# Applications of the Cesium Low Temperature Ion Source (LoTIS)

*High Resolution FIB and SIMS*

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M&M 2021

# Technology Highlights

## LoTIS is a new $Cs^+$ ion source

AV Steele, et al., Nano Futures, Volume 1, Number 1 (2017)

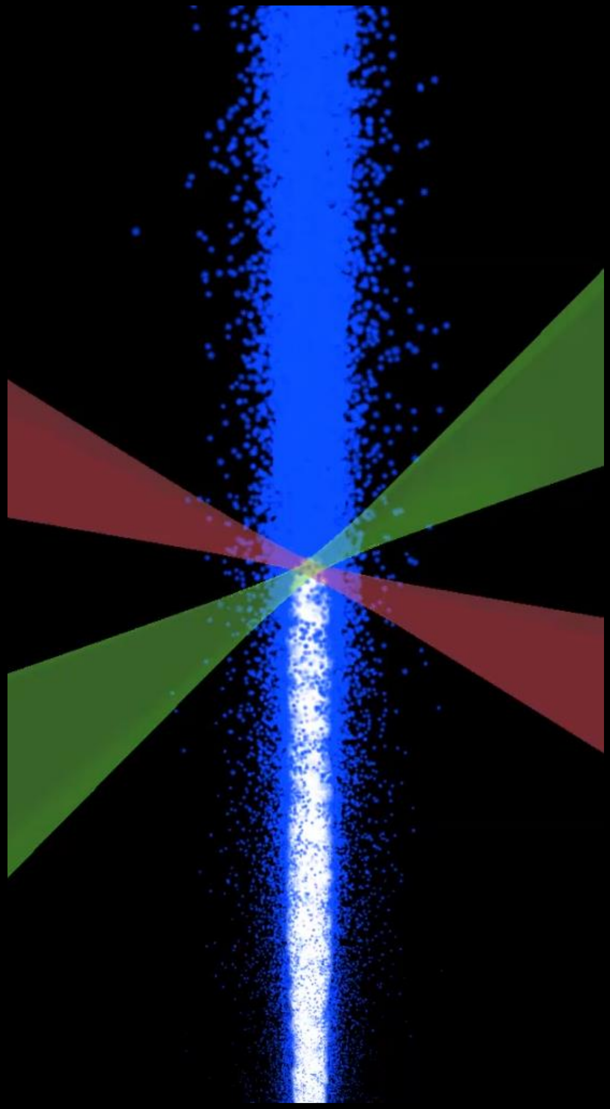
## A LoTIS FIB instrument has been built and tested

- Successful circuit edits on 10 nm node chips
- Imaging and milling demonstrations

## LoTIS Beam Performance

- Demonstrated 2 nm spots with 1 pA, at 10 kV beam
- Provides currents >10 nA (so far)
- 1 kV to 18 keV
  - Performs very well at low-energy relative to $Ga^+$
- Yields large numbers of secondary ions

## Available in FIB and SIMS variants



# FIB:ZERO

Modified Thermo-Fischer v600 platform

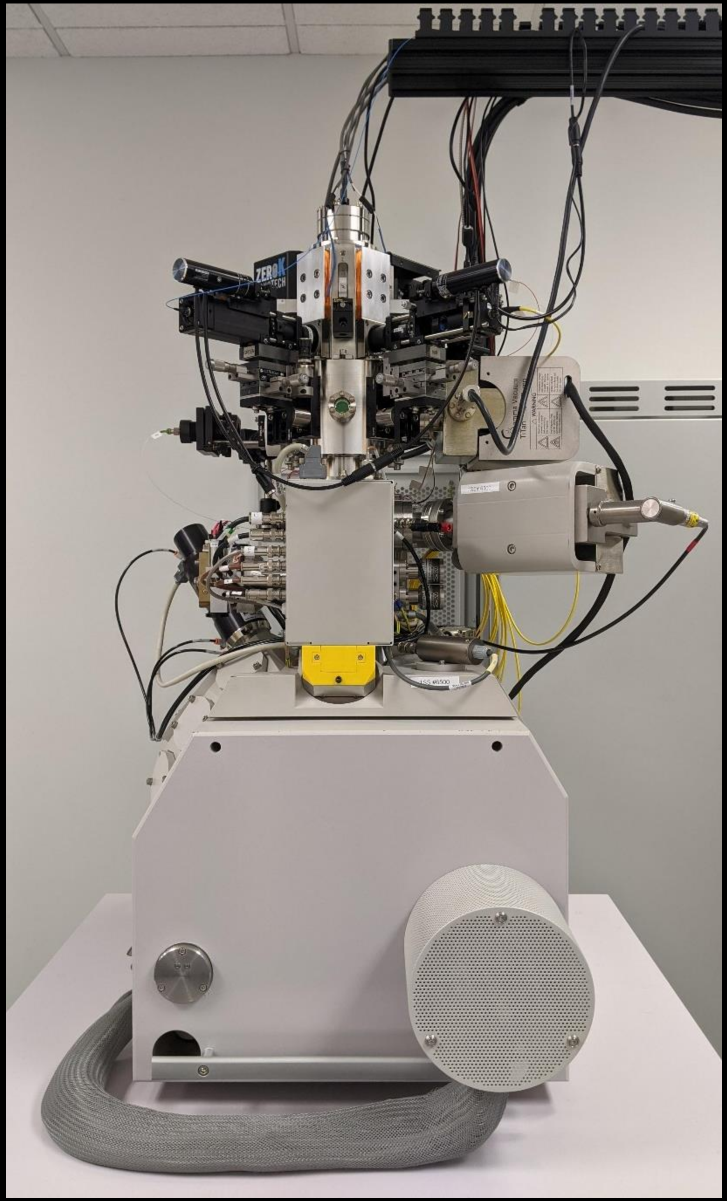
2 nm resolution at 1 pA, 10 keV
(2-3x better spot sizes and at 3x lower beam energy than LMIS)

<1 pA to 10+ nA

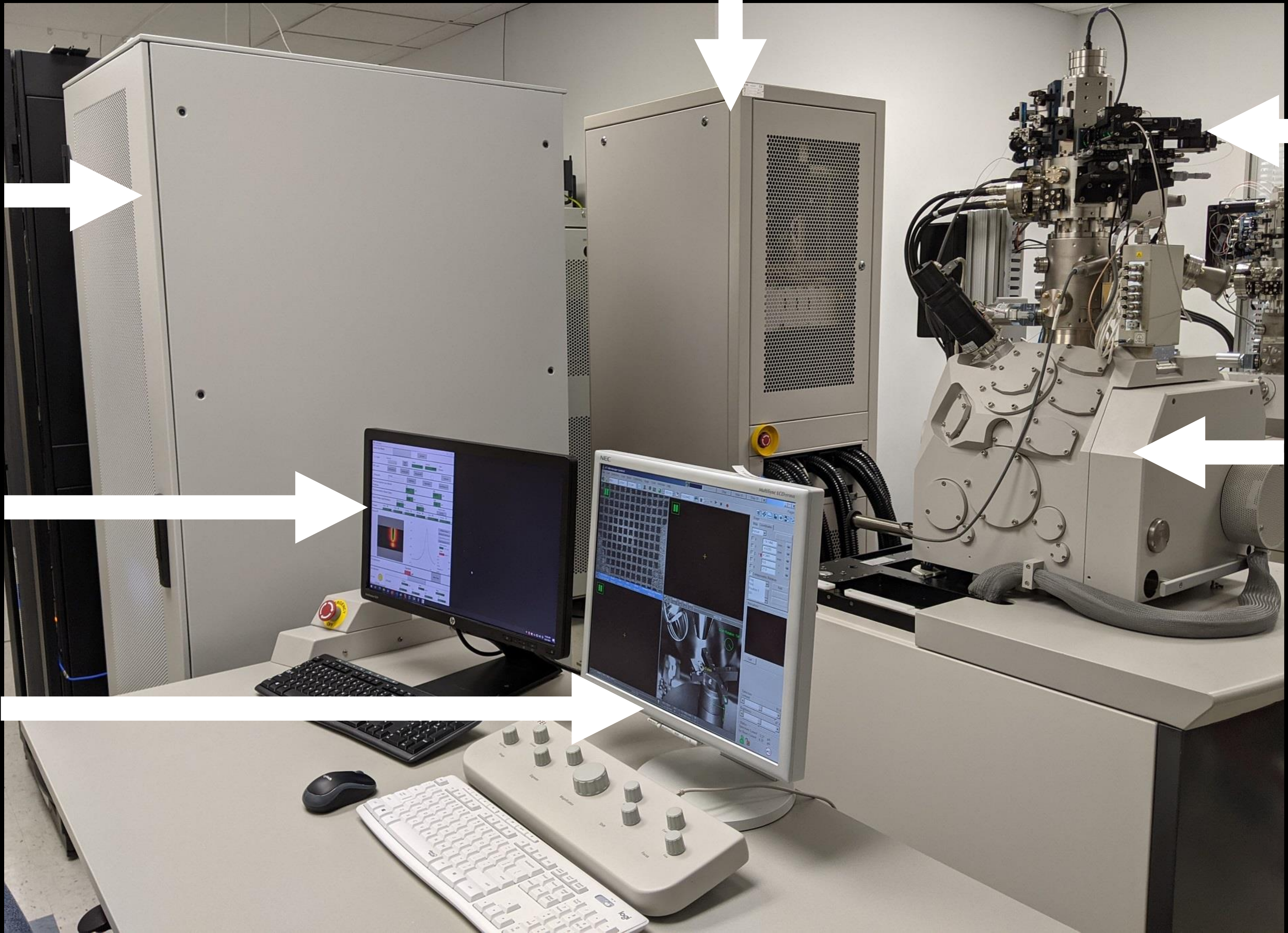
Platinum GIS (others available)

Generates secondary ion images as well

Demonstrations available



# FIB:ZERO



# Spot Sizes

## Selected Beam Energies and Currents

Results given as a $\sigma$

- $d_{50} = 2.2\ \sigma$
- $d_{35-65} = \frac{\sigma}{1.3}$,
- $d_{16-84} = 2\sigma$

Spot sizes are about 2x smaller than Helios ($Ga^{+}$) at <10 pA, and at lower energy

Bit worse spot sizes than Zeiss He/Ne, but better machining performance in many cases

Methodology in [1]

### 16 kV

| Current (pA) | Spot Size (1-$\sigma$ nm) |
|---|---|
| 1.5 | <2.0 |
| 3.0 | 2.3 |
| 10 | 4.0 |
| 30 | 7.5 |
| 100 | 23 |
| 300 | 57 |
| 1000 | 175 |
| 5500 | 580 |

### 8 kV

| Current (pA) | Spot Size (1-$\sigma$ nm) |
|---|---|
| 1.5 | 2.3 |
| 3 | 2.5 |
| 10 | 4.7 |
| 30 | 7.6 |
| 100 | 55 |
| 300 | 150 |
| 1000 | 244 |
| 2600 | 510 |

### Low-Energy

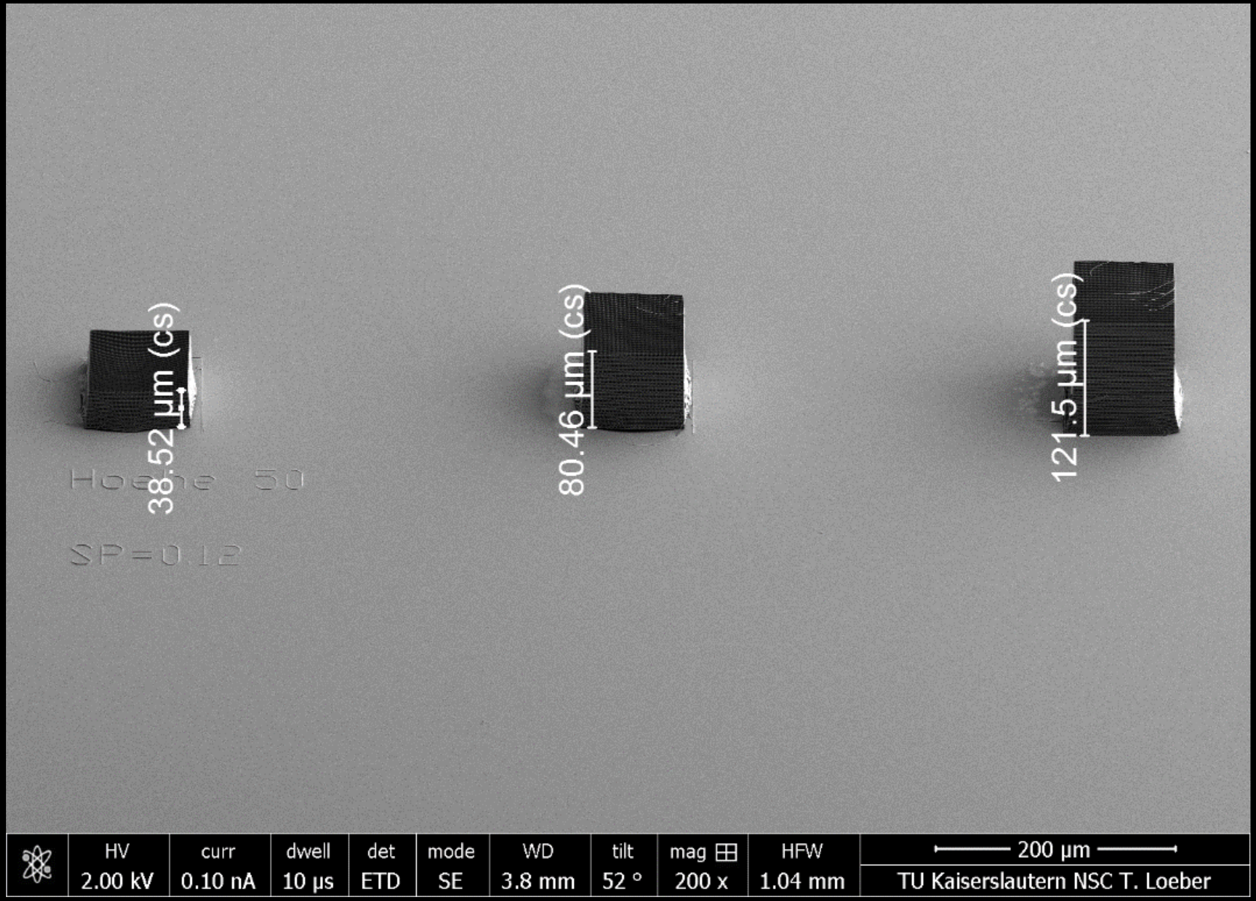
| Energy (kV) | Current (pA) | Spot Size (1-$\sigma$ nm) |
|---|---|---|
| 5 | 3.5 | 15 |
| 2 | 3.5 | 44 |

[1] A V Steele *et al* 2017 *Nano Futures* **1** 015005
Open access link: https://iopscience.iop.org/article/10.1088/2399-1984/aa6a48

# Depth of Focus Comparison

(Results on slides that follow)

FEI: SEM image




## "Wood Pile" Nanostructures

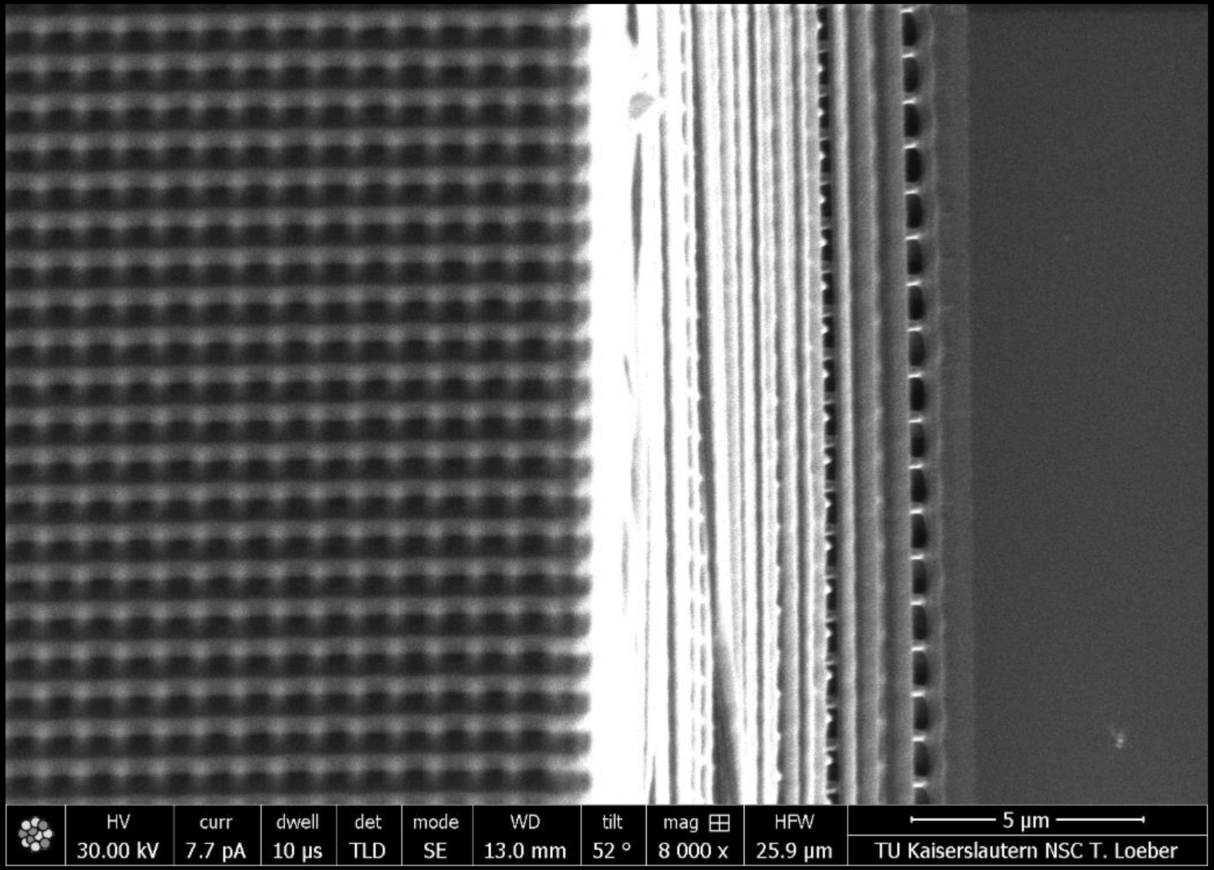
- Heights: 40 µm, 80 µm, 120 µm
- In the following slides we acquire an image containing both the top and bottom of such the 120 µm (tallest) structure
- We can compare the depth of focus of various beams by comparing the 'blurriness' of the top of the structure

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

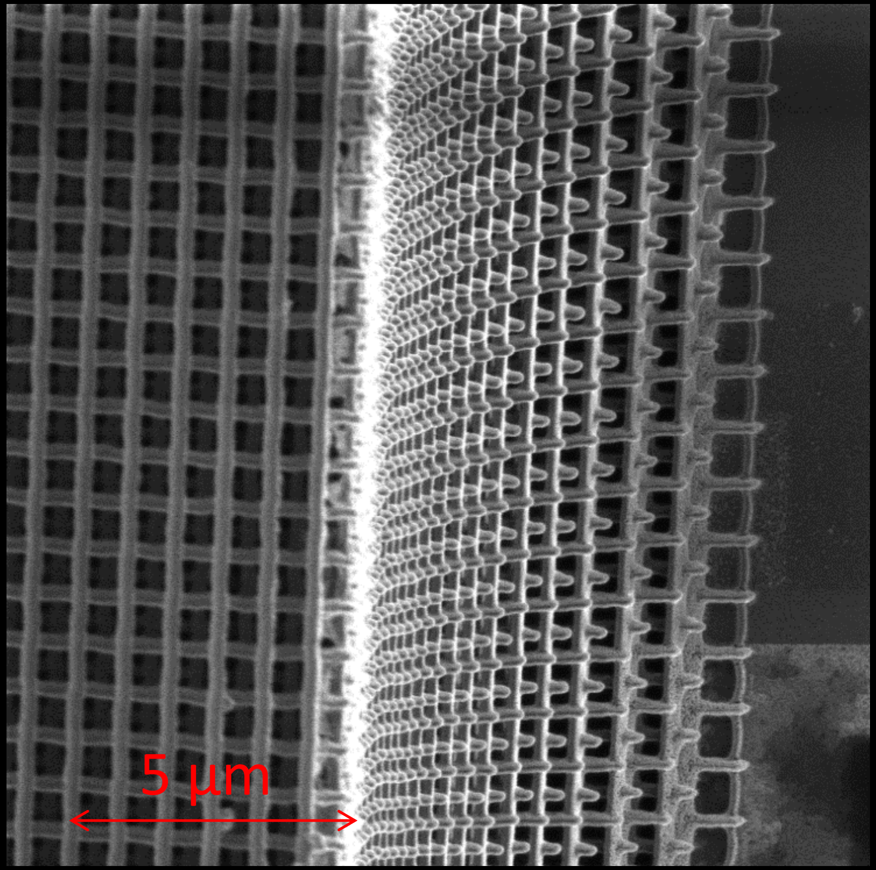
# Depth of Focus Comparison

## →LoTIS depth of focus substantially better than Ga

Ga⁺ LMIS (30 kV)




Cs⁺ LoTIS (10 kV)

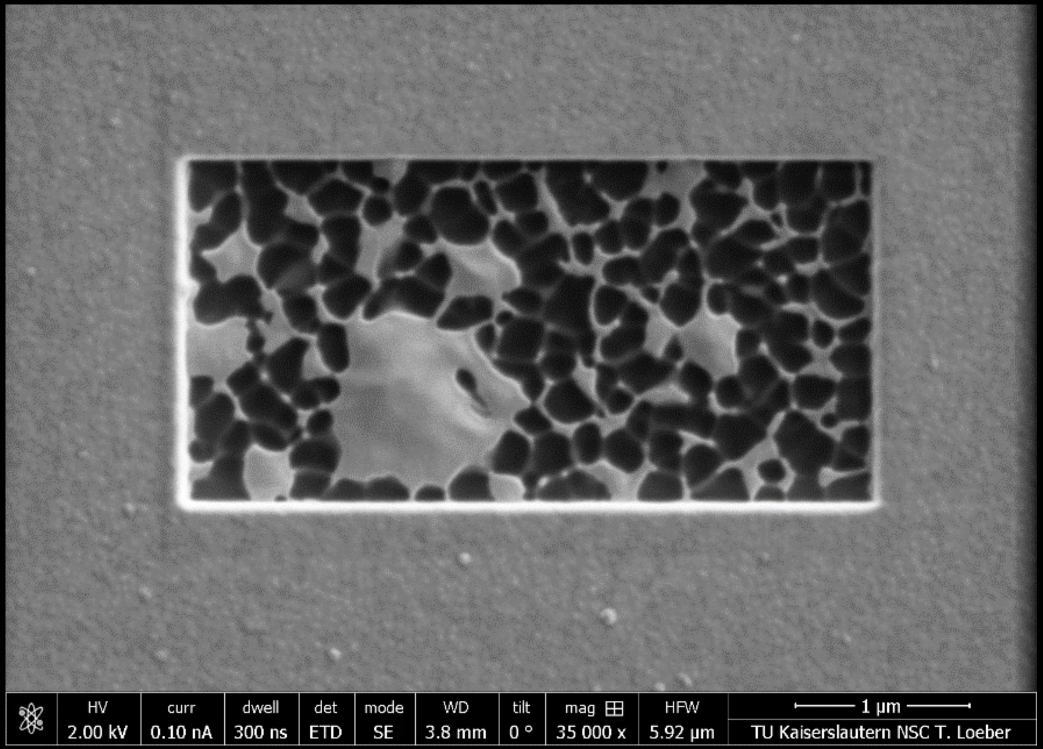



"Wood Pile" Height 120 µm

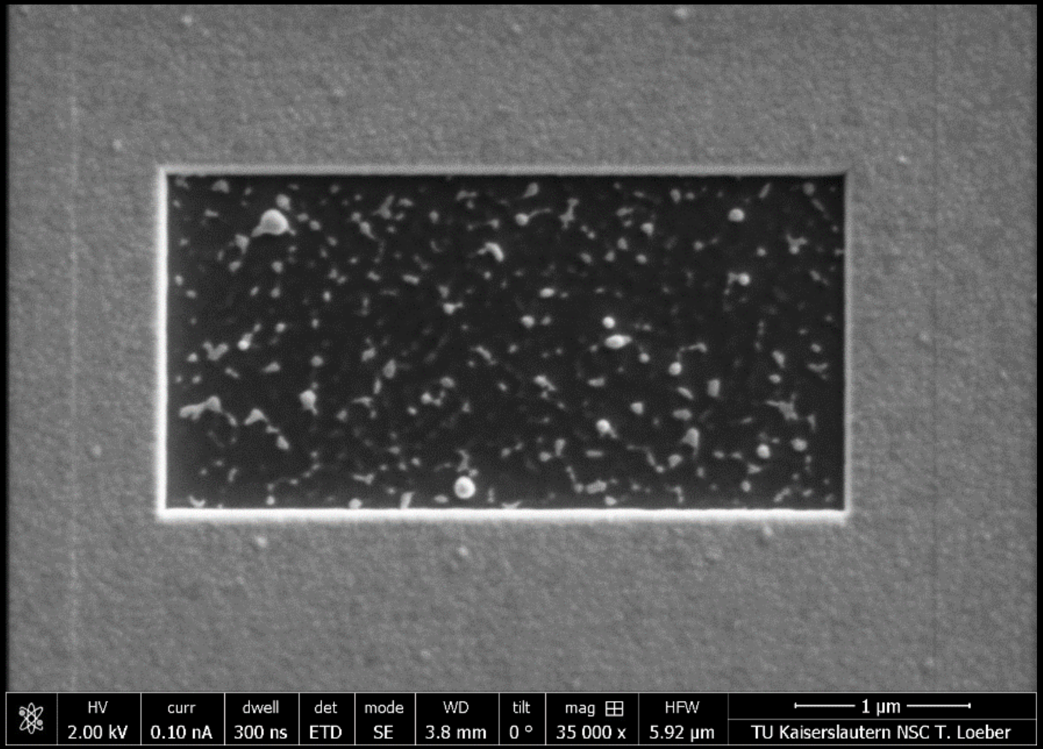
# Milling Homogeneity: 150 nm Au on Si

→$Cs^+$ LoTIS proves even touchdown

Milled with $Ga^+$ LMIS




Milled with $Cs^+$ LoTIS

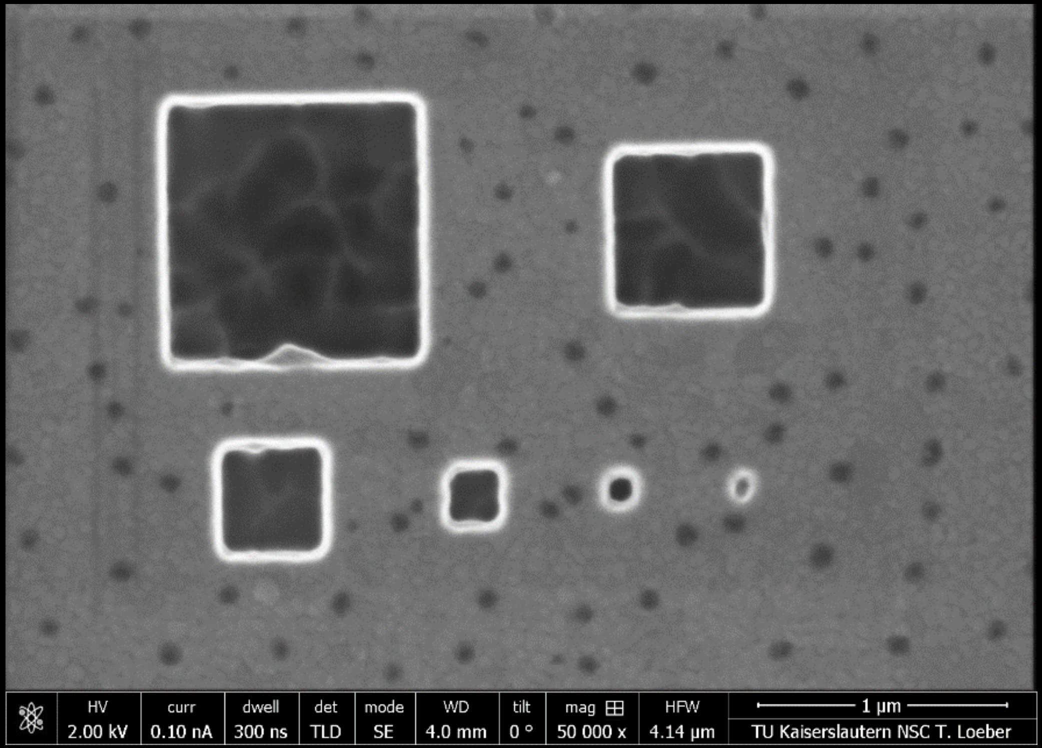



- milled rectangle ‘almost through’ the Au layer
- milling time Ga and Cs almost the same

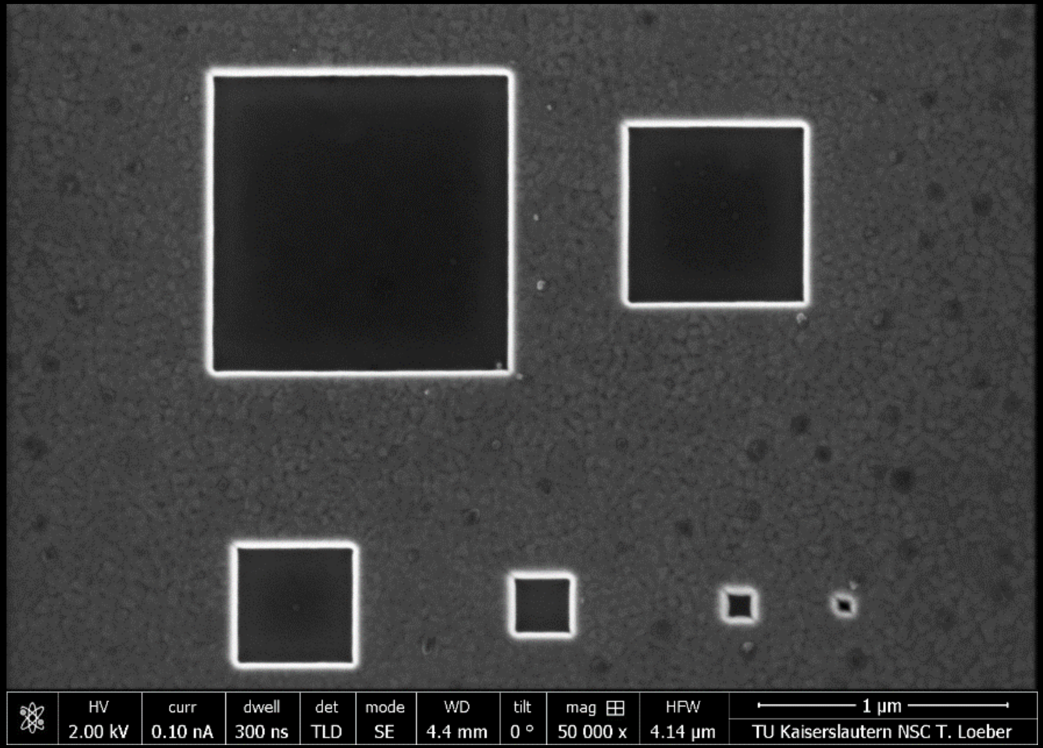
# Milling Accuracy: 110 nm Au on Si

→ LoTIS provides clean mill boxes with sharp corners

Milled with Ga⁺ LMIS



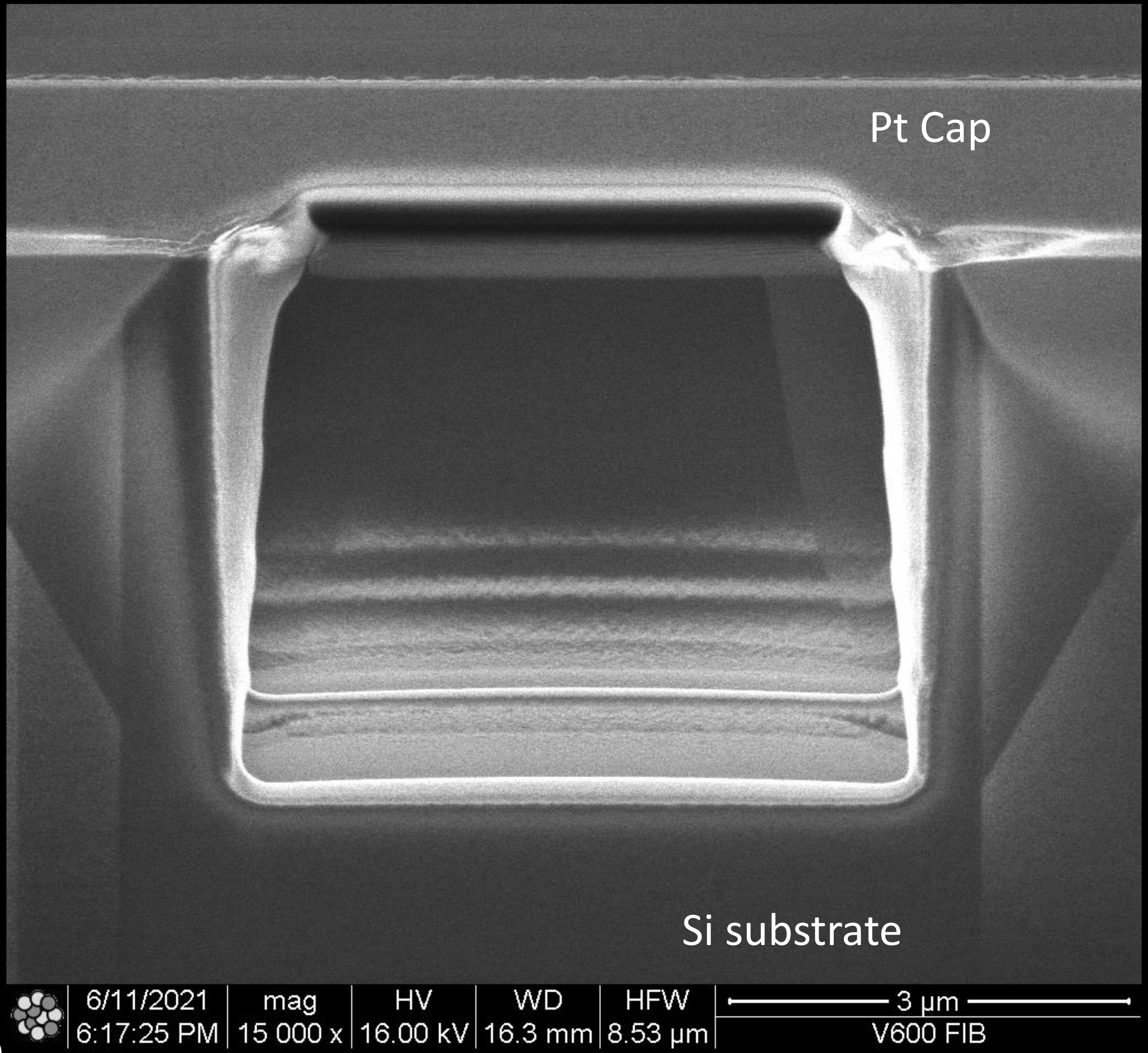

Milled with Cs⁺ LoTIS

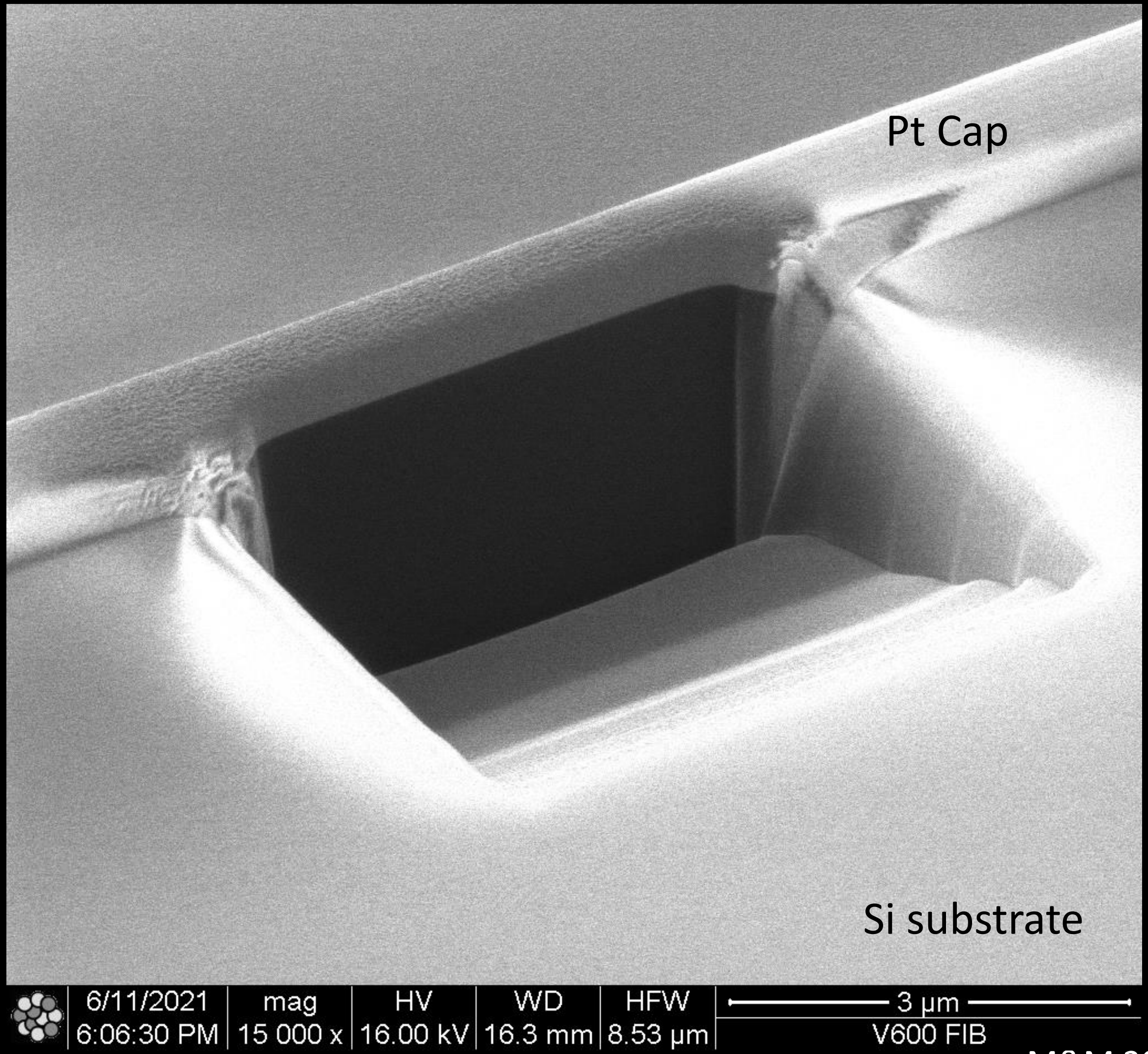



- squares with 1, 0.6, 0.4, 0.2, 0.1 and 0.05 µm length
- milled through the Au layer
- milling time Ga and Cs almost the same

# FIB:ZERO Cross Section Example

Done with 200 pA beam, 30 pA 'cleanup' afterwards

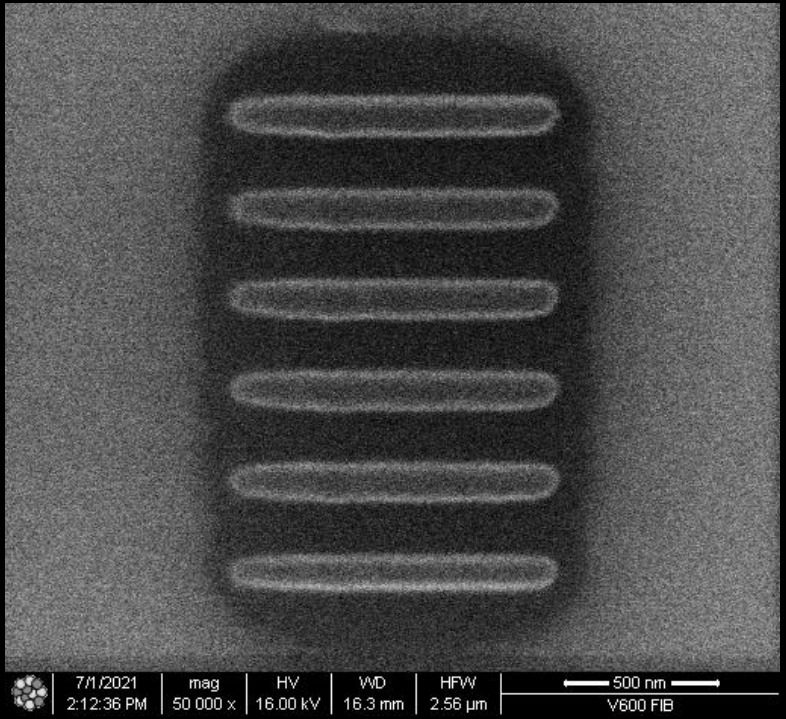


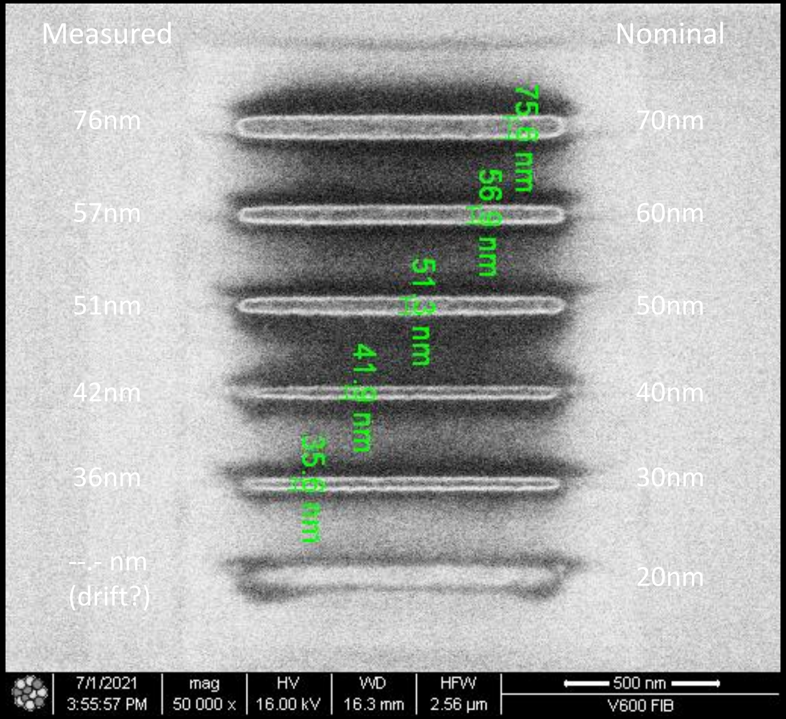
# Platinum Deposition – Narrow Lines

→ FIB:ZERO can provide very narrow metal lines

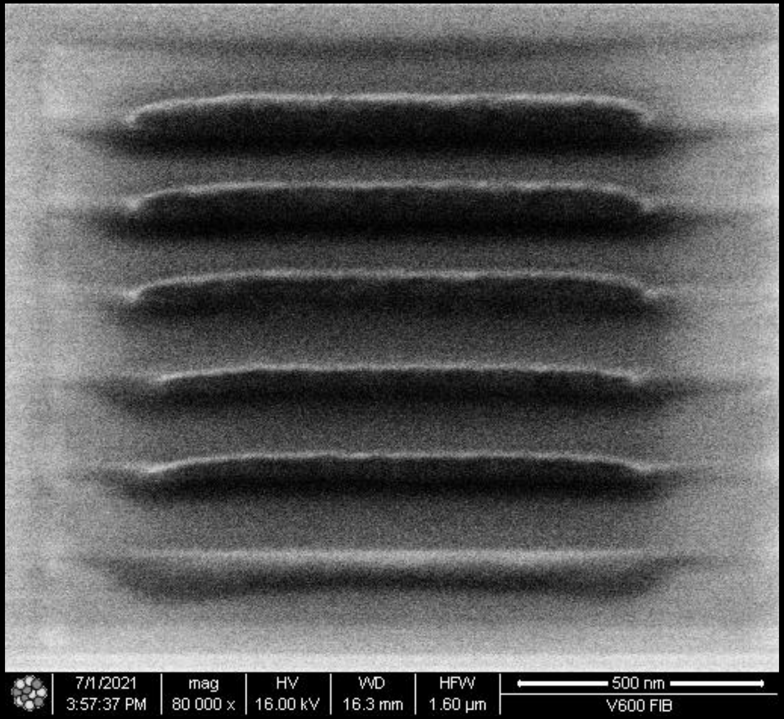
Pt lines with 100nm width as deposited on Si.




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.

# Pain Points of 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**

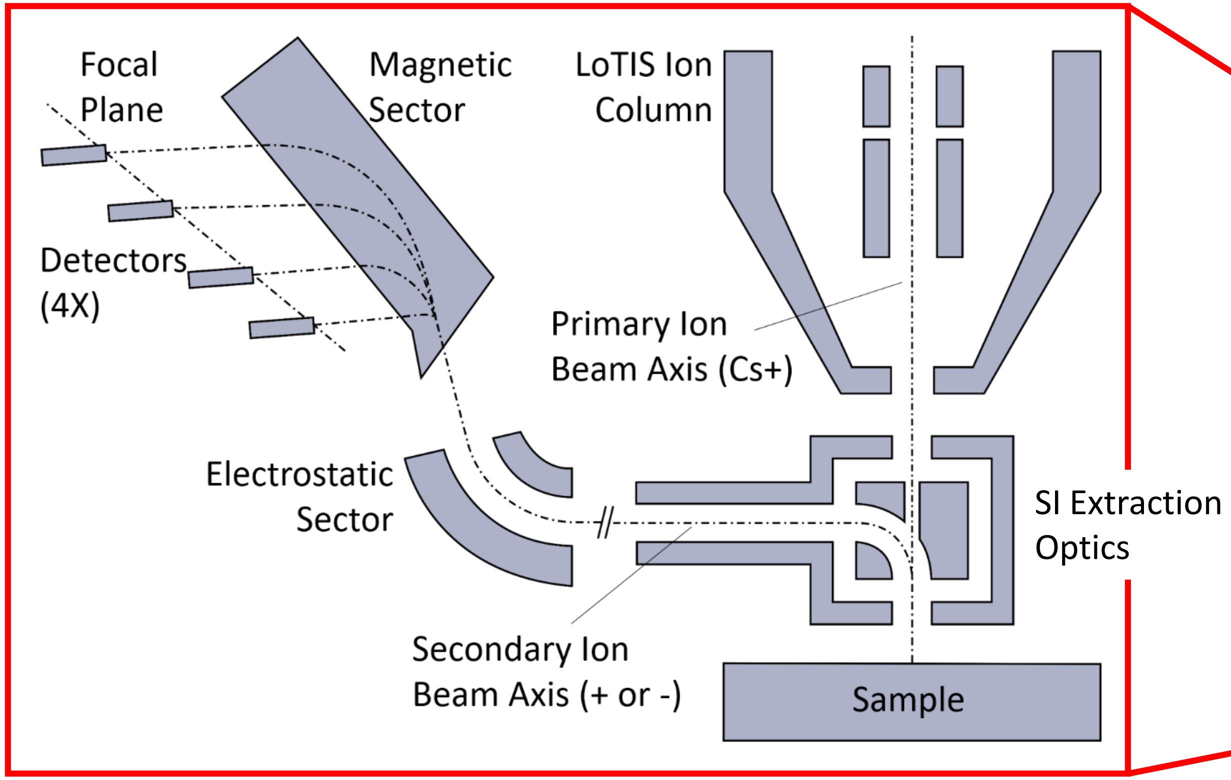
- Few-nanometer resolution (slide 17)
- High secondary ion yield (slides 19,20)
- Integrated sample-prep and analysis capability (slides 21-27)

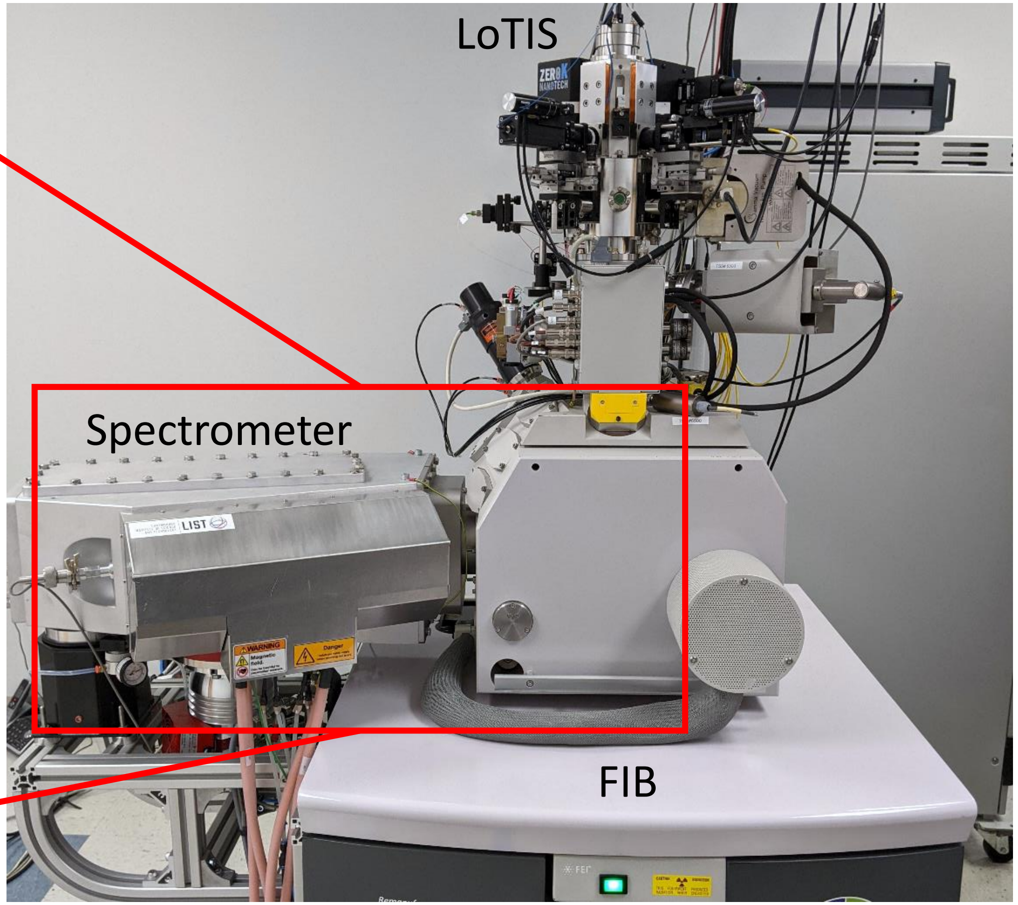
These slides are the first public data presented from SIMS:ZERO

# SIMS:ZERO

## Instrument Overview

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

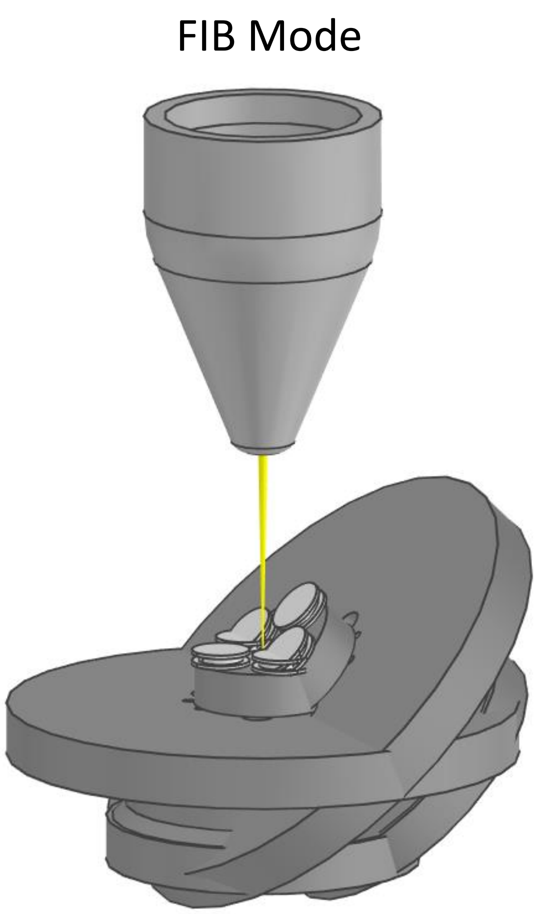


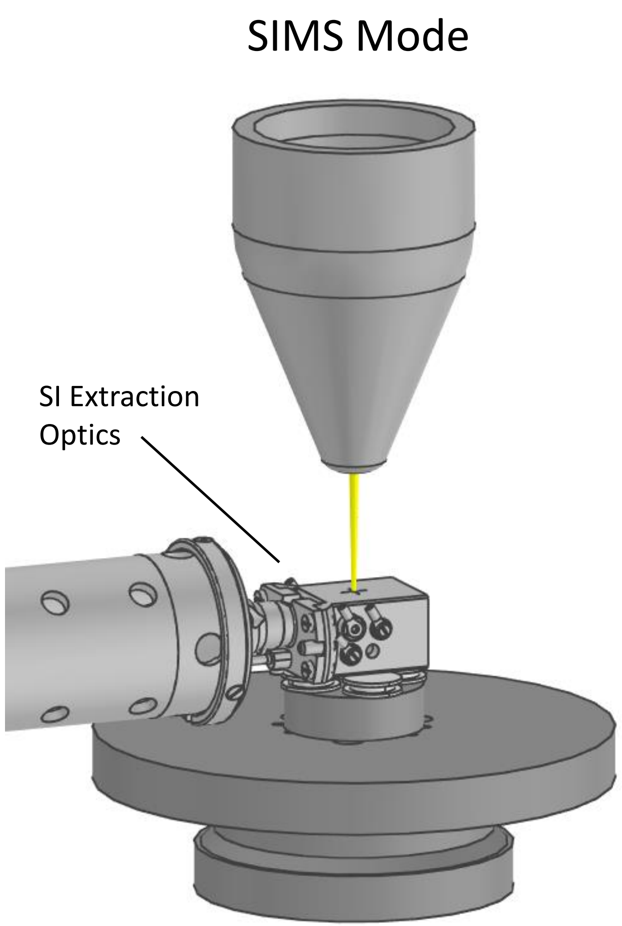




- FIB online 6/2020
- SIMS online 5/2021

# FIB / SIMS Combination

## Sample Prep, Nanofabrication / Analysis, Process Control




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

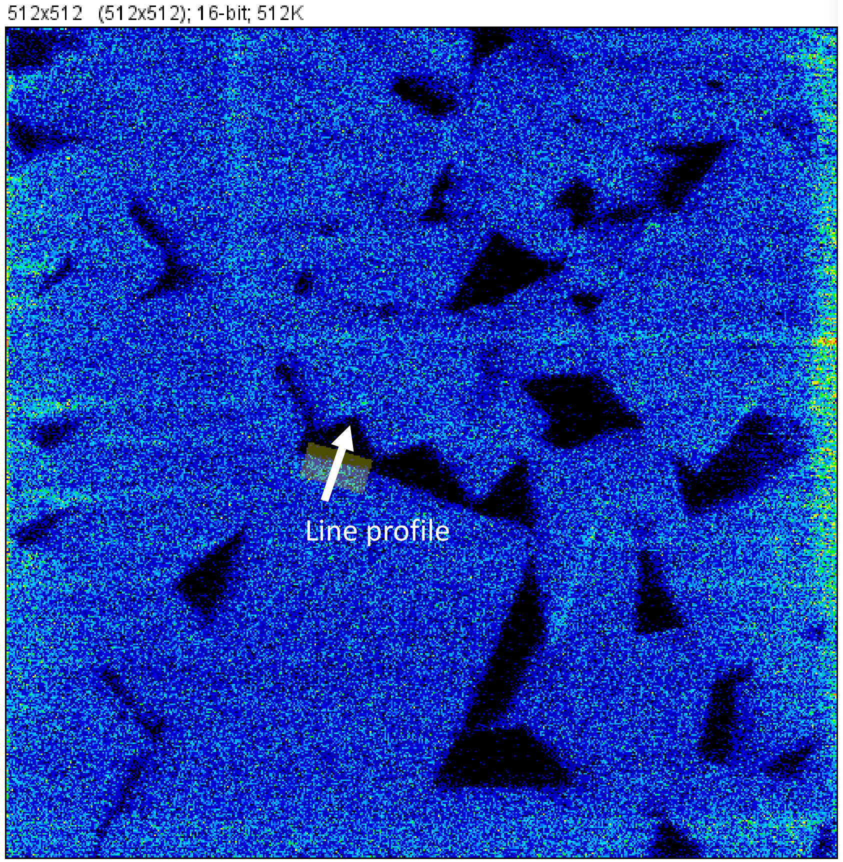
**FIB Mode (SIMS Extraction Optics Retracted)**
- Milling
- Sample Preparation (eg Sectioning, Polishing)
- Nanofabrication
- Gas-assisted processes (eg Platinum Deposition)
- Tilt stage

**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)

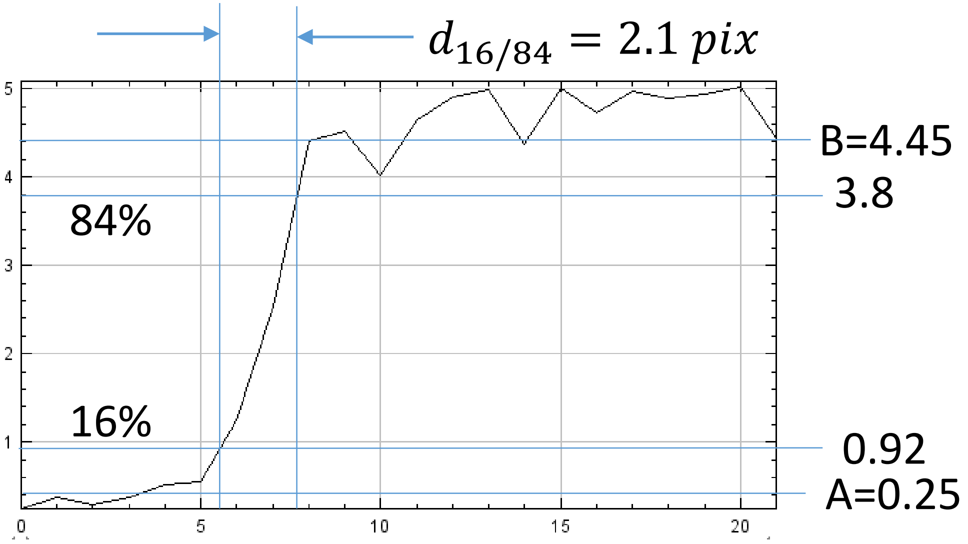
# SIMS:ZERO Resolution

## 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




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

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

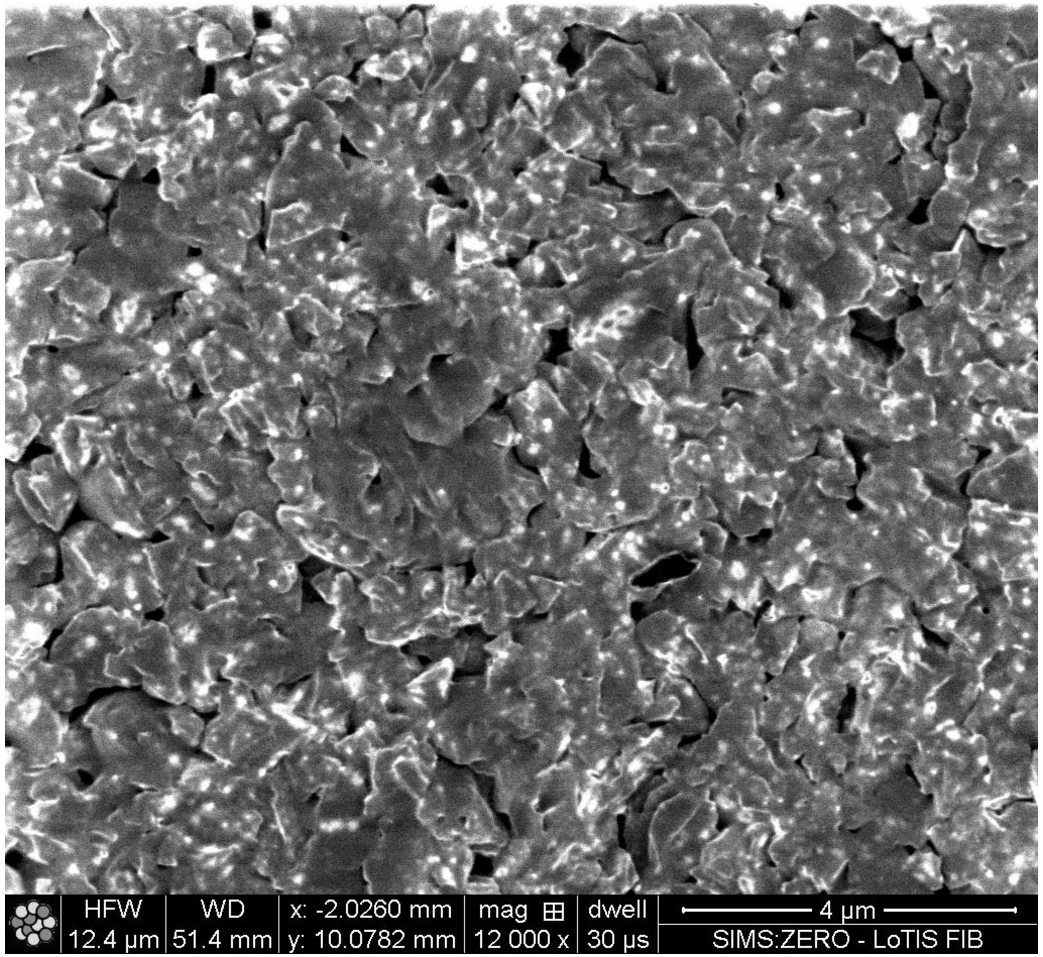
Working Distance = 51.6mm
272s acquisition time.

Negative Ions

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

# SIMS Analysis Example

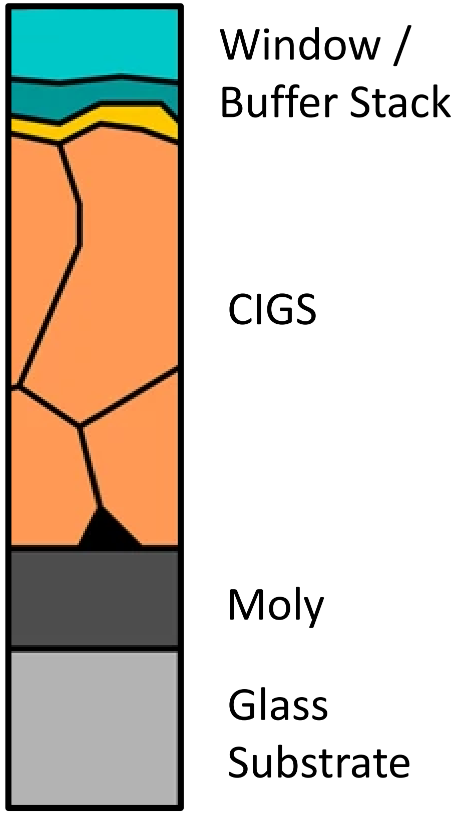
## CIGS $Cu(In,Ga)Se_2$ – Rb doped




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

## Summary

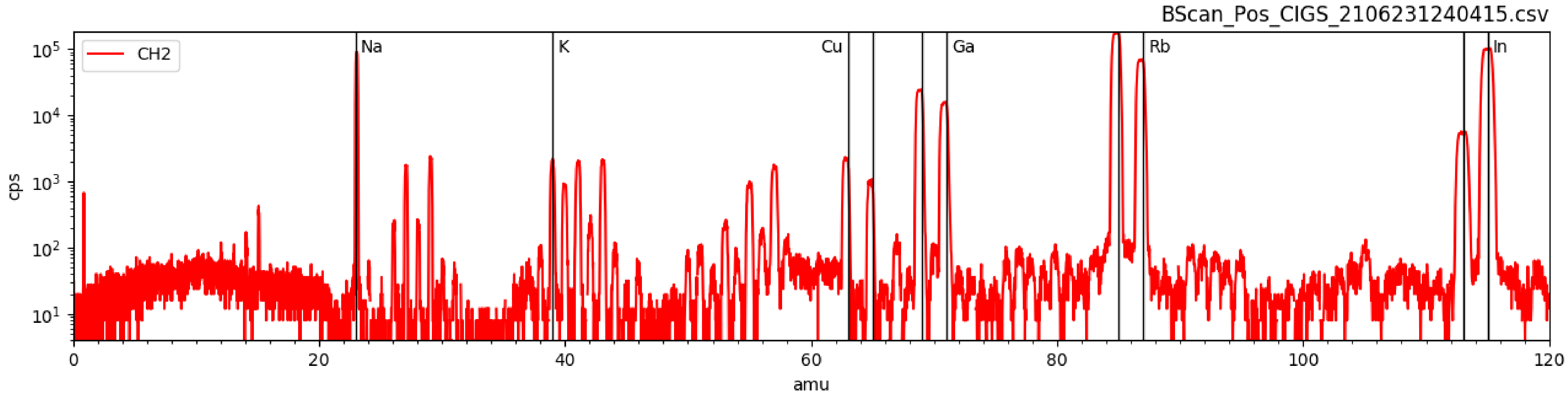
- CIGS is a solar cell absorber material
  - Rubidium doping increases conversion efficiency
- SIMS spectra clearly show all CIGS elements:
  - Cu, In, Ga, Rb in Positive Mode
  - Se in Negative Mode
- Secondary ion imaging channels show distribution of elements in sample, eg Rb dopants concentrated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

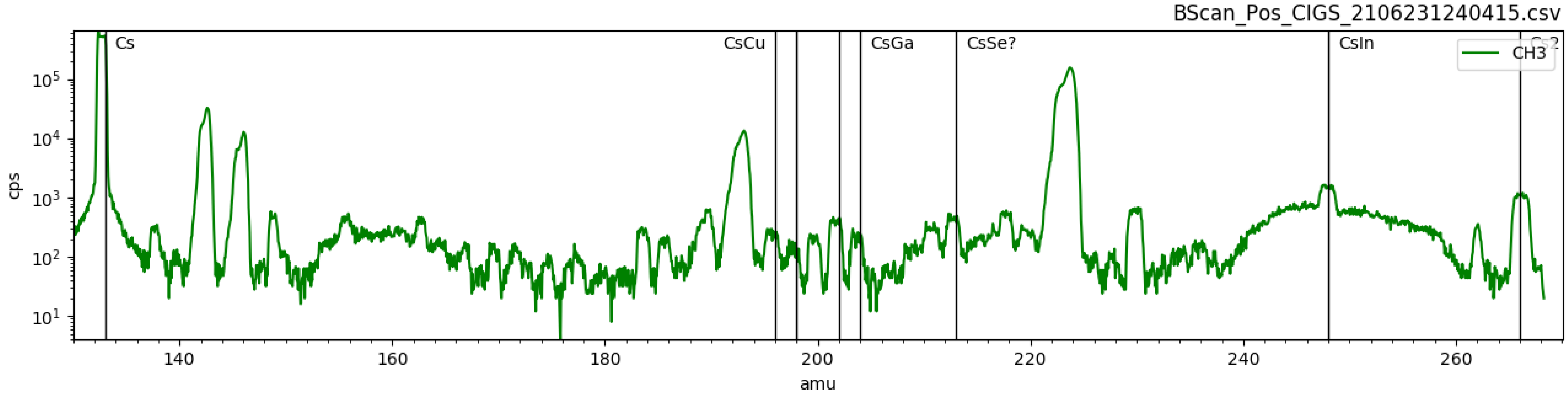



Werner, et al. *Scientific Reports* **volume 10**, 7530 (2020)

# CIGS $Cu(In,Ga)Se_2$ – Rb doped

## Mass Spectra – Positive Ions



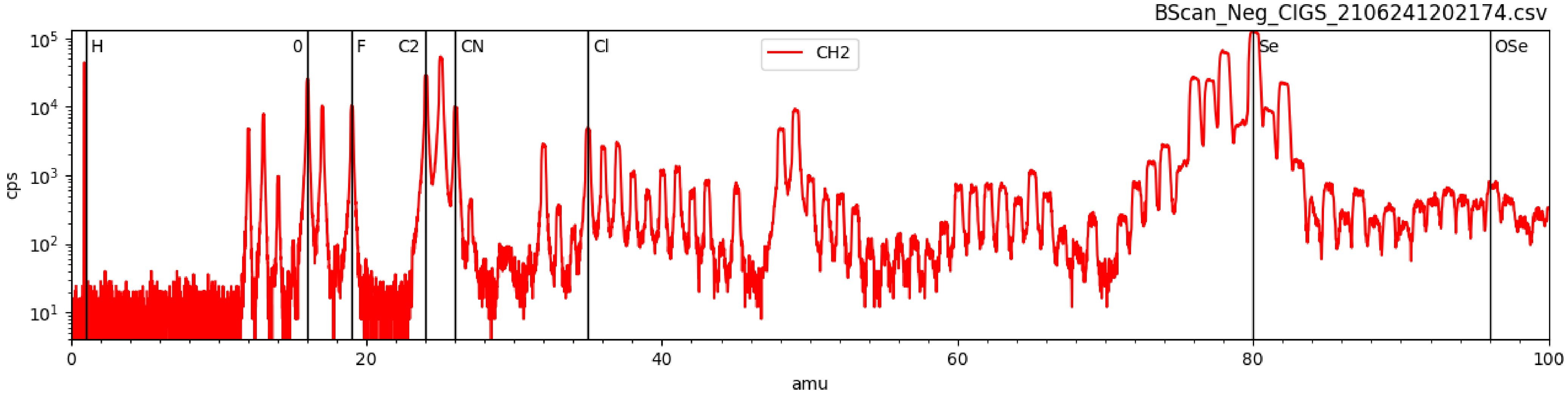



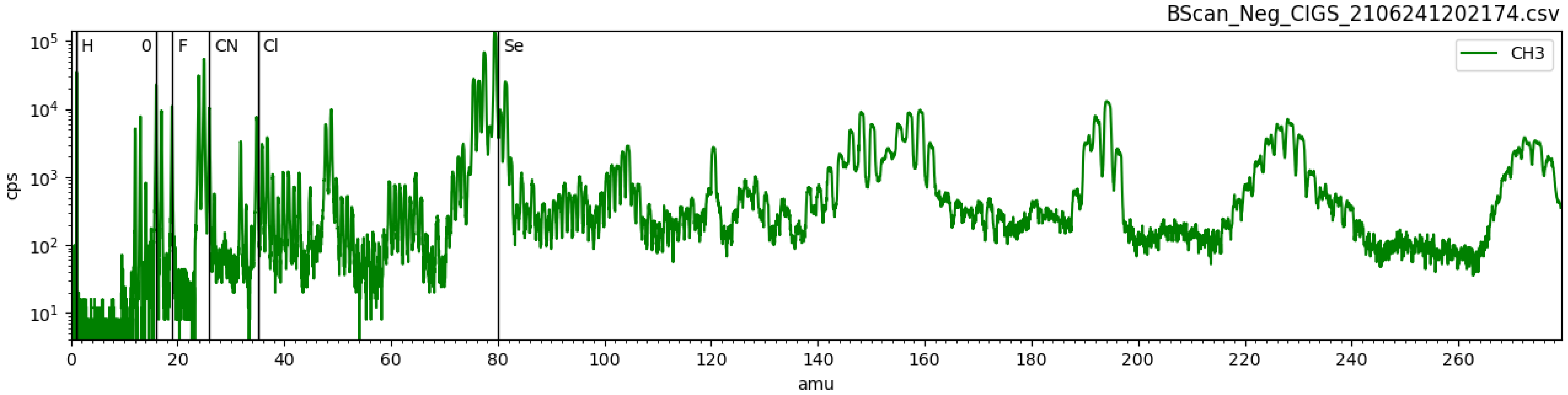

Start (mT) : 30.000000
Stop (mT) : 700.000000
Delta (mT) : 0.100000
Sampling rate (ms) : 250.000000
Waiting time (s) : 0.250000
Period of beam acq : 0
Pos CH1 : 100.000675
Pos CH2 : 200.000362
Pos CH3 : 299.999717
Pos CH4 : 390.000000

| | |
|---|---|
| Date | 06/23/2021 |
| Sample | CIGS |
| Aperture Slit | 100um |
| FOV (um) | 43 |
| I (pA) | 10 |
| U (kV) | 16 |

# CIGS Cu(In,Ga)$Se_2$ – Rb doped

## Mass Spectra – Negative Ions



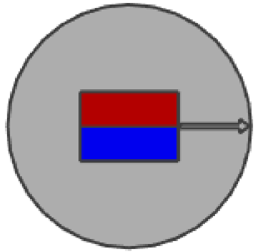




Start (mT) : 30.000000
Stop (mT) : 700.000000
Delta (mT) : 0.100000
Sampling rate (ms) : 250.000000
Waiting time (s) : 0.250000
Period of beam acq : 0
Pos CH1 : 100.000675
Pos CH2 : 200.000362
Pos CH3 : 299.999717
Pos CH4 : 390.000000

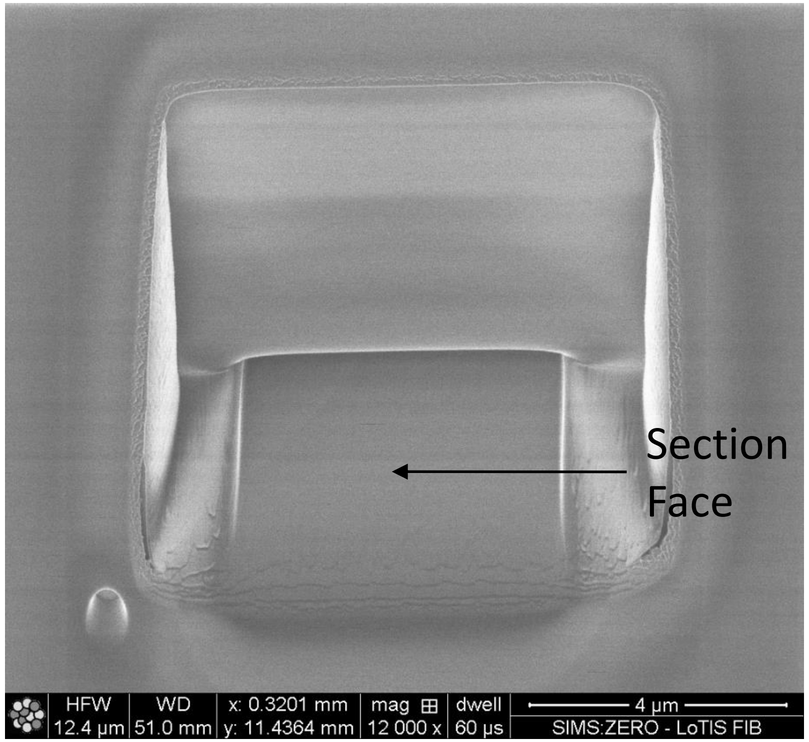
| | |
|---|---|
| Date | 06/24/2021 |
| Sample | CIGS |
| Aperture Slit | 100um |
| FOV (um) | 43 |
| I (pA) | 10 |
| U (kV) | 16 |

# SIMS-Compatible Section View

## 45° Angle Cut - Example



View with Sample Normal to Beam; Ready for SIMS on Section



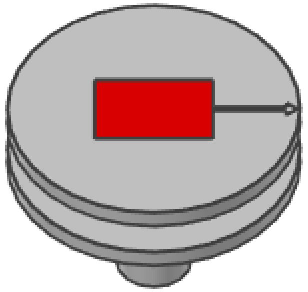

For many samples, working with a section view is a sensible choice

1. Reveal sub-surface structure
2. Obtain depth profile data 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

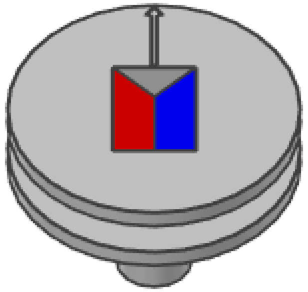
In SIMS:ZERO, sample must be normal to ion beam in SIMS Mode, so section face is cut at 45° to sample surface

# SIMS-Compatible Section View

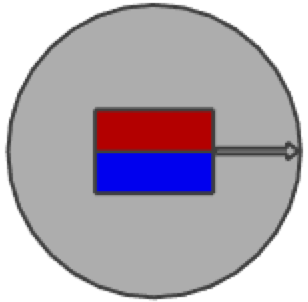
## 45° Angle Cut – Sample Prep Example



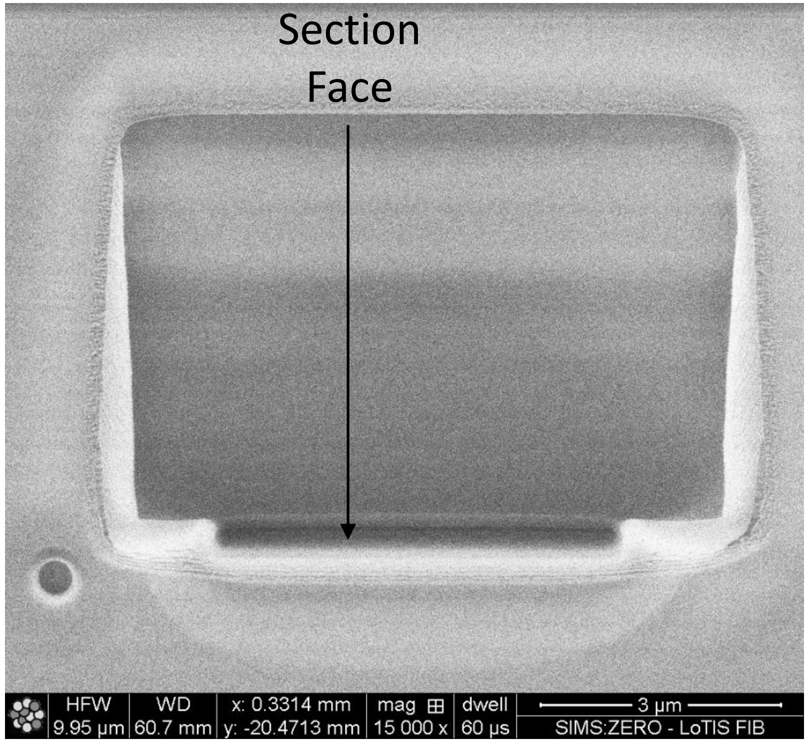
Section Cut with
Sample Tilted at 45°

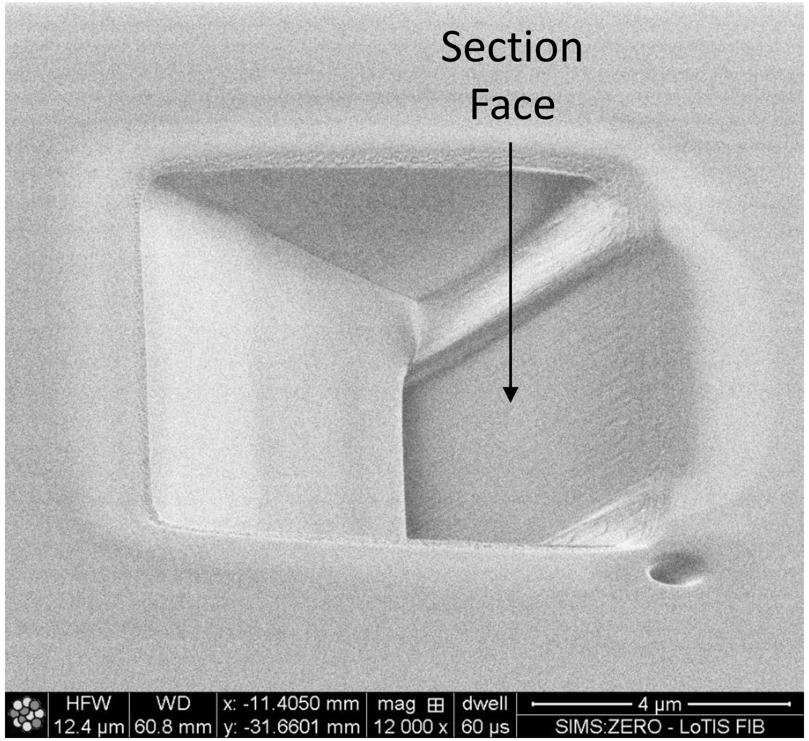


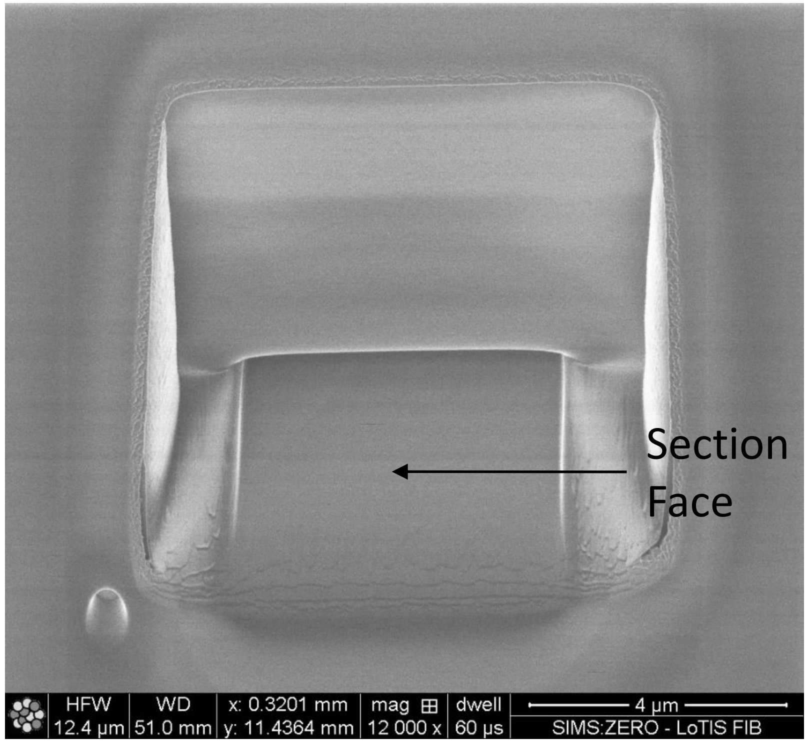
View with
Sample Tilted at 45°
& Rotated 90°



View with Sample
Normal to Beam; Ready
for SIMS on Section



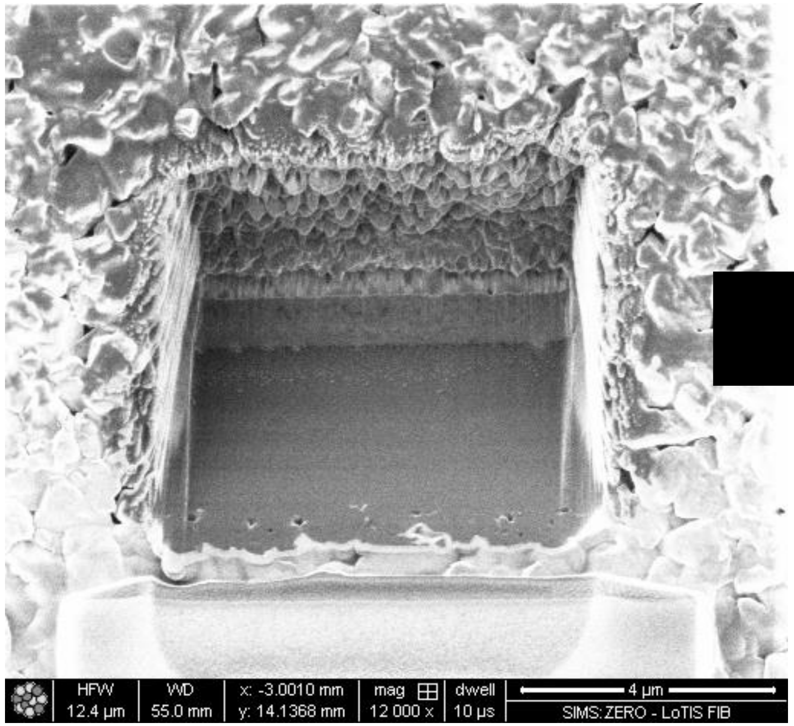



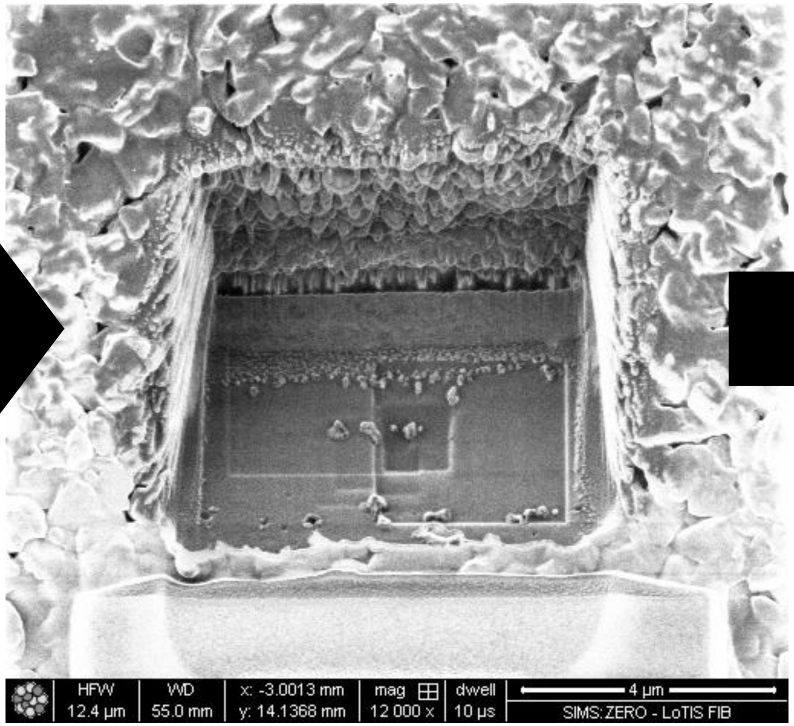
# CIGS Cu(In,Ga)$Se_2$ – Rb doped

## Serial Sectioning / Imaging / Polishing Work-Flow

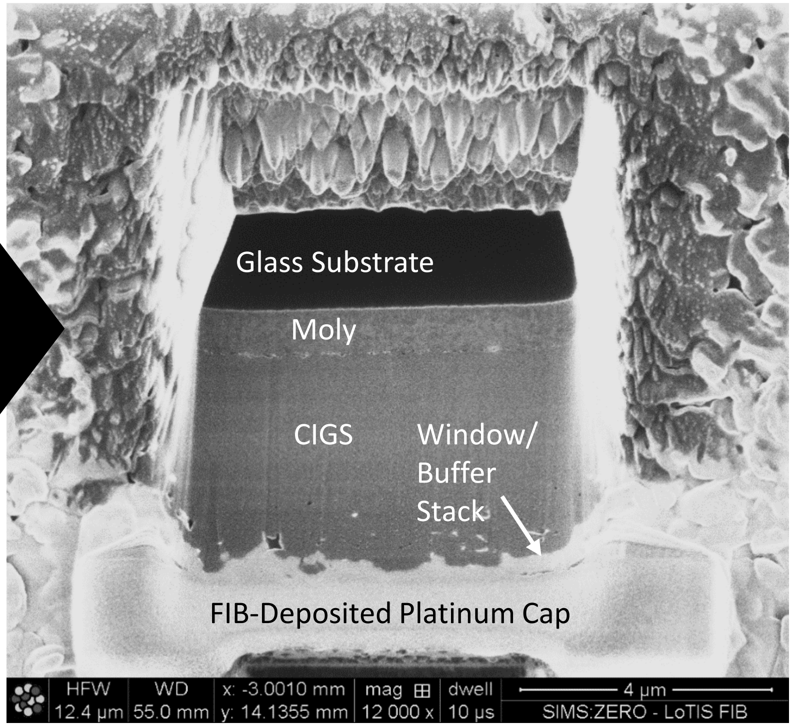
SE Images




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



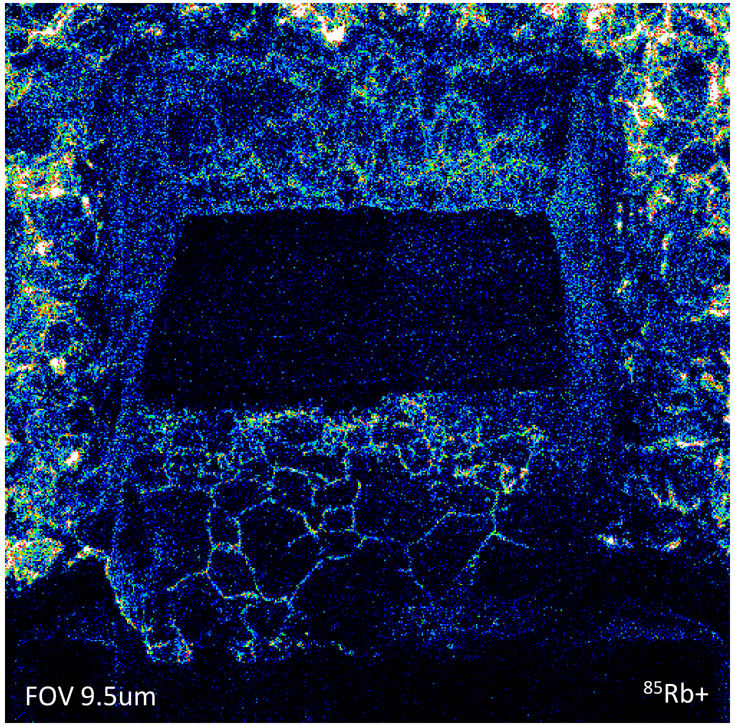

Section face after cleanup mill. Ready for SIMS on next layer

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

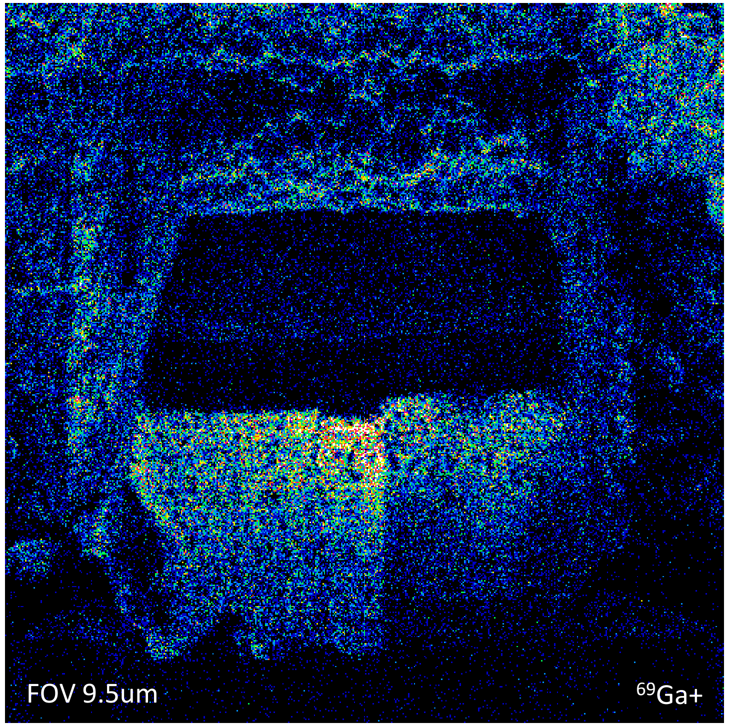
# CIGS Cu(In,Ga)$Se_2$ – Rb doped

## Section View – Positive Ions

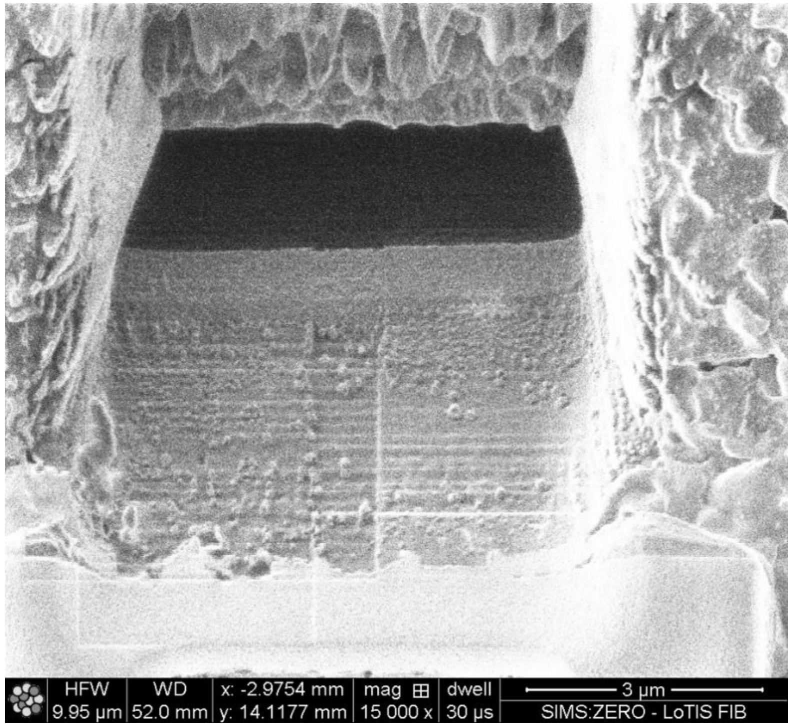
Grain size gradient ↓




Ga concentration gradient ↑



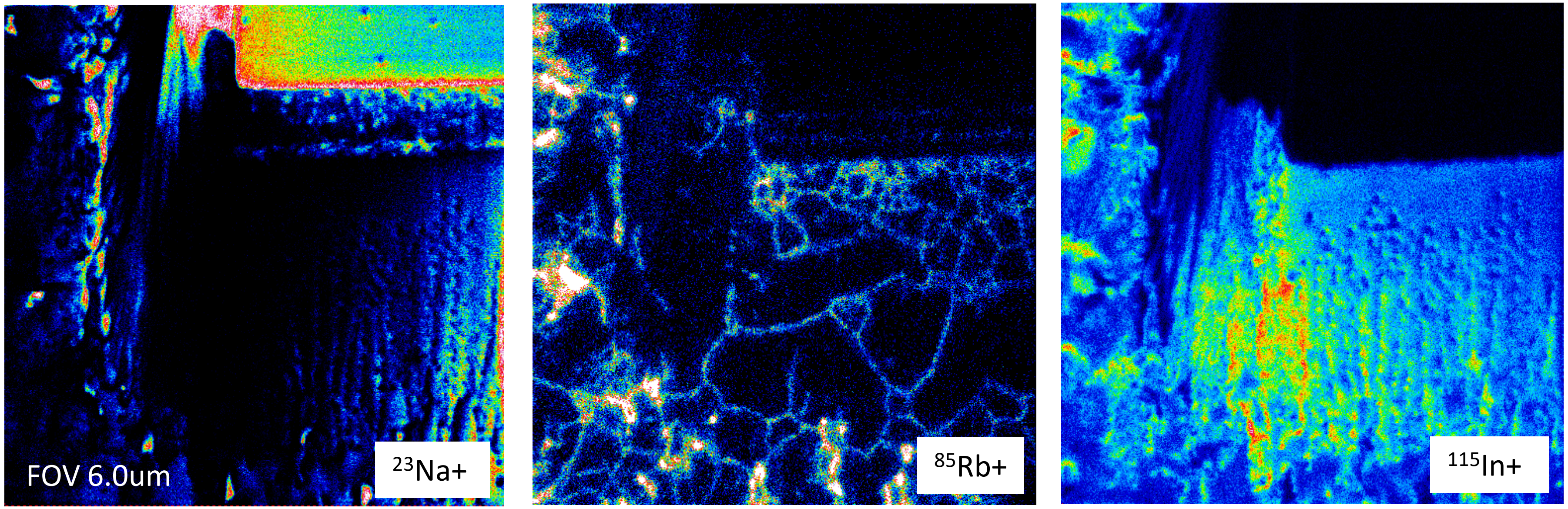

SE Image – Post Imaging




Cs+, 16keV, 3.5pA, 51.6mm WD
CIGS_Pos_2107151438259.csv

# CIGS $Cu(In,Ga)Se_2$ – Rb doped

## Section View – Positive Ions

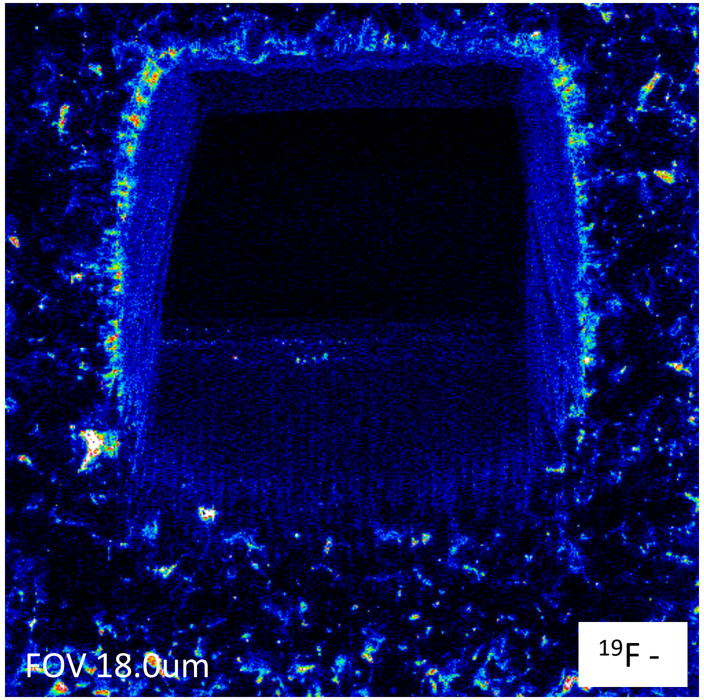


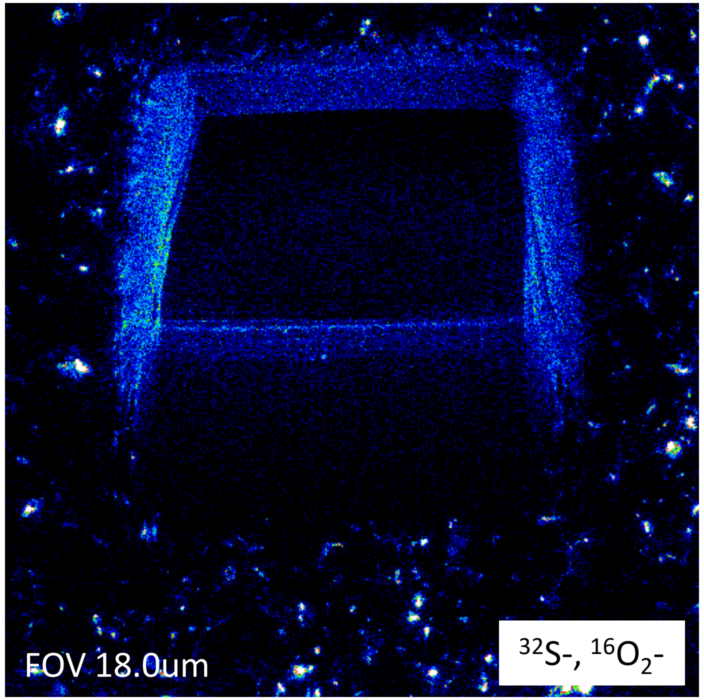

- $Na^+$ signal in glass substrate (soda-lime)
- $Rb^+$ in CIGS grain boundaries
  - Grain sizes are smaller near Molybdenum (Moly) layer
- $Na^+$, $Rb^+$ in Moly layer
- $In^+$ signal confined to the CIGS & Buffer / Window Stack

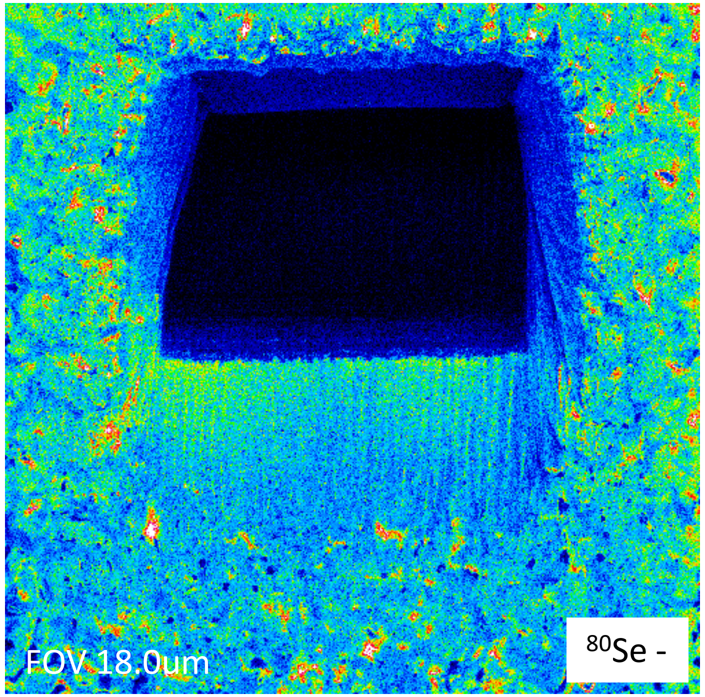
Cs+, 16keV, 10pA, 51.6mm WD
CIGS_Pos_2107121154464.csv

# CIGS $Cu(In,Ga)Se_2$ – Rb doped

## Section View – Negative Ions



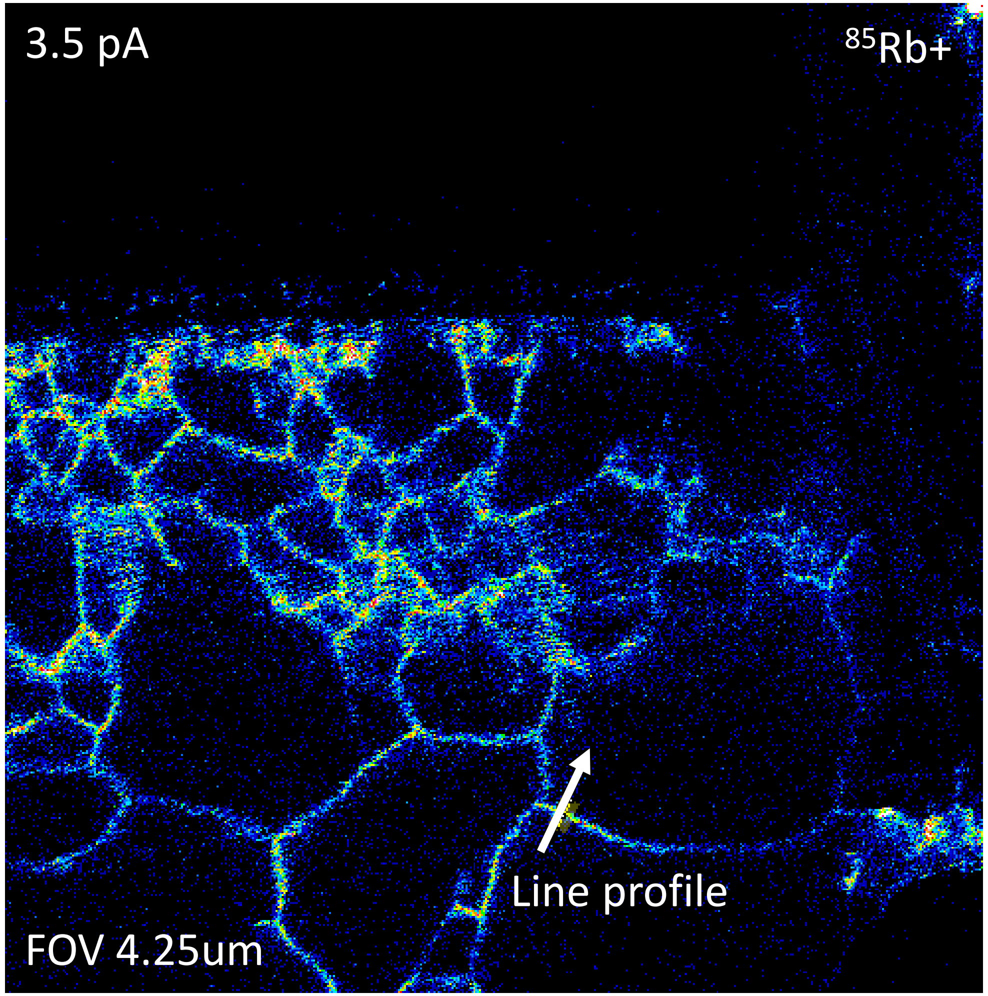



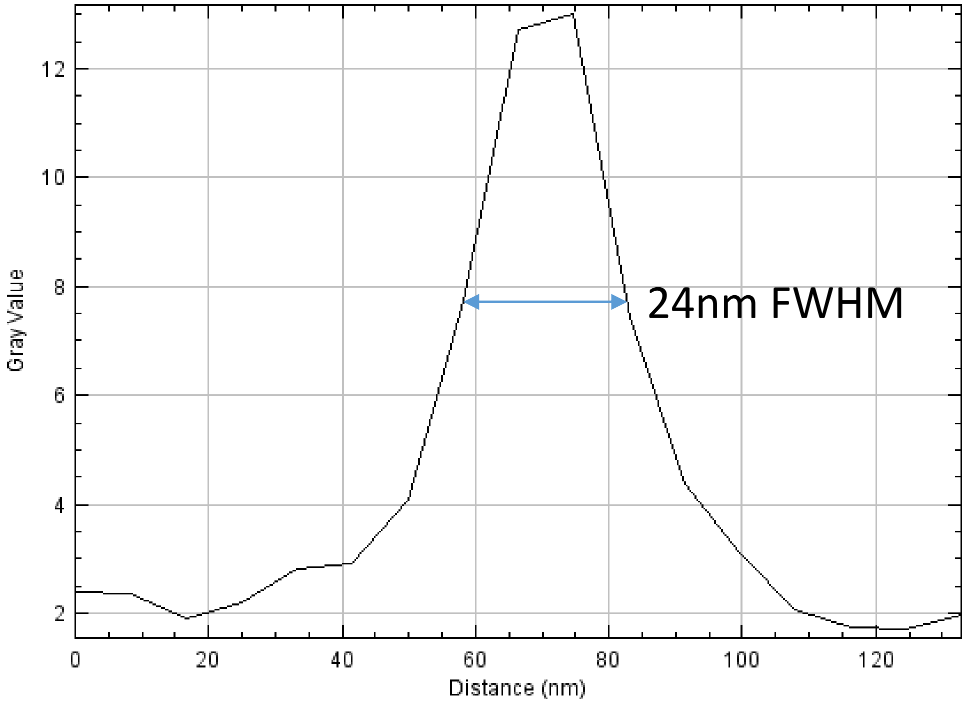

Cs+, 16keV, 10pA, 51.6mm WD
CIGS_Neg_2107121556386.csv

# CIGS $Cu(In,Ga)Se_2$ – Rb doped

## Section View – Positive Ions




Apparent width of Rubidium signal between grains

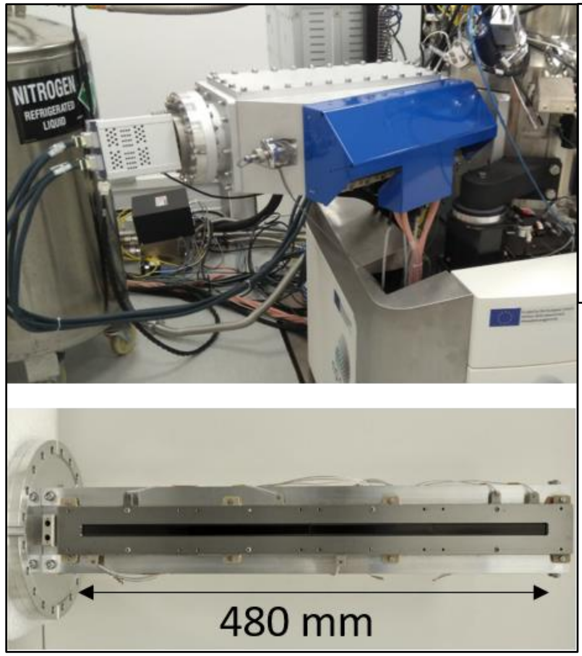



Cs+, 16keV, 3.5pA, 51.6mm WD
CIGS_Pos_2107151409368.csv

# Continuous Detector

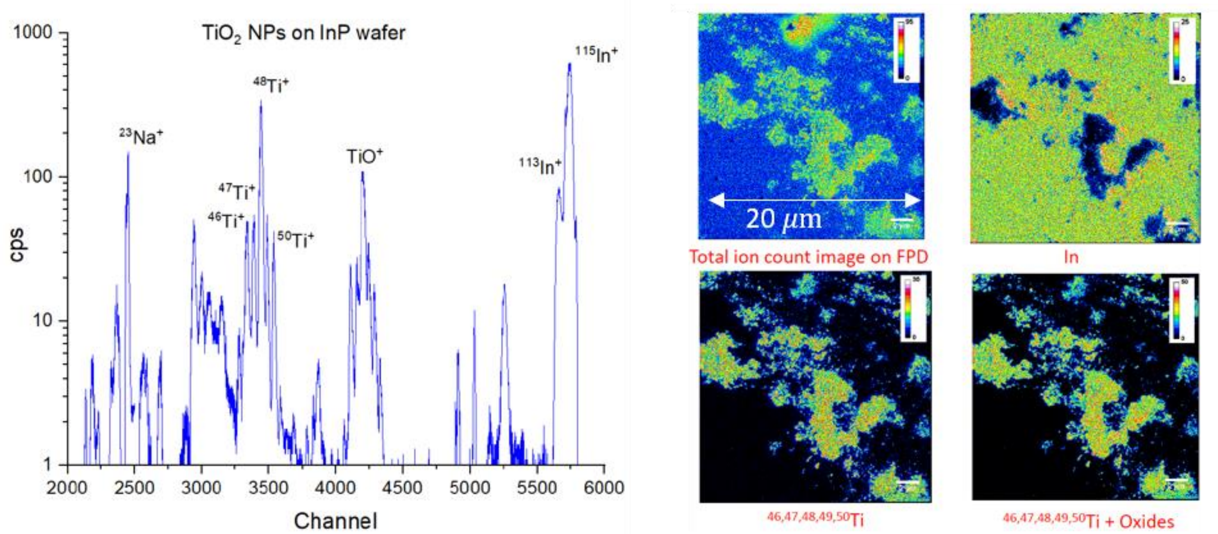
## A SIMS:ZERO Option

- SIMS signals for a given element are split into many lines (e.g. Ti, TiO etc..) .
- In discrete-detector systems this leads to a loss of information and lower SNR.
- With continuous detector technology we can sample the entire mass spectrum at once.
- Now we can collect the entire spectrum as in TOF systems, but without painfully long acquisition times.




(Top) Photo of SIMS spectrometer (at LIST) and the continuous focal plane detector mounted to a vacuum flange.

(Bottom) A 480mm micro-channel plate that spans the focal plane of the spectrometer.




Data from by a continuous focal plane detector. Mass spectrum (Left) and surface compositional maps (Right). The sample under interrogation was titanium oxide nanoparticles on an indium phosphide substrate.