Plasma tools for QIS Fabrication





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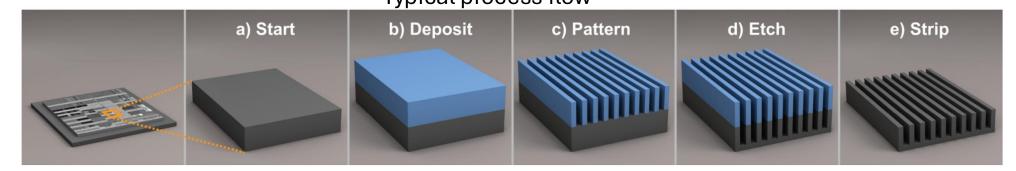


Overview

• How does process develop with plasma tools look like and flow in a smaller scale R&D facility like the ones found in universities.

 A couple of examples of plasma etching and processing of diamond for QIS.

To pattern a material from the top down. We must have masks! Typical process flow



J. Vac. Sci. Technol. A 38(3) May/Jun 2020; doi: 10.1116/1.5141863

First things to think about!

What are your critical dimension:

Horizontally and lateral.

Vertical etch depths and anisotropy or isotropy required.

What are your material compatibility issues:

With mask materials.

With chemicals for resist development or cleaning.

With etch tools (some tools have strict material limitations).

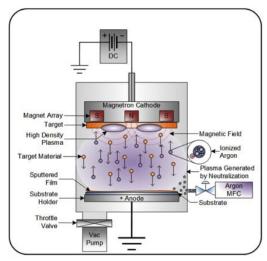
Plasma Cleaning wafer or substrates

A cylindrical chamber ("barrel") where the plasma is generated and reactive species diffuse to the sample, enabling non-directional (isotropic) etching

Great at removing organic contamination



Plasma tools for deposition of mask or device materials

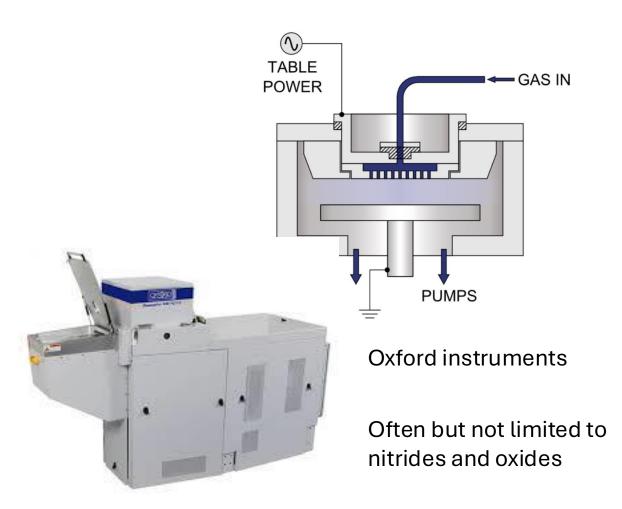


https://www.semicore.com/news/94-what-is-dc-sputtering



http://www.imajeenyus.com

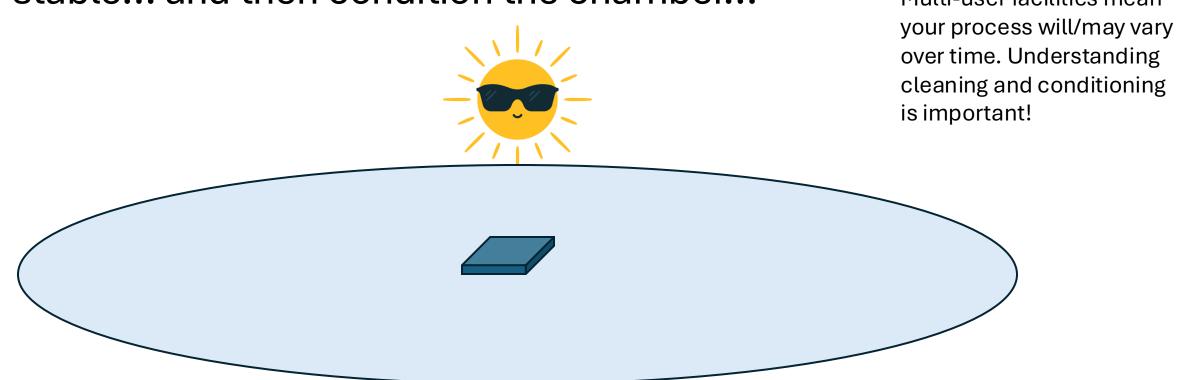
Magnetron Plasma used in sputtering systems



Plasma Enhanced Chemical Vapour Deposition PECVD

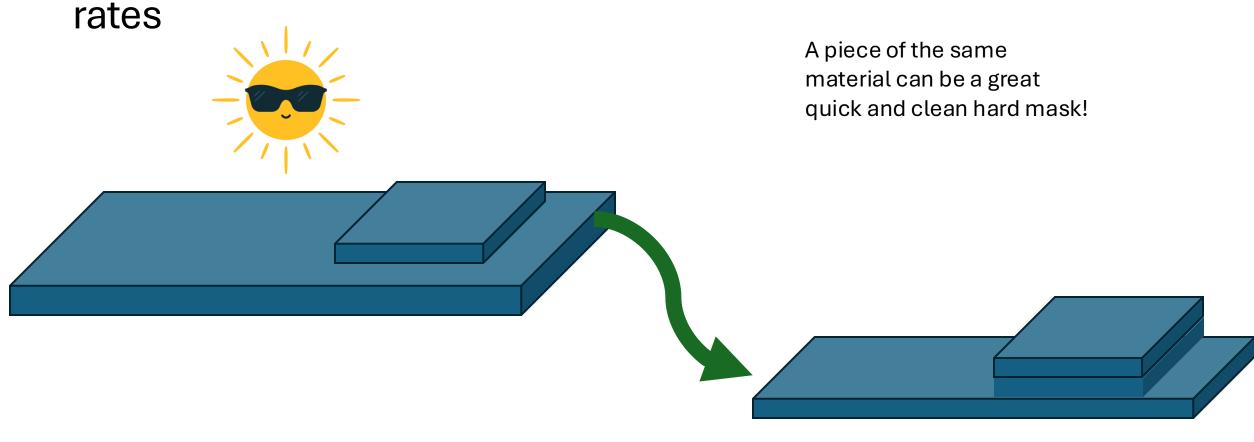
Now we want to do some plasma etching. You've done a literature review, or thought about appropriate chemistry and physicality...

Run some chamber cleaning. Does the plasma strike, is it stable... and then condition the chamber...



Now we want to etch. You've done a literature review, or thought about appropriate chemistry and physicality...

Shadow masks for quick rough test on chemistry and etch



Things we can check without making complicated masks:

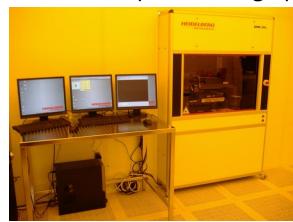
- Does it etch!!
- Is the etch relatively smooth
 - Could our plasma be making lots of polymer causing micromasking
 - This could be seen by eye (is it no longer shiny!) if really bad.
 - Was our substrate or chamber clean... has this caused lots of micro masking.
- What is the approximate etch rate
 - Approx. as etch rates may depend on
 - Feature size and pitch
 - Mask plasma and substrate chemistries, loading effects, edge effects and plasma uniformity

Photolithography and Electron beam lithography for lift off and or patterning of resist masks an when you need to be quick... Shadow masks.



larger than ~1µm

Hard Mask-UV photo-lithography



Direct write photo-lithography



Elionix ELS -F125



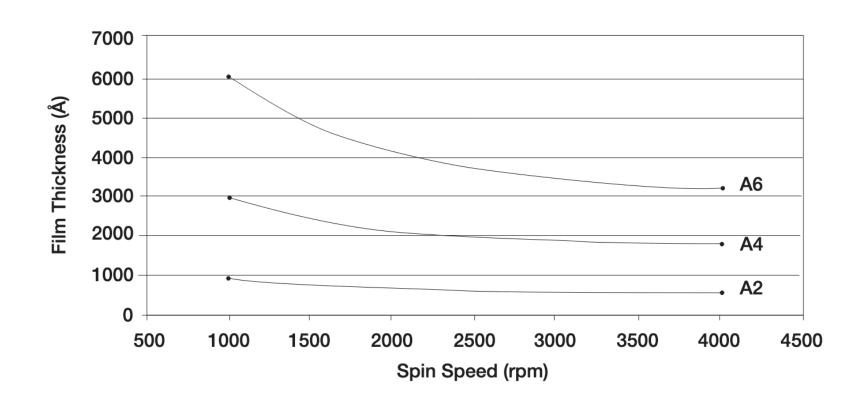
EBPG 5150 plus

10nm and upwards

Probably you'll next spin some resist.

495PMMA A Resists

Solids: 2% - 6% in Anisole



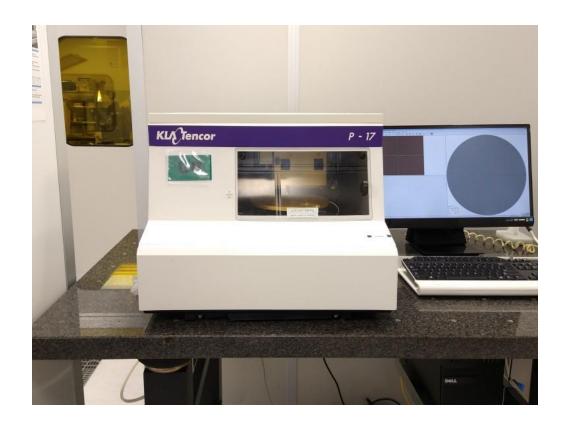
But is it really that thick and uniform on your weird little piece of material..?



spectroscopic reflectometry to measure the thickness and optical constants (n and k), wavelengths between 190 and 1100 nm.

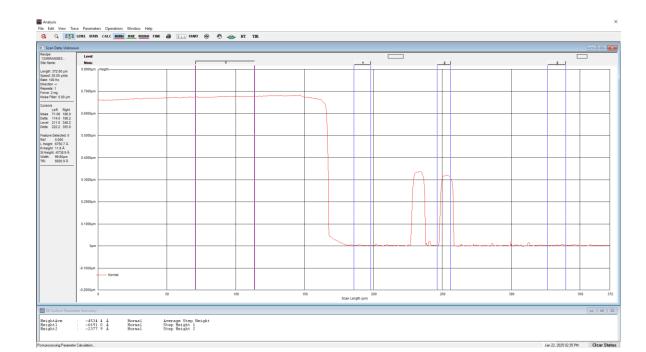
Or use **ellipsometry**

Or expose, develop and use a profiling tool



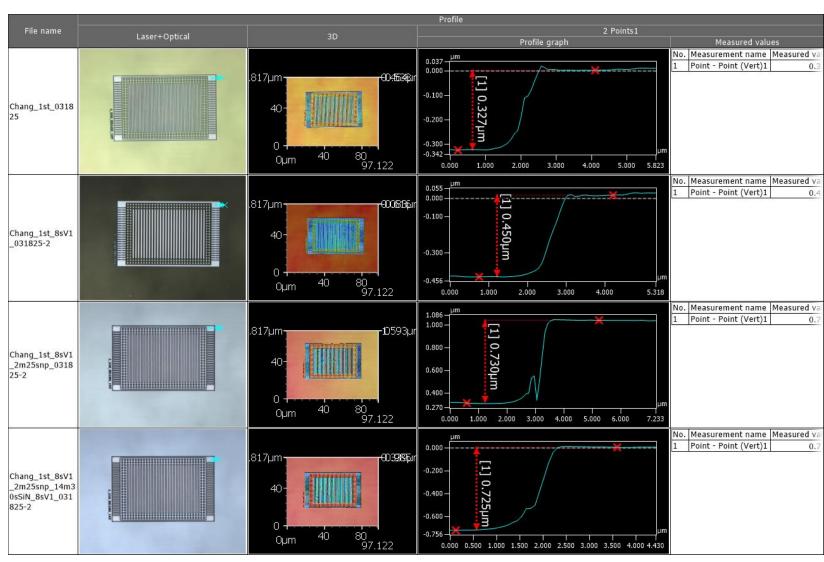
Step height: Nanometers to 1000µm

No time for optimising writing/exposure and development of resist optimisiation..!



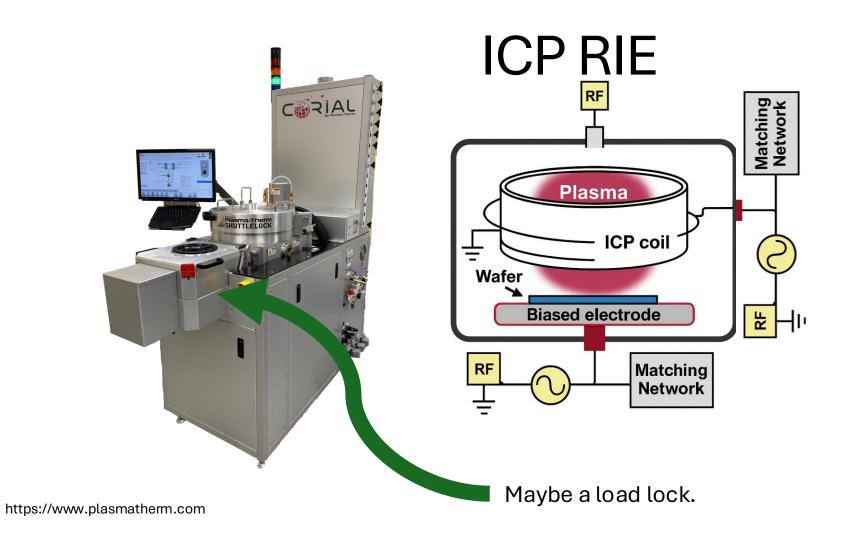
Or expose develop and use optical profilometer



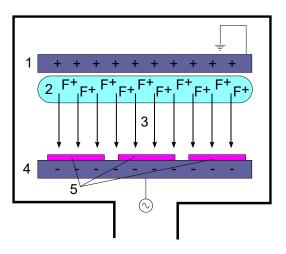


Do Metrology, do it often, and if possible, in different ways, and keep good notes/lab books.

You've made some sort of mask what next!



RIE



By Dollhous, modified by Adove1018 to show correct electric charges. - https://en.wikipedia.org/

How are you mounting your sample and to what sort of carrier wafer.

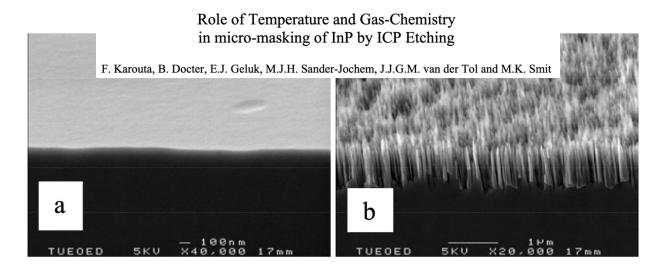


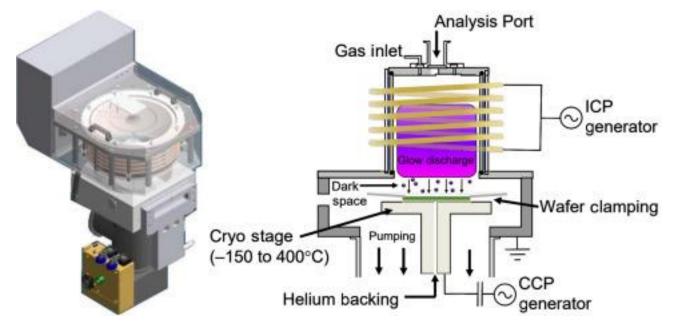
Figure 1- SEM photographs of etched s.i. InP samples with heat sink paste (a) and w/o (b).

Sapphire or Silicon carrier wafers are common. Will the carrier interact with your plasma?

Boron nitride pastes as well as crystal bond and oils are common mounting/heat sinking...glues

What is your substrate temperature?

RIE tools often have Helium back side cooling and a resulting helium leak rate



Amit Solanki, Handon Um, Chapter Two - Top-Down Etching of Si Nanowires, Semiconductors and Semimetals,

Temperature control... and maybe a bit of helium in your plasma. Carrier wafer must be clean!!!

Measure your selectivity, this will feedback to the plausibility of your design!

A few ways to do this:

Make a film, run your plasma for a bit, measure film thickness with elispometer, make enough points to make a straight line.

Etch silicon oxide or silicon nitride using a mask of PMMA or CSAR

Here is an etch recipe for an ordinary parallel-plate etcher such as the Oxford-80, with a carbon bottom plate:

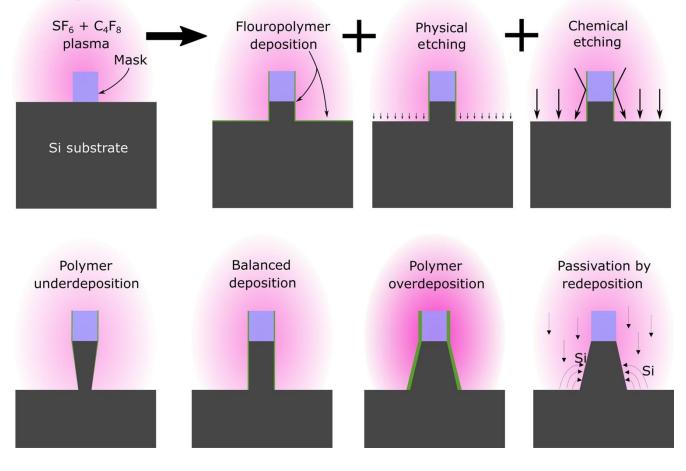
| CHF ₃ | 40 sccm |
|------------------|----------|
| 02 | 2 sccm |
| pressure: | 30 mTorr |
| RF power: | 100 W |
| | |

This etches silicon nitride at about 10 nm/min, while etching the PMMA mask at about 5 to 10 nm/min.

https://nano.yale.edu/etch-recipes

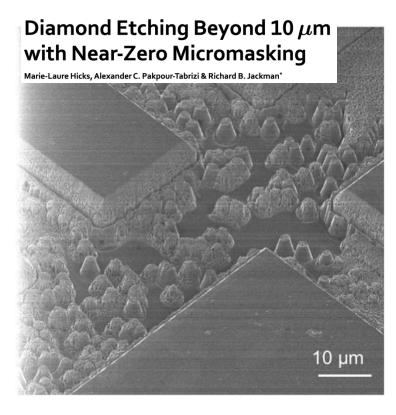
Is your selectivity the same in the middle of your mask/a feature as near edges..

Process optimizations: ICP & RF power, flow rates and pressure, temperature and time



Amit Solanki, Handon Um, Chapter Two - Top-Down Etching of Si Nanowires, Semiconductors and Semimetals,

Micro-grass/nano-wiskers or pillars

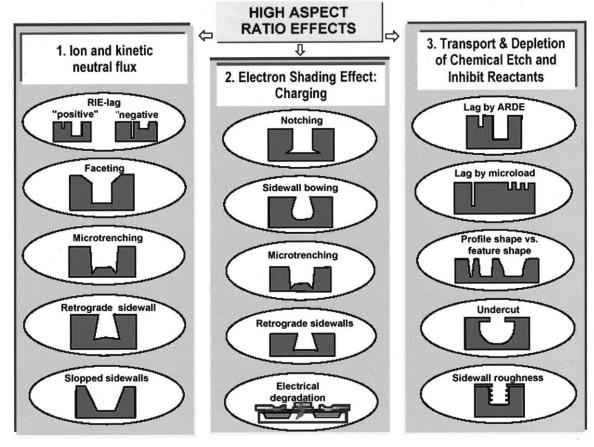


Sputtering from mask or substrate can put down micro or nano particles that can act as a mask, creating unwanted roughness.

Polymer creating plasmas can also cause micromasking...

Figure 2. Surface Roughening and Damage Observed after 233 Minute Etch with Ar/O₂ ICP RIE (HIM). Significant micron-scale pillar-like features were observed across the etched surface. The mesa sidewalls presented a rough surface and trenching at the base.

Area dependent etching.



High aspect ratio dependent effects in high-density plasma etching of silicon

Invited Paper: Critical tasks in high aspect ratio silicon dry etching for microelectromechanical systems

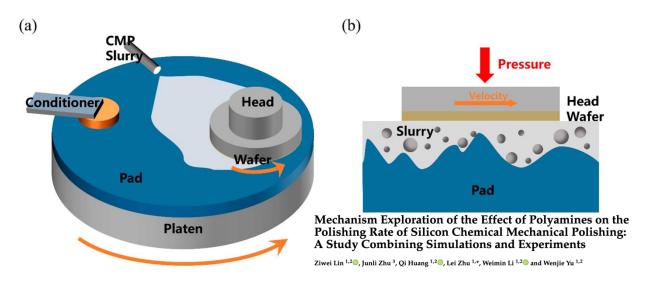
Ivo W. Rangelow^{a)}
Institute of Microstructure Technologies and Analytics (IMA), University of Kassel, Heinrich Plett Straße 40, 34132 Kassel, Germany

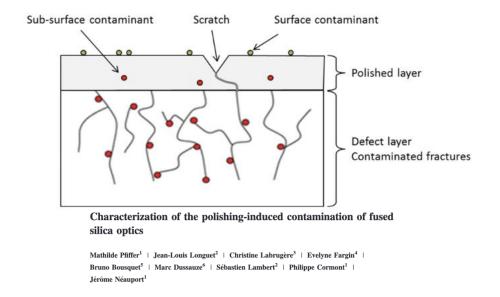
These are just some of the gremlins waiting to entertain you!

An application of ICP-RIE for QIS

 Removing polish damage layer from diamond to enable quantum sensing with shallow Nitrogen Vacany centers on diamond.

Improving surfaces with plasma processing.



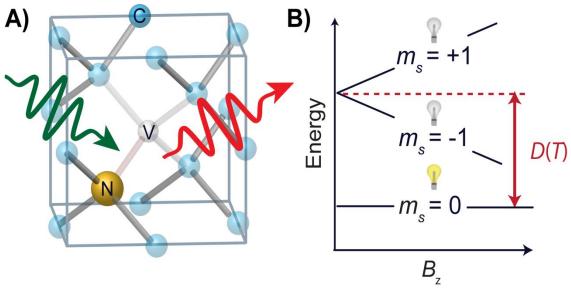


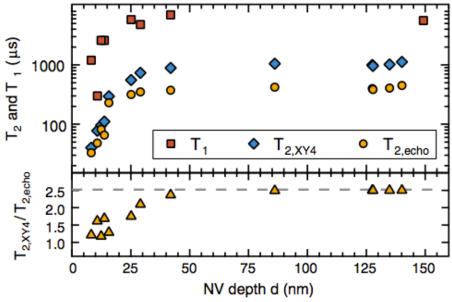
Problems: Diamond is hard and brittle. Can we remove polish damage with plasma etching without amplifying roughness, preferably smoothing the surface.

For QIS can we also remove Ion mixing at the surface from RIE plasma processing

What is a nitrogen Vacancy and why do we want it close to surfaces.

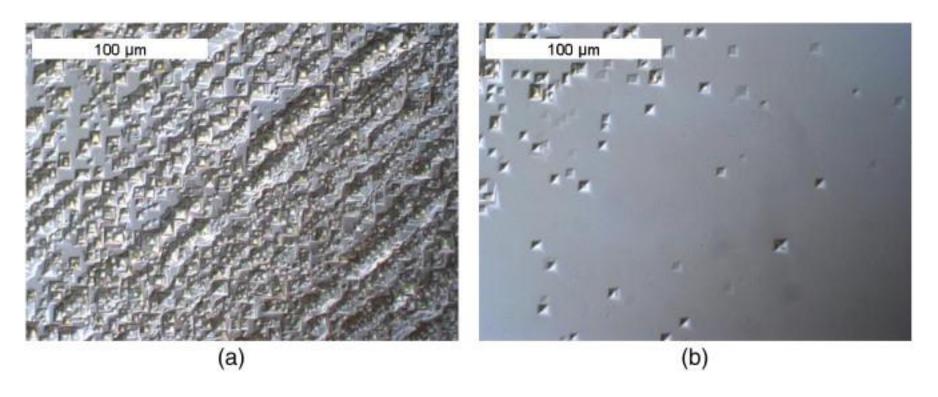
NV coherence degrades with proximity to surface Myers et. al, Phys. Rev. Lett. (2014)





Toyli et al., PNAS 110, 21 8417-8421

Using an oxygen plasma to reveal Sub surface damage:



DIC micrographs of single crystal samples etched in an oxygen-containing plasma with (a) high levels and (b) lower levels of sub-surface damage.

Control of surface and bulk crystalline quality in single crystal diamond grown by chemical vapour deposition Author links open overlay panell. Friel

, S.L. Clewes, H.K. Dhillon, N. Perkins, D.J. Twitchen, G.A. Scarsbrook

Developing damaged surface removal, smoothing diamond etch.

| Treatment | Ar (sccm) | O ₂ (sccm) | CF ₄ (sccm) | ICP (W) | Platen (W) | Pressure (mTorr) | Etch rate (nm/min) |
|-----------|-----------|-----------------------|------------------------|---------|------------|------------------|--------------------|
| AOCF4 | 100 | 11 | 4 | 250 | 300 | 5 | 61.5 |

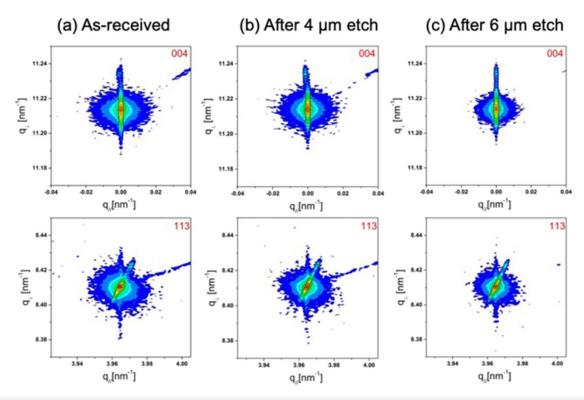
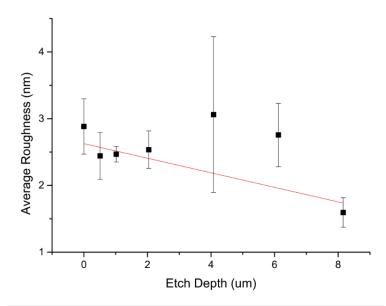


FIG. 9. Reciprocal space mapping with HRXRD: comparison between (a) as-received, (b) after $4\mu m$ etch, and (c) after $6\mu m$ etch (b) for (004) and (113). The diffuse scattering remained unchanged between (a) and (b) and reduced following treatment AOC to $6\mu m$ depth.



Marie-Laure Hicks, Alexander C. Pakpour-Tabrizi, Verena Zuerbig, Lutz Kirste, Christoph Nebel, Richard B. Jackman; Optimizing reactive ion etching to remove sub-surface polishing damage on diamond. *J. Appl. Phys.* 28 June 2019; 125 (24): 244502. https://doi.org/10.1063/1.5094751

HRXRD: monochromatic X-ray beam at a sample and measuring the intensity of the diffracted X-rays as a function of the incident angle

Etching and micro-optics fabrication in diamond using chlorine-based inductively-coupled plasma

C.L. Lee a,*, E. Gu a, M.D. Dawson, I. Friel, G.A. Scarsbrook

^a Institute of Photonics, University of Strathclyde, Glasgow, G4 0NW, United Kingdom
^b Element Six Ltd, King's Ride Park, Ascot, Berkshire SL5 8BP, United Kingdom

Available online 12 January 2008

Chlorine base Ar/Cl₂ plasma etching

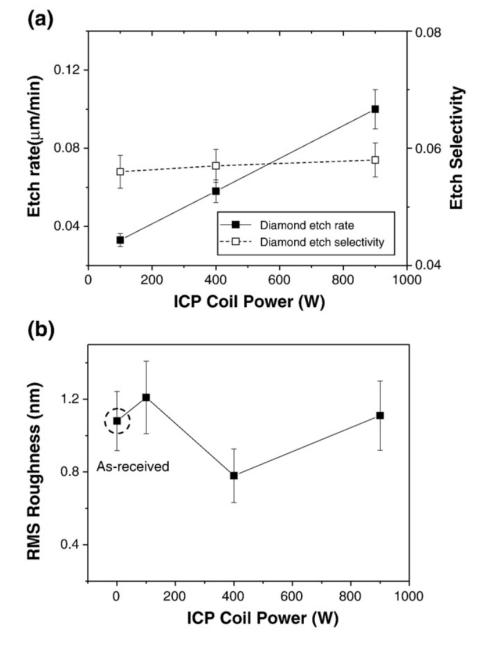


Fig. 1. (a) Etch rate and selectivity of natural diamond as a function of ICP coil power with the ICP platen power of 300 W, and (b) corresponding surface rms roughness for a 5 μ m \times 5 μ m area.

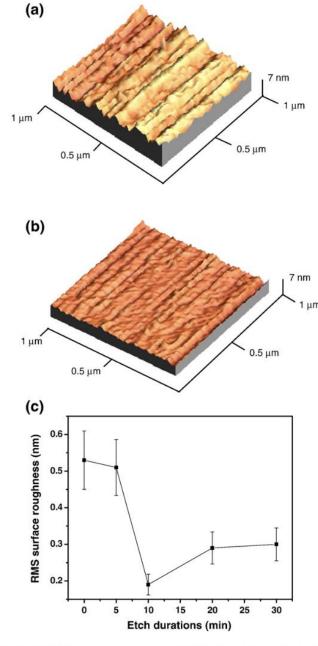
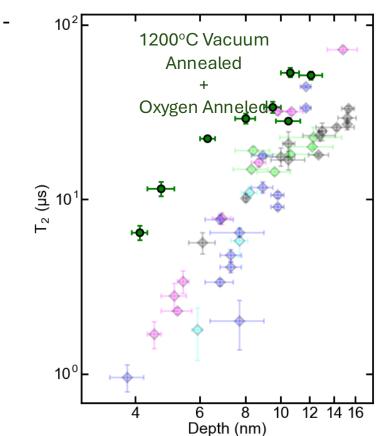
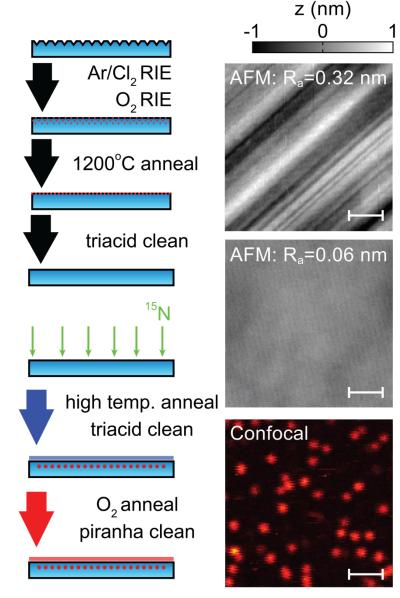


Fig. 2. (a) AFM images of an as-received HPHT diamond, and (b) the HPHT diamond etched by Ar/Cl_2 plasma for 10 min at an ICP platen and coil powers of 300 W and 400 W, respectively. (c) Surface rms roughness versus etch durations for a 1 μ m x 1 μ m area after etched with ICP Ar/Cl_2 plasma.

Preparing a Pristine Diamond Surface for quantum sensing

- Morphologically smooth surface
- Low strain and minimal subsurface defects





Origins of diamond surface noise probed by correlating single spin measurements with surface spectroscopy- arXiv: 1810:00144

High quality diamond etching for nanoscale quantum sensing substrates.





The devils at the Interfaces:

Charge-state instability

Band bending

NEA surface termination



Charge traps

centers, vacancy centers



e.g., sp² carbon, P1



Acceptor states

photoluminescence quenching

Surface roughness

source of charge state instability, magnetic noise & electronic noise

Magnetic noise

Proximal spins

unpaired electrons

surface nuclear spins



Electronic noise

Unscreened charge

surface termination, or adsorbed molecules









vacancy complex



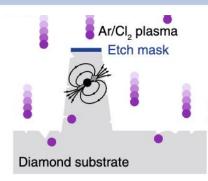
NV- → NV0





P1 center

Etching generally makes things worse!



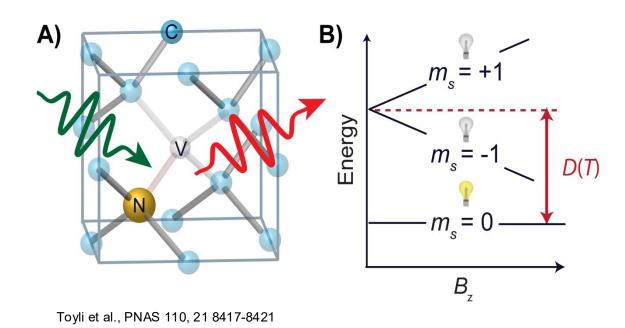
Materials challenges for quantum technologies based on color centers in diamond

Lila V. H. Rodgerso, Lillian B. Hugheso, Mouzhe Xieo, Peter C. Maurer[®], Shimon Kolkowitz[®], Ania C. Bleszynski Jayich[®], and Nathalie P. de Leon*0

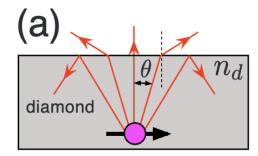
Diamond surface engineering for molecular sensing with nitrogen—vacancy centers

Erika Janitz, Konstantin Herb, Laura A. Völker, D. William S. Huxter, D. Christian L. Degen and John M. Abendroth **

Why do we want to etch diamond for NV sensing experiments:



1. Collection efficiency

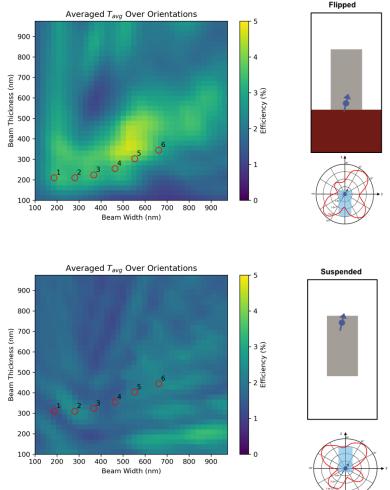


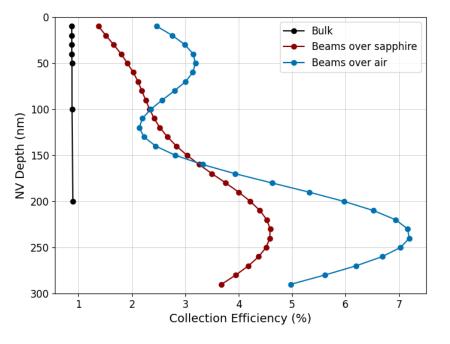
Monolithic diamond optics for single photon detection **⊘**

P. Siyushev; F. Kaiser; V. Jacques; I. Gerhardt; S. Bischof; H. Fedder; J. Dodson; M. Markham; D. Twitchen; F. Jelezko; J. Wrachtrup

Simulation and design goals. Beam orientation and interfacing with a dielectric.

FDTD simulations for 5nm deep nvs in a sweep of beam thickness and widths, on sapphire





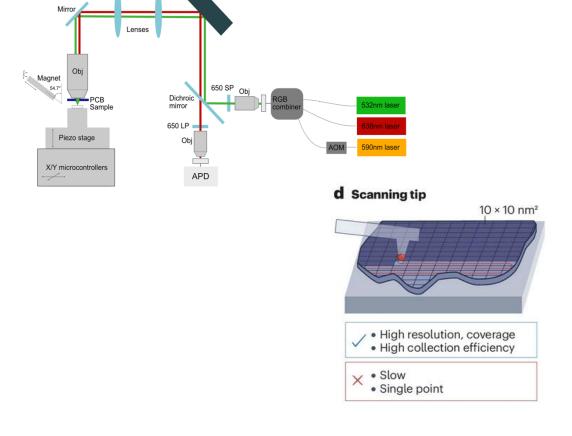
Collection efficiency for 300nm wide 250nm deep beam on sapphire or air

Far-field, half-space projection of the electric field magnitude for the two configurations along the y/z cross section at x=0. NV depth of 20 nm and at 700 nm wavelength.

Experimental configurations:

2. Enable Sensing platforms and modalities:

Galvanometer



B Resolvable layer C Ensemble layer 500 × 500 nm² ✓ • High resolution • Simultaneous readout ✓ • Low coverage • Inconvenient optical access C Ensemble layer ✓ • High coverage • Fast imaging ✓ • Low resolution • Challenging MW homogeneity C Ensemble layer

· High collection efficiency

· Difficult to fabricate and handle

No sample obstruction

× • Low coverage

Nanoscale diamond quantum sensors for many-body physics

Jared Rovny®¹, Sarang Gopalakrishnan¹, Ania C. Bleszynski Jayich², Patrick Maletinsky®³, Eugene Demler & Nathalie P. de Leon®¹⊠

Single crystal diamond is (nearly) always homoepitxy... so to make free standing nano beams you need to be creative! Triangular diamond nanobeams

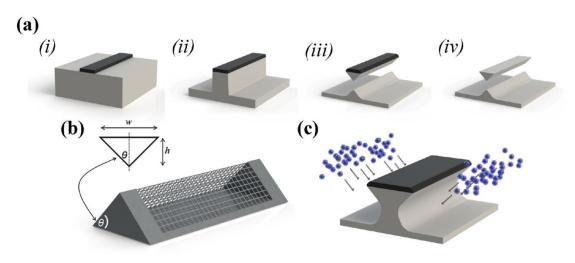


Figure 1. (a) Angled-etching fabrication schematic: (i) an etch mask was defined by standard electron beam lithography and thin film deposition techniques, (ii) the etch mask pattern was then transferred into diamond substrate by conventional top down plasma etching, (iii) angled-etching is then employed to realize suspended nanobeam structures, (iv) residual etch mask is removed. (b) Schematic of triangular prism Faraday cage design with inset showing the relationship between the prescribed etch angle and the nanobeam bottom apex. (c) Illustration of angled-etching from two directions accomplished with the triangular prism Faraday cage design.

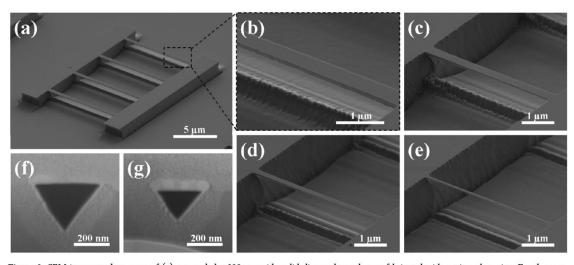


Figure 2. SEM images and an array of (a) suspended \sim 500 nm wide solid diamond nanobeams fabricated with a triangular prism Faraday cage. Close-up SEM images of (b) \sim 500 nm, (c) 350 nm, (d) 200 nm, and (e) 75 nm wide solid diamond nanobeams. SEM images of FIB cross-sectioned (f) \sim 350 nm and (g) \sim 250 nm wide solid diamond nanobeams. All SEM images were taken at a 60° stage tilt.

Free-Standing Mechanical and Photonic Nanostructures in Single-Crystal Diamond

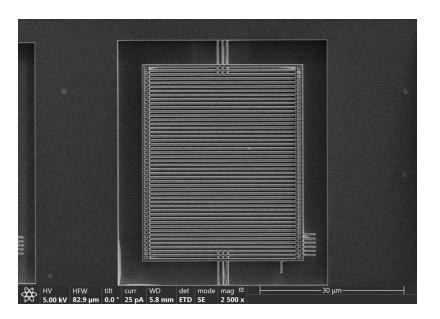
Michael J. Burek,[†] Nathalie P. de Leon,^{‡,§} Brendan J. Shields,[‡] Birgit J. M. Hausmann,[†] Yiwen Chu,[‡] Qimin Quan,[†] Alexander S. Zibrov,[‡] Hongkun Park,^{†,§} Mikhail D. Lukin,[‡] and Marko Lončar*,[†]

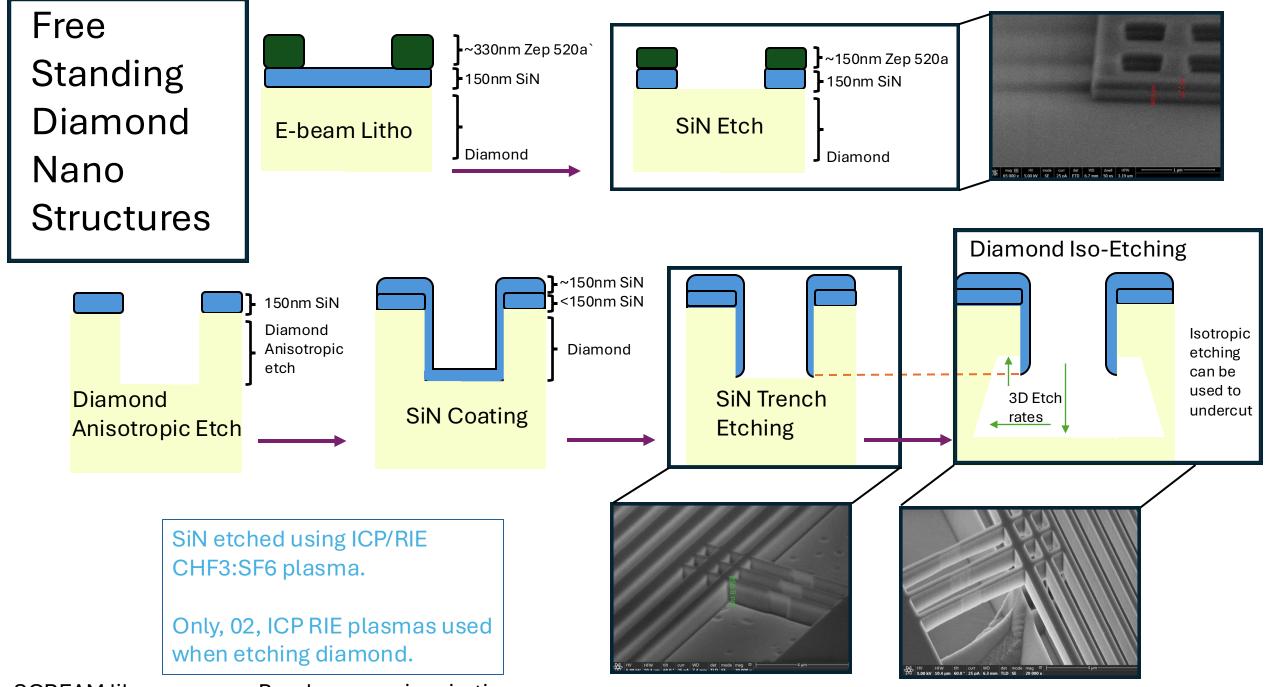
Goal: Enable sensing experiments!!

- Advantages of nanowire frames
- Versatile feature fabrication (nm to microns).
- Many beams to a frame. Enable covariance and larger multiplexed NV experiments.
- Easily transferable over sensing targets.
- Tunable NV depth.
- Material stacks, if dielectric layers are needed.
- Enhanced collection efficiency.

Take the state of the art, best features of existing unstructured and nanostructured diamond and make a versatile sensing platform.

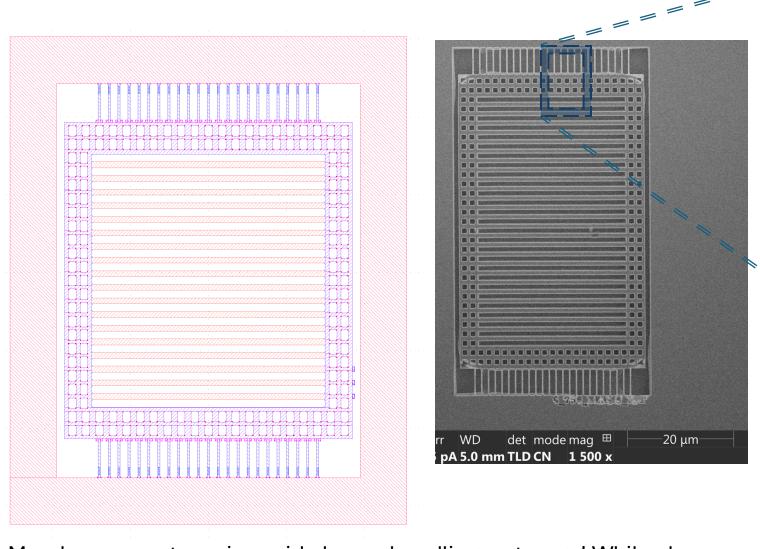
- Research questions:
- Geometries/topography to maximise collection efficiency?
- How to reliably fabricate beams, and frames of beams, yield..?
- Are the NV properties affected by fabrication processes?

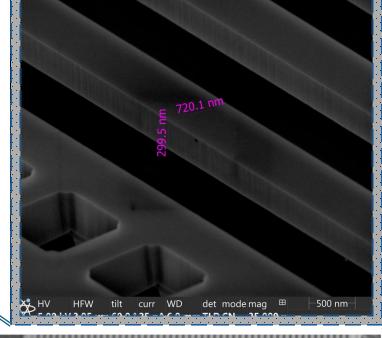


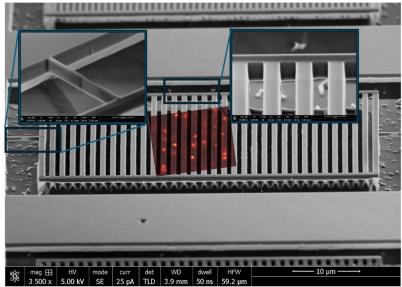


SCREAM like process... Barclay group inspiration:

Transferable Membrane of Nanowires

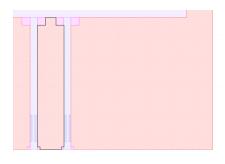


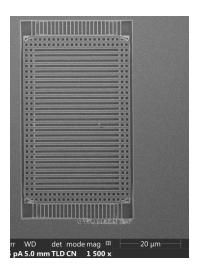


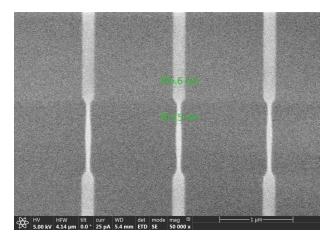


Membrane must survive acid cleans, handling... storage! While also being able to be removed at will

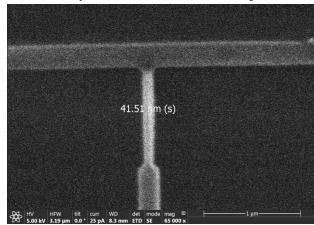
Tethers to host crystal. Area dependent etching informs all design choices

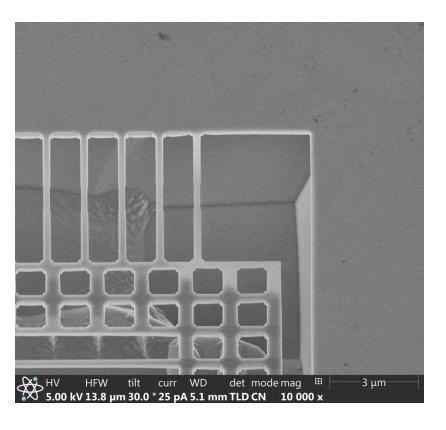




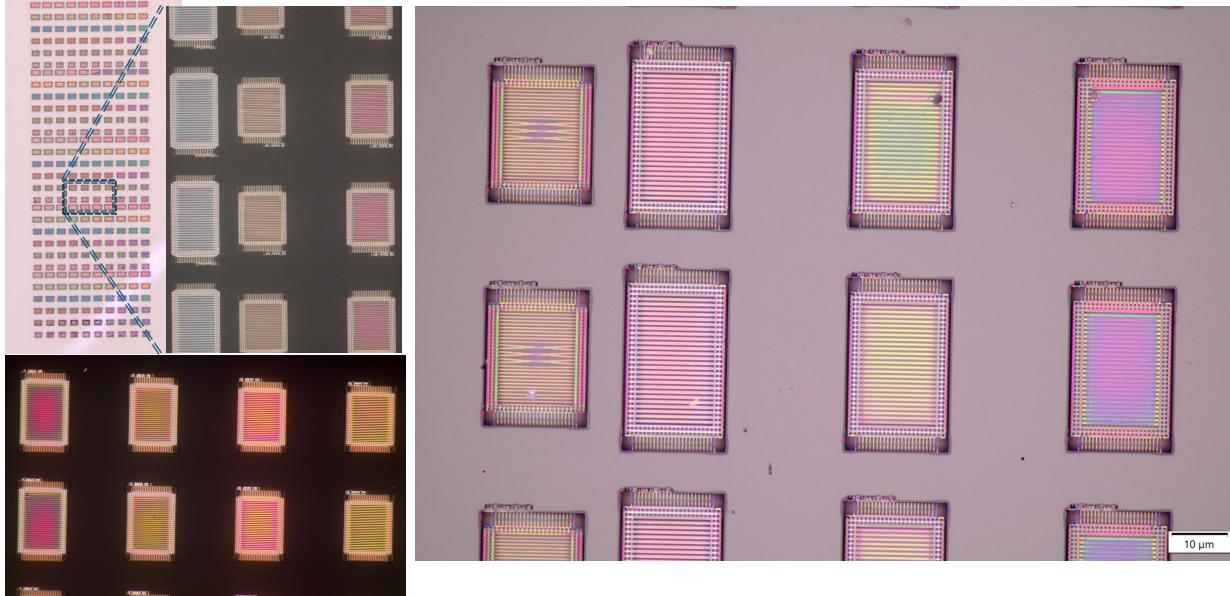


Can be pushed to be very thin





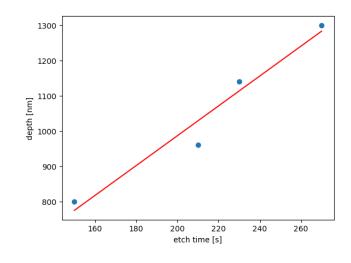
But it doesn't need to be pushed too far. Memframe must survive many rounds of aggressive cleaning while still being removeable.



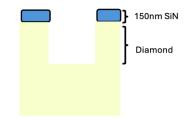
White light optical microscope images.

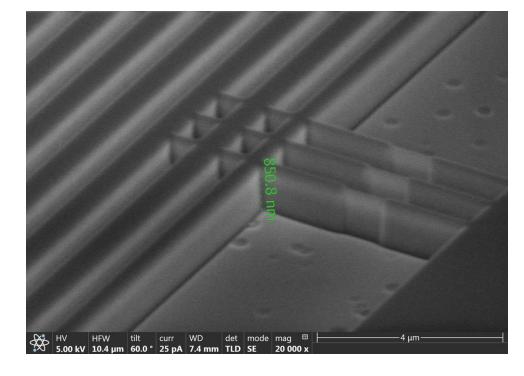
The Anisotropic diamond etch: etch fast but without messing everything up!

| achi Recipe nanopillar aniso | Step 4 |
|---|--|
| | Etch |
| Time | 3min30s |
| Bias Power (W) | 110 |
| ICP Power (W) | 1000 |
| Coil V | <mark>273.5</mark> |
| DC bias | 94 |
| Chamber Pressure (mTorr) | 10 |
| 02 flow rate (sccm) | 30 |
| Chamber Temperature (degree C) | 40 |
| Electrode Temperature (degree C) | 20 |
| He backing pressure (Torr) | 5 |
| He backing flow rate (sccm) | 3.82 |
| Throttle pos | <mark>13.8</mark> |
| Coil V DC bias Chamber Pressure (mTorr) 02 flow rate (sccm) Chamber Temperature (degree C) Electrode Temperature (degree C) He backing pressure (Torr) He backing flow rate (sccm) | 273.5 94 10 30 40 20 5 3.82 |



Etch Rate ~ 50nm/s

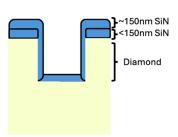




Low-No micromasking, no edge erosion, minimal trenching and smooth vertical sidewalls

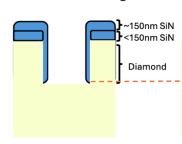
PECVD of SiN and etching it!





| Takachi Recipe Si N | Step 6 |
|--|-------------|
| | Etch |
| Time | 9s |
| Bias Power (W) | 25 |
| ICP Power (W) | 1000 |
| Chamber Pressure (mTorr) | 5 |
| CHF3flow rate (sccm) | 14 |
| SF6 flow rate (sccm) | 14 |
| Ar flow rate (sccm) | 0 |
| 0 flow rate (sccm) | 0 |
| Chamber Temperature (degree C) | - |
| Electrode Temperature (degree C) | - |
| Throttle pos | 29%(stable) |

SiN Trench Etching



GaAs test structures can get you so far...



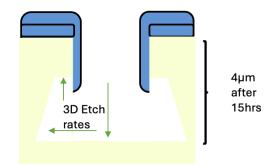
Etch to punch through bottom not sides or top.

Deposition...and etch Rates may vary...do careful metrology at every step!

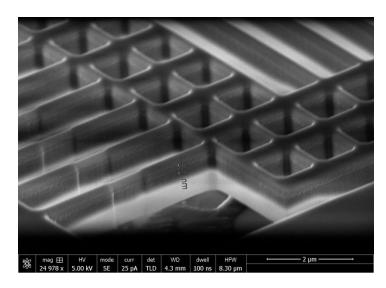


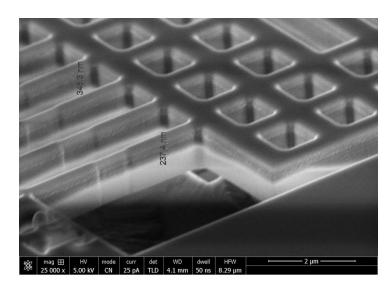
Isotropic etch. Etch slow... but without messing everything up...

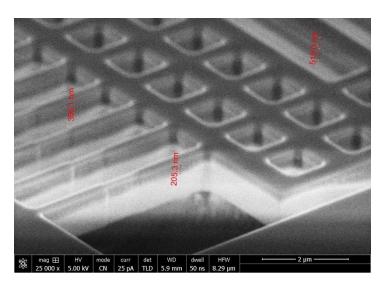
Diamond Isotropic-Etching



40sccm O2, ICP power 1000 W, RF bias 0W, 20 mTorr, 140°C

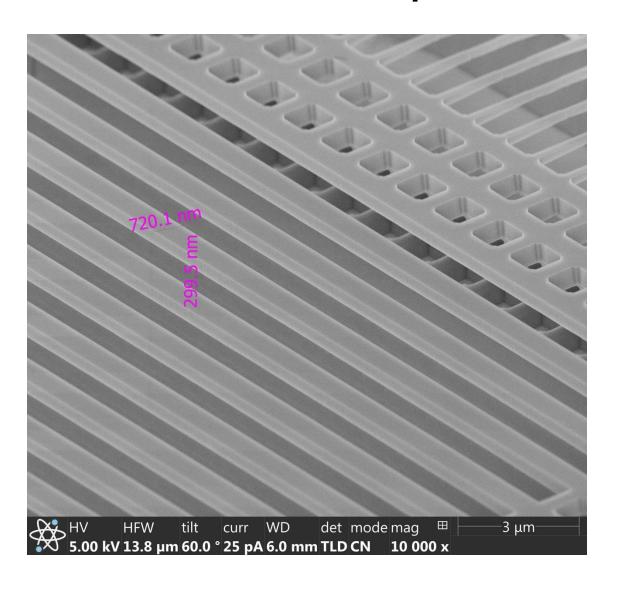


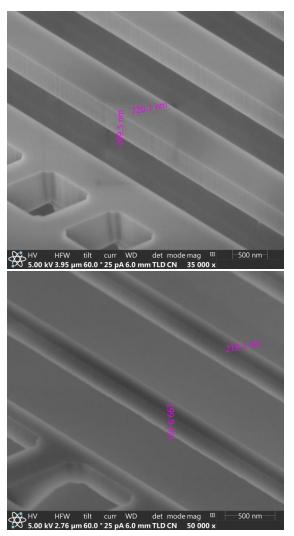


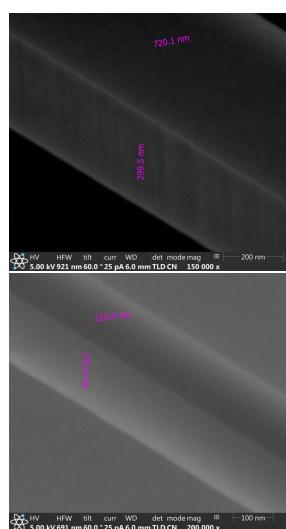


15hrs 20hrs 30hrs

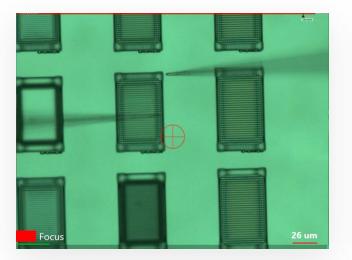
Our beams: suspended, 24hrs HF to remove SiN

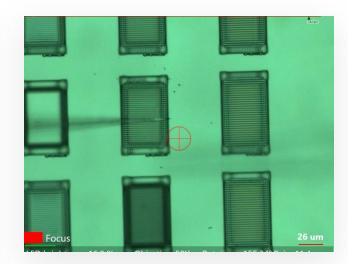


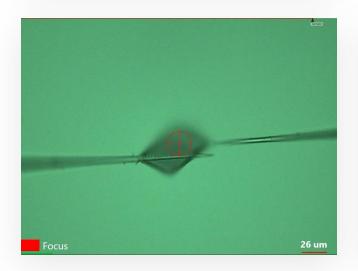


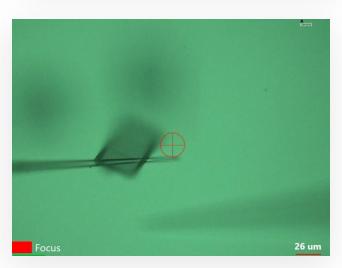


Transferring memframes

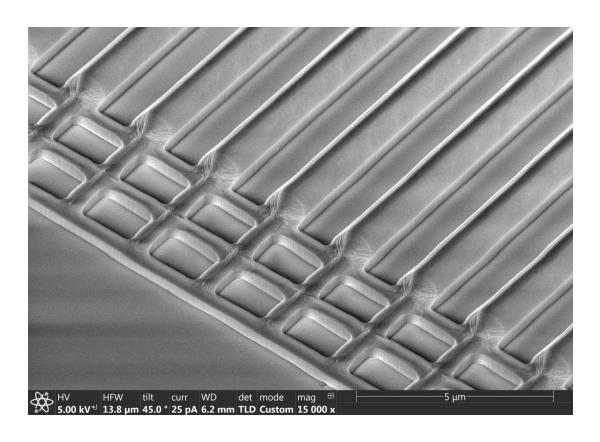


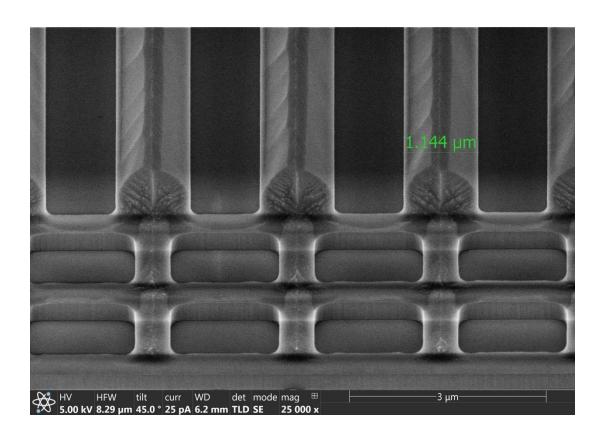






Beams: transferred and flipped: 1.15µm wide beams.

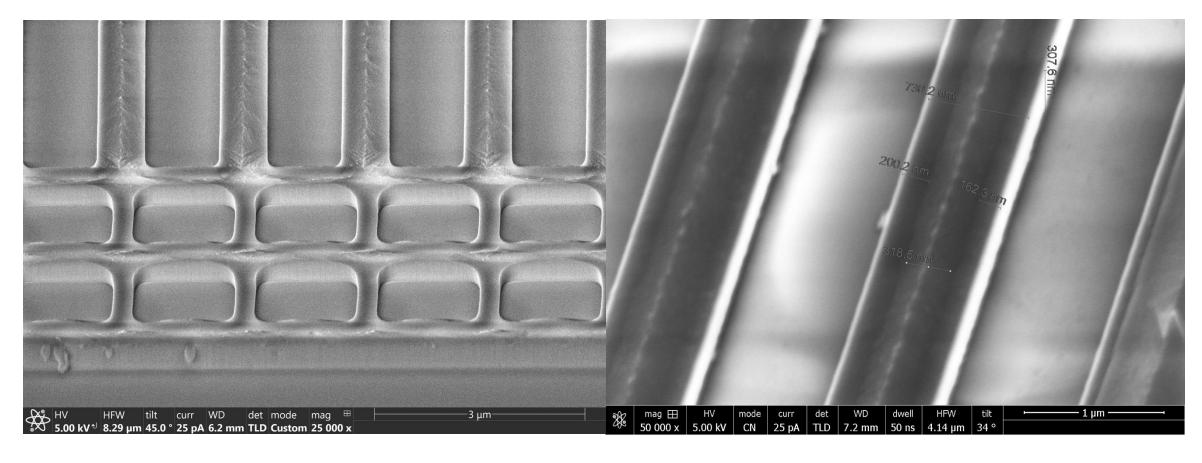




Standard trellis frame is the same as we vary beam width, keeping 1µm gap between wires.

Bottom of beams evolves between release and over etching

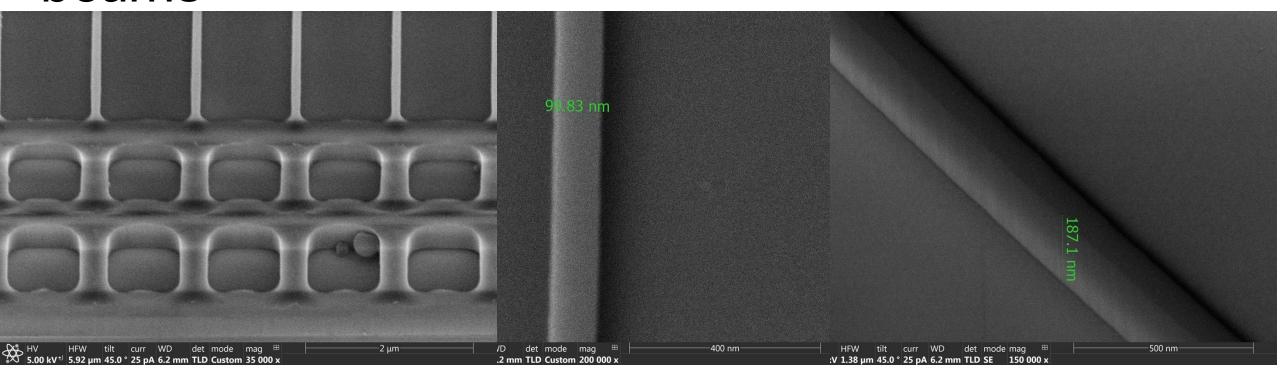
Our beams: transferred and flipped: 720nm wide beams.



Standard trellis frame is the same as we vary beam width, keeping 1µm gap between wires.

Bottom of beams evolves between release and over etching

Our beams: transferred and flipped:100nm wide beams

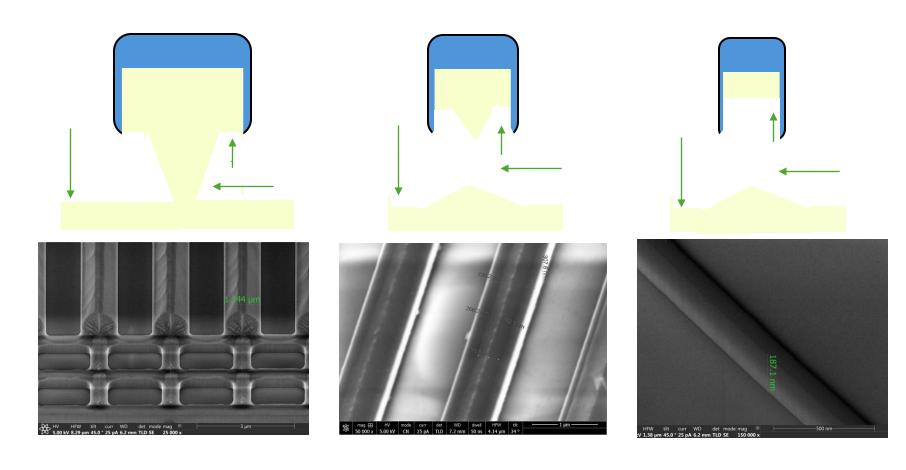


Standard trellis frame is the same as we vary beam width, keeping 1µm gap between wires.

Bottom of beams evolves between release and over etching

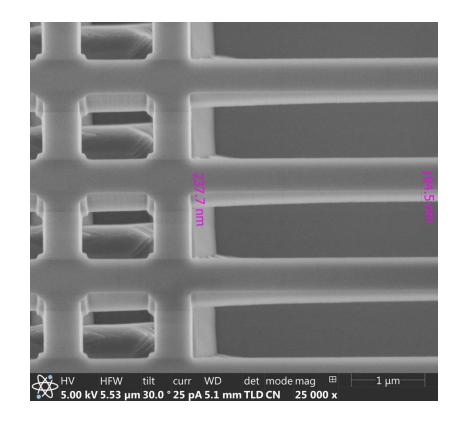
Toy model for Evolution of beam bottom

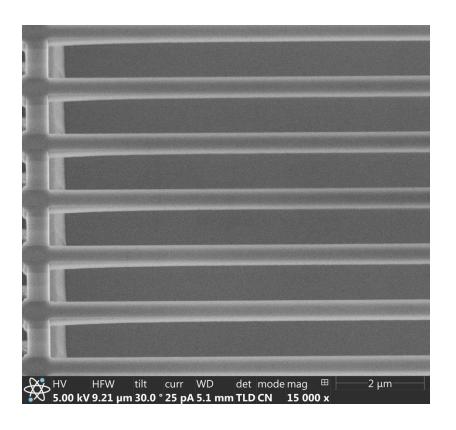
Keeping isotropic etch constant. Mask design and layout can radically change etch rates



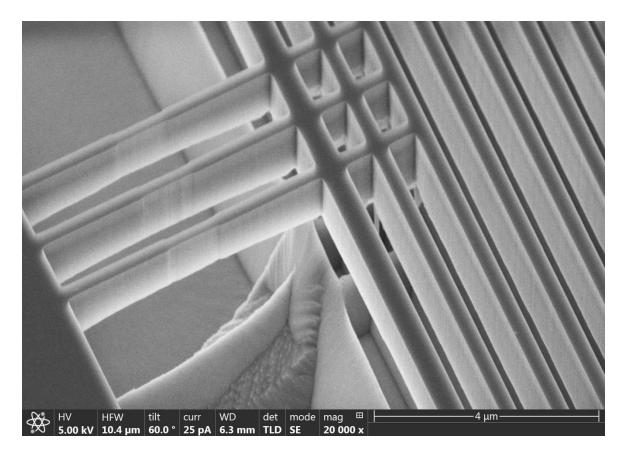
20hrs of isotropic etch time for different widths of beams

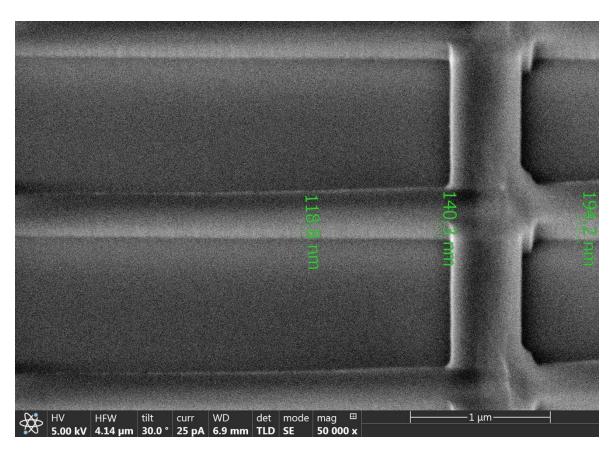
Thickness Variation across beams length:





Membrane thickness: by varying anisotropic etch time





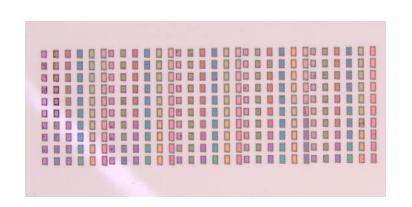
Ranging from over 1um deep to under 100nm deep with new tethers, xy limited by usual lithographic constraints.

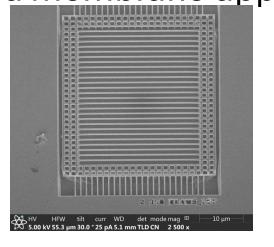
Very high aspect ratio and range of feature dimensions possible with one, robust, process.

Recap:

• Transferrable Nanostructured membranes optimised for collection efficiency.

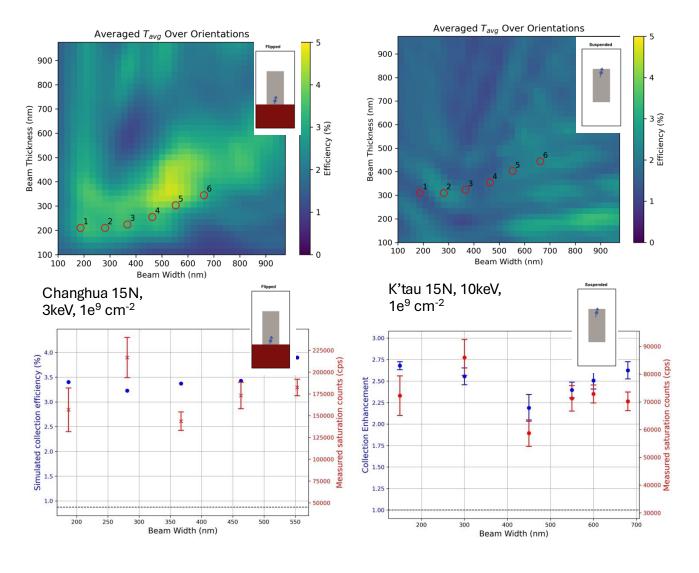
 Reproducible versatile patterning and fabrication process for making the nanostructured membrane appropriate for your task



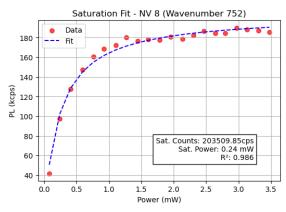


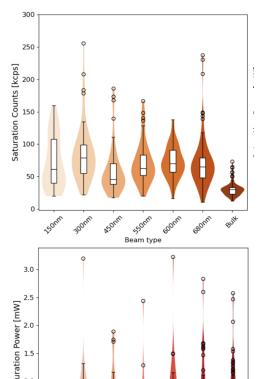
Do quantum defects have acceptable properties in etched membranes?

NVs in nanobeams... Always brighter than bulk (inair):



*Diagonal cut through space as beam depth/thickness varies for one given iso-etch time

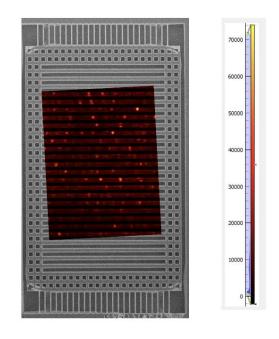




Tecla Bottinelli Montandon Master thesis in Materials Science and Engineering, EPFL

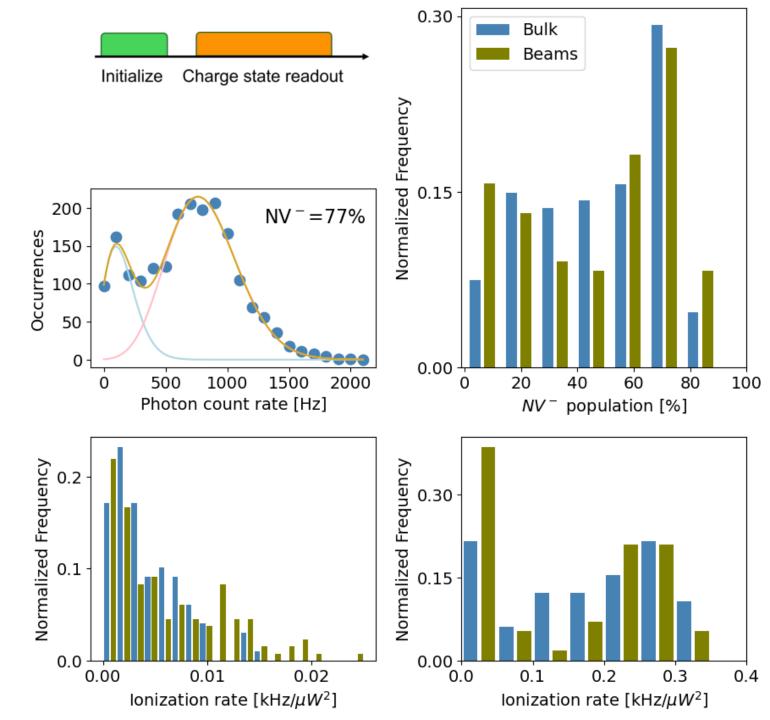
Beam type

Preliminary Charge state comparisons between shallow nvs in bulk and beams:

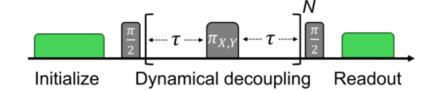


Charge state dynamics and optically detected electron spin resonance contrast of shallow

Zhiyang Yuan, Mattias Fitzpatrick , Lila V. H. Rodgers, Sorawis Sangtawesin , Srikanth Srikan



Spin properties:



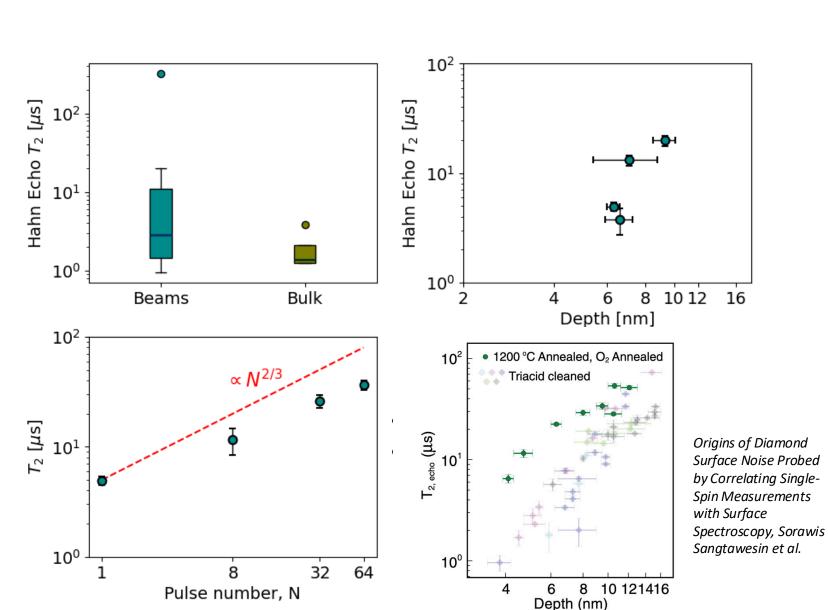
Looks like T2 in nanostructures with shallow NV centers behaves as expected/hoped for.

We can also measure the depth of the shallow nvs by looking at the proton nmr signature of oil.

 λ =2/3. P1 densities are below 100 ppm.

Quantum decoherence of nitrogen-vacancy spin ensembles in a nitrogen spin bath in diamond under dynamical decoupling

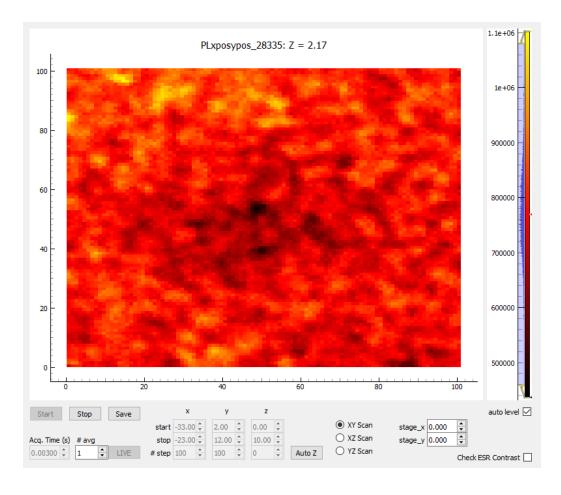
Huijin Park^{1,2,3}, Mykyta Onizhuk³, Eunsang Lee⁴, Harim Lim⁴, Junghyun Lee^{3,4,5*}, Sangwon Oh^{2*}, Giulia Galli^{3,6,7}, and Hosung Seo^{1,2,4,8*}

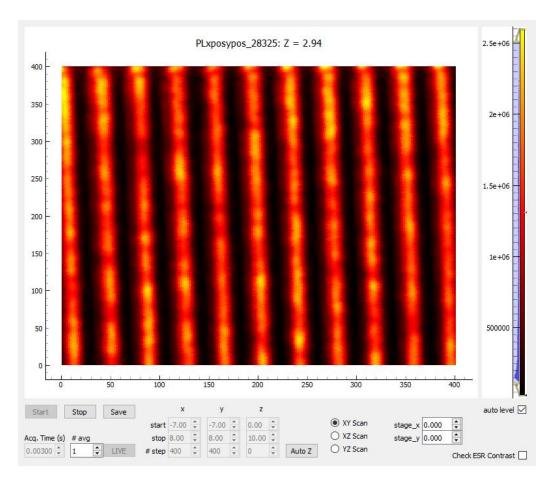


Future Work

- T1, T2 CPMG and depth check, spin characterisation...Stats!
- Sensor integration, low temperature measurements.
- Characterisation of sidewall roughness, AFM, TEM, measurements of losses in Photonic structures and Electrical devices such as hall bars, Schottky diodes, MESFETs etc
- Models to capture true beam shape, fab scatter and variation in defect location.
- A broad range of non-nv experiments!!

Other samples: Ensemble implantation, conditions: 15N, 5keV, 2e¹² cm⁻²

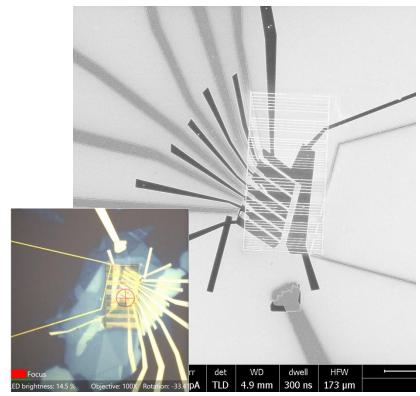




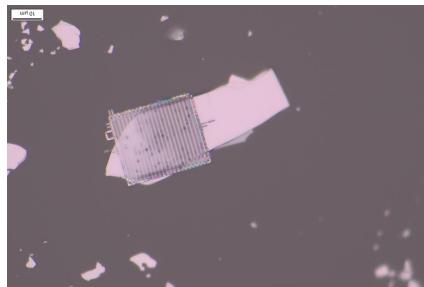
Bulk

Beams

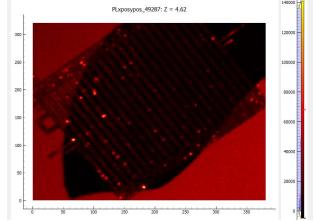
Integration and packaging.

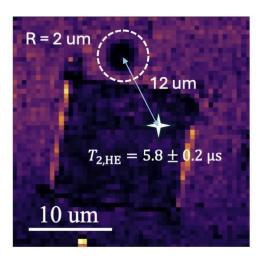


Frame on graphene transistor.

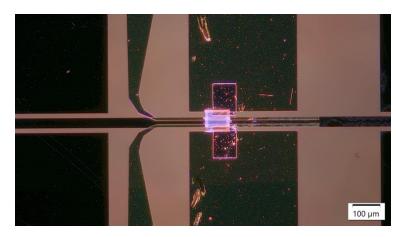


Beams on TaCoTe²





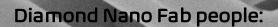
Beams on YBCO with levitating magnet, *Trisha Madhavan, Lukin group*



Hydrogen terminated Nanowires with source and drain contacts on cpw with voltage probes.

Acknowledgments: Whole De Leon group... Particularly diamond Nano factory!





- Tecla Bottinelli Montandon
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- Jared Rovny
- Alex Abulnaga
- Lila Nassar
- · Zeeshawn Kazi
- Marjana Mahdia
- Zhiyang Yuan
- Nathalie De Leon





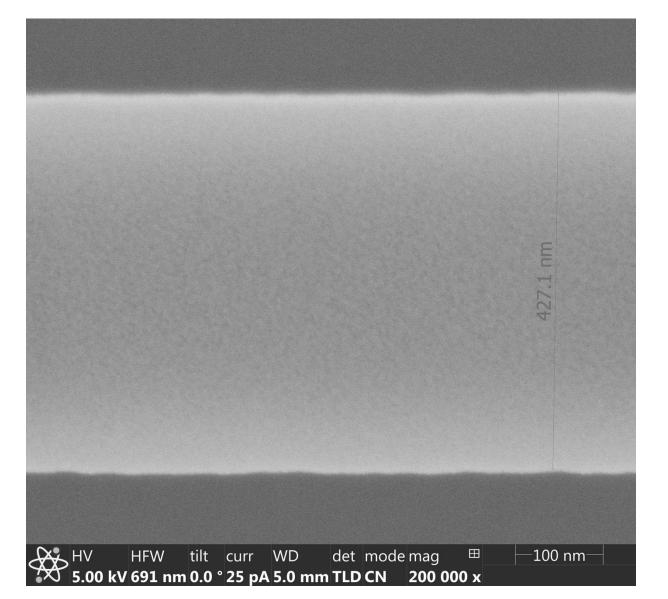




Honorable mention to superconducting qubits team.

Ray Chang, Apoorv, Artharv, Izze, Faranak, Mathew, Andrew Houck etc!

Edge roughness



1-2nm of wiggle

Switching gases to remove non-volatile re-doposited micro masks can work...

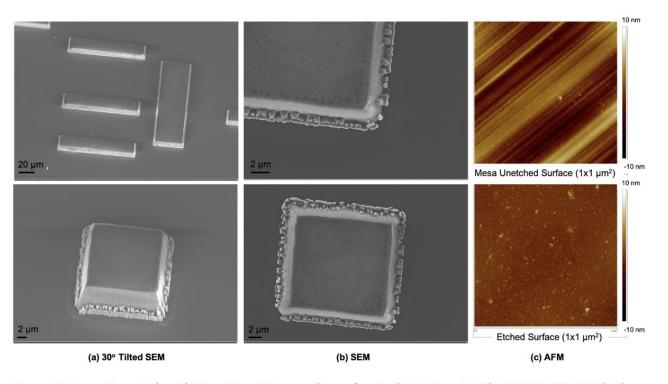
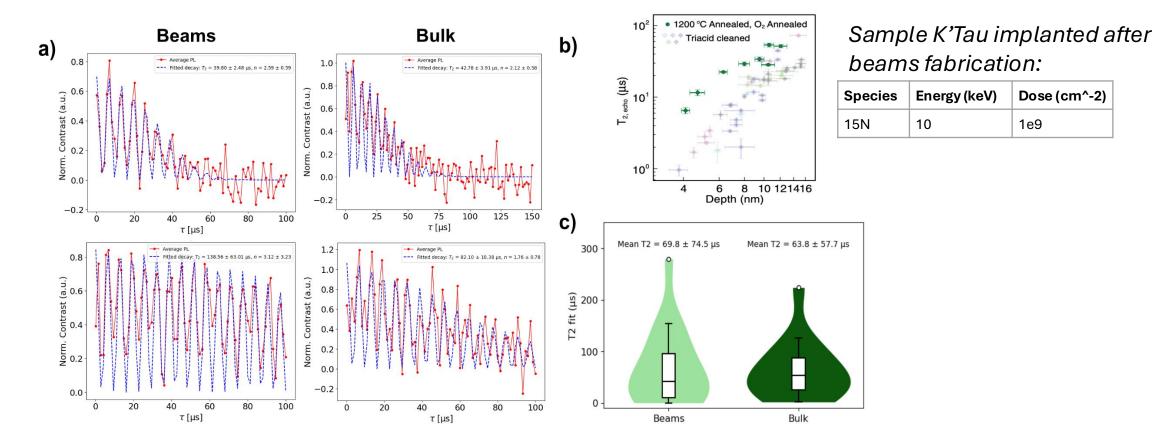


Figure 3. 8 μ m Deep Etch with Near-Zero Micromasking after Cyclic Ar/O₂ - Ar/Cl₂ ICP RIE: (a) 30° tilted SEM, (b) no tilt SEM and (c) AFM scans of the unetched mesa surface with 1.7 nm roughness and etched surface with 0.47 nm roughness. The etched surface was smooth, with micromasking only observed within a 1 micron radius around the base of the mesa. The mesa sidewalls were also smooth with defects observed at the base and without trenching.

T2, on average as expected for NVs implantation



40 beams NVs from three different width categories (from 300 to 650 nm width). This data is all taken in the same region of the diamond.



Grapes in a microwave

https://www.youtube.com/@veritasium