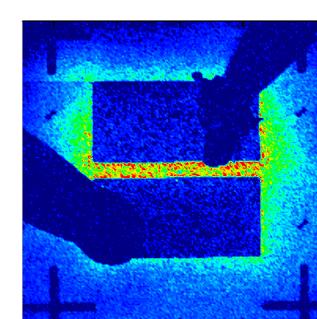
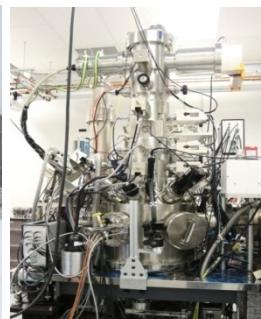




Sandia  
National  
Laboratories

# Towards Deterministic Defect Center Fabrication Using Sandia's Focused Ion Implantation Capabilities



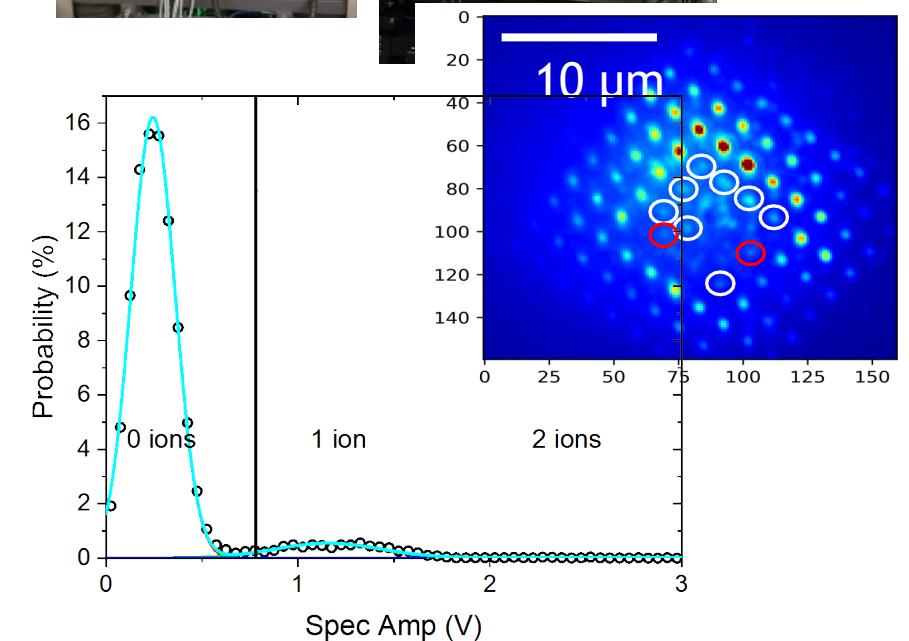
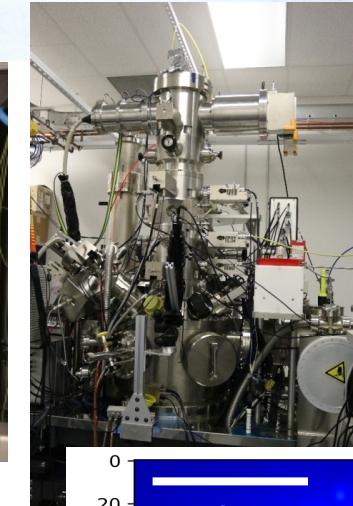
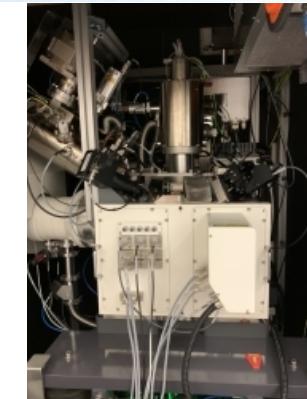
E. Bielejec

Sandia National Laboratories, Albuquerque, NM 87185

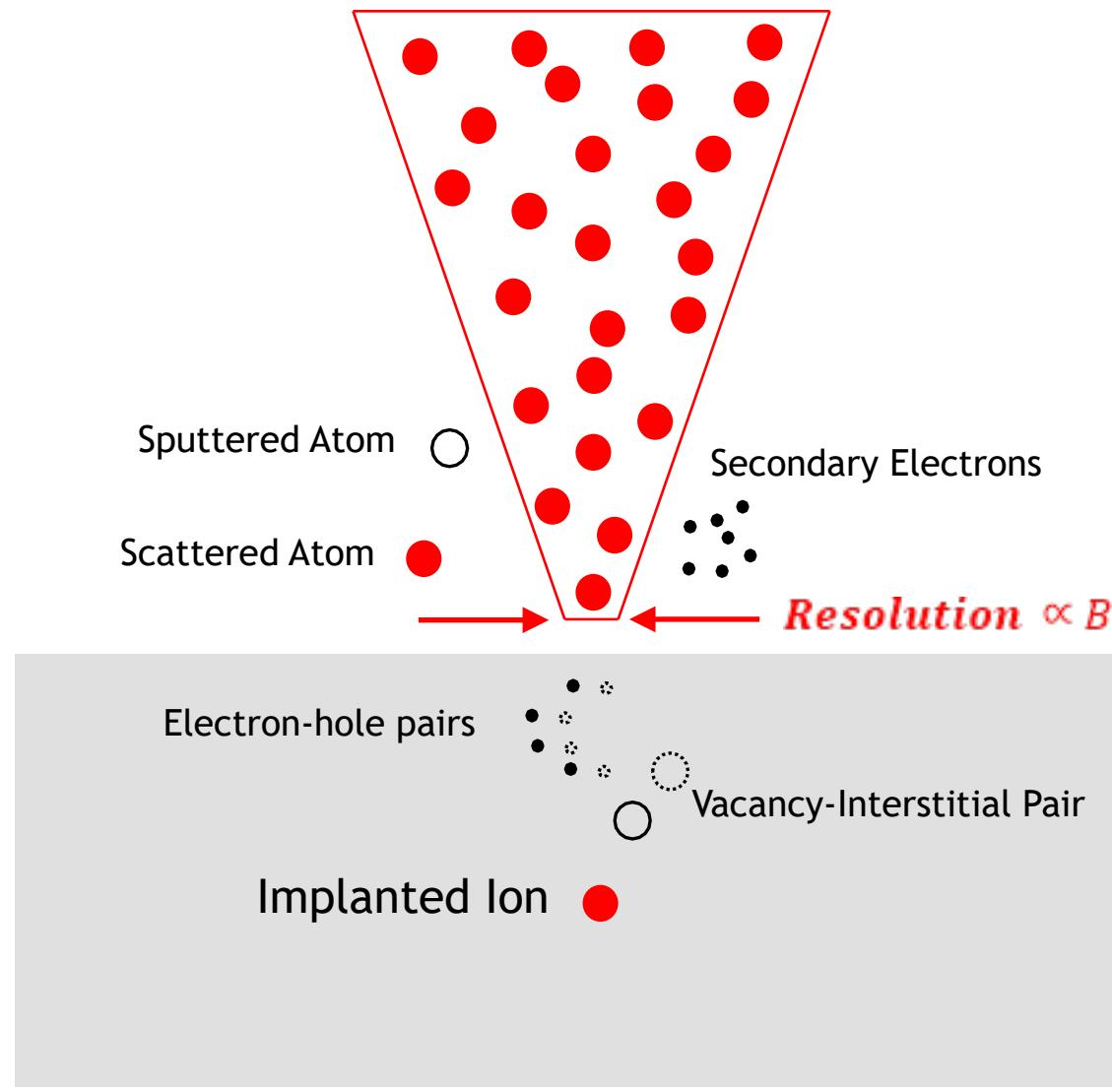


# Outline

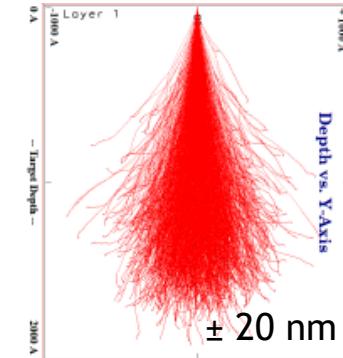
- Introduction to Focused Ion Beam Capabilities at Sandia's Ion Beam Laboratory (IBL)
- Multi-species Focused Ion Beam (FIB)
  - Sandia's nanolimplanter and Raith Velion
  - New Liquid Metal Alloy Ion Source Development
- Single Defect Centers in Wide Bandgap Substrates
  - In-situ Counting - control the # of implanted ions
  - In-situ Photoluminescence - verify defect centers
- Summary and Outlook for FIB Implantation



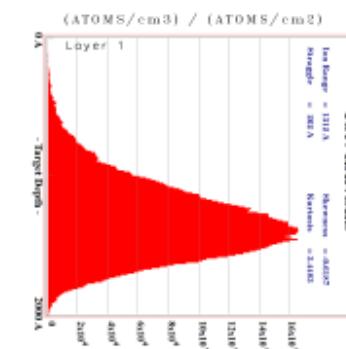
# Nanofabrication using Focused Ion Beam (FIB) Implantation



SRIM simulation - XY straggle  
200 keV Si into diamond



*Depth  $\propto$  Energy + Z Straggle*

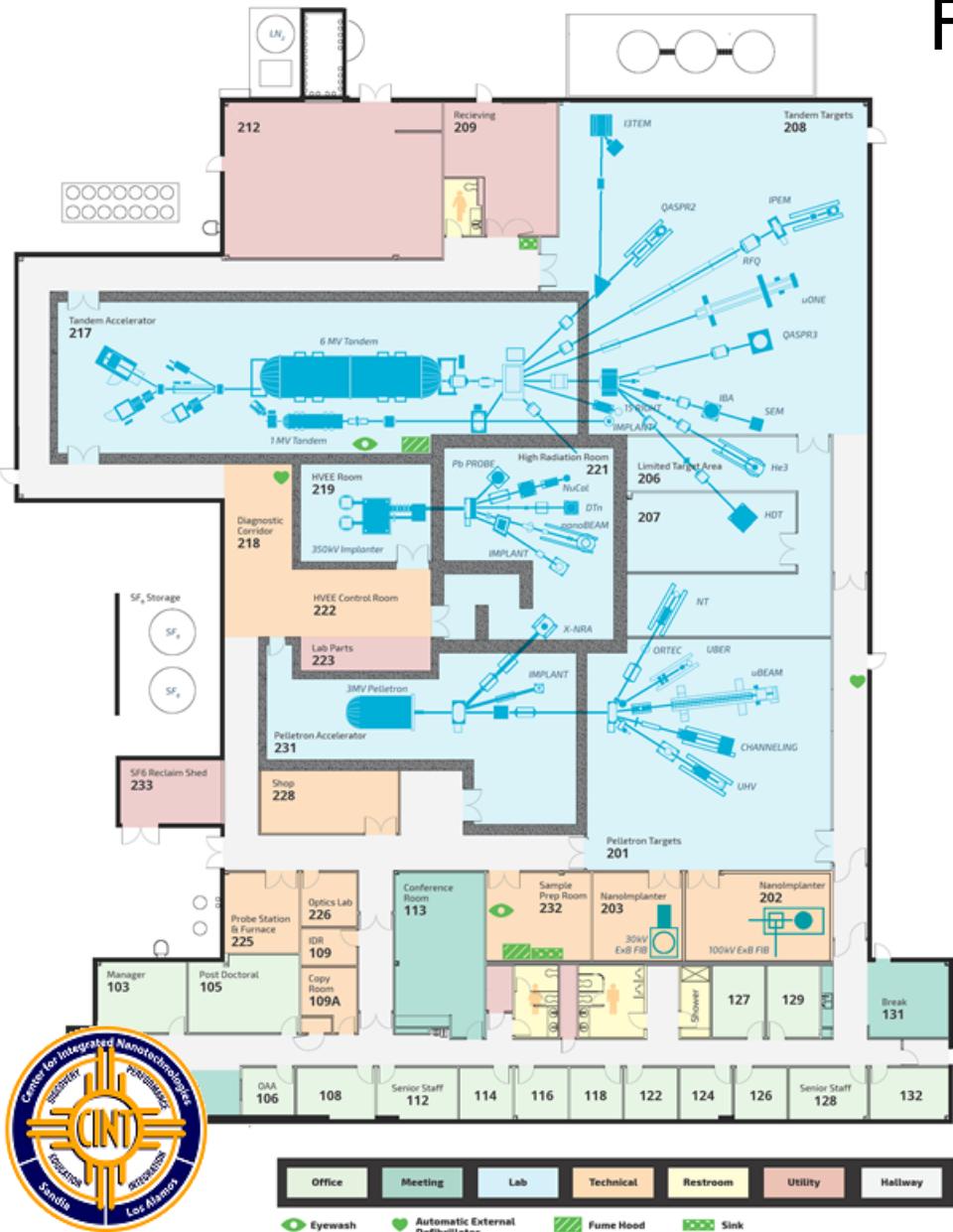


SRIM simulation - Z straggle

200 keV Si into diamond  
 $131 \pm 26$  nm



# Overview of Sandia's Ion Beam Laboratory (IBL) Focused Ion Beam (FIB) Capabilities



## Operational

- (1) 6 MV Tandem Accelerator
- (2) 3 MV Pelletron Accelerator
- (3) 1 MV Tandem Accelerator
- (4) 350 kV HVEE Implanter
- (5) 100 kV ExB FIB nanolplanter
- (6) 35 kV ExB FIB Raith Velion
- (7) 35 kV Zeiss HelM

## Installing

- (8) 35 kV Plasma FIB

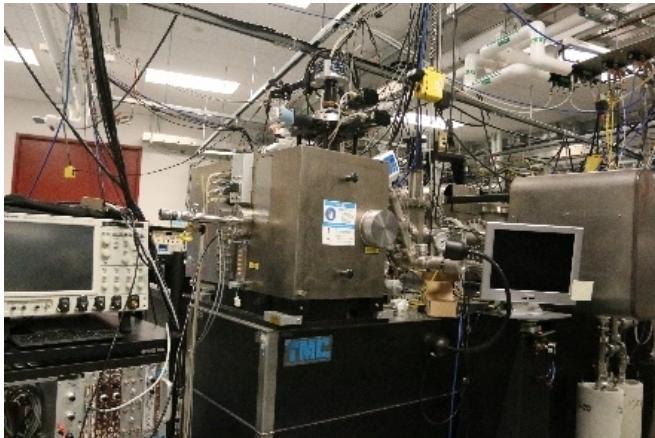
High energy  
focused  
micobeams  
1  $\mu$ m

Low energy  
focused  
nanobeams  
<1 to 20 nm

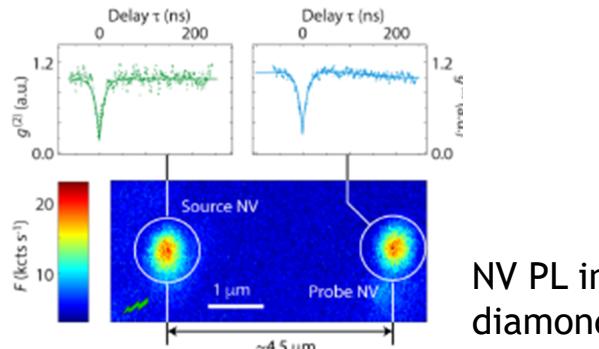
25 end-stations including *in-situ* DLTS, PL, TEM, SEM, 1200°C heating, etc...

# High Energy Focused Ion Beams $\sim 1 \mu\text{m}$

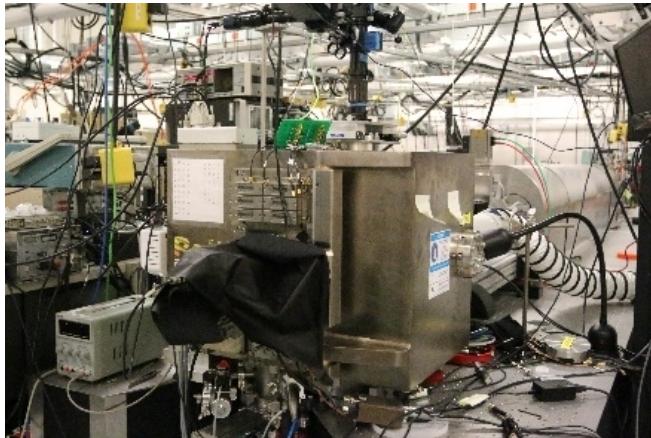
- 6 MV Tandem microbeam (microONE)



- High resolution laser stage
- Spot size  $<1 \mu\text{m}$
- Energy 0.8 - 70 MeV
- H to Au



- 3 MV Pelletron microbeam (Light Ion Microbeam)

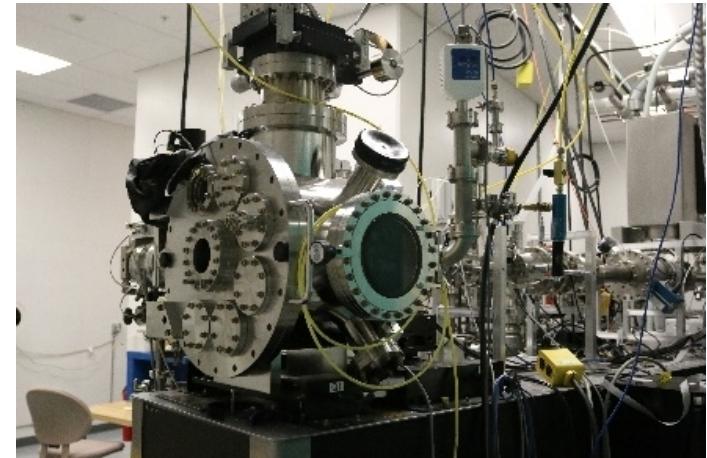


- High resolution laser stage
- Spot size  $<600 \text{ nm}$
- Energy 0.25 - 3 MeV
- H, He, N, Ar, Xe, ...

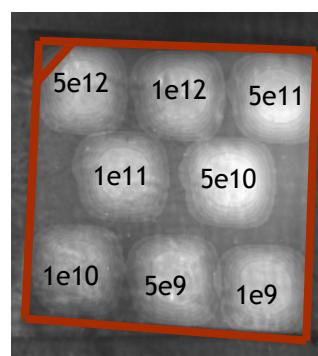


IBIC on PIN diode

- 350 kV HVEE microbeam (NanoBeamLine)



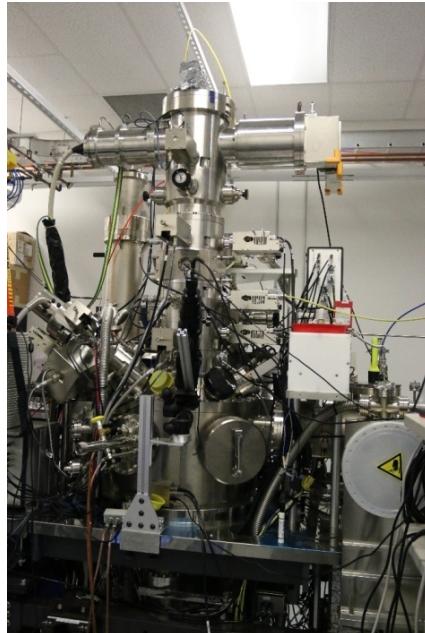
- Piezo stage
- Spot size  $<1 \mu\text{m}$
- Energy 20 - 350 keV
- H to Au



Ni into diamond

# Low Energy Focused Ion Beams <1 to 20 nm

- 100 kV A&D FIB100NI  
(nanolmplanter)



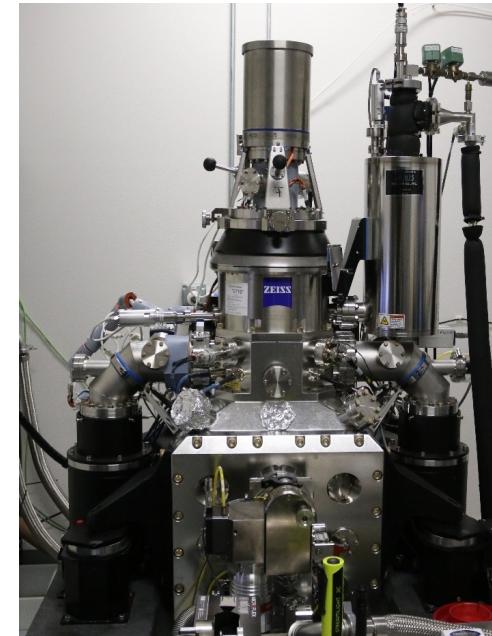
- High resolution laser stage
- Spot size <10 nm (Ga)
- Energy 10 - 200 keV
- 1/3 periodic table

- 35 kV Raith Velion  
(Velion)



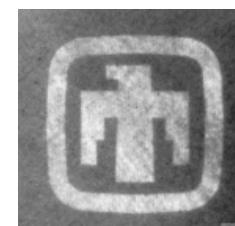
- High resolution laser stage
- Spot size <6 nm (Ga)
- Energy 5 - 70 keV
- 1/3 periodic table

- 35 kV Zeiss Orion Plus  
(HeLM)

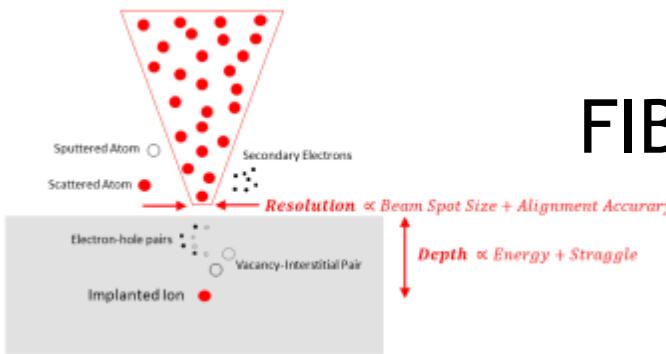


- Piezo stage
- Spot size <1 nm
- Energy 10 - 35 keV
- He

*All three equipped with Raith lithography software for patterning*



# FIB Implantation Resolution



## *What our resolution?*

### High Energy FIB

Spot Size  $\sim 1 \mu\text{m}$

Alignment accuracy  $\sim 1 \mu\text{m}$

Overall resolution  $\sim 1\text{-}2 \mu\text{m}$

### Low Energy FIB

Spot Size  $\sim$  few nm

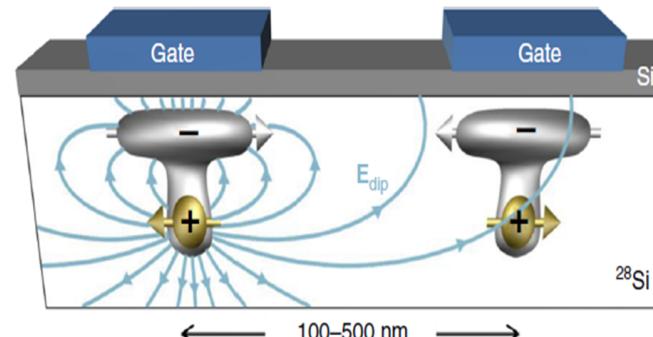
Alignment accuracy  $\sim 10\text{'s nm}$

Overall resolution  $\sim 20\text{-}50 \text{ nm}$

## *What is the needed resolution?*

Silicon quantum processor with robust long-distance qubit couplings

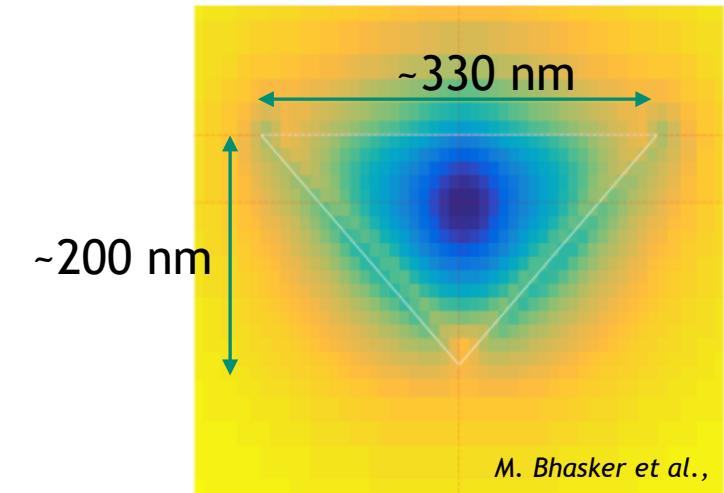
Guilherme Tosi<sup>1</sup>, Fahd A. Mohiyaddin<sup>1,3</sup>, Vivien Schmitt<sup>1</sup>, Stefanie Tenberg<sup>1</sup>, Rajib Rahman<sup>2</sup>, Gerhard Klimeck<sup>2</sup> & Andrea Morello<sup>1</sup>



G. Tosi et al., Nat. Comm. 8, 450 (2017)

- Depth  $z_d = 15\text{-}20 \text{ nm}$
- Separation of 100-500 nm

### Nanophotonic Applications



Center of mode is  $\sim 55 \text{ nm}$  below the surface of the waveguide

Low Energy Implantation? Lateral Positioning - OK, Target Depth - OK!

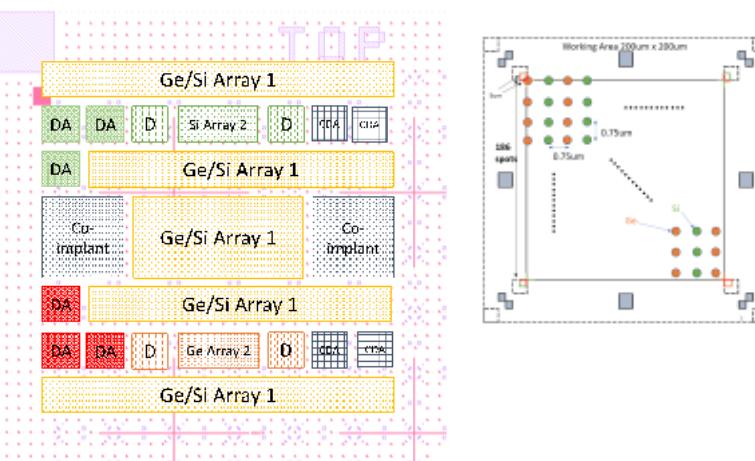
# Practical Example of FIB Implantation



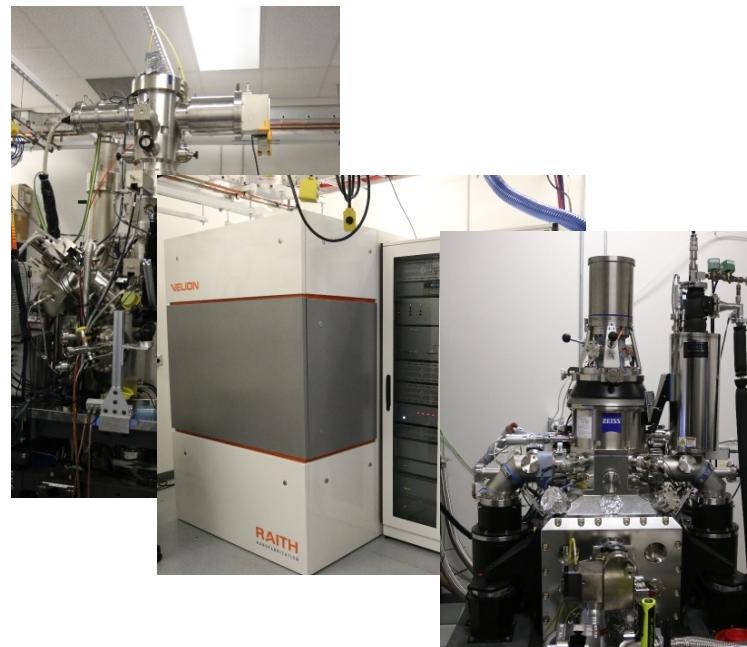
## Conversation of what is needed?



## Design and layout of sample



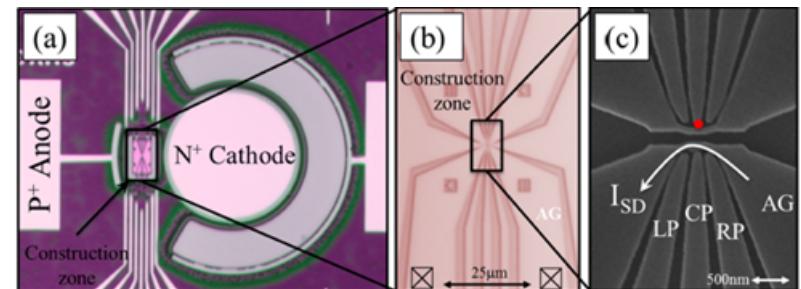
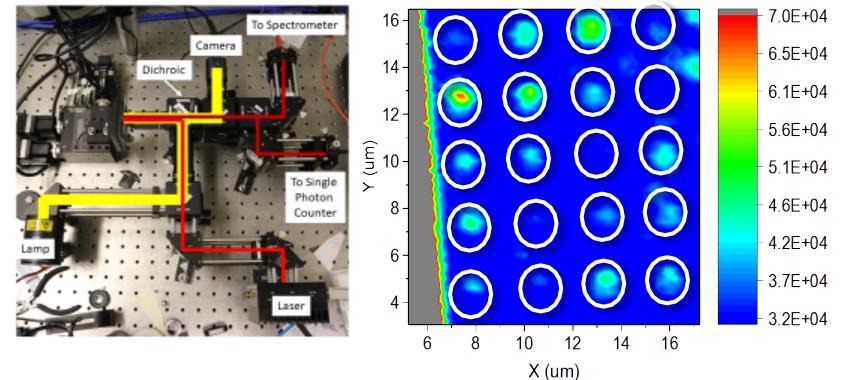
## FIB Implantation/Irradiation



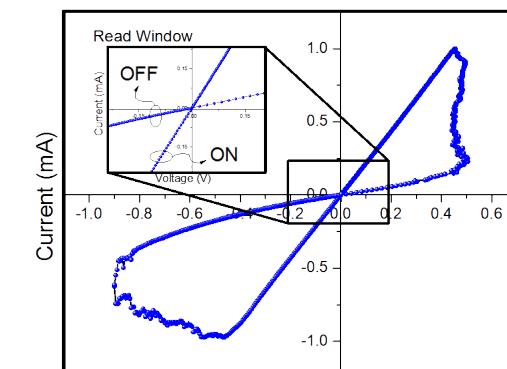
## Post Implantation Sample Prep



## Post Implantation Characterization



J. Pachaco, et al., Rev. of Sci. Instr. (2017)



J. Pachaco, et al. Appl. Phys. A 124 626 (2018)

# How can you access these capabilities? CINT User Proposals





## THE CENTER FOR INTEGRATED NANOTECHNOLOGIES



**One Scientific Community Focused on Nanoscience Integration**

The Center for Integrated Nanotechnologies (CINT) is a Department of Energy Office of Science Nanoscale Science Research Center. CINT offers world-leading scientific expertise and specialized capabilities to create, characterize, and integrate nanostructured materials at a range of length scales, from the nano- to meso-scale. It is jointly operated by Los Alamos and Sandia national laboratories and leverages the unmatched scientific and engineering expertise of the host labs.

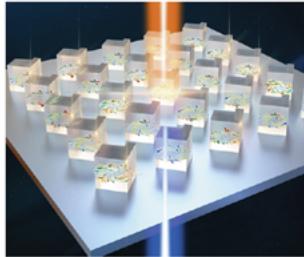
### Science Thrusts

Integration is the key to exploiting the novel properties of nanoscale materials and creating new technologies. CINT's scientific staff and capabilities are organized around four interdisciplinary science thrusts which address different challenges in nanoscience integration.



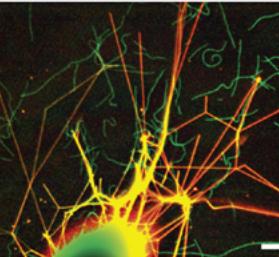
#### *In-Situ Characterization and Nanomechanics*

Developing and implementing world-leading capabilities to study the dynamic response of materials and nanosystems to mechanical, electrical, or other stimuli.



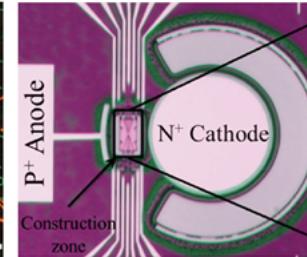
#### *Nanophotonics & Optical Nanomaterials*

Synthesis, excitation, and energy transformations of optically active nanomaterials and collective or emergent electromagnetic phenomena (plasmonics, metamaterials, photonic lattices).



#### *Soft, Biological & Composite Nanomaterials*

Synthesis, assembly, and characterization of soft, biomolecular, and composite nanomaterials that display emergent functionality.



#### *Quantum Materials Systems*

Understanding and controlling quantum effects of nanoscale materials and their integration into systems spanning multiple length scales.

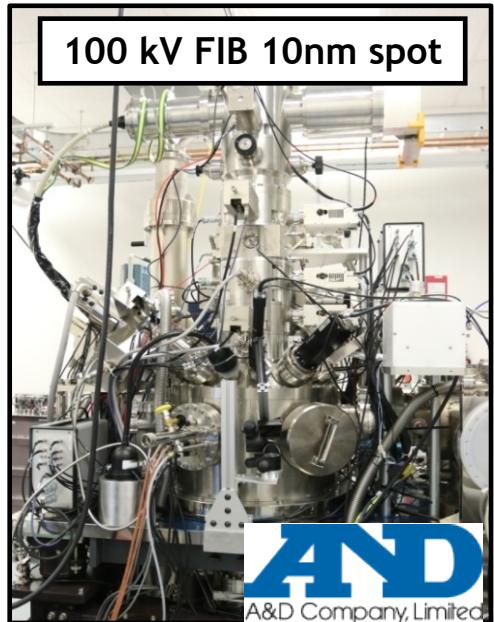
### User Program

CINT is an Office of Science national user facility. CINT helps the international research community perform cutting-edge research in the areas of nanoscience and nanotechnology, and is available free of charge for open science. As a user facility, CINT has the structure and mission to collaborate widely across academia, industry, and within DOE labs. Access is via peer-reviewed technical proposals. Proprietary research may be conducted in accordance with Federal regulations for full-cost recovery. CINT cannot provide funding to users.

The Center for Integrated Nanotechnologies (CINT) has both regular Fall and Spring call for proposals and Rapid Access available

<https://cint.lanl.gov/>

# FIB Implantation using the nanolimplanter and Velion

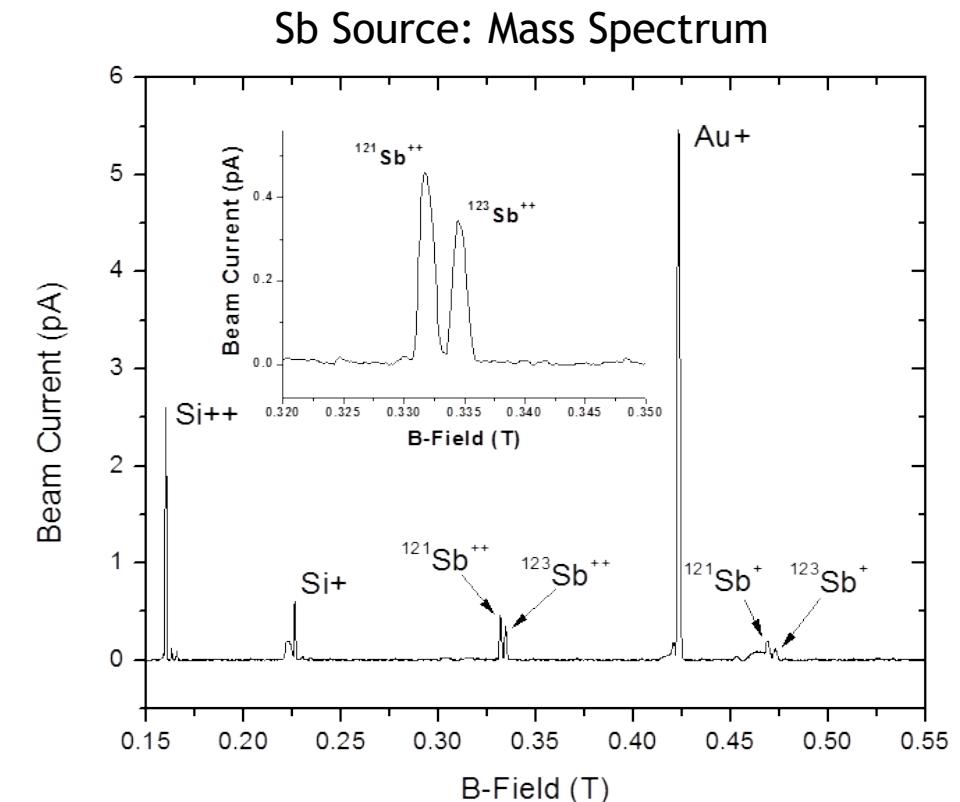
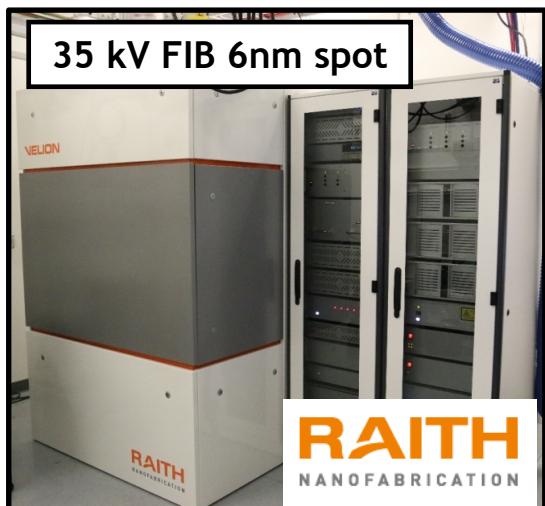


- Focused ion beam system (FIB)  
→ nm beam spot size on target
- ExB Filter (Wien Filter)  
→ Multiple ion species  
e.g., Li, Si, P, Sb, etc... (separating out  $^{28}\text{Si}$ ,  $^{29}\text{Si}$ , etc...)

- Fast blanking and chopping  
→ Single ion implantation

- Direct-write lithography  
→ nm targeting accuracy

- Low temperature stage
- In-situ electrical probes



# Available Liquid Metal Alloy Ion Sources (LMAIS) for FIB Implantation



hydrogen	1	H	1.0079
lithium	3	beryllium	4
Li	6.941	Be	9.0122
sodium	11	magnesium	12
Na	22.990	Mg	24.305
potassium	19	calcium	20
K	39.098	Ca	40.078
rubidium	37	strontium	38
Rb	85.468	Sr	87.62
caesium	55	barium	56
Cs	132.91	Ba	137.33
francium	87	radium	88
Fr	[223]	Ra	[226]

Purple - running at SNL  
 Yellow - attempting at SNL  
 Green - demonstrated at other labs

scandium	21	titanium	22	vanadium	23	chromium	24	manganese	25	iron	26	cobalt	27	nickel	28	copper	29	zinc	30	gallium	31	aluminum	13	boron	5	carbon	6	nitrogen	7	oxygen	8	fluorine	9	helium	2		
Sc	44.966	Ti	47.867	V	50.942	Cr	51.996	Mn	54.938	Fe	55.845	Co	58.933	Ni	58.693	Cu	63.546	Zn	65.39	Ga	69.723	Al	10.811	B	12.011	C	14.007	O	15.999	Sulfur	16	Neon	10				
Yttrium	39	Zirconium	40	Niobium	41	Molybdenum	42	Technetium	43	Ruthenium	44	Rhodium	45	Palladium	46	Silver	47	Cadmium	48	Indium	49	Tin	50	Antimony	51	Germanium	31	As	33	Sulfur	16	Neon	10				
Rb	88.906	Zr	91.224	Nb	92.906	Mo	95.94	Tc	[98]	Ru	101.07	Rh	102.91	Pd	106.42	Ag	107.87	Cd	112.41	In	114.82	Sn	118.71	Te	121.76	Ge	72.61	As	74.922	Se	78.96	Br	83.80				
caesium	55	barium	56	lutetium	71	hafnium	72	tantalum	73	tungsten	74	rhenium	75	osmium	76	iridium	77	platinum	78	gold	79	mercury	80	thallium	81	lead	82	bismuth	83	polonium	84	astatine	85	radon	86		
Cs	132.91	Ba	137.33	*	Lu	Hf	Ta	W	Re	Os	186.21	190.23	192.22	196.08	196.97	196.97	Pt	200.59	Hg	204.38	Tl	207.2	Pb	208.98	Bi	[209]	Po	[210]	At	[221]	Rn	[222]					
francium	87	radium	88	lawrencium	103	rutherfordium	104	dubnium	105	seaborgium	106	bohrium	107	hassium	108	meitnerium	109	unnilmillsium	110	unnilonium	111	ununbium	112	ununquadium	114	Uuq	[289]										

\* Lanthanide series

lanthanum	57	cerium	58	praseodymium	59	neodymium	60	promethium	61	samarium	62	europerium	63	gadolinium	64	terbium	65	dysprosium	66	holmium	67	erbium	68	thulium	69	ytterbium	70
La	138.91	Ce	140.12	Pr	140.91	Nd	144.24	Pm	[145]	Sm	150.36	Eu	151.96	Gd	157.25	Tb	158.93	Dy	162.50	Ho	164.93	Er	167.26	Tm	168.93	Yb	173.04
actinium	89	thorium	90	protactinium	91	uranium	92	neptunium	93	plutonium	94	americium	95	curium	96	berkelium	97	californium	98	einsteinium	99	fermium	100	mendelevium	101	nobelium	102
Ac	[227]	Th	232.04	Pa	231.04	U	238.03	Np	[237]	Pu	[244]	Am	[243]	Cm	[247]	Bk	[247]	Cf	[251]	Es	[252]	Fm	[257]	Md	[258]	No	[259]

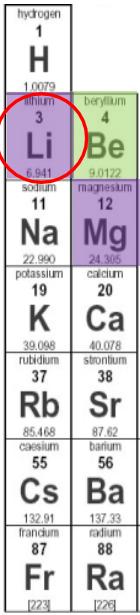
\*\* Actinide series

After A. Weick University of Bochum

L. Bischoff, *et al.*, Applied Physics Reviews 3, 021101 (2016)

Wide Variety of Ion Species Available and Developing More!

## Examples of New Sources - Li, V, Cr, Fe, Zn, Sn, Tm (easy-ish)



## Purple - running at SNL

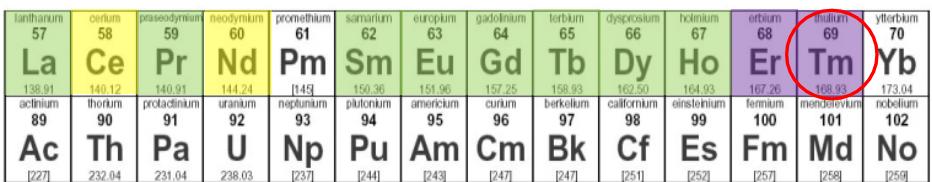
## Yellow - attempting at SNL

## Green - demonstrated at other labs



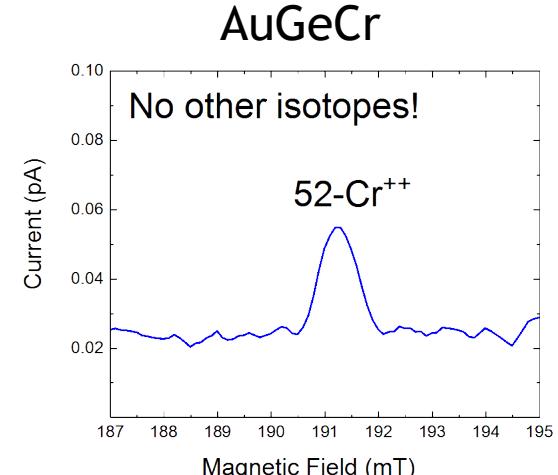
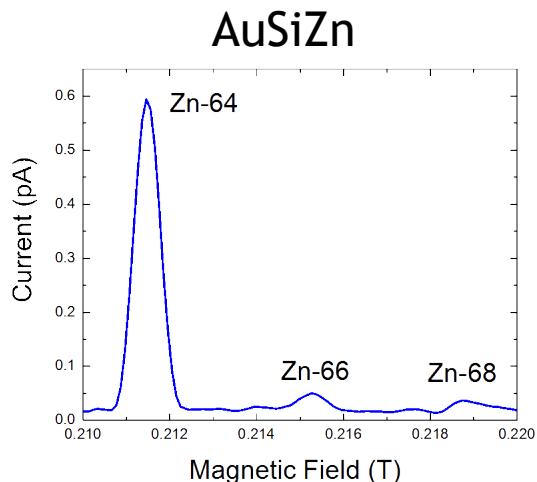
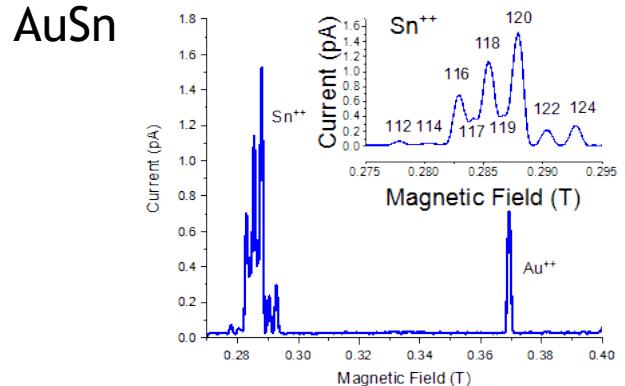
\* Lanthanide series

### \*\*Actinide series



- Based on AuSiX or AuGeX alloys
- Easy to wet the tip and easy to run
- For example, AuSiLi

M. Titze *et al.*, JVST B, 39, 012802 (2021)





# Examples of **HARD** New Sources - Pb and N

hydrogen	1	H	1.0079
beryllium	4	Be	9.0122
lithium	3	Li	6.941
magnesium	12	Mg	24.305
sodium	11	Na	22.990
potassium	19	K	39.098
calcium	20	Ca	40.078
rubidium	37	Rb	85.468
strontium	38	Sr	87.62
cesium	55	Cs	132.91
barium	56	Ba	137.33
franidium	87	Fr	223
radium	88	Ra	226

Purple - running at SNL

Yellow - attempting at SNL

Green - demonstrated at other labs

scandium	21	Sc	44.96
titanium	22	Ti	47.867
vanadium	23	V	50.942
chromium	24	Cr	51.996
manganese	25	Mn	54.028
iron	26	Fe	55.845
cobalt	27	Co	58.033
nickel	28	Ni	58.693
copper	29	Cu	63.546
zinc	30	Zn	65.39
gallium	31	Ga	69.723
germanium	32	Ge	72.61
arsenic	33	As	74.922
selenium	34	Se	79.96
bromine	35	Br	79.994
krypton	36	Kr	83.80
rubidium	37	Rb	85.468
strontium	38	Sr	87.62
yttrium	39	Y	88.906
zirconium	40	Zr	91.224
niobium	41	Nb	92.906
molybdenum	42	Mo	[96]
technetium	43	Tc	101.07
ruthenium	44	Ru	102.91
rhenium	45	Rh	106.42
palladium	46	Pd	107.87
silver	47	Ag	112.41
cadmium	48	Cd	114.82
indium	49	In	118.71
tin	50	Sn	121.76
antimony	51	Sb	127.60
tellurium	52	Te	126.90
iodine	53	I	131.29
xenon	54	Xe	132.91
cesium	55	Cs	137.33
barium	56	Ba	*
lutetium	71	Lu	174.97
hafnium	72	Hf	178.49
tantalum	73	Ta	183.84
tungsten	74	W	186.21
rhodium	75	Re	190.23
osmium	76	Os	192.22
iridium	77	Ir	196.97
platinum	78	Pt	199.08
gold	79	Au	200.59
mercury	80	Hg	204.38
thallium	81	Tl	207.2
lead	82	Pb	208.98
bismuth	83	Bi	[209]
polonium	84	Po	[210]
astatine	85	At	[221]
radon	86	Rn	[222]
franidium	87	Fr	*
radium	88	Ra	*
lawrencium	103	Lr	[262]
rutherfordium	104	Rf	[261]
dubnium	105	Db	[262]
seaborgium	106	Sg	[266]
bohrium	107	Bh	[264]
hassium	108	Hs	[269]
meitnerium	109	Mt	[268]
ununnilium	110	Uun	[271]
ununnilium	111	Uuu	[272]
ununquadium	112	Uub	[277]
ununquadium	114	Uuq	[289]

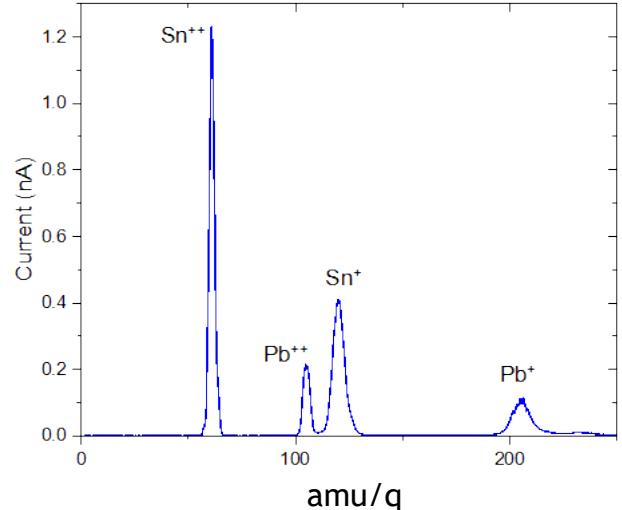
\*Lanthanide series

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La	138.91	Ce	140.12	Pr	140.91	Nd	144.24	Pm	[149]	Sm	150.36	Eu	151.96	Tb	157.25	158.93	Dy	162.50	164.93	Ho	167.26	Er	168.93	Tm	169.3	Yb	173.04	
actinium	89	thorium	90	protactinium	91	uranium	92	neptunium	93	plutonium	94	americium	95	curium	96	berkelium	97	californium	98	einsteiniun	99	fermium	100	mandeleium	101	nobelium	102	No
Ac	[227]	Th	232.04	Pa	231.04	Np	238.03	Pu	[237]	Am	[244]	Cm	[247]	Bk	247	Cf	[251]	Es	[252]	Fm	[257]	Md	[258]	No	[259]			

\*\*Actinide series

- Pb

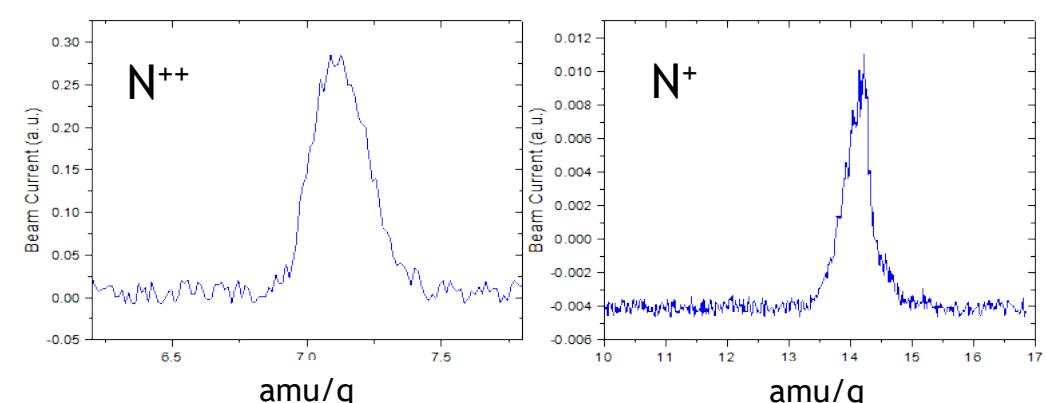
→ PbSn alloys with W tip, use Kovar tips!



- N

→ Liquid metal alloys with implanted N

i.e., AuSn+N





# Examples of **HARD** New Sources - C

hydrogen	1	H	1.0079
lithium	3	beryllium	4
Li	6.941	Be	9.0122
sodium	11	magnesium	12
Na	22.990	Mg	24.305
potassium	19	calcium	20
K	39.098	Scandium	21
Rb	85.468	Titanium	22
Ca	40.078	Vanadium	23
Yttrium	39	Chromium	24
Rb	87.62	Manganese	25
Sr	88.906	Iron	26
Sc	44.96	Cobalt	27
Nb	50.942	Nickel	28
Ti	47.867	Copper	29
Zr	51.996	Zinc	30
Mo	54.028	Gallium	31
Y	55.942	Germanium	32
Re	55.945	Arsenic	33
Lu	56.996	Selenium	34
Hf	57.996	Bromine	35
Ta	58.996	Krypton	36
W	59.996	Phosphorus	15
Os	60.996	Sulfur	16
Ir	61.996	Chlorine	17
Pt	62.996	Argon	18
Au	63.996	Aluminum	13
Hg	64.996	Silicon	14
Tl	65.996	Phosphorus	15
Pb	66.996	Sulfur	16
Bi	67.996	Chlorine	17
Fr	68.996	Argon	18
Ra	69.996	Scandium	13
Fr	87.996	Titanium	22
Ra	88.996	Vanadium	23
Lawrencium	103	Chromium	24
Rutherfordium	104	Manganese	25
Db	105	Iron	26
Lr	106	Cobalt	27
Rf	107	Nickel	28
Sg	108	Copper	29
Bh	109	Zinc	30
Hs	110	Gallium	31
Mt	111	Germanium	32
Uun	112	Arsenic	33
Uuu	113	Selenium	34
Uub	114	Bromine	35
Uq	115	Krypton	36

hydrogen	1	H	1.0079
lithium	3	beryllium	4
Li	6.941	Be	9.0122
sodium	11	magnesium	12
Na	22.990	Scandium	21
K	39.098	Titanium	22
Rb	85.468	Vanadium	23
Ca	40.078	Chromium	24
Yttrium	39	Manganese	25
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W	59.996	Sulfur	16
Os	60.996	Chlorine	17
Ir	61.996	Argon	18
Pt	62.996	Scandium	13
Au	63.996	Titanium	22
Hg	64.996	Vanadium	23
Tl	65.996	Chromium	24
Pb	66.996	Manganese	25
Bi	67.996	Iron	26
Fr	68.996	Cobalt	27
Ra	69.996	Nickel	28
Fr	87.996	Copper	29
Ra	88.996	Zinc	30
Lawrencium	103	Gallium	31
Rutherfordium	104	Germanium	32
Db	105	Arsenic	33
Lr	106	Selenium	34
Rf	107	Bromine	35
Sg	108	Krypton	36
Bh	109	Phosphorus	15
Hs	110	Sulfur	16
Mt	111	Chlorine	17
Uun	112	Argon	18
Uuu	113	Scandium	13
Uub	114	Titanium	22
Uq	115	Vanadium	23

Purple - running at SNL

Yellow - attempting at SNL

Green - demonstrated at other labs

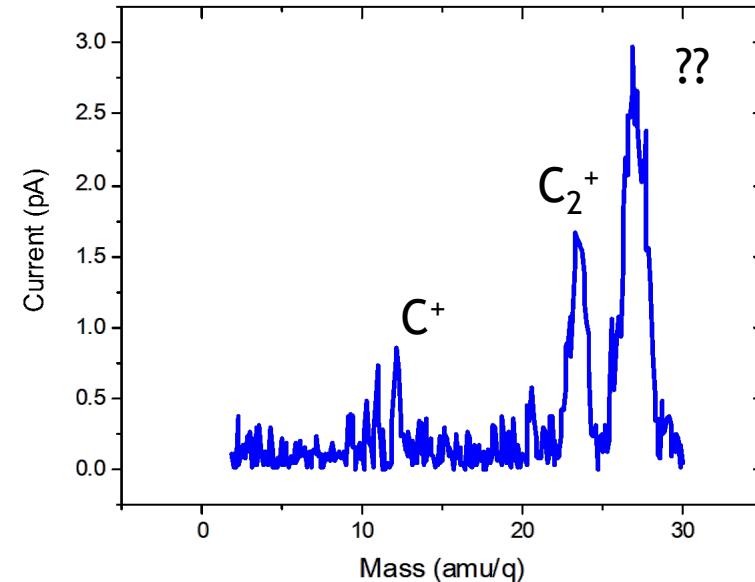
\* Lanthanide series

\*\* Actinide series

Lanthanum	57	cerium	58	praseodymium	59	neodymium	60	promethium	61	samarium	62	europium	63	gadolinium	64	terbium	65	dysprosium	66	holmium	67	erbium	68	thulium	69	ytterbium	70
La	138.91	Ce	140.12	Pr	140.91	Nd	144.24	Pm	149.1	Sm	150.36	Eu	151.96	Gd	157.25	Tb	158.93	Dy	162.50	Ho	164.93	Er	167.26	Tm	168.93	Yb	173.04
actinium	89	thorium	90	protactinium	91	uranium	92	neptunium	93	plutonium	94	americium	95	curium	96	berkelium	97	californium	98	einsteiniun	99	fermium	100	mandeleivium	101	nobelium	102
Ac	[227]	Th	232.04	Pa	231.04	U	238.03	Np	[237]	Pu	[244]	Am	[243]	Cm	[247]	Bk	[247]	Cf	[251]	Es	[252]	Fm	[257]	Md	[258]	No	[259]

- C

→ CeC alloy with



(1) Source fabricated at Helmholtz-Zentrum Dresden-Rossendorf

(2) Source tested at Raith

(3) Source used at SNL to implant XXX

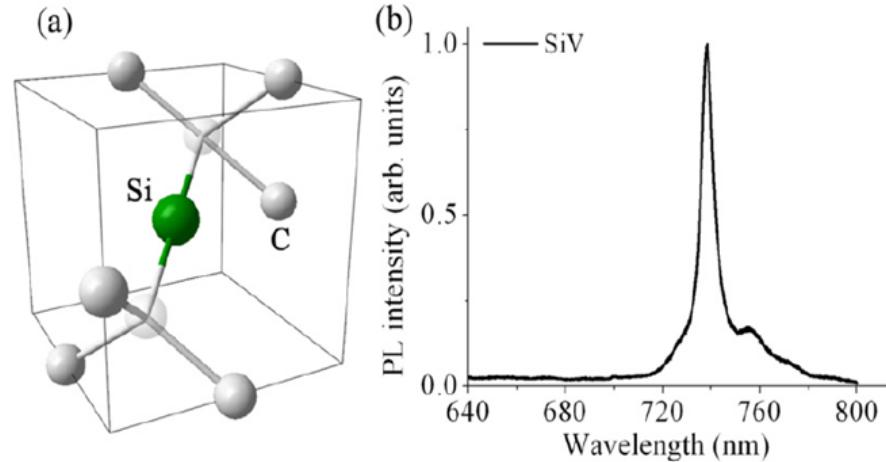
# Creation of Single Defect Centers in Wide Bandgap Substrates



- Defect centers in wide bandgap substrates have applications ranging from metrology, quantum computing, quantum sensing to biological

Ex. Silicon Vacancy Centers in diamond

The ion beam implantation and detection techniques are mainly material agnostic!

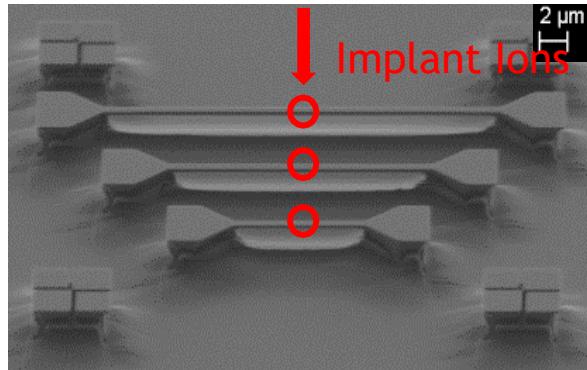


I. Aharonovich *et al.*, Rep. Prog. Phys. **74**, 076501 (2011)

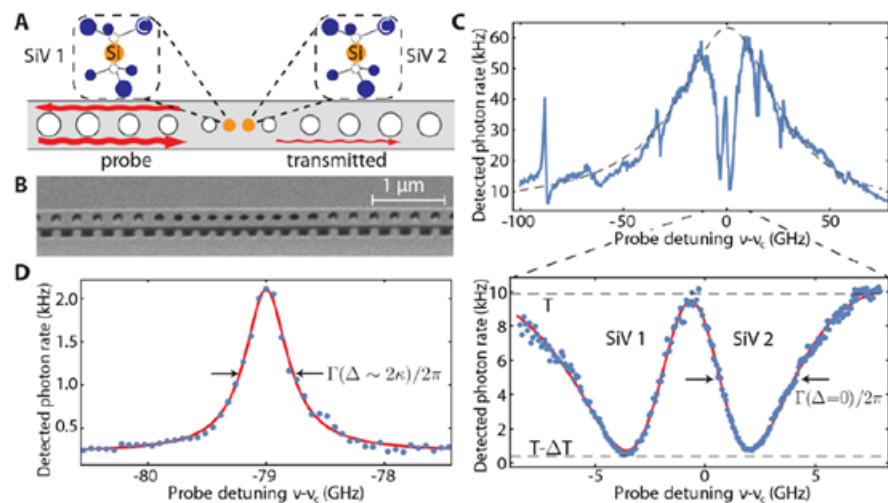
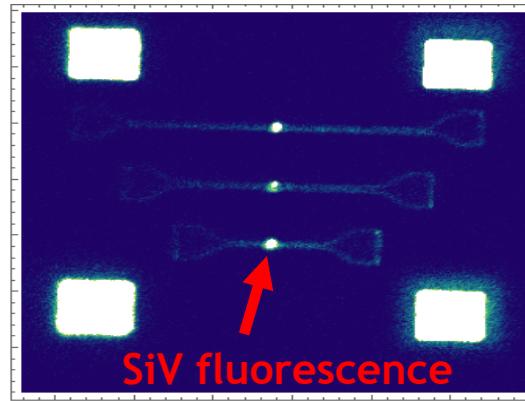
- How to produce a single defect center where you want it?
  - 1.) Location - focused ion beam implantation to control location
  - 2.) Yield - counted implantation to control the number of ions and in-situ PL to confirm an optically active defect center

# Location solved\*, use FIB implantation to control the spatial location

## Diamond Nanobeams (with Harvard)

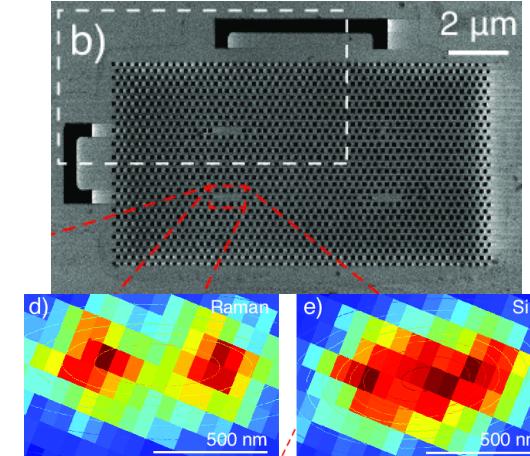


A. Sipahigil, *et al.*, *Science* (2016)

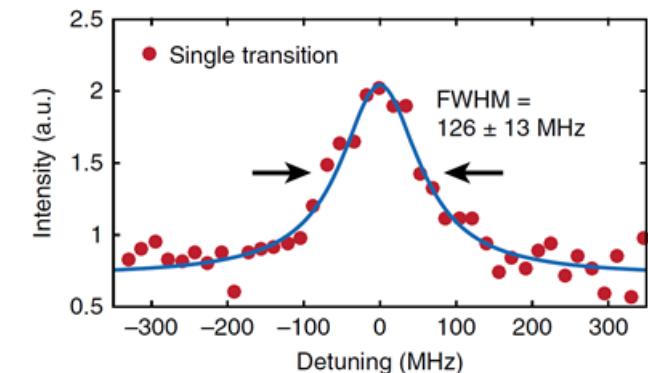


R. E. Evans, *et al.*, *Science* (2018)

## 2D Photonic Crystals (with MIT)



T. Schroder, *et al.*, *Nature Communications* (2017)



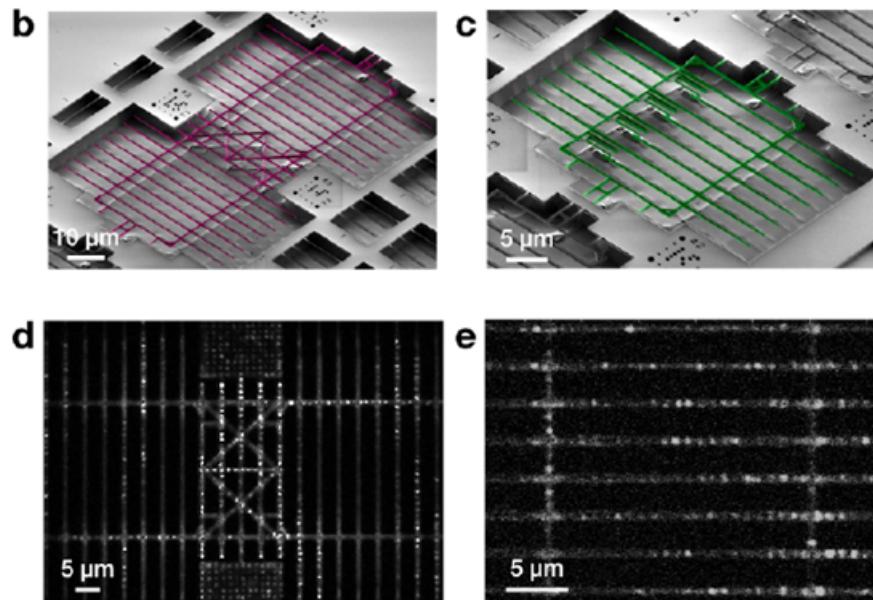
\* Application dependent

Spatial Accuracy is <50 nm

# Another set of CINT user projects using FIB Implantation

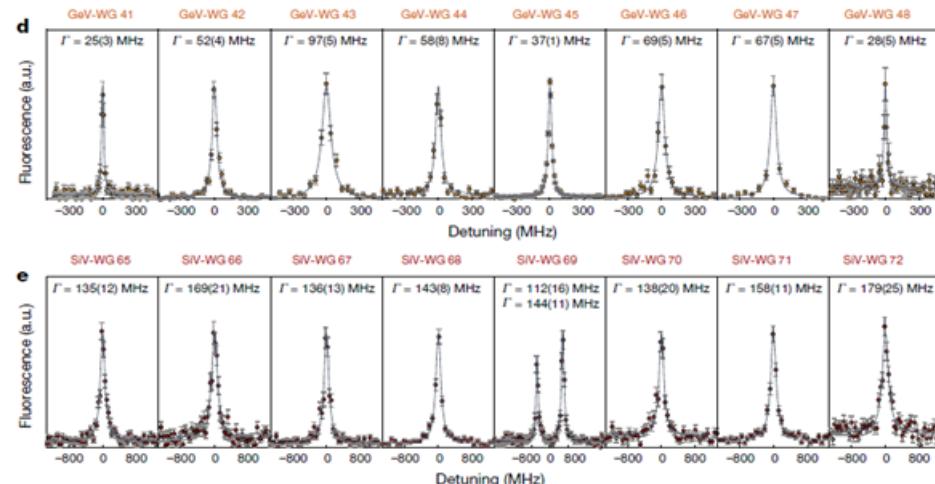


## Diamond waveguides with AlN photonics (with MIT)



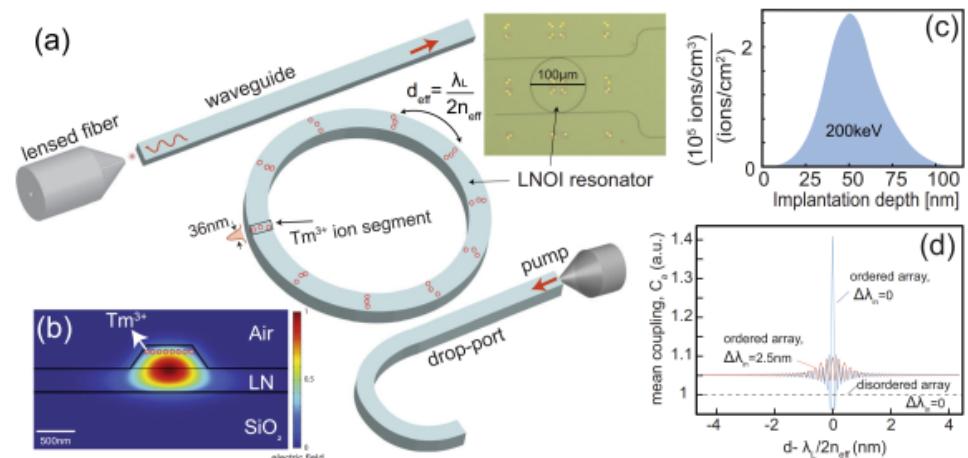
16 and 8 channel  
“quantum micro-  
chiplets”

Noel H. Wan *et al.*, *Nature*,  
583, 226-231(2020)

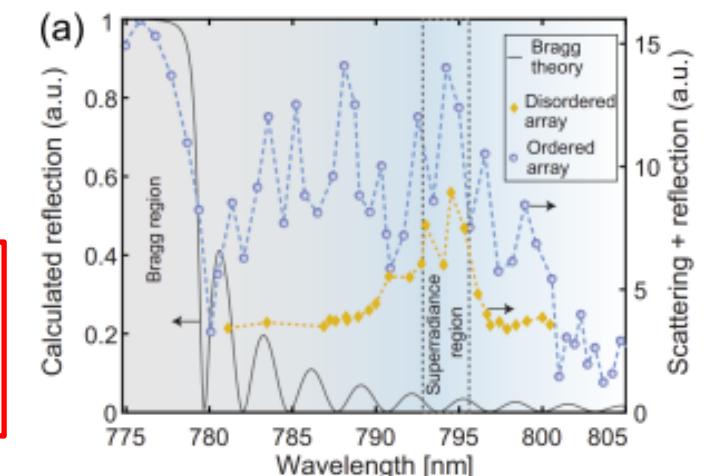


FIB implantation works  
for a wide range of  
substrates

## Lithium Niobate Photonics (with Purdue)



D. Pak *et al.*, *Comm. Physics*, 5, 89 (2022)



# We Use Counted Implantation and Photoluminescence to Better Understand the Yield

$$\text{Yield} = \# \text{ measured SiV} / \# \text{ implanted Si}$$



## In-situ Photoluminescence

- Low activation yield limits our ability to make high yield arrays

(Yield numbers are typically 3-10%)

- In-situ photoluminescence can reduce the error in the number of defect centers



## In-situ Counted Implantation

- Timed Implantation dominated by Poisson statistics for small numbers

(Uncertainty in number of ion is  $\sqrt{N}$  )

- In-situ counting can reduce the error in the number of implanted ions

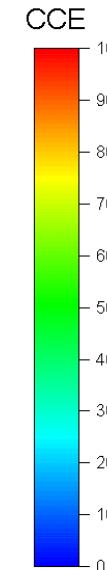
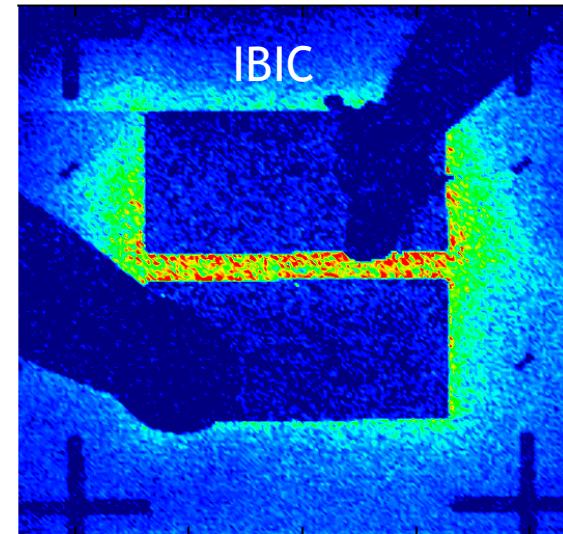
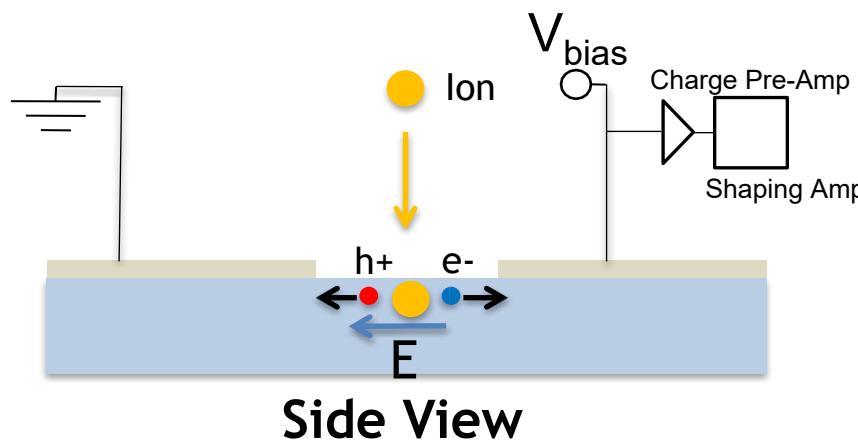
# In-situ Counted Ion Implantation to control the number of implanted ion

Yield = # measured SiV / # implanted Si

## Poisson Statistics

(Uncertainty in number of ion is  $\sqrt{N}$  )

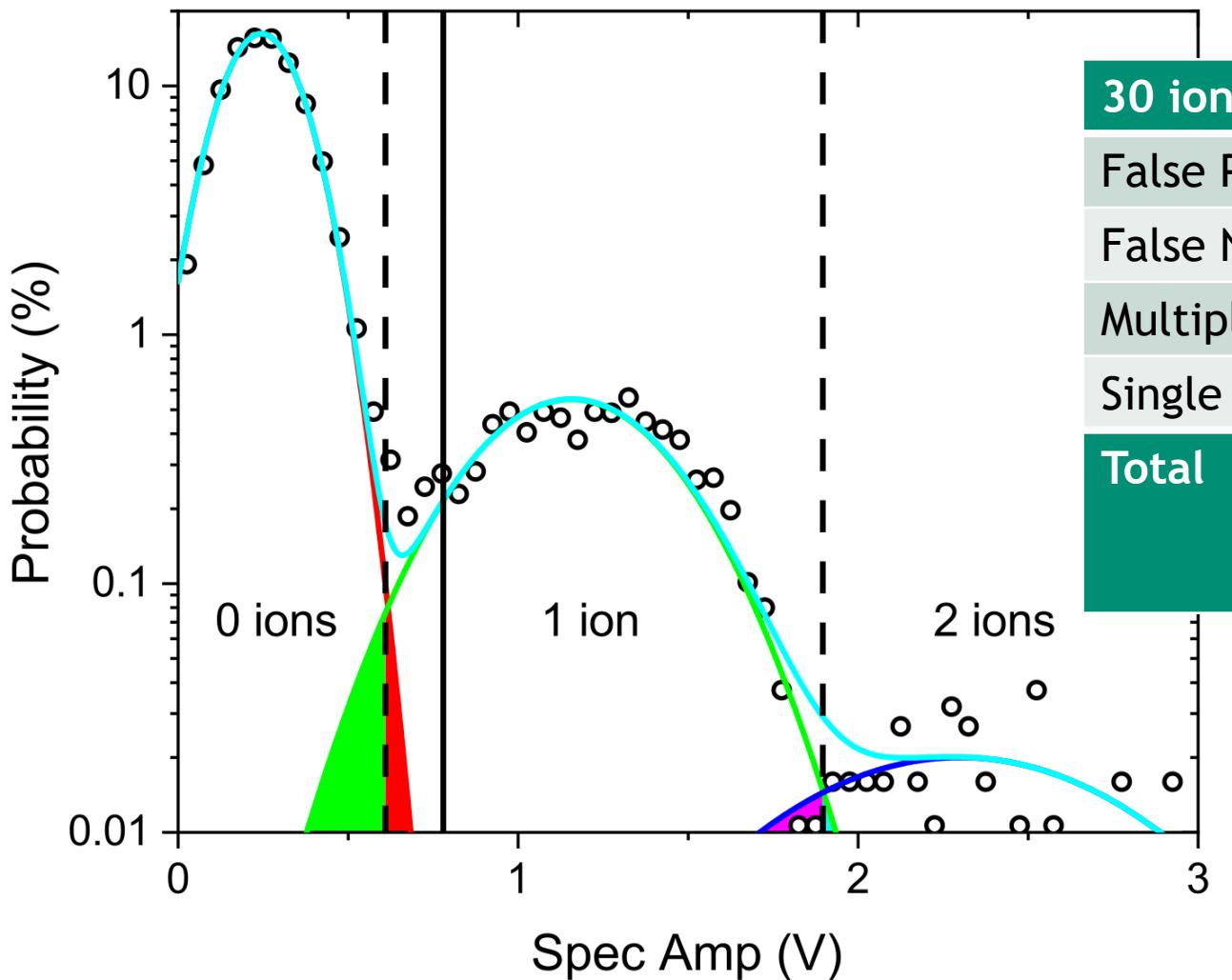
### Ion Beam Induced Charge (IBIC)



$$CCE = \frac{\text{Charge Collected}}{\text{Charge Deposited}} \times 100$$

IBIC/detection demonstrated for low energy heavy ions

# Demonstrated Single Ion Diamond Detection at $<0.1>$ ions/pulse



30 ions / $<1>$ SiIV	Timed	In-Situ	Post-Analysis
False Positives	-	< -1 ppb	2.3 %
False Negatives	-	8.6 %	-0.9 %
Multiple Ions	-	5.8 %	1.7 %
Single as Double	-	-	-0.2 %
<b>Total</b>	+18.3 / -18.3 %	+14.4 / -0 %	+4.0 / -1.1 %

- 7x improvement in the error in implanted ions as compared to timed implantation

Titze *et al*, Nano Letters 22, 3212-3218 (2022)

- Aside,  $<0.1>$  ions/pulse using a 1  $\mu$ s pulse length needs ~30 fA of beam current or ~100k ions/s

In-situ counting to reduce the error in the number of implanted ions

# In-situ Photoluminescence to confirm optical defect center



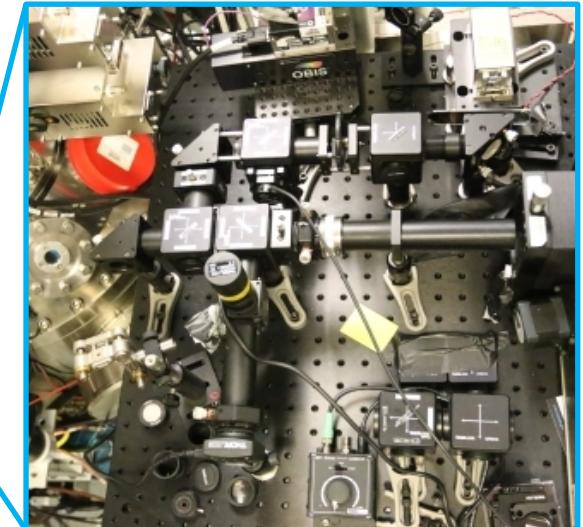
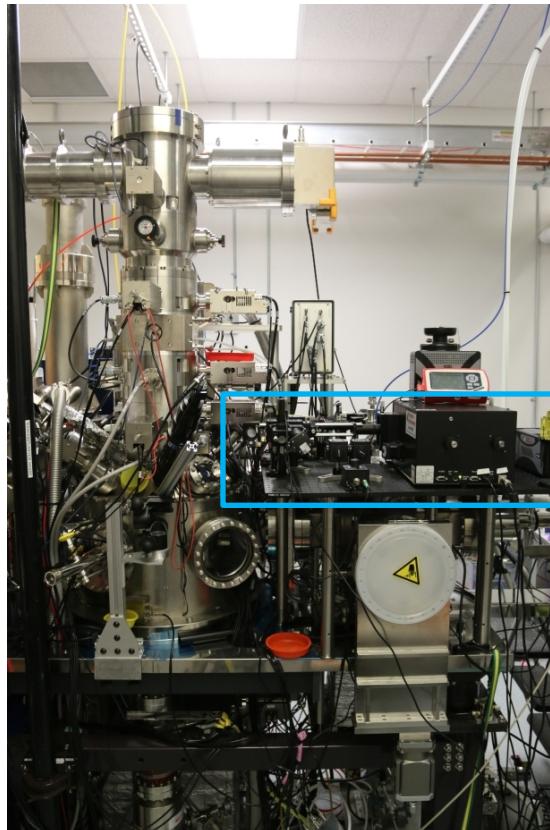
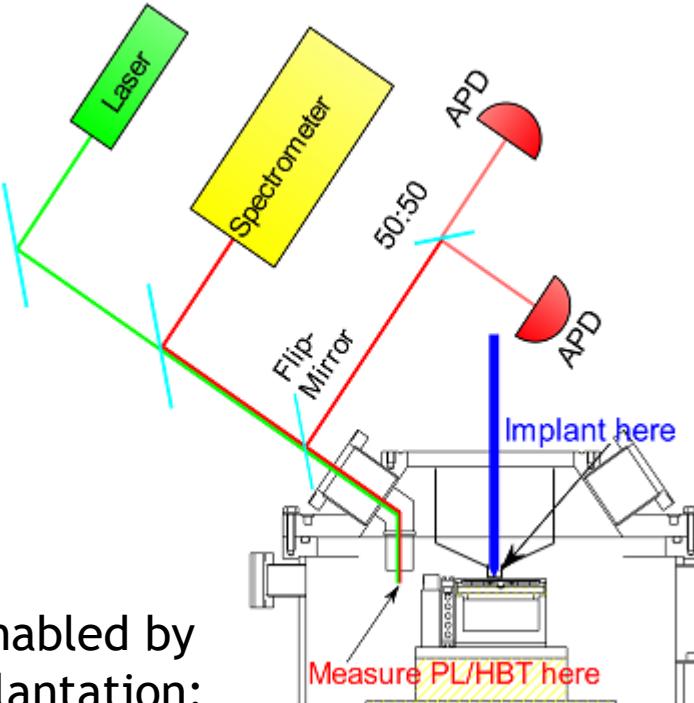
$$\text{Yield} = \# \text{ measured V}_{\text{Si}} / \# \text{ implanted Si}$$

We switched from SiV in diamond to V<sub>Si</sub> in SiC as can measure as implanted samples without annealing

Two-step process enabled by high resolution implantation:

- (1) Aligned implantation, <40 nm
- (2) Detect PL

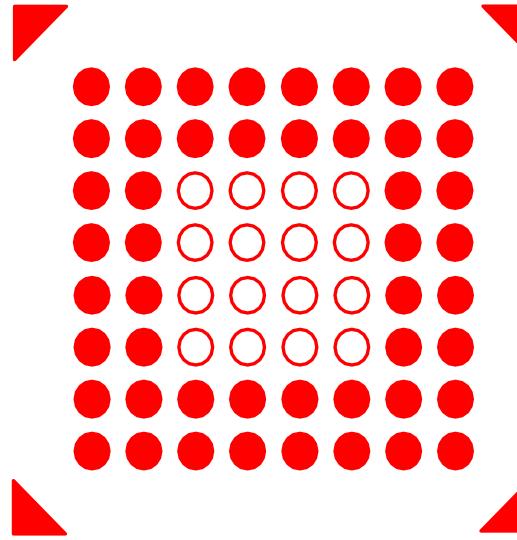
In-situ PL to confirm the optically emission from the defect centers



# Demonstration of In-situ Photoluminescence in SiC



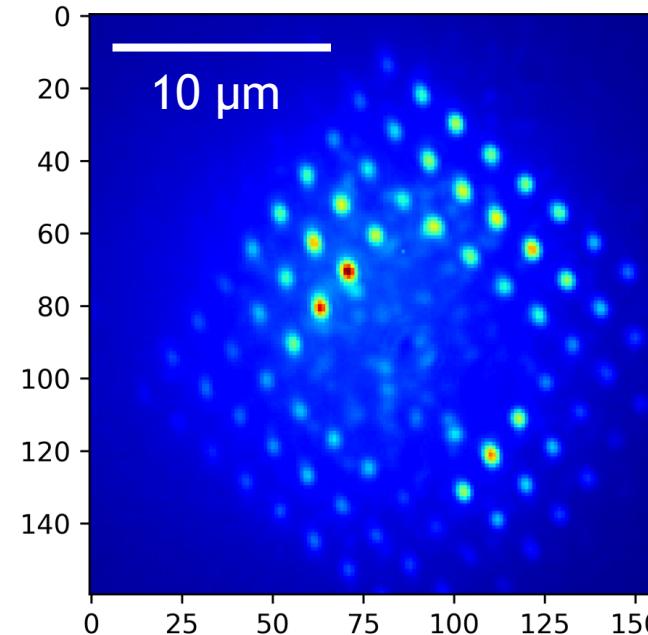
Design of array



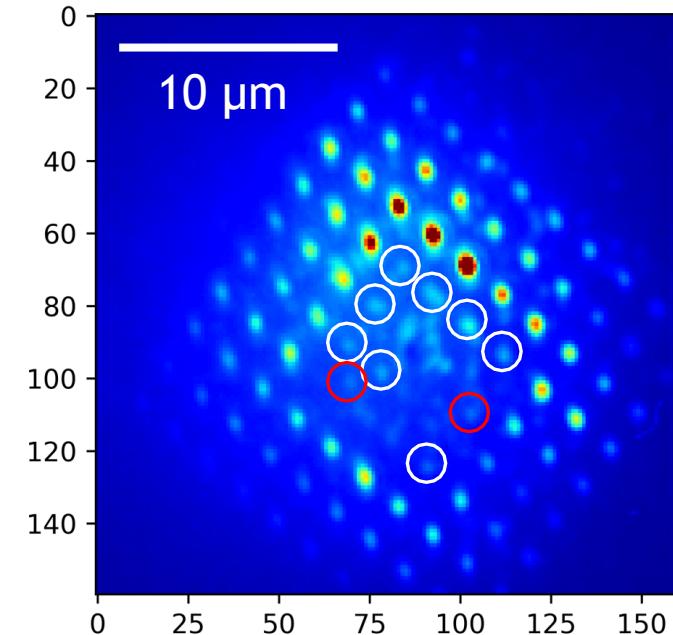
► Alignment markers

- <500> Ions/spot implanted once that acts as landmark to find in-situ arrays during PL check
- In-situ array points to be filled

Implant Alignment Grid



Implant/PL Repeat to fill Array



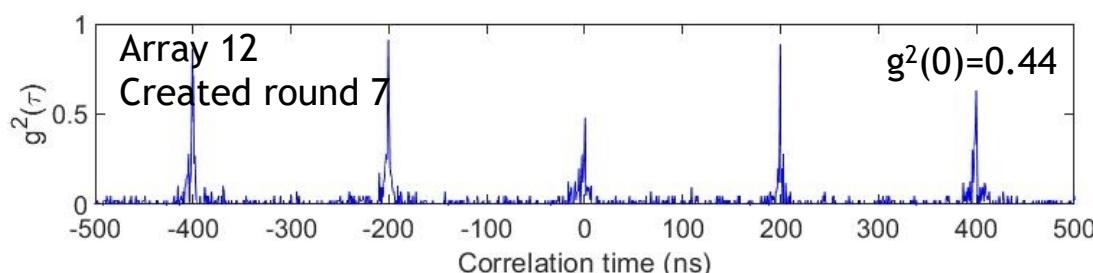
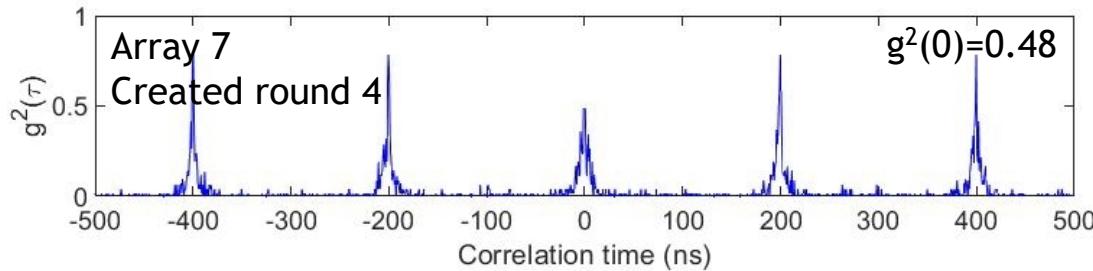
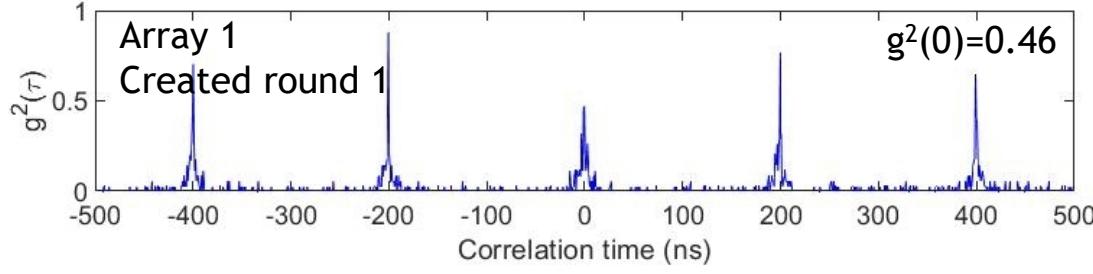
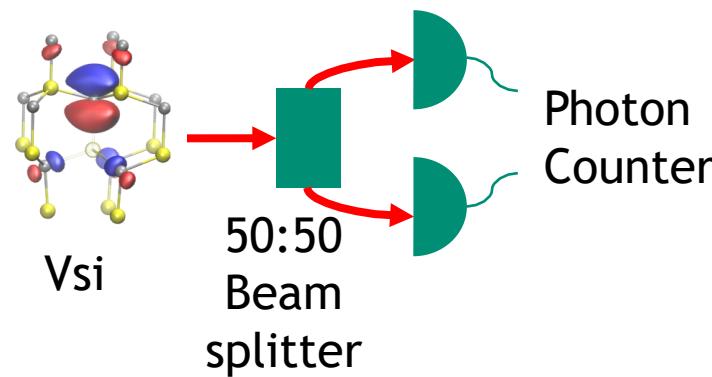
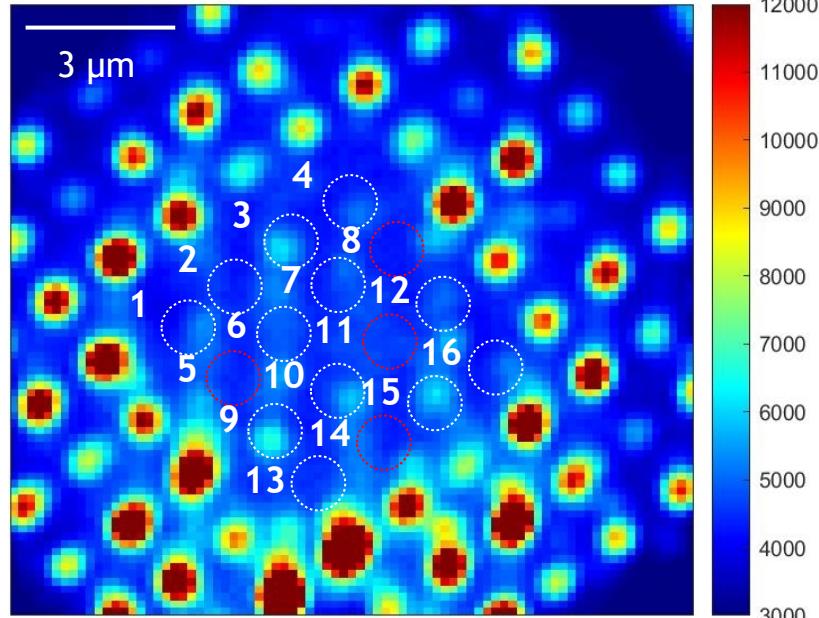
Chandrasekaran *et al*, Adv. Sci., 2300190 (2023)

Preliminary results suggest the in-situ PL works, BUT is limited by high background counts



# Ex-situ $g^2(\tau)$ check on <3> ions/spot arrays shows single defects

3 representative examples (okay, good examples 😊!)



\*No background correction

Chandrasekaran et al, Adv. Sci., 2300190 (2023)

9/16 (56%) of filled array points showed  $g^2(0) < 0.5$  without background correction → single defects

# Acknowledgements



Sandia has developed strong internal **ion beam implantation** and **optical** groups

M. Titze, W. Hardy, J. L. Pacheco, J. B. S. Abraham, G. Burns, A. Flores, G. Vizkelethy (SNL)

M. Zaibari, Jacob Henshaw, L. Basso, H. Byeon, A. Mounce, P. Kehayias, M. Lilly (SNL)

V. Chandrasekaran, Han Htoon (LANL)

V. Acosta (UNM)

And we have continued to support a wide range of user groups through CINT



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HARVARD  
UNIVERSITY



THE UNIVERSITY OF  
CHICAGO

Los Alamos  
NATIONAL LABORATORY  
EST. 1943

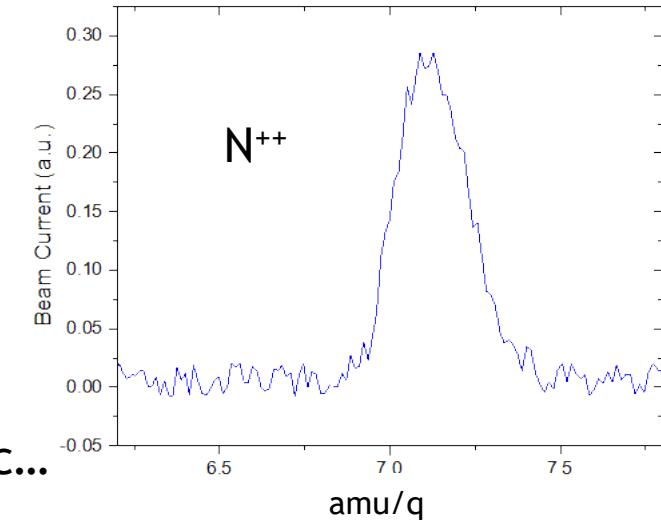
PURDUE  
UNIVERSITY

# Summary



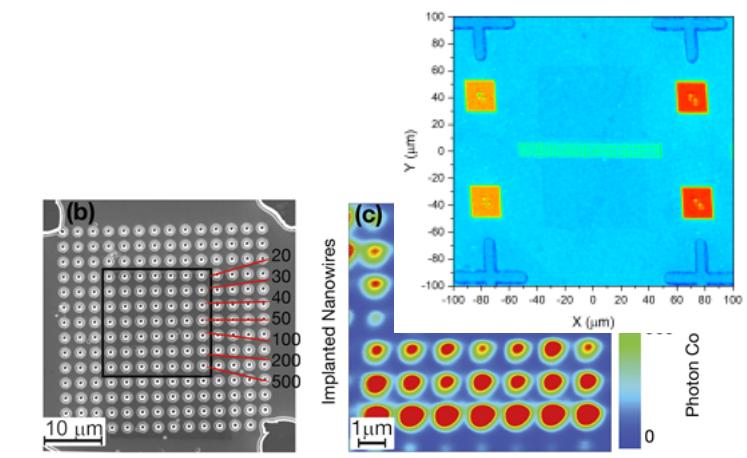
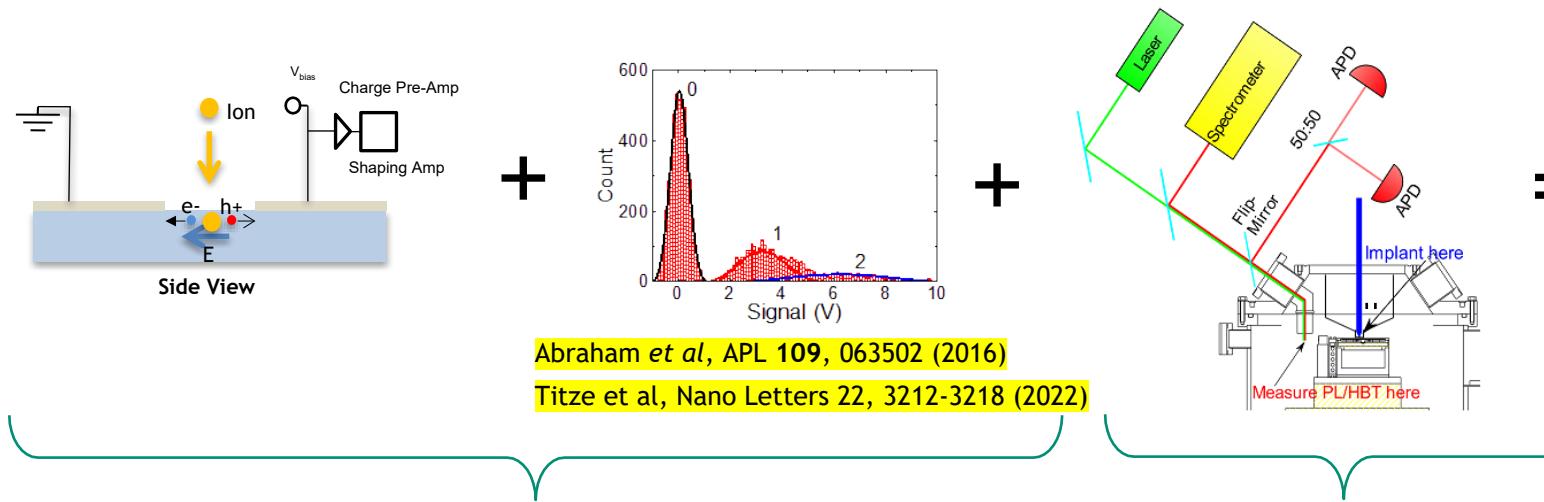
- We have demonstrated focused ion implantation for fabrication of single atom devices and nanofabrication

- Viable solution for prototyping - fast and easy!
- On-going work in diamond, SiN, SiC, hBN, GaN, AlN, GaOx, etc...
- LMAIS for 42/114 elements and development still on-going



Multiple sources run, with ~1.5 days of runtime

- Pathway Towards Deterministic Defect Centers in Wide Bandgap Materials using FIB Implantation



L. Marseglia, *et al.*, Optics Express (2018)

Control the number of ions

Confirm Optically Active Defect Centers

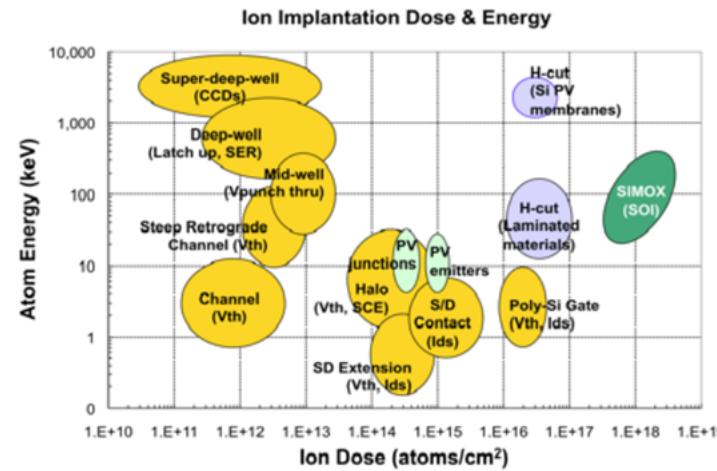
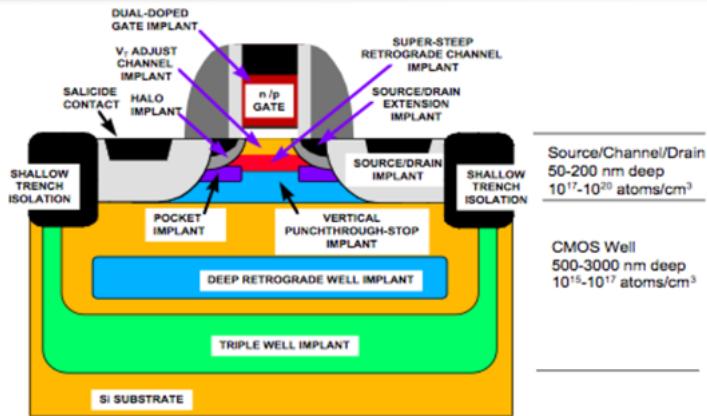
<https://cint.lanl.gov/>



# Ion Implantation and Irradiation for Device Fab

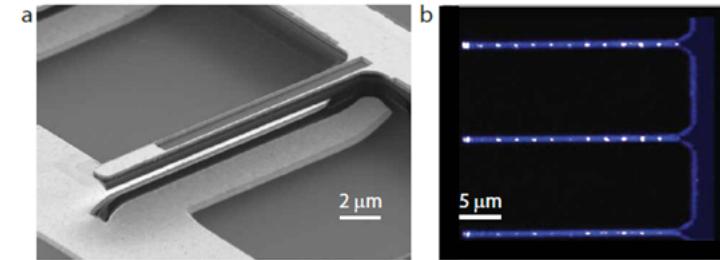


Ion Implantation has been a work-horse for the semiconductor industry

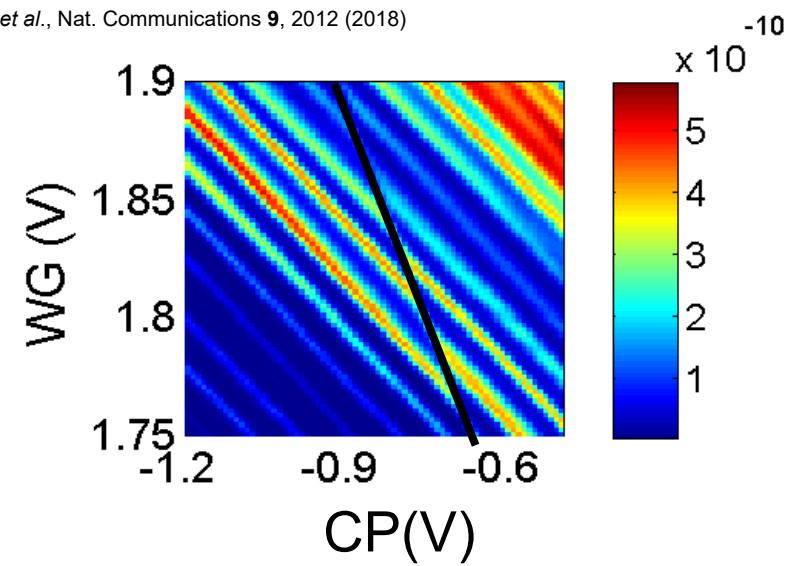


S. B. Felch, et al., Proceedings of PAC2013, Pasadena, CA (2013)

Our work is centered on localized implantation and fabrication based on a deterministic number of implanted ions



Y. Sohn, et al., Nat. Communications 9, 2012 (2018)

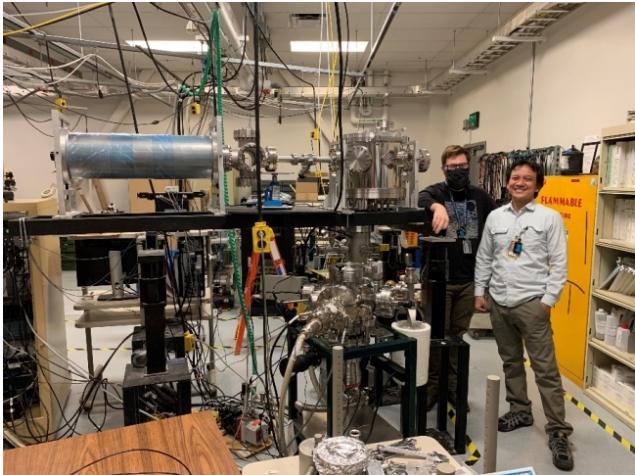


Singh et al. Appl. Phys. Lett. 108, 062101 (2016)

**Focused Ion Beam implantation for fabrication of single atom devices and nanofabrication**

# Capability Currently Under Development

## Pulsed Power Electron Gun

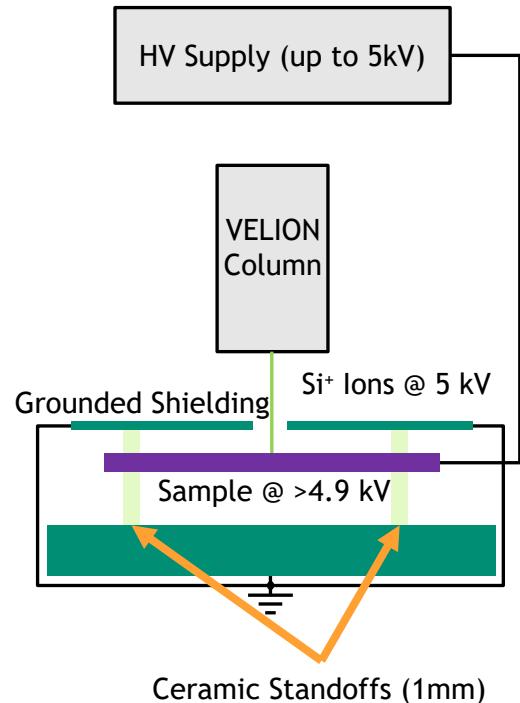


## Specifications

- 300 keV
- $>>10$  A of beam current
- $<25$  ns pulse lengths

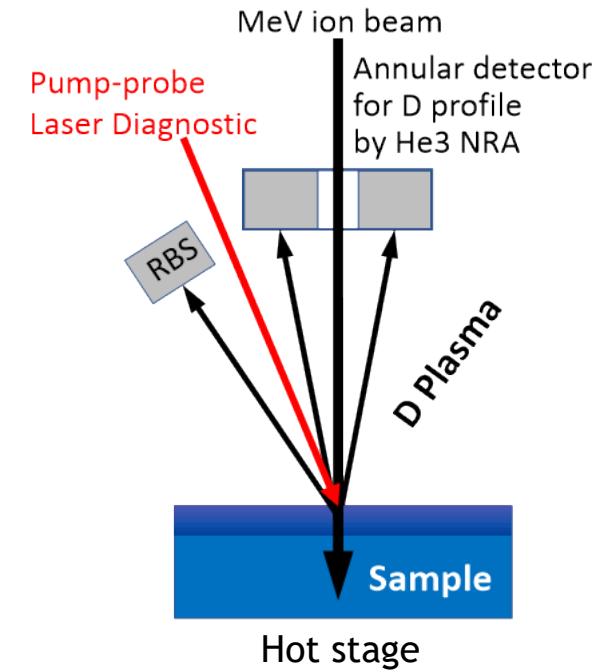
*Enables improved yield for wide bandgap defect centers*

## Low Energy Implantation Stage



*Enables lower energy implantation, albeit with a loss of spatial resolution*

## Combined Ion Irradiation + Ion Beam Analysis + Plasma Exposure



*Enables exploration of real-time evolution in sample properties including erosion and redeposition*

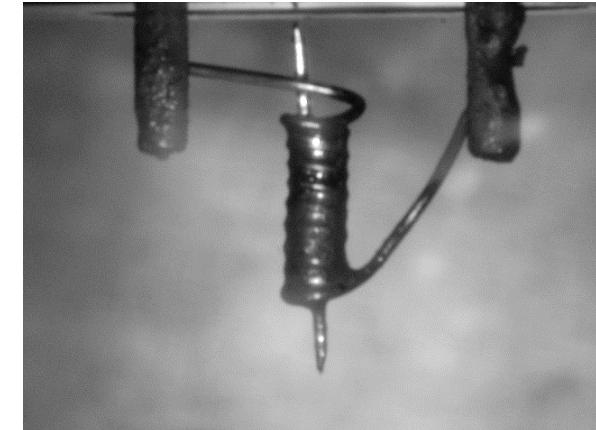
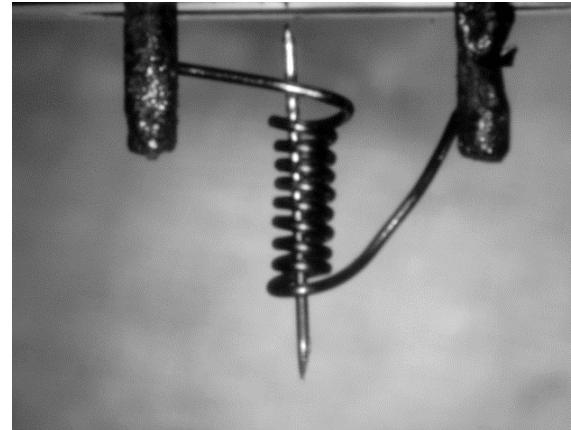


# Pb Source based on SnPb alloy

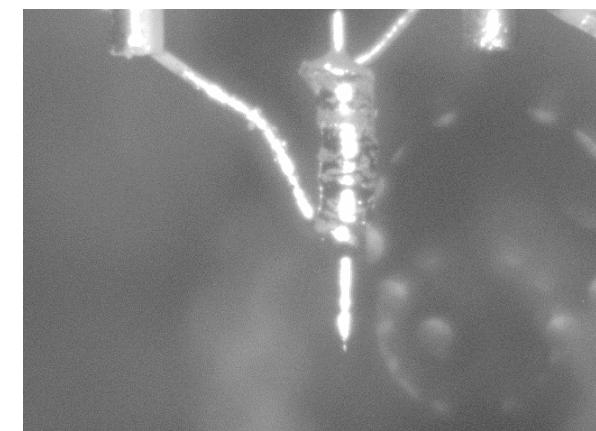
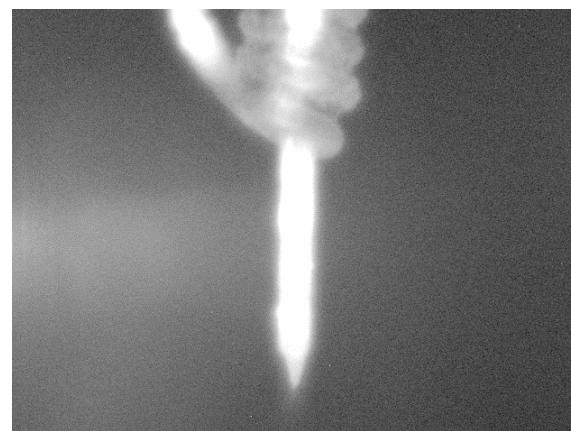
- Solder (SnPb) does not wet W, but Solder does wet a soldering iron
- We attempted multiple homemade sources made from Fe, Monel, Kanthal, Kovar. Of these Kovar (Fe-Ni-Co) gave the best performance
- We tested both mechanically cut and electrochemically etched tips welded to the reservoir to improve alloy flow and heating

As tested in the Raith Velion these tips achieve lifetimes of  $<2000 \mu\text{A}^*\text{hr}$  and spot sizes of  $<40 \text{ nm}$

Mechanically cut tips



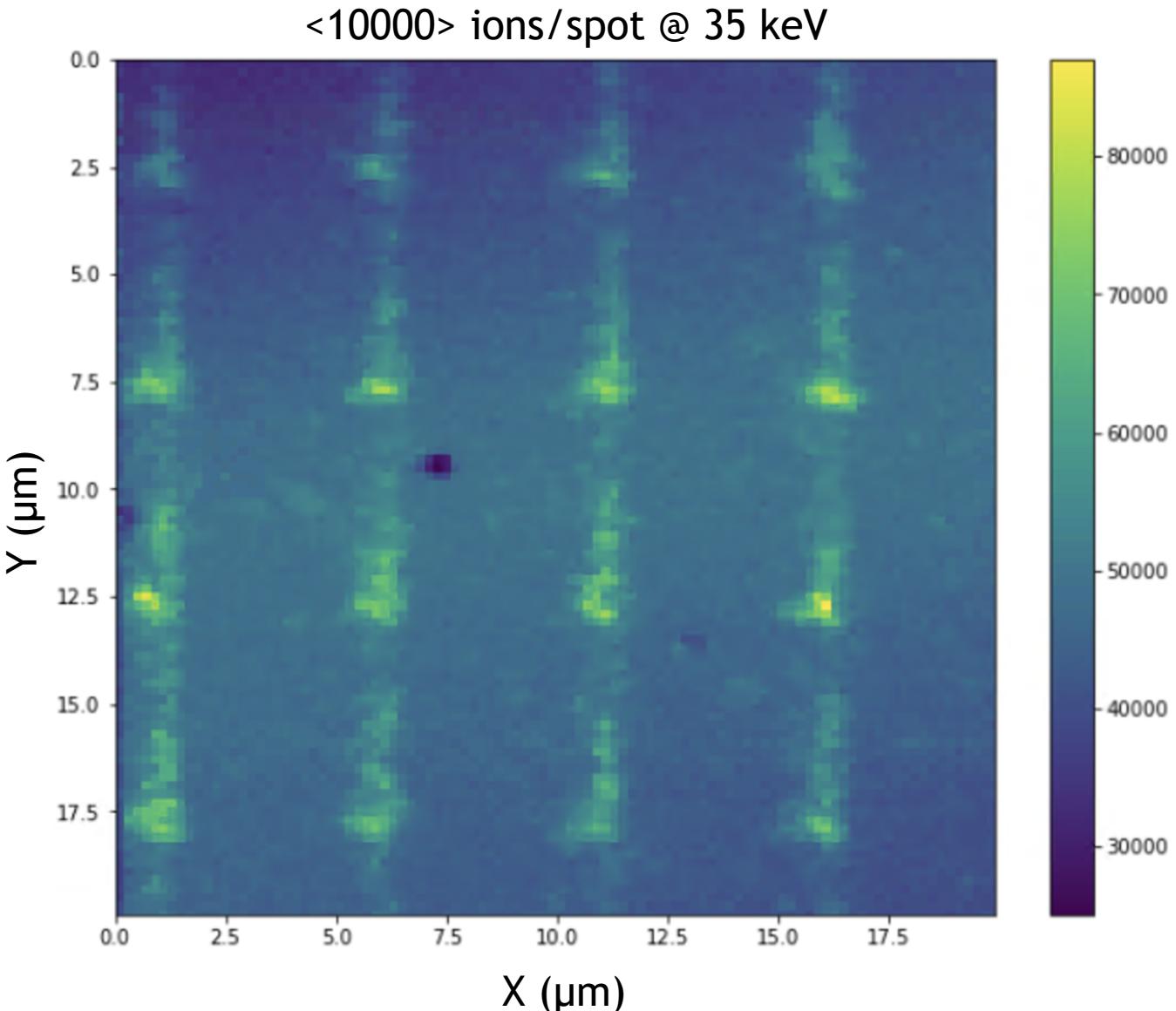
Electrochemically etched tips using Iron Chloride





# Preliminary Example of PbV in Diamond Substrate

- Observed PbV spectral signature in diamond substrates using FIB implantation
- Vertical stripes in the datasets?
  - Different Pb isotopes?
    - $^{208}\text{Pb}$  ( $\approx 52\%$ )
    - $^{206}\text{Pb}$  ( $\approx 24\%$ )
    - $^{207}\text{Pb}$  ( $\approx 23\%$ )
    - $^{204}\text{Pb}$  ( $\approx 1\%$ )
  - In the Raith Velion our  $m/\Delta m$  is not large enough to isolate the individual isotopes





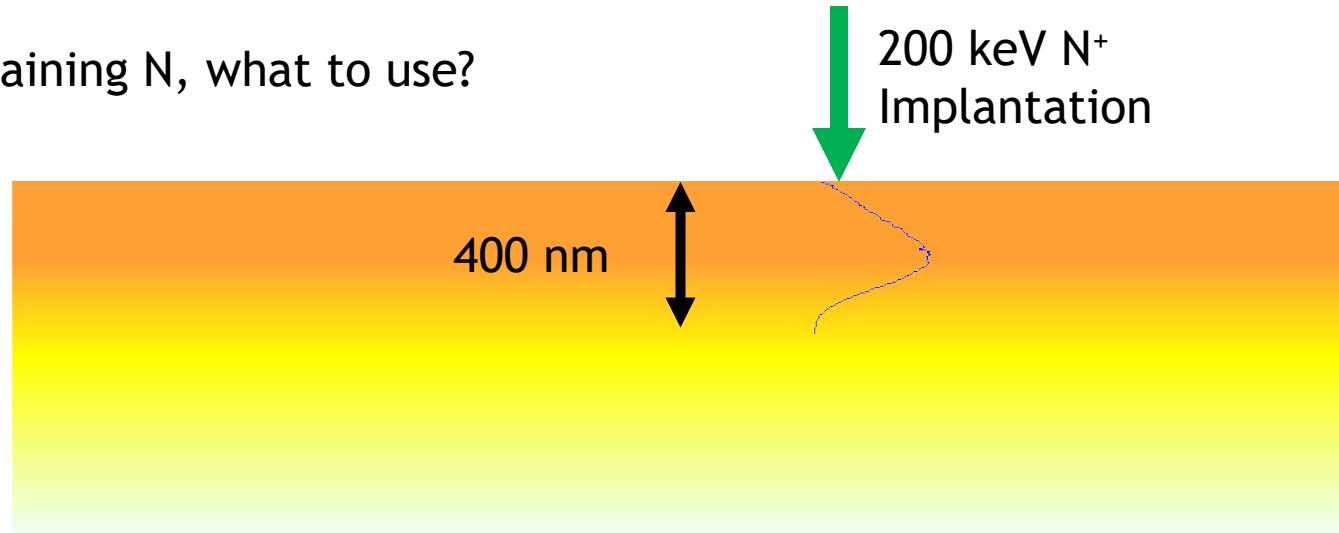
# Fabrication of a Nitrogen Liquid Metal Alloy Ion Source

Need a low melting point (200-500C) alloy containing N, what to use?

## Implantation

Implanted a 200  $\mu\text{m}$  thick  $\text{Au}_{80}\text{Sn}_{20}$  foil with Nitrogen to  $5 \times 10^{17}$  ions/cm<sup>2</sup>

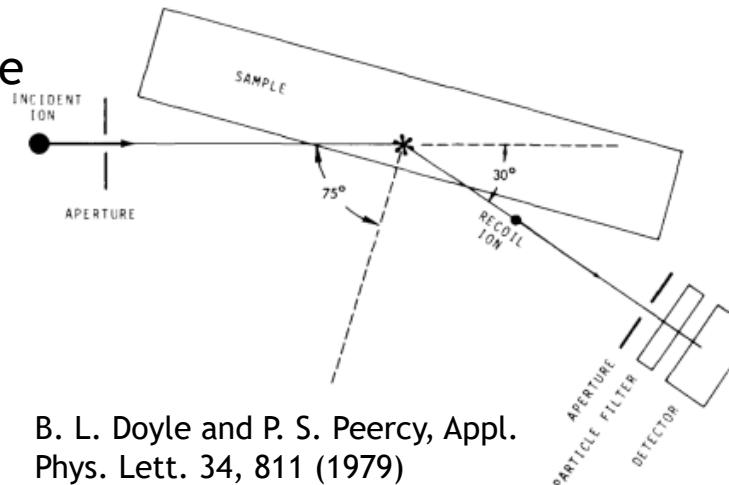
→ Implantation level chosen based on solid-solubility limit for H



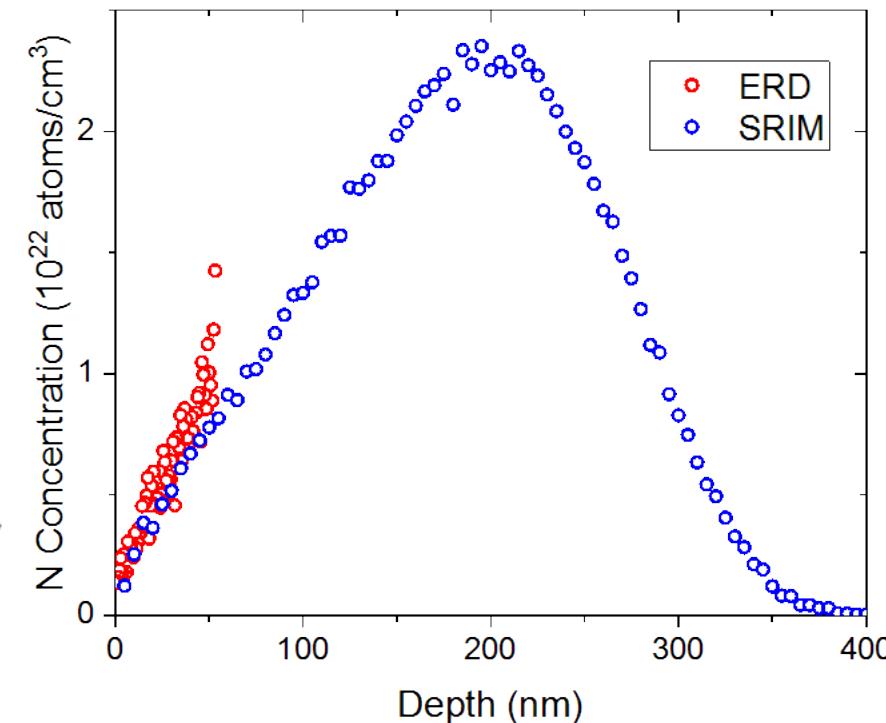
## Characterization

Elastic Recoil Detection (ERD) is used to probe the first 100 nm of the implanted foil

→ Limited interrogate depth due to multiple scattering of ions



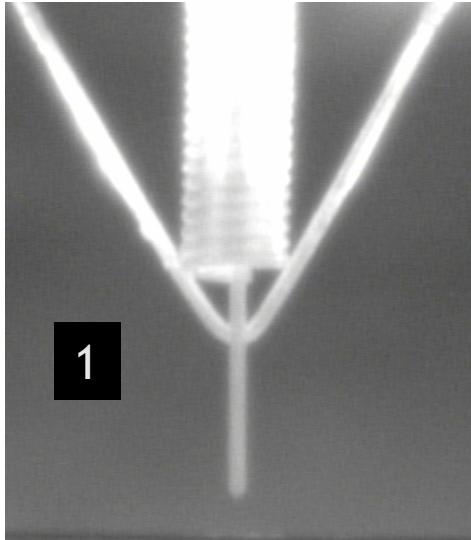
We can produce the material, but can we make a usable LMAIS?



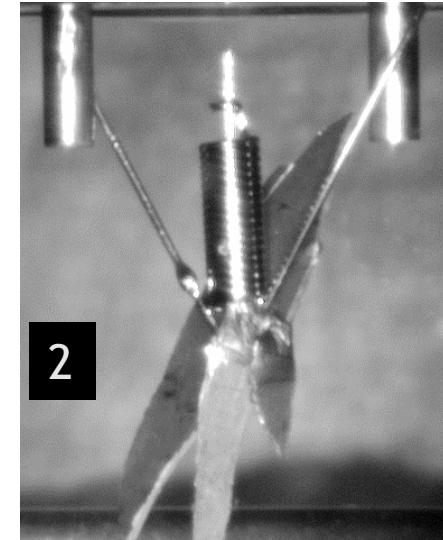


# Tip fabrication from implanted AuSnN foil

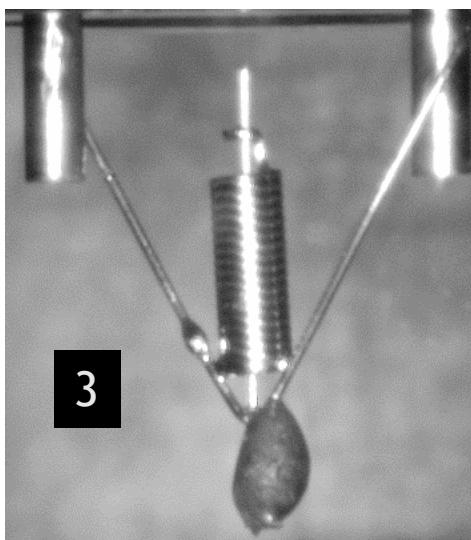
(1) Heat W tip to remove surface contaminants



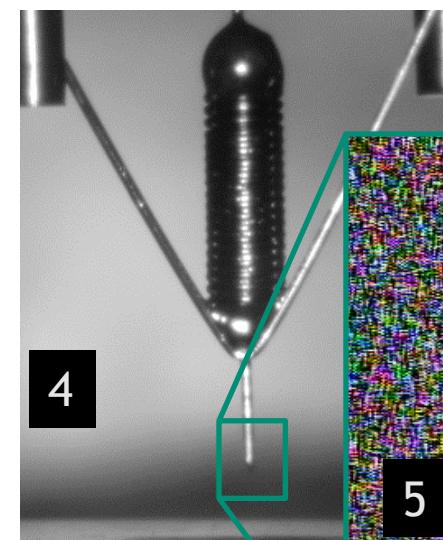
(2) Tack weld on implanted foil to the tip



(3) Heat tip to melt the foil onto the tip

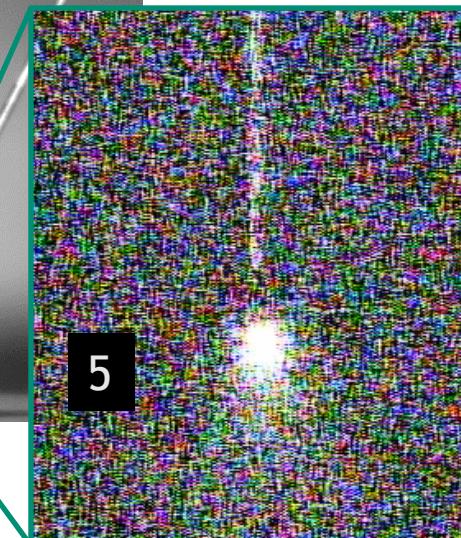


(4) Repeat steps (2) and (3) until tip and the reservoir is filled



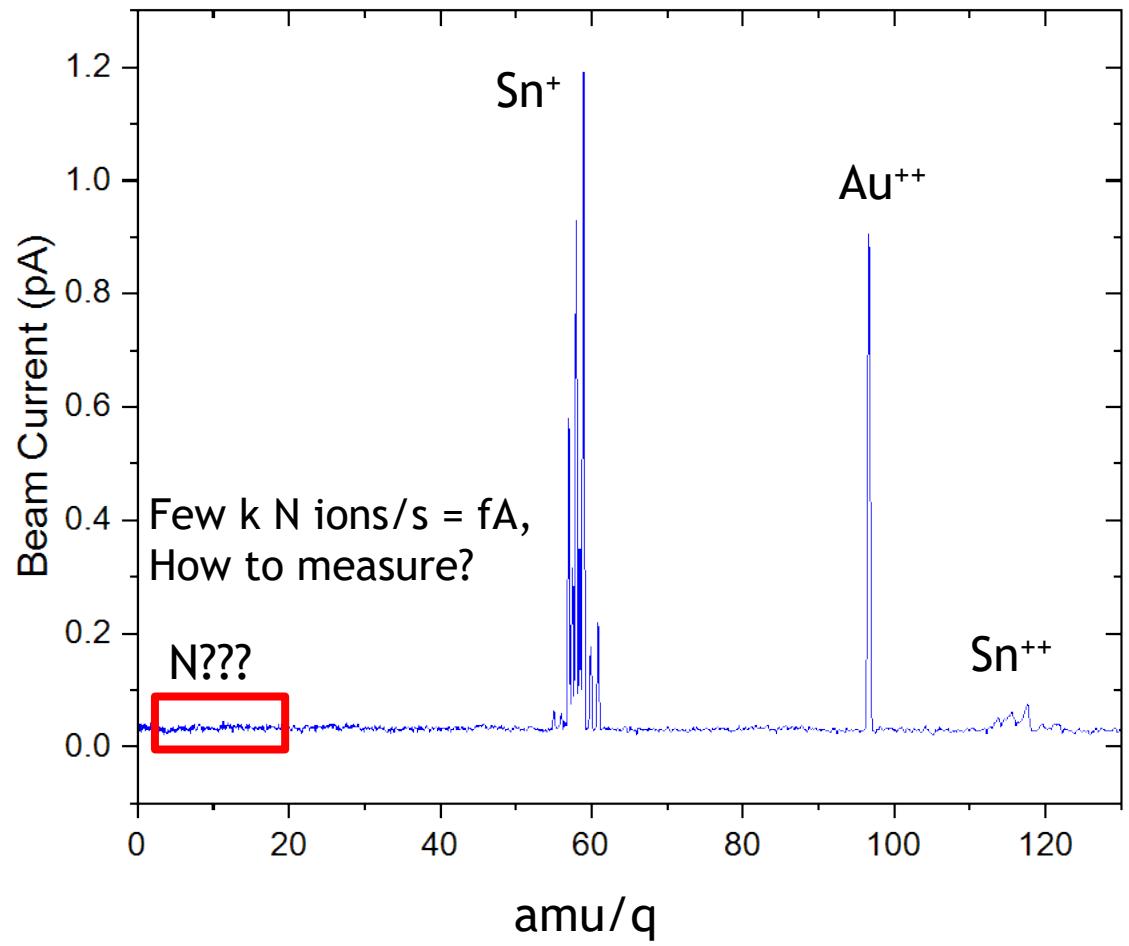
(5) Test tip for emission

(6) Install tip into nanolimplanter

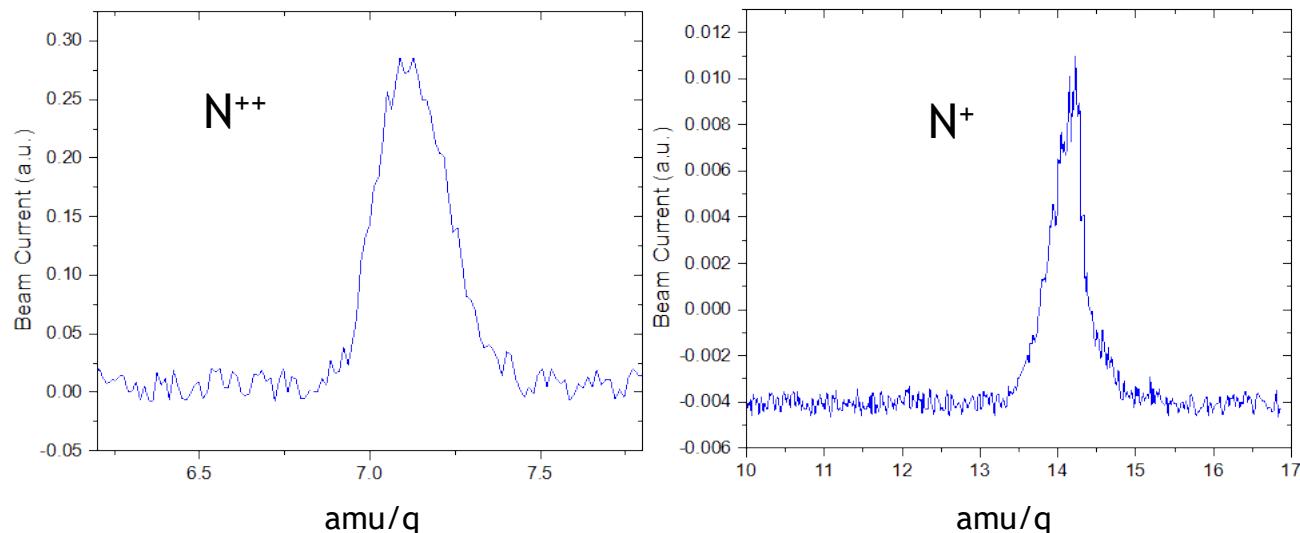
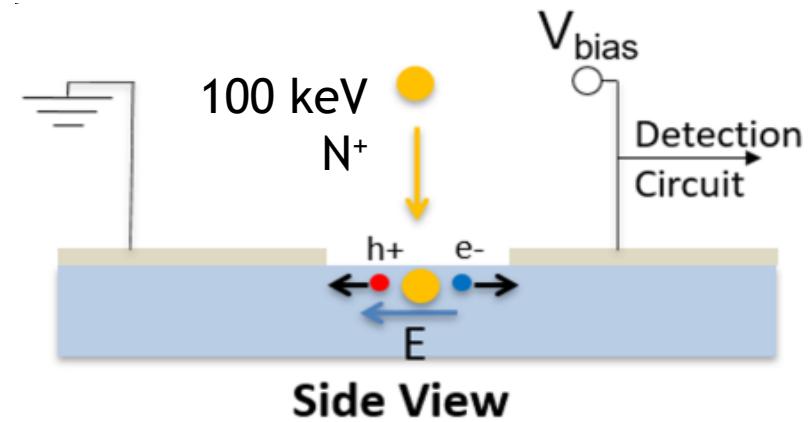


Very challenging source preparation! How well does the N LMAIS run?

# N Source Characterization - Mass Spectrum via IBIC

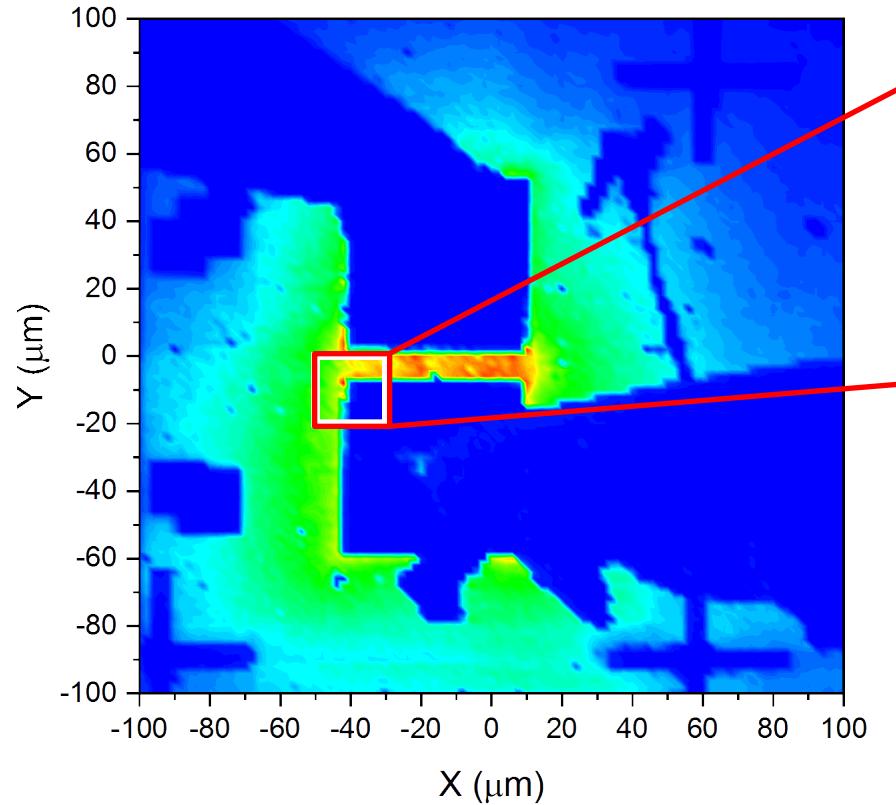


Ion Beam Induced Charge (IBIC) to measure N beam

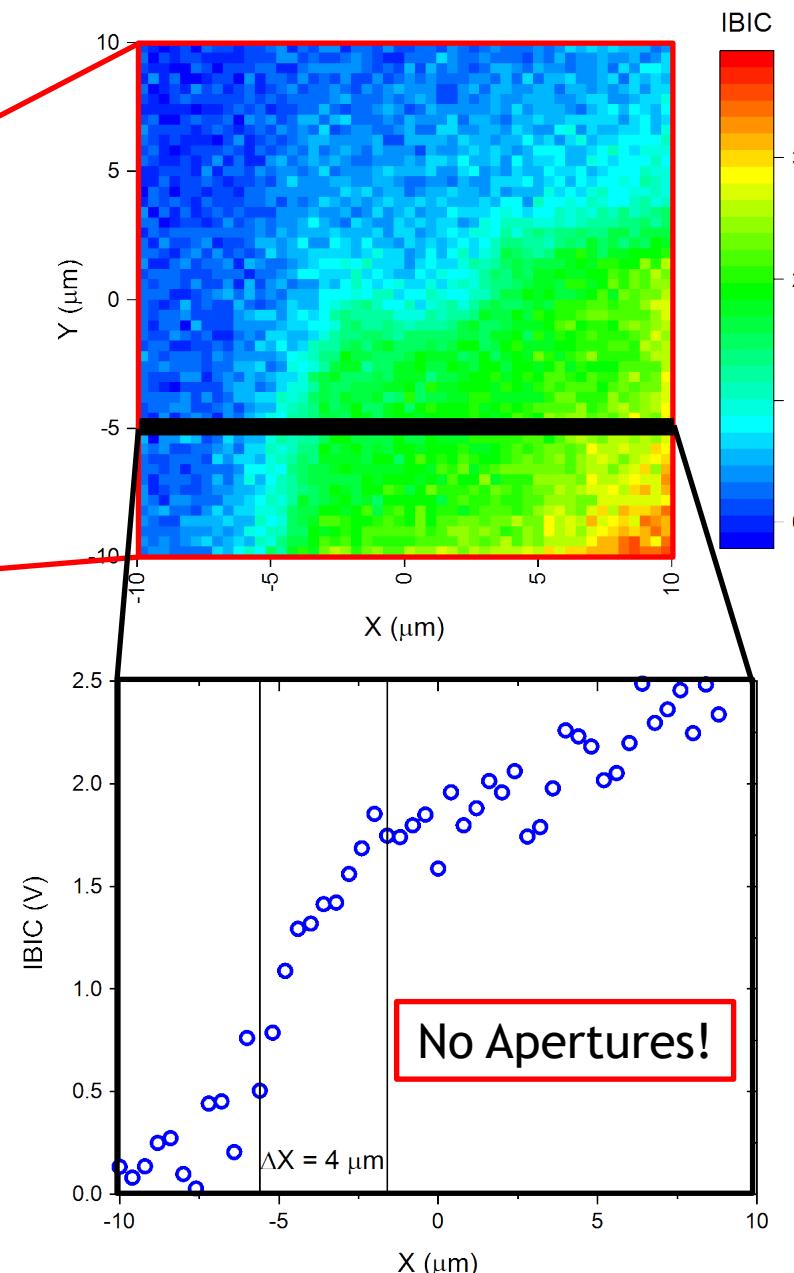


Observe Atomic nitrogen single and doubly charged ions in the source at ~20k ions/s

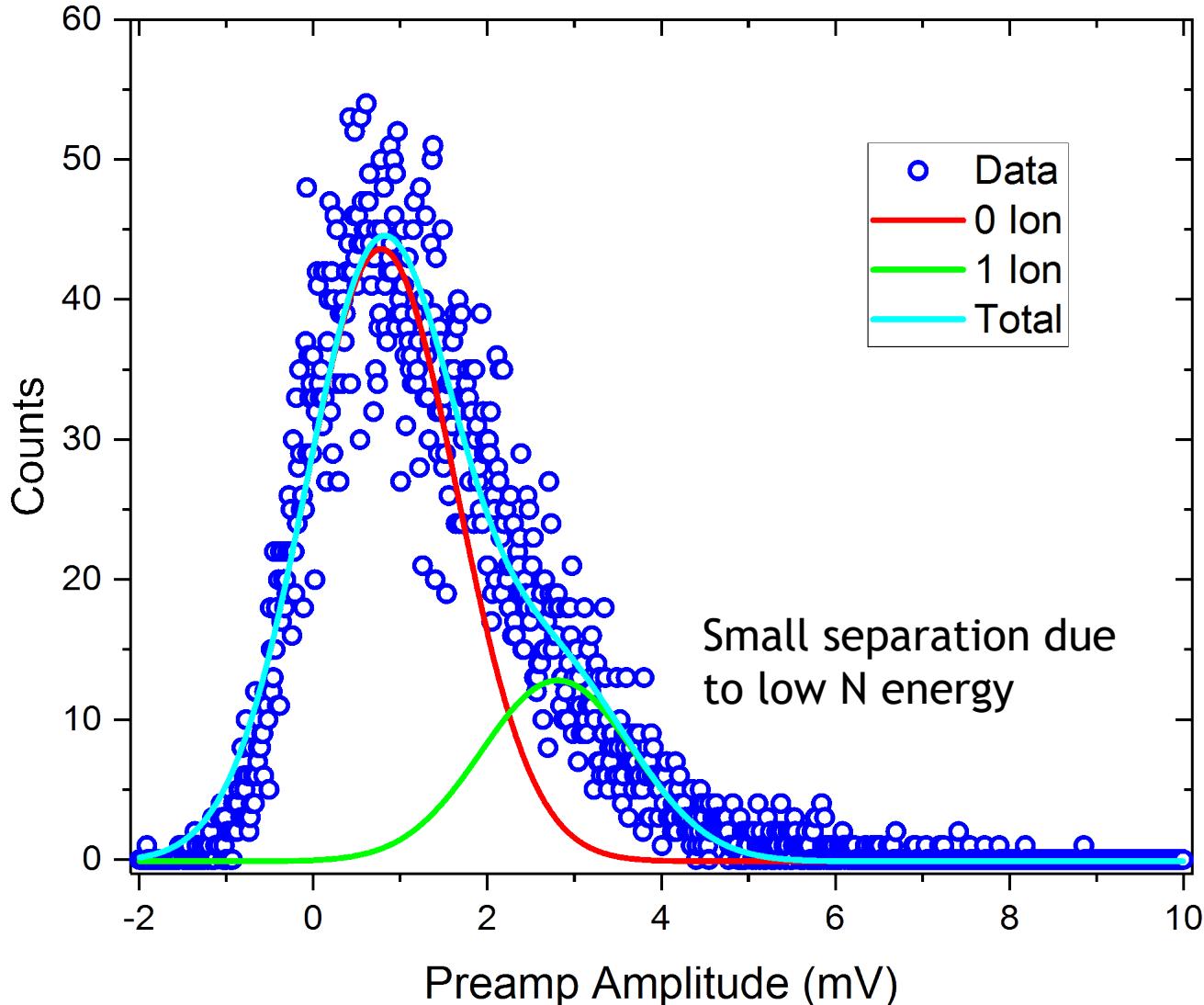
# N Source Characterization - Spot Size via IBIC



As tested in the A&D nanolimplanter these tips achieve lifetimes of  $\sim 100\text{-}200 \mu\text{A}^*\text{hr}$  and spot sizes of  $< 4 \mu\text{m}$



# Characterization - Beam Current measured via IBIC



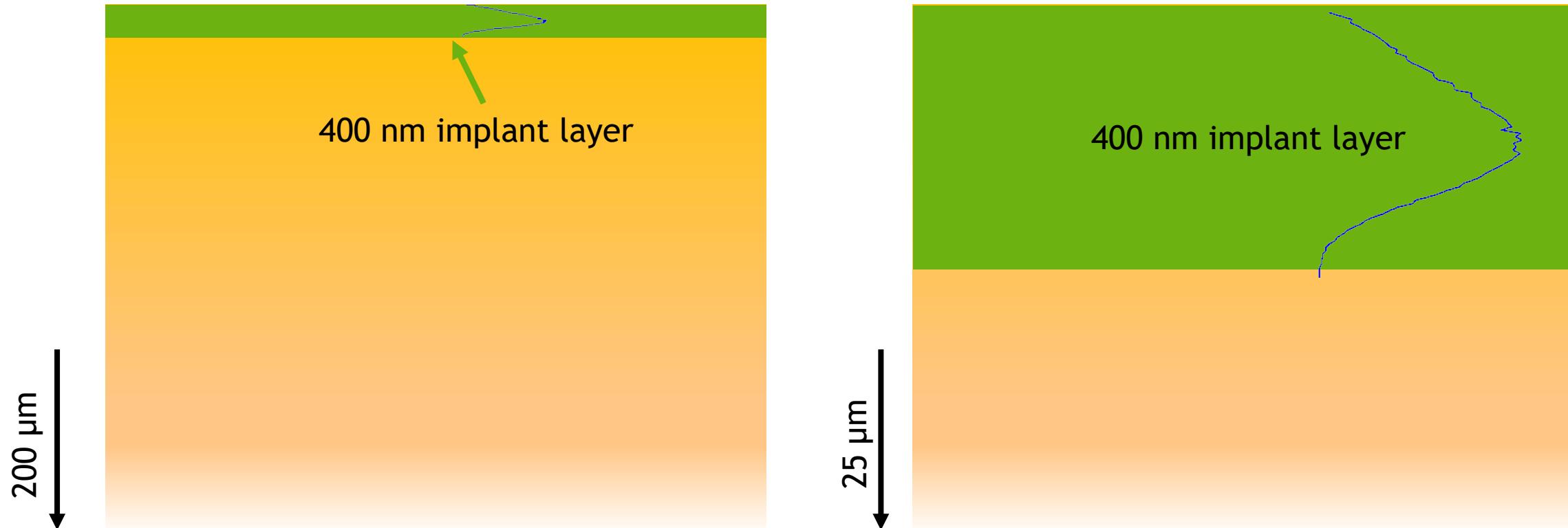
1. Fit peak to Gaussians
2. Area under peak corresponds to likelihood of event

$\langle 1 \rangle$  ion every 50  $\mu$ s  $\equiv$  20 k ions/s

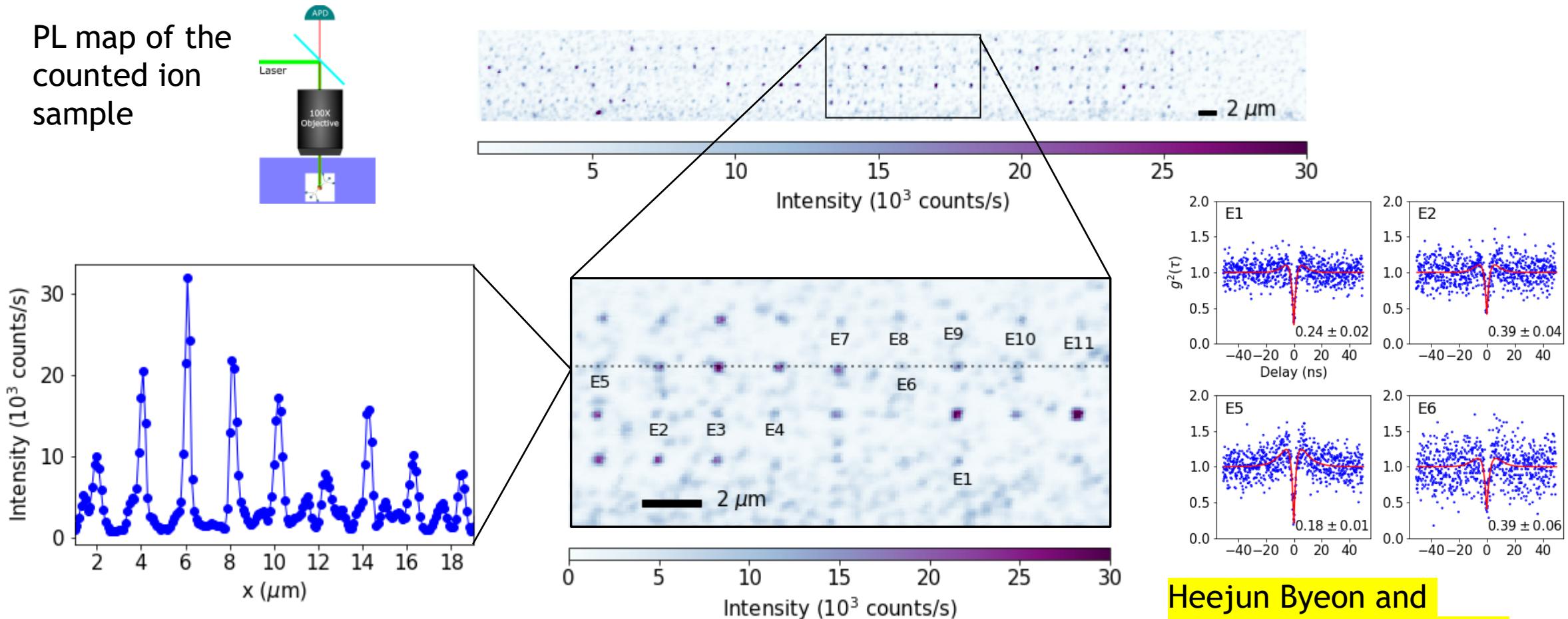
# N Source – Improvements

Higher beam current

- Thinner foil to same fluence → higher overall concentration
- Fabrication in N atmosphere → additional N absorption



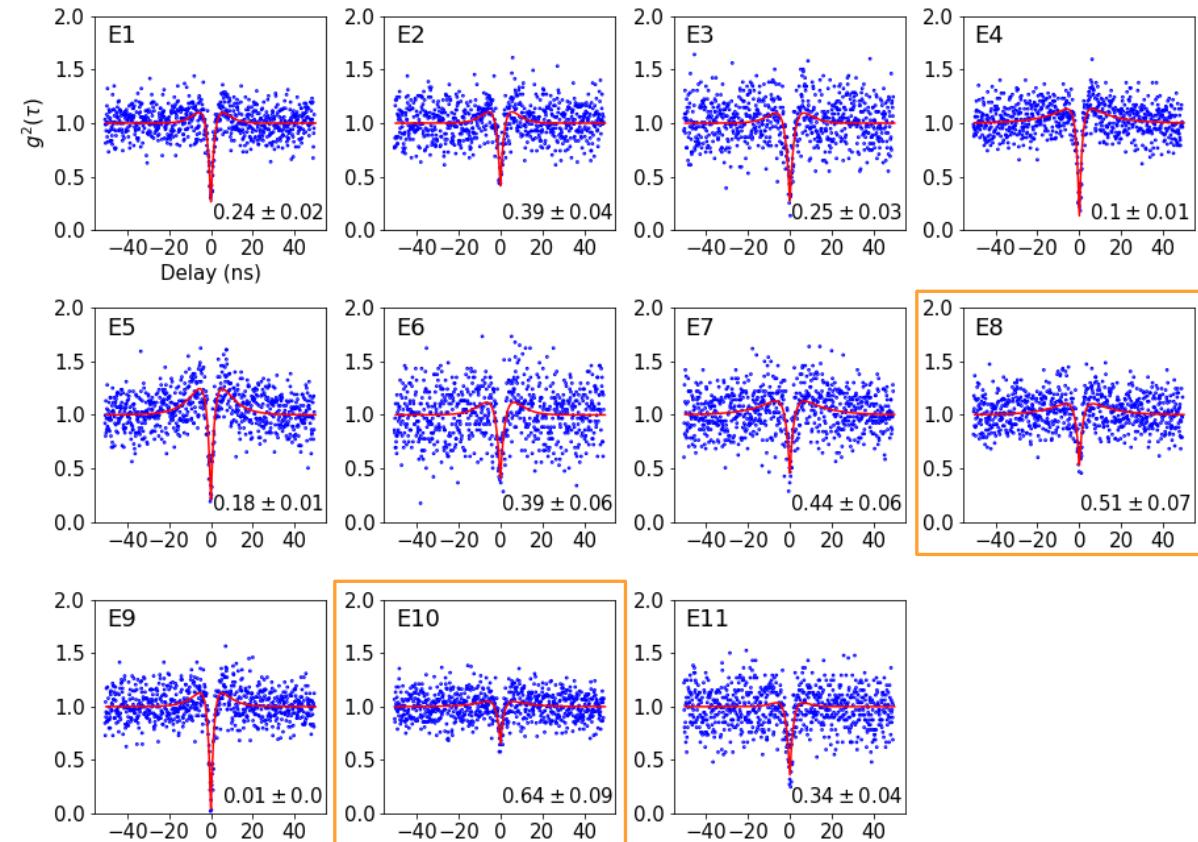
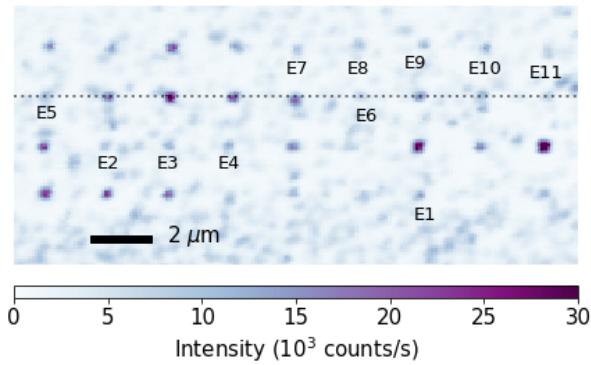
# Photoluminescence of the counted sample shows single defects



Heejun Byeon and  
Andrew Mounce at CINT

High confidence in the number of implanted Si ions → confirmed by  $g^2(\tau)$

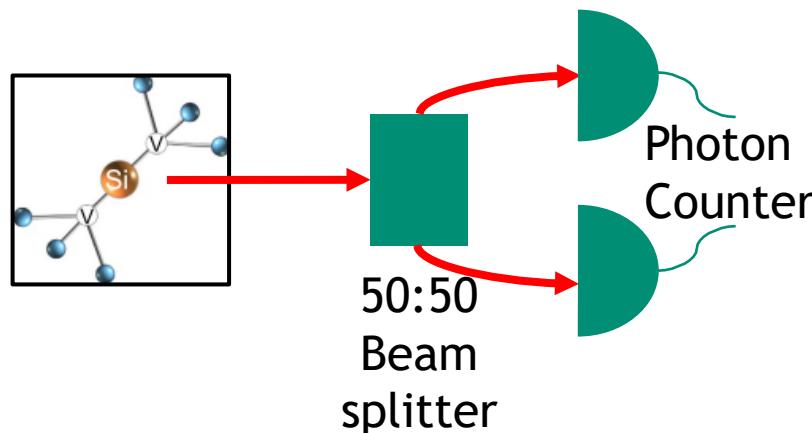
# Hanbury-Brown-Twiss Interferometry



Single emitter will emit only 1 photon at a time

2<sup>nd</sup> order autocorrelation

Only 1 photon at each counter at any time



Not all locations identified as single emitters from PL are single emitters