ZEROK NANOTECH

High-Resolution FIB and SIMS with the Cesium Low Temperature Ion Source (LoTIS)

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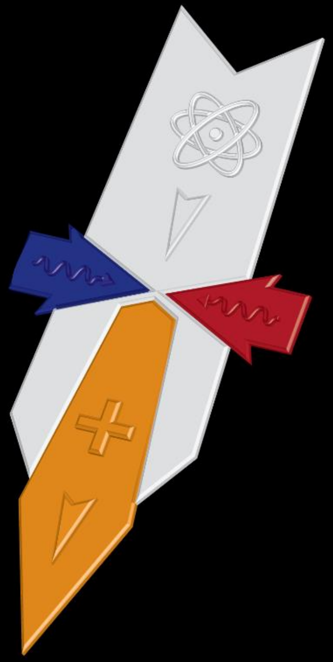
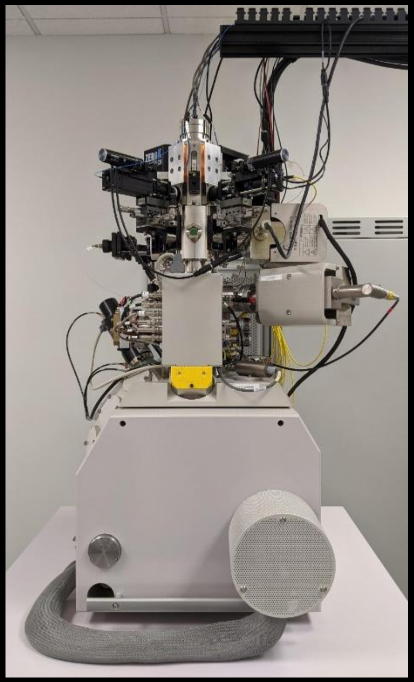
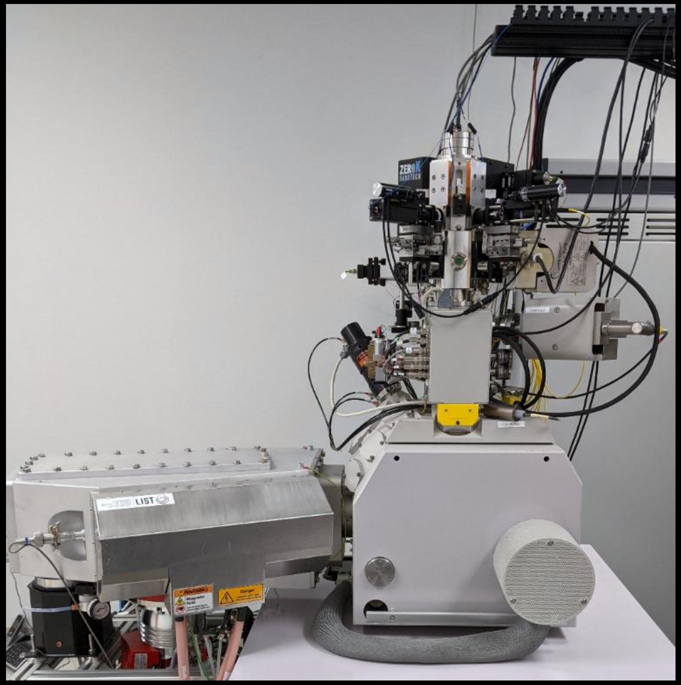
Slides will be posted at zeroK.com/news.html

EIPBN 2024

Technology and Applications







Cs⁺ LoTIS	FIB:ZERO	SIMS:ZERO

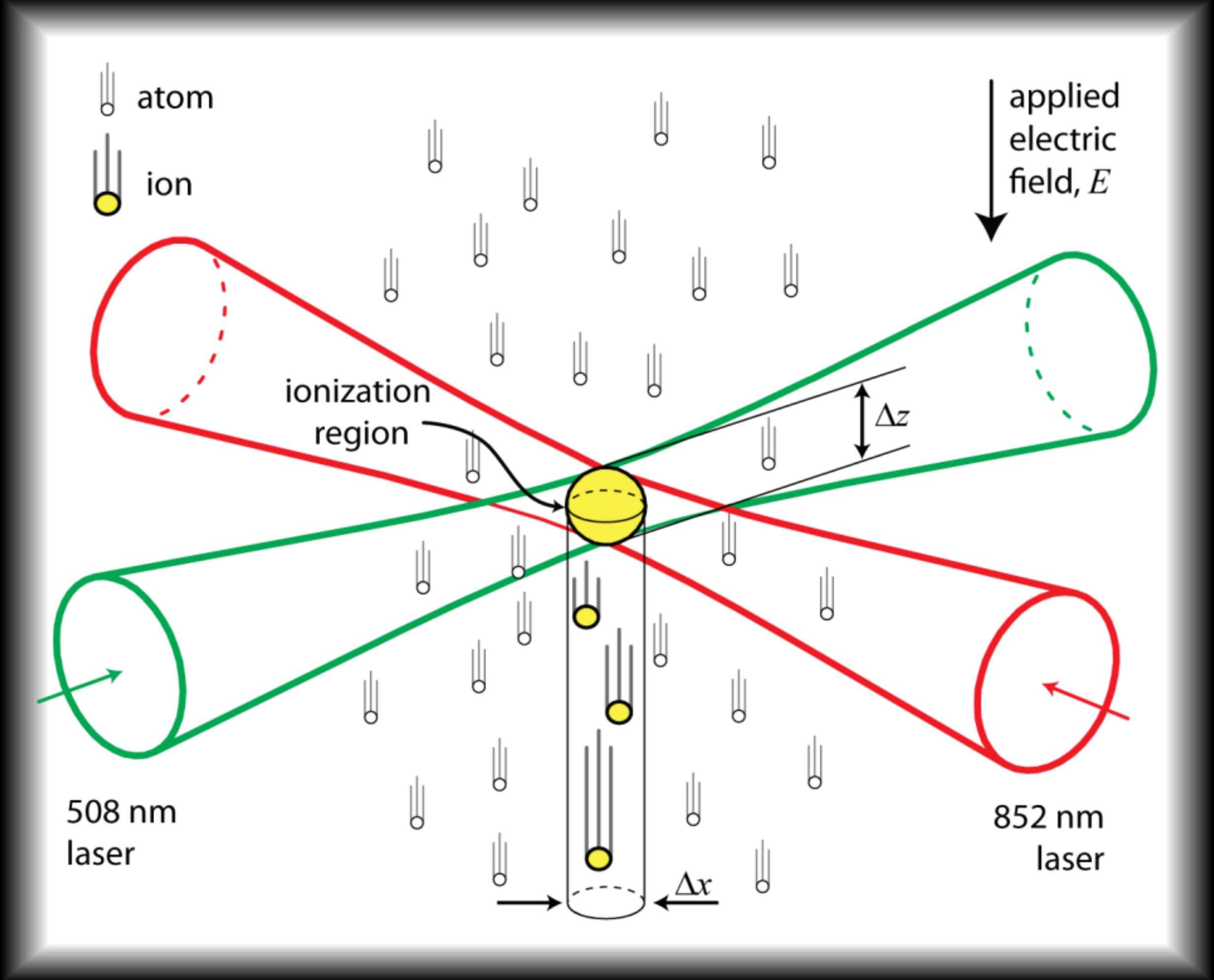
• Low Temperature Ion Source
  • Laser-cooling + Photoionization
• Heavy ion nanomachining
• Small spot sizes
• Excellent resolution at low energy (~2 nm resolution at 1 pA, 16 kV)
• 1 pA - 10 nA
	• LoTIS + FIB
	• Comparable to standard Ga^+ FIB, with 2x higher resolution at low beam currents
	• Compatible 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

Creates only the current to be used.

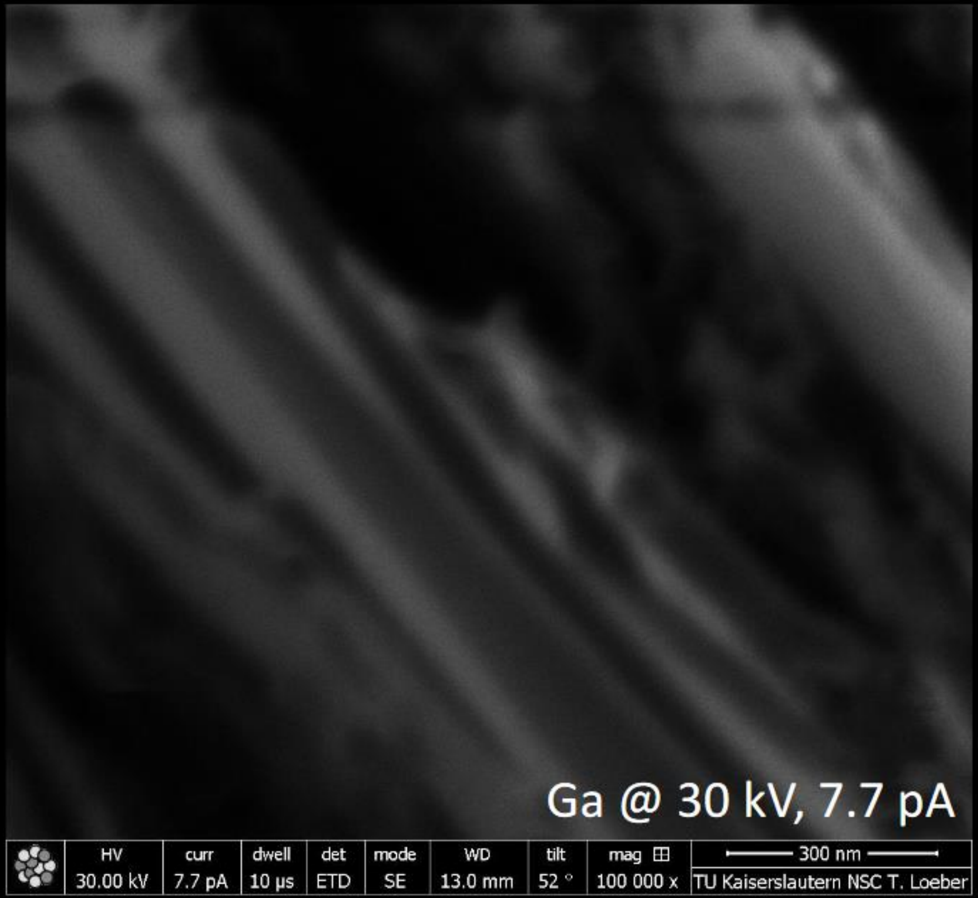


FIB:ZERO

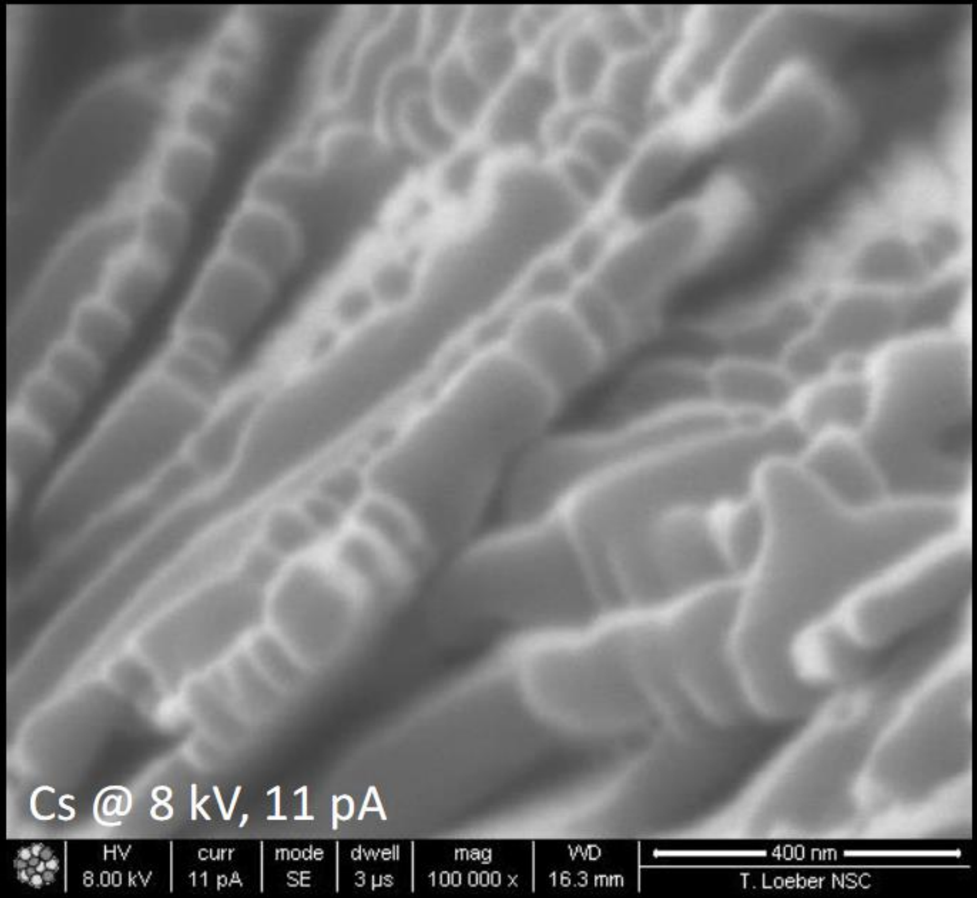
Resolution

PHYSIK

Ga ion image



Cs ion image

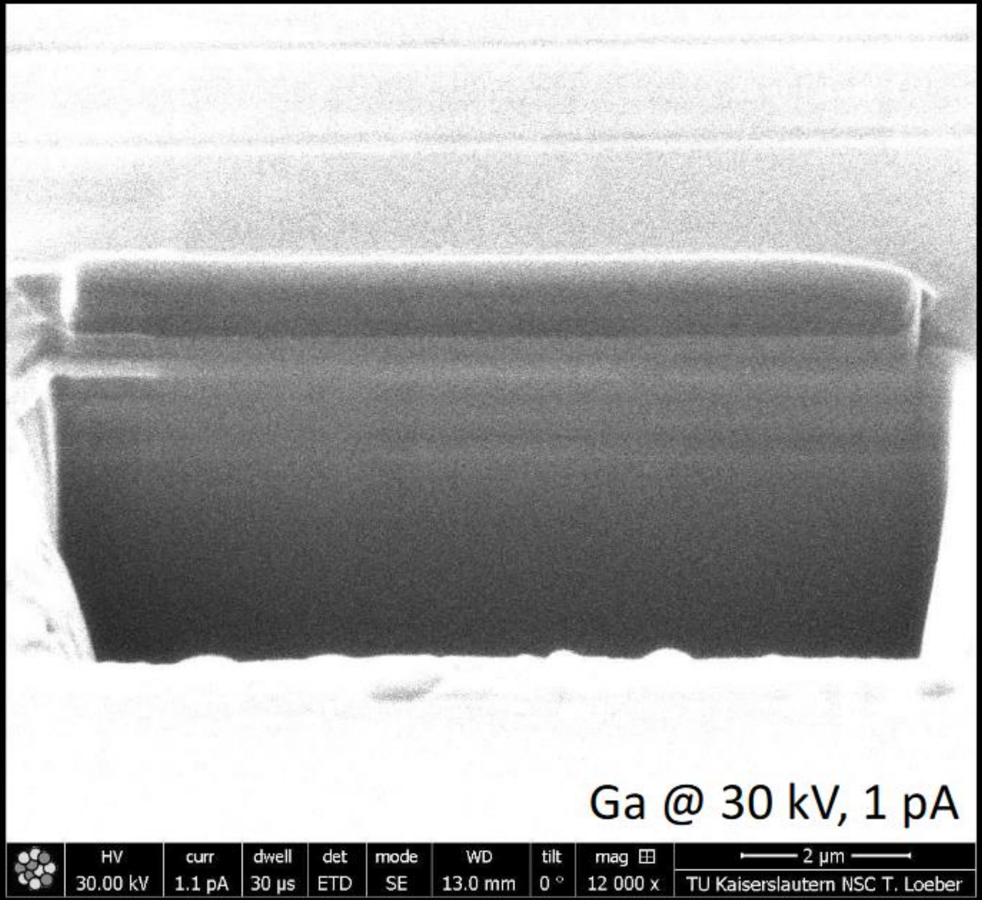


graphite pen: magnification 100k

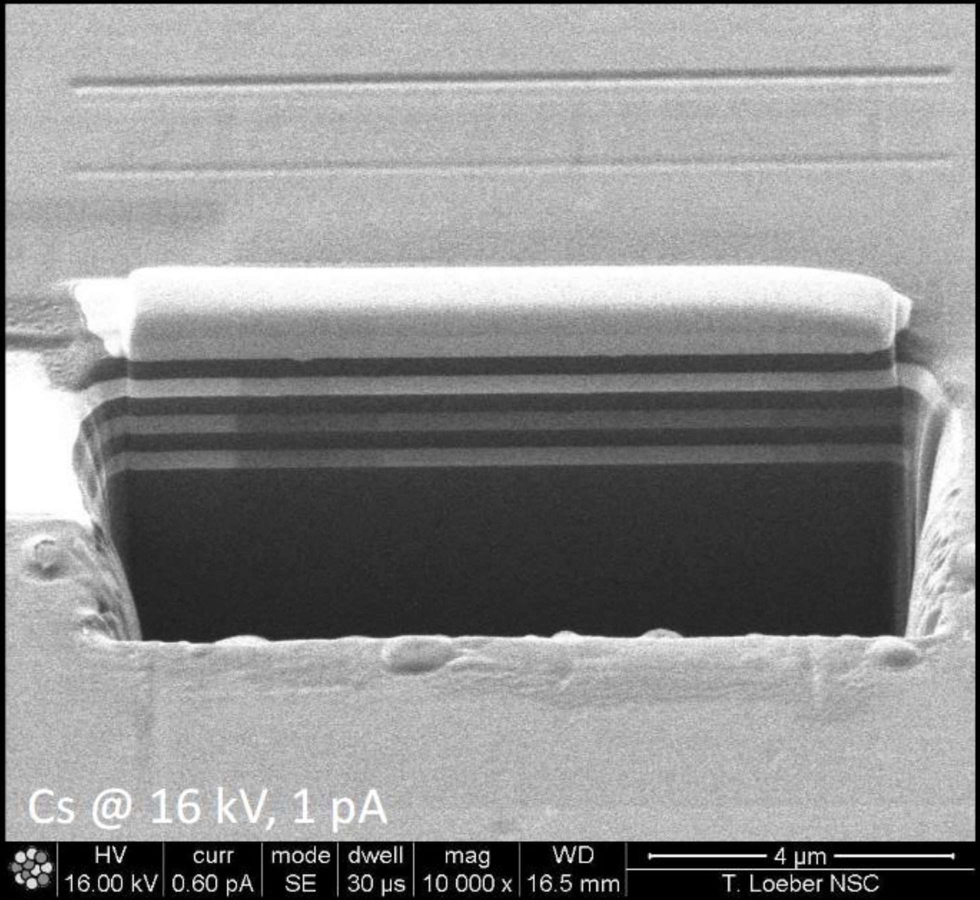
Data by T. Lober, presented at Micro and Nano Engineering Conference (MNE) 2023

Material contrast

Ga ion image



Cs ion image

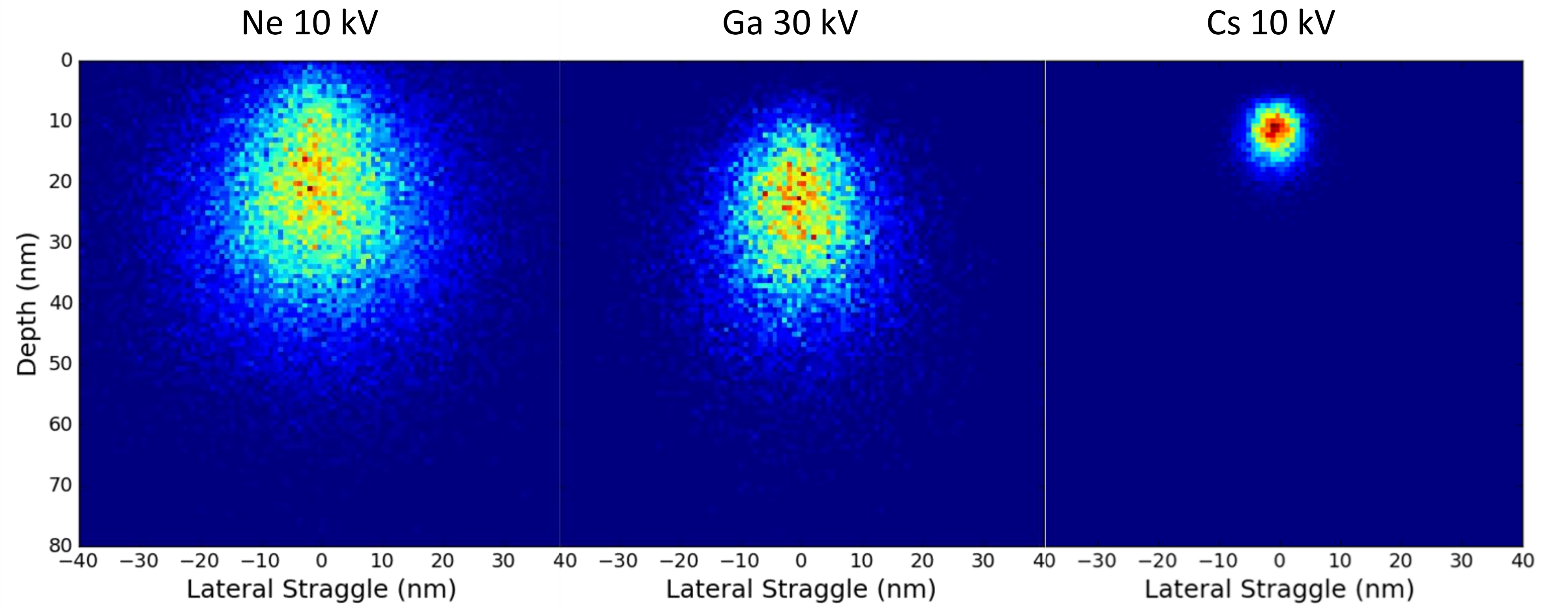


Pt layer contrast of Ga inverted to Cs: dark <-> light

Data by T. Lober, presented at Micro and Nano Engineering Conference (MNE) 2023

Implant Depth Comparisons (SRIM simulation)

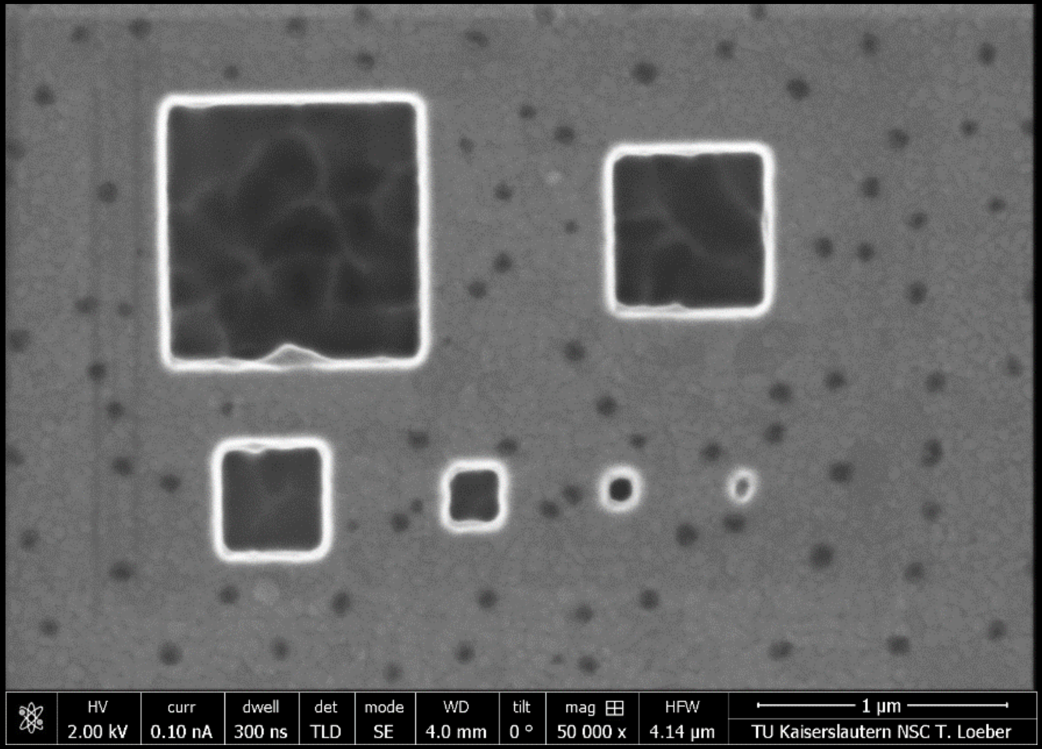
- Comparison of three scenarios where spot size might be 'good enough'
- Cs has significantly reduced straggle and implant depth



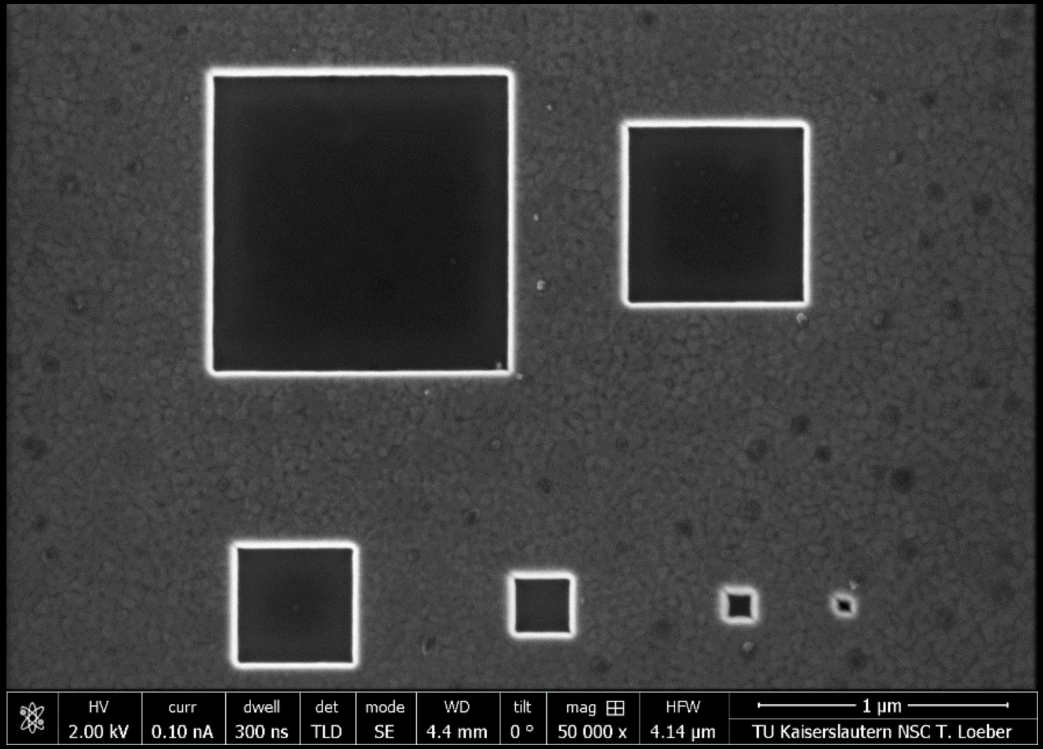
Milling Accuracy: 110 nm Au on Si

→ LoTIS provides clean mill boxes with sharp corners

Milled with Ga^+ LMIS



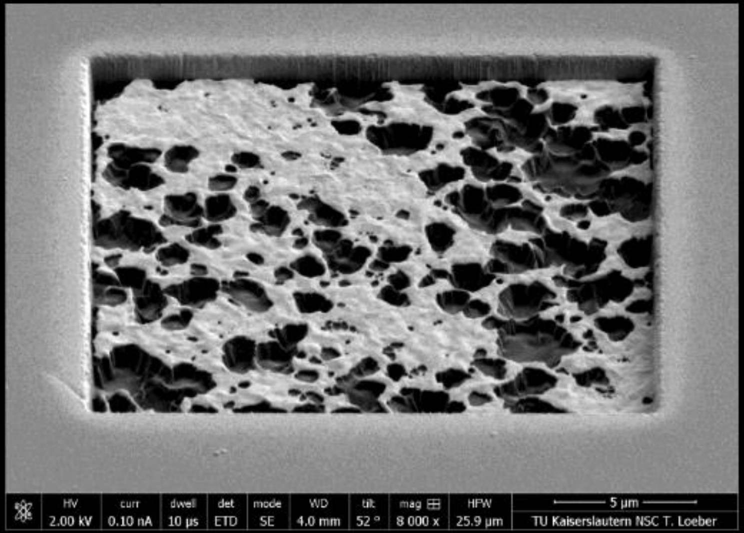
Milled with Cs^+ LoTIS



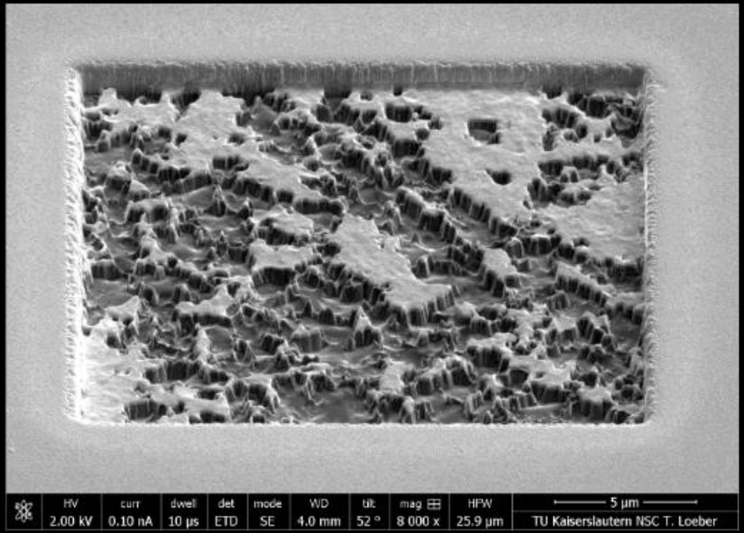
- squares with 1, 0.6, 0.4, 0.2, 0.1 and 0.05 µm length
- milled through the Au layer
- milling time Ga and Cs almost the same

Milling in Copper

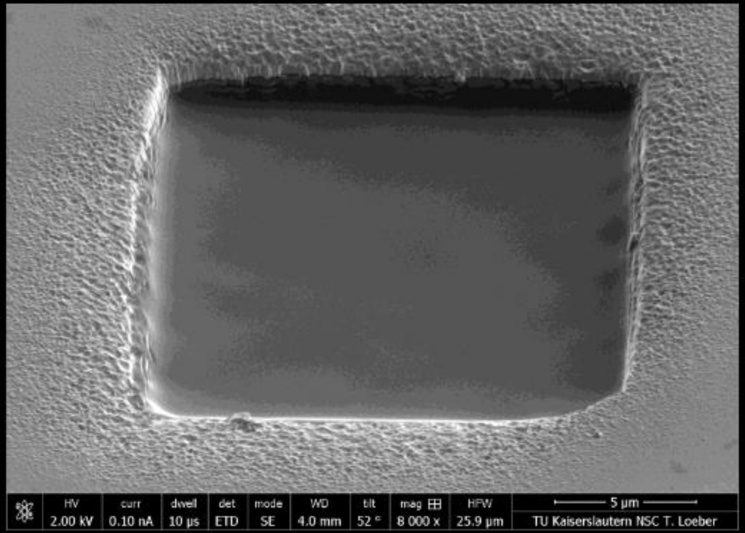
Ga ion: 30 kV @ 2640 pA



Ga ion: 16 kV @ 1440 pA



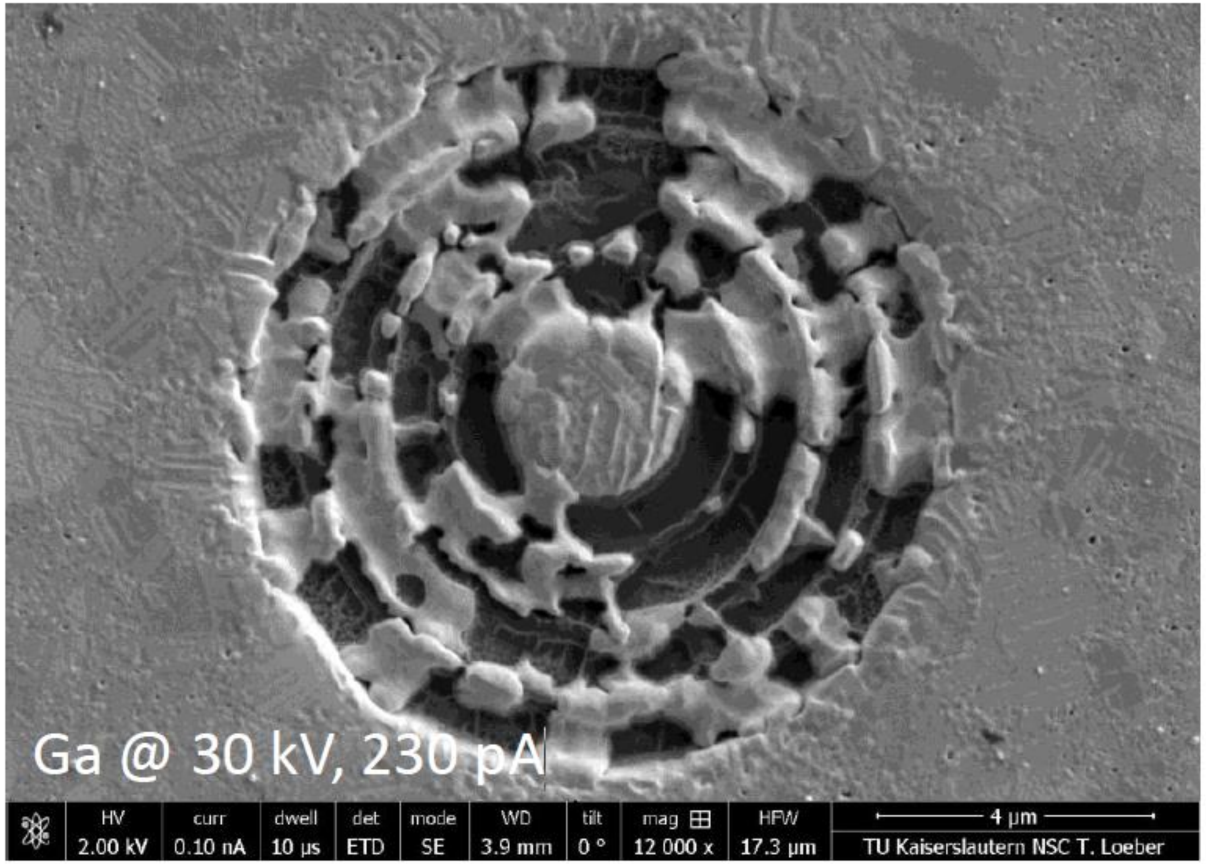
Cs ion: 16 kV @ 1070 pA

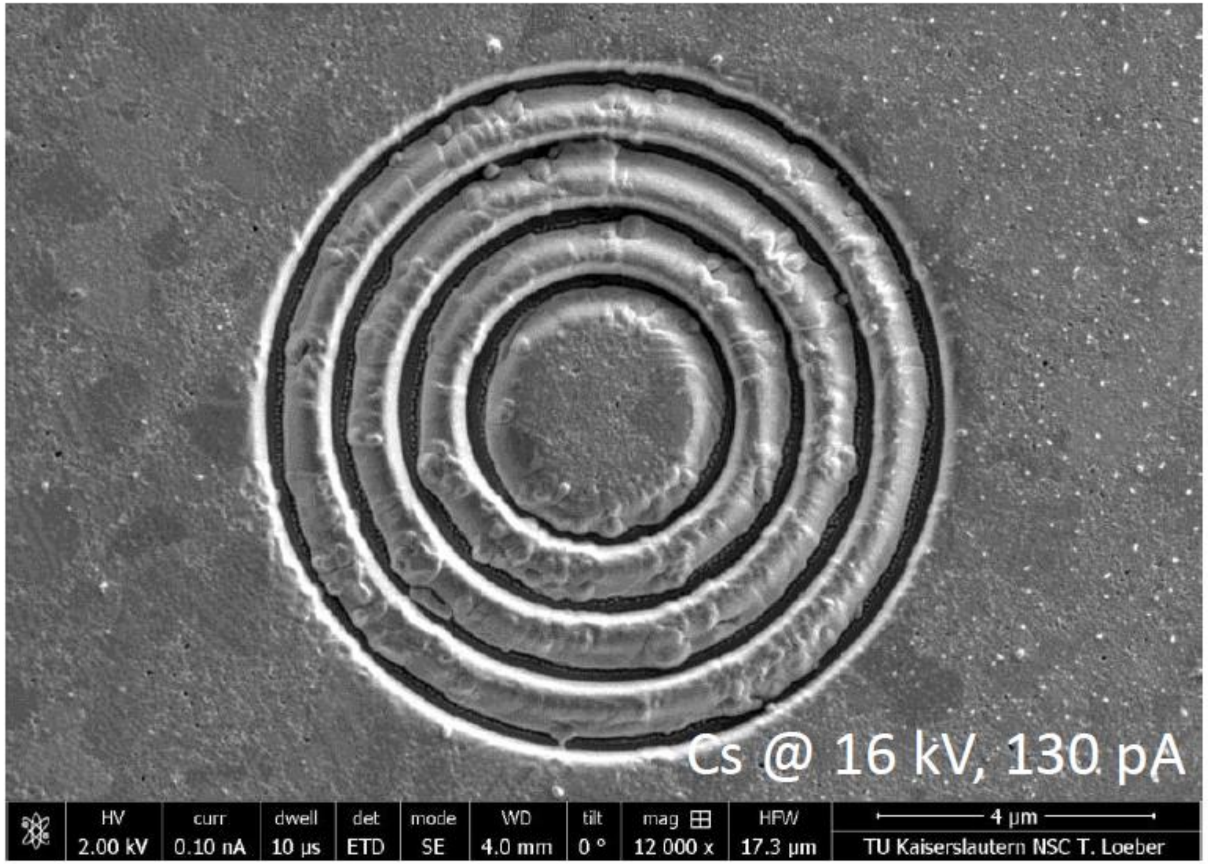


- sputtered Copper layer on Silicon
- layer thickness 1150 nm
- rectangle 20 µm x 20 µm
- milling time about 20 min
- dose about 4500 pC/µm^2

Data by T. Lober, presented at Micro and Nano Engineering Conference (MNE) 2023

Milling in silver





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

Spot Sizes

Selected Beam Energies and Currents

Results given as a σ

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

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

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

Methodology in [1]

16 kV

Current (pA)	Spot Size (1-σ 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-σ 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-σ 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

Summary- FIB:ZERO

- High resolution FIB nanomachining tool
- … with a Cs^+ ion source
- … excellent at milling small structures
- … compatible with depo & etch gas chemistries
- … demo tool available for collaborations
- … available as a FIB-SEM (Thermo-Fisher SCIOS)

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NSC Nano Structuring Center

Milling Accuracy: 110 nm Au on Si

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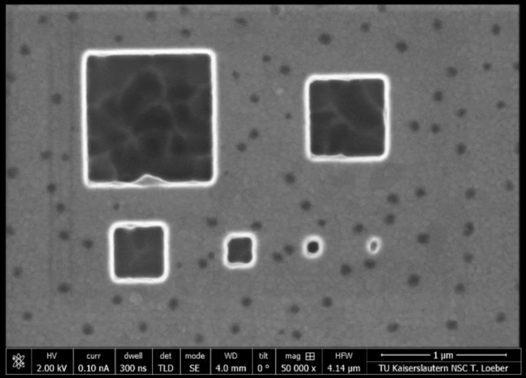
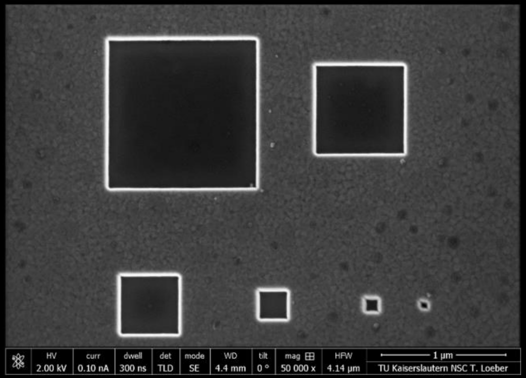
Milled with Ga^+ LMIS



Milled with Cs^+ LoTIS



- squares with 1, 0.6, 0.4, 0.2, 0.1 and 0.05 µm length
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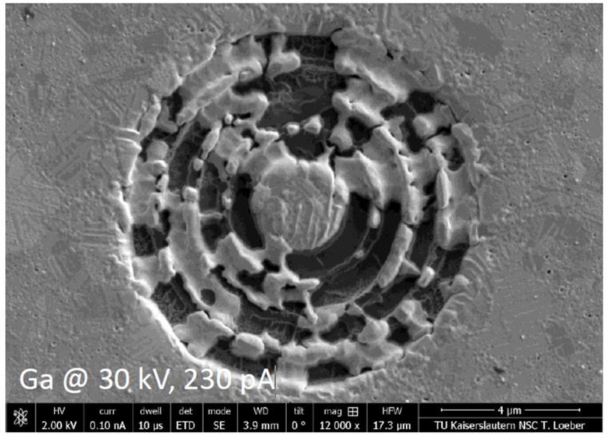
PHYSIK

Milling in silver

TECHNISCHE UNIVERSITÄT KAISERSLAUTERN



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

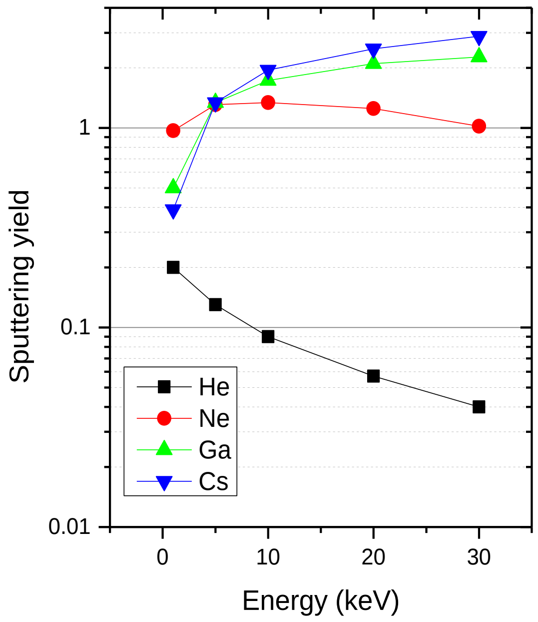
NSC Nano Structuring Center

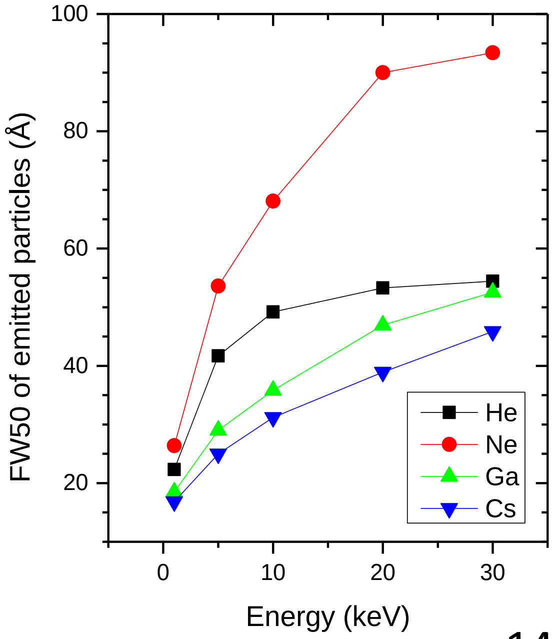
SIMS: Primary Ion Species Matters

Differing Sputter Rates
→ Analysis Time

Differing interaction Volumes
→ Resolution

Differing Yields
→ Sensitivity Floor, SNR





Existing Elemental Analysis Techniques and a New Solution

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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

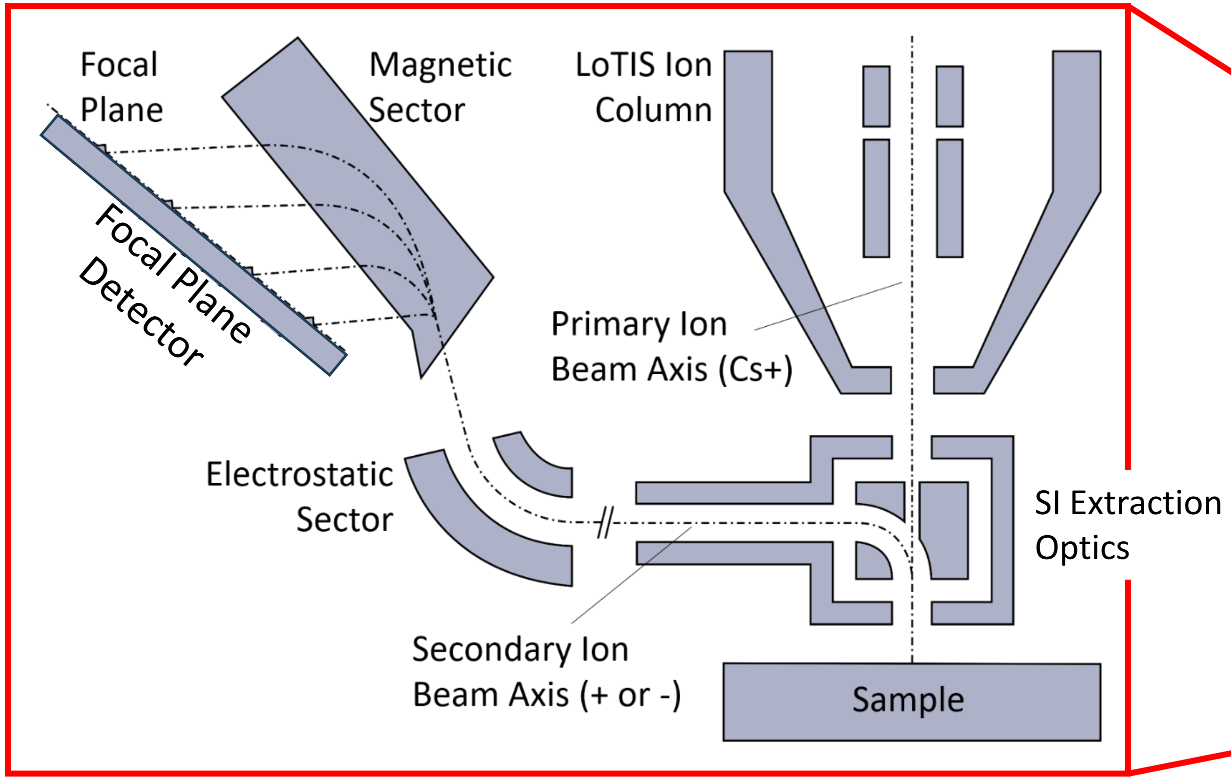
SIMS:ZERO

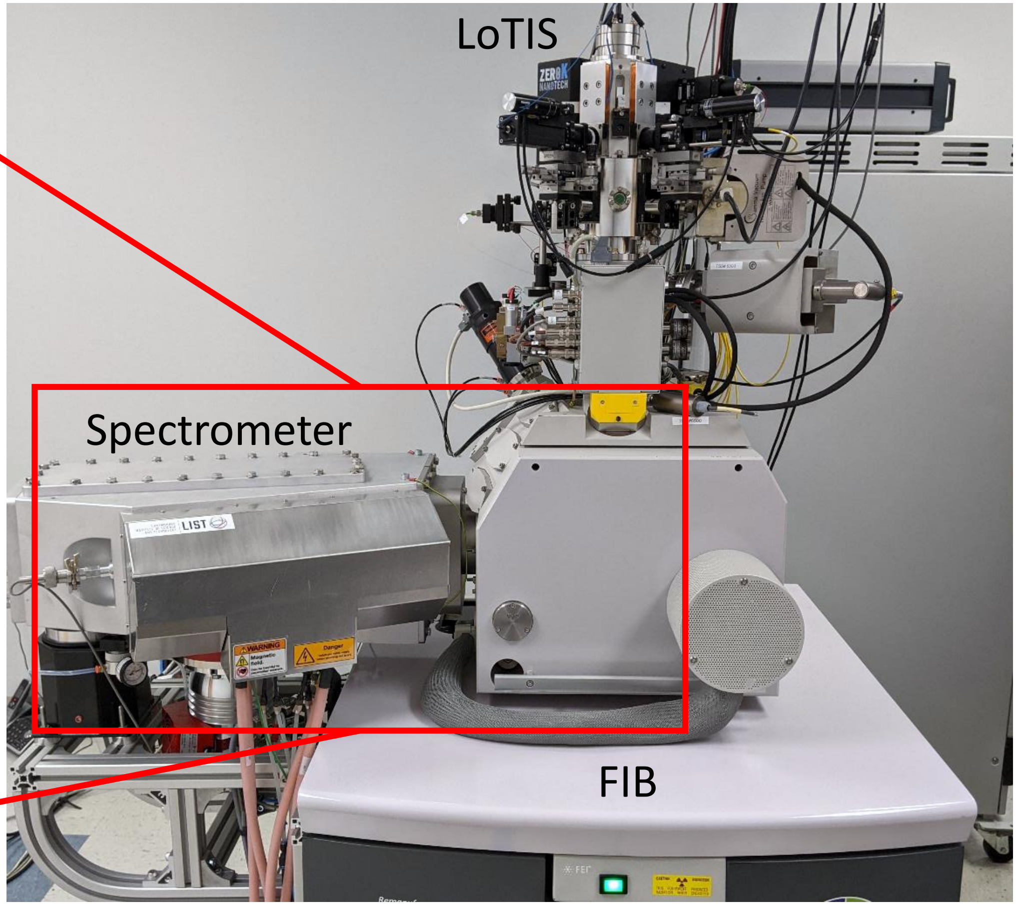
- Few-nanometer resolution (slide 21)
- High secondary ion yield (slides 23,24)
- Integrated sample-prep and analysis capability (slides 25-31)

SIMS:ZERO

Instrument Overview

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



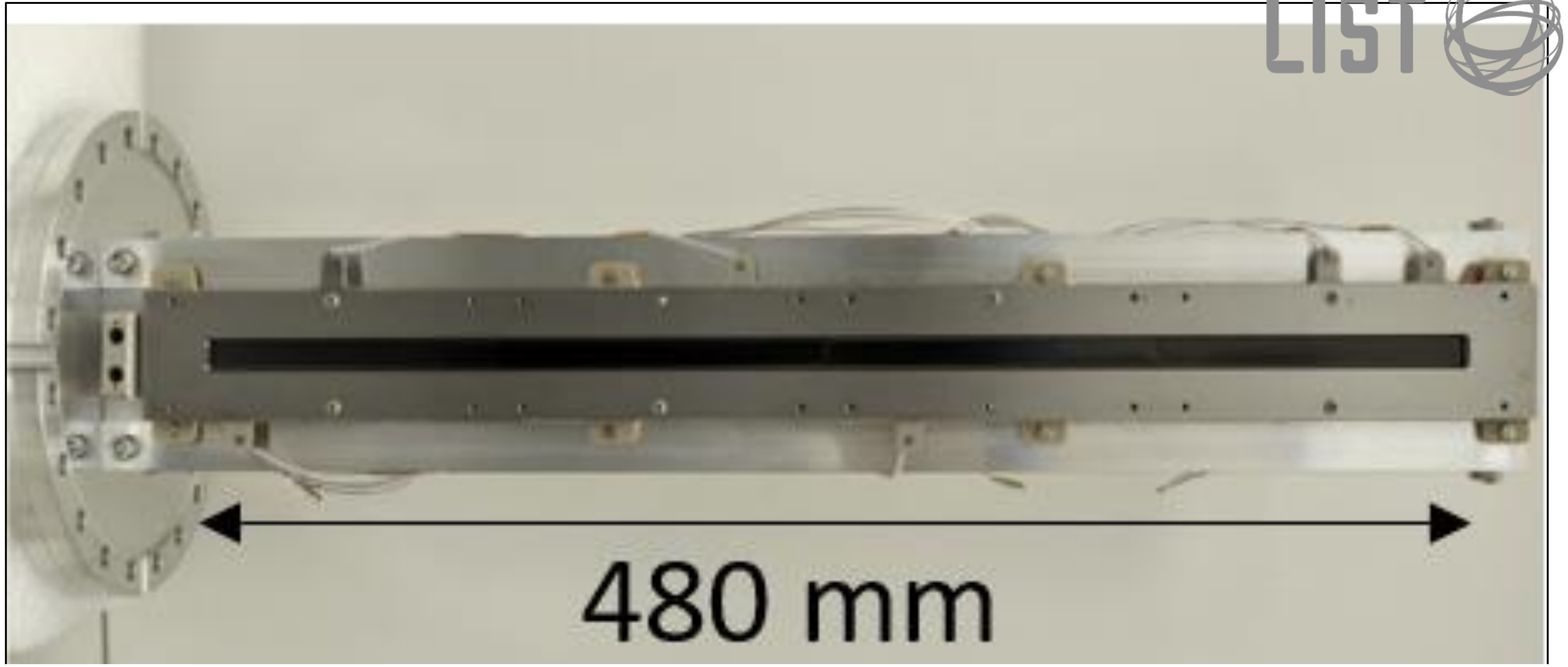


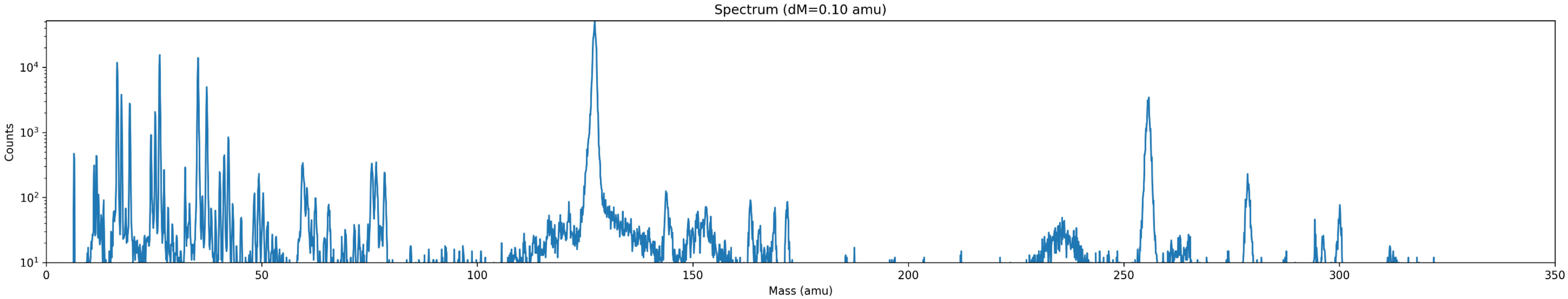
- FIB online 6/2020
- SIMS online 5/2021

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

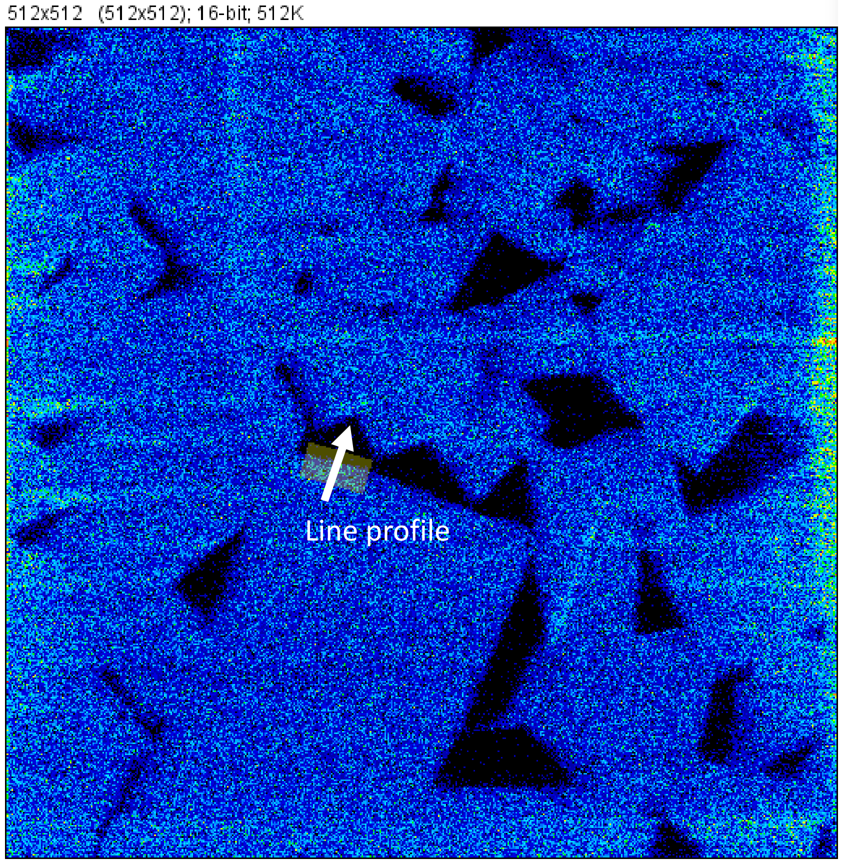




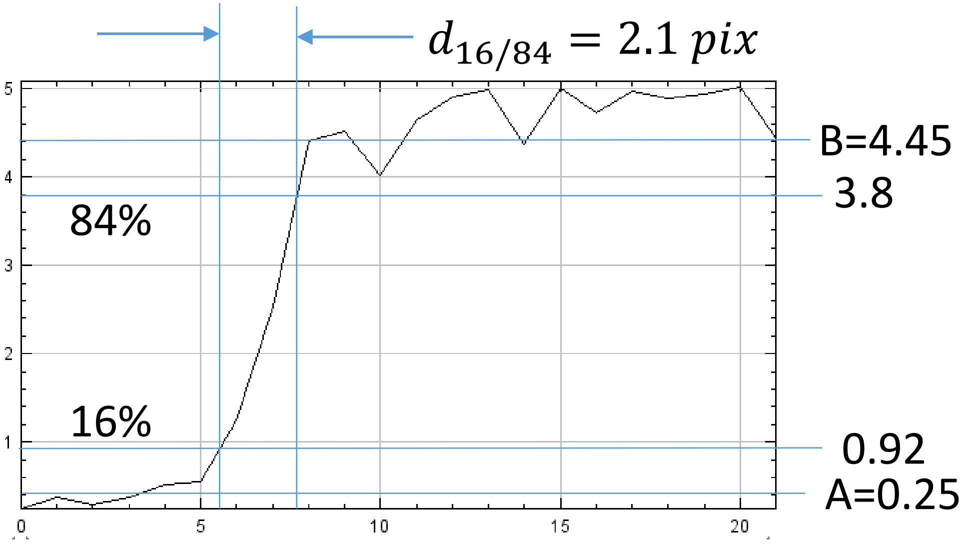
SIMS:ZERO Resolution

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



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

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

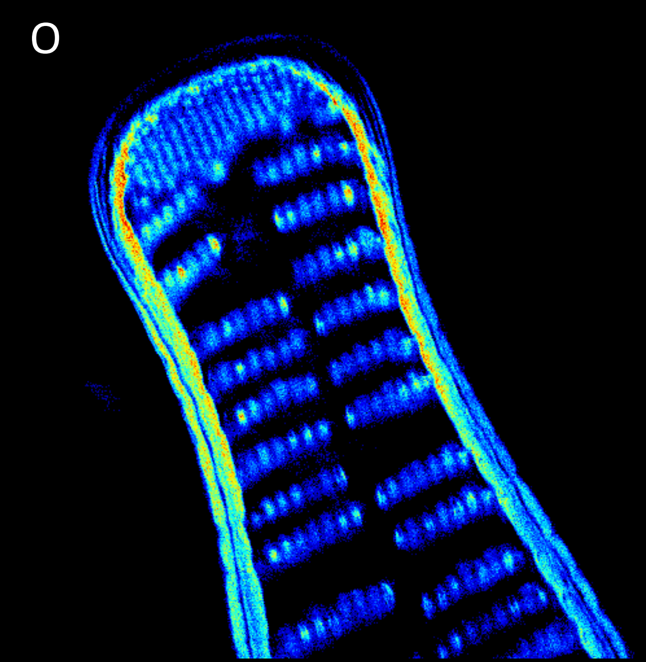
Working Distance = 51.6mm
272s acquisition time.

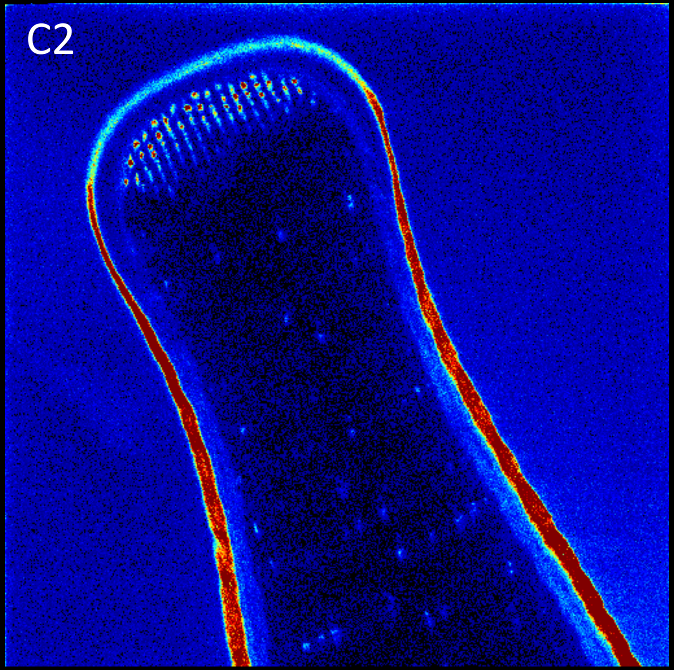
Negative Ions

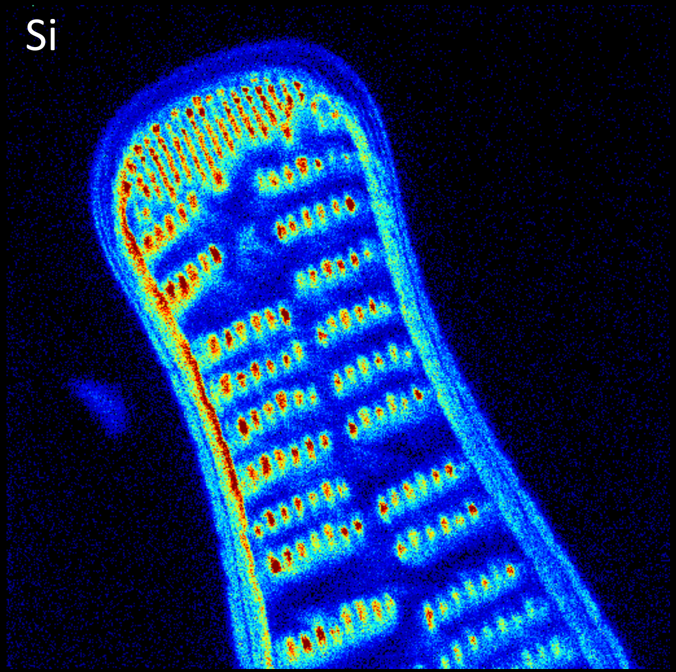
Date	05/12/2021
Sample	WC (184 amu)
FOV (um)	2.97um
I (pA)	2.5
U (kV)	16

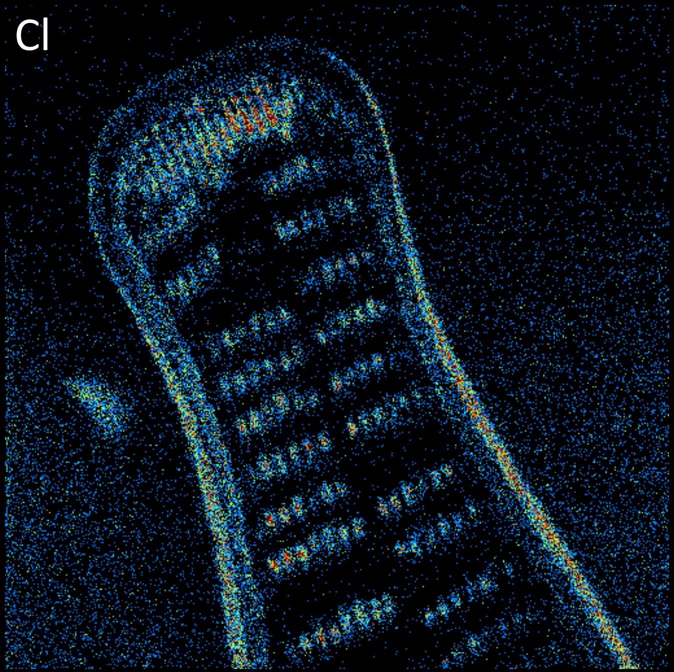
Diatoms (Silica –shelled algae)

7.5 um FoV



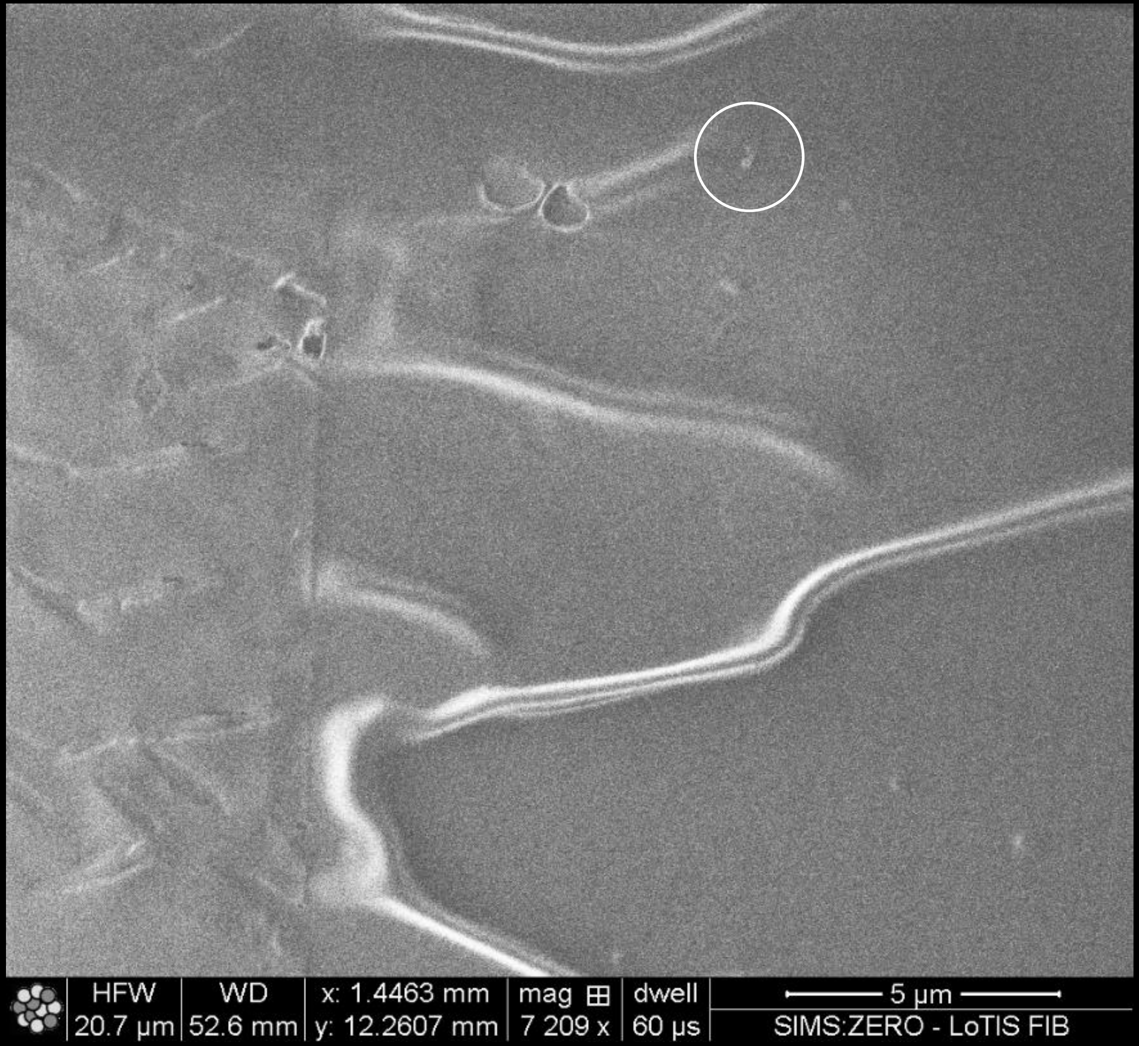


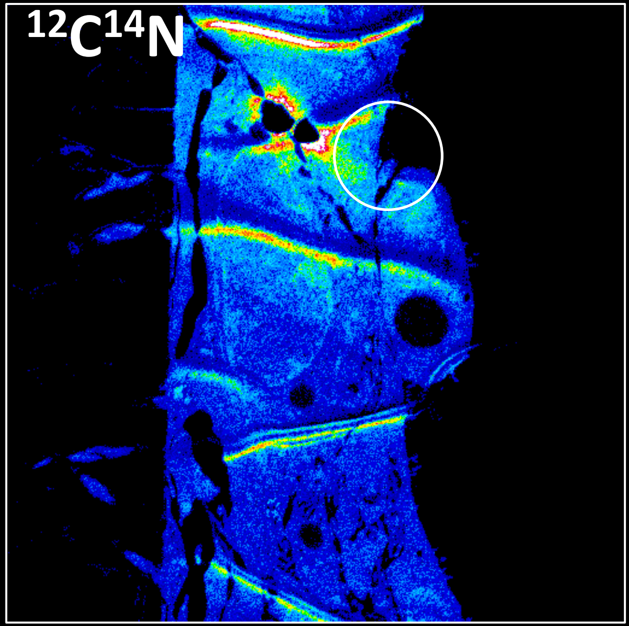




Needle in a haystack:

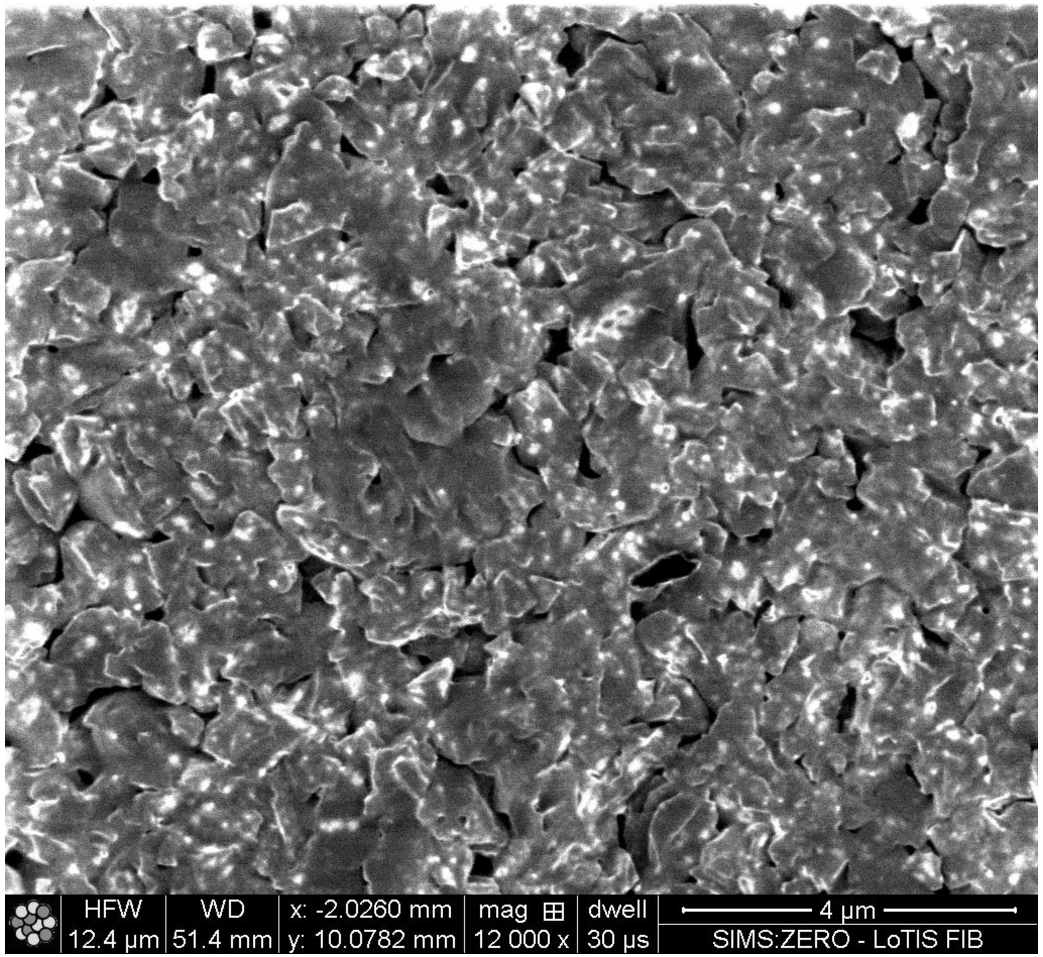
Find the TiO nanoparticle in the huge, fixed cell





SIMS Analysis Example

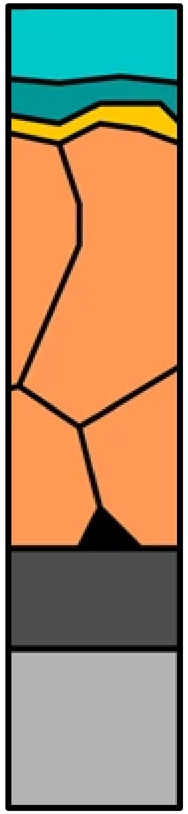
CIGS Cu(In,Ga)Se_2 – Rb doped



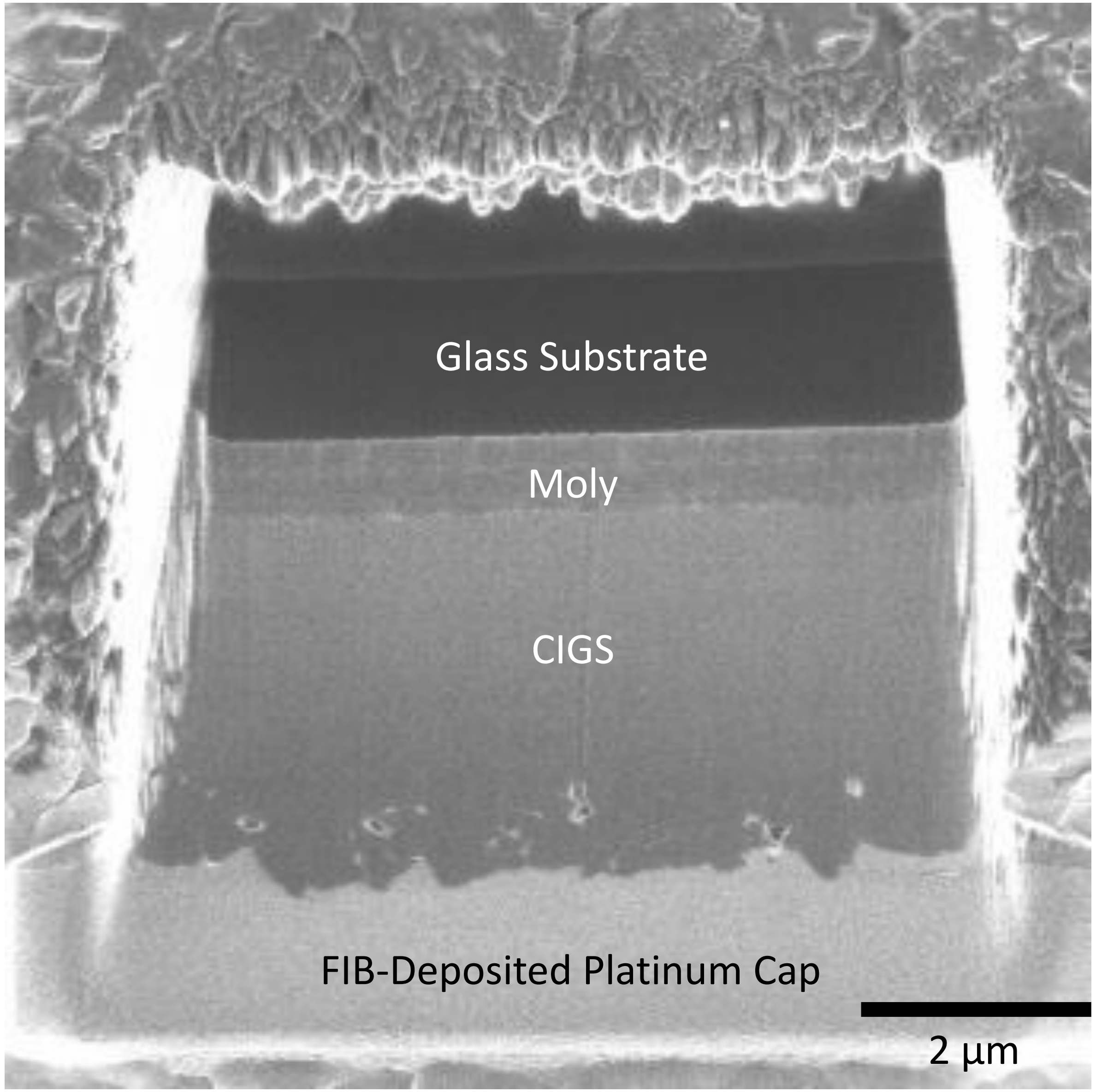
SE Image Cs+, 16keV, 10pA, 51.6mm WD

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

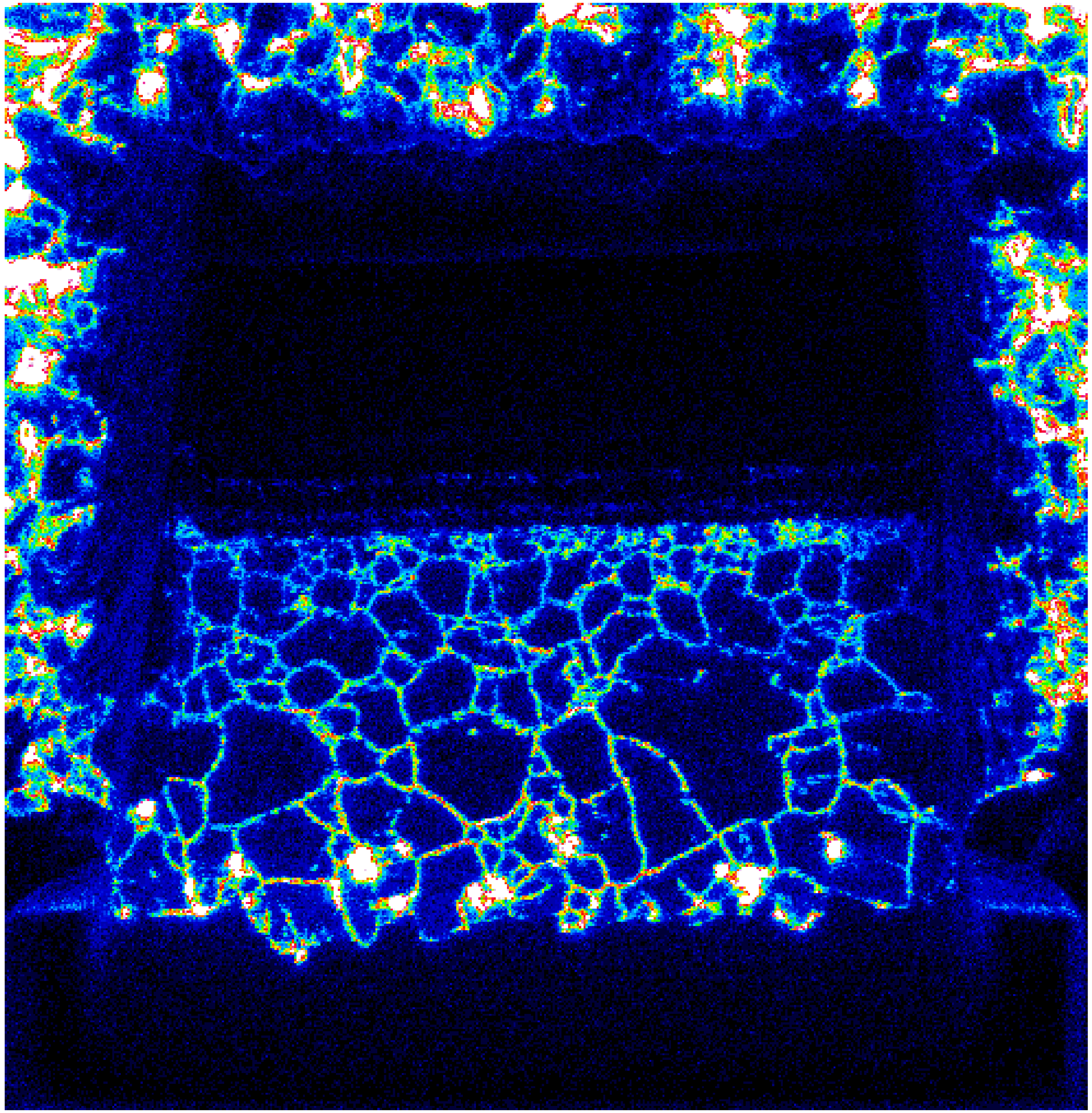


Werner, et al. *Scientific Reports* **volume 10**, 7530 (2020)



Secondary electron image

- Sample polished, ready for SIMS
- 9.5 µm FOV

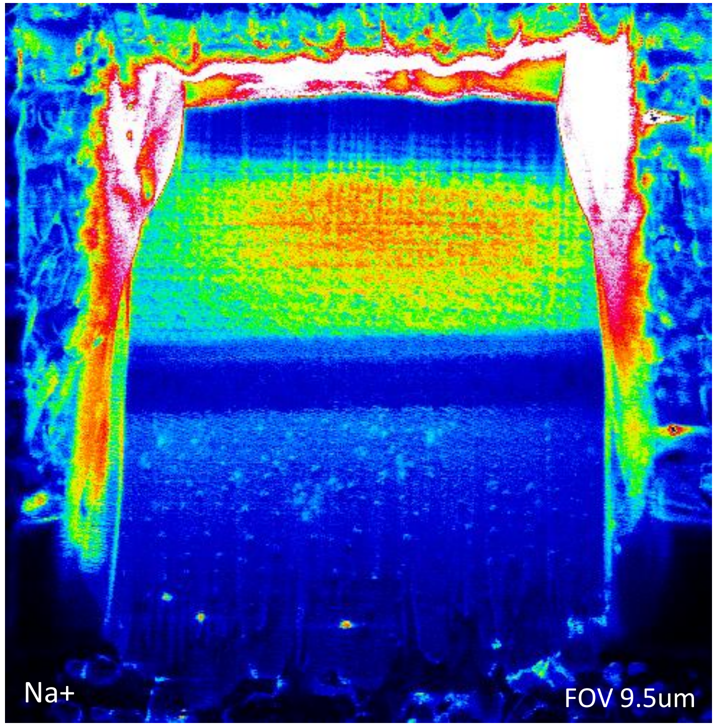


Rb$^+$ SIMS Image

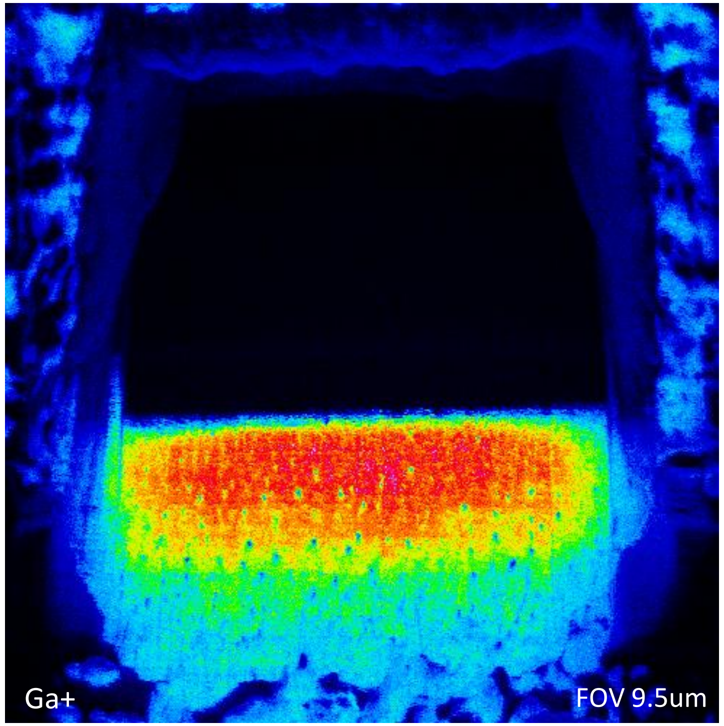
- Rb confined to grain boundaries
- Grains are smaller near the interfaces
- Bilayer structure in the Moly layer

CIGS $Cu(In,Ga)Se_2$ – Rb doped

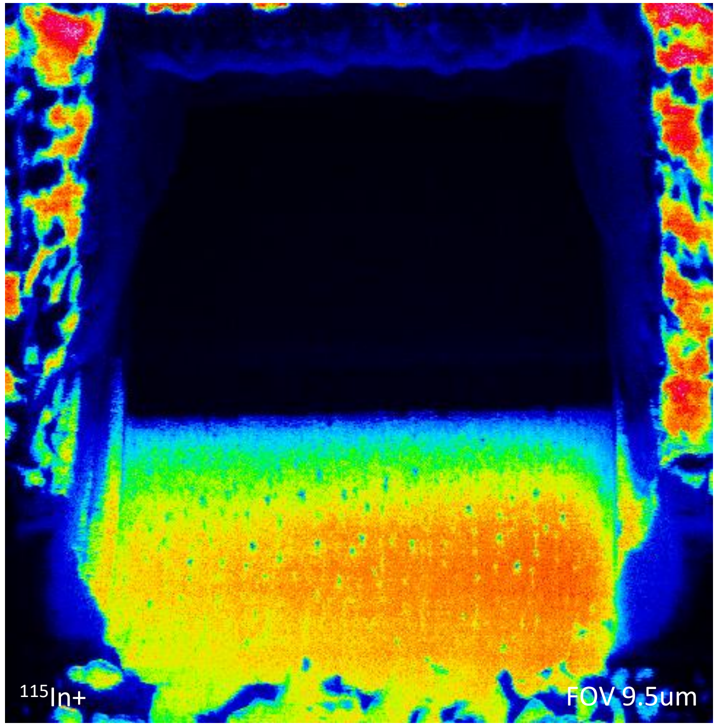
Section View – Positive Ions – Post 3rd Polish



Na – Soda Lime Glass



Ga concentration gradient ↑

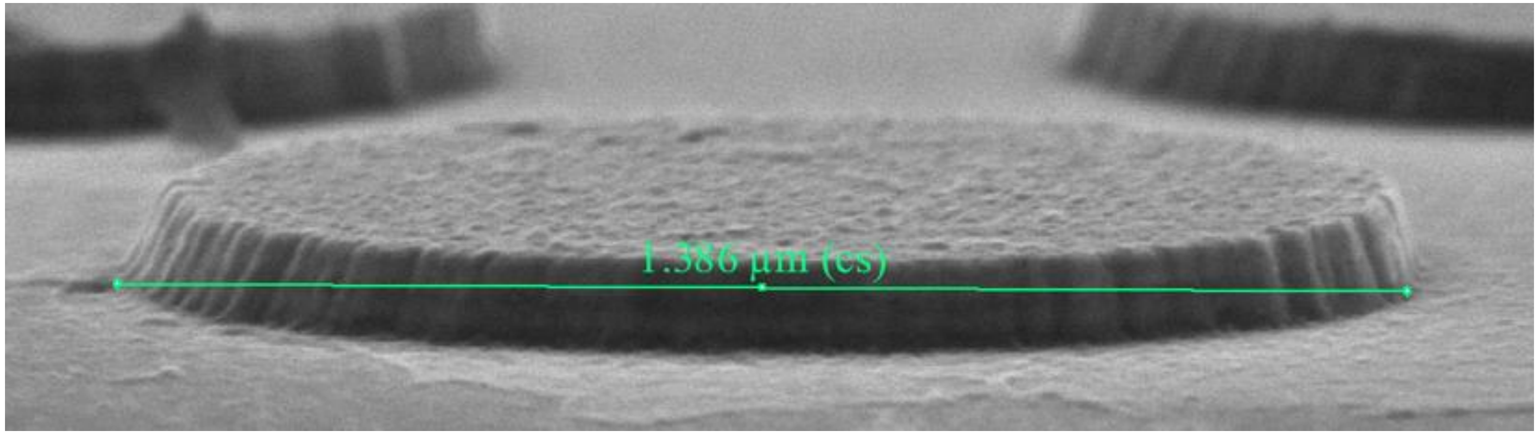


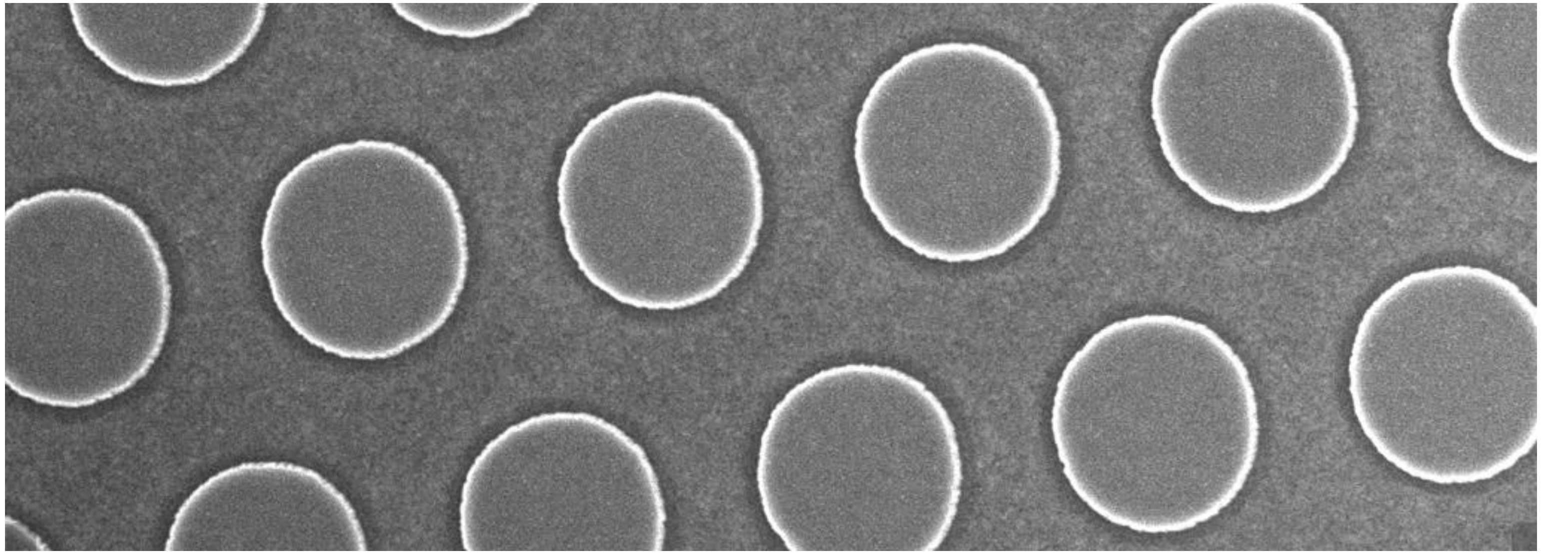
In concentration gradient ↓

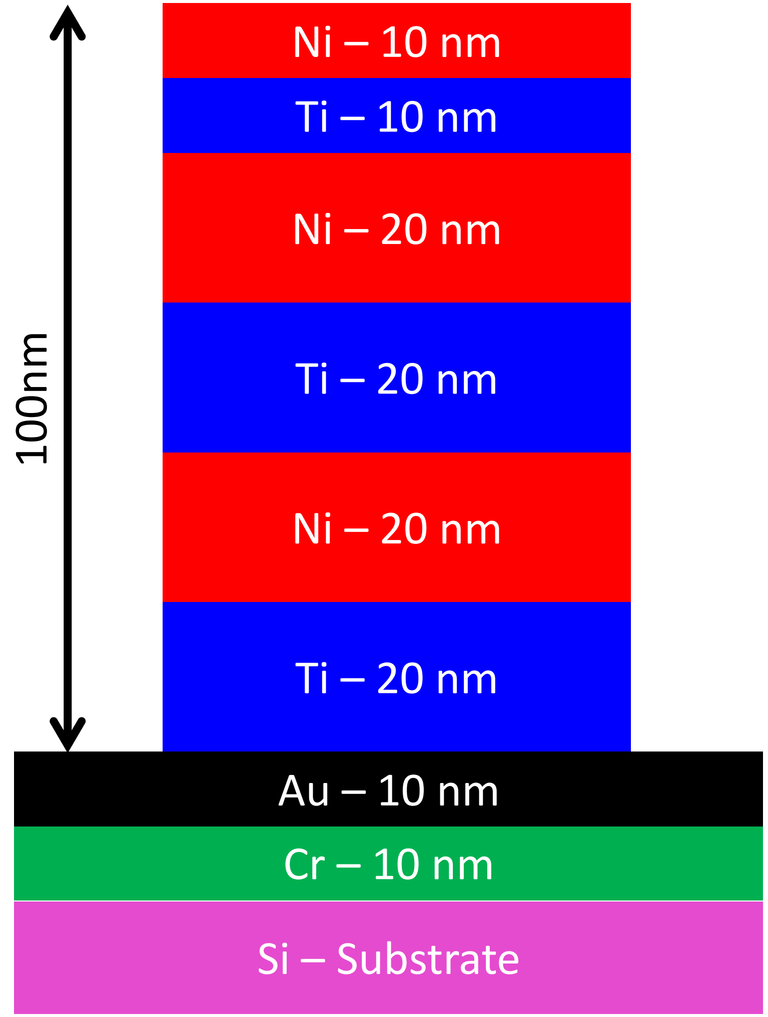
Cs+, 16keV, 10pA, 51.6mm WD
CIGS_Pos_2107201626359.csv

TiNi Pillars

Multilayer Ti / Ni Pillars on Au/Cr/Si Substrate

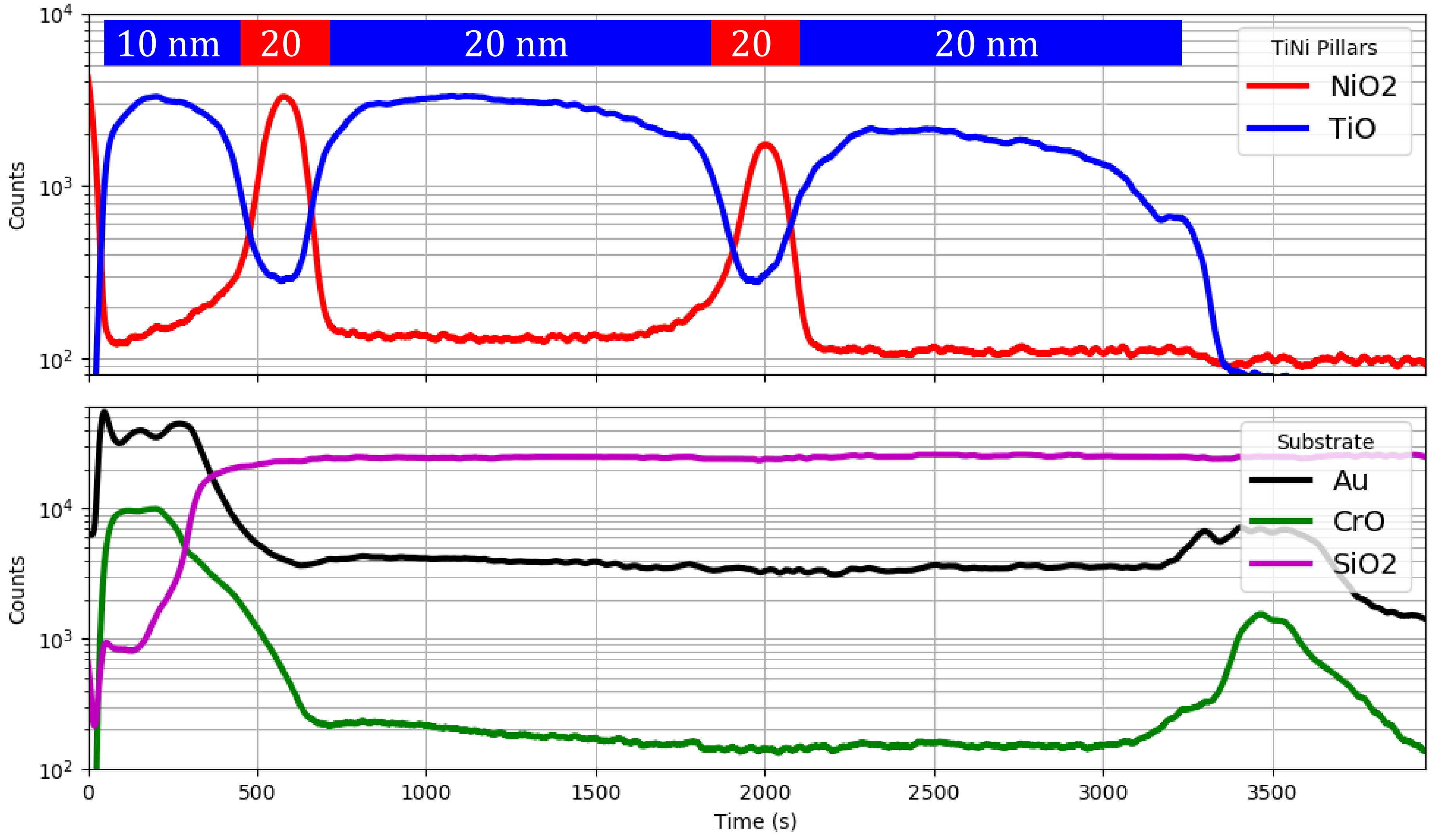


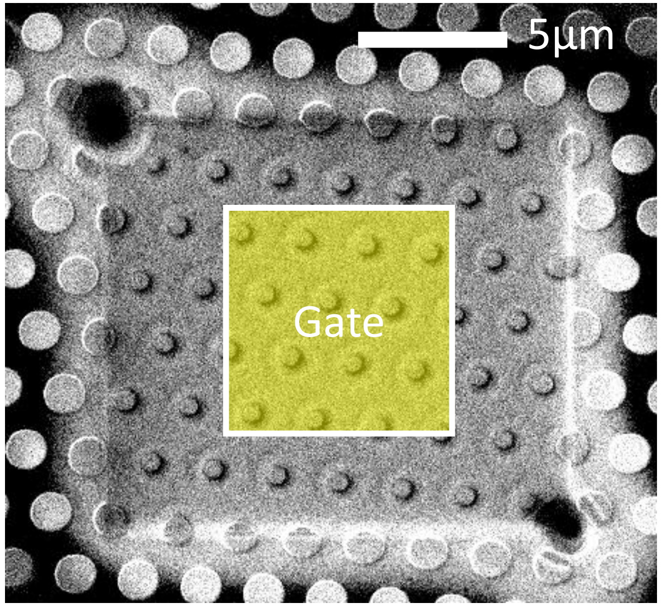




TiNi Pillars

‘Large’ Area Depth Profile (7.5µm Gate)

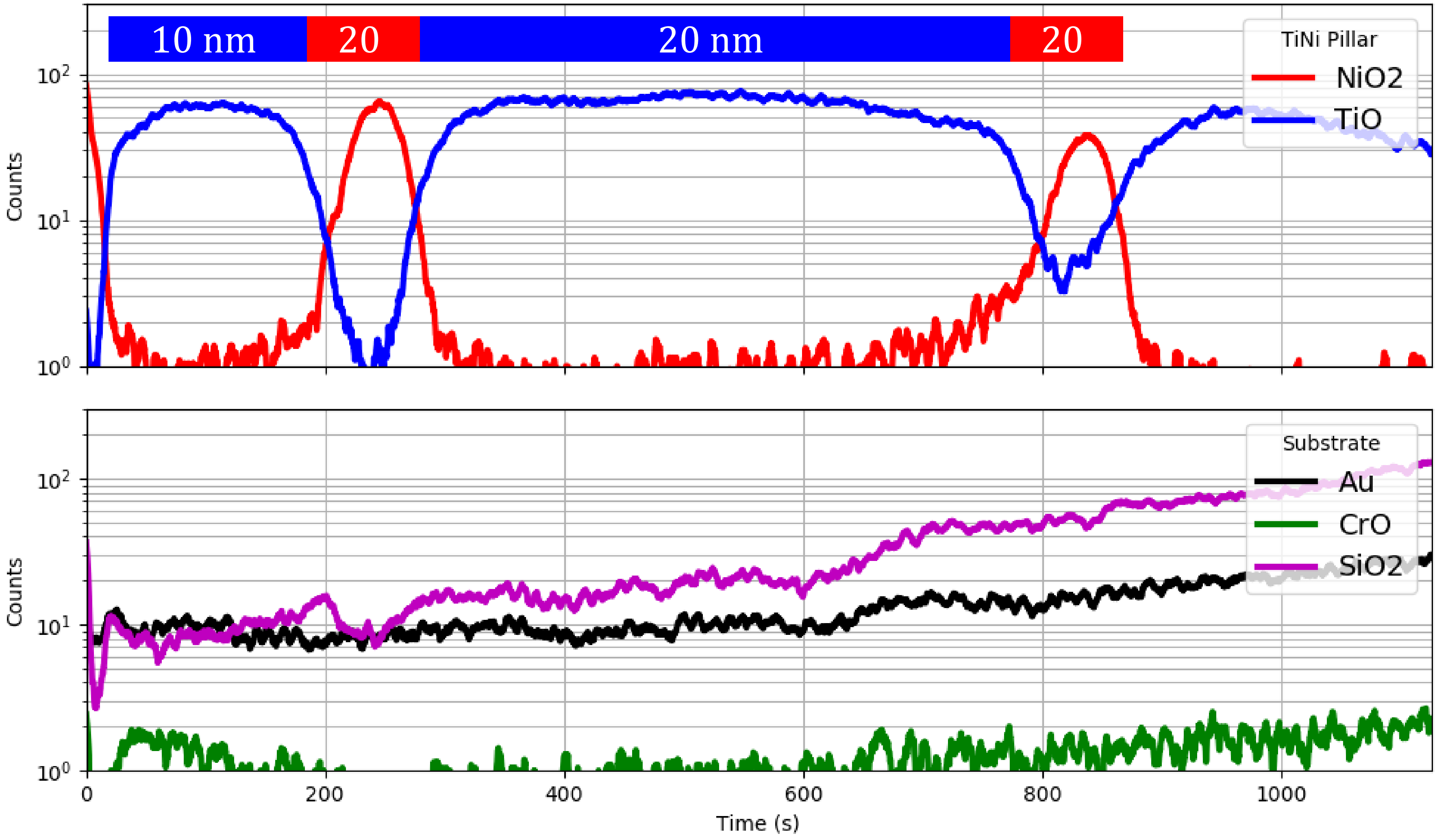


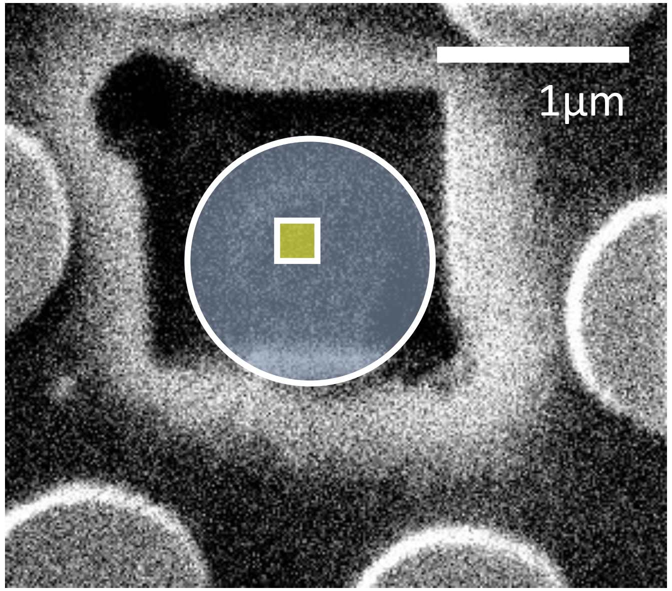


Parameter	Value
Current	5pA
Energy	5kV
Polarity	Neg
Gate FOV	7.5 µm
Integration time	1000ms
Field	600mT

TiNi Pillars

‘Small’ Area Depth Profile (200nm Gate)



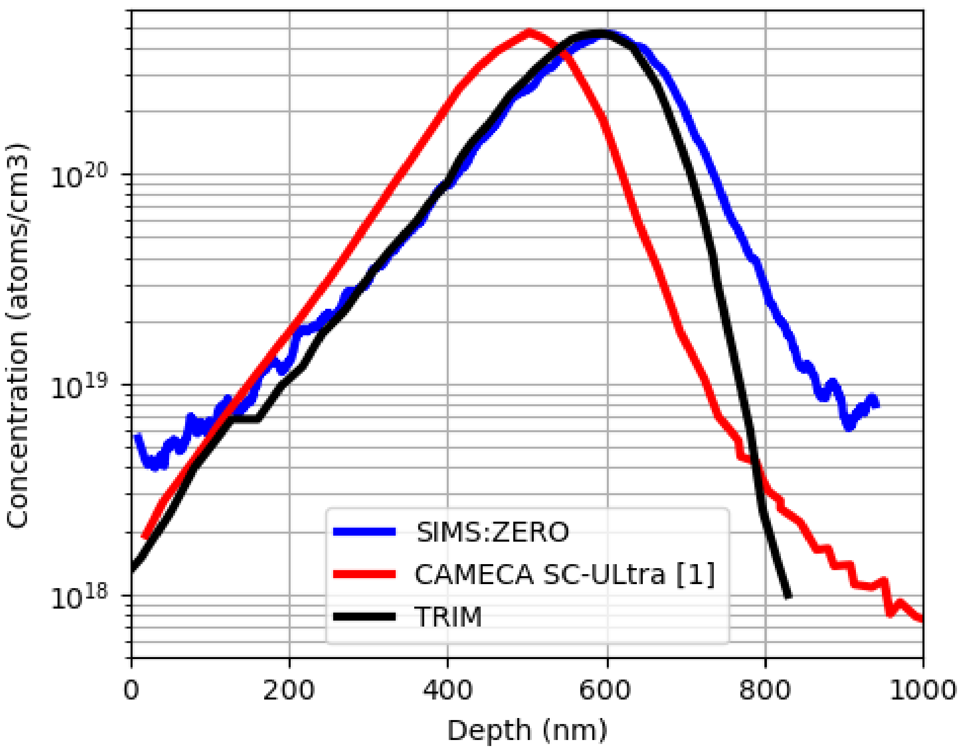


Parameter	Value
Current	2pA
Energy	5kV
Polarity	Neg
Gate FOV	200 nm
Integration time	250ms
Field	600mT

Boron Doped Silicon

Depth Profile Comparison – Reference Sample

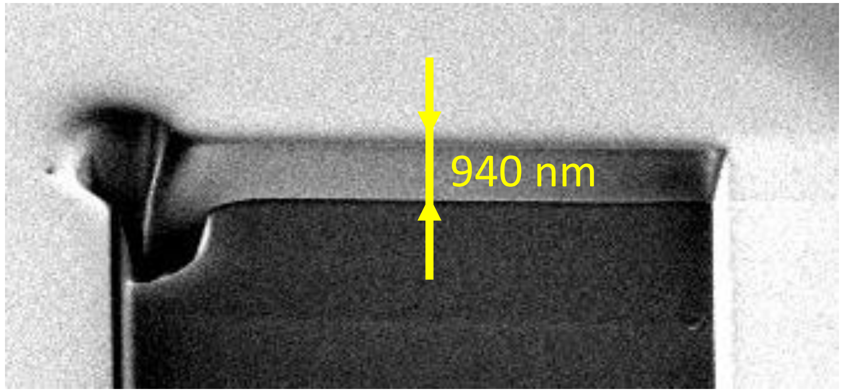
Implantation of B at 190 keV; Dose 10^{16} ions/cm^2



[1] Eswara, et al. MRS COMMINCATIONS. Volume 9, Issue 3 (2019) 10.1557/mrc.2019.89

	SIMS:ZERO	SC Ultra
Primary Ion	Cs^+	O_2^+
Energy	16kV	4.5kV
Current	25 pA	85000 pA
Area	4.2um x 4.2um	?
Polarity	Neg	Pos
Secondary Ion	BO2-	B+

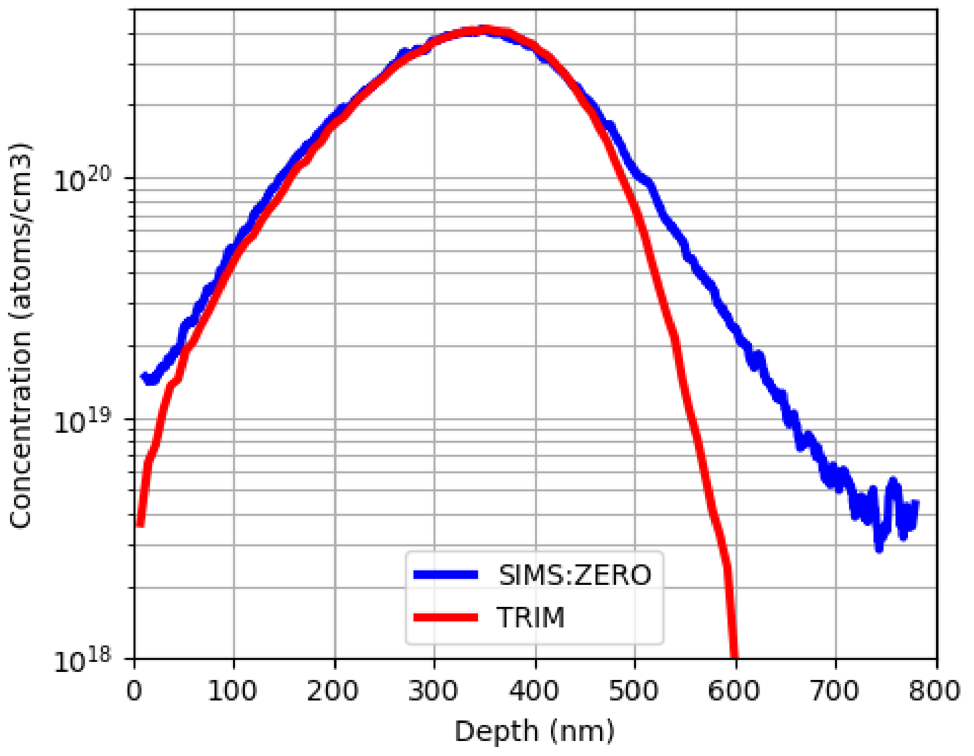
Si:B Crater



Aluminum Doped Silicon

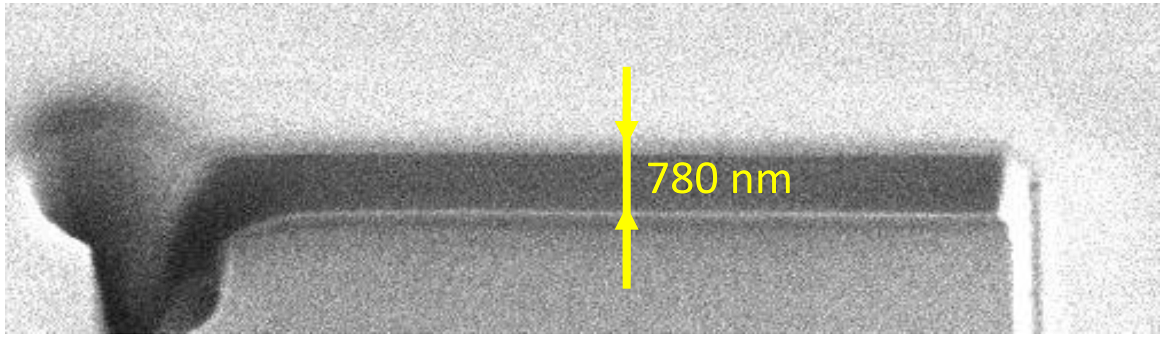
Depth Profile Comparison – Reference Sample

Implantation of Al at 190 keV; Dose 10^{16} ions/cm^2



	SIMS:ZERO
Primary Ion	Cs+
Energy	16kV
Current	25pA
Area	4.2um x 4.2um
Polarity	Neg
Secondary Ion	AlO-

Si:Al Crater



SUMMARY SIMS:ZERO

Strengths:

… 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 analysis of complex, multi-element, or light element samples

… new opportunities for FIB beam control via SIMS signal

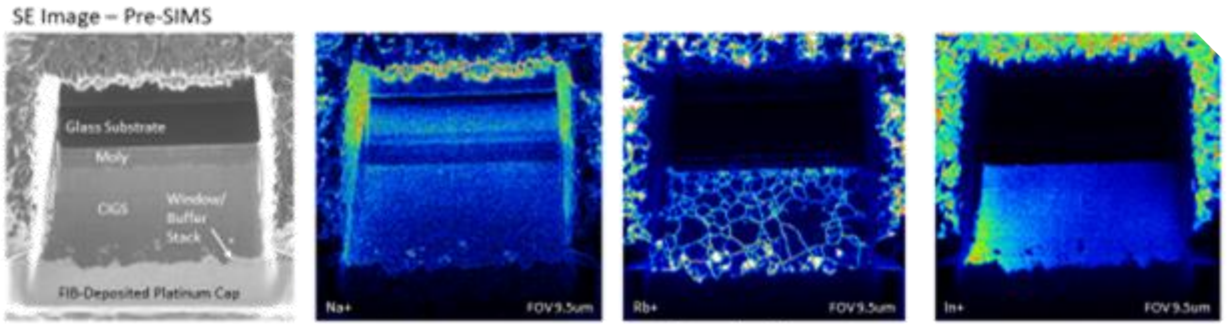
Weaknesses:

Lower mass-resolving power than most dedicated SIMS systems

Quantification of concentrations harder than EDX

CIGS Cu(In,Ga)Se_2 – Rb doped

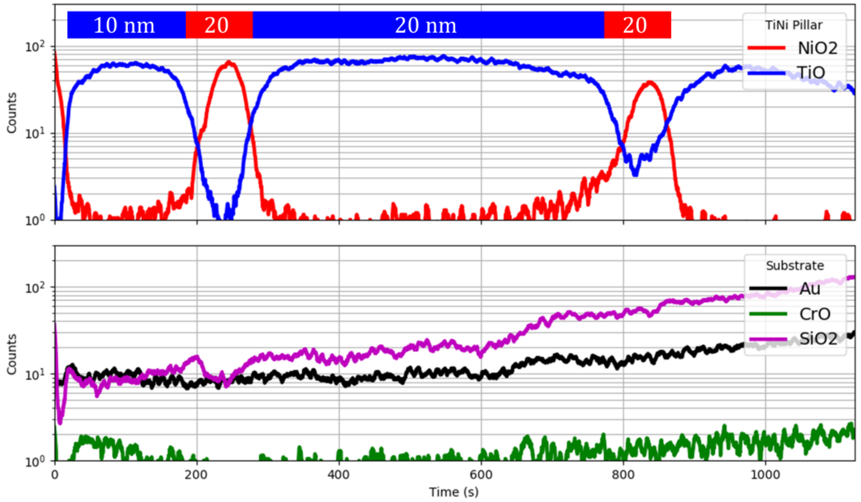
Section View – Positive Ions

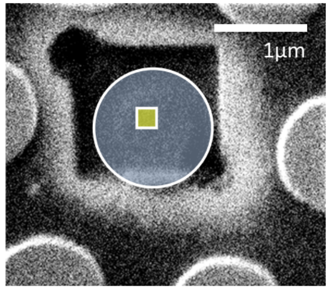


- Rb confined to grain boundaries
- Grains are smaller near the interfaces

TiNi Pillars

'Small' Area Depth Profile (200nm Gate)





Parameter	Value
Current	2pA
Energy	5kV
Polarity	Neg
Gate FOV	200 nm
Integration time	250ms
Field	600mT