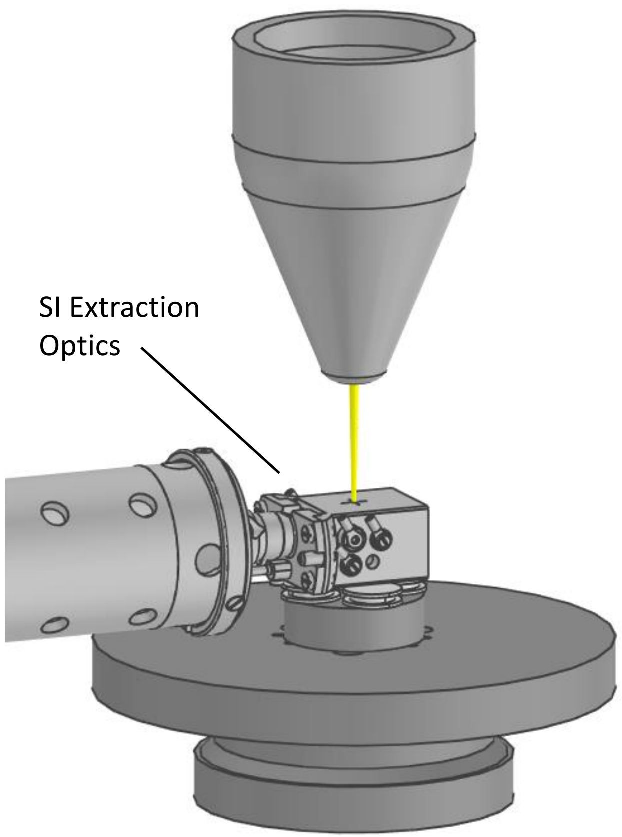
FIB / SIMS Combination

Sample Prep, Nanofabrication / Analysis, 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

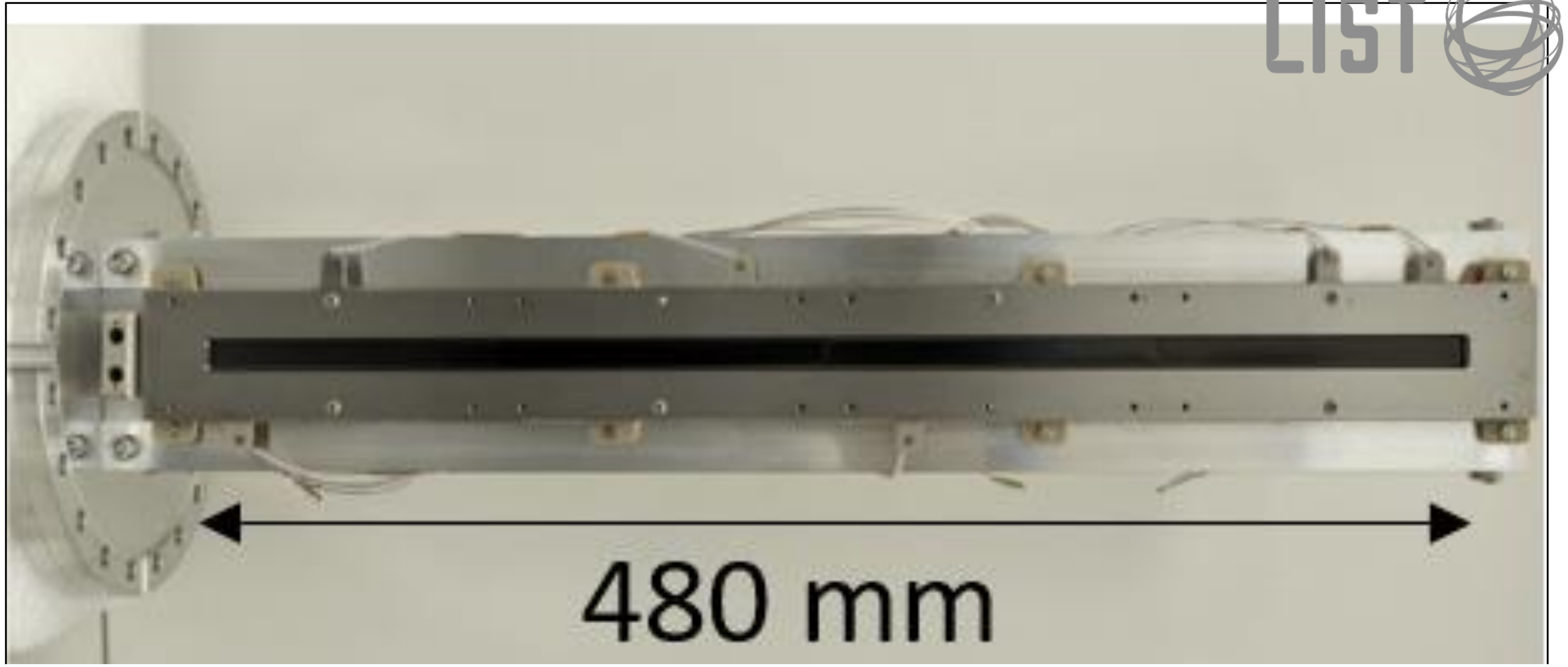
SIMS Mode (SIMS Extraction Optics Inserted)

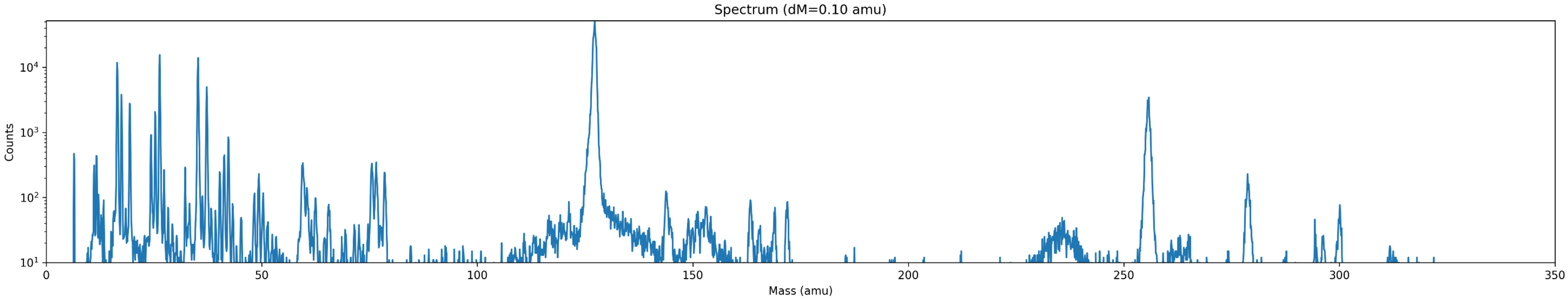
- Highest spatial resolution SIMS imaging
  - $\sigma = 6\ \mathrm{nm}$ demonstrated
- Mass resolution $M/\Delta M = 400$
- Mass range up to 300 amu
- High secondary ion throughput (~40% simulated)
- 4-Channel Detector Standard (Continuous Focal Plane Detector available)

Continuous Detector

- Sample the entire mass spectrum for every pixel (e.g. 6-350 amu)
- Collect the entire spectrum (as in ToF SIMS), but without painfully long acquisition times

- 480 mm micro-channel plate
- Delay lines, discriminators allow for pulse counting along the full length

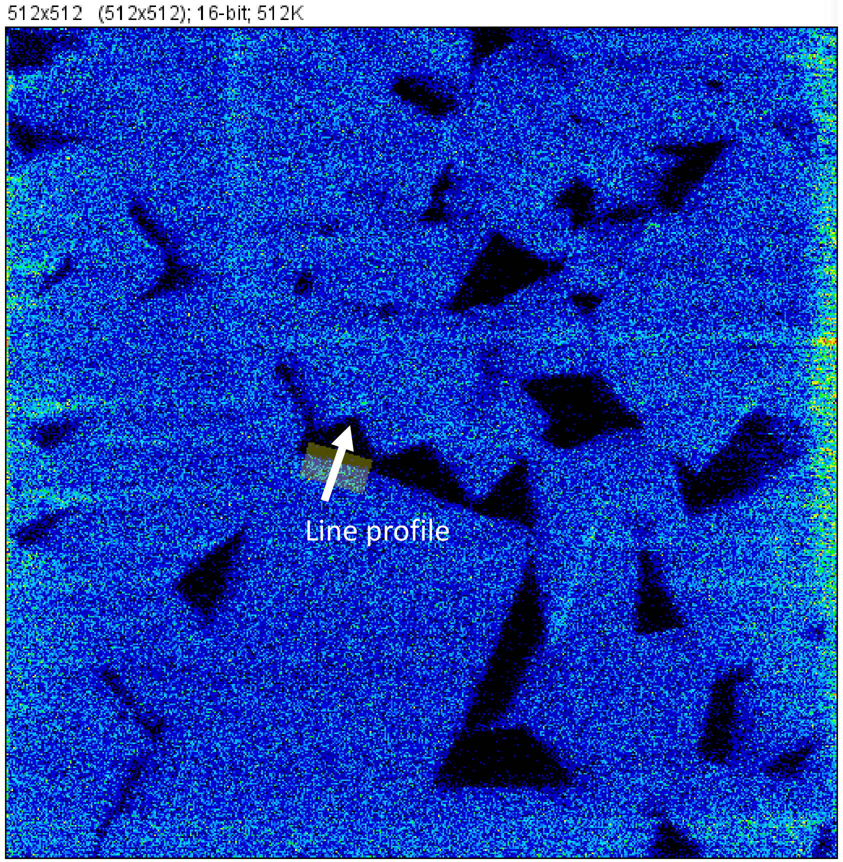




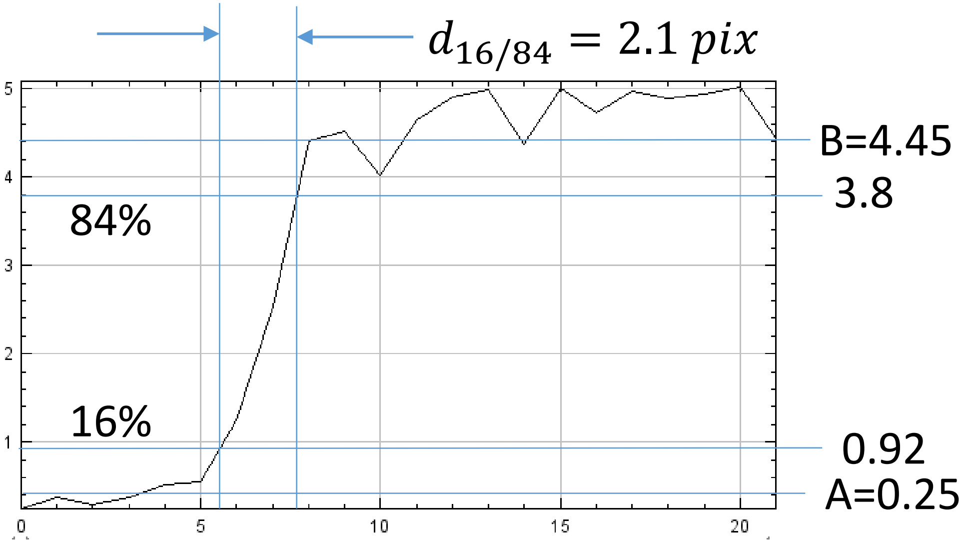
SIMS:ZERO Resolution

Tungsten Carbide

- **SIMS:ZERO can provide higher resolution SIMS scans than any other instrument**
- SIMS resolution is a function of abundance, yield, and spot size
- SIMS:ZERO has a focused ion beam with <3 nm spot size, and since it's Cs^+ we achieve high yields for many materials
- In samples with high abundances, resolution at near the physical limits of SIMS can be achieved (see right)



Multi_WC_2105121624015_CH1.TIF



$$d_{16/84} = 2.97\ \mu m * \frac{2.1}{512} = 12.2\ nm$$

$$\boldsymbol{\sigma = 6.1nm}\ \textbf{(!)}$$

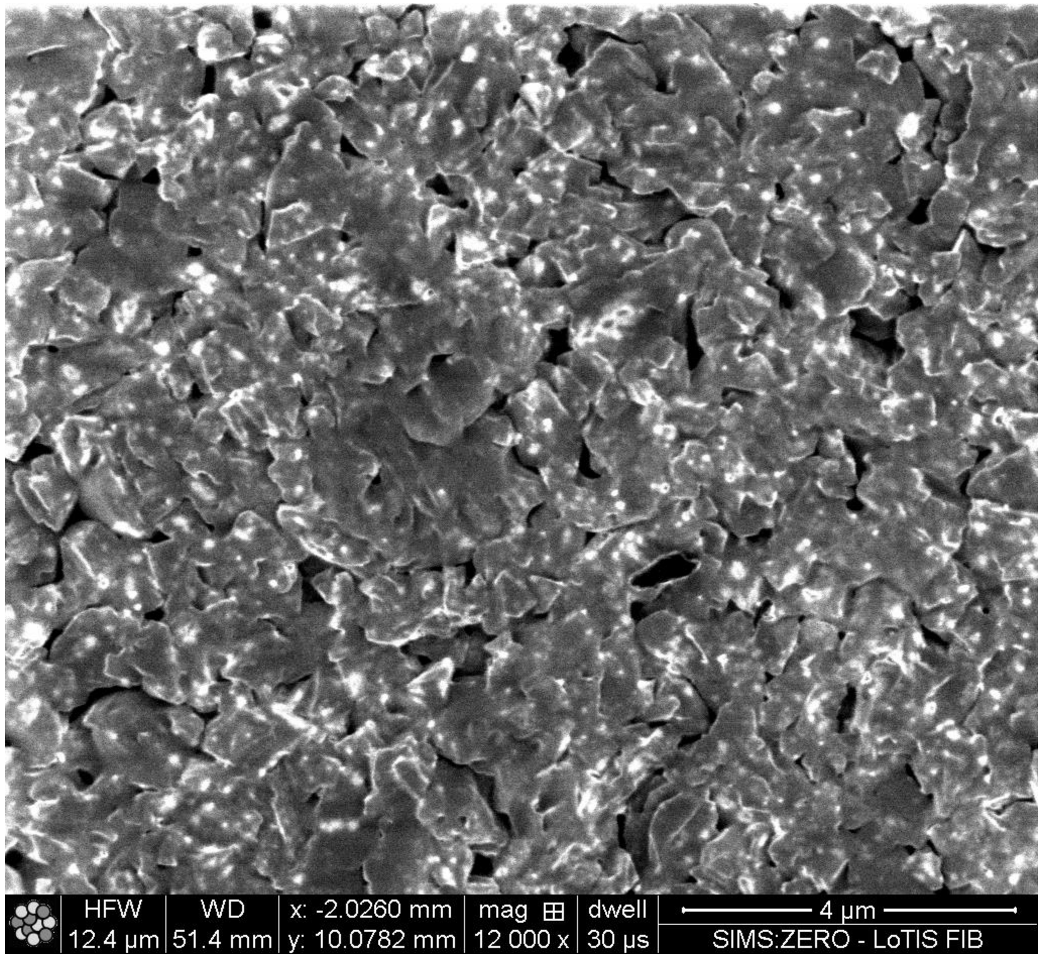
Working Distance = 51.6mm
272s acquisition time.

Negative Ions

Date	05/12/2021
Sample	WC (184 amu)
FOV (um)	2.97um
I (pA)	2.5
U (kV)	16

SIMS Analysis Example

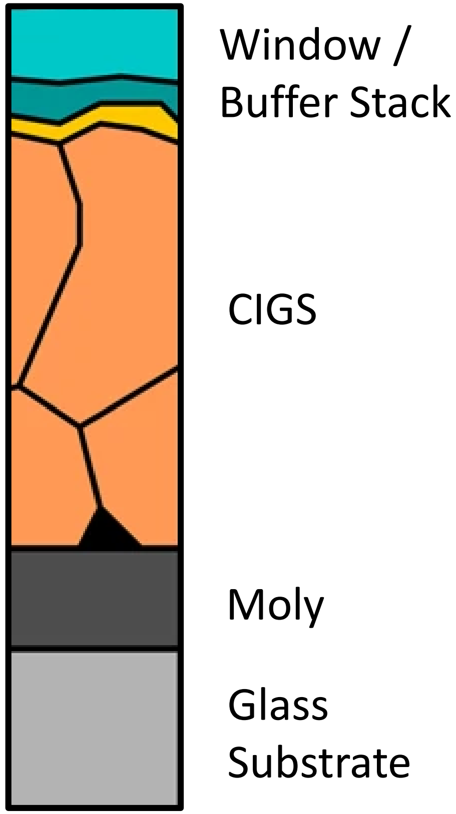
CIGS $Cu(In,Ga)Se_2$ – Rb doped



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

Summary

- CIGS is a solar cell absorber material
  - Rubidium doping increases conversion efficiency
- SIMS spectra clearly show all CIGS elements:
  - Cu, In, Ga, Rb in Positive Mode
  - Se in Negative Mode
- Secondary ion imaging channels show distribution of elements in sample, eg Rb dopants concentrated in grain boundaries
- Secondary electron images provide complementary information at high resolution
- Section view technique provides superior SIMS data

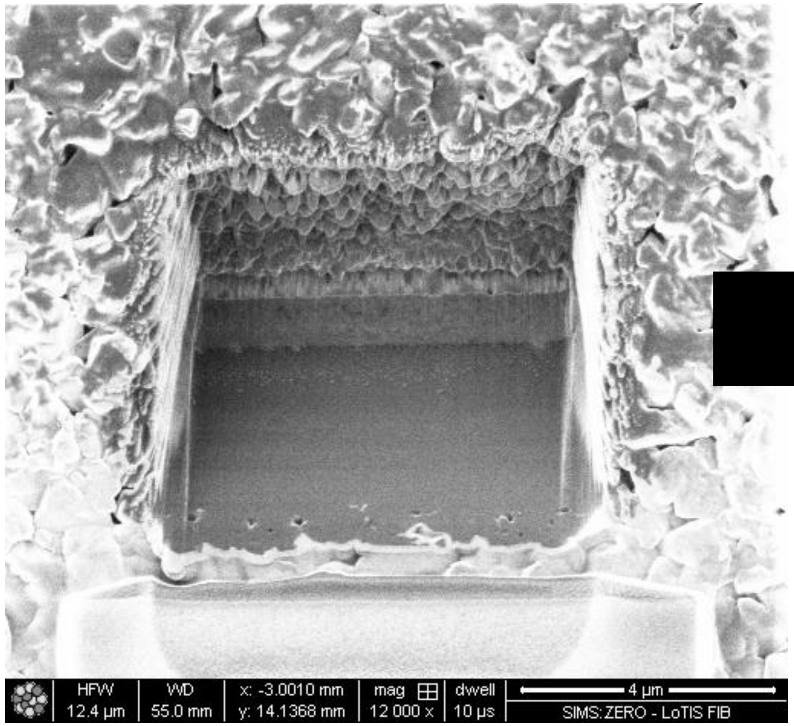


Werner, et al. *Scientific Reports* **volume 10**, 7530 (2020)

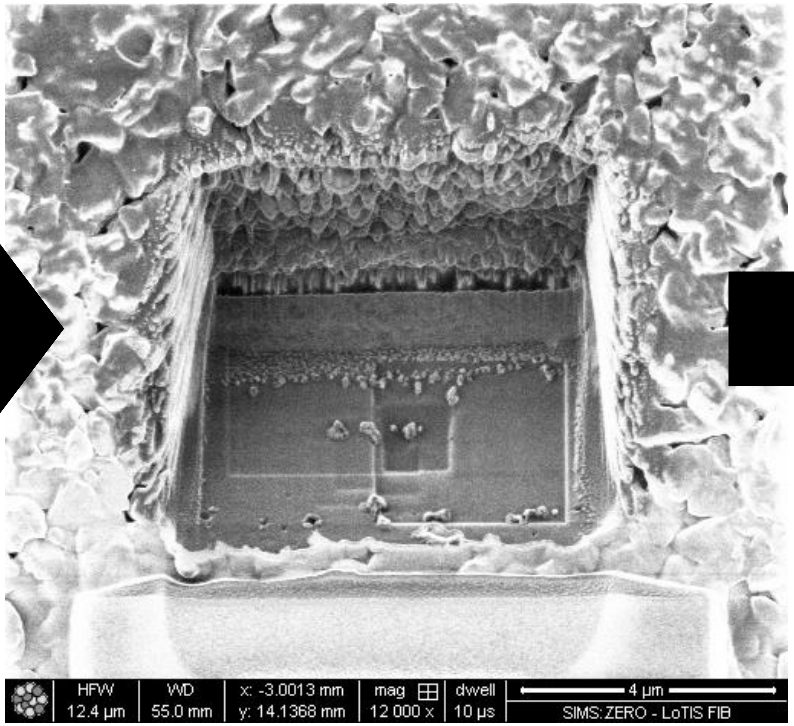
CIGS $Cu(In,Ga)Se_2$ – Rb doped

Serial Sectioning / Imaging / Polishing Work-Flow

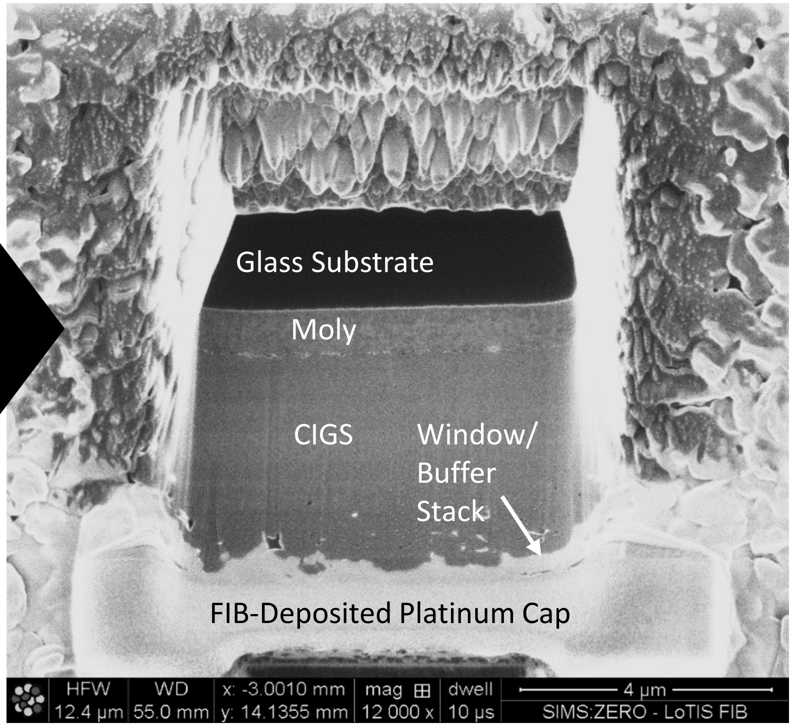
SE Images



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

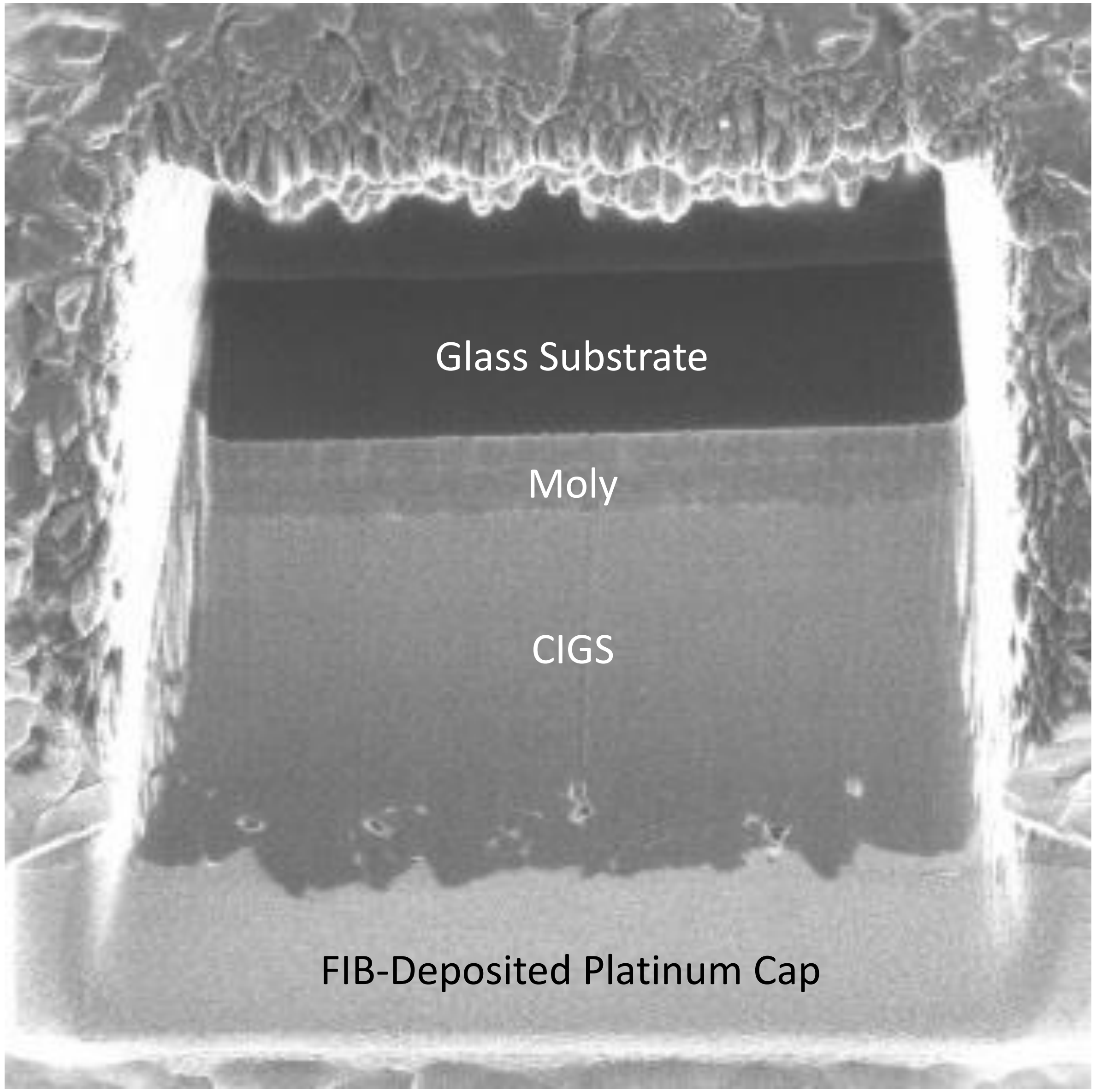


After SIMS Imaging, section face develops topography which obscures elemental contrast / distribution information



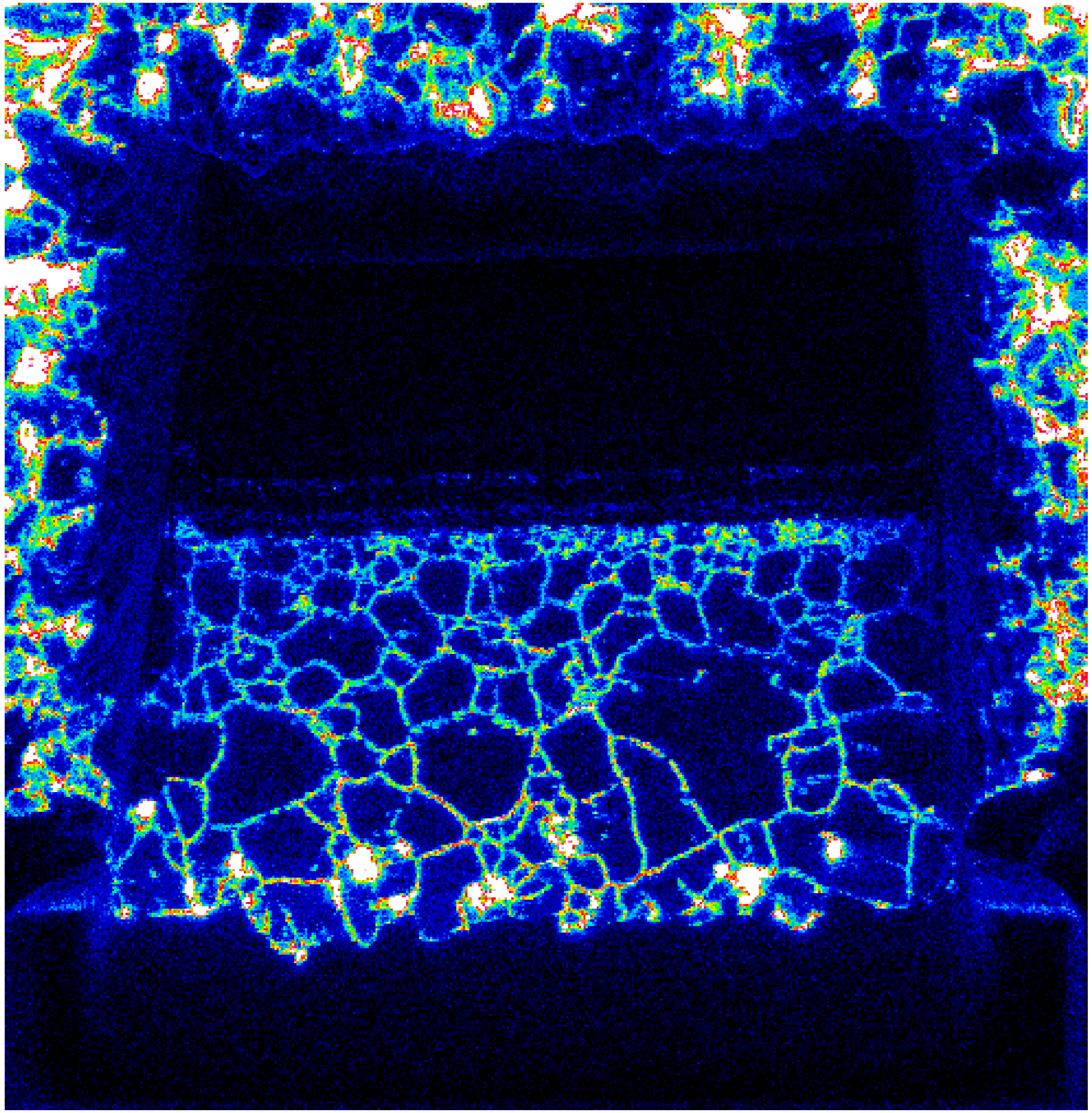
Section face after cleanup mill. Ready for SIMS on next layer

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



<u>Secondary electron image</u>

- Sample polished, ready for SIMS
- 9.5 µm FOV

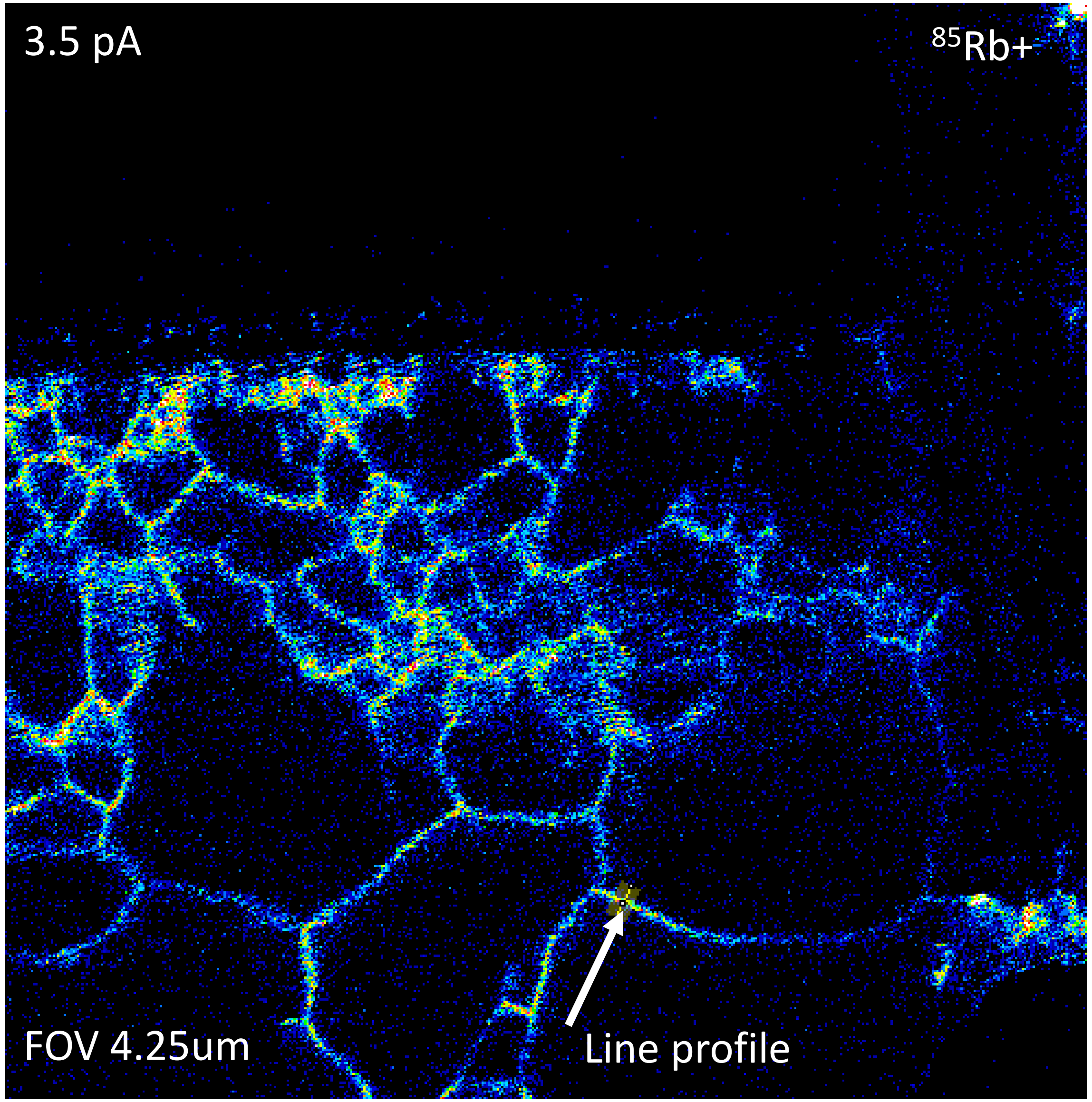


<u>Rb^+ SIMS Image</u>

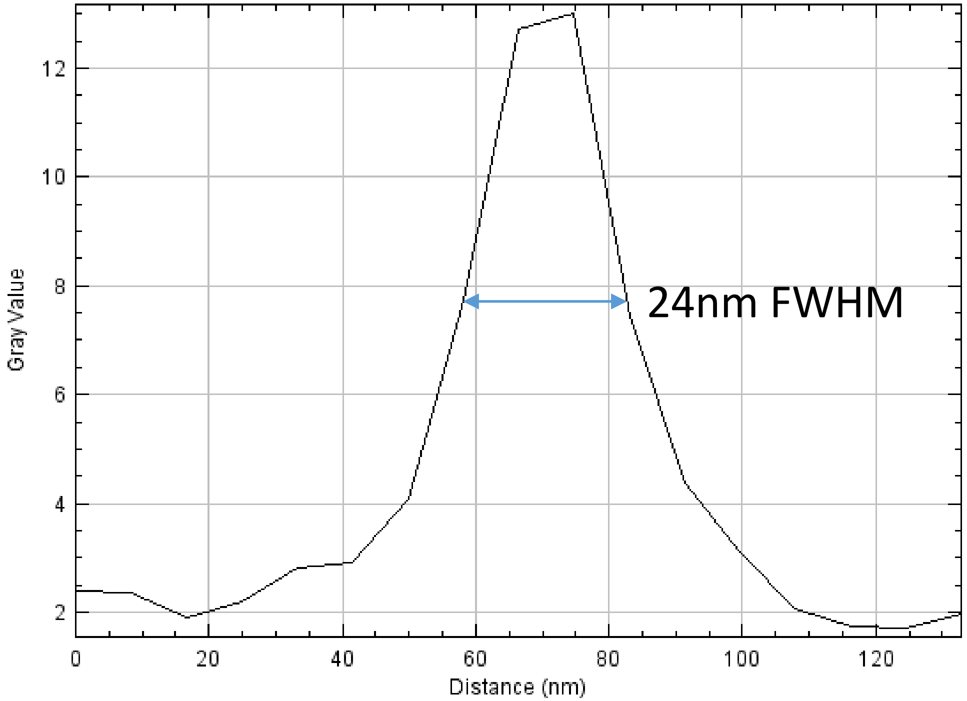
- Rb confined to grain boundaries
- Grains are smaller near the interfaces
- Bilayer structure in the Moly layer

CIGS Cu(In,Ga)Se_2 – Rb doped

Section View – Positive Ions



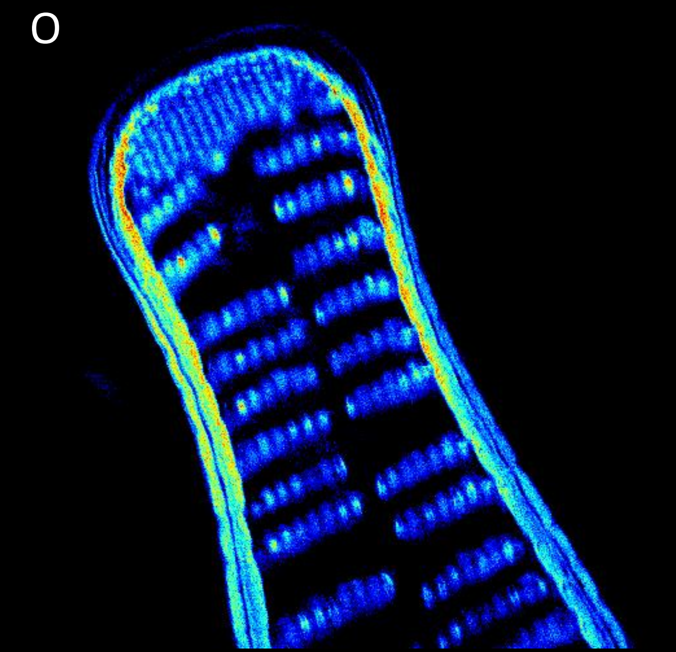
Apparent width of Rubidium signal between grains

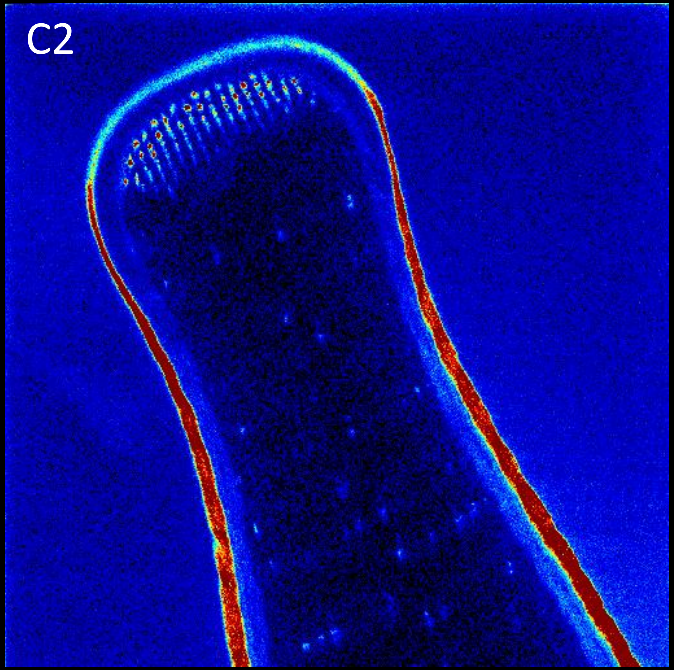


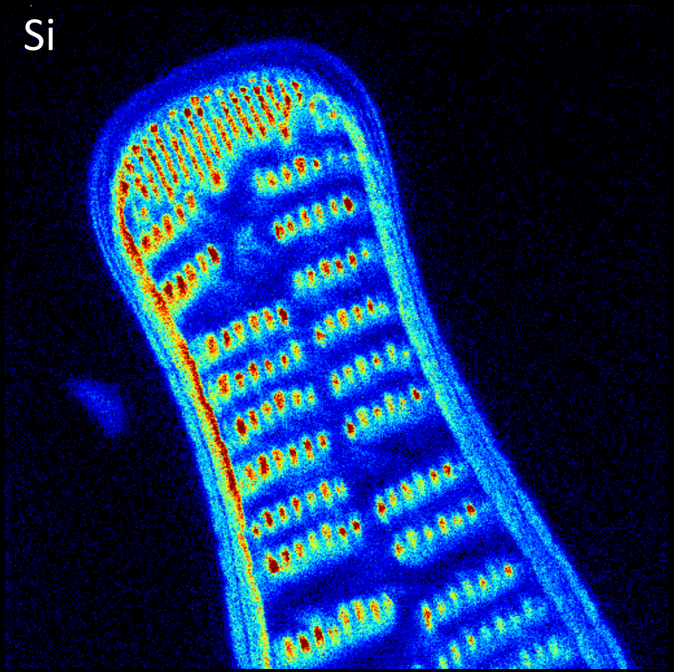
Cs+, 16keV, 3.5pA, 51.6mm WD

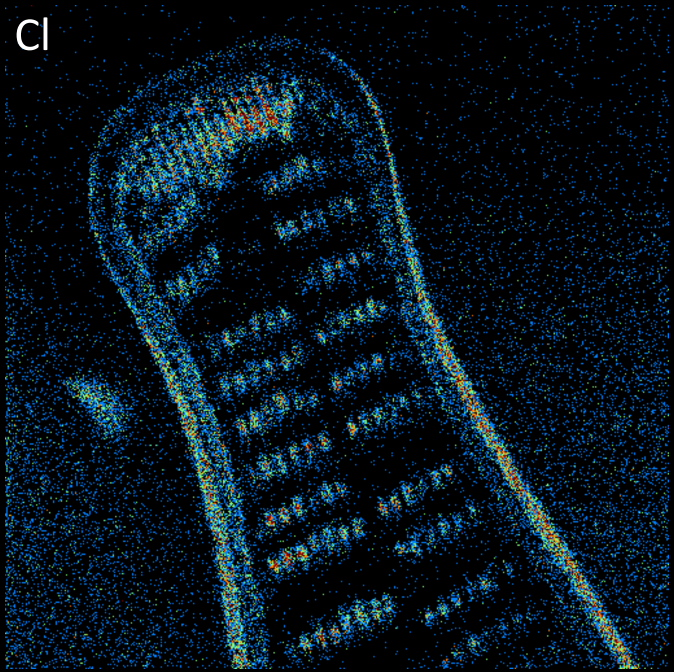
CIGS_Pos_2107151409368.csv

Diatoms
(Silica –shelled algae)
7.5 um FoV



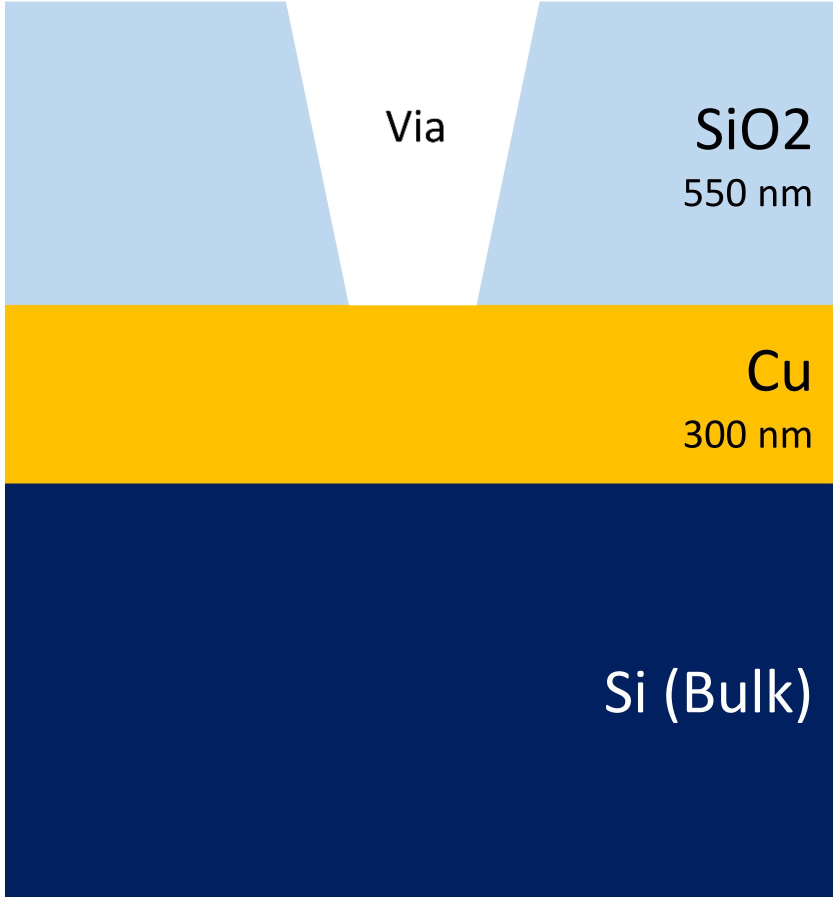






Endpointing Example

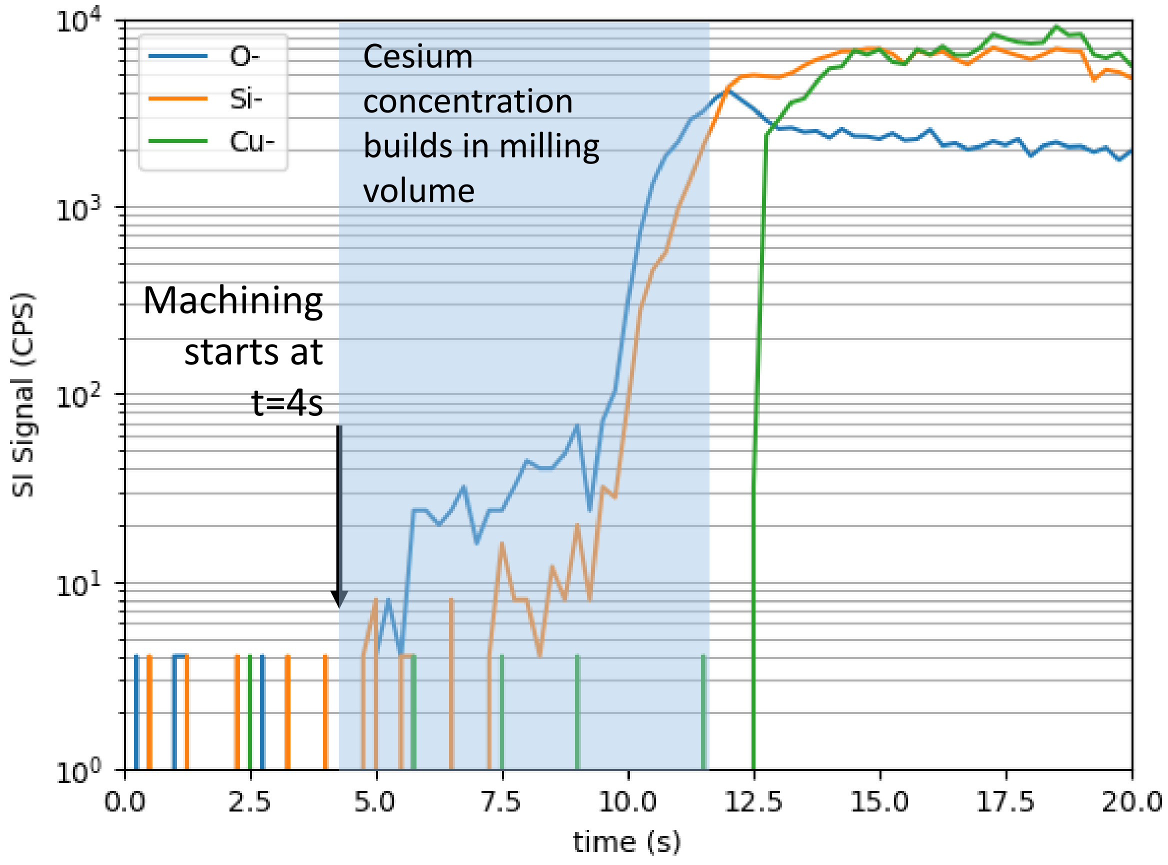
Test sample SiO_2 on Cu



- 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

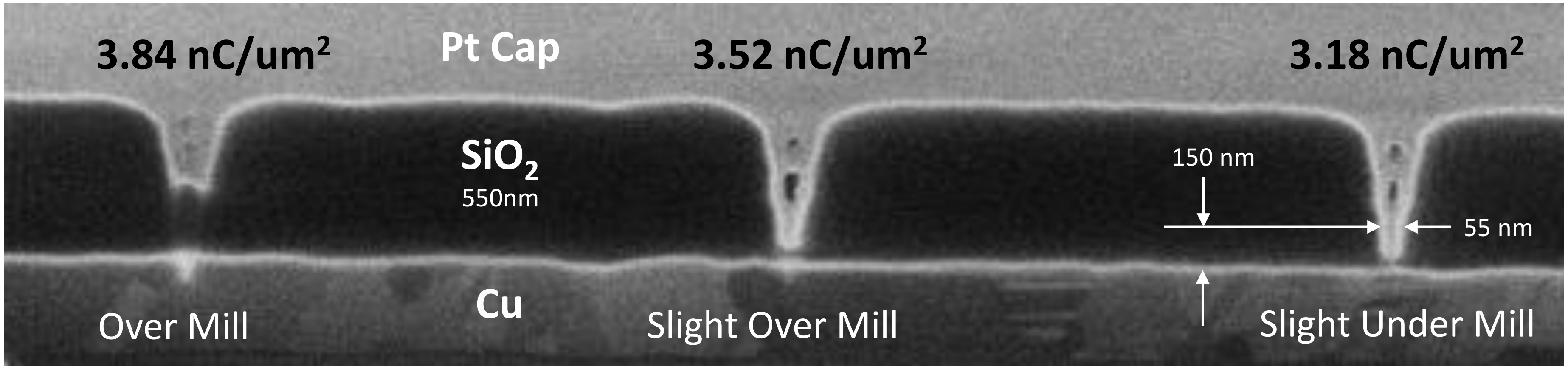
100nm Square Mill Box, 5 pA, 16 kV, 54 mm WD, Negative SIs

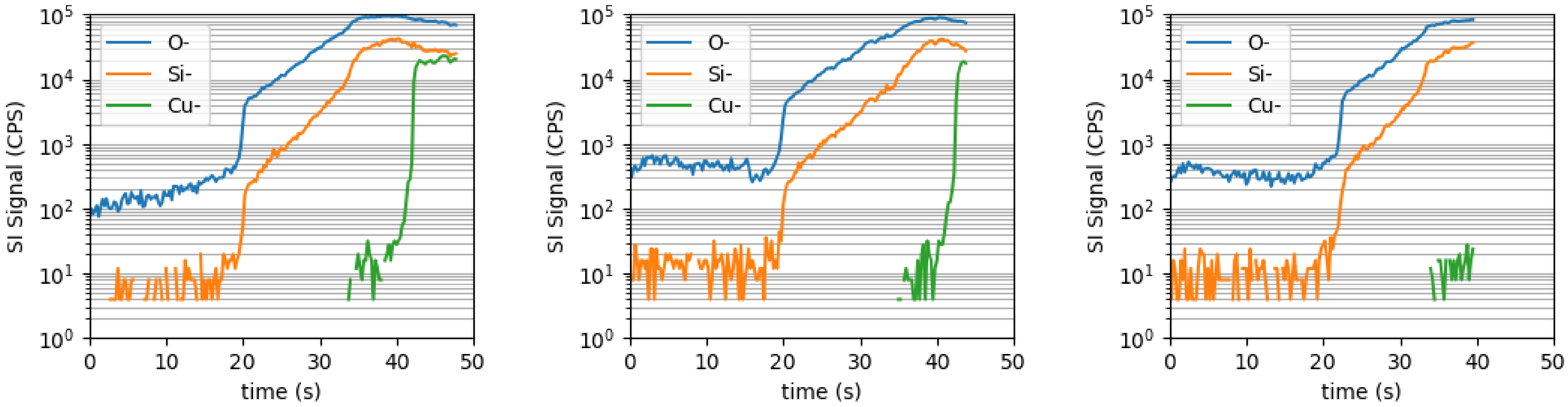


- Initial signal levels rise as cesium concentration builds to enhance the SI yield by ~3 orders of magnitude
- All 3 channels show abrupt changes when crossing the SiO2-Cu interface
- Si- and O- exhibit clear features just prior to Cu- appearance
  - Could be used as advance "predictors" of the endpoint, 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

Section View of 50nm Rectangular Vias

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



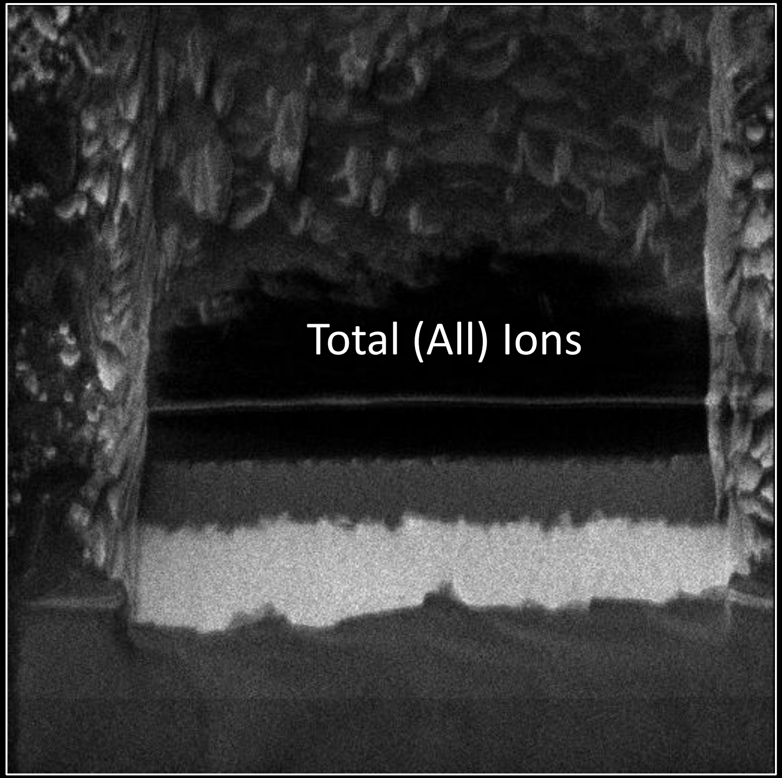


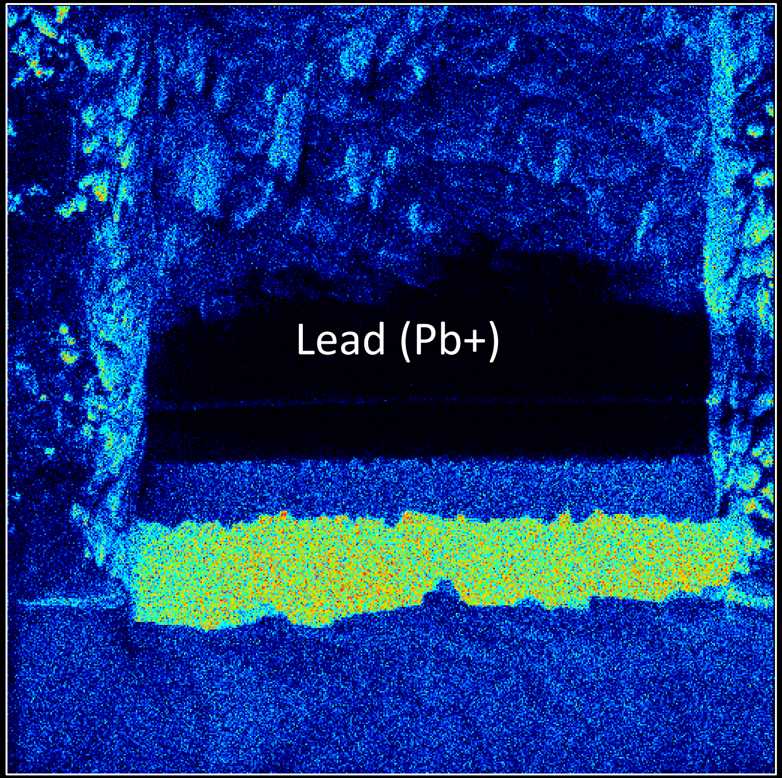
SIMS signals Predictive of Milling Results

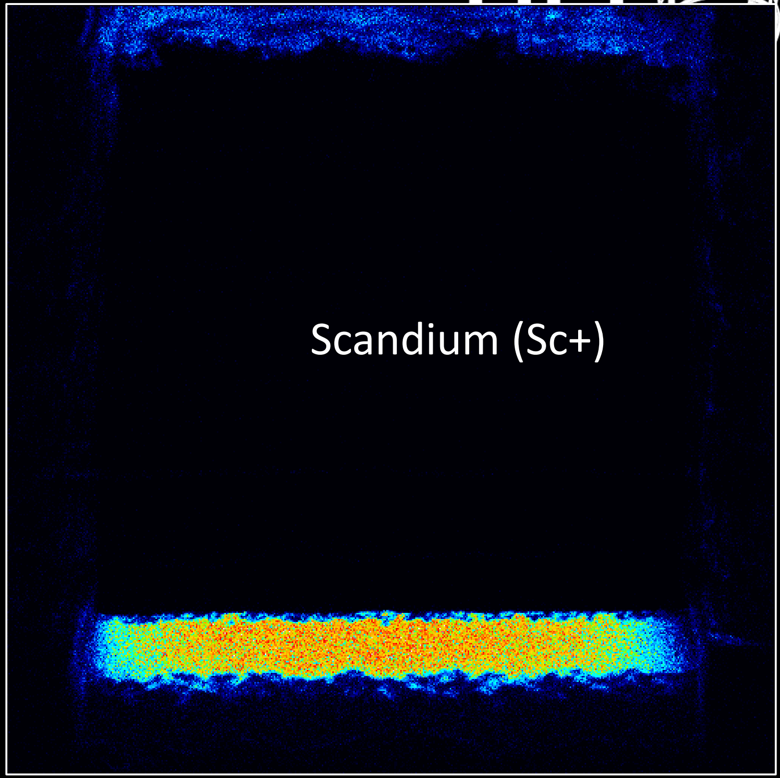
Signal Level Remains High Despite Higher Aspect Ratio

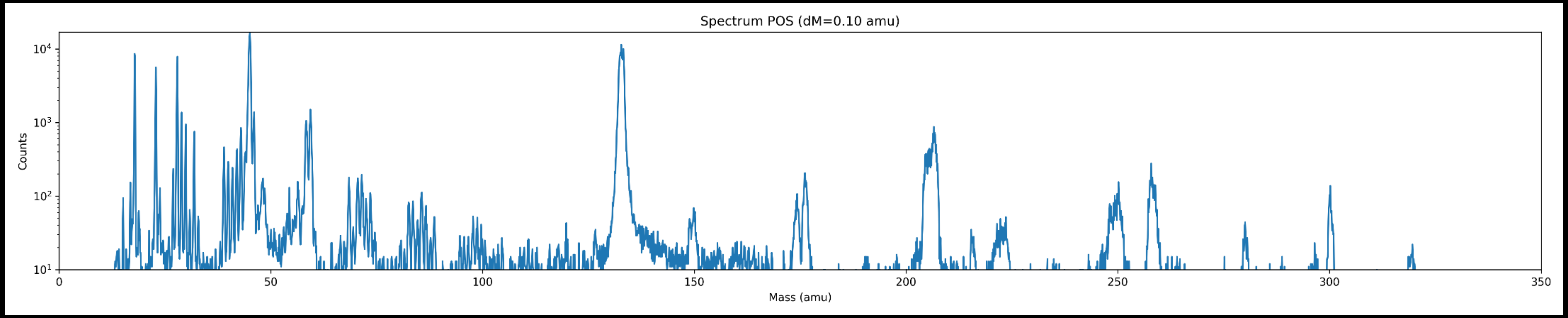
Perovskite SIMS (Positive ions)

Complex Multi-Element Analysis with continuous detector



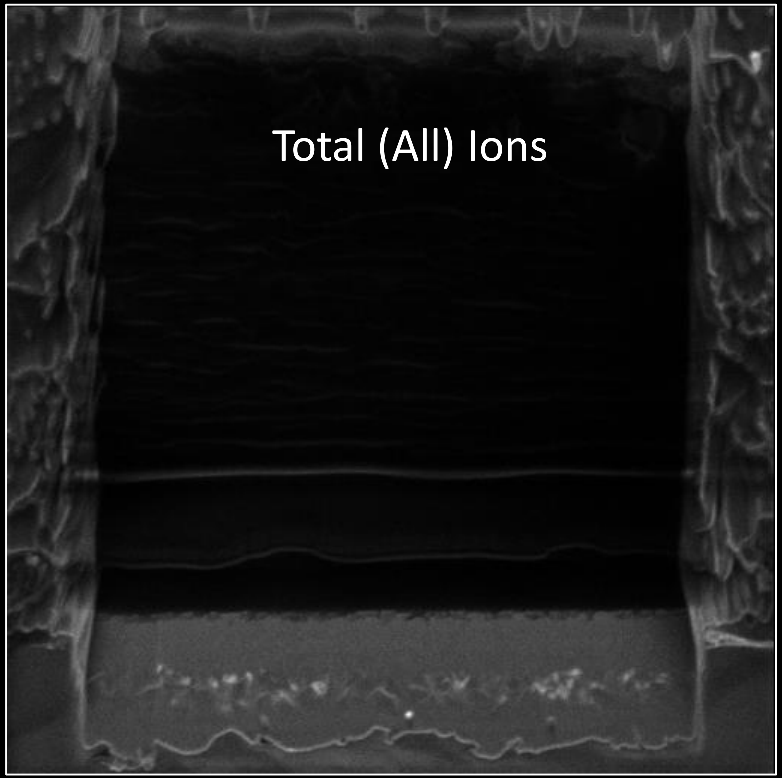


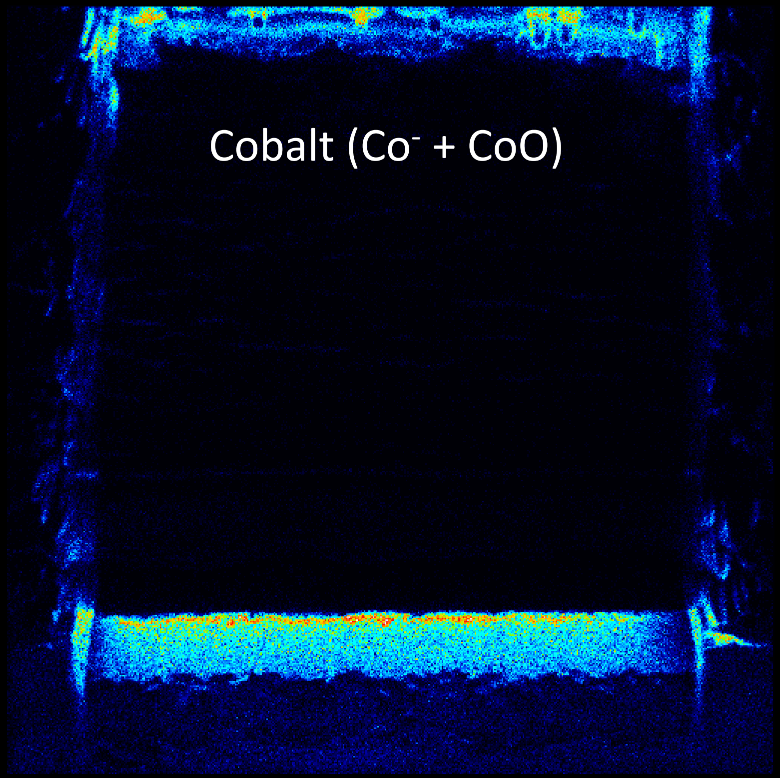


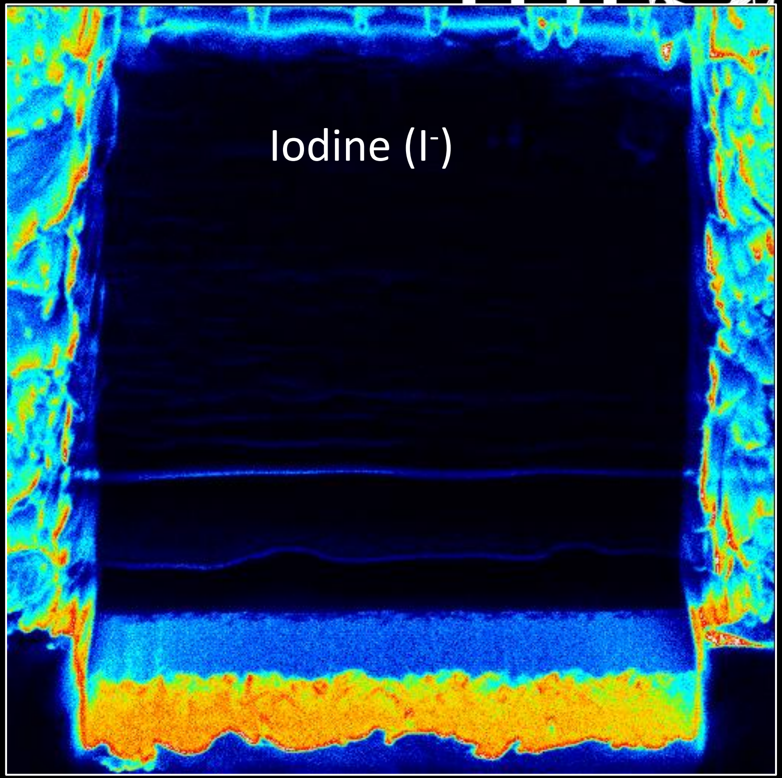


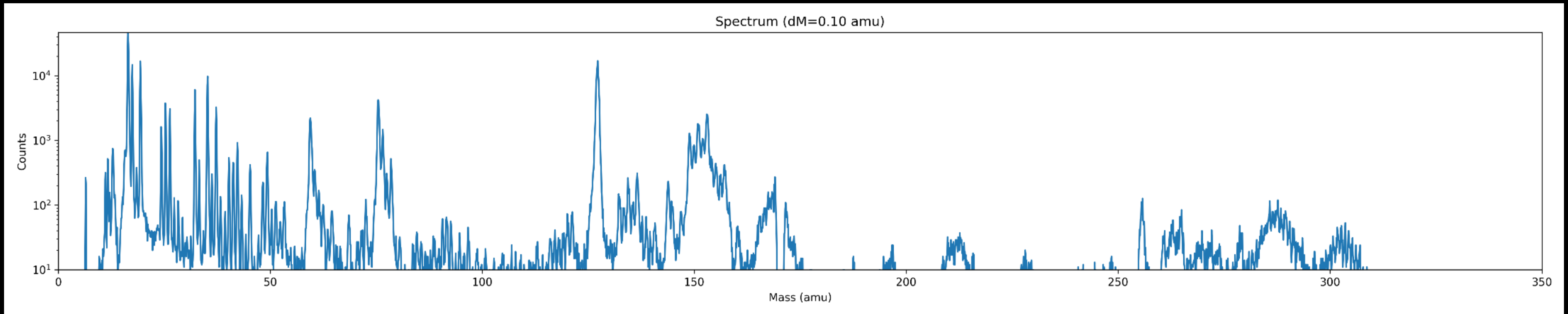
Perovskite SIMS (Negative Ions)

Complex Multi-Element Analysis with continuous detector









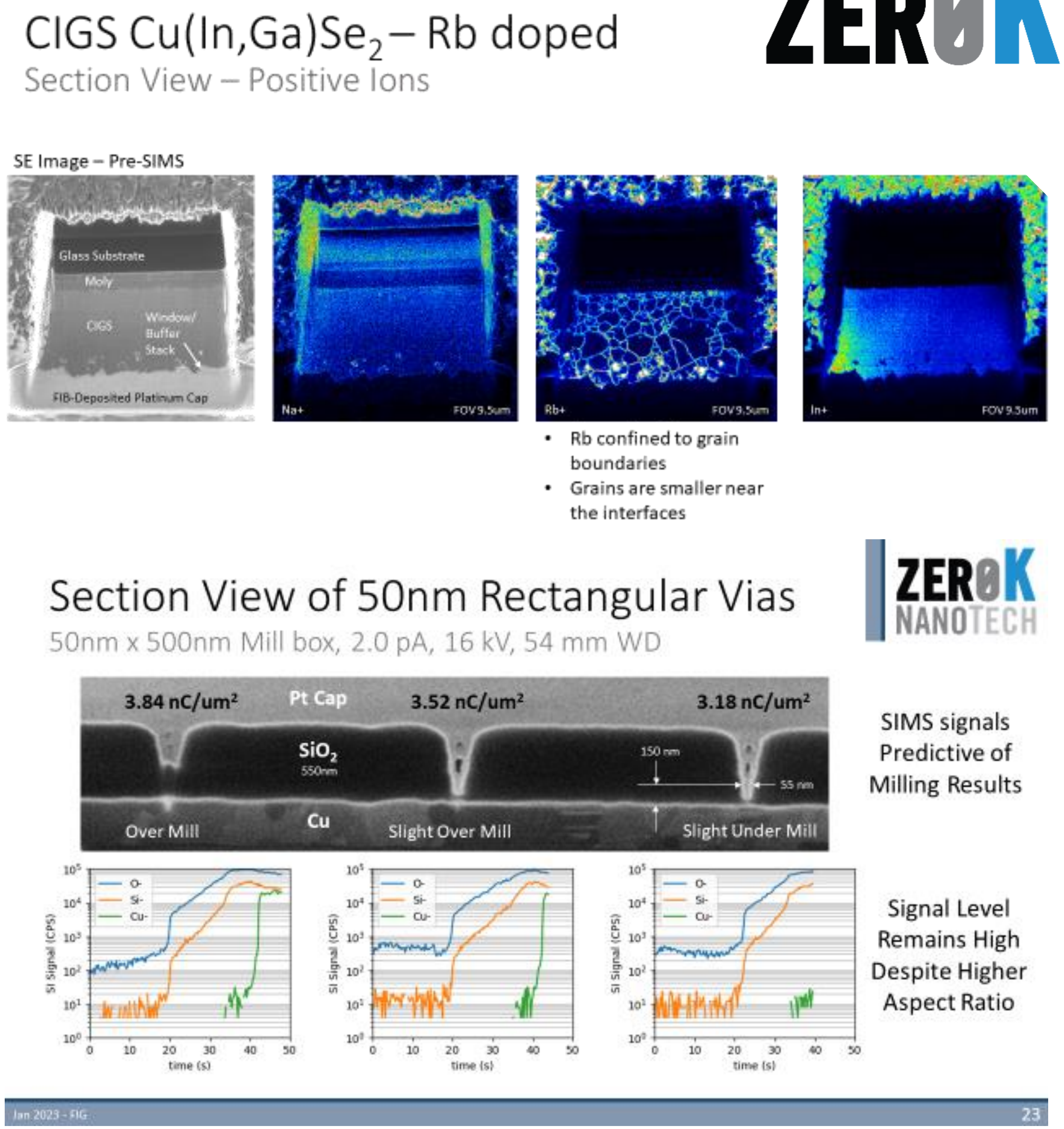
SUMMARY
SIMS:ZERO

... has all the capabilities of FIB:ZERO

... adds high-resolution, high-sensitivity, high speed elemental analysis

... consider in lieu of EDX or ToF SIMS for anlaysis of complex, multi-element samples

... new opportunities for FIB beam control via SIMS signal



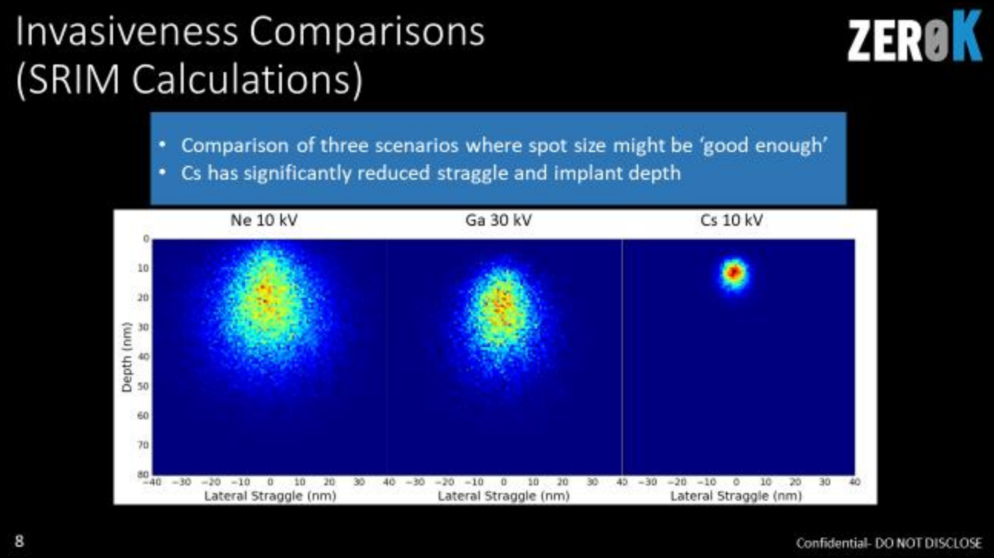
SUMMARY
FIB:ZERO

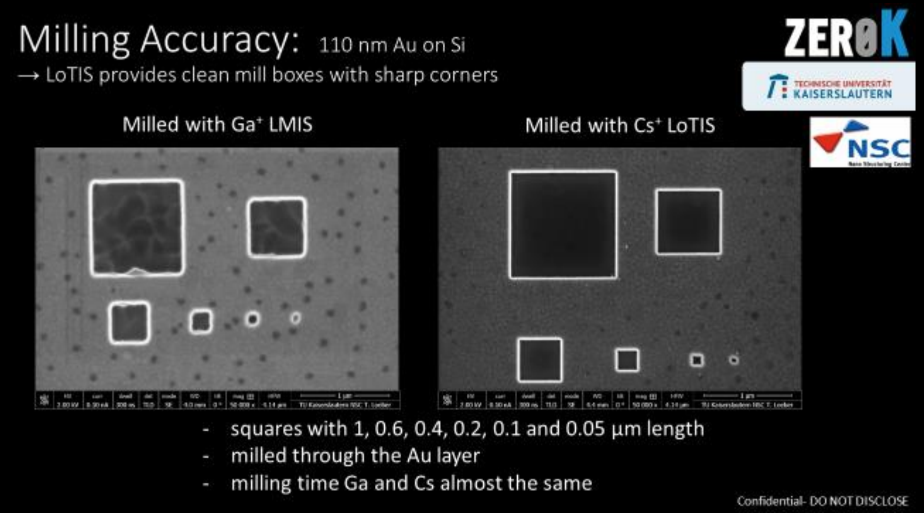
... is a ‘nanomachining’ tool

... has industry-leading performance at low beam currents and low energy

... is compatible with gas precursors for deposition or etch just like other FIBs

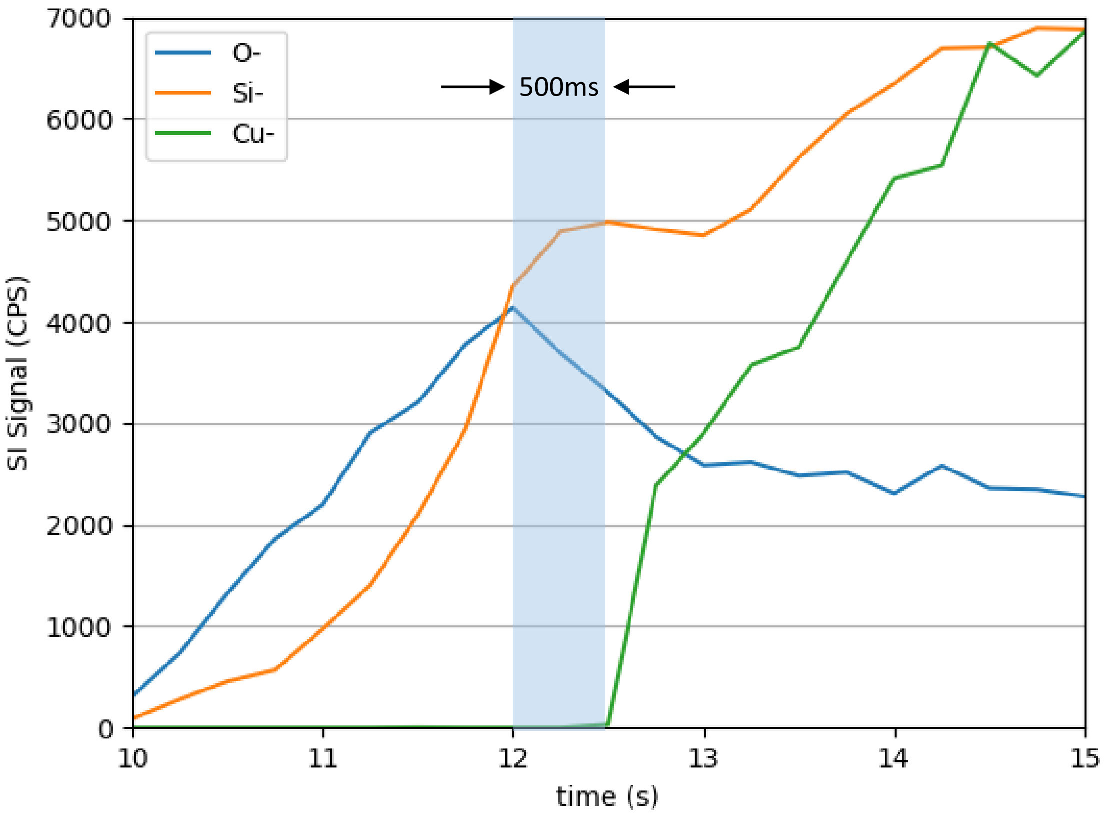
Data pictured right, implant depth and milling fidelity, summarize the story best





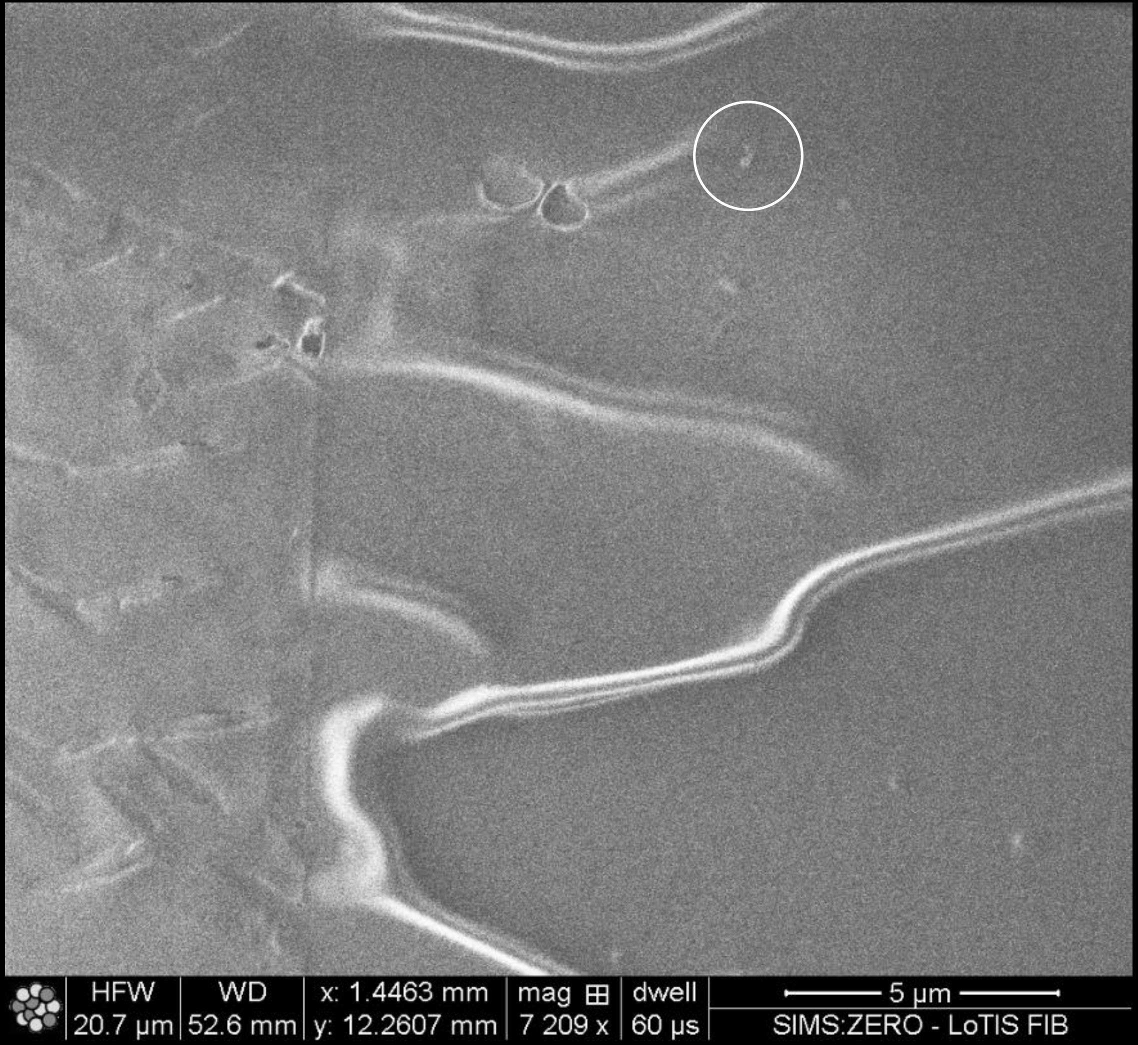
Zoom on SIMS Signal at interface

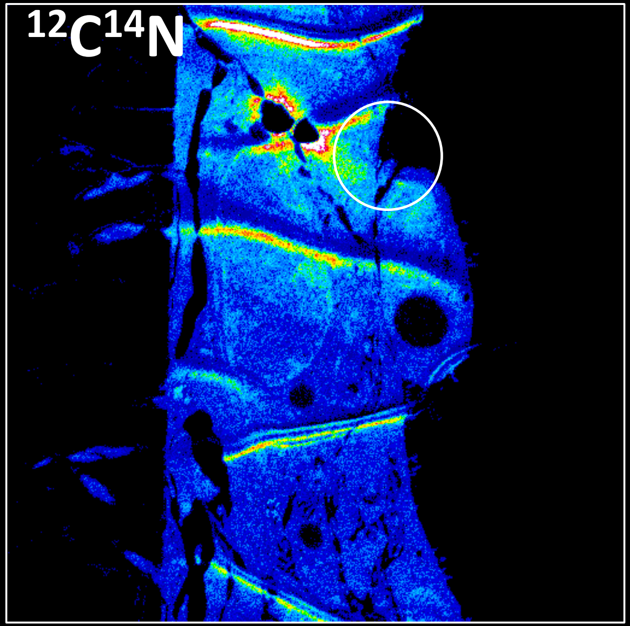
100nm Square Mill Box, 5 pA, 16 kV, 54 mm WD, Negative SIs



- 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

Location of TiO nanoparticle within in huge, fixed cell

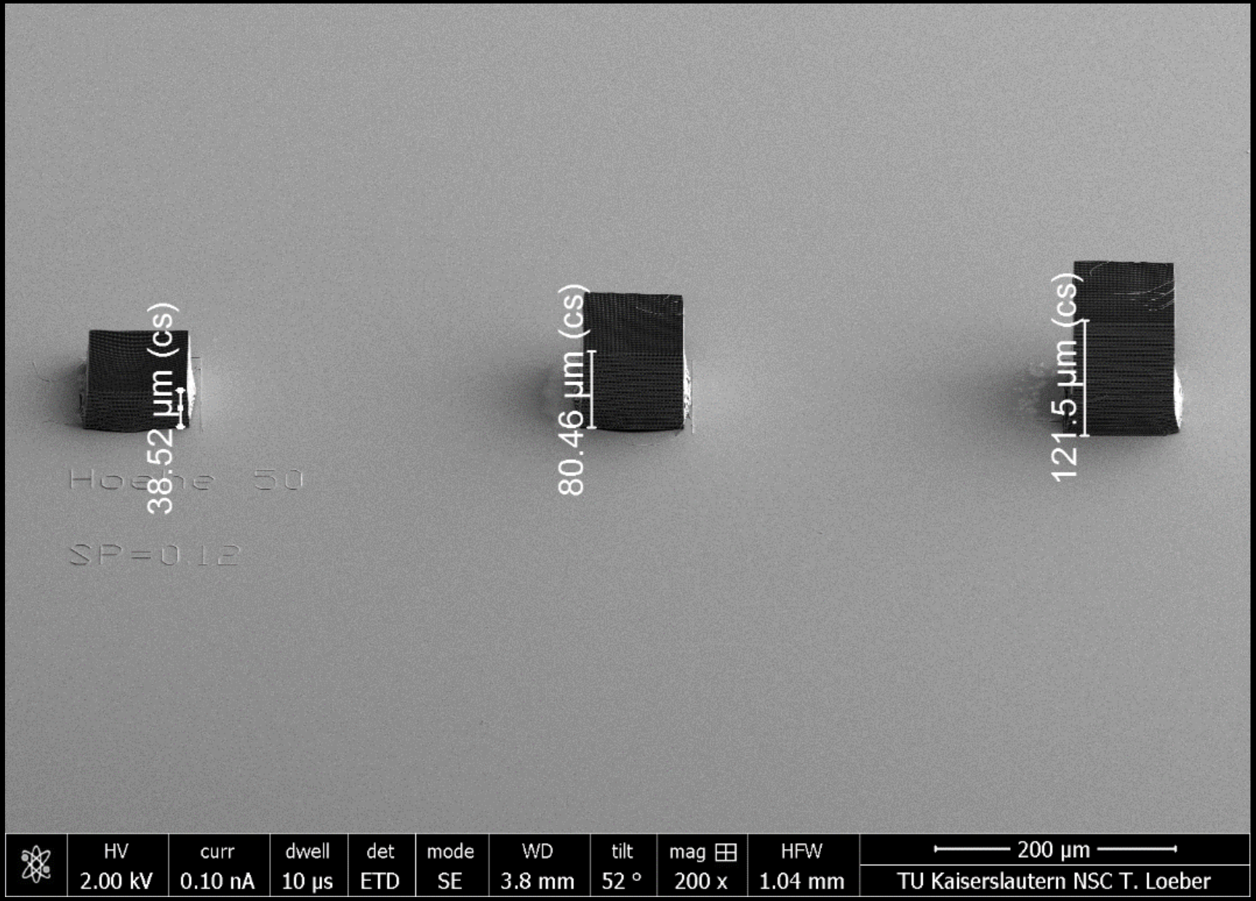




Depth of Focus Comparison

(Results on slides that follow)

FEI: SEM image



"Wood Pile" Nanostructures

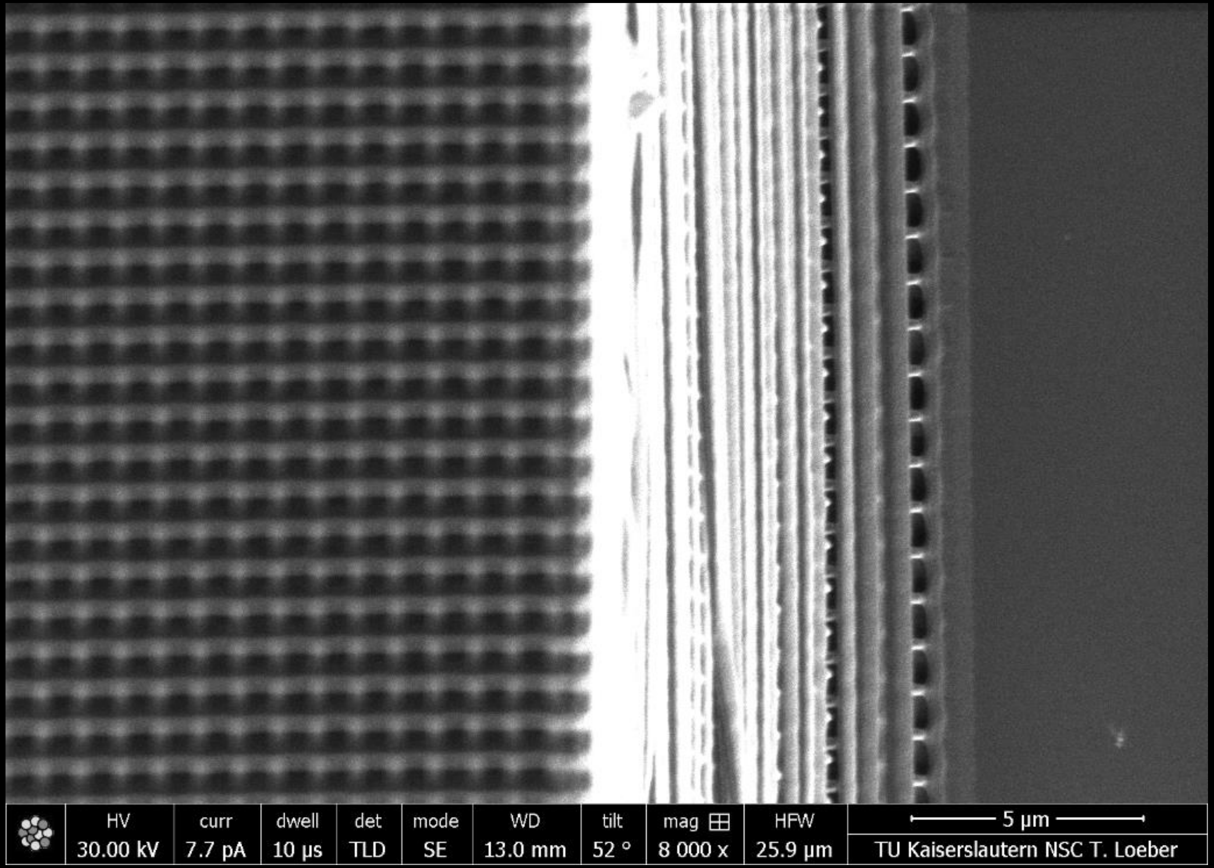
- Heights: 40 µm, 80 µm, 120 µm
- In the following slides we acquire an image containing both the top and bottom of such the 120 µm (tallest) structure
- We can compare the depth of focus of various beams by comparing the 'blurriness' of the top of the structure

A better depth of focus aids in the milling and imaging of 'deep' or 'tall' structures.

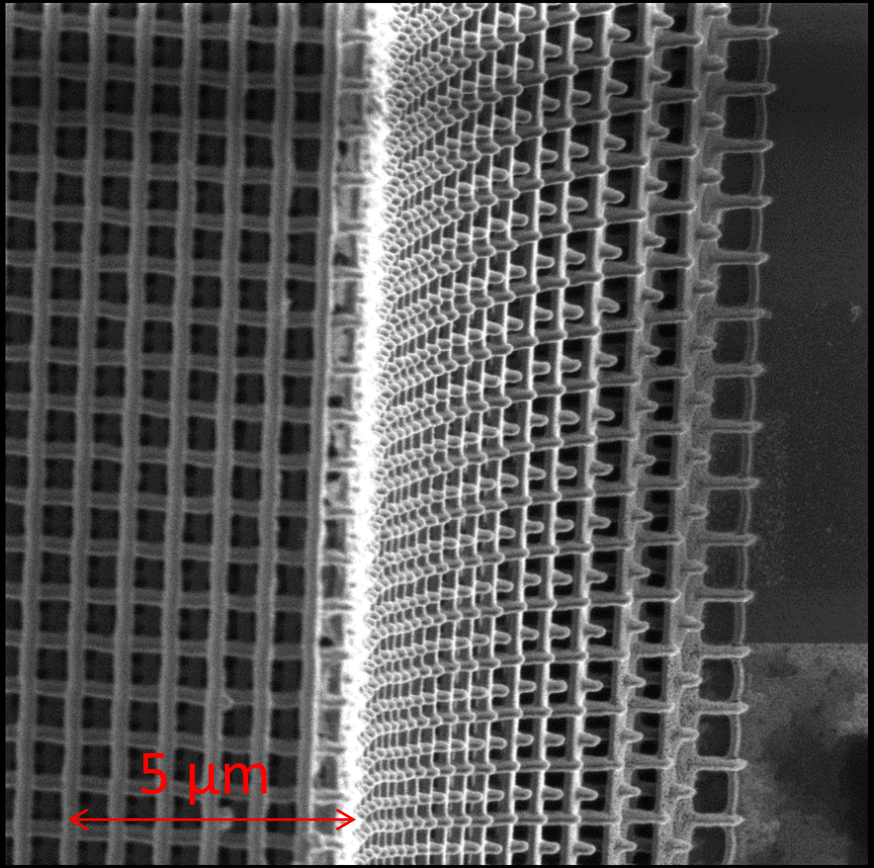
Depth of Focus Comparison

→LoTIS depth of focus substantially better than Ga

Ga⁺ LMIS (30 kV)



Cs⁺ LoTIS (10 kV)

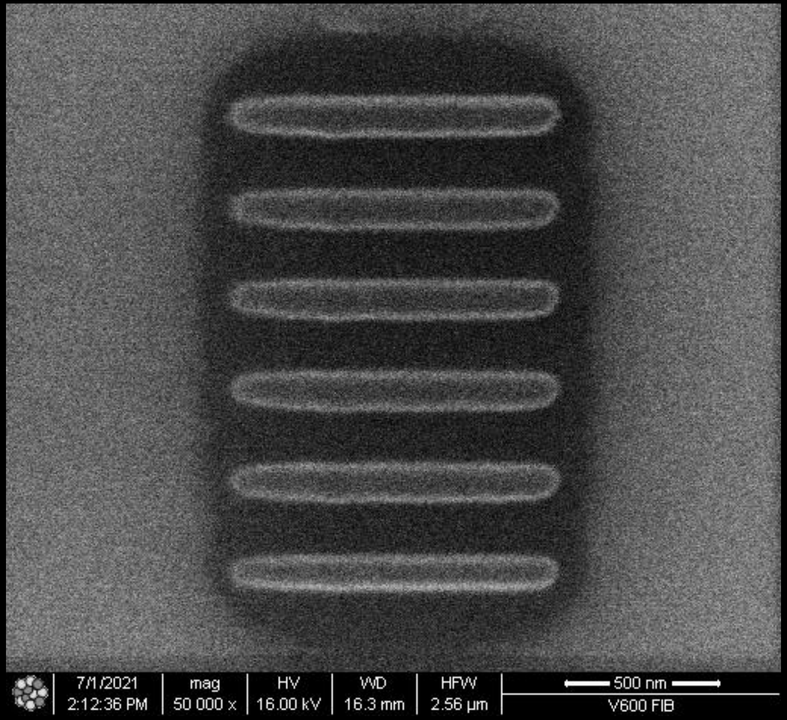


"Wood Pile" Height 120 µm

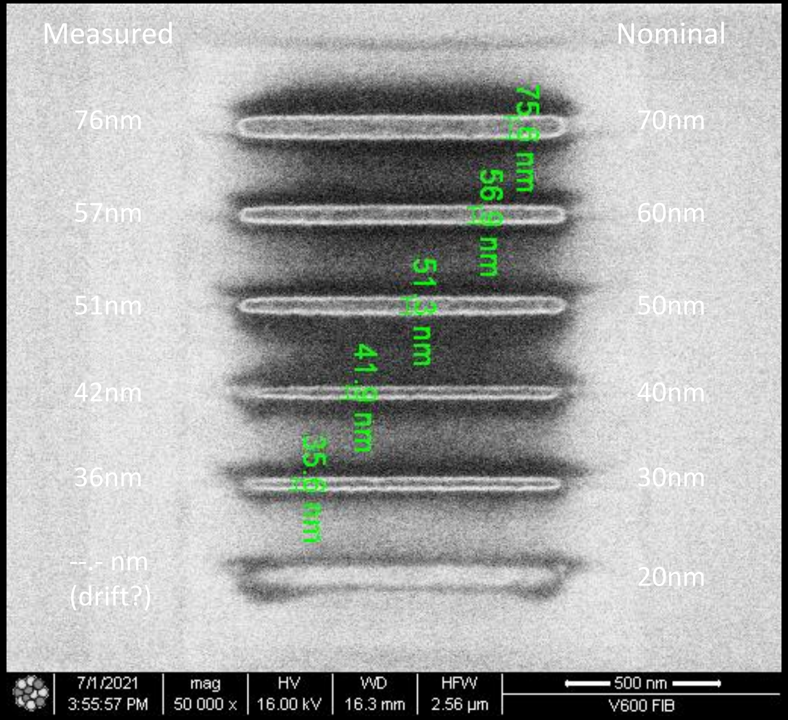
Platinum Deposition – Narrow Lines

→ FIB:ZERO can provide very narrow metal lines

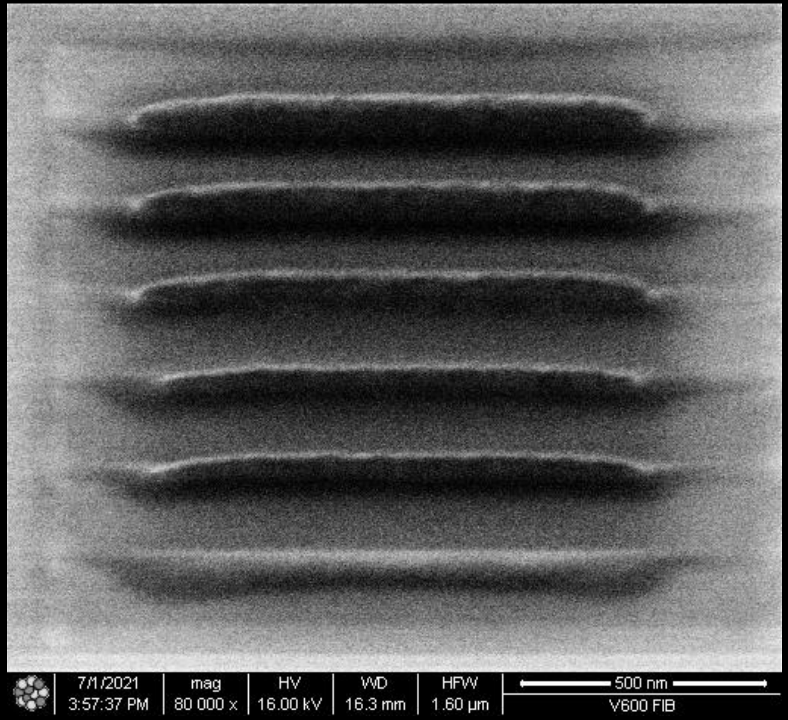
Pt lines with 100nm width as deposited on Si.



Pt lines after cleanup mill.



Tilted view to show height of Pt lines above Si substrate.

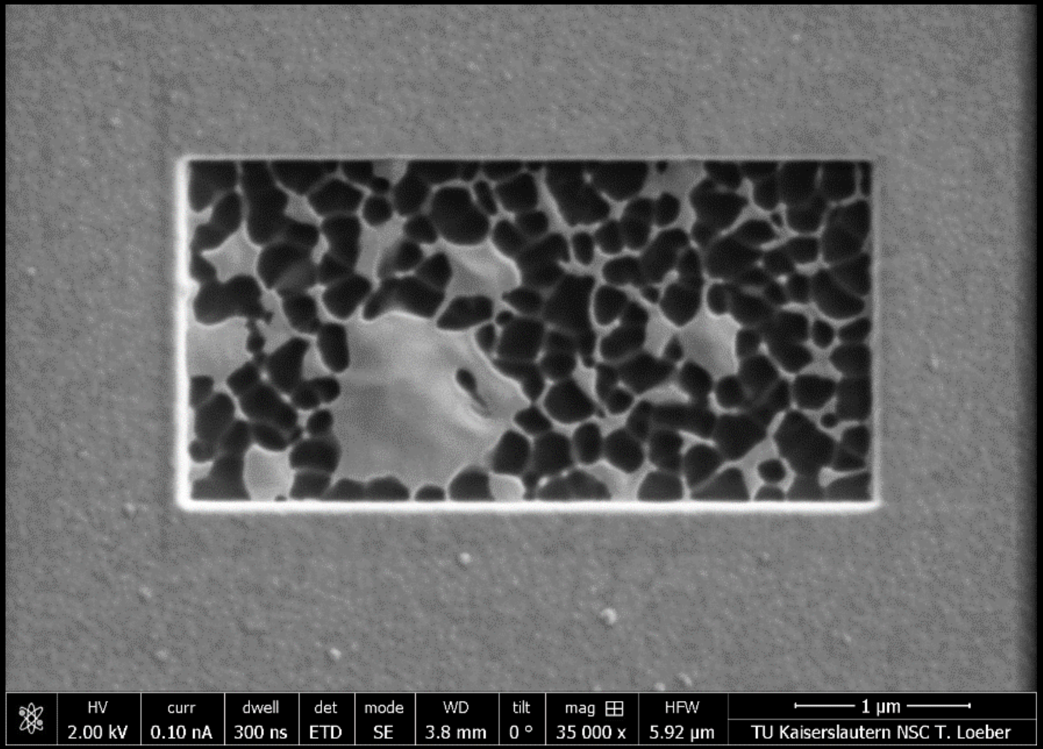


Deposition, milling, and imaging performed with 1.0pA Cs+ ion beam.

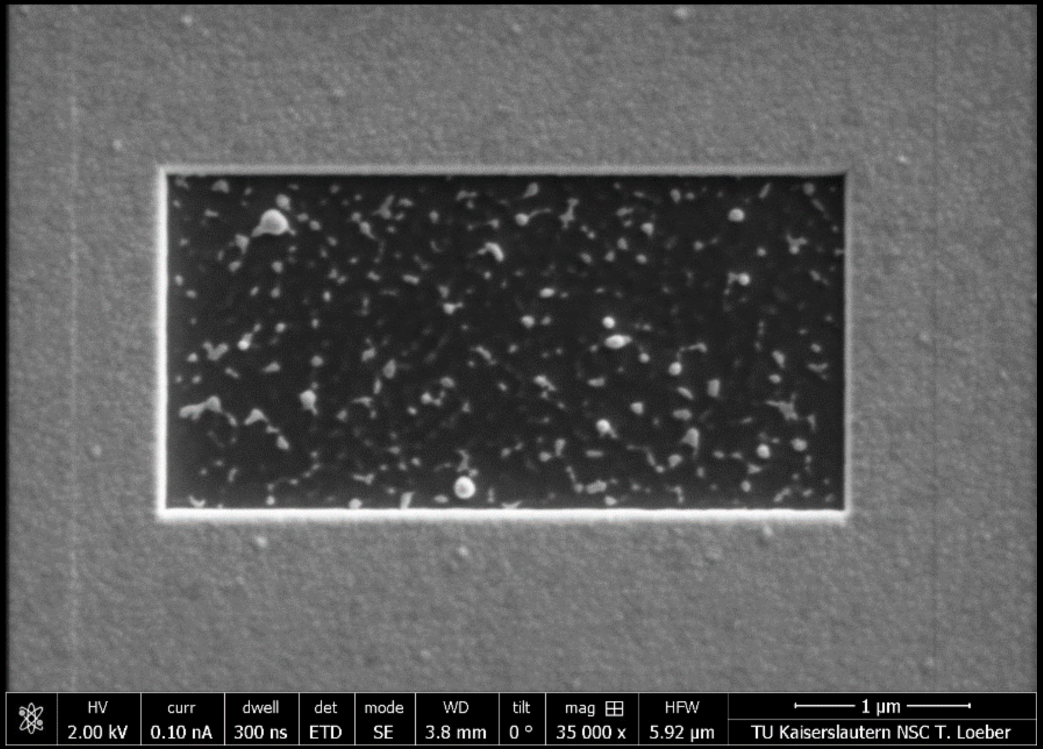
Milling Homogeneity: 150 nm Au on Si

→Cs^+ LoTIS proves even touchdown

Milled with Ga^+ LMIS

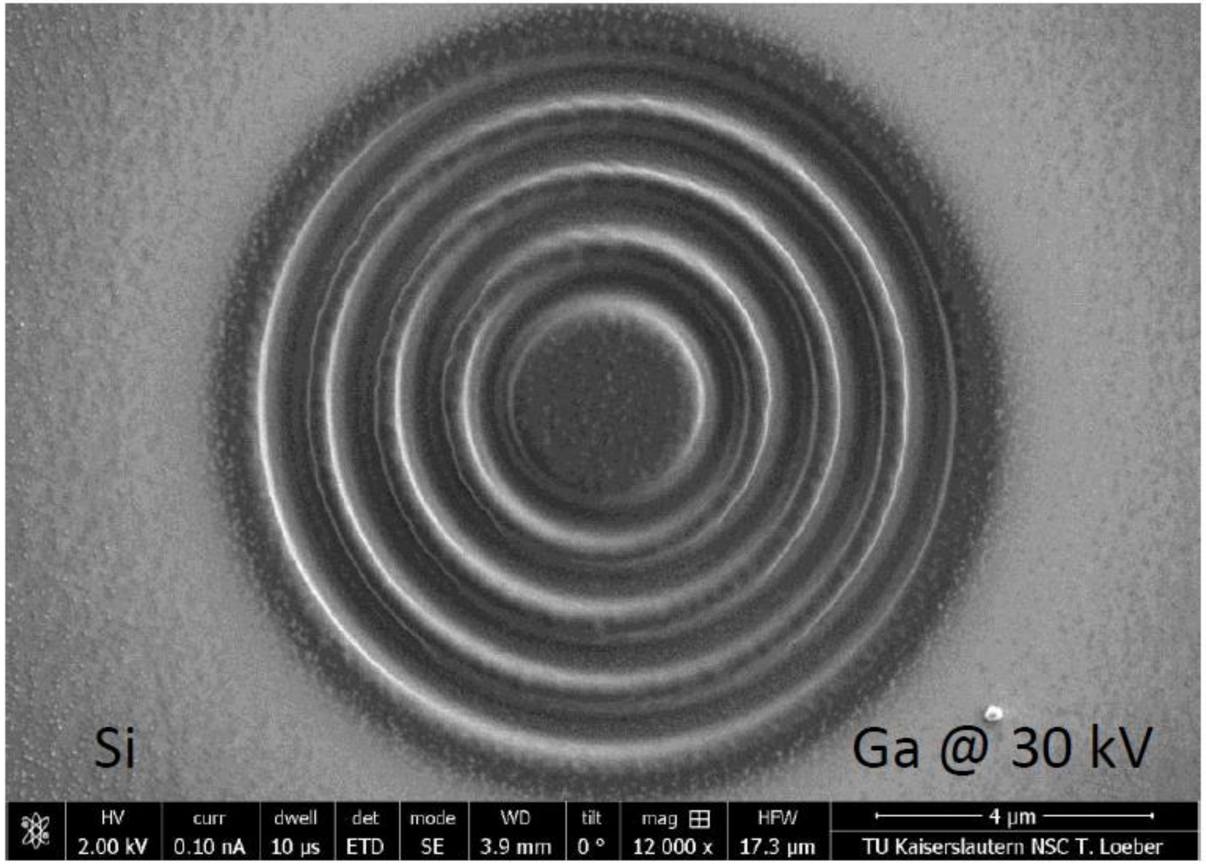


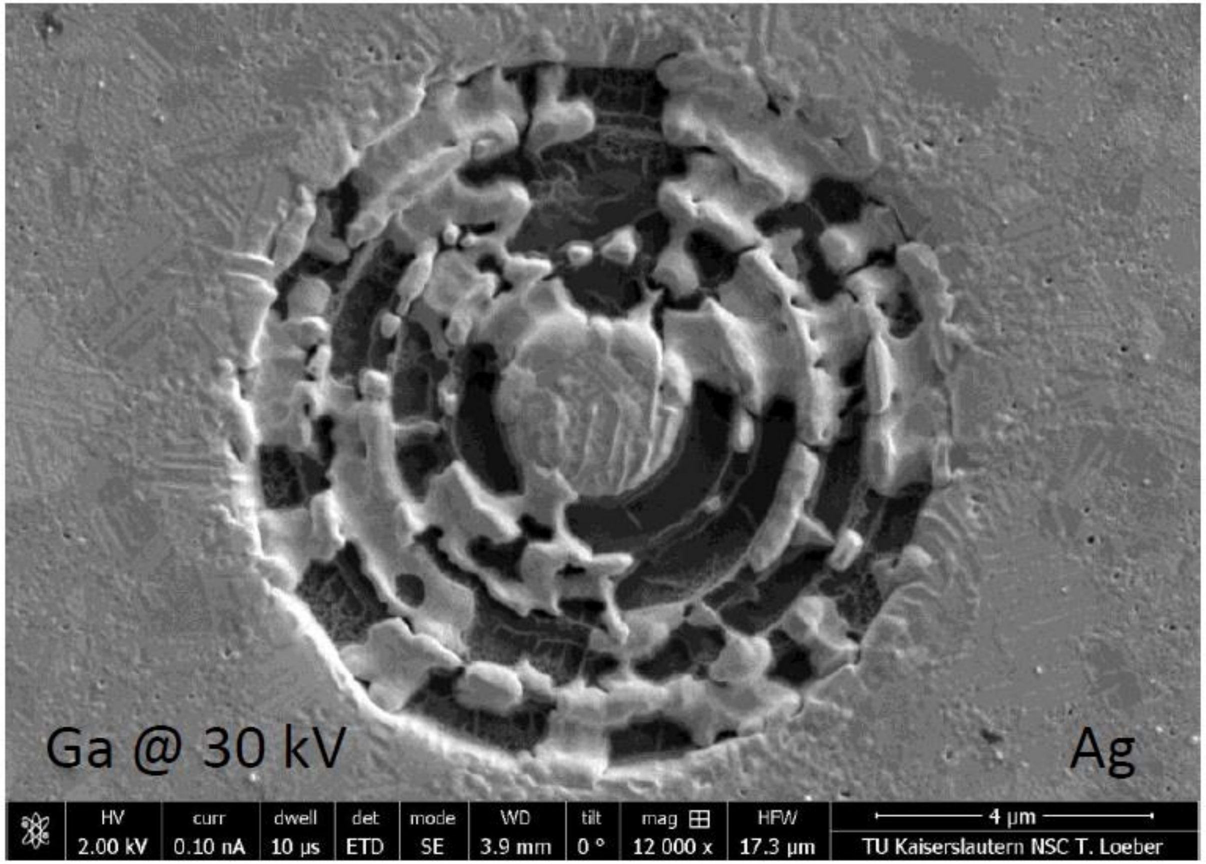
Milled with Cs^+ LoTIS



- milled rectangle ‘almost through’ the Au layer
- milling time Ga and Cs almost the same

Milling in silver

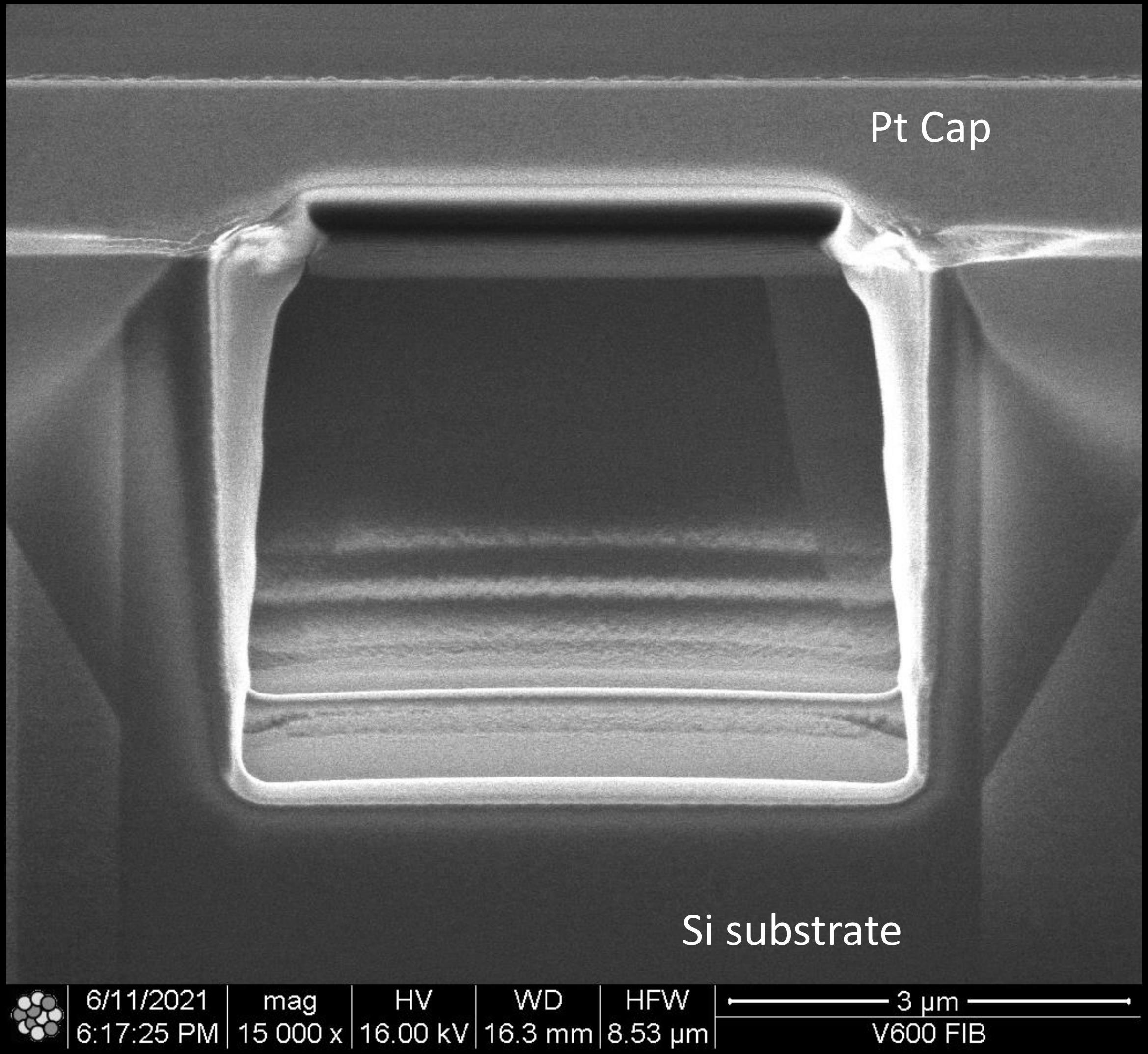


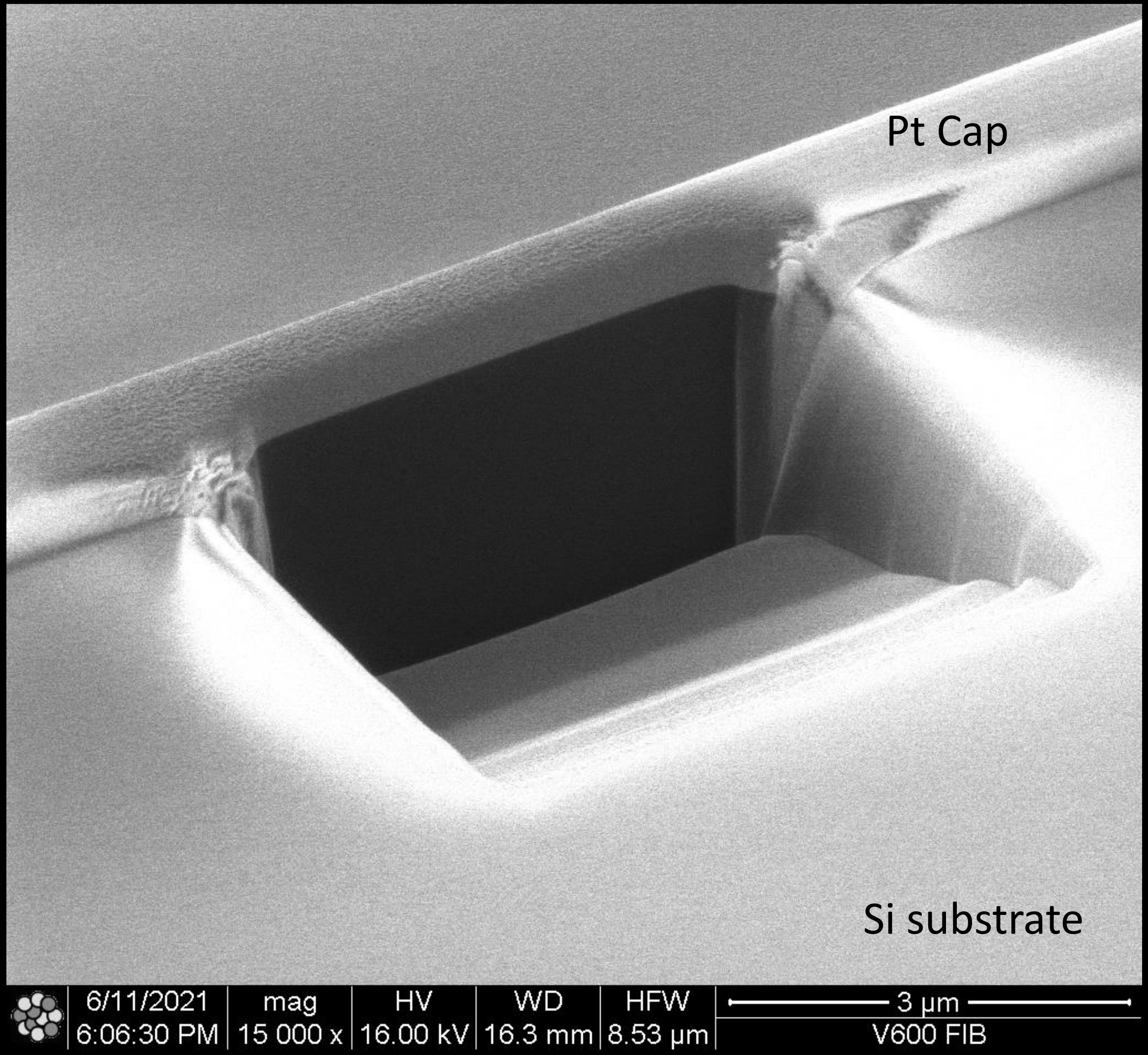


- demonstration: plasmonic ring structures
- no problem in silicon
- inhomogeneous milling in polycrystalline silver

FIB:ZERO Cross Section Example

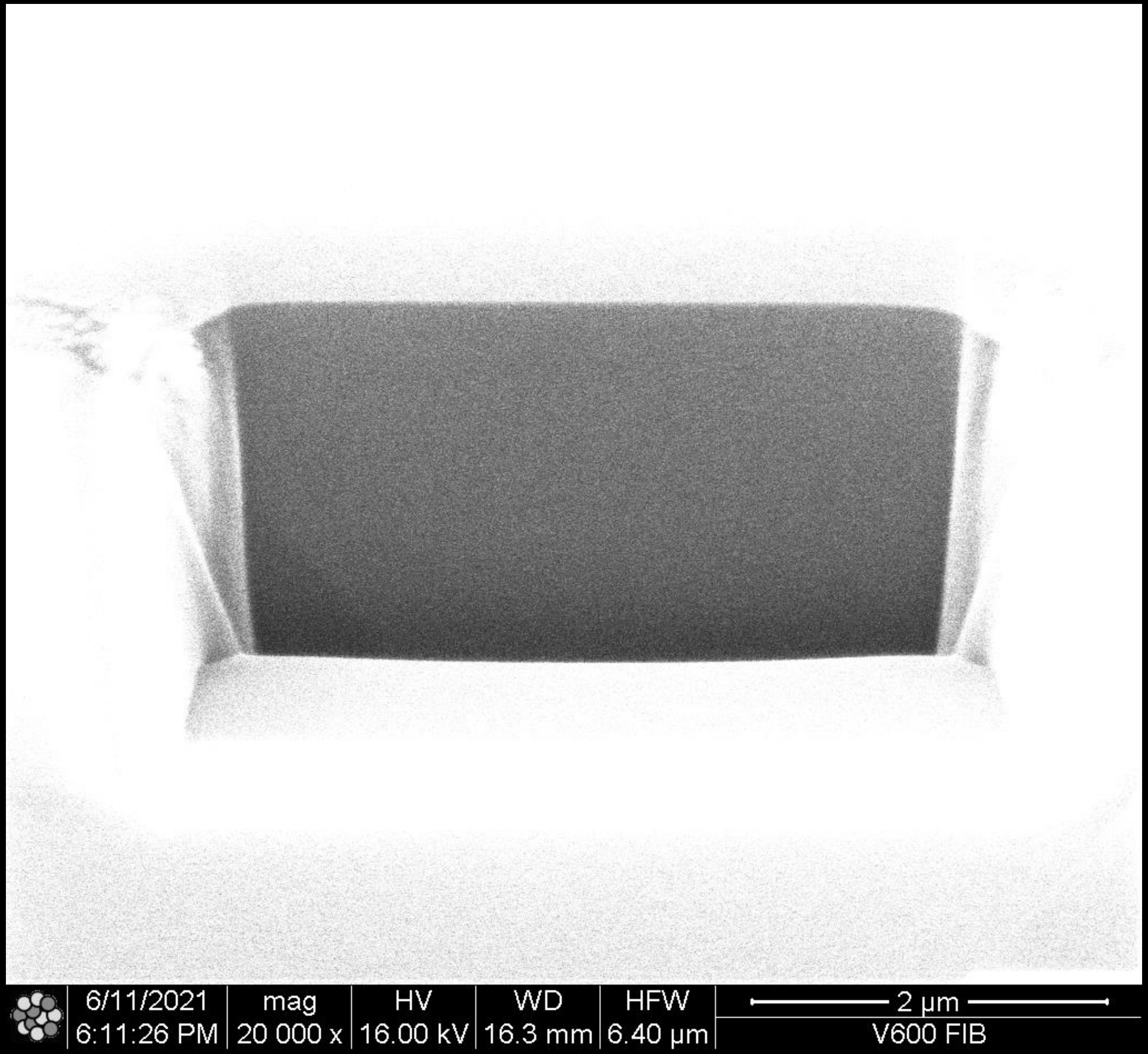
Done with 200 pA beam, 30 pA ‘cleanup’ afterwards

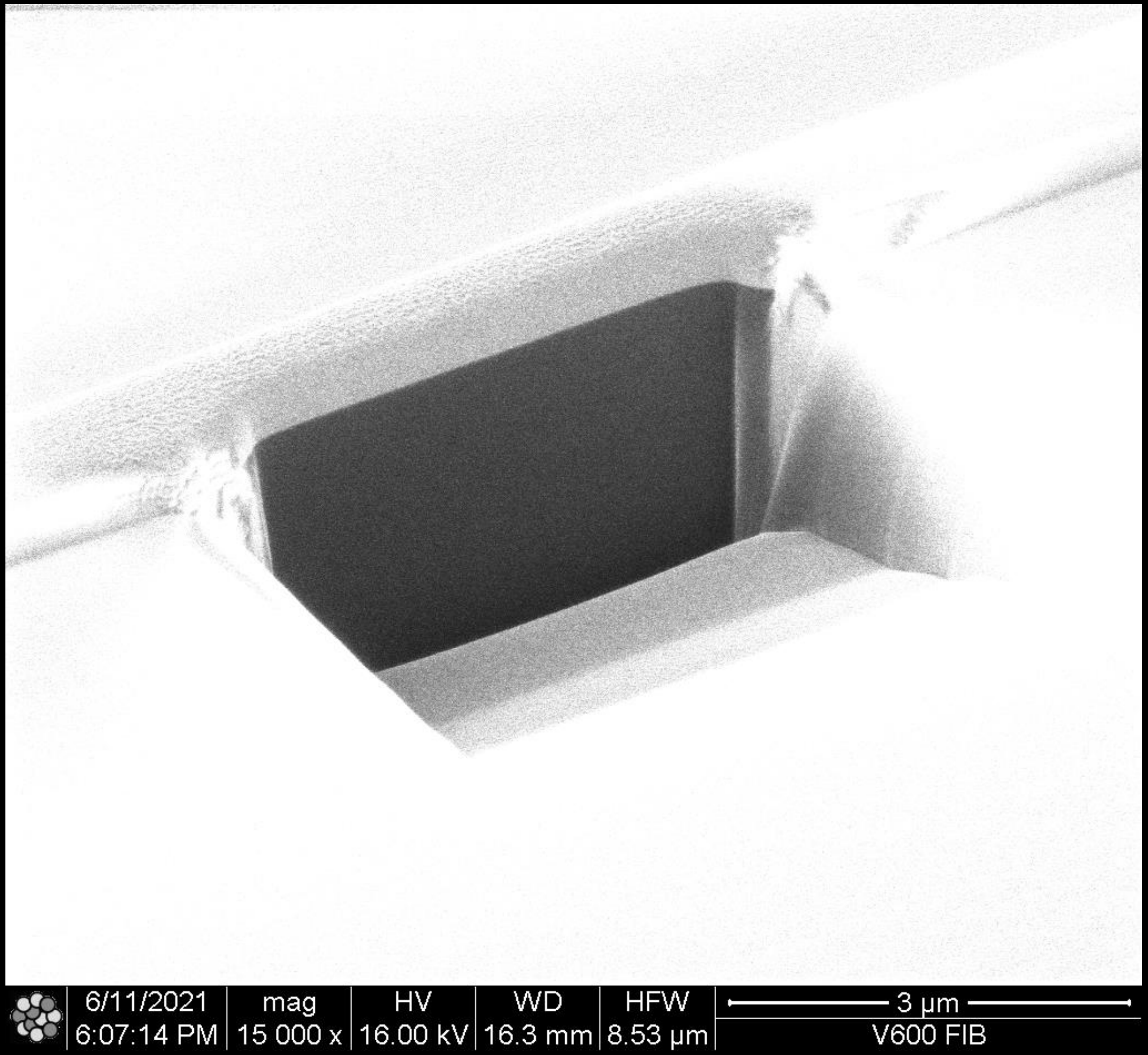




FIB:ZERO Cross Section Example

Oversaturated images to show lack of curtaining

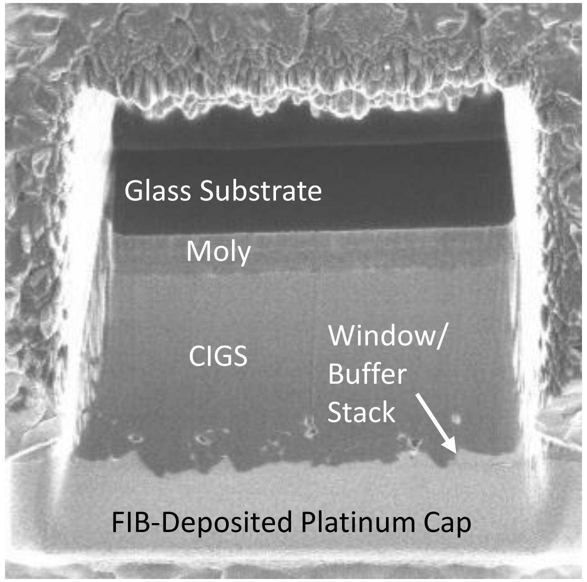


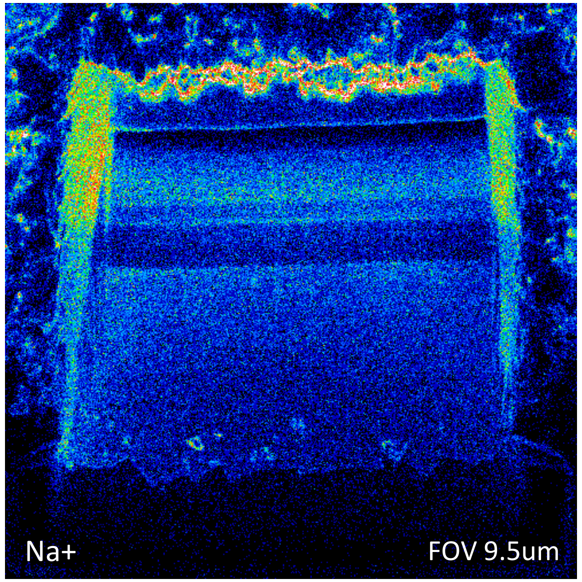


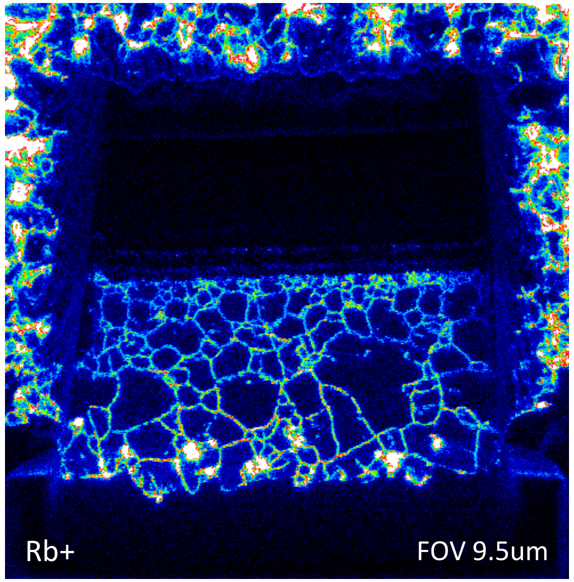
CIGS $Cu(In,Ga)Se_2$ – Rb doped

Section View – Positive Ions

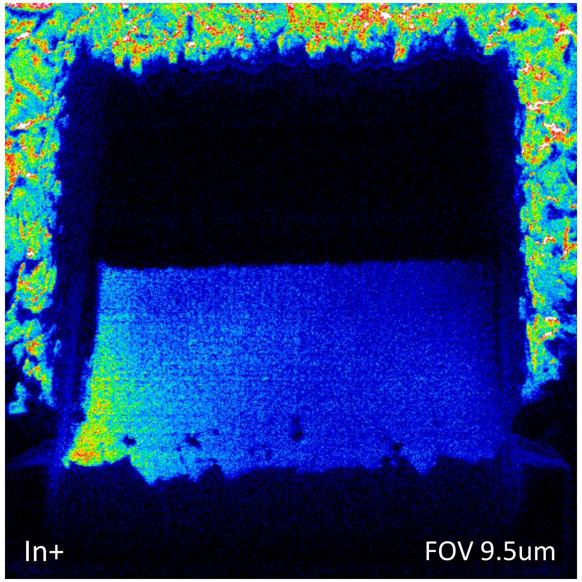
SE Image – Pre-SIMS







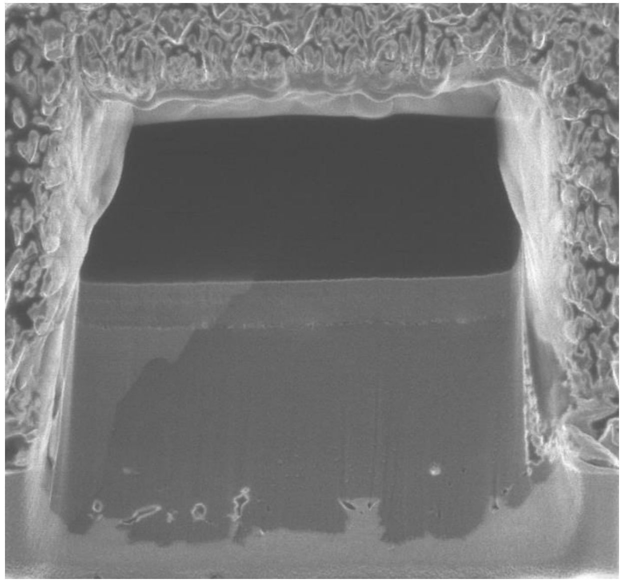
- Rb confined to grain boundaries
- Grains are smaller near the interfaces



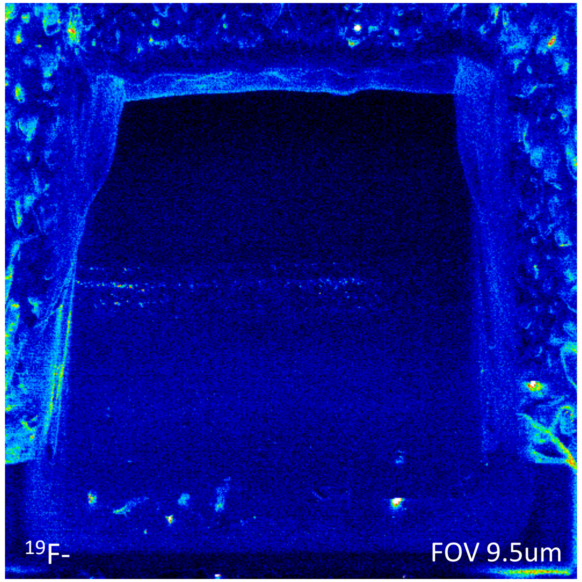
Cs+, 16keV, 3.5pA, 51.6mm WD
CIGS_Pos_2107161606287.csv
CIGS_Pos_2107161613425.csv

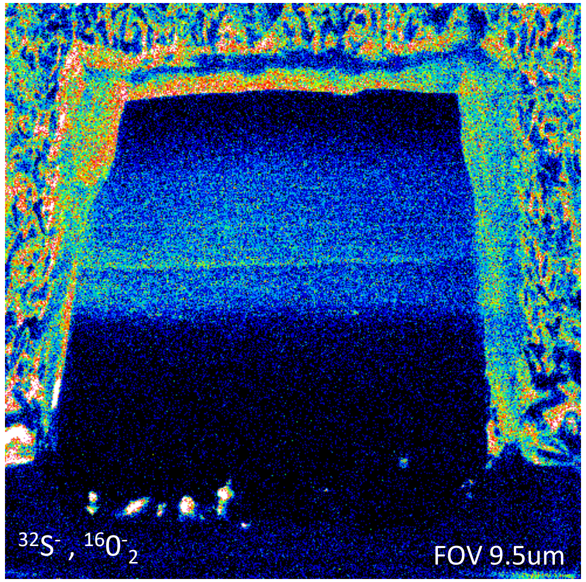
CIGS $Cu(In,Ga)Se_2$ – Rb doped

Section View – Negative Ions – Post 2^{nd} Polish

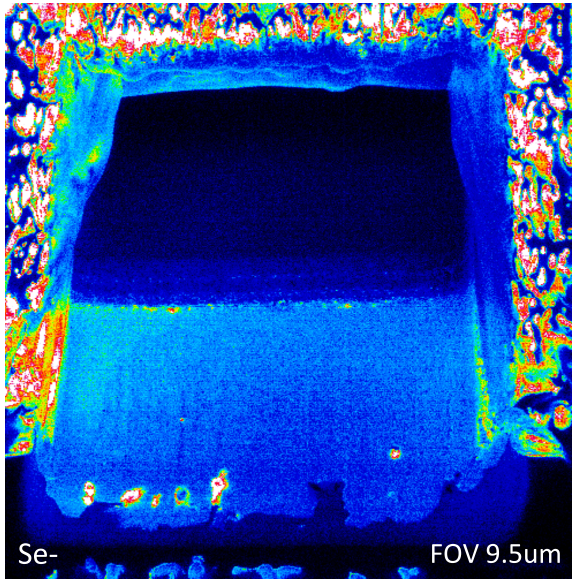


SE Image – Post Polish
Low topography restored





Signal band in CIGS layer near moly may be sulfur, commonly used in CIGS fabrication process; inclusions near surface

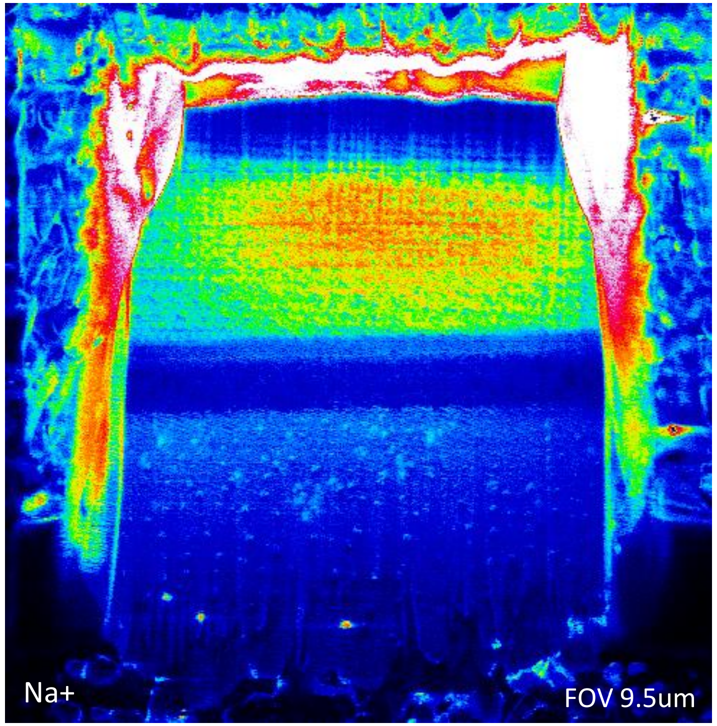


Se is more uniformly distributed in CIGS layer; droplets at moly interface, a few inclusion near surface

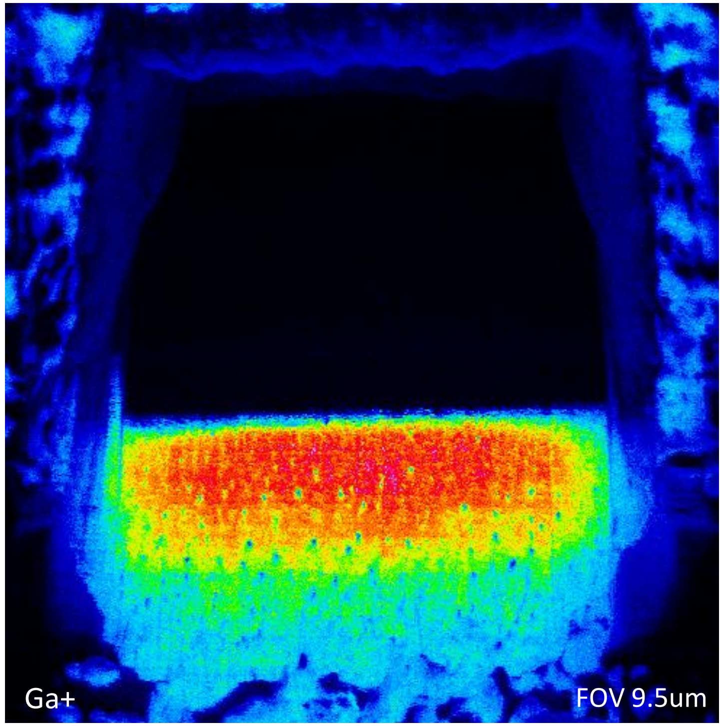
Cs+, 16keV, 10pA, 51.6mm WD
CIGS_Neg_2107201513310.csv

CIGS $Cu(In,Ga)Se_2$ – Rb doped

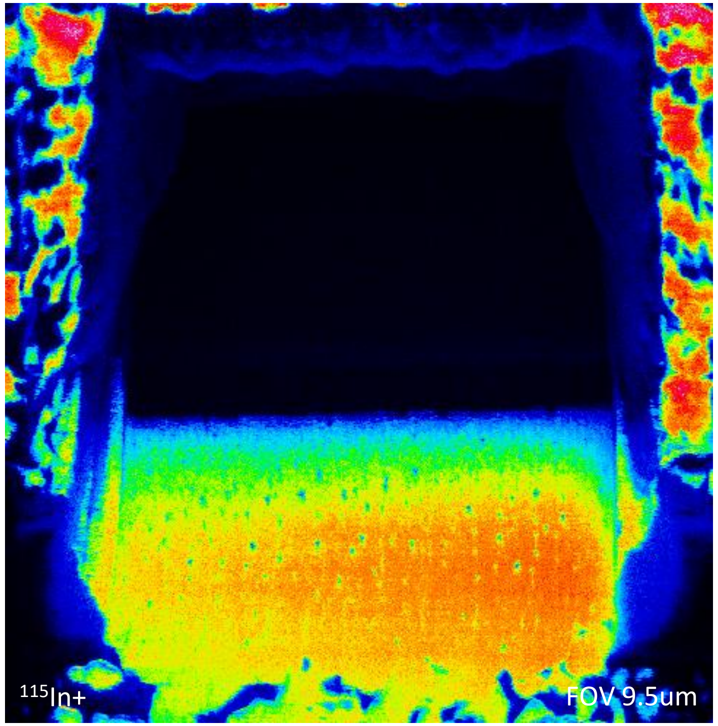
Section View – Positive Ions – Post 3rd Polish



Na – Soda Lime Glass



Ga concentration gradient ↑



In concentration gradient ↓

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