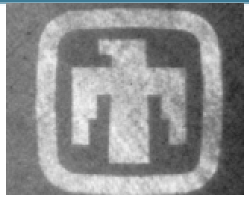
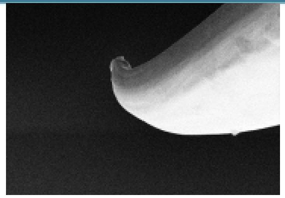
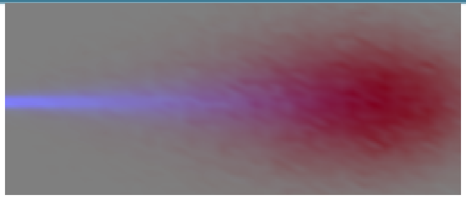
SAND2021-11706C

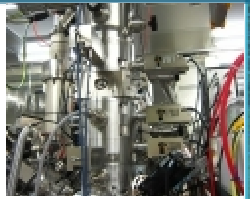
Sandia National Laboratories

Focused Ion Beams for Deterministic Nanometer-Scale Quantum Device Fabrication









Michael Titze, Heejun Byeon, Vigneshwaran Chandrasekaran, Anthony Flores, Han Htoon, Andrew Mounce, Ed Bielejec

08/17/2021

U.S. DEPARTMENT OF ENERGY NNSA

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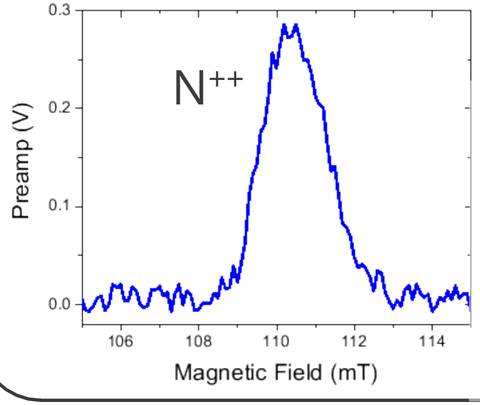
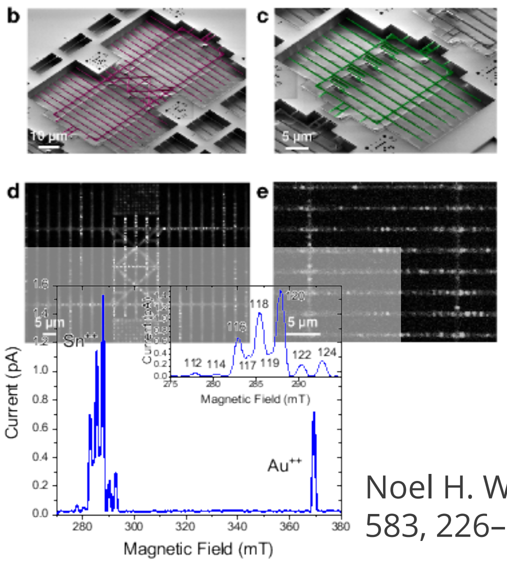
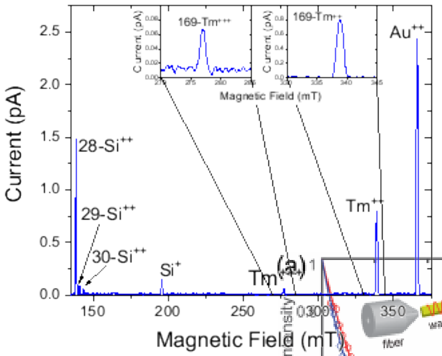
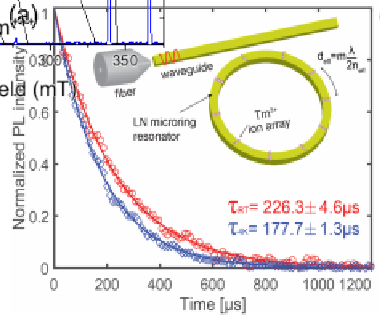
Outline

LMAIS Source Development



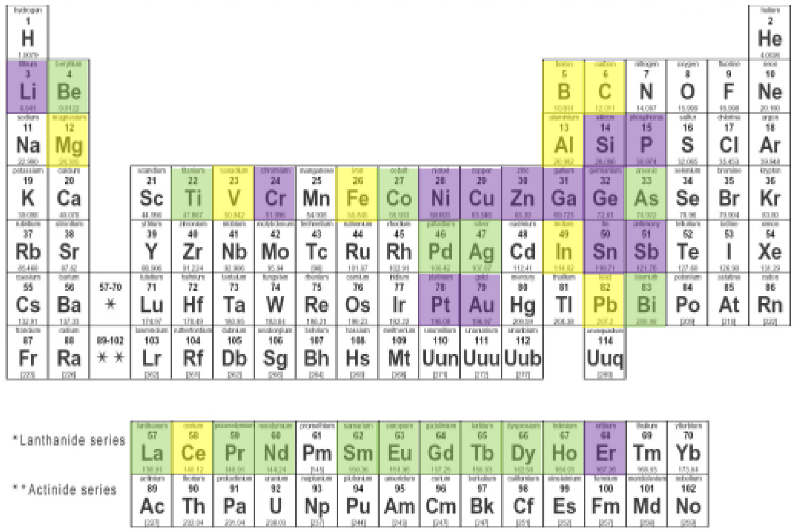


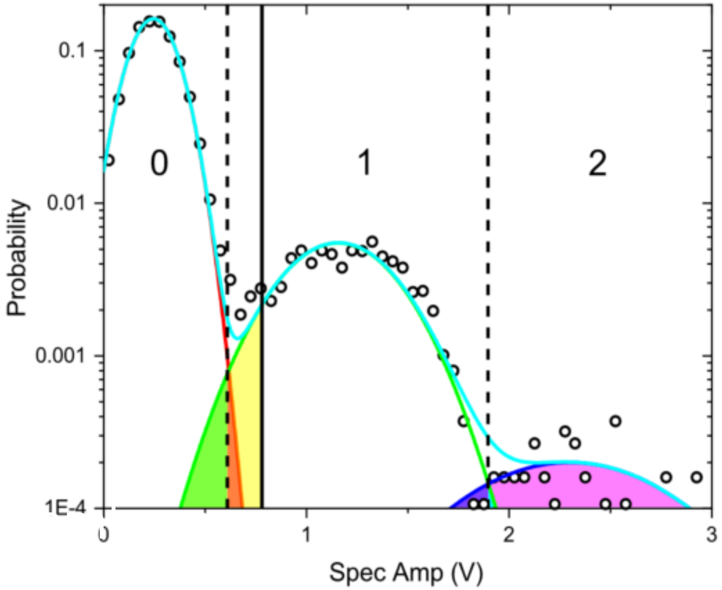




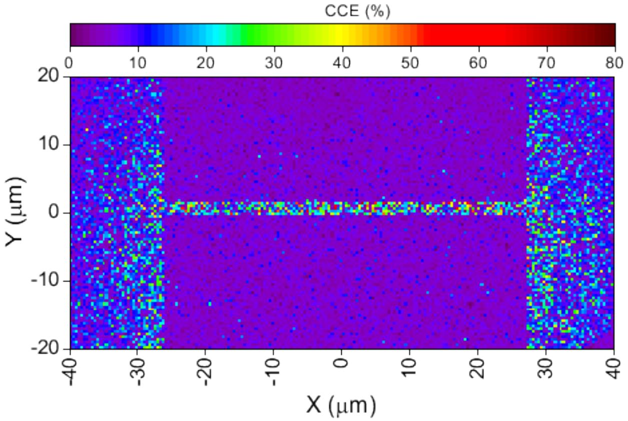
Noel H. Wan et al., Nature, 583, 226–231(2020)

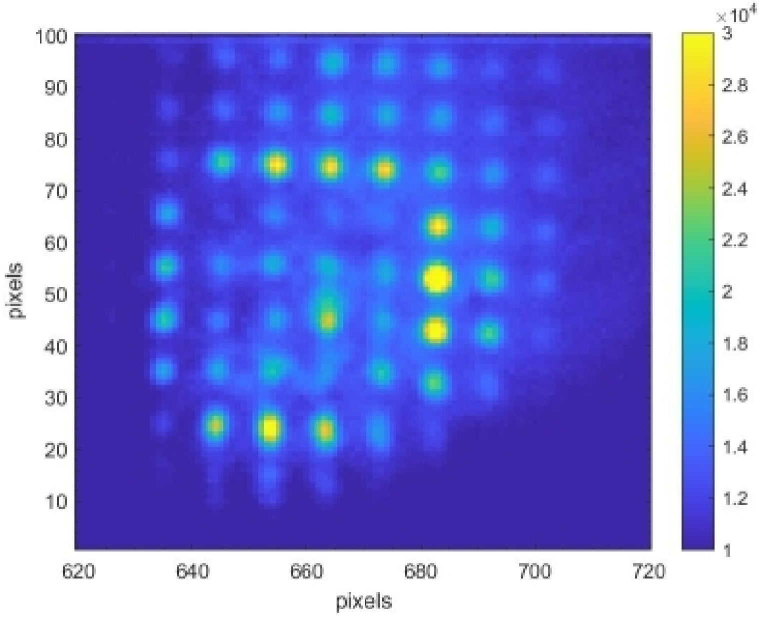
D. Pak et al., submitted





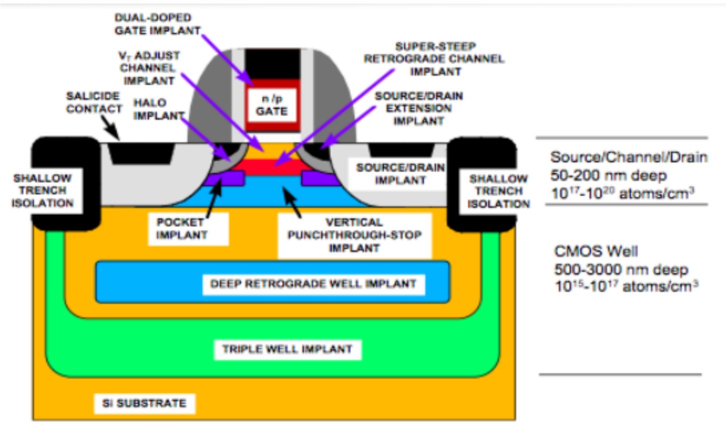
In-Situ Counting + Photoluminescence

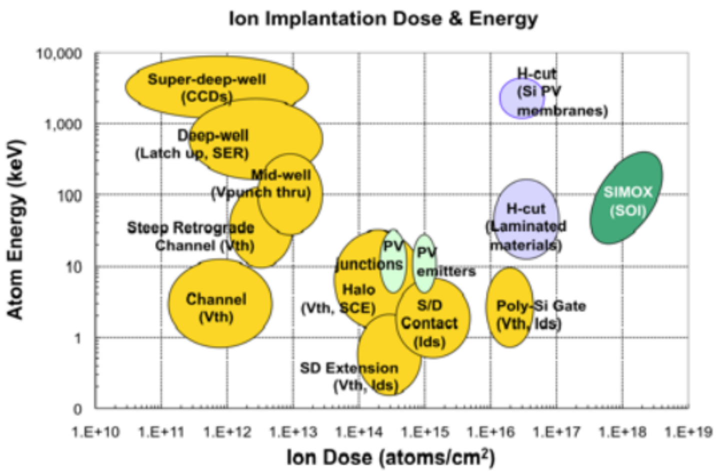




Ion Implantation and Irradiation for Device Fab

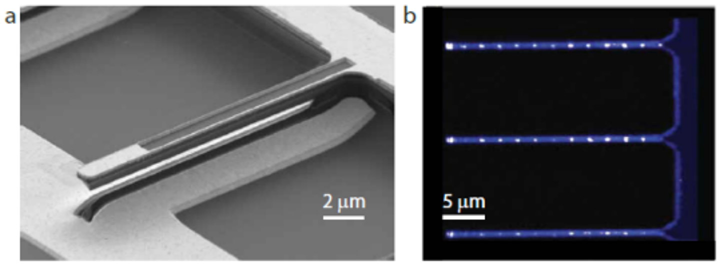
Ion Implantation has been a work-horse for the semiconductor industry



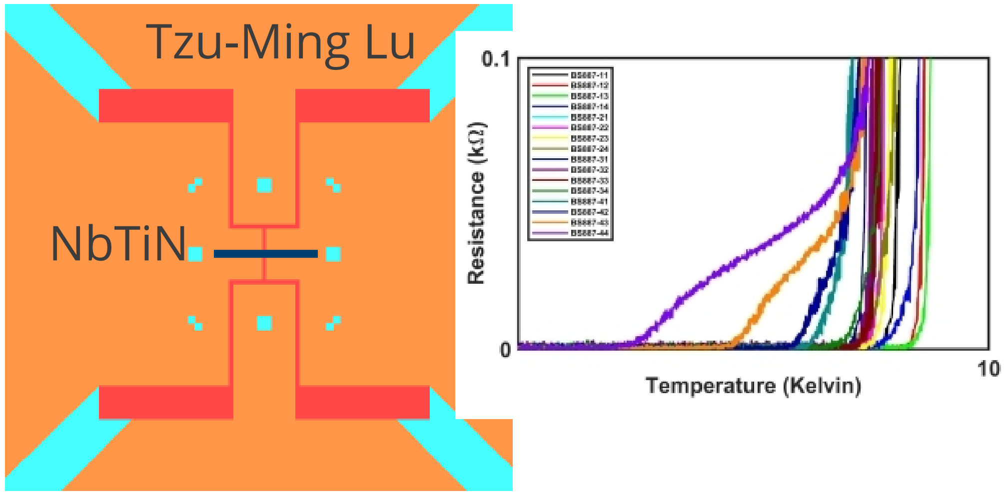


S. B. Felch, *et al*., Proceedings of PAC2013, Pasadena, CA (2013)

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

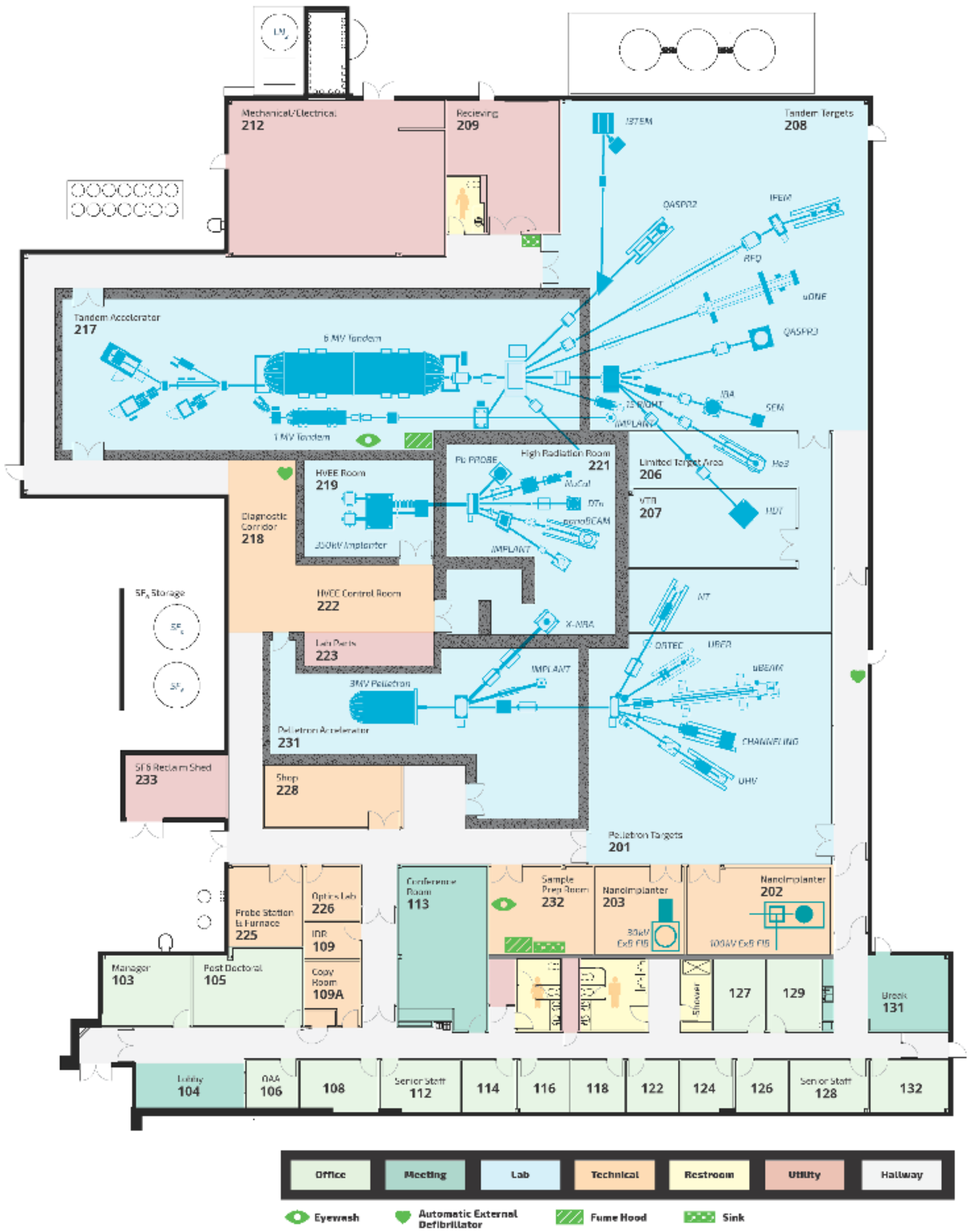


Y. Sohn, *et al*., Nat. Communications **9**, 2012 (2018)



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

building **720**
ION BEAM LABORATORY



Operational

(1) 6 MV Tandem Accelerator

Radiation effects testing

(2) 3 MV Pelletron Accelerator

Ion beam analysis and displacement damage

(3) 1 MV Tandem Accelerator

Support I3TEM and "replace" HVEE for ions

(4) 350 kV HVEE Implanter

>90% usage for mono-energetic neutrons

(5) 100 kV ExB FIB nanoImplanter

High spatial resolution implantation

(6) 35 kV ExB FIB Raith Velion

Higher spatial resolution at lower energies

(7) 35 kV Zeiss HeIM

<0.5 nm beam spot, high resolution imaging

Installing

(8) 35 kV Plasma FIB

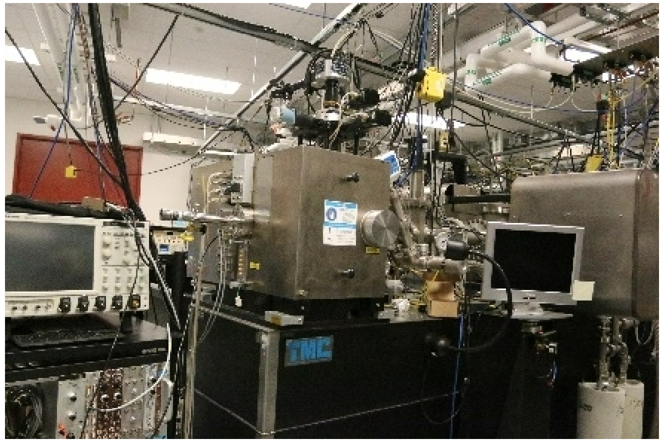
Gas sources – O, N, Ar, Xe, etc…

High energy focused micobeams
1 μm to mm's

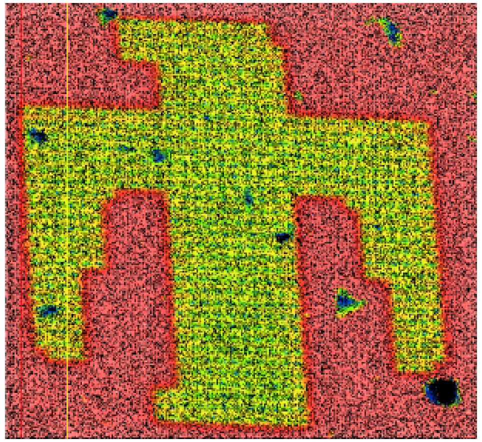
Low energy focused nanobeams
<1 to 20 nm

High Energy Microbeams ~1 μm to mm's

- 6 MV Tandem microbeam (microONE)

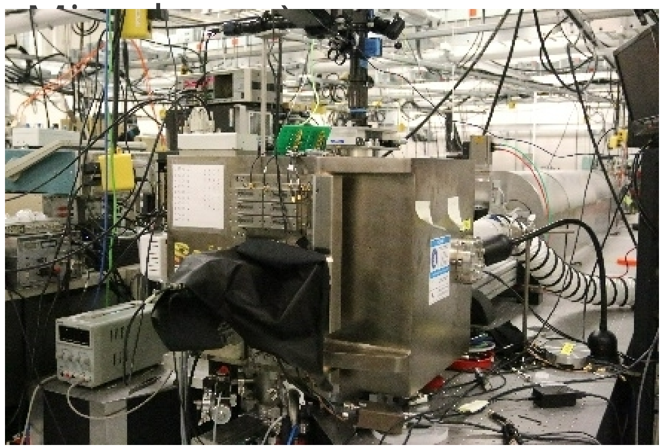


- High resolution laser stage
- Spot size <1 μm
- Energy 0.8 – 70 MeV
- H to Au



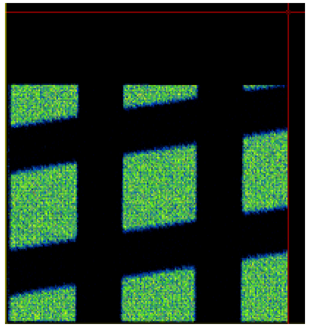
IBIC on PIN diode

- 3 MV Pelletron microbeam (Light Ion



- High resolution laser stage
- Spot size <600 nm
- Energy 0.25 – 3 MeV
- H, He, N, Ar, Xe, …

12.5 μm



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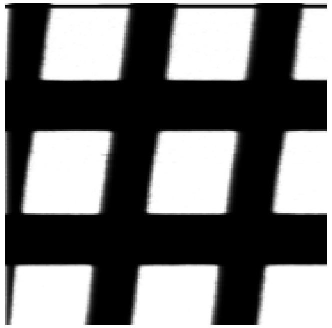
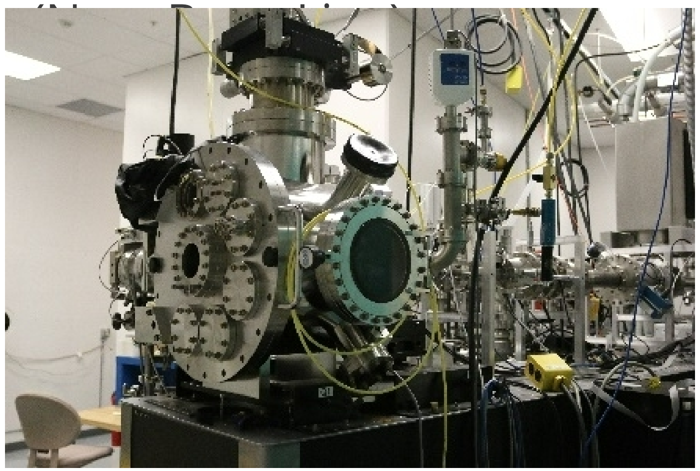
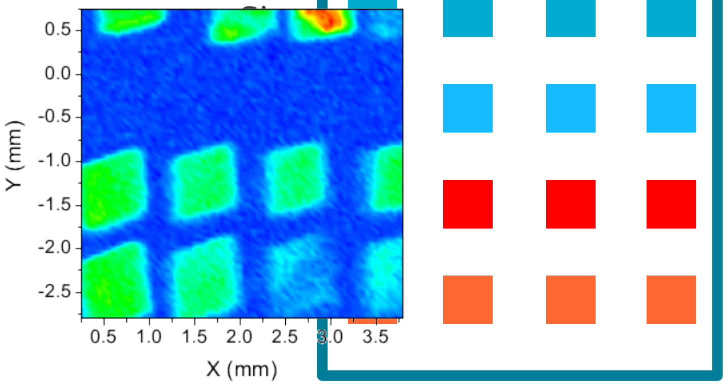


SED on PIN

- 350 kV HVEE microbeam

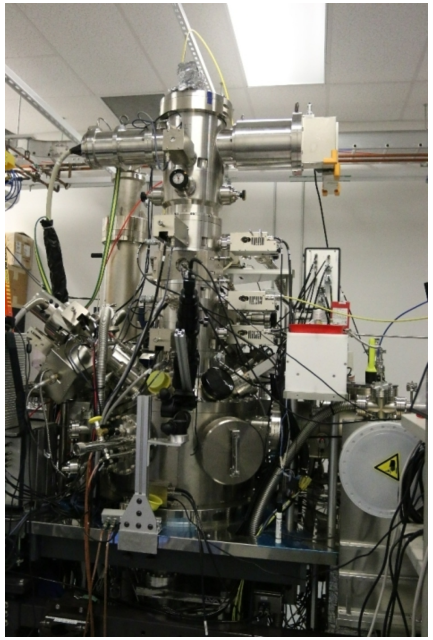


- Piezo stage
- Spot size <1 μm
- Energy 20 – 350 keV
- H to Au



Low Energy Nanobeams <1 to 20 nm

- 100 kV A&D FIB100NI (nanoImplanter)

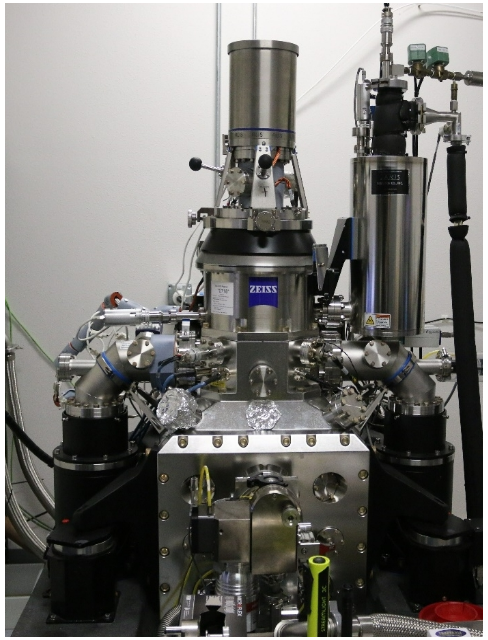


- High resolution laser stage
- Spot size <10 nm (Ga)
- Energy 10 – 200 keV
- 1/3 periodic table

- 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 (HeIM)



- Piezo stage
- Spot size <1 nm
- Energy 10 – 35 keV
- He, **N**, likely other gases

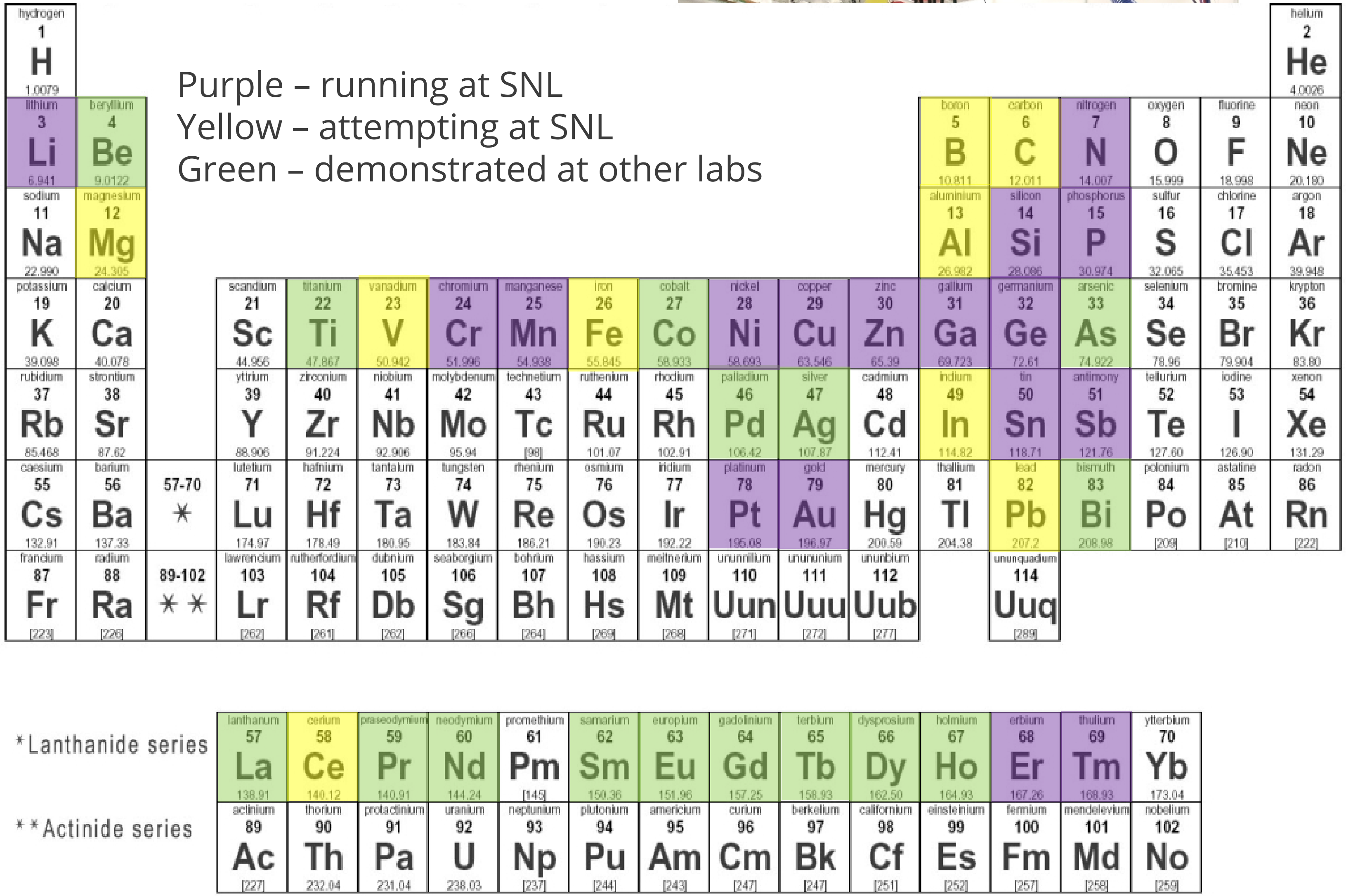
All equipped with Lithography Software for Patterning

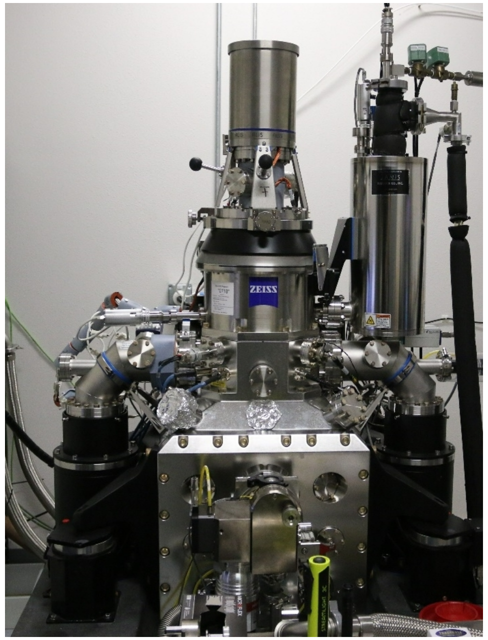
Low Energy Nanobeams <1 to 20 nm

- 100 kV A&D FIB100NI (nanoImplanter)

- 35 kV Raith Velion (Velion)

- 35 kV Zeiss Orion Plus (HeIM)





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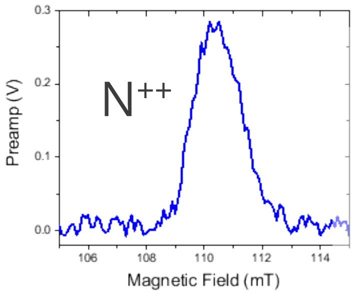
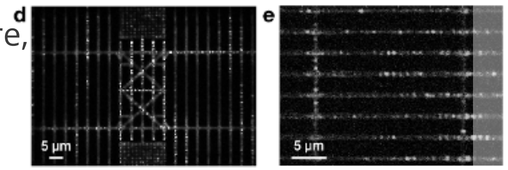
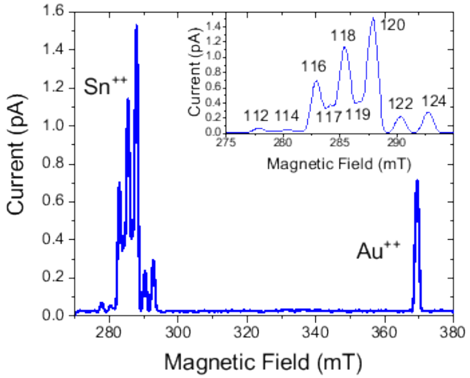
erning

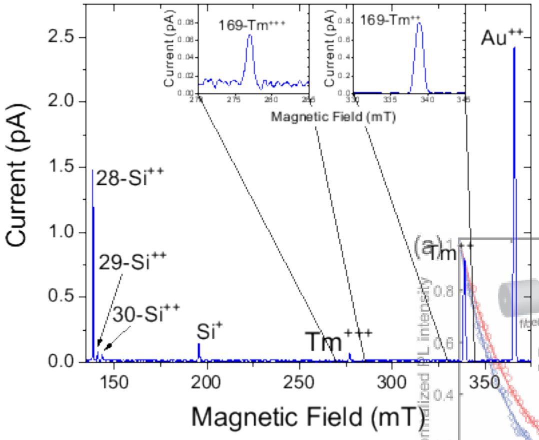
LMAIS Source Development

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

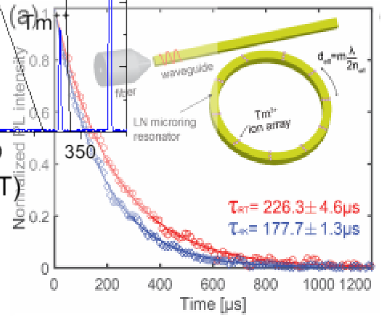


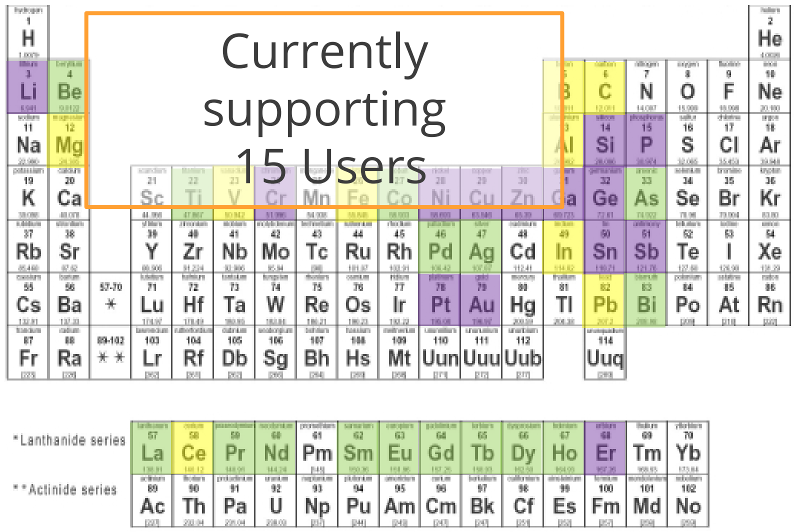




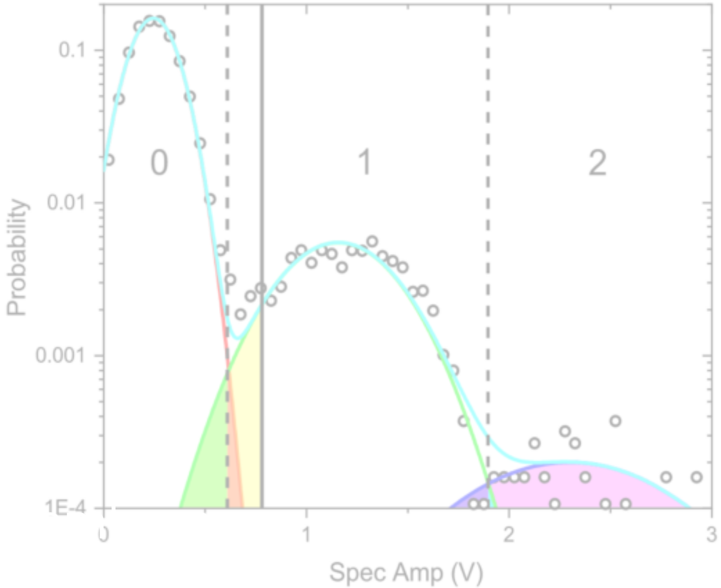


D. Pak et al., submitted





Currently supporting 15 Users



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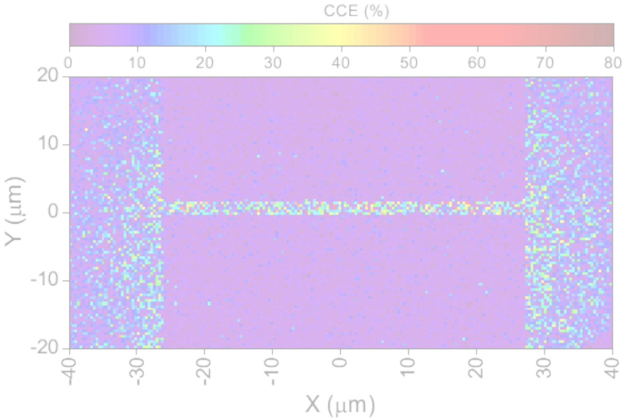
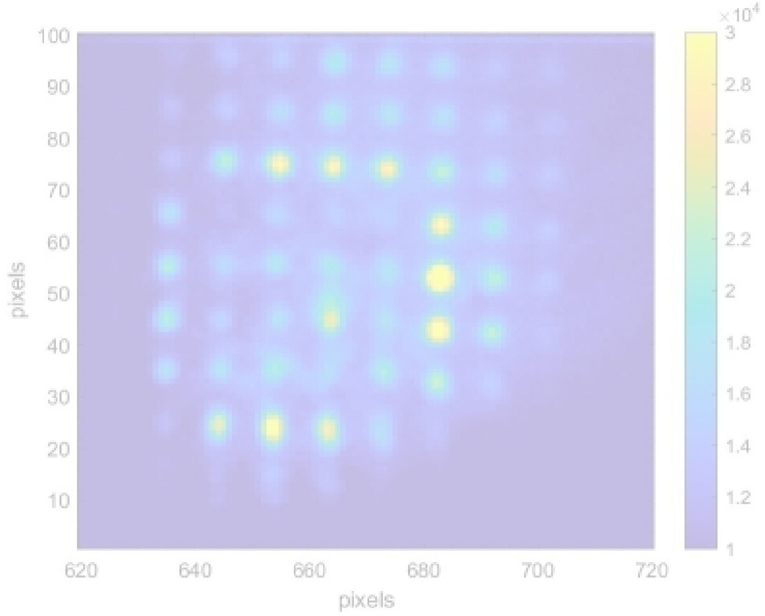
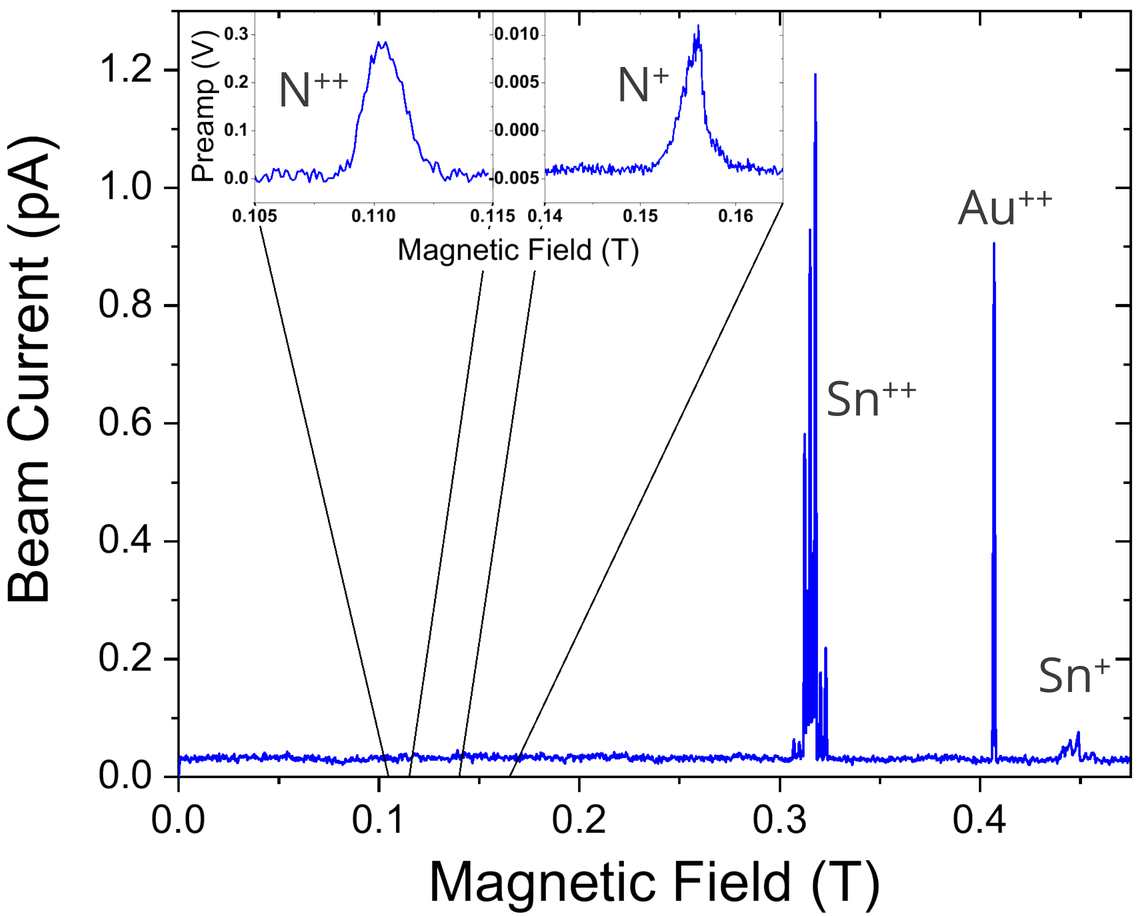




N Source - Characterization

Three parameters are important for ion sources

1. Mass Spectrum



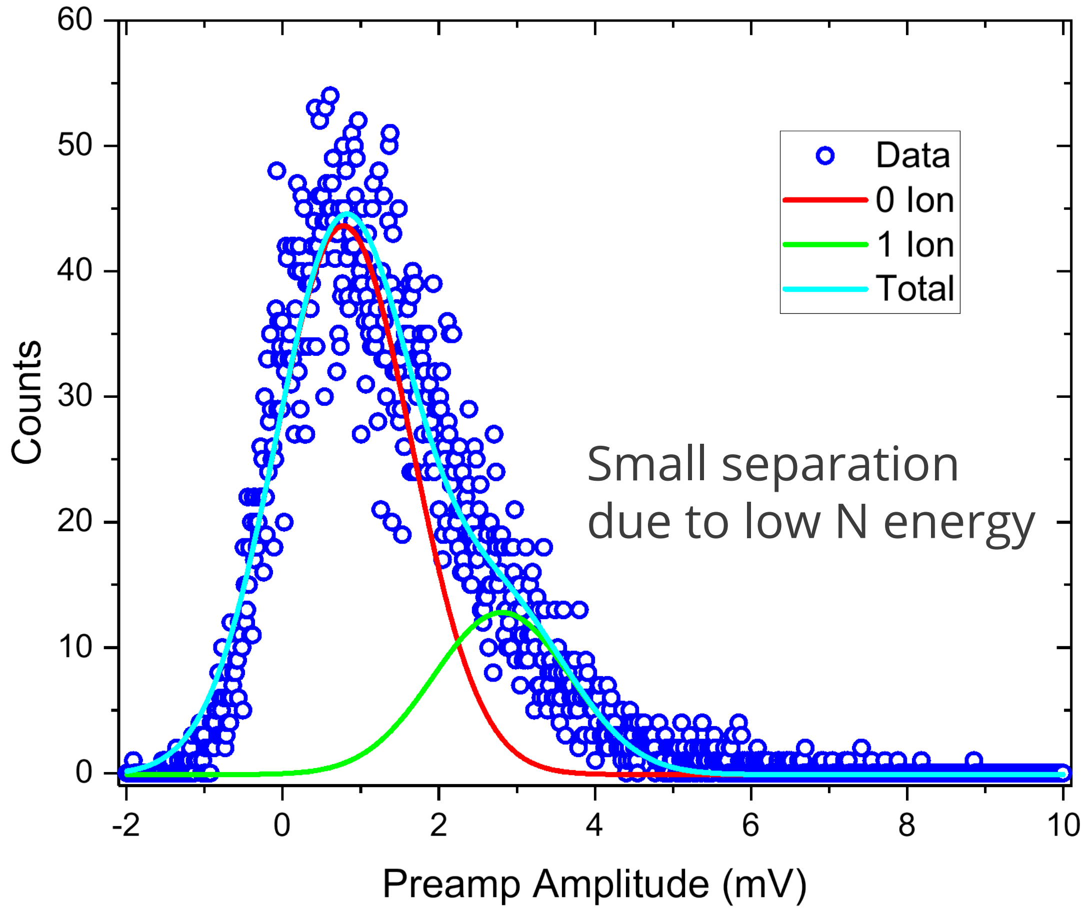
N Source - Characterization

Three parameters are important for ion sources

1. Mass Spectrum ☑
2. Beam Current

- 25 μs pulselength
- 0.48 ions/pulse

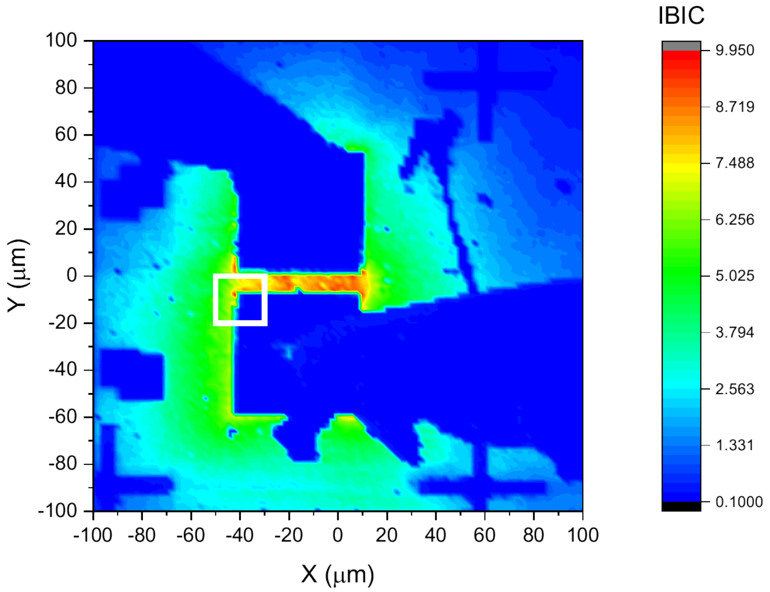
<1> ion every 50 μs ≡ 20 k ions/s

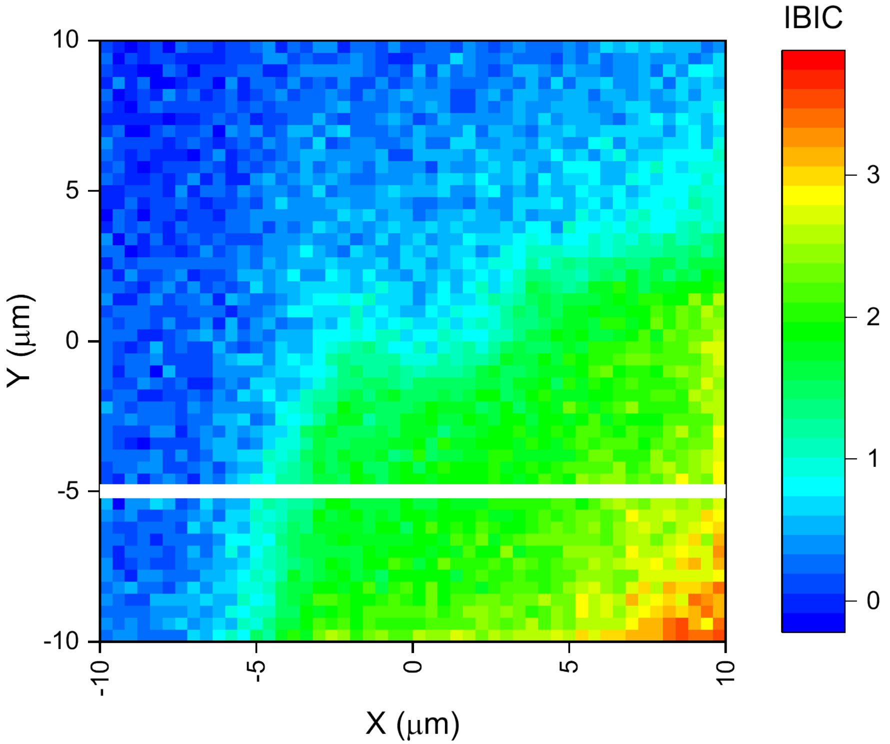


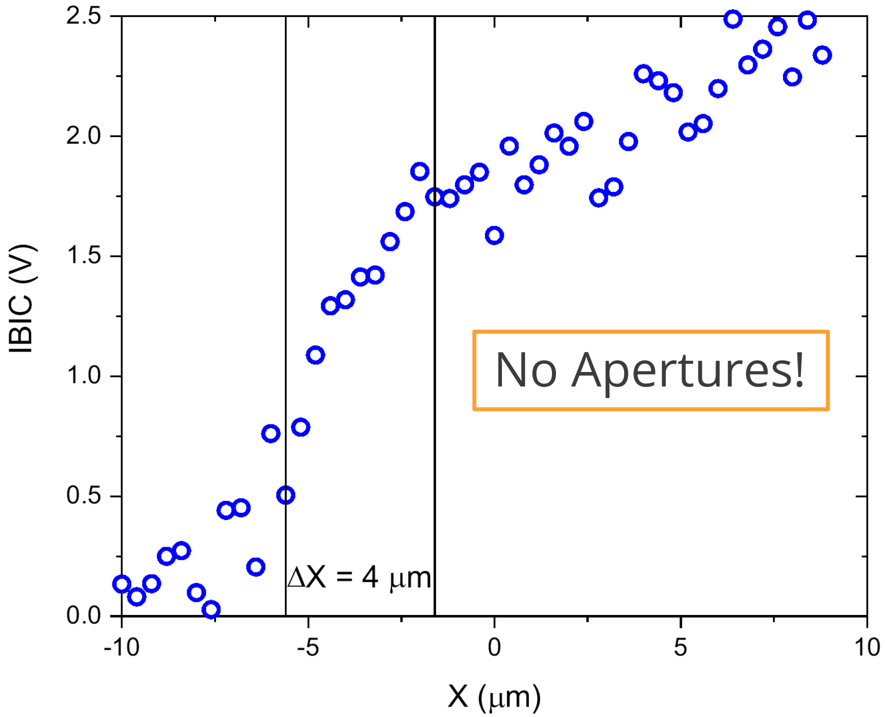
N Source - Characterization

Three parameters are important for ion sources

1. Mass Spectrum ☑
2. Beam Current ☑
3. Spot Size







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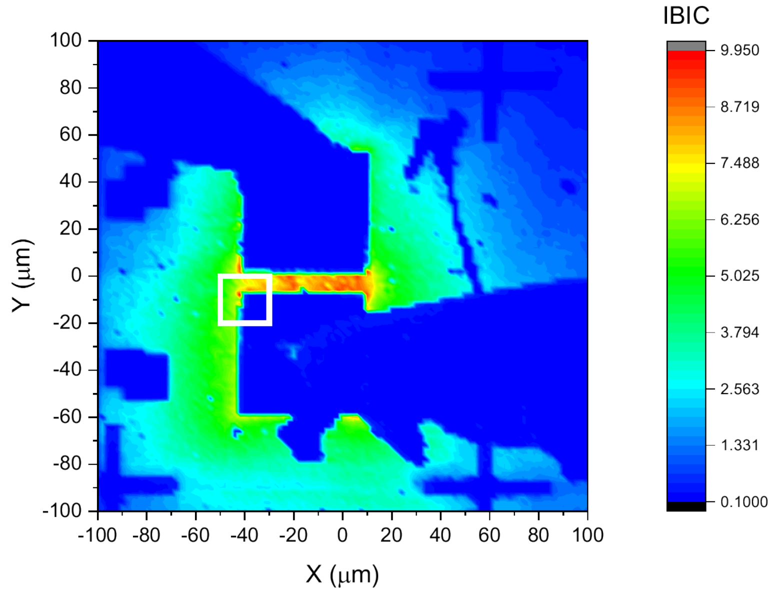

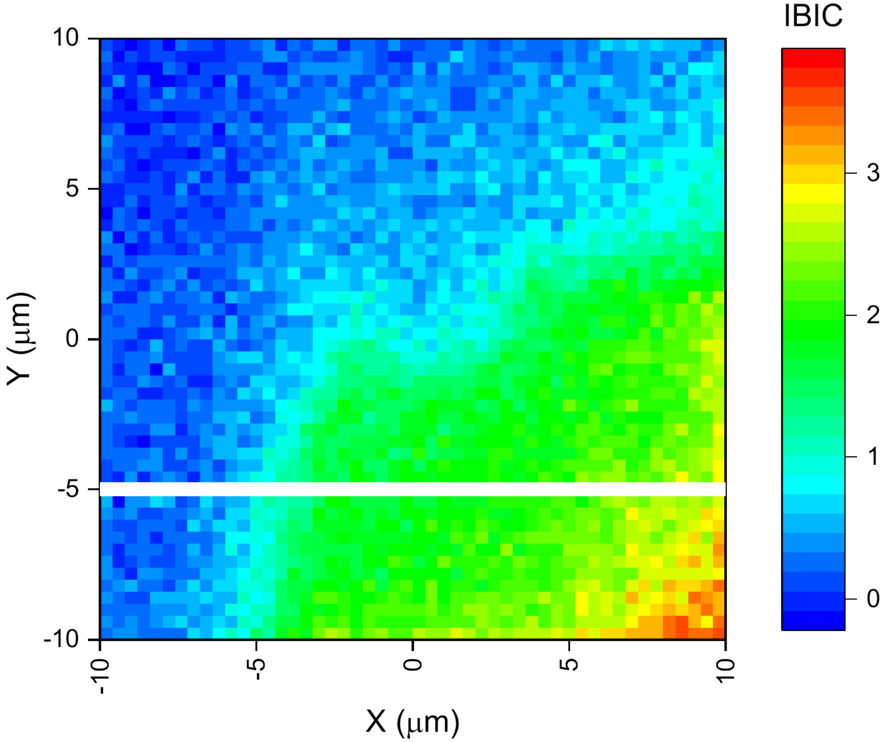

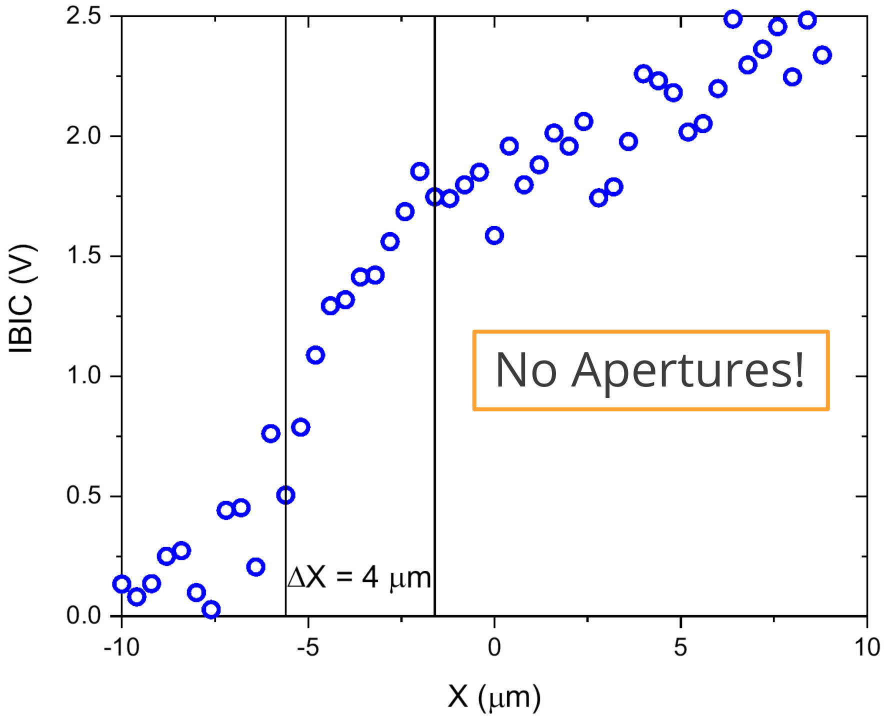

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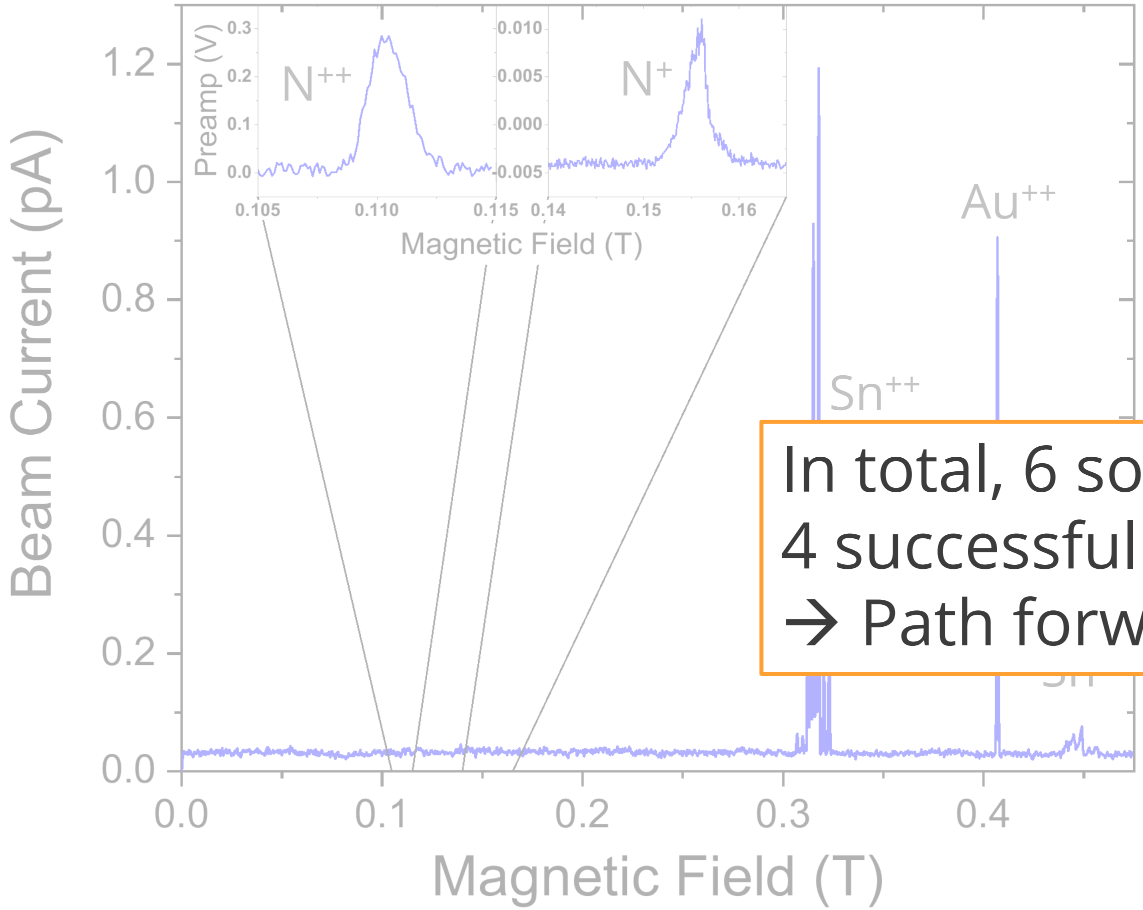
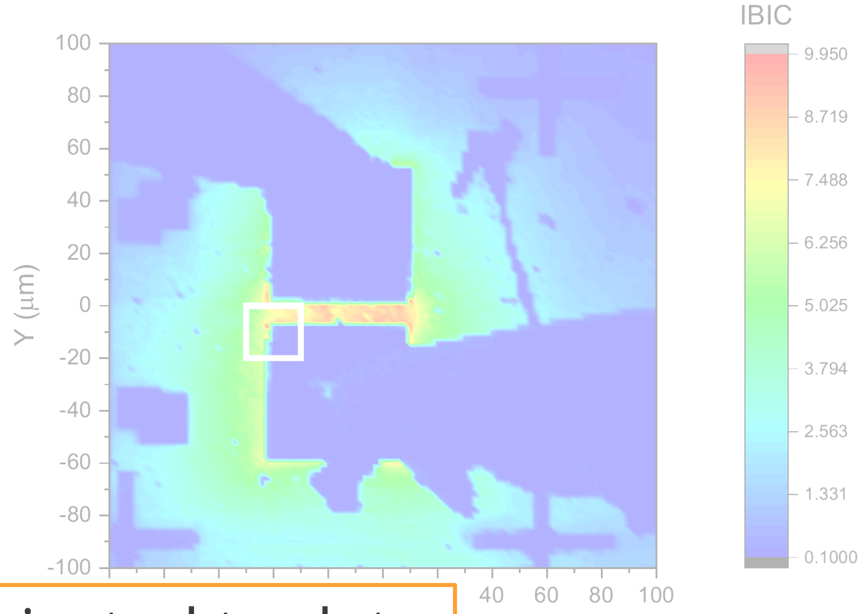
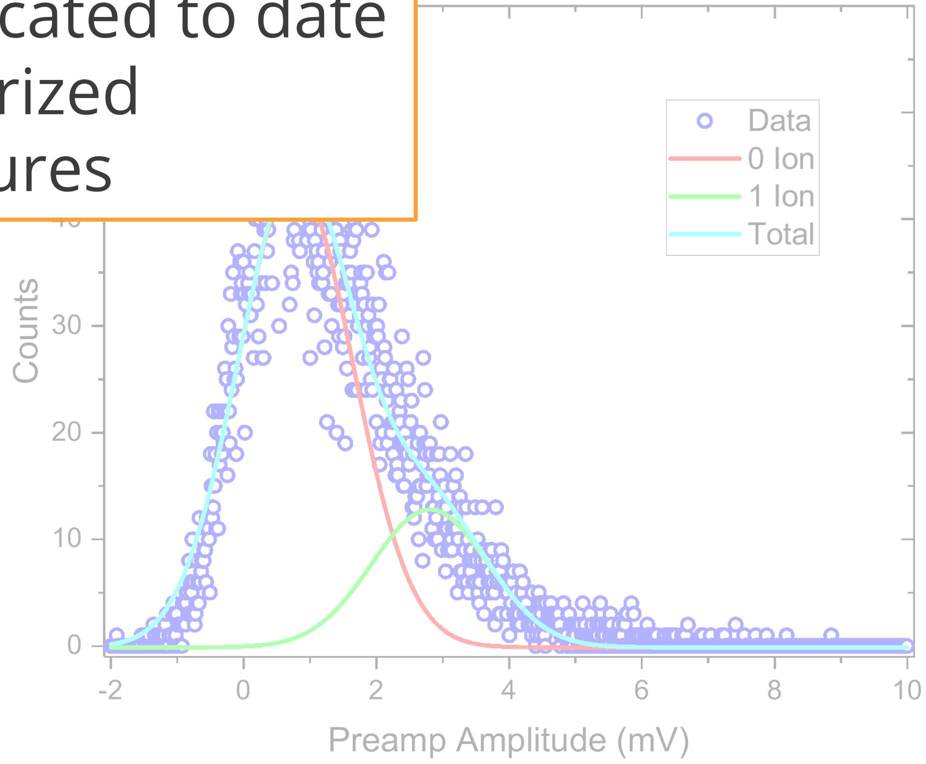




In total, 6 sources fabricated to date

4 successfully characterized

→ Path forward: Apertures



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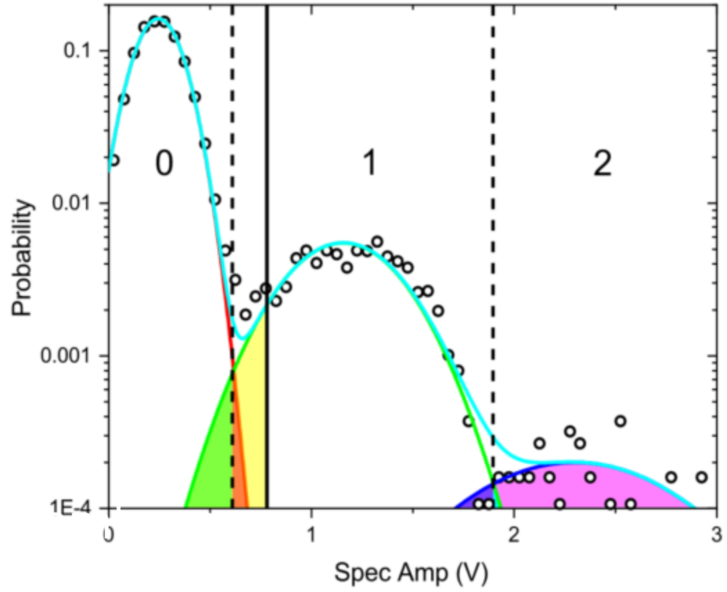

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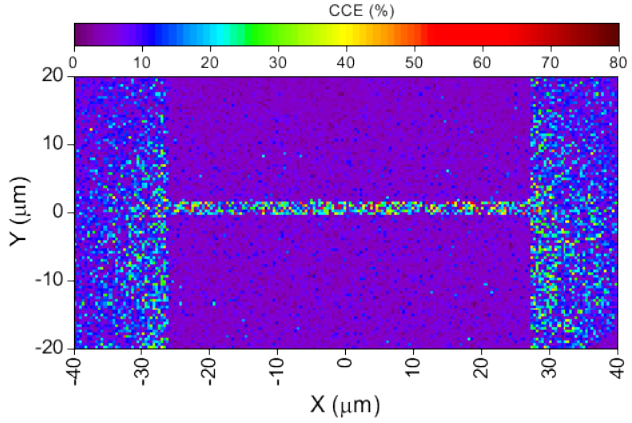
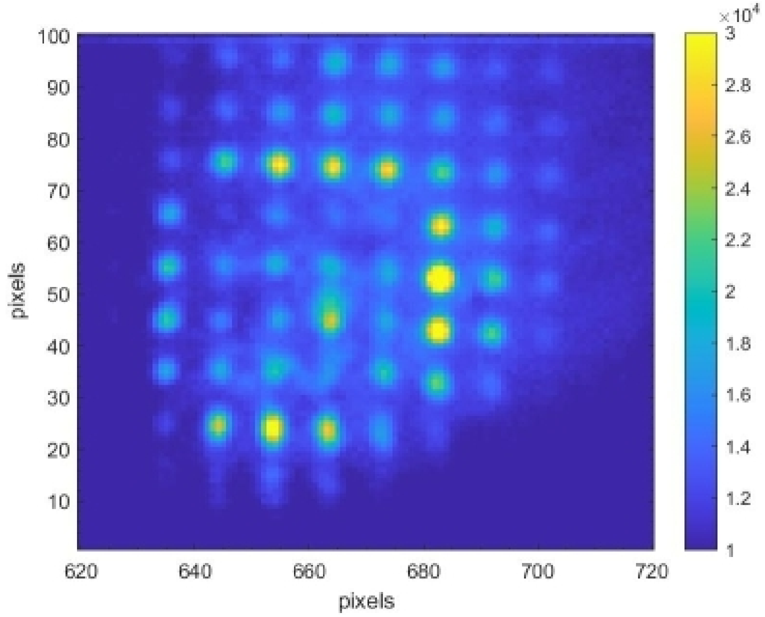




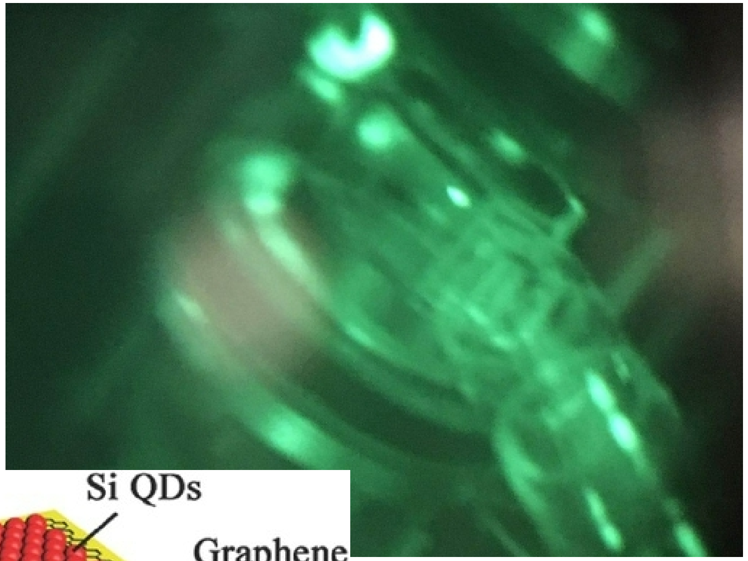
Single Photon Sources

Three main candidates

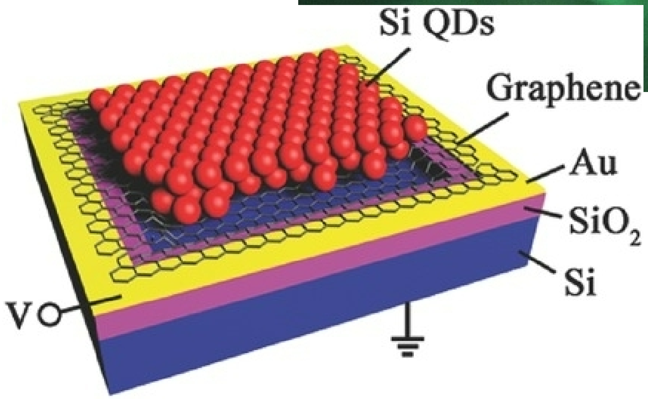
1. Atoms & Ions
   - Requires ultracold atoms
   - Hard to scale up
   - Fixed emission at transition lines
2. Quantum Dots
   - High brightness
   - Poor indistinguishability
3. Defect Emitters
   - Artificial Atoms embedded in semiconductor lattice
   - Potential for scalable fabrication
   - Examples are SiV in diamond, V_{Si} in SiC, unknown defects in GaN, unknown defects in 2D TMDs

Quantum Applications require single photon sources to distribute information

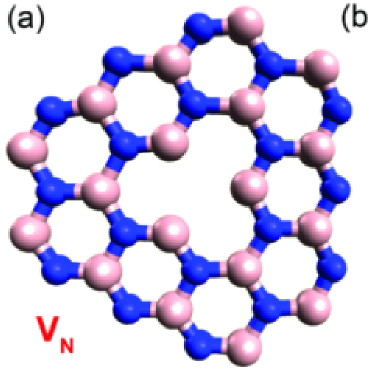
Defect emitters combine benefit of atom-based high fidelity sources with room-temperature operation and scalability of solid-state system



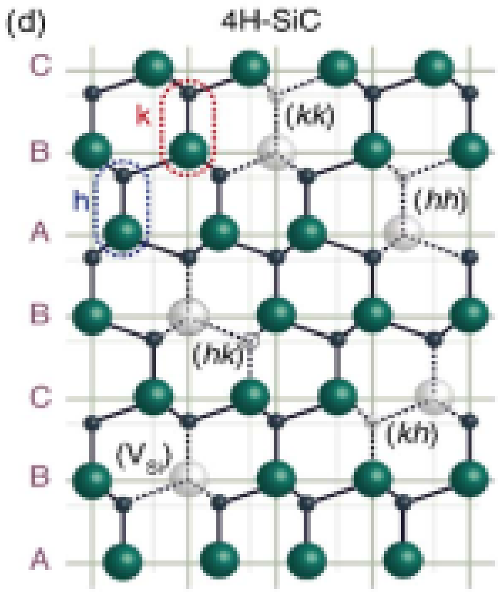
Li Lab @ FIU



T. Yu, Adv. Mater., 28: 4912 (2016)



T.T. Tran, arXiv1504.06521

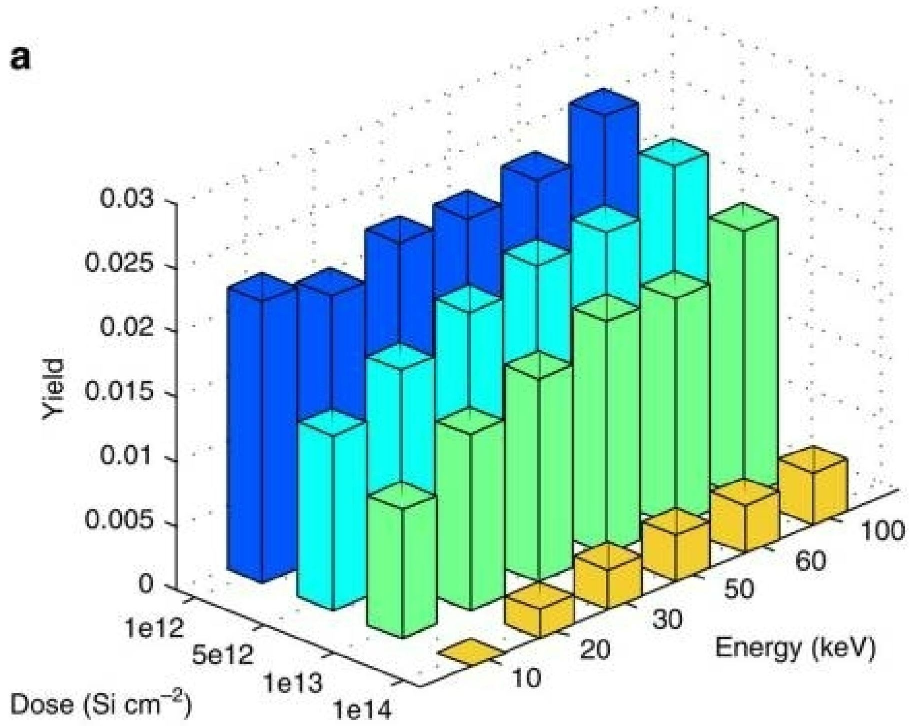


F. J. Heremans et al., Proc. IEEE 104 , 10 (2016)

Deterministic Creation of Single Photon Sources

Task: Create single optically active defect / impurity emitter at a defined location (i.e. inside optical cavity)
Issue: Poisson Statistics for low #s

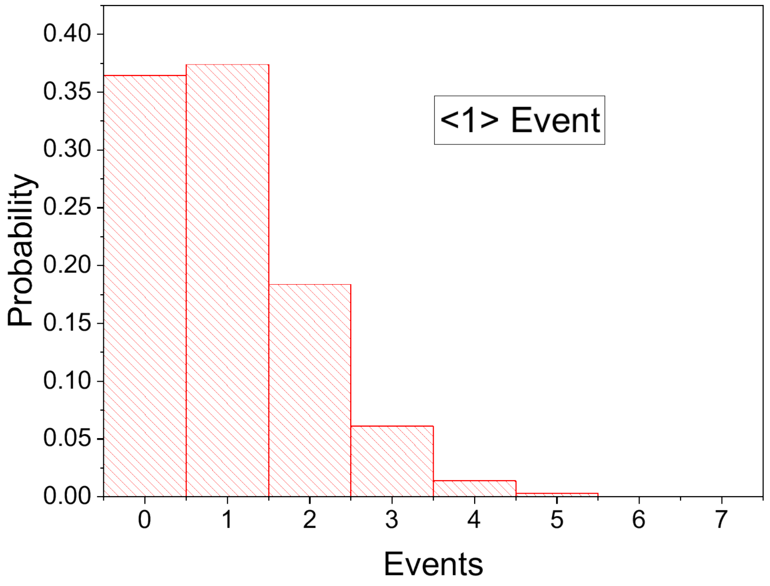
$$\text{Yield} = \frac{\# SiV}{\# Si}$$



T. Schröder et al., Nat. Commun., 8, 15376 (2017)

<30> Si/location

Fabricating Device



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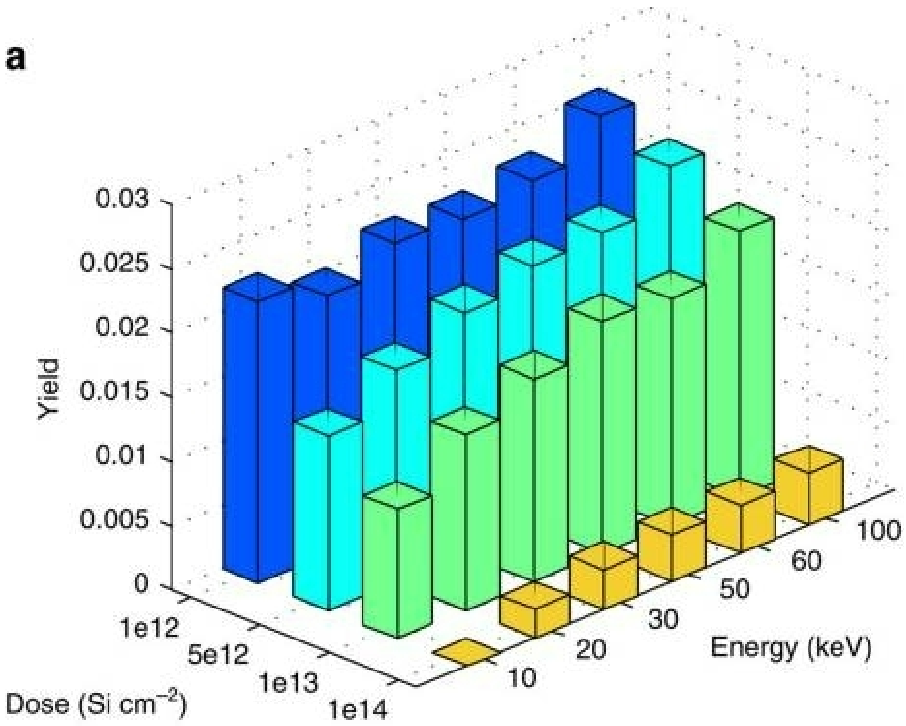
In-situ counting: Improve #Si



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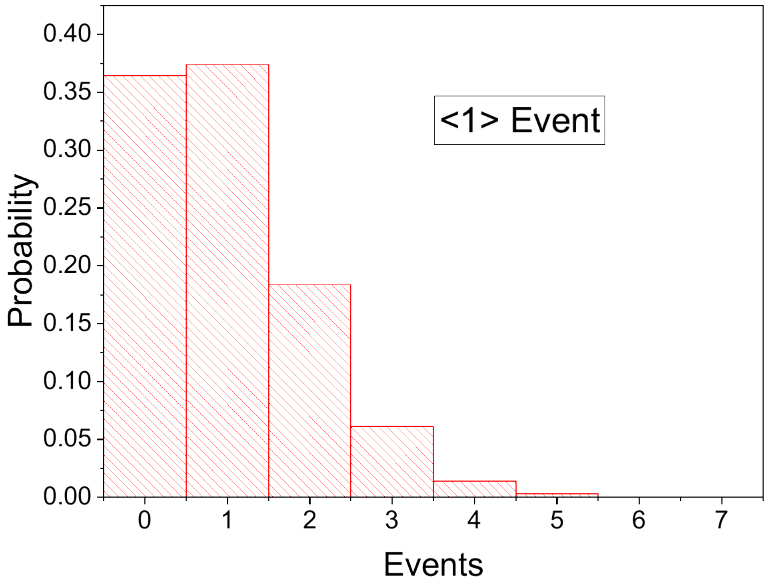

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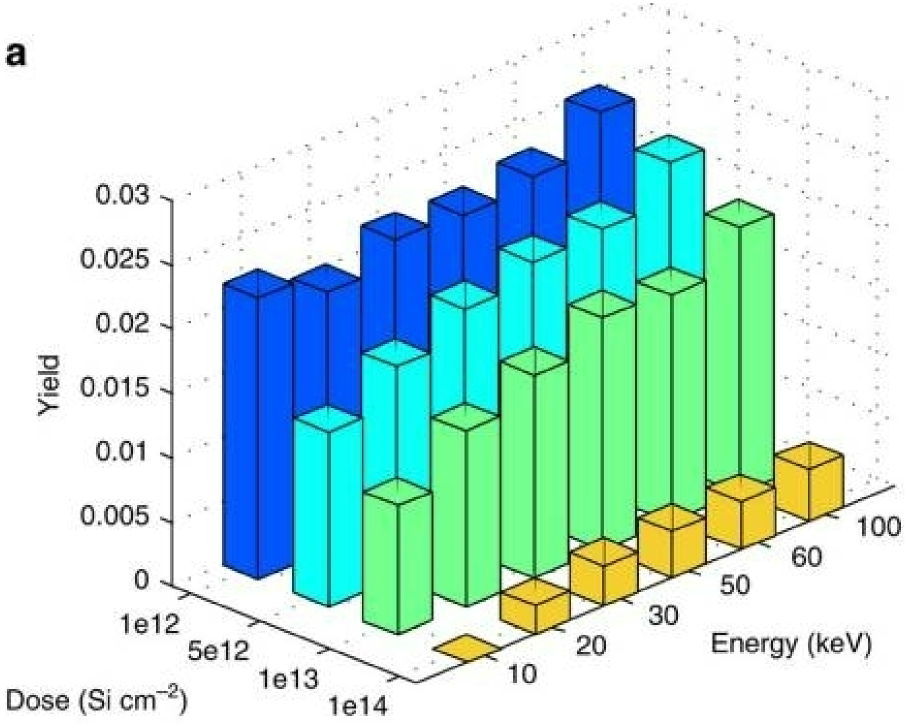
In-situ PL: Improve #SiV

In-situ counting: Improve #Si



T. Schröder et al., Nat. Commun., 8, 15376 (2017)

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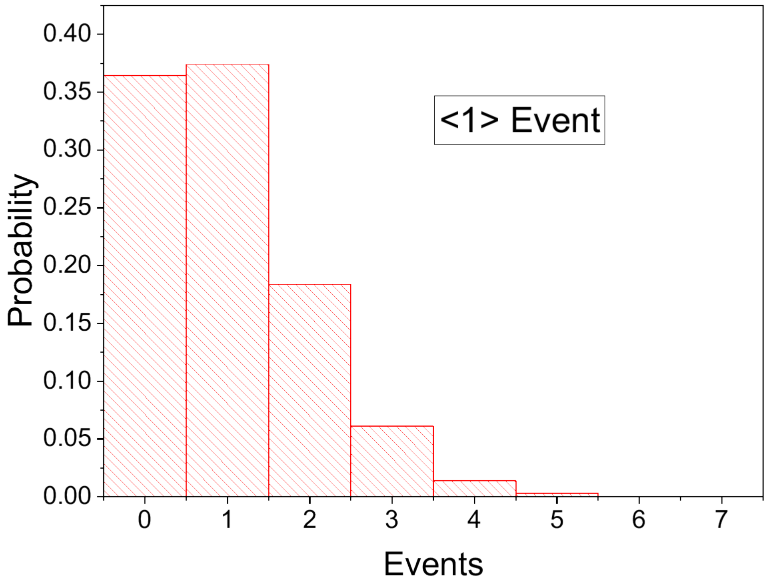
Fabricating Device



Deterministic Creation of Single Photon Sources

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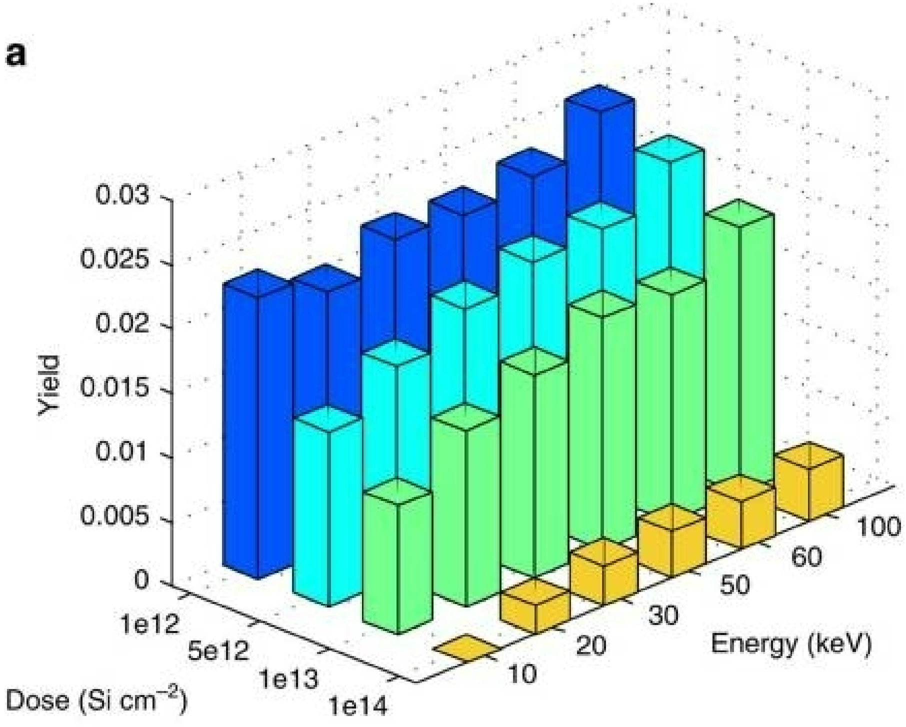
Issue: Poisson Statistics for low #s



T. Schröder et al., Nat. Commun., 8, 15376 (2017)

$$\text{Yield} = \frac{\#\,SiV}{\#\,Si}$$

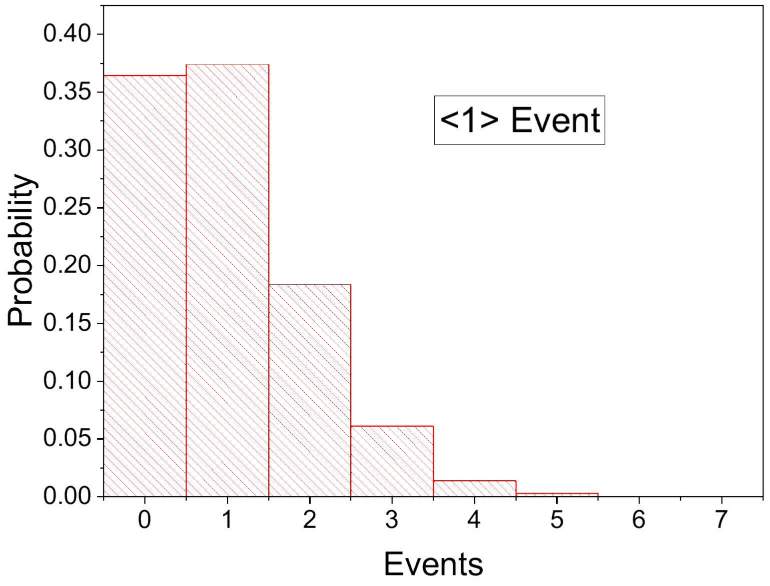
In-situ PL:
Increase #SiV

In-situ counting:
Improve #Si

In-situ anneal:
Increase Yield

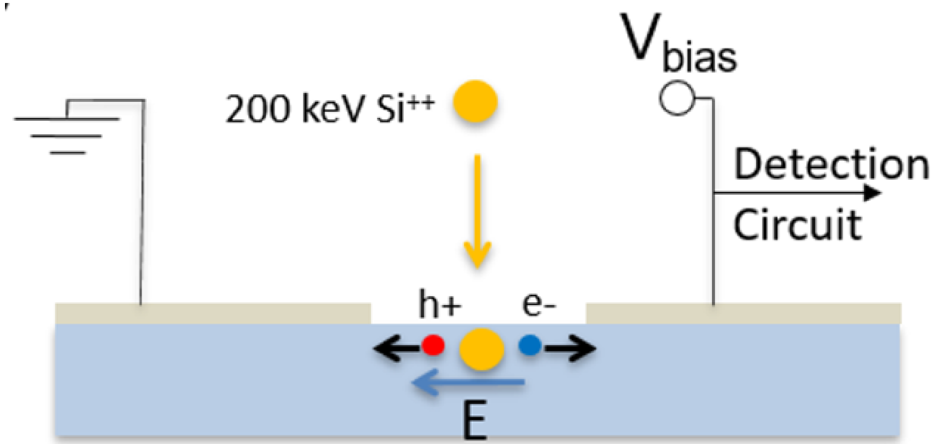
<30> Si/location

Fabricating Device

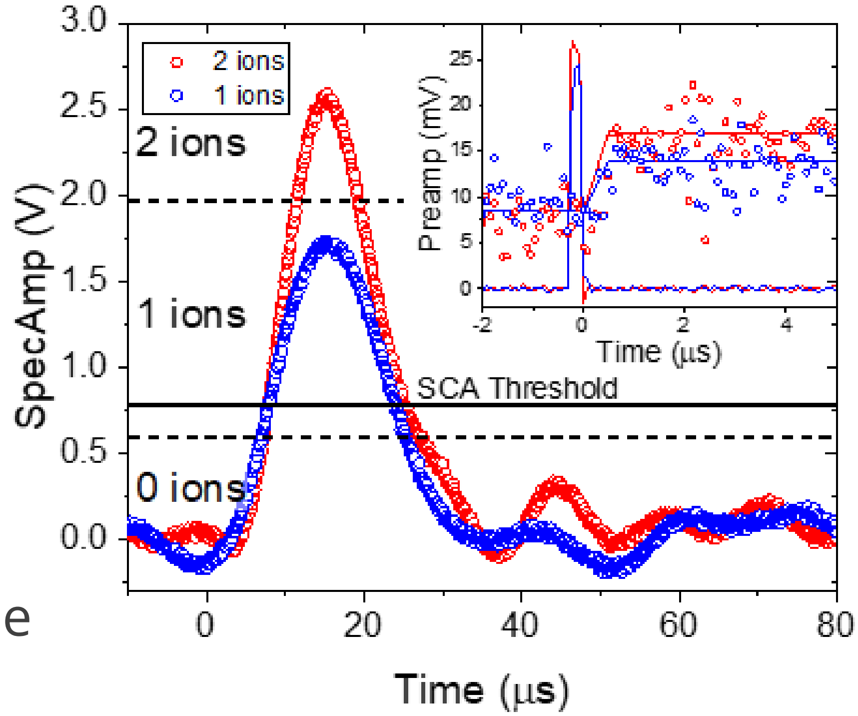


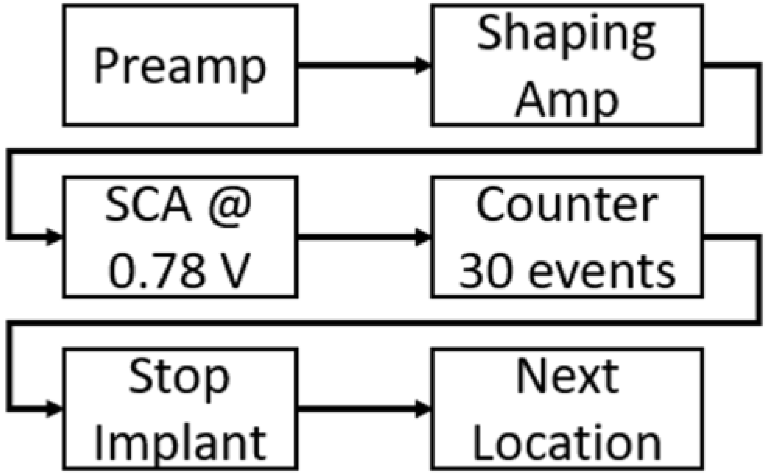
Experiment Setup

- Ion counting relies on e-h pairs generated by ion
  - ~12000 e-h pairs per ion
- Other counting techniques use secondary electrons
  - Fails for non-flat geometries
- Two in-situ electrical probes for contacting the sample
  - Bias field makes e-h pairs separate
- Induced charge gets collected by probes



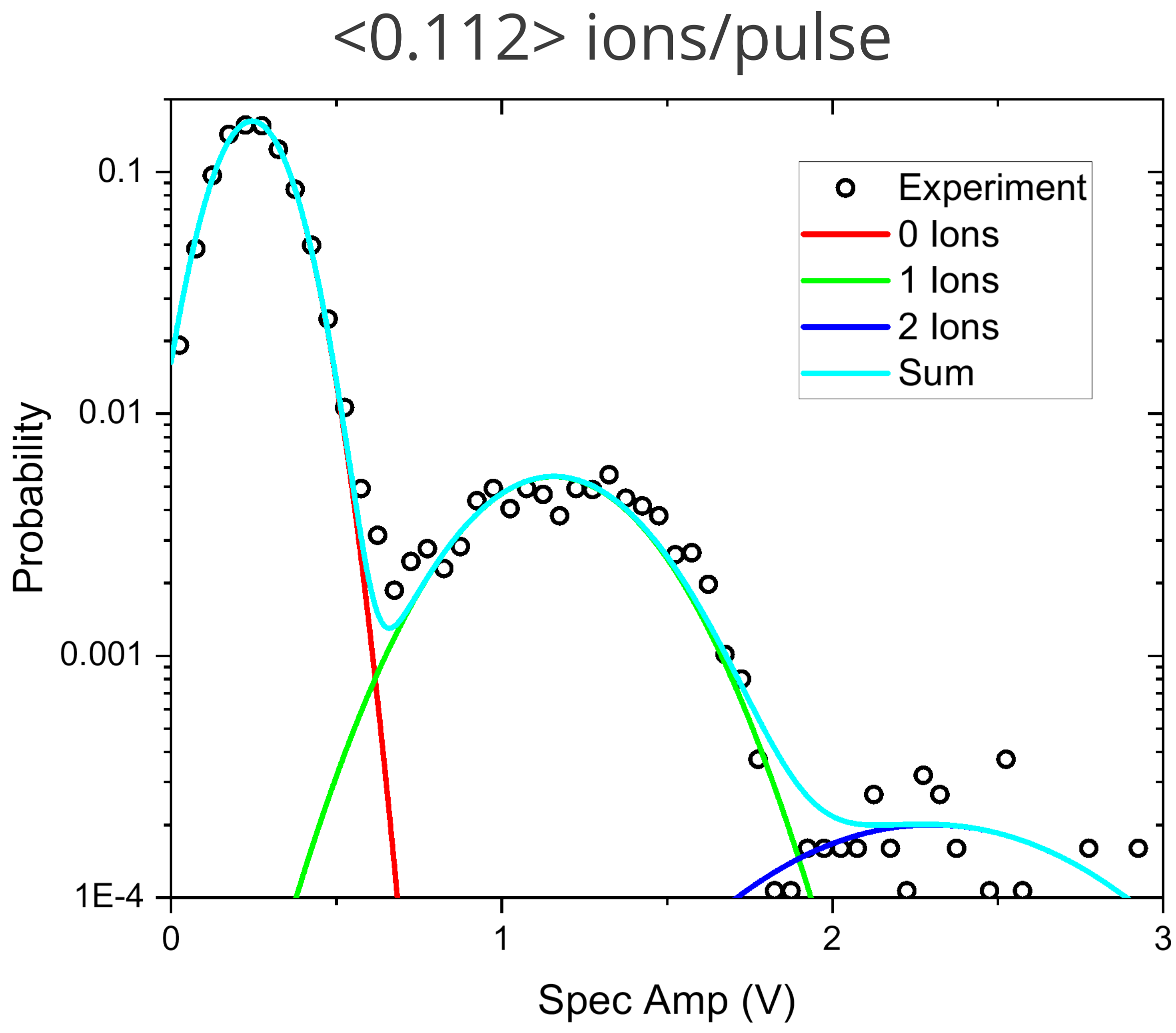
Side View





In-Situ Result

- Histogram of SpecAmp amplitude shows Poisson statistics of ion emission
- From histogram derive errors on counting measurement



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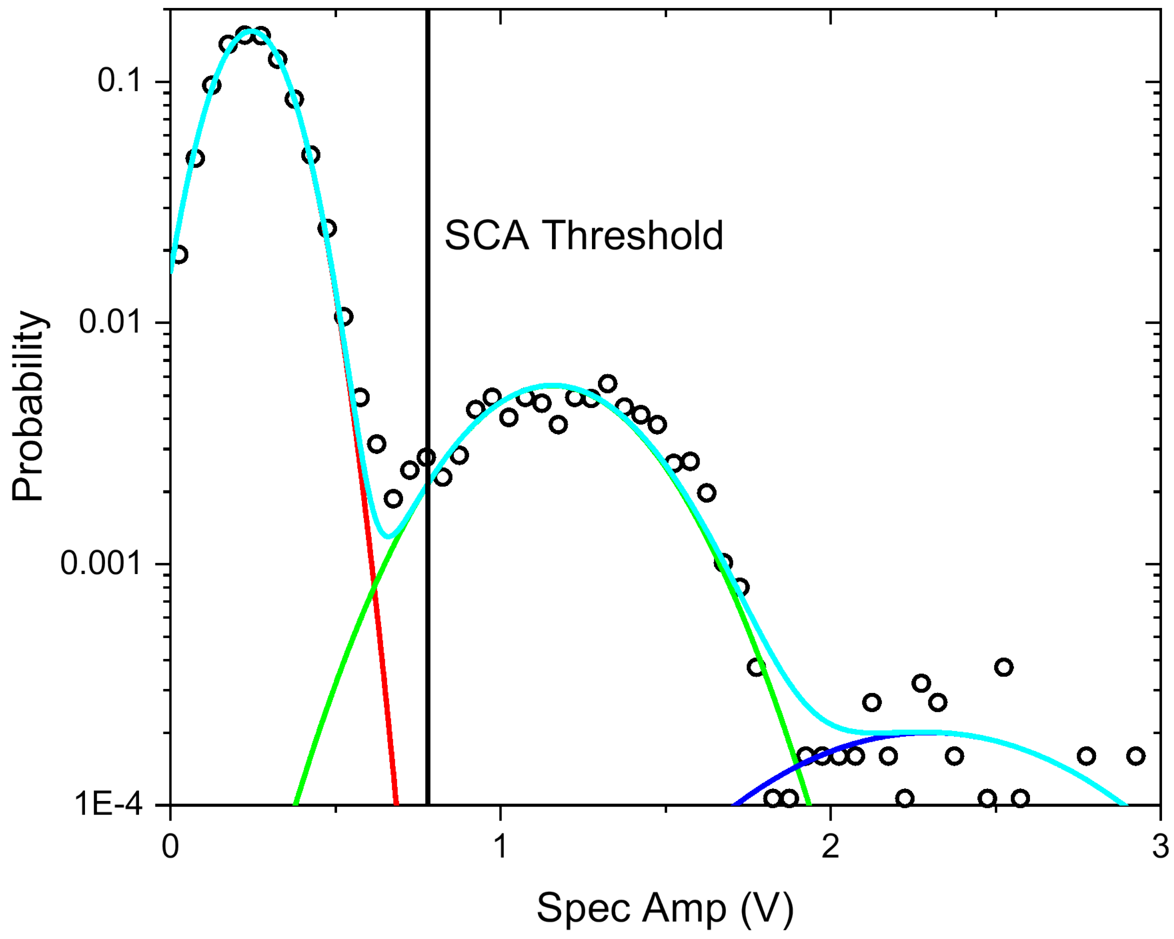
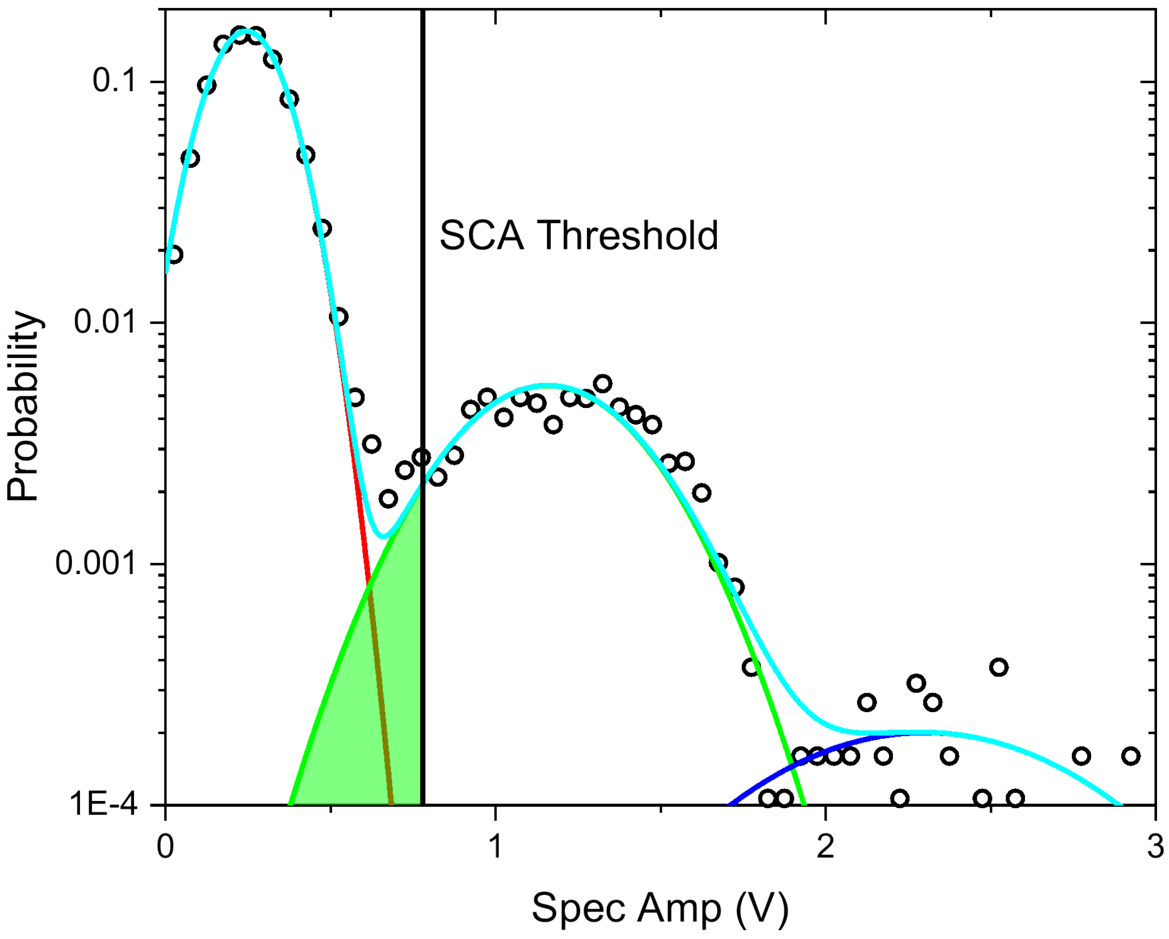
  1. False Positives - < 1 ppb



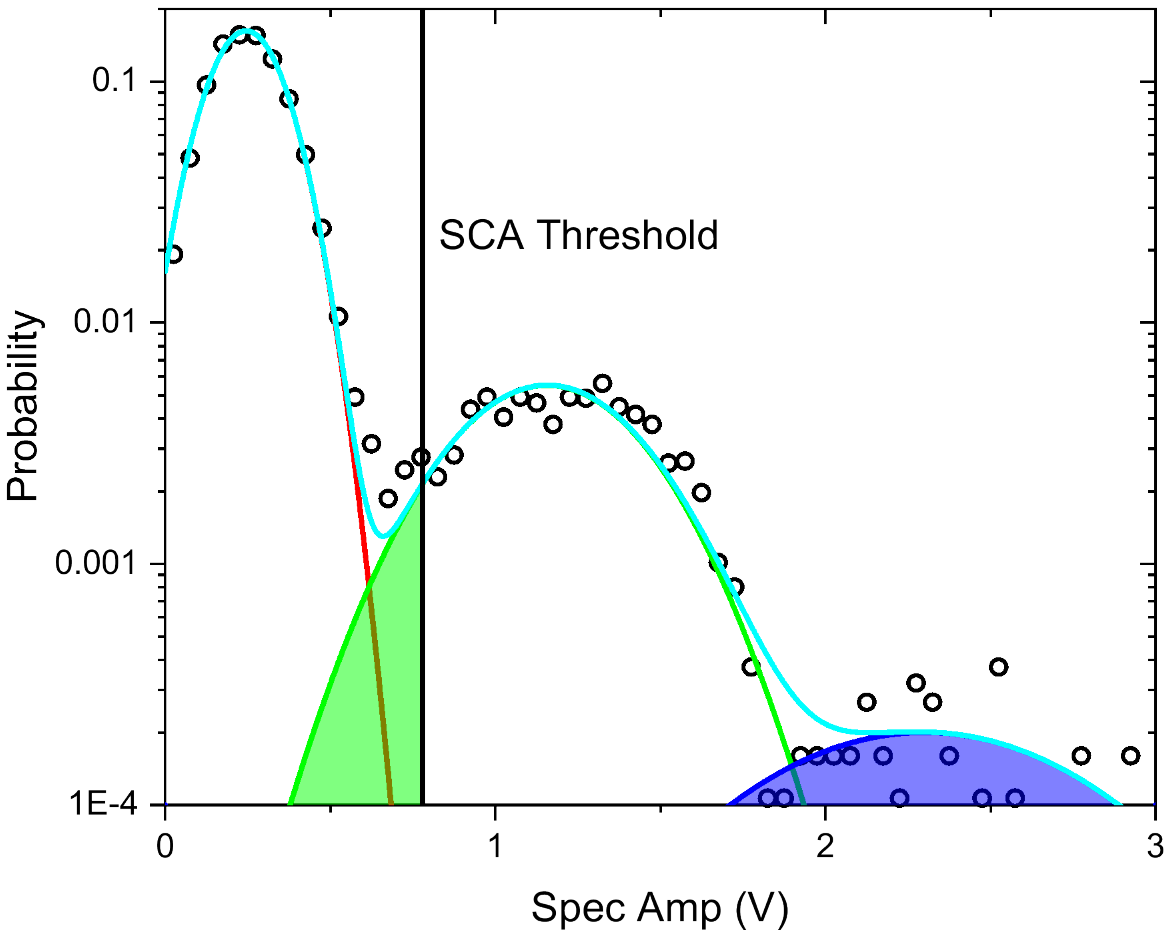
In-Situ Result

- Histogram of SpecAmp amplitude shows Poisson statistics of ion emission
- From histogram derive errors on counting measurement
  1. False Positives - < 1 ppb
  2. False Negatives +8.6 %



In-Situ Result

- Histogram of SpecAmp amplitude shows Poisson statistics of ion emission
- From histogram derive errors on counting measurement
  1. False Positives - < 1 ppb
  2. False Negatives +8.6 %
  3. Multiple Ions +5.8 %



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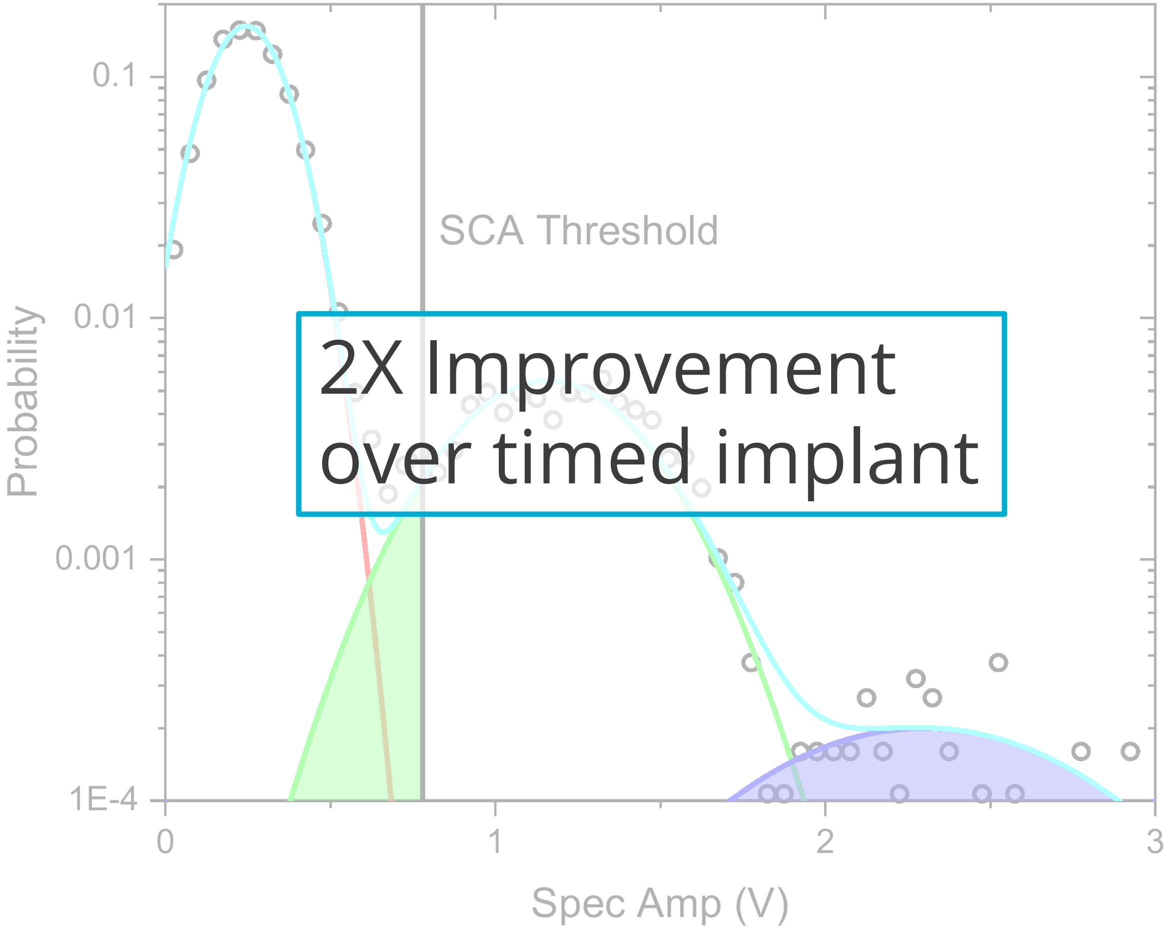
- From histogram derive errors on counting measurement
  1. False Positives - < 1 ppb
  2. False Negatives +8.6 %
  3. Multiple Ions +5.8 %
- For implantation of 30 ions

  Total Error: + 14.4 % / - 0 %

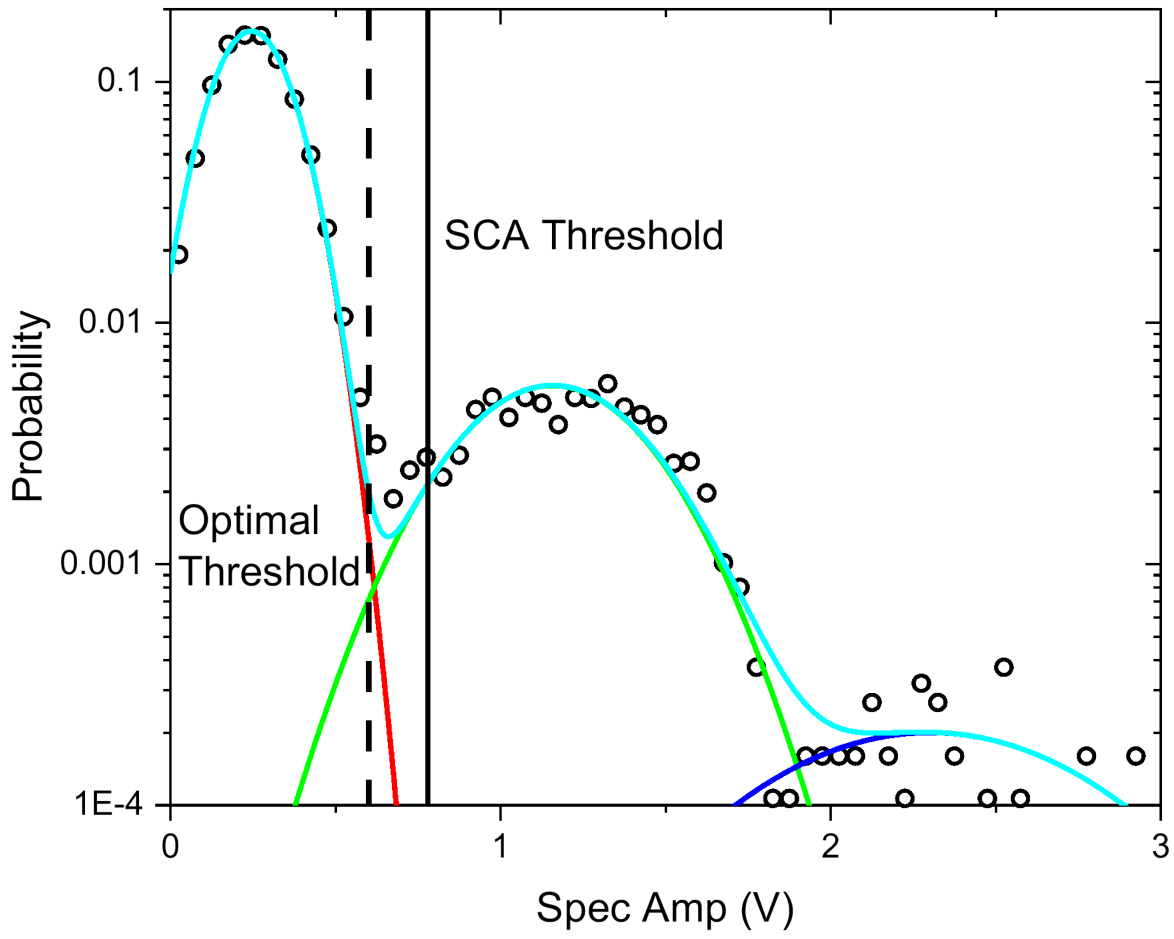
  Timed Error: +/- 18.3 %



2X Improvement over timed implant

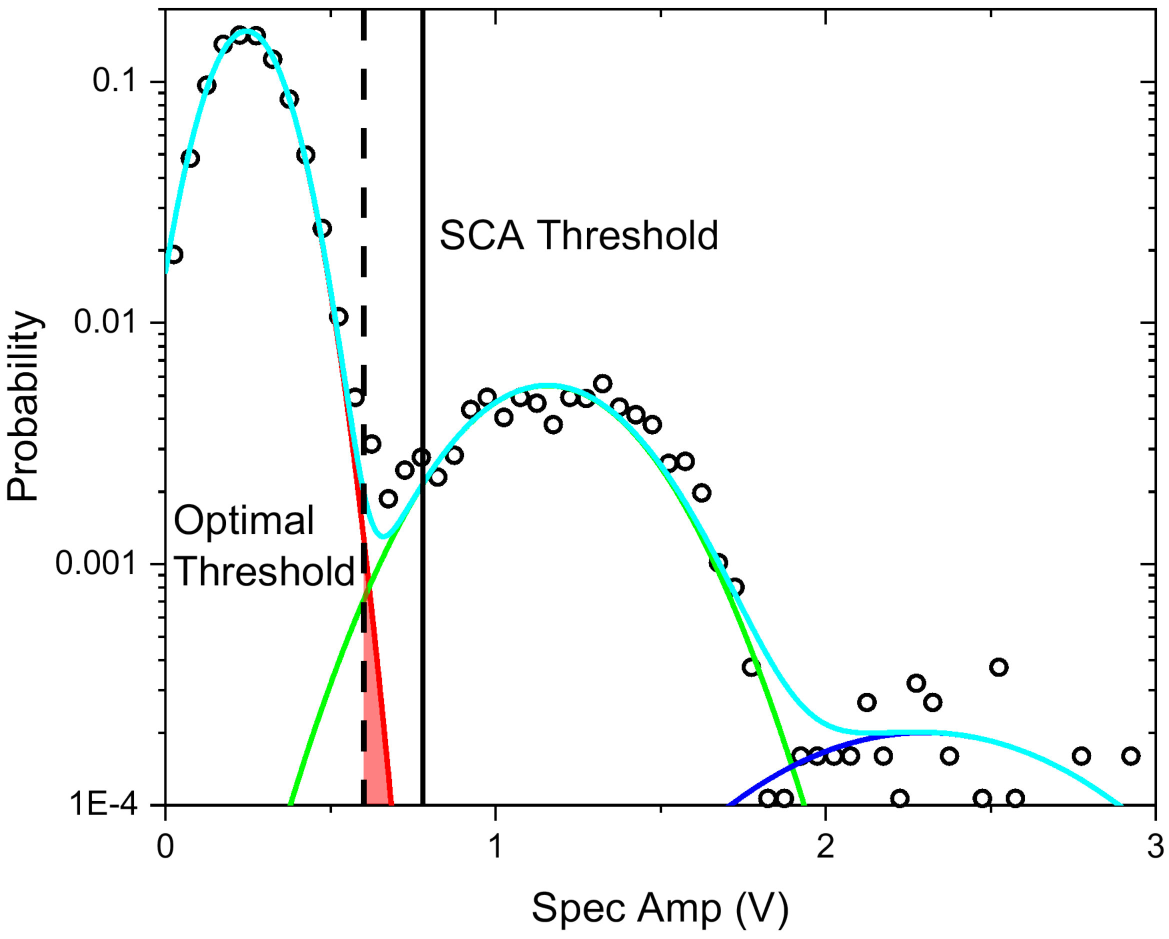
Ex-Situ Analysis

- In-situ measurement allows determination of number of ions per implantation spot post -implantation
- New and reduced errors



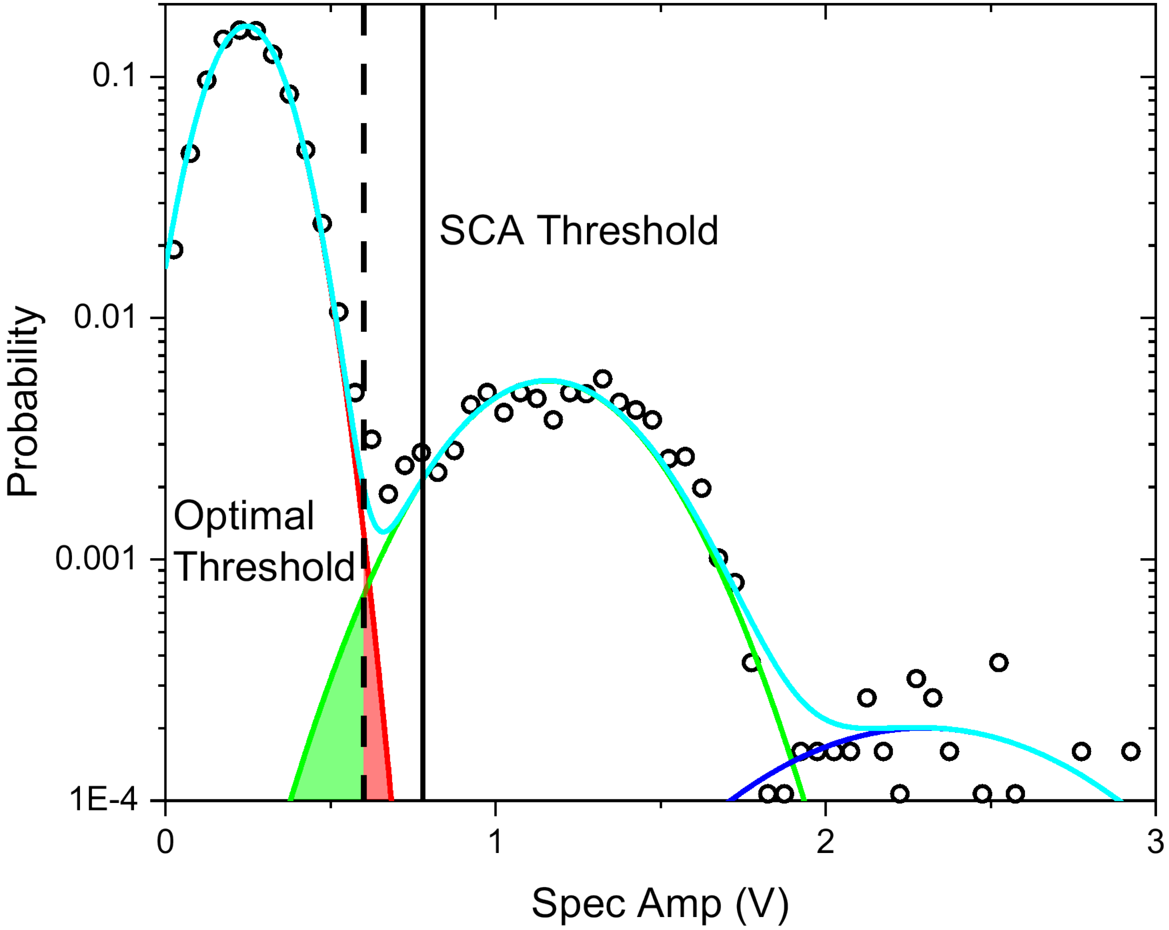
Ex-Situ Analysis

- In-situ measurement allows determination of number of ions per implantation spot post -implantation
- New and reduced errors
  1. False Positives -0.9 %



Ex-Situ Analysis

- In-situ measurement allows determination of number of ions per implantation spot post -implantation
- New and reduced errors
  1. False Positives -0.9 %
  2. False Negatives +2.3 %



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- In-situ measurement allows determination of number of ions per implantation spot post -implantation
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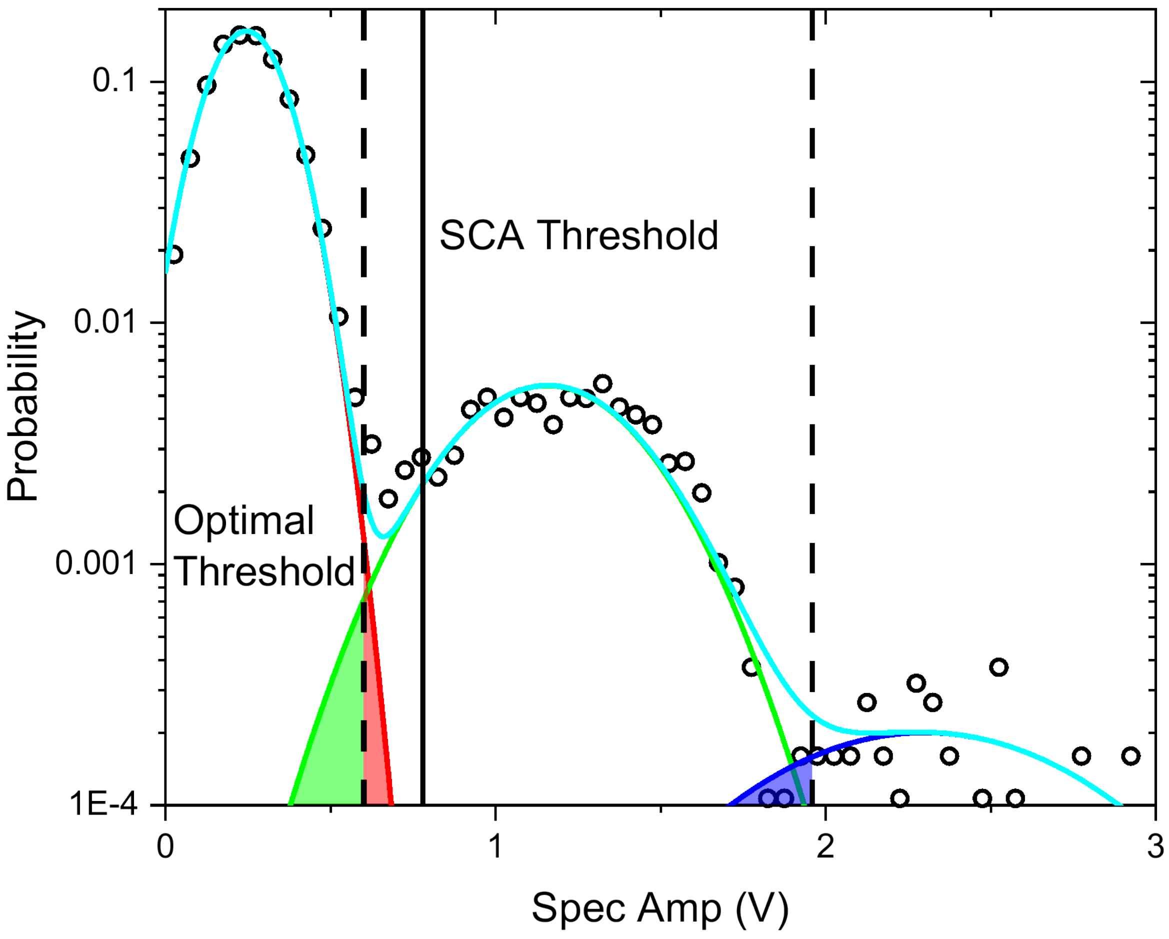
  3. Multiple Ions +1.7 %



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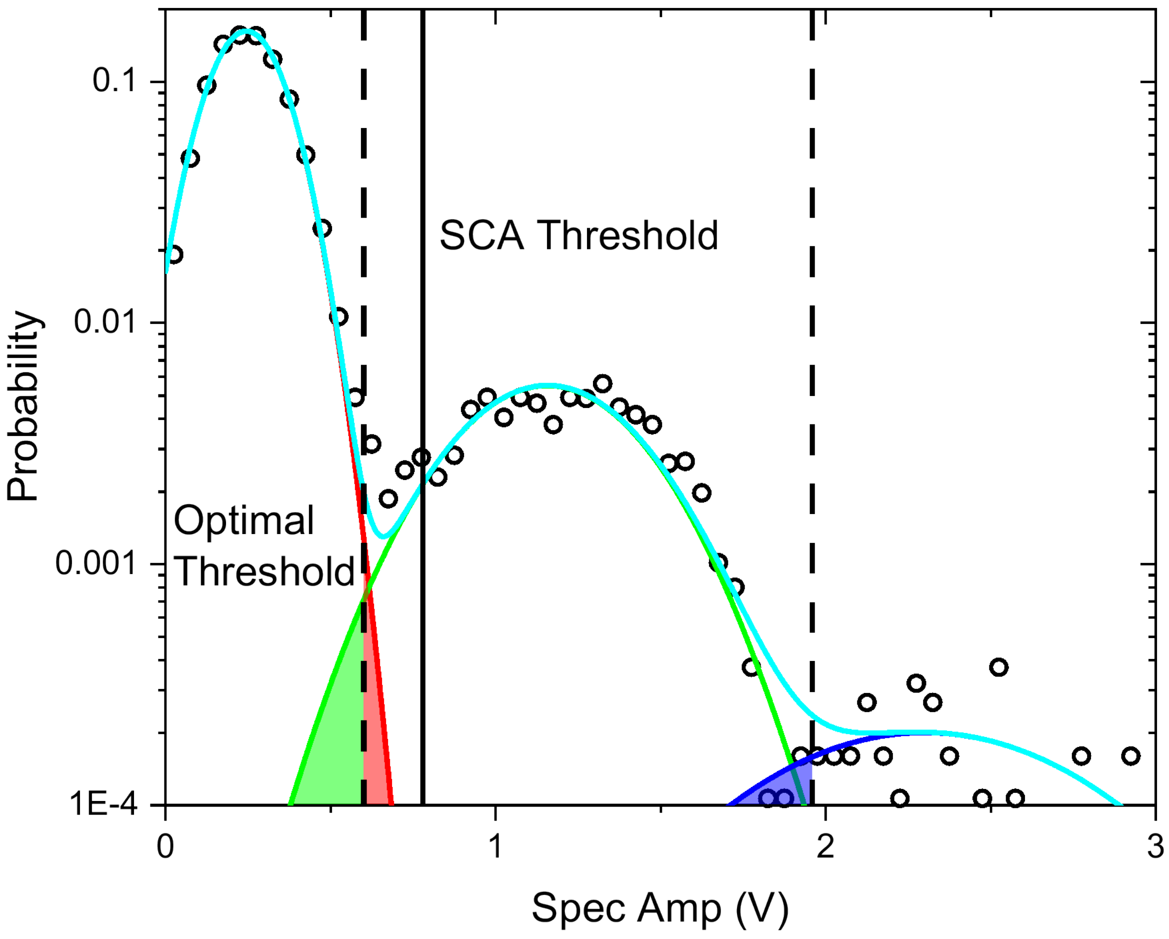
  4. Single as Double -0.2 %



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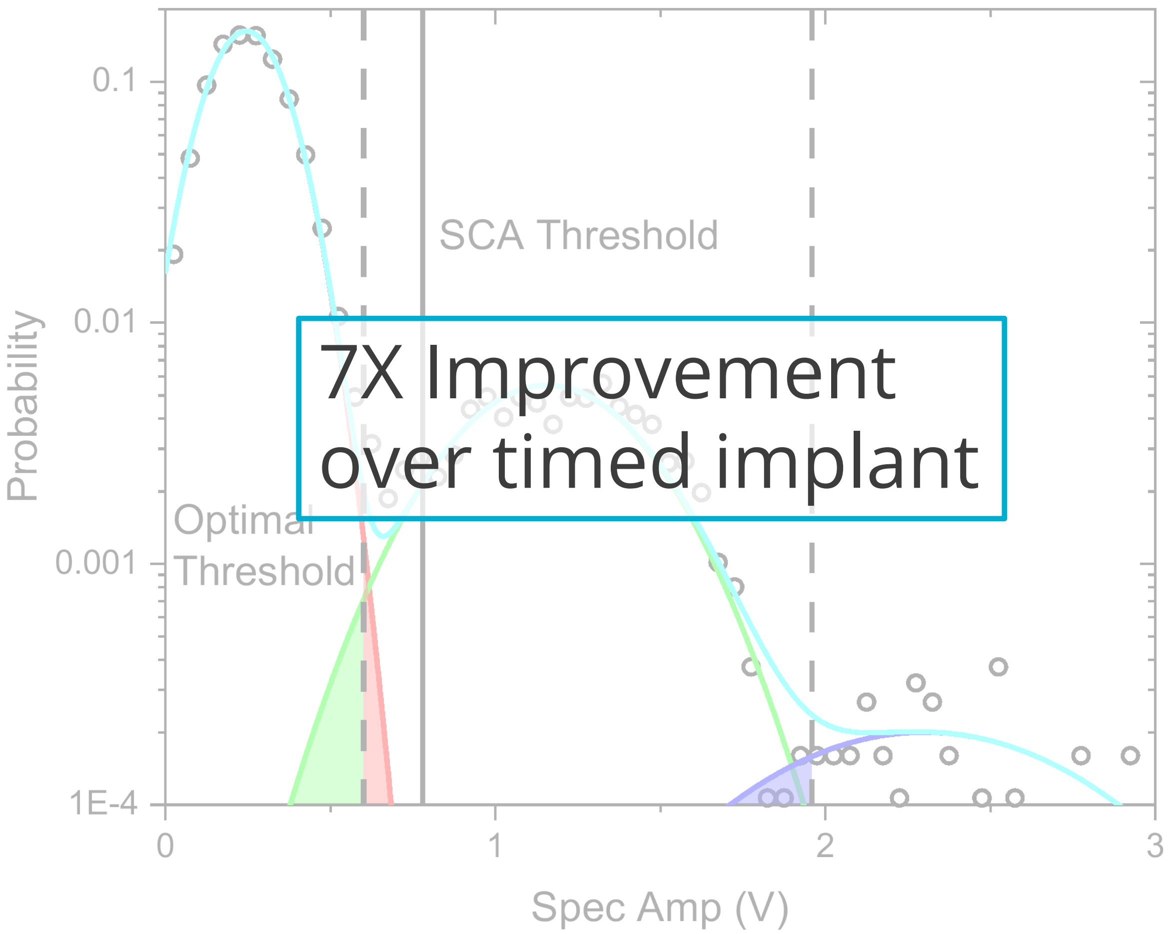
  4. Single as Double -0.2 %

- For implantation of 30 ions

  Total Error: + 4.0 % / - 1.1 %

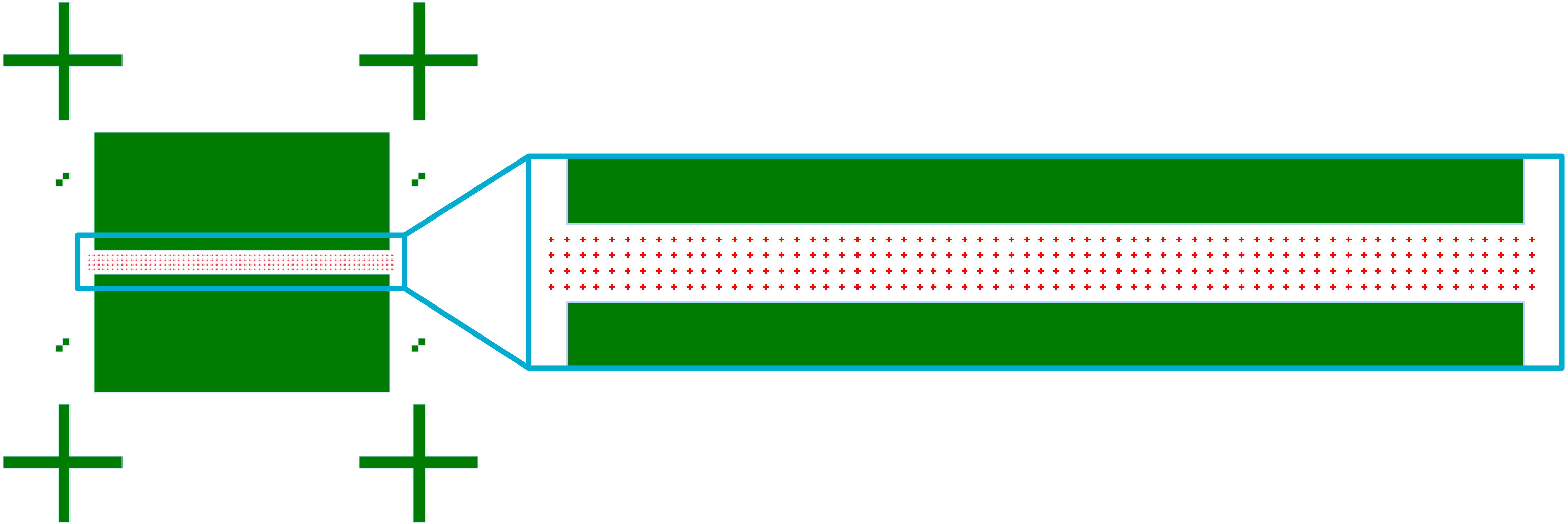
  In-Situ Error: + 14.4 % / - 0 %

  Timed Error: +/- 18.3 %



Implanted array

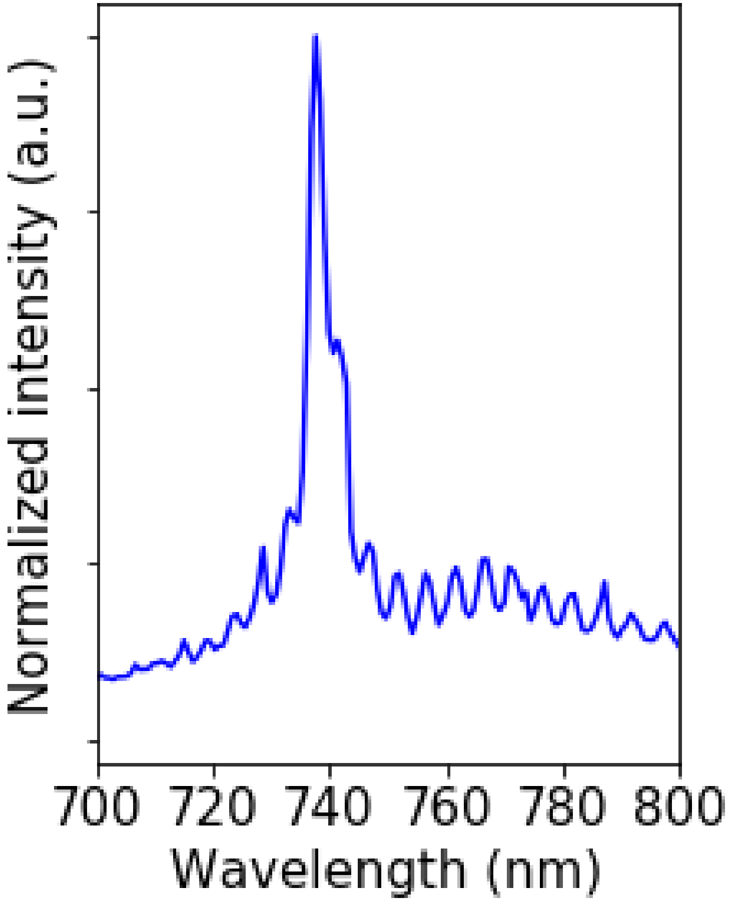
- In total implanted 5 working areas with 10, 20, 30 ions/location
- Each location has counted number of ions
- Can determine number of ions per spot with 5% error bar for each location



Photoluminescence Experiment

H. Byeon

- Photoluminescence (PL) spectrum confirms emission due to SiV
  - 738 nm emission wavelength
  - ~4 nm full-width half maximum linewidth



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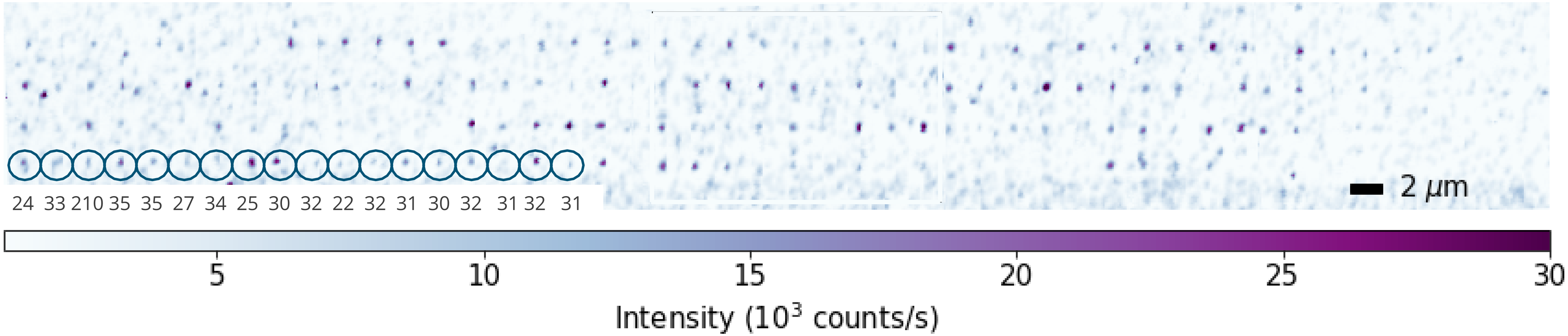

Ex-situ analysis of in-situ data allows determination of #ions/spot with 5% accuracy

Photoluminescence Experiment

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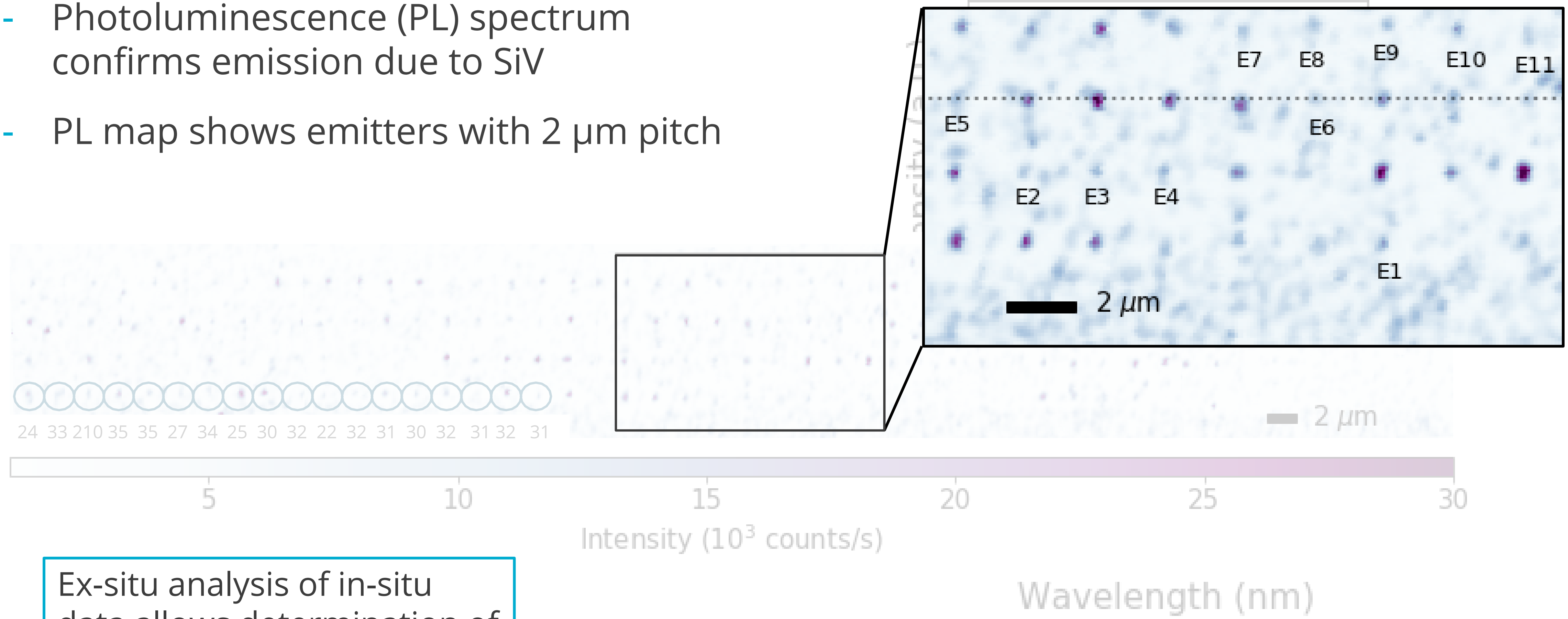
- PL map shows emitters with 2 μm pitch



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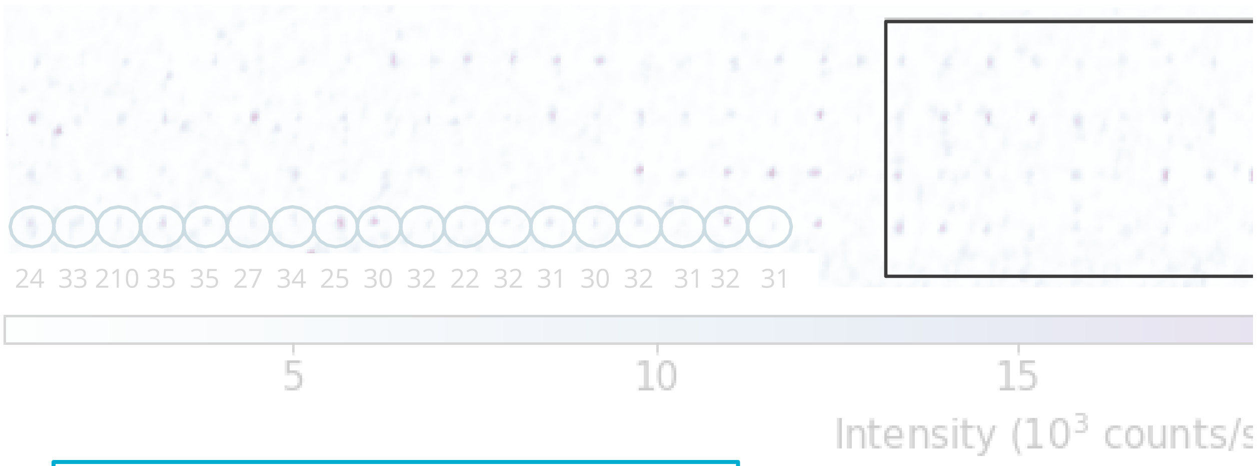
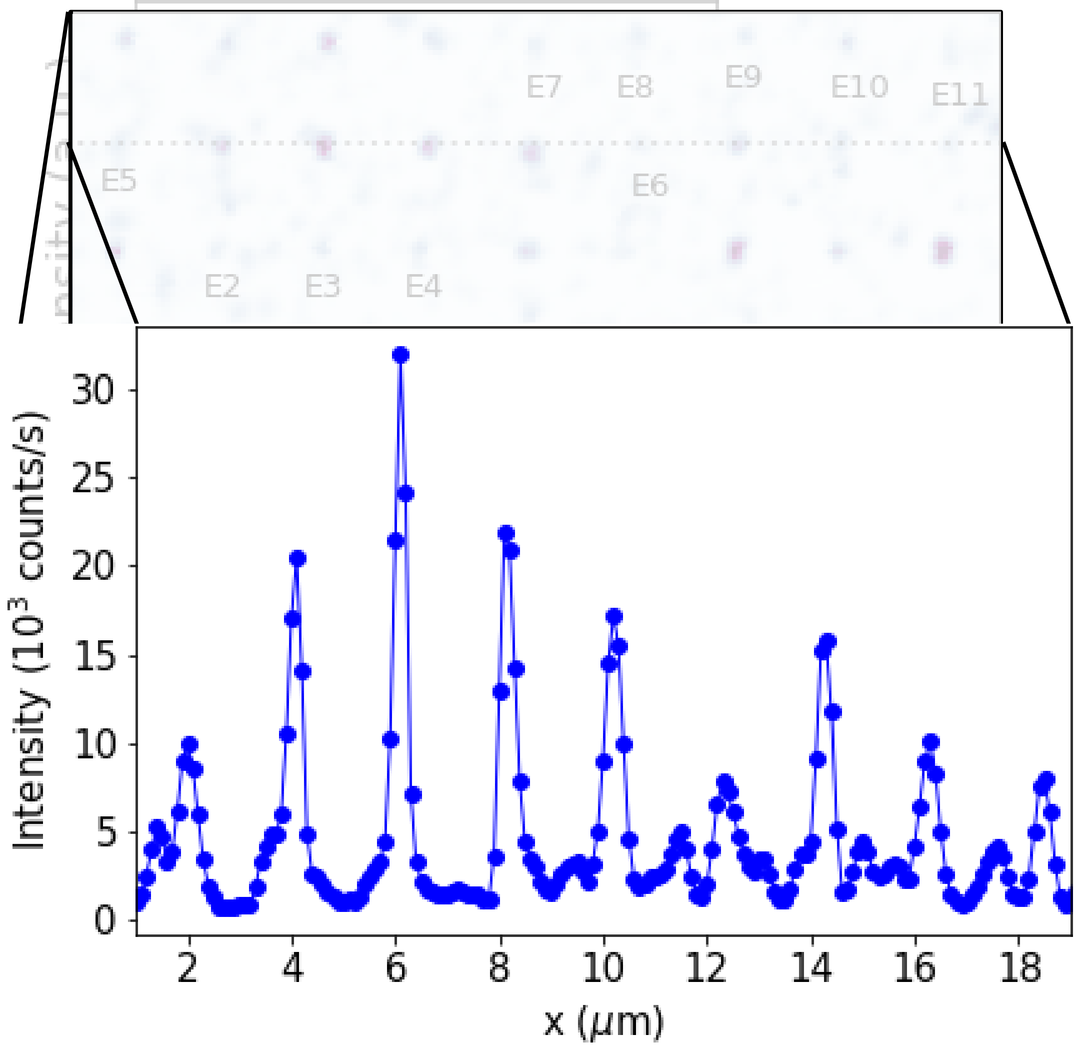




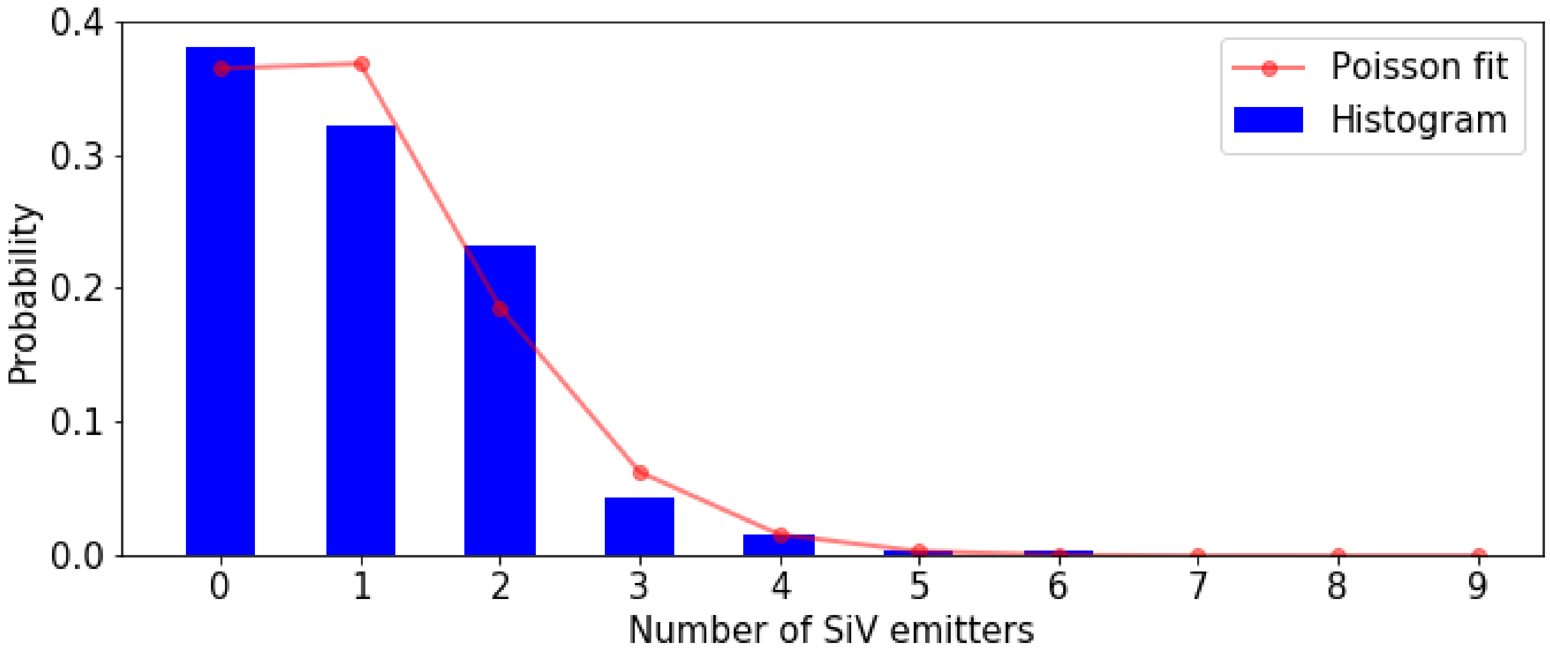
Ex-situ analysis of in-situ data allows determination of #ions/spot with 5% accuracy

Photoluminescence Experiment

H. Byeon

- Bin PL into 0,1,2,... SiV
  - Countrate of single emitter 10.7 ± 0.2 kcps
- From binning get frequency of 0,1,2,... emitters
- Fit histogram to Poisson distribution

→ Avg. # emitters = 1.01±0.07

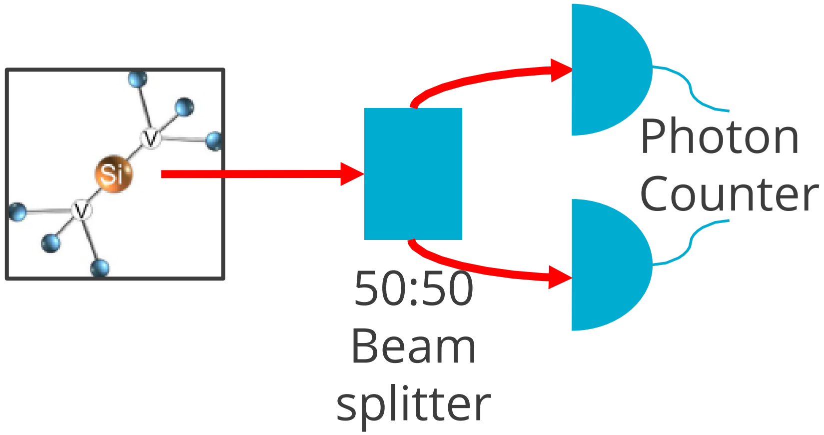


Error on # emitters matches counting error

Hanbury-Brown-Twiss

H. Byeon

- PL alone not enough to confirm single emitter
- Use Hanbury-Brown-Twiss interferometry to confirm single photon emission
  - If single, only detect at one photon counter



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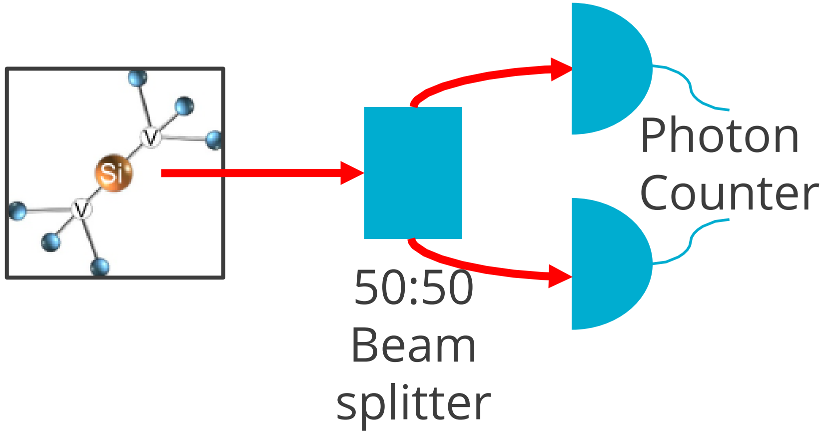
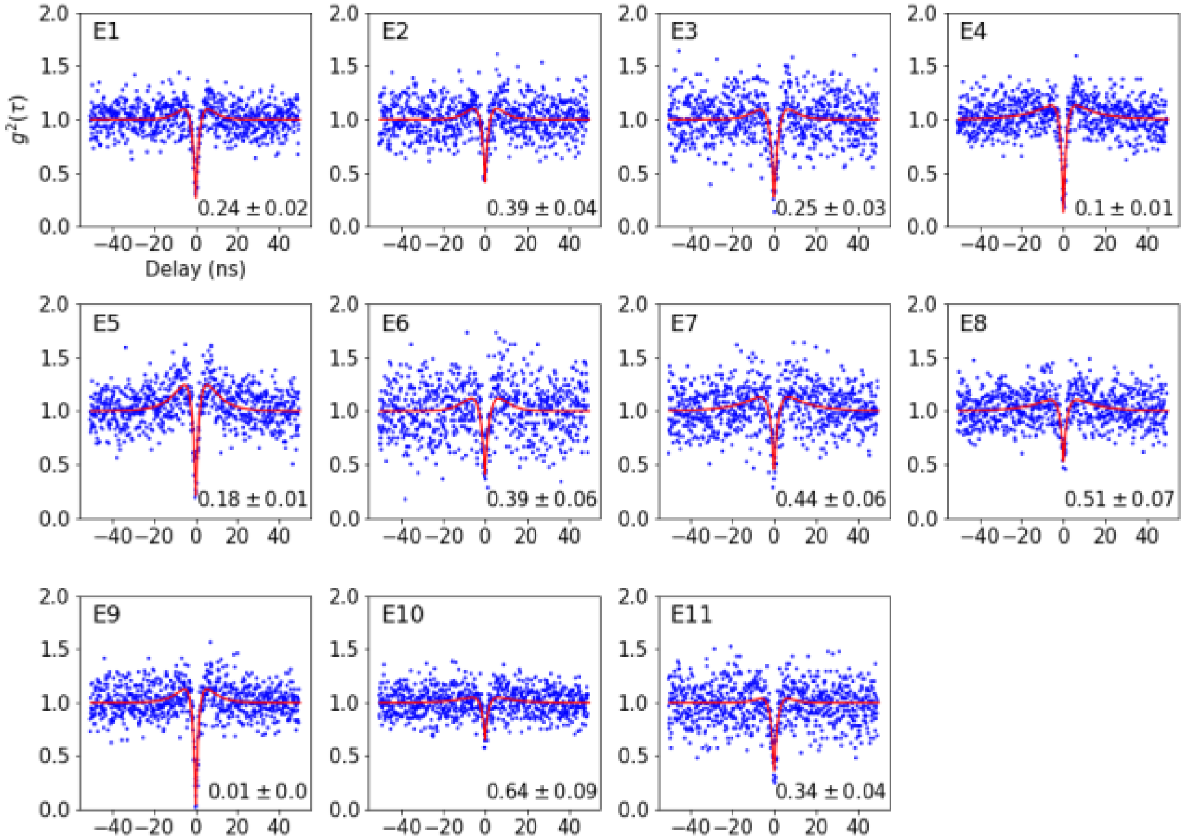
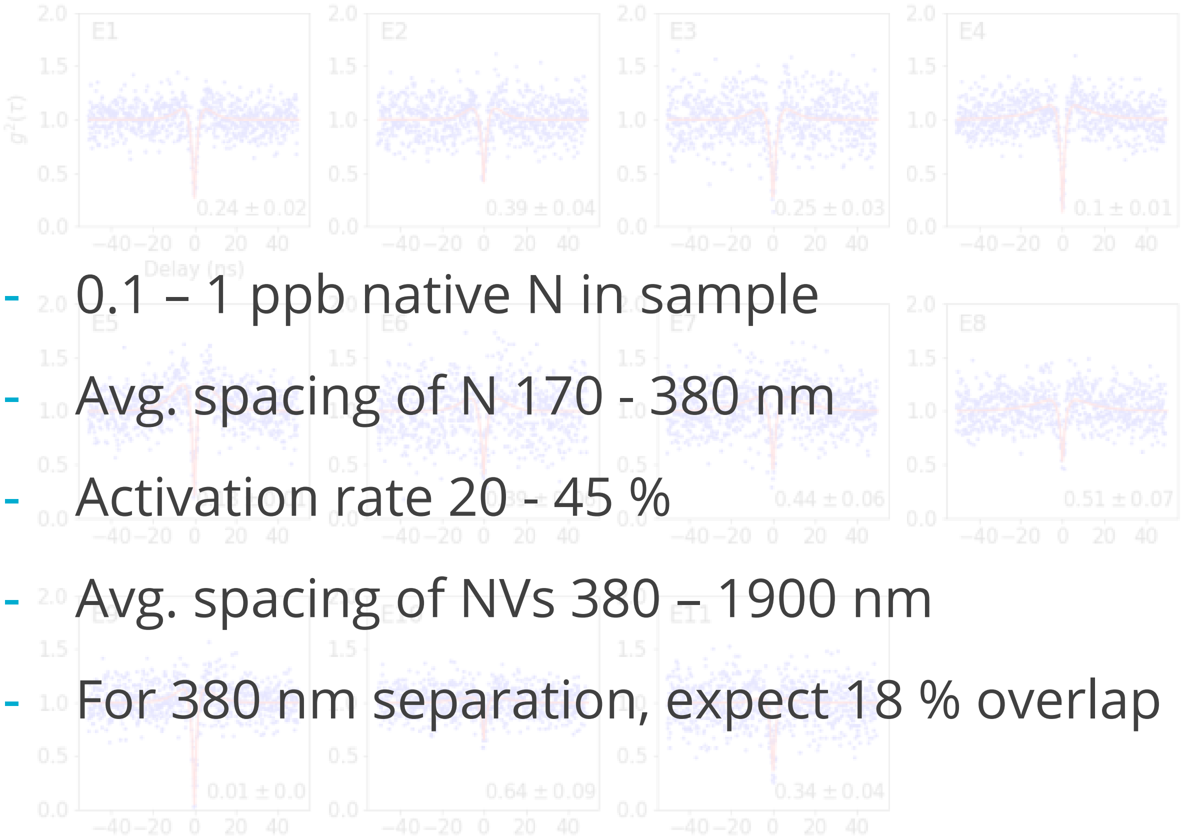




Not all locations identified as single emitters from PL are single emitters

Hanbury-Brown-Twiss

- 18 % of emitters classified as single emitters based on their PL are not single emitters
- Possible explanation is overlap between SiV and NV
  - NV would be activated during high-temperature activation anneal for forming SiV

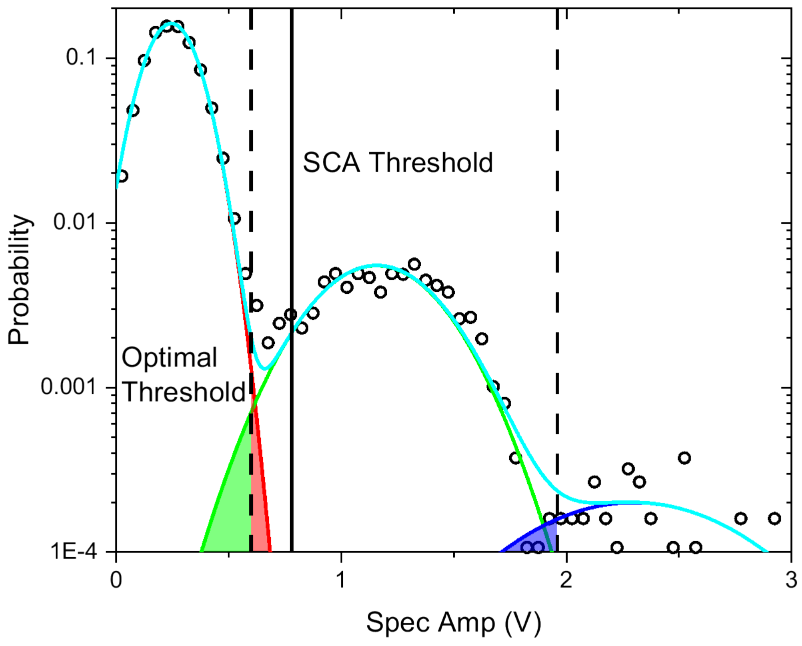


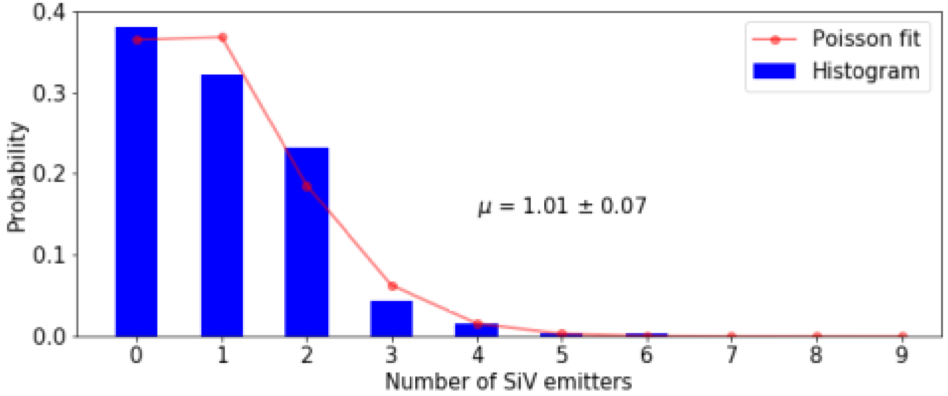
- 0.1 – 1 ppb native N in sample
- Avg. spacing of N 170 - 380 nm
- Activation rate 20 - 45 %
- Avg. spacing of NVs 380 – 1900 nm
- For 380 nm separation, expect 18 % overlap

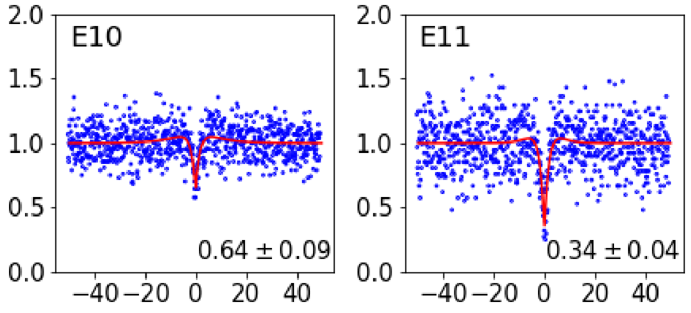
Not all locations identified as single emitters from PL are single emitters

Conclusion of In-Situ Counting

- In-situ single ion counting can improve confidence on number of ions to 5 %
- For implantation of 30 ions / <1> SiV, 7X improvement over timed implantation
  - For implantation of fewer ions, improvement will be even greater
- PL confirms we implanted <1> SiV
- HBT shows that 18 % of locations classified as single emitters based on PL are not single emitters
  - Likely due to natively occurring N being converted to NV during high-temperature annealing







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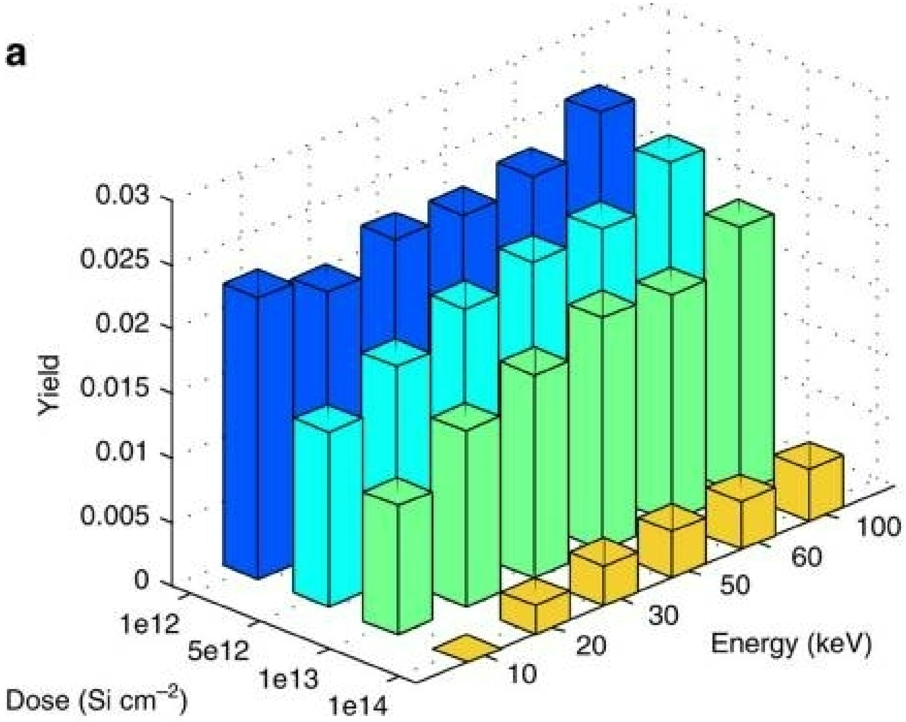
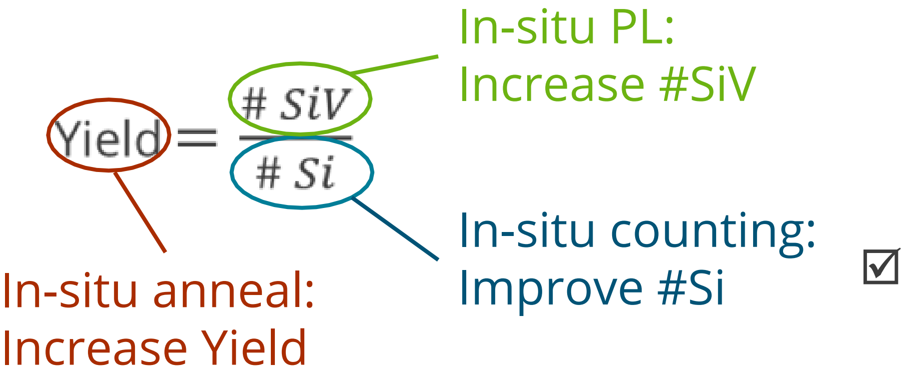
Issue: Poisson Statistics for low #s



T. Schröder et al., Nat. Commun., 8, 15376 (2017)



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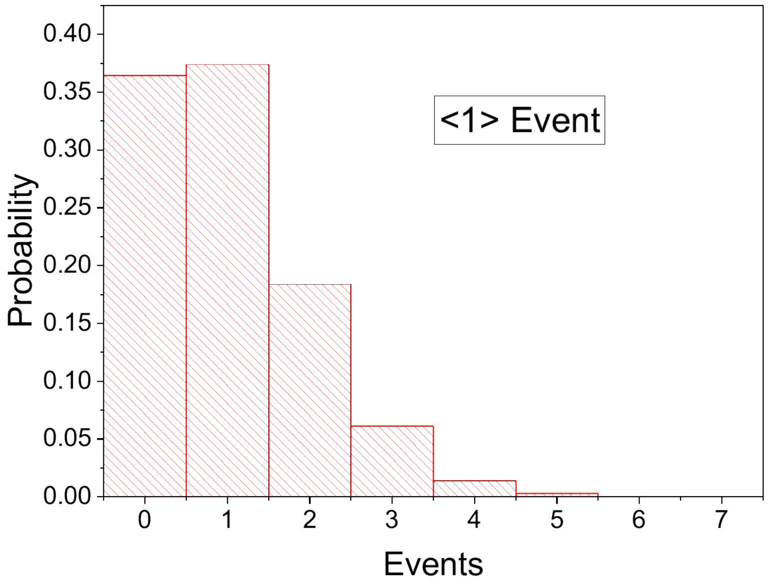
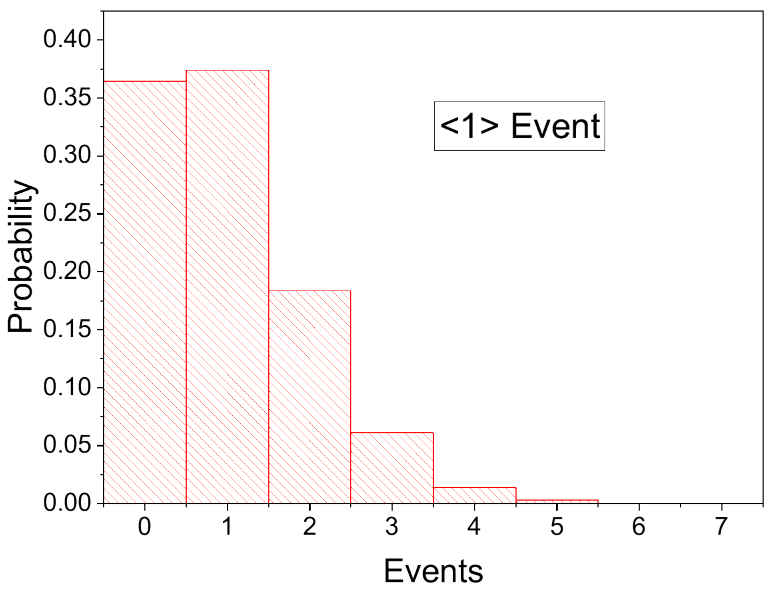
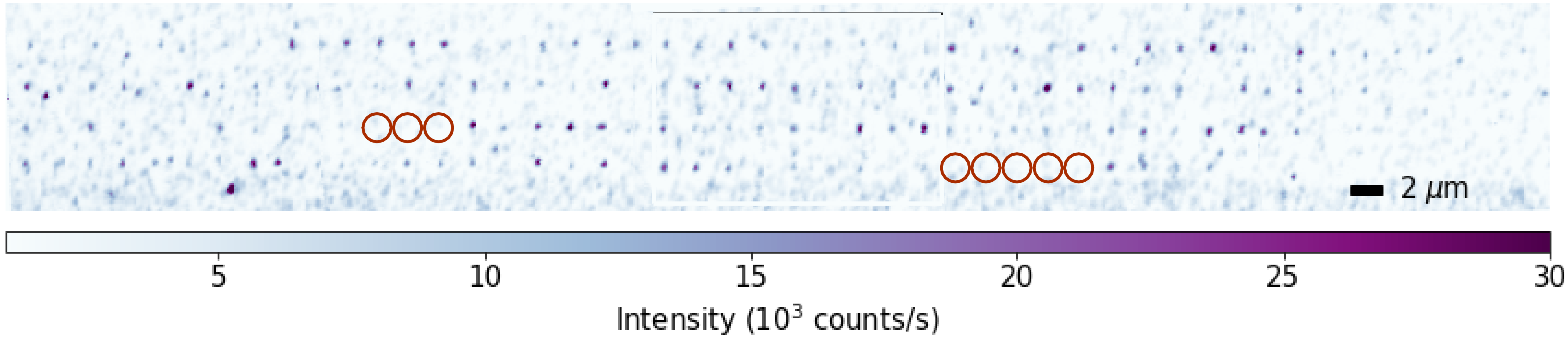


Improvements - In-Situ Photoluminescence

- Low conversion yield from implanted ions to optically active emitters requires in-situ feedback
- Array of <1> emitters will have 40 % no emitters and 20 % double or more emitters, only 40 % “useful” sites
- Photoluminescence is a way to confirm emitters are created
  - Caveat: Not necessarily singles





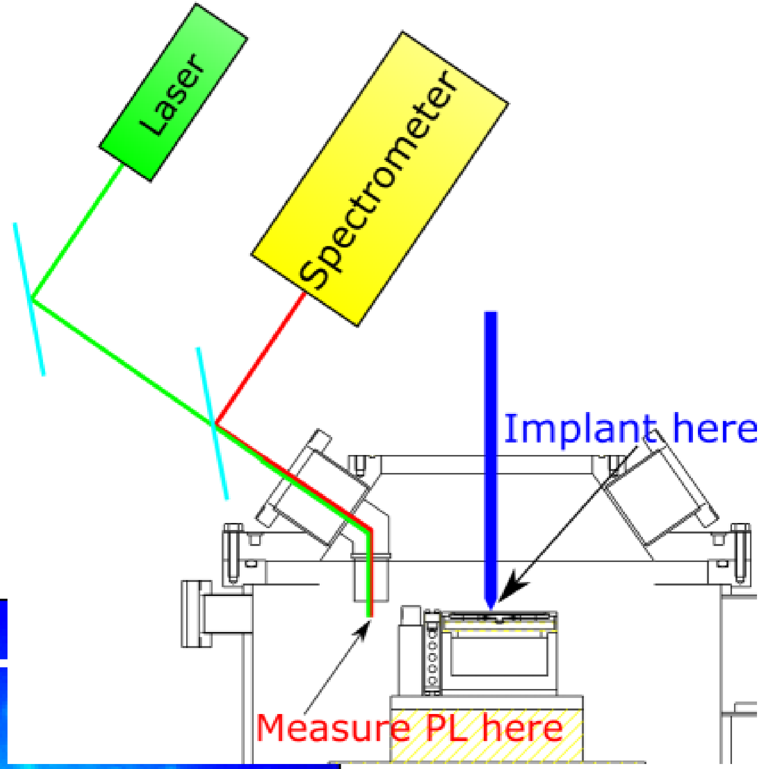
In-Situ Photoluminescence - Experiment

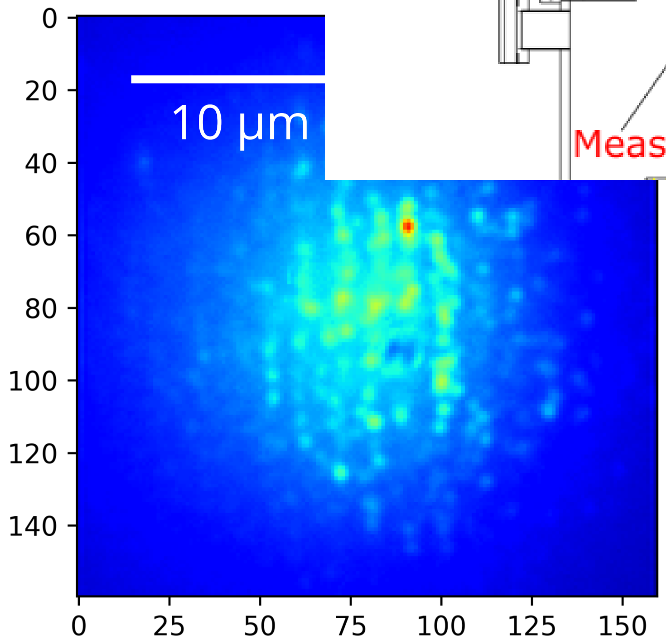
V. Chandrasekaran

- V_{Si} in SiC has ~10 % conversion yield from implanted Li to optically active emitter [1]
- V_{Si} does not require high-temperature annealing to activate

In-situ method:

1. Measure background PL





[1]: S. P. Pavunny et al., Sci. Rep. 11:3561 (2021)

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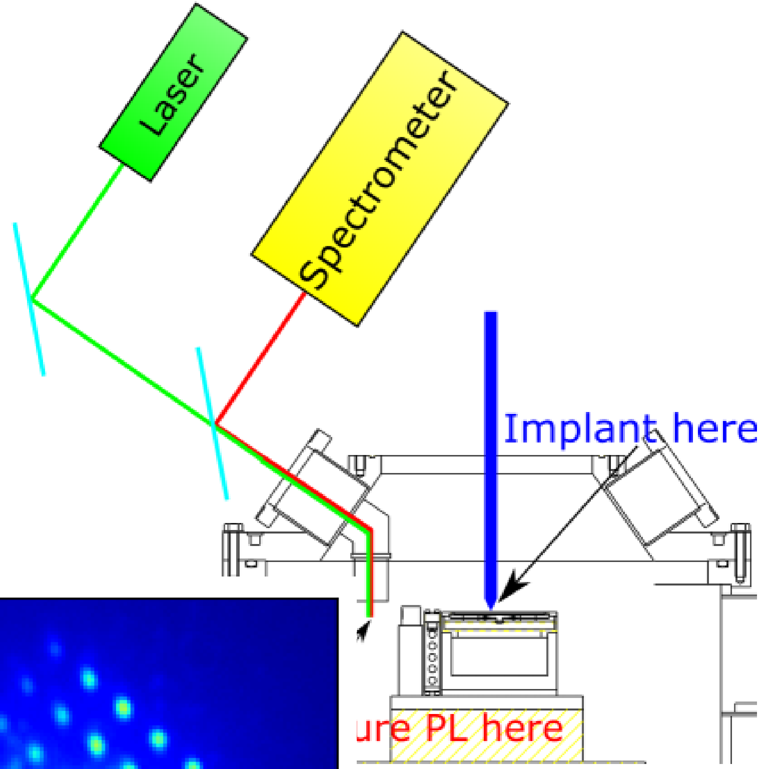

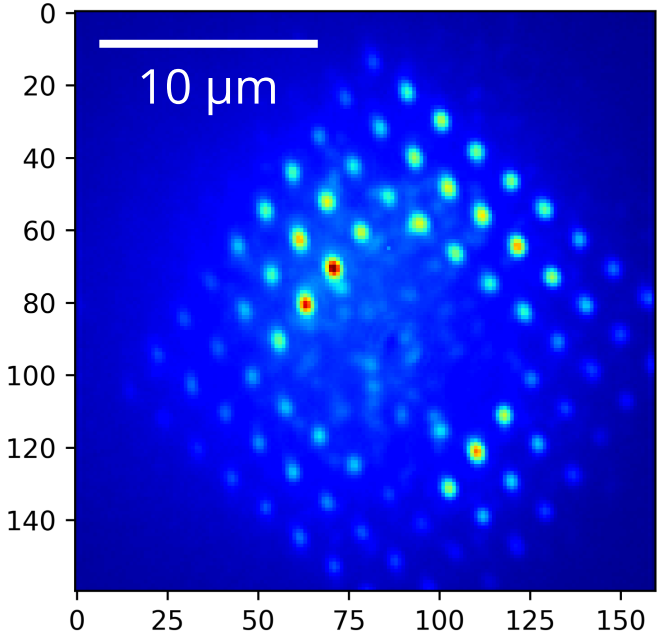

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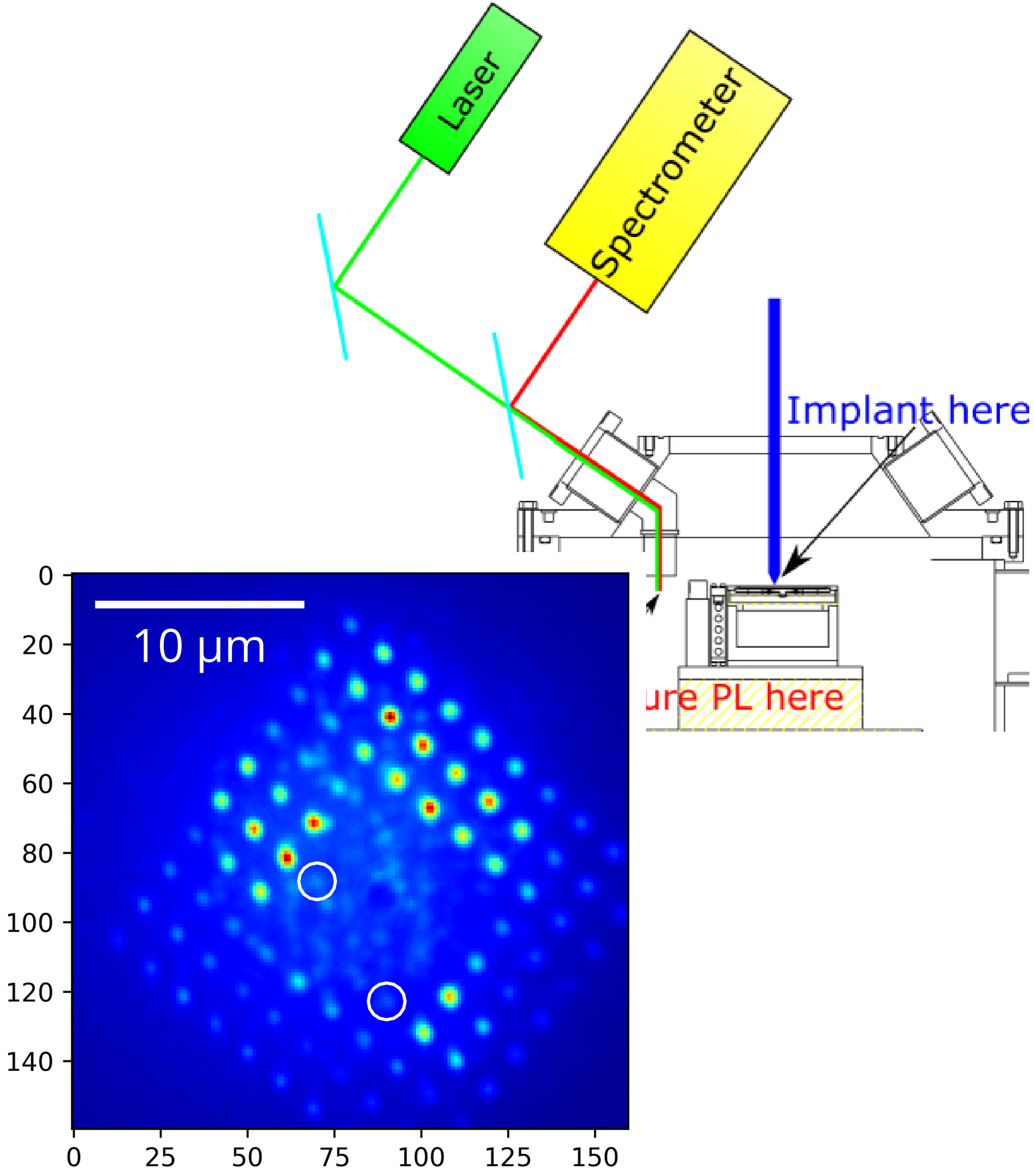

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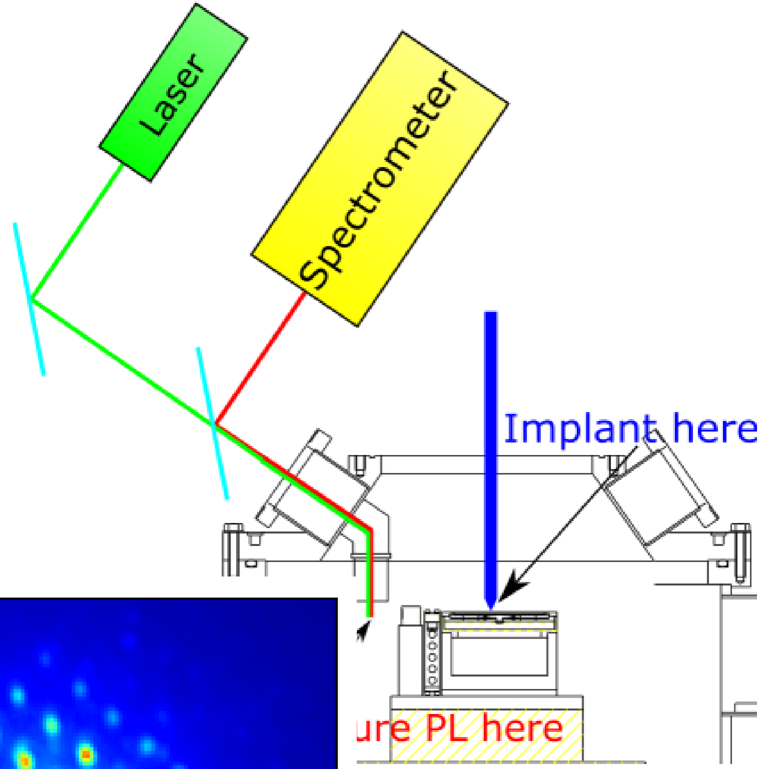
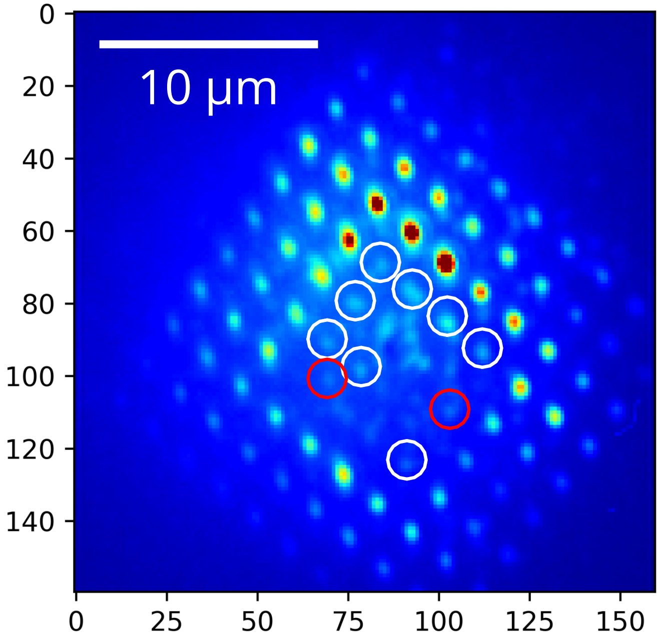




Preliminary work

[1]: S. P. Pavunny et al., Sci. Rep. 11:3561 (2021)

Acknowledgements

Anthony Flores, George Burns, Aaron Katzenmeyer, Barney Doyle, Ed Bielejec

All Projects — Membranes — N Source — All Projects

Tom Harris, Deanna Lopez

Diamond — Si, SiC, GaN, AlN

Andrew Mounce, Heejun Byeon, Jacob Henshaw, Luca Basso, Maziar Ziabari, Mike Lilly

N Source + Counting — Membranes — Hot Implant

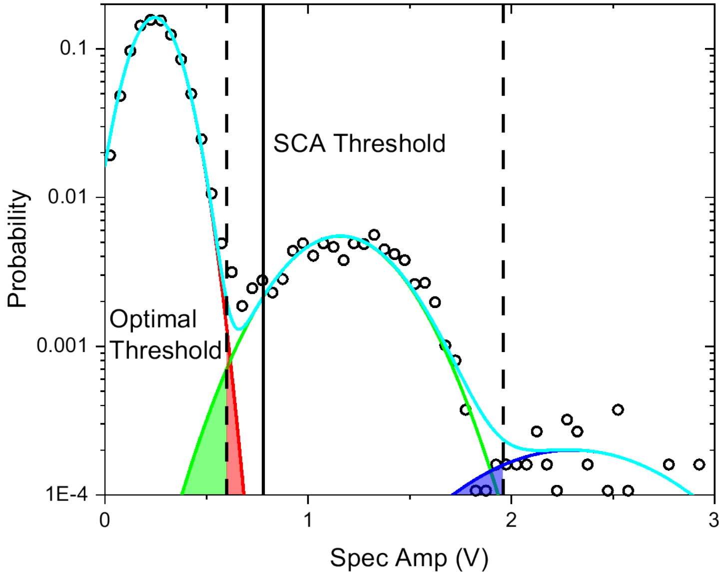
Vigneshwaran Chandrasekaran (LANL), Han Htoon (LANL), Yongqiang Wang (LANL)

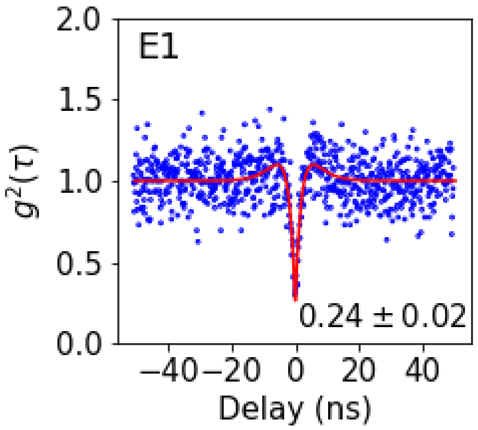
In-Situ PL — N Source

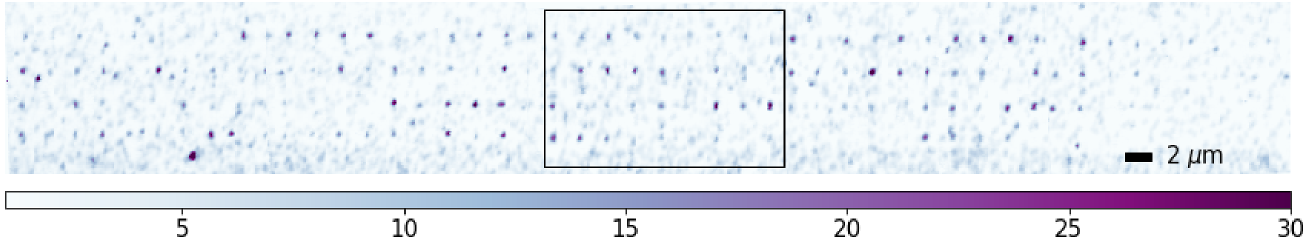
IBL, Fab, Optics

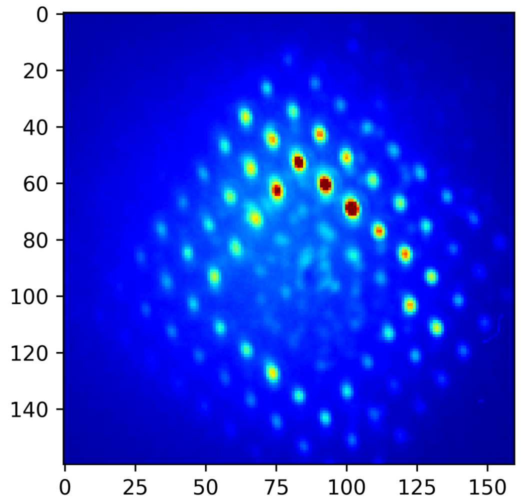
Summary

- In-situ ion counting improves confidence in ion number
  - Crucial for high-yield emitter fabrication
  - Better understanding of the yield for low number of ions
- In-situ photoluminescence allows fully deterministic creation of emitters
  - Caveat: At the moment, no high-temperature annealing, only vacancy-type defects
  - So far only made arrays, not yet confirmed single emitters









N Source - Improvements

- Higher beam current
  - Thinner foil implanted to the same level gives higher relative concentration
  - Fabrication in higher pressure N atmosphere may lead to more N absorption during melting
- Detection fidelity
  - N^{++} creates about 3.5x more e-h-pairs → Better separation between 0 and 1 peak in histogram
- Spot size
  - Currently align using Sn^{++} beam → $\Delta v/v > 50\%$, large error in aperture positions
  - Instead align to N while monitoring IBIC signal

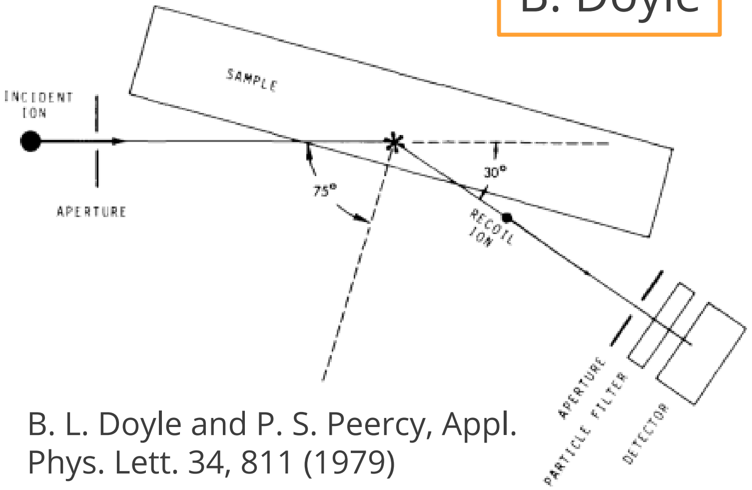
Motivation

- Quantum Devices require nm-scale positioning
  - Exchange Coupling: 10s of nm
  - Optical Cavities: 10s of nm
    - FIB has 10s of nm spot size, ideally suited
- Tailoring of quantum properties requires implantation of different ion species
  - ExB FIB with LMAIS
    - N Source
- To get quantum properties in a device, need 1 color center / donor / etc.
  - Poisson statistics kick in
    - Counted implantation
    - In-situ PL
  - Ways to improve yield
    - Hot Implants
    - In-situ Annealing

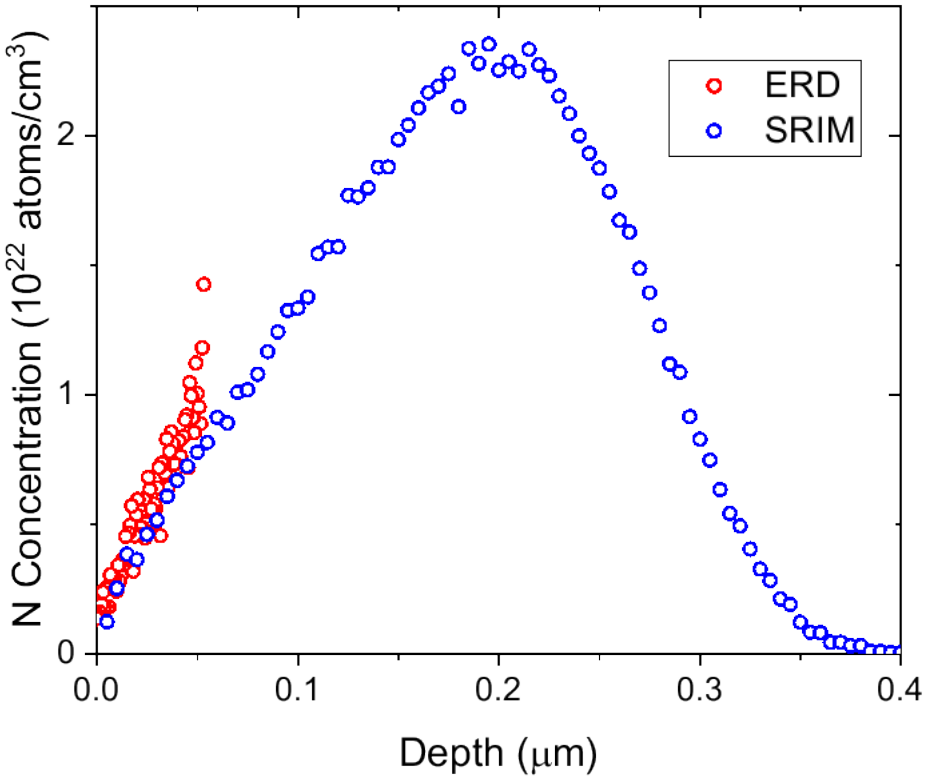
N Source - Implantation + ERD

Y. Wang
B. Doyle

- Implanted a $Au_{80}Sn_{20}$ 200 μm thick foil with N up to $5x10^{17}$ ions/cm^2
  - Level based on solid-solubility limit for H
- ERD: 50 nm of foil as expected
  - Cannot interrogate deeper due to multiple-scattering of deep ions
- From N concentration expect 3k N ions/s at 1 pA total beam current



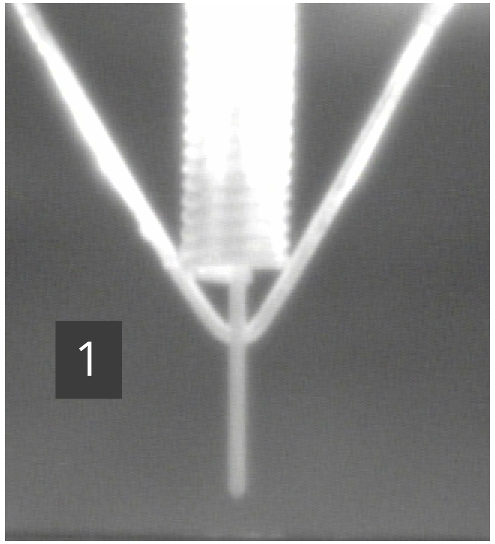
B. L. Doyle and P. S. Peercy, Appl. Phys. Lett. 34, 811 (1979)



N Source - Tip Fabrication

A. Katzenmeyer

1. W tip is heated to remove surface contaminants

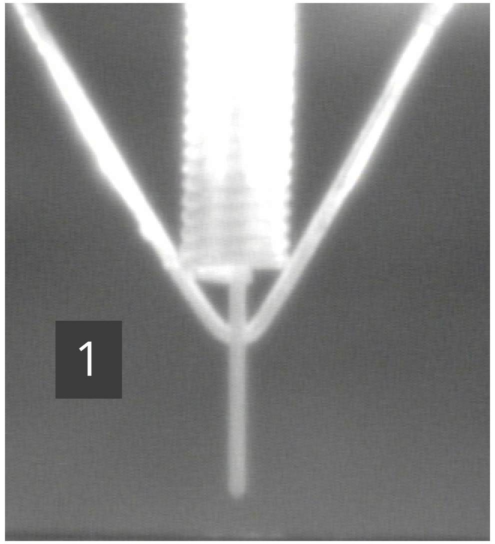


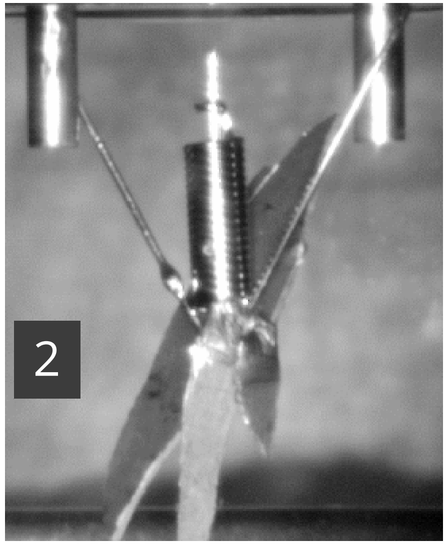
N Source - Tip Fabrication

A. Katzenmeyer

1. W tip is heated to remove surface contaminants

2. Foil is tack welded to the tip

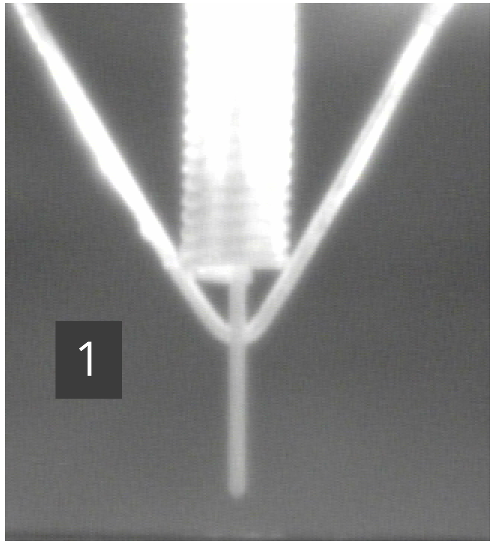


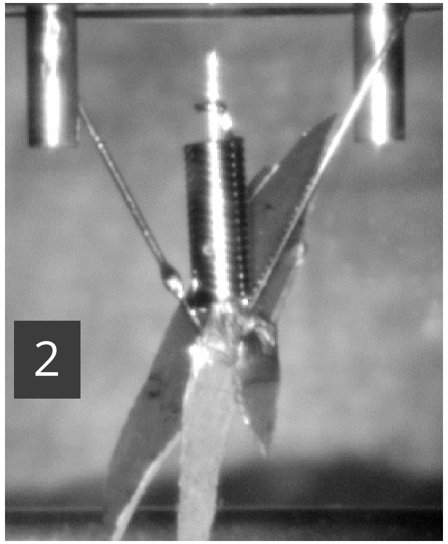


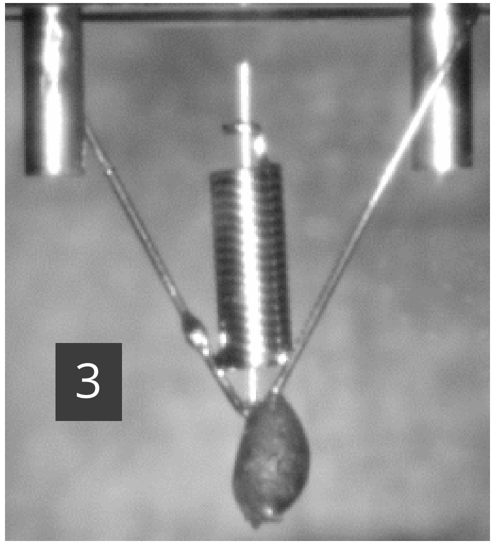
N Source - Tip Fabrication

A. Katzenmeyer

1. W tip is heated to remove surface contaminants
2. Foil is tack welded to the tip
3. Tip is heated to melt foil onto tip



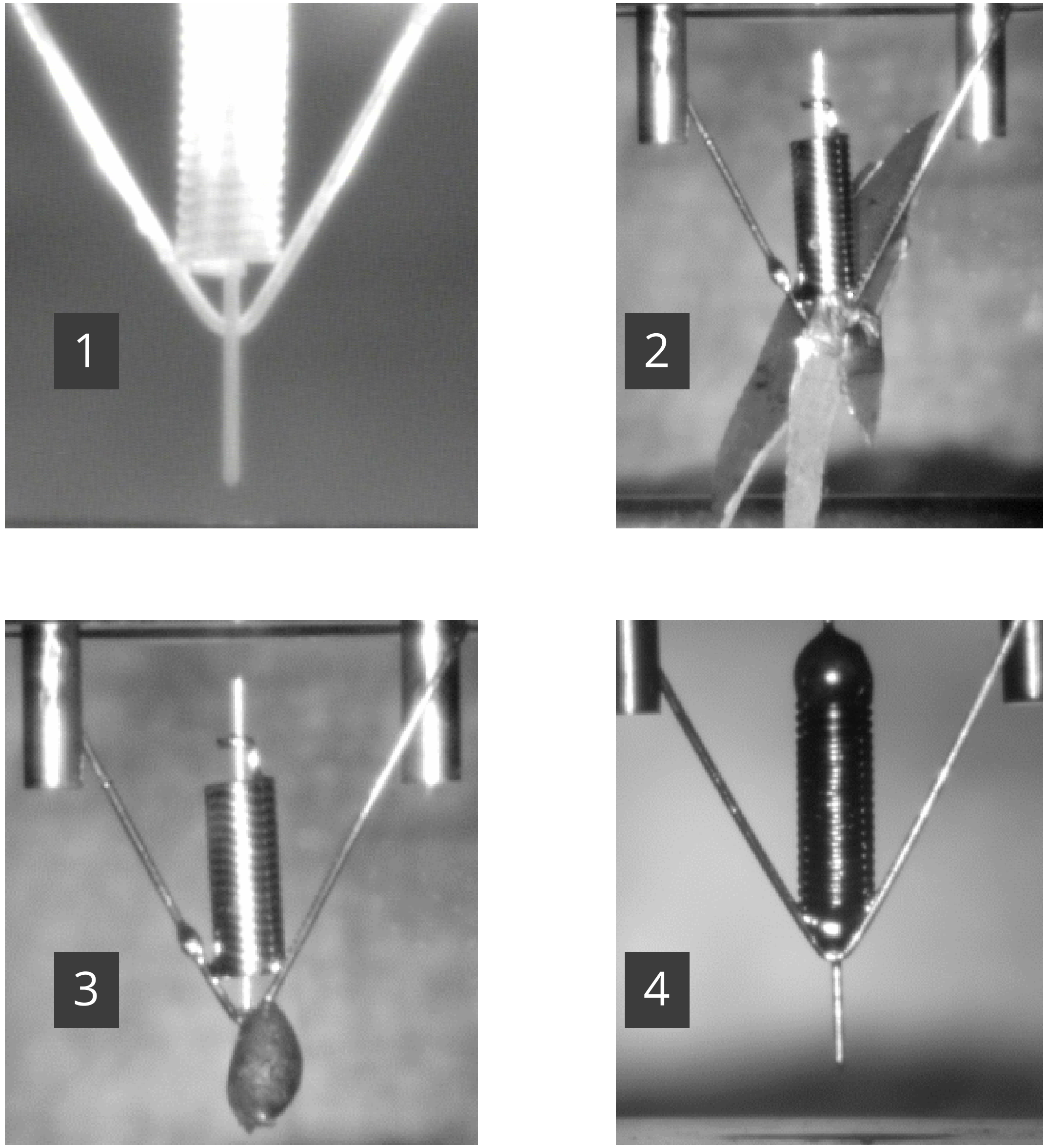




N Source - Tip Fabrication

A. Katzenmeyer

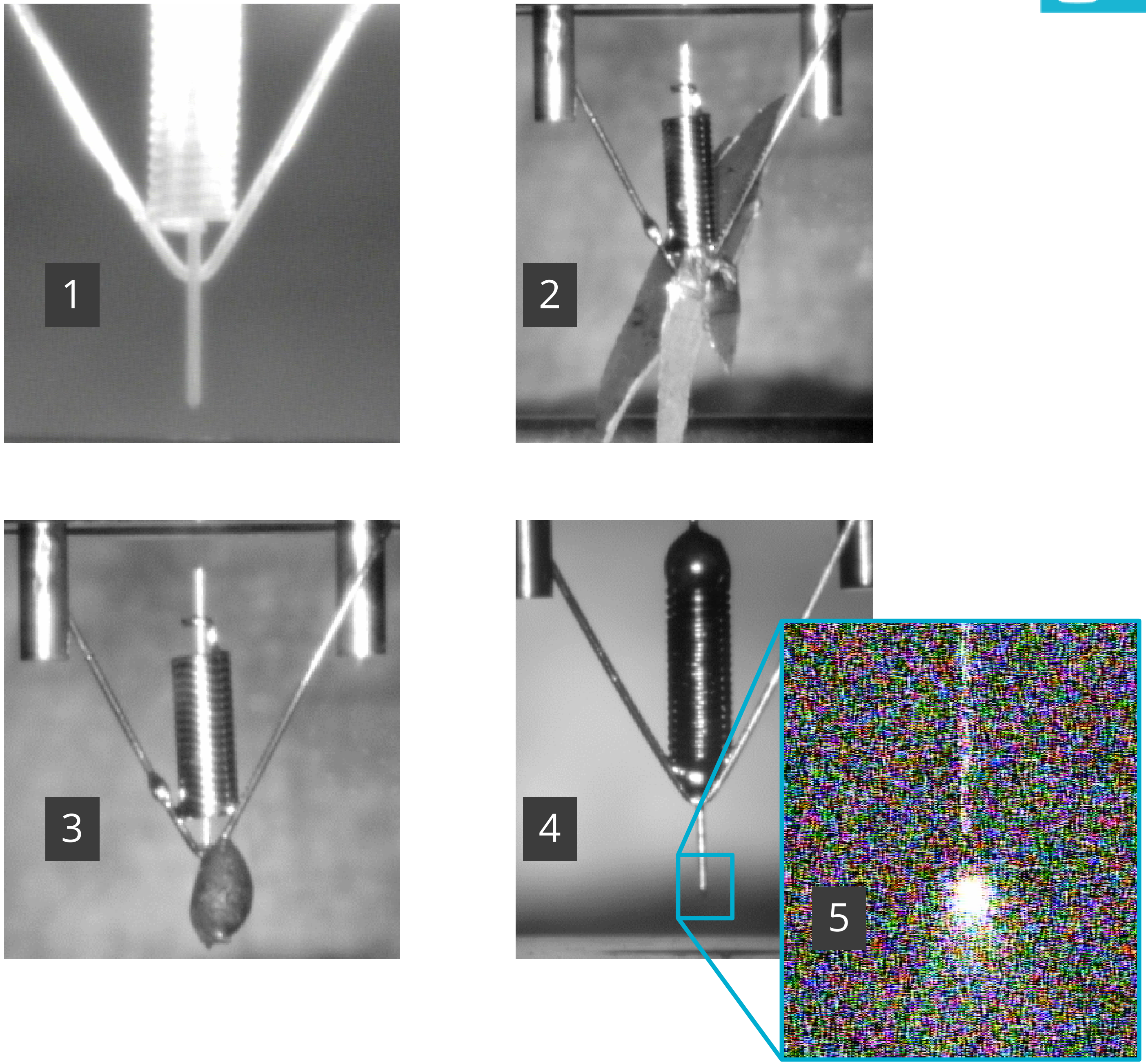
1. W tip is heated to remove surface contaminants
2. Foil is tack welded to the tip
3. Tip is heated to melt foil onto tip
4. Repeat steps 2. + 3. until tip + parts of reservoir is filled



N Source - Tip Fabrication

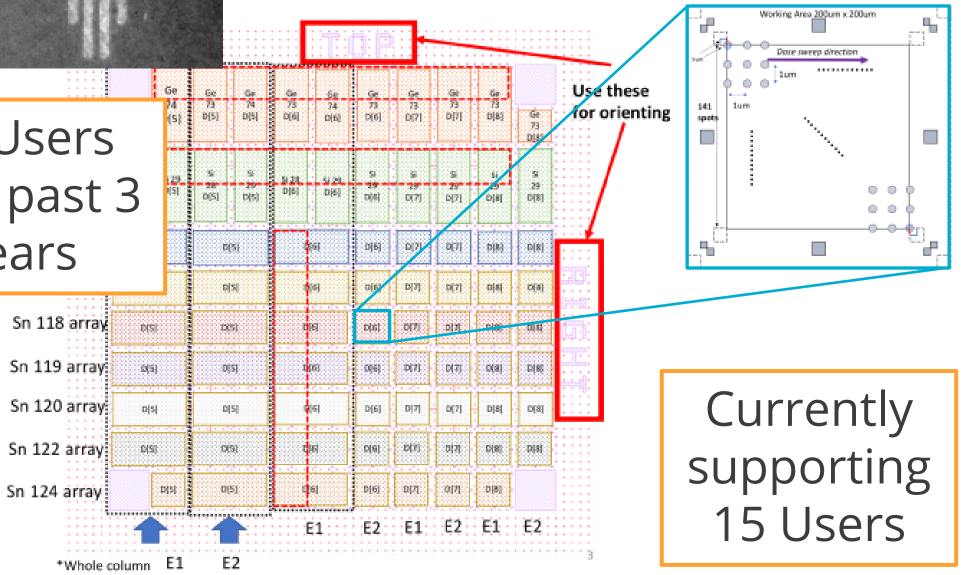
A. Katzenmeyer

1. W tip is heated to remove surface contaminants
2. Foil is tack welded to the tip
3. Tip is heated to melt foil onto tip
4. Repeat steps 2. + 3. until tip + parts of reservoir is filled
5. Test tip emits
6. Install tip into nanoImplanter



User Support

25 Users over past 3 years



Currently supporting 15 Users

LMAIS Source Development

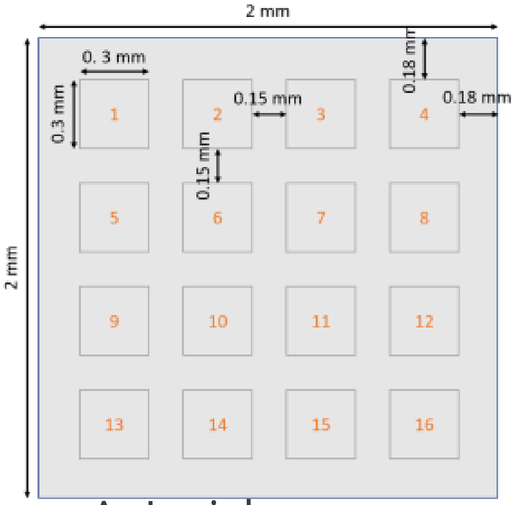
In-Situ Counting + Photoluminescence

User Support

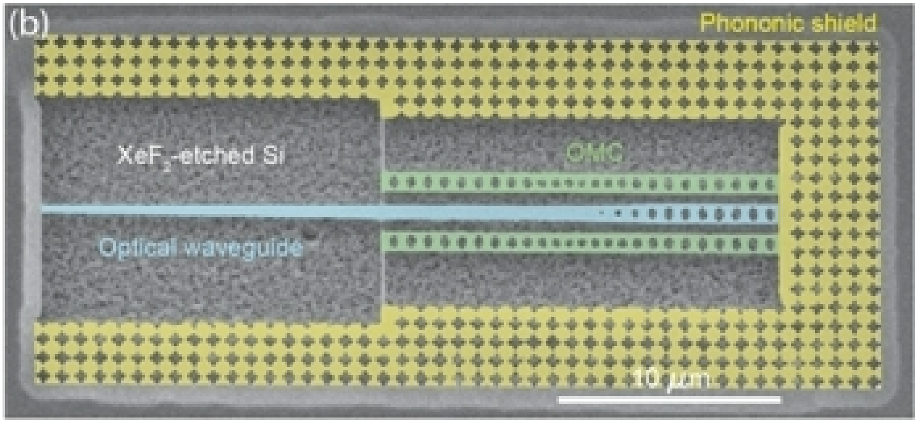
Localized Smartcuts for Diamond Membranes

HVEE Nanobeamline

Rapid prototyping enabled by focused ion beams
- Different Energy / Dose in each area



A. Jayich @ UCSB



J. V. Cady et al., Quantum Sci. Technol., 4, 2 (2018)

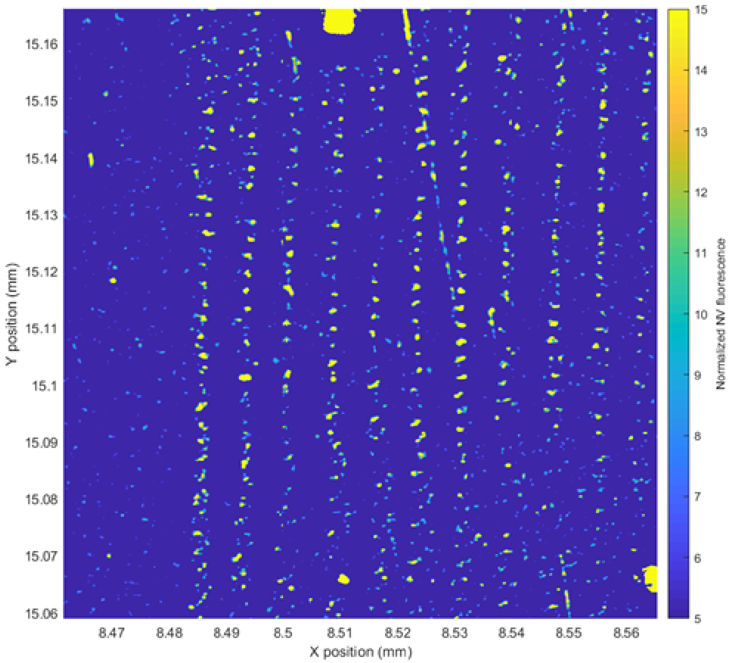
Local Damage to Simulate Dark Matter Strikes

Tandem MicroOne beamline

36 working areas over a 1x1mm^2 area
- Different Energy / Dose at each area

1x1 μm^2 beamspot for localized damage events

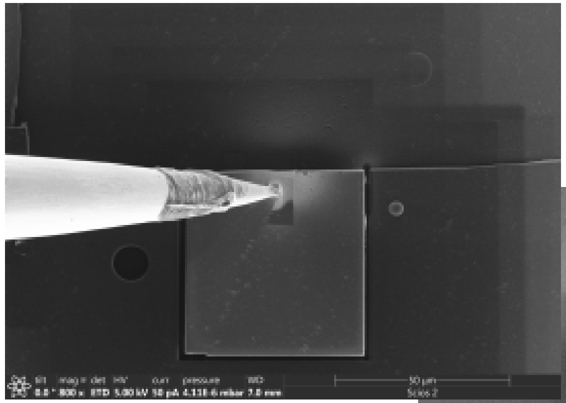
R. Walsworth @ U Maryland



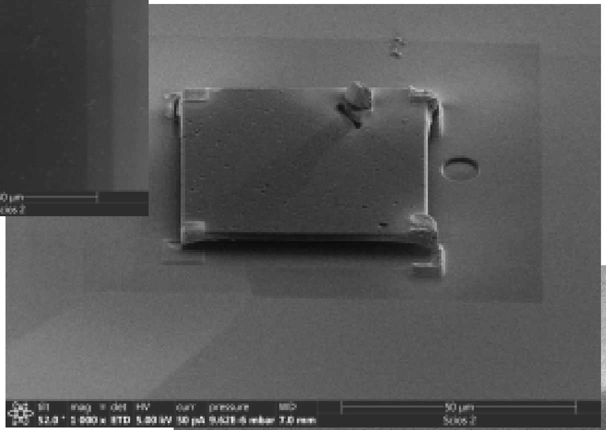
User Support

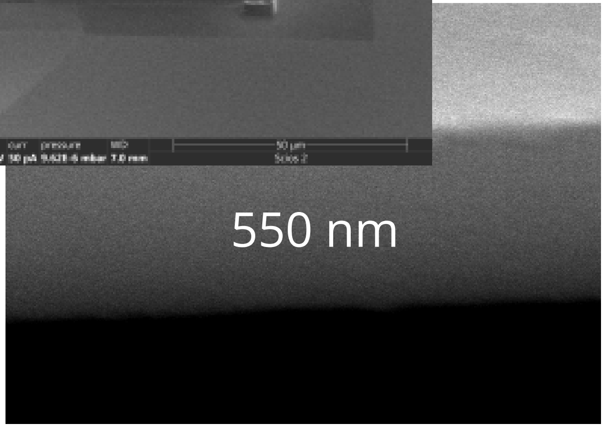
Membrane Formation for High-Sensitivity Sensors

Pelletron implant beamline



L. Basso
@ CINT



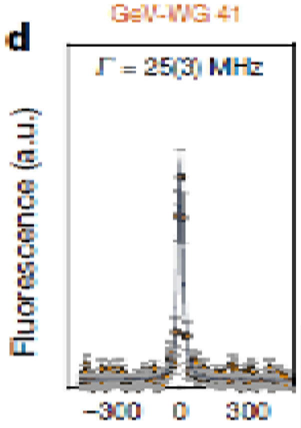


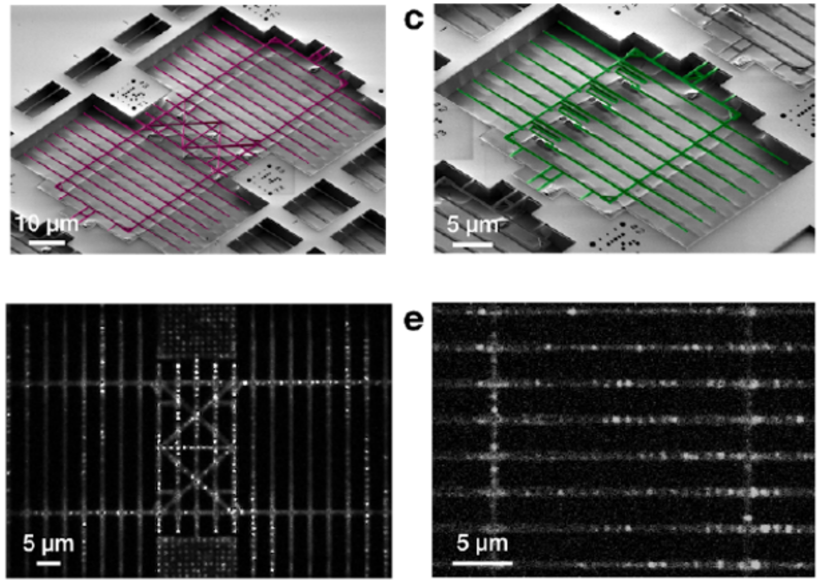
M. Loncar
@ Harvard

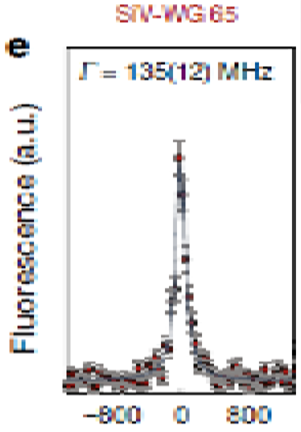
A. Mounce
@ CINT

Nanoimplantation for Diamond-Based Photonics

NanoImplanter for <50 nm spot @ 200 keV





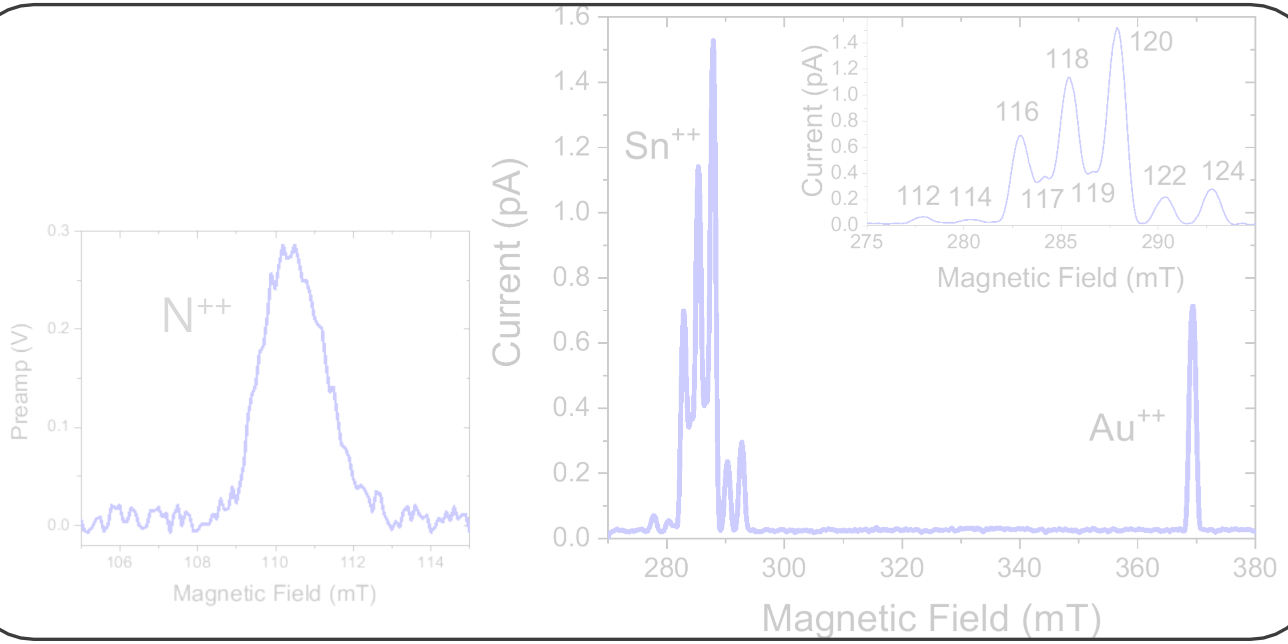


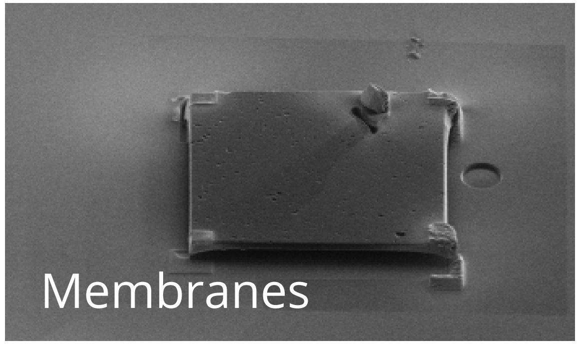
MIT measuring 2 additional samples with Si, Ge, **Sn**

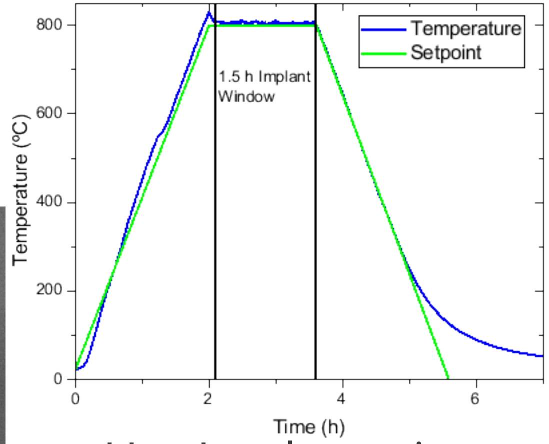
D. Englund
@ MIT

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

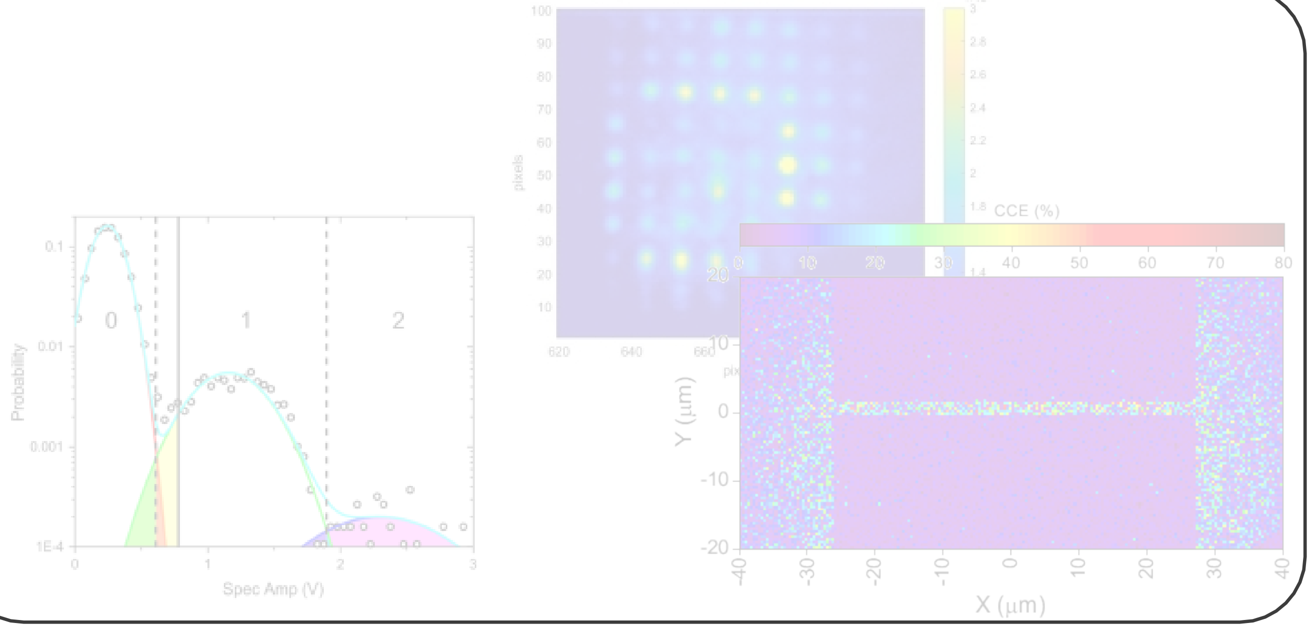
Improving Diamond NV Sensors







Hot Implantation



Hot Implantation for Improved NV Formation

- NV creation is mostly vacancy-limited

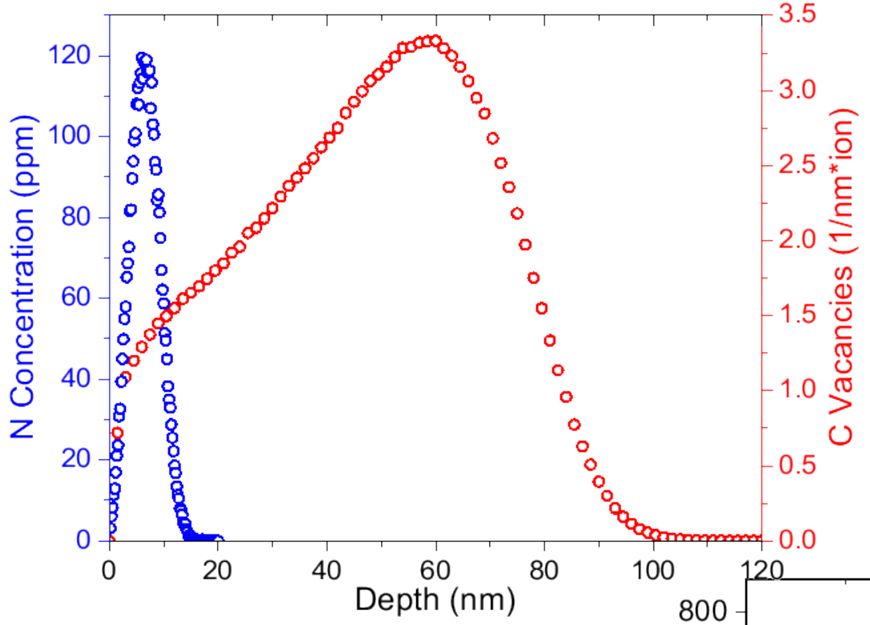
Solution:

1. Implant 46 keV C into diamond

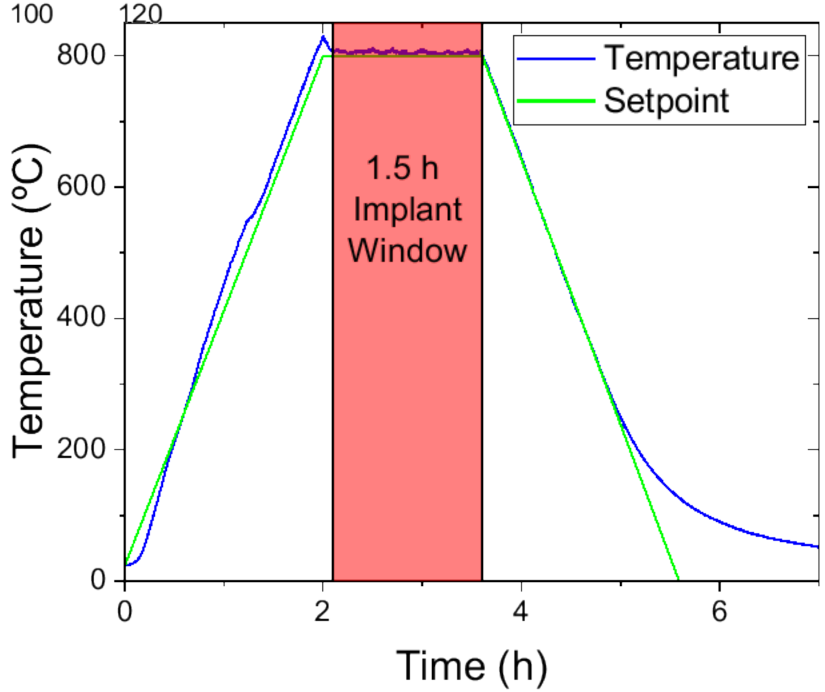
   → Create vacancies

2. Heat sample during implant

   → NV activation anneal



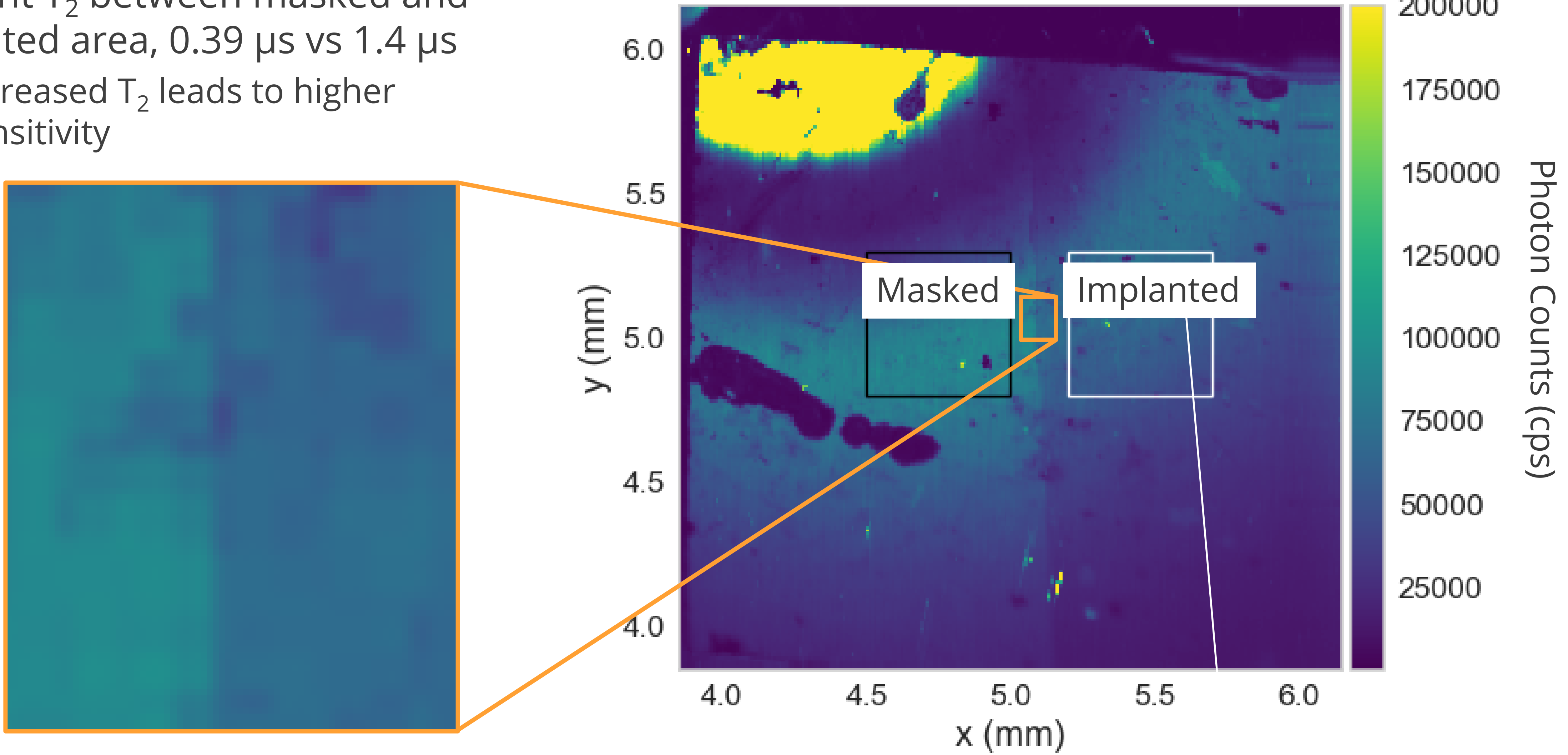
Ramp rate not to exceed 400 °C/h



Results for Hot Implanted Diamonds

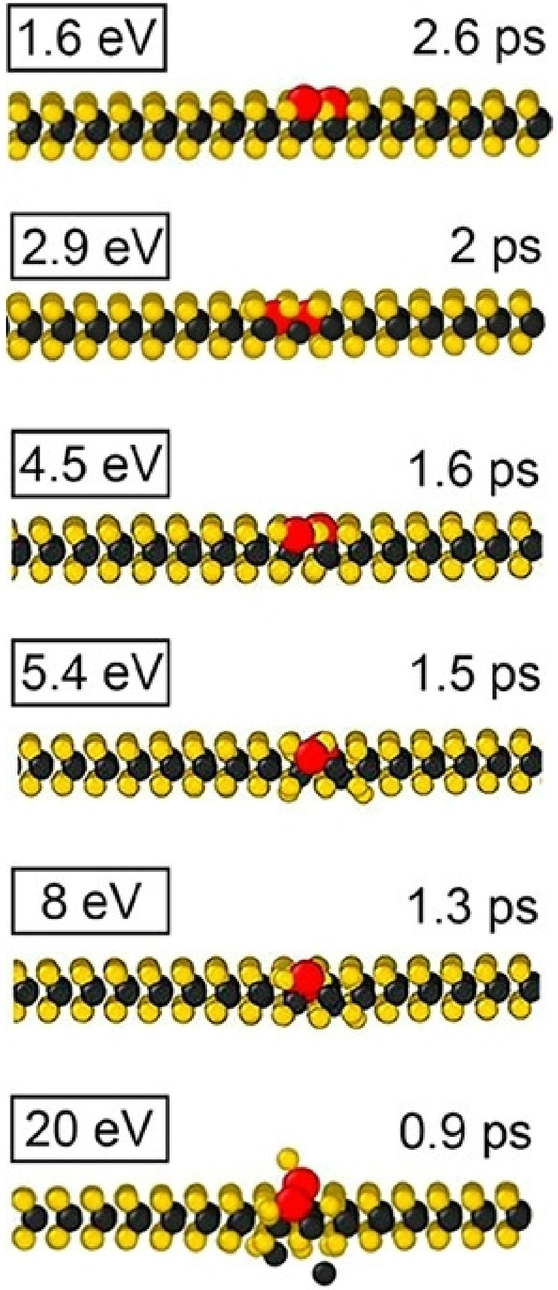
M. Ziabari
M. Lilly

- Very preliminary results, but show different T_2 between masked and implanted area, 0.39 μs vs 1.4 μs
  - Increased T_2 leads to higher sensitivity



Impurity Doping in 2D Materials

- To implant 2D materials require ion stopping within atomic monolayers
  - 10s to 100s of eV ion energy for deterministic stopping in atomic monolayers
- New VELION can implant < 5 keV @ <100 nm spot size

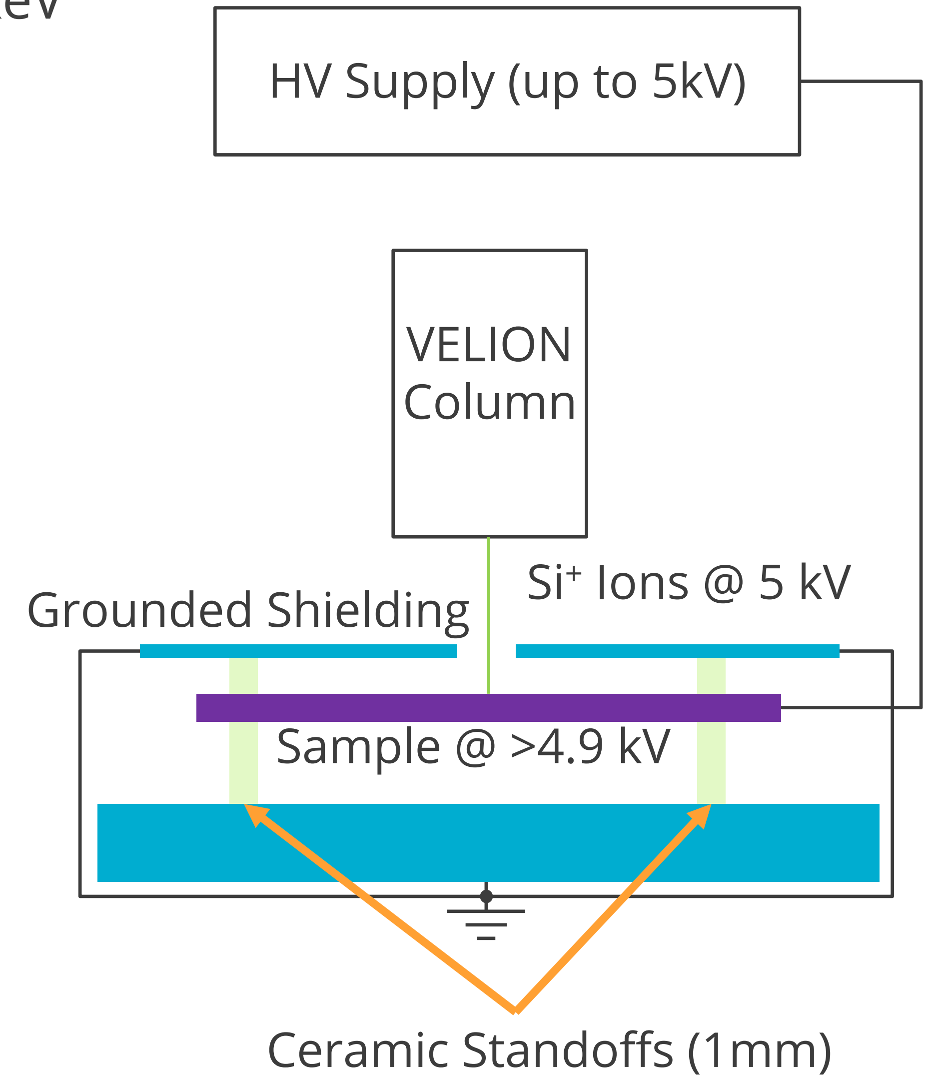


Lin et al., ACS Nano,14, 4, 3896–3906 (2020)

Ultra-Low Energy Implantation

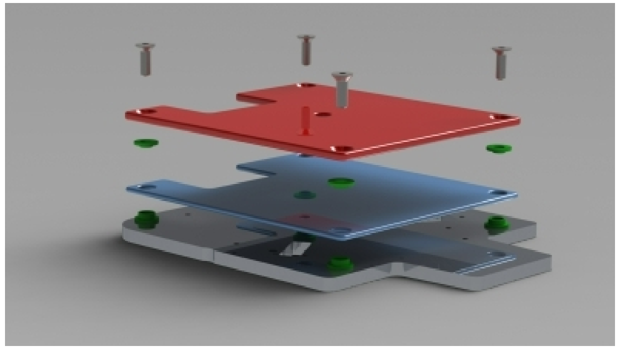
- New VELION can implant < 5 keV @ <100 nm spot size
  - Bias sample to reduce ion landing energy to eV range
- Caveat: What will be our spot size?
  → SIMION simulations
  - Few μm: Useful for yield measurement
  - < 1 μm: Can be useful for photonics

Currently pursuing through CNMS user project with A. Belianinov



FY 22 LDRD Idea, PI

BES infrastructure



Future Directions

CINT

Actively reaching out to engage new users

11 high-profile publications + 4 under review over past 3 years

LMAIS Source Development

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

Atomic N source for FIB applications (in preparation)

Improving Diamond NV Sensors

P. Kehayias, J. Henshaw, M. Ziabari, M. Titze et al., NIM B, 500, 52 (2021)

P. Kehayias, E.V. Levine, L. Basso, J. Henshaw, M. Titze et al., "Simulation and Measurement of the Magnetic Fields from a 555 Timer Integrated Circuit using a Quantum Diamond Microscope and Finite Element Analysis" (in preparation)

A. Mounce: Quantum Sensing for Quantum Materials (BES Infrastructure, renewed)

In-Situ Counting + Photoluminescence

Towards Deterministic Creation of Single Photon Sources in Diamond using In-Situ Ion Counting (in preparation)

A. Katzenmeyer: In-situ laser annealing (FY22 LDRD Idea)

H. Htoon: Deterministic Placement and Integration of Quantum Defects (BES Infrastructure)