SQMS Fermilab U.S. DEPARTMENT OF ENERGY Office of Science

Micro/Nano Fabrication of Superconducting Quantum Devices

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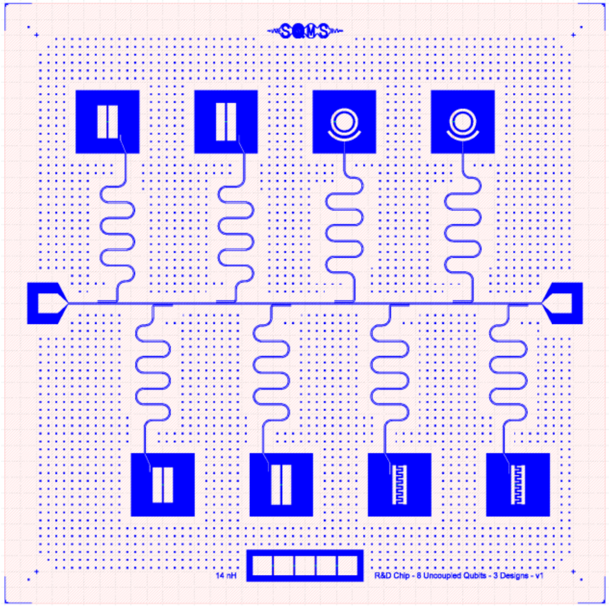
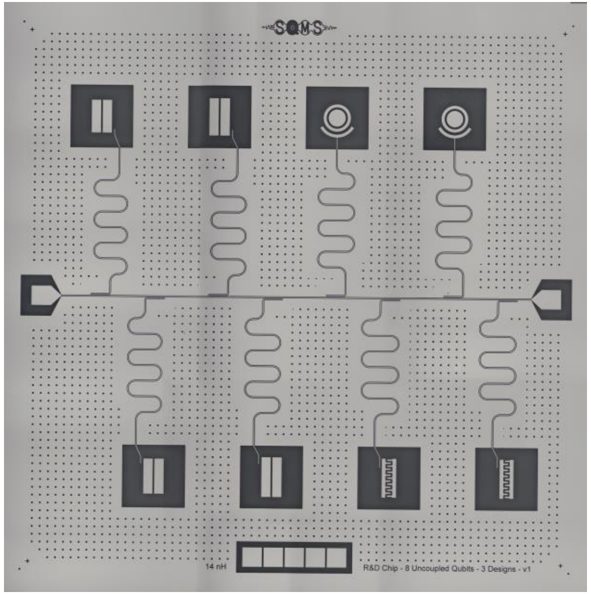
Design to Device

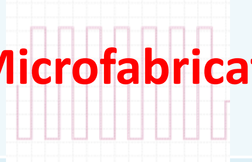
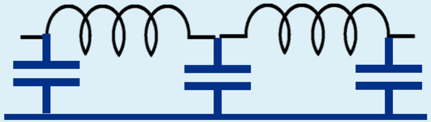
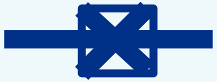
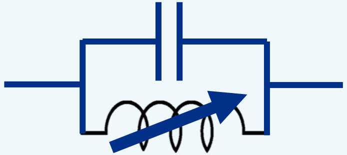


Micro/Nano

Fabrication



Basic components in the design

Name	Symbol	Equivalent rep.	Top view
Capacitor			
Inductor			
Transmission line (CPW)			
Josephson Junction			

Microfabrication

Nanofabrication

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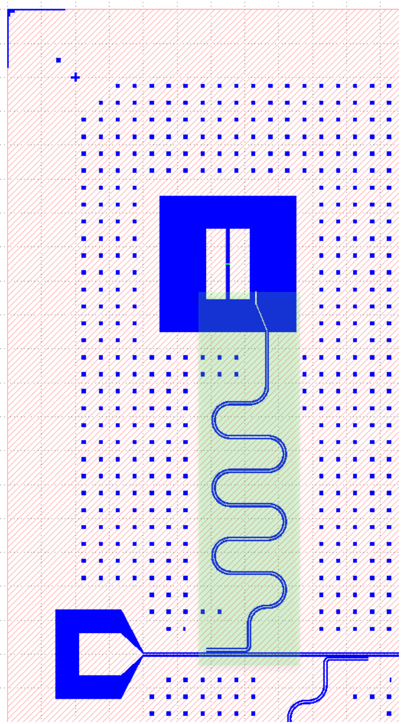
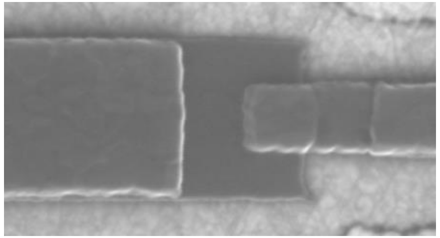
1st key component of superconducting circuits - resonators

- Typically meander CPWs, $l = {}^{\lambda}/_{4}$ $(\text{or}\, {}^{\lambda}/_{2})$; $f_r = \frac{v}{l} \sim 6 - 7\ GHz$ coupled to feedline
  - Loss/cycle, $\tan\delta \sim 10^{-6}$ - 10^{-8}
  - Can define Q = f_r / Δf $= 1/\tan\delta$
  - long lifetime, τ = Q/ $2\pi f_r$
- Measure amplitude & phase of output probe tone
  - Useful as test structures to evaluate loss (δ=1/Q)
  - Extensively used circuits as readouts & couplers for qubits
  - Equally space energy levels => difficult to use directly as qubits

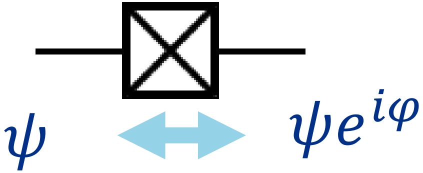


2nd key component – Josephson junction

- Wavefunction tunnels across barrier from top electrode to bottom electrode
- Ultra-thin amorphous−AlOX
  - small to reduce loss
  - Thermal oxidation, thickness d~1.8 nm:
    - tunneling current $I \propto \exp(-d/\lambda)$
  - φ = phase across junction, Josephson relations

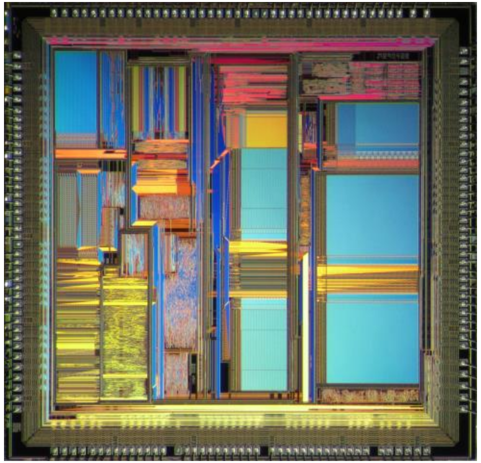


- $$\begin{cases} I = I_0 \sin(\varphi) \\ V = \frac{\Phi_0}{2\pi}\frac{d\varphi}{dt} \end{cases}$$



$$I_0 = \frac{\pi}{2e}\frac{\Delta_{S.C.}}{R_n}$$ (Ambegoaker – Baratoff Relation)

Micro/Nano Fabrication of ICs



- Tremendous progress has been achieved in integrated circuit fabrication.
- The same technology provides the basis for micro/nano fabrication of superconducting quantum chips

Semiconductor IC Fabrication "Toolkit"

- **Insulating Layers**
  - Oxidation, nitridation
  - Deposition (LPCVD, PECVD, APCVD)
- **Selective doping of silicon**
  - Diffusion (in-situ doping)
  - Ion implantation
  - Epitaxy (in-situ doping)
- **Material deposition (silicon, metals, insulators)**
  - LPCVD
  - PECVD
  - PVD
- **Patterning of Layers**
  - Lithography (UV, deep UV, e-beam & x-ray)
- **Etching of (deposited) material**
  - Dry etches—plasma, RIE, sputter etch, DRIE
  - Wet etches—etch in liquids, CMP etc

LPCVD: low pressure chemical vapor deposition.

PECVD: plasma enhanced CVD.

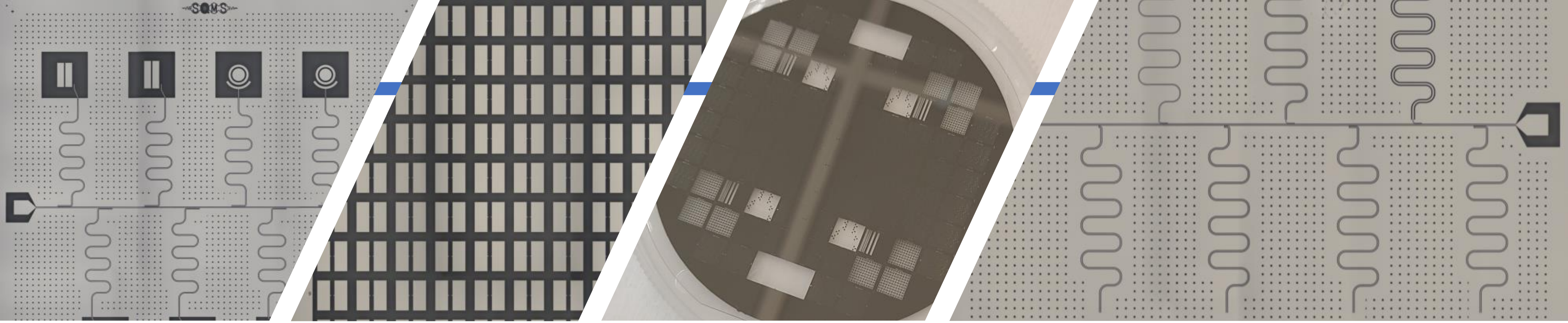
APCVD: atmospheric pressure CVD

RIE: reactive ion etching

DRIE: deep RIE.

CMP: chemical mechanical polishing

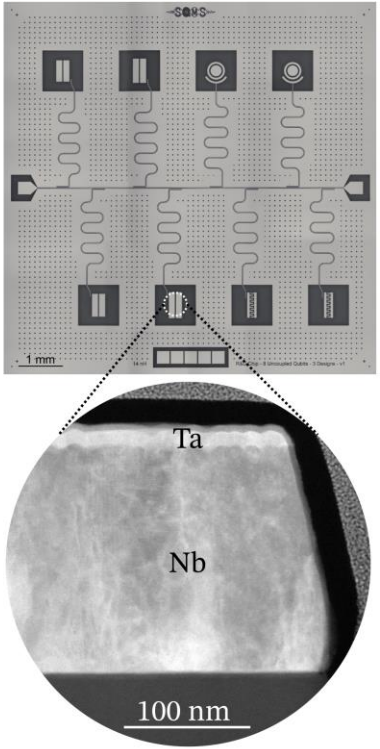
Lecture notes on micro/nano fabrication by Bo Cui, ECE, University of Waterloo; http://ece.uwaterloo.ca/~bcui/
Textbook: Nanofabrication: principles, capabilities and limits, by Zheng Cui



What processing is needed to turn a substrate into superconducting quantum devices?

Basic Processing Steps

A sequence of additive and subtractive steps with lateral patterning.



Three components for micro/nano fabrication of superconducting quantum devices:

Lithography (lateral patterning): generate pattern in a material *called resist*
photolithography, electron-beam lithography…

Thin film deposition (additive): spin coating, chemical vapor deposition, molecular beam epitaxy, sputtering, evaporation, electroplating…

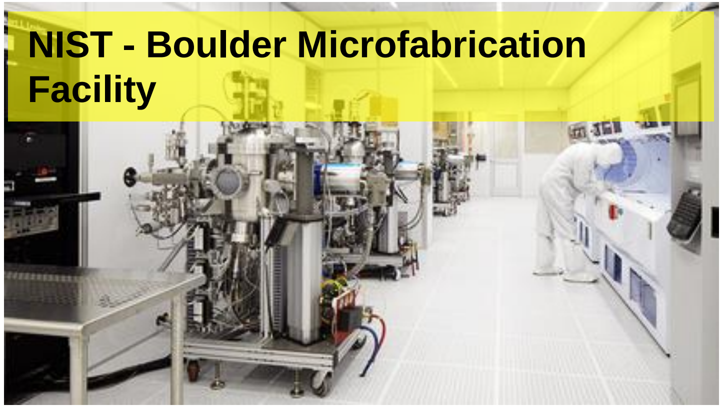
Etching (subtractive): reactive ion etching, ion beam etching, wet chemical etching…

Cleanroom

Cleanroom is cleaned by:

- HEPA filters and recirculation for the air.
- “Bunny suits” for workers.
- Filtration of chemicals and gases.
- Manufacturing protocols.

FNAL/UChicago - Pritzker Nanofabrication Facility

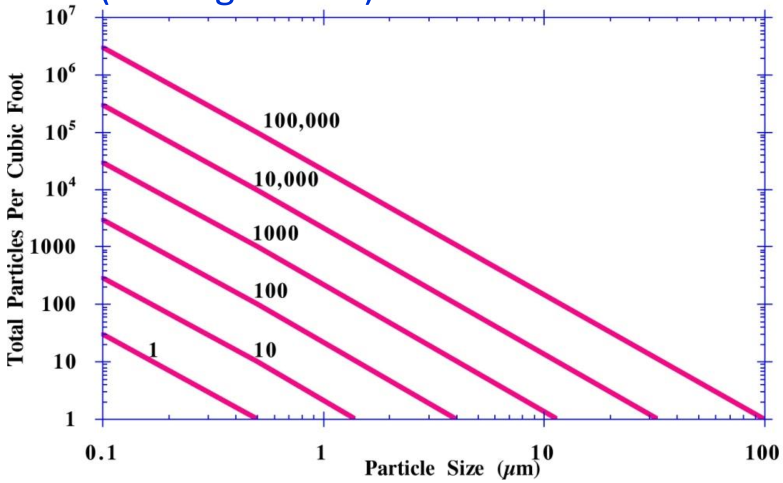


NIST - Boulder Microfabrication Facility

Rigetti - Fab-1 Integrated Circuit Foundry, Fremont CA

Class of a Cleanroom

- Air quality is measured by the “class” of the facility.
- Class 1-100,000 mean number of particles, greater than 0.5μm, in a cubit foot of air.
- A typical office building is about class 100,000.
- The particle size that is of most concern is 10nm – 10μm. Particles <10nm tend to coagulate into large ones; those >10μm are heavy and precipitate quickly.
- Particles deposit on surfaces by Brownian motion (most important for those <0.5μm) and gravitational sedimentation (for larger ones).



Particle diameter (μm)

Class	0.1	0.3	0.5	5.0
1	35	3	1	
10	350	30	10	
100		300	100	
1000			1000	7
10000			10000	70
100000			100000	700

Cleanroom Processing

Deposition Tools

Wet Processing Benches

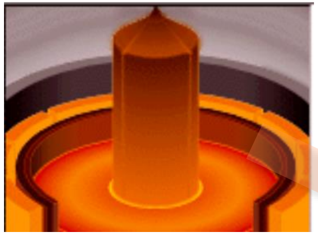
Etching Tools

Imaging & Characterization Tools

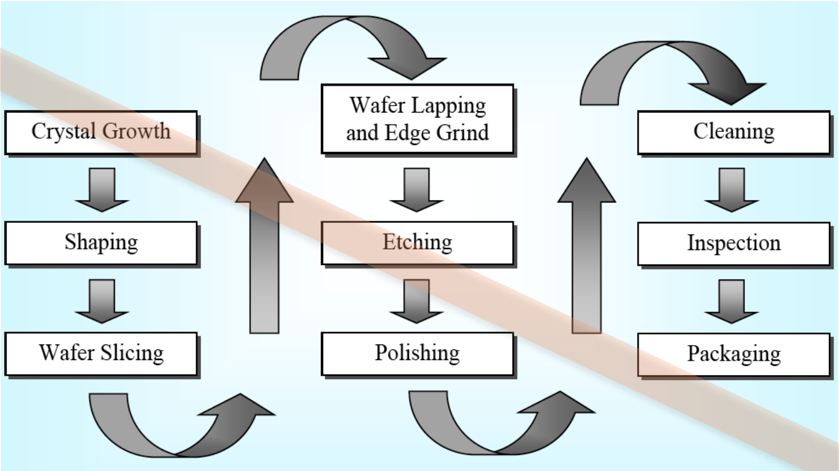
Lithography Tools

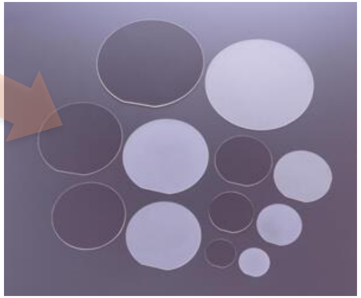
Post-processing & Packaging Tools

Substrates: Si, Sapphire,…



CZ growth





Single crystal wafers

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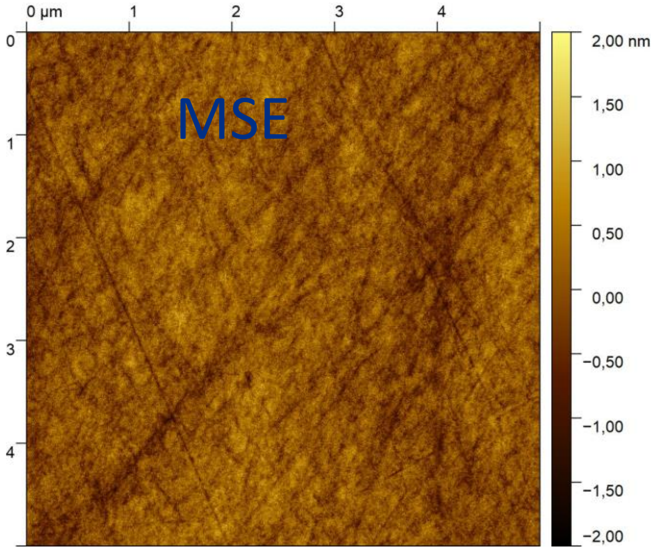
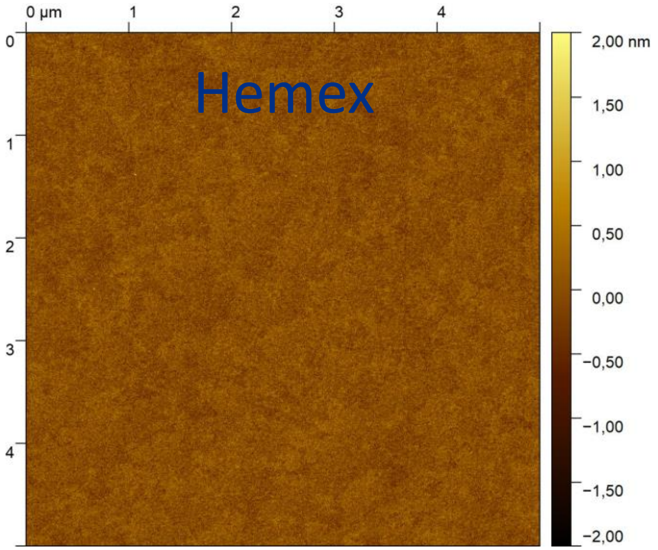
Surface of Hemex and MSE Sapphire Substrates as received





RMS Roughness = 0.35 nm
Scratched surface (as much as ~ 1nm deep)

RMS Roughness = 0.21 nm
Smooth overall

Substrate Surface Treatment/Cleaning

- Treatment/cleaning involves removing particles, organics, oxides, and metals from substrate surfaces.
- Particles are largely removed by ultrasonic agitation during cleaning.
- Organics (photoresist) are removed in O_2 plasma or in H_2SO_4/H_2O_2 (Piranha) solutions.
- The “RCA clean” is used to remove metals and any remaining organics.
- Oxides are removed by BOE, HF, or BHF solutions

Standard RCA Cleaning Procedure for Si Substrates

H_2SO_4/H_2O_2 1:1 to 4:1	120 - 150 °C 10 min	Strips organics especially photoresist
↓		
HF/H_2O 1:10 to 1:50	Room T 1 min	Strips chemical oxide
↓		
DI H_2O Rinse	Room T	
↓		
$NH_4OH/H_2O_2/H_2O$ 1:1:5 to 0.05:1:5 SC-1	80 - 90 °C 10 min	Strips organics, metals and particles
↓		
DI H_2O Rinse	Room T	
↓		
$HCl/H_2O_2/H_2O$ 1:1:6 SC-2	80 - 90 °C 10 min	Strips alkali ions and metals
↓		
DI H_2O Rinse	Room T	

Sapphire Surface Treatment

RCA SC-1:

NH_4OH(30%):H_2O_2(30%):H_2O=1:1:5; 70-80°C, 5 min, high pH.

- Oxidize organic contamination (form CO_2, H_2O…)
- Slowly dissolve native oxide and grow back new oxide, which removes particles on oxide.
- But NH_4OH etches Si and make the surface rough, thus less NH_4OH is used today.

Piranha:

H_2SO_4:H_2O_2(30%)=3:1; 50°C; 5 min, low pH.

- Strong oxidizer, removes organics.
- Could be replaced by Nanostrip.

Other Cleaning Methods:

Energy may come from plasma, ion beam, short-wavelength (UV) radiation or heating.

- HF/H_2O vapor cleaning
- UV-ozone cleaning (UVOC)
- H_2/Ar plasma cleaning
- Thermal Annealing

Thin Film Deposition: Key Parameters & Modes of Deposition

- Deposition rate, Film uniformity (across wafer & run-to-run), Film density, Defect density, Film texture, Grain size, Film conformality, Impurities
- Electrical properties of film: resistivity, dielectric characteristics…
- Mechanical properties: residual stress, adhesion…
- Optical properties: transparency, refractive index…

- Physical vapor deposition (PVD): sputtering, e-beam or thermal evaporation
- Chemical vapor deposition (CVD): metal-organic CVD, plasma-enhanced CVD, low pressure CVD…
- Epitaxy: molecular beam epitaxy (MBE), liquid-phase epitaxy…
- Electrochemical deposition: electro- and electroless plating (of metals)
- Oxidation (growth of thermal SiO_2)
- Spin-on and spray-on film coating (resist coating)

Physical vapor deposition (PVD): evaporation and sputtering

Plassys MEB550S

Evaporation:

- Material source is heated to sublimation temperature in vacuum either by thermal or e-beam methods.
- Material is vapor transported to target in vacuum.
- Easier to change evaporation material than sputtering target.

EVAPORATION	SPUTTERING
low energy atoms	higher energy atoms
high vacuum path • few collisions • line of sight deposition • little gas in film	low vacuum, plasma path • many collisions • less line of sight deposition • gas in film
larger grain size	smaller grain size
fewer grain orientations	many grain orientations
poorer adhesion	better adhesion

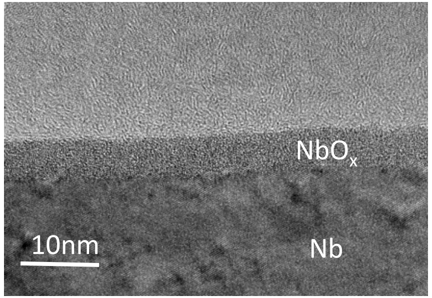
AJA ATC 2200 UHV Sputtering System

Sputtering:

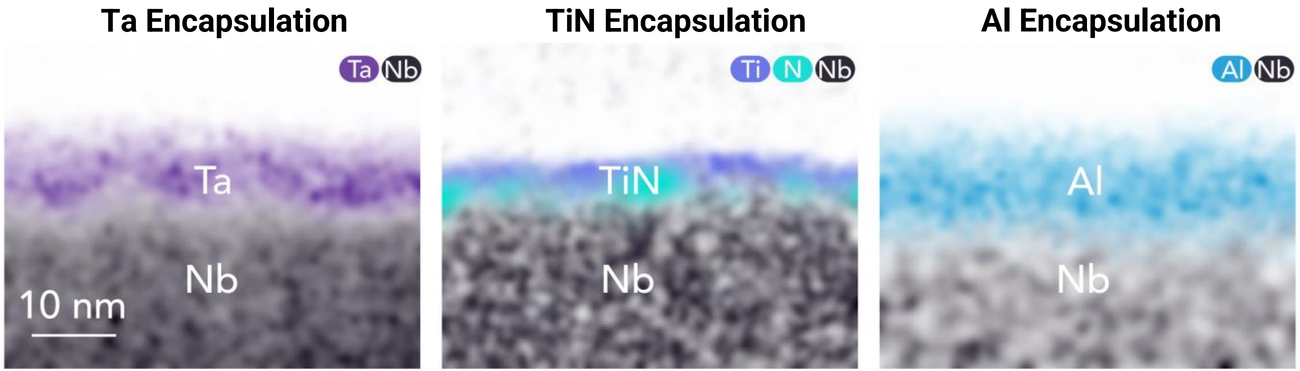
- Material is removed from target by momentum transfer.
- Gas molecules are ionized in a glow discharge (plasma), ions strike target and remove mainly neutral atoms.
- Sputtered atoms condense on the substrate.
- Not in vacuum, gas (Ar) pressure 5-50mTorr.

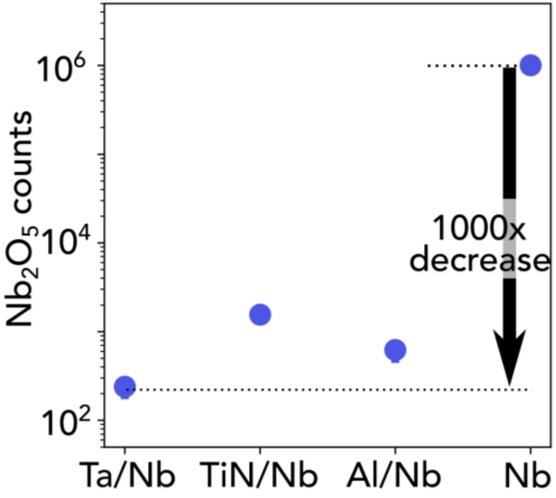
Novel Surface Encapsulation as Mitigation Strategy to eliminate Nb_2O_5

- Nb is frequently used is S.C. quantum devices. It has a surface oxide which is very lossy.



- Avoid niobium oxidation by stable surface **encapsulating layer**
  - Thin (~5-10 nm) => small contribution to conductive losses
  - But TLS-hosting dissipative surface Nb_2O_5 is absent => reduction of the TLS dielectric losses => better coherence





M. Bal et al, arXiv:2304.13257

Lithography – Patterning

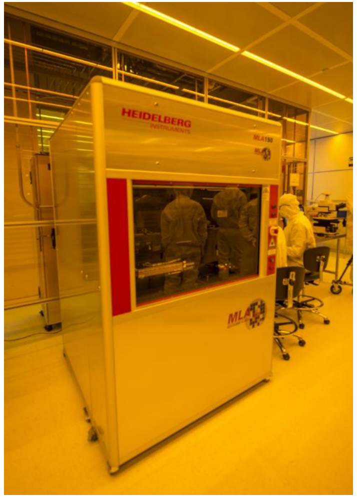
Lithography on surfaces

- Optical/UV lithography
- E-beam lithography
- FIB lithography
- X-ray lithography
- SPM-lithography
  - AFM
  - STM
  - DPN (dip-pen nanolithography)
- Imprint lithography
  - Soft lithography
  - Hot embossing
  - UV imprinting
- Stencil mask lithography

Lithography in volume

- Two photon absorption
- Stereo-lithography

Heidelberg MLA 150
Maskless aligner



- Critical dimension (CD) control

  Size of features must be controlled within wafer and wafer-to-wafer

- Overlay (alignment between different layers)

  For high yield, alignment must be precisely controlled

- Defect control

  Other than designed pattern, no additional patterns must be imaged

Raith EBPG5200 E-Beam lithography system (100 kV)

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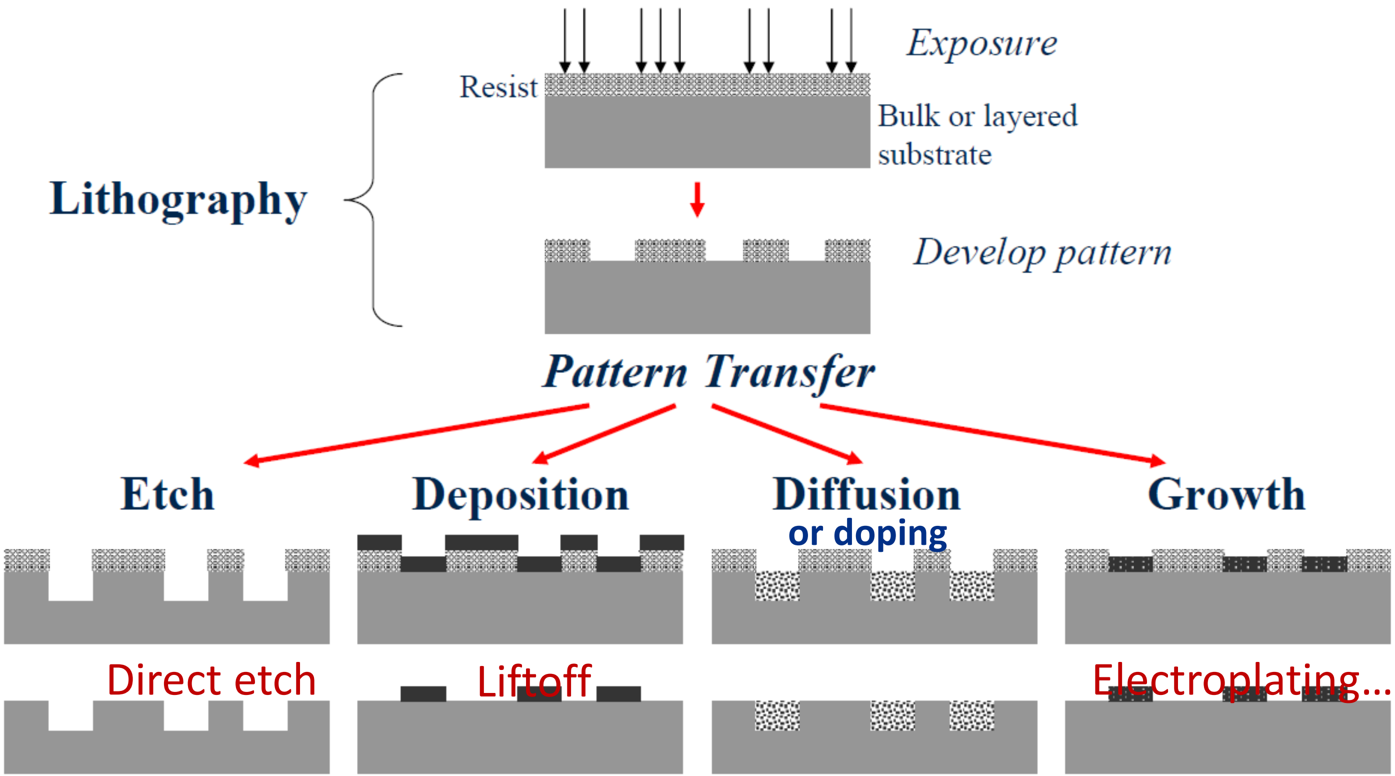
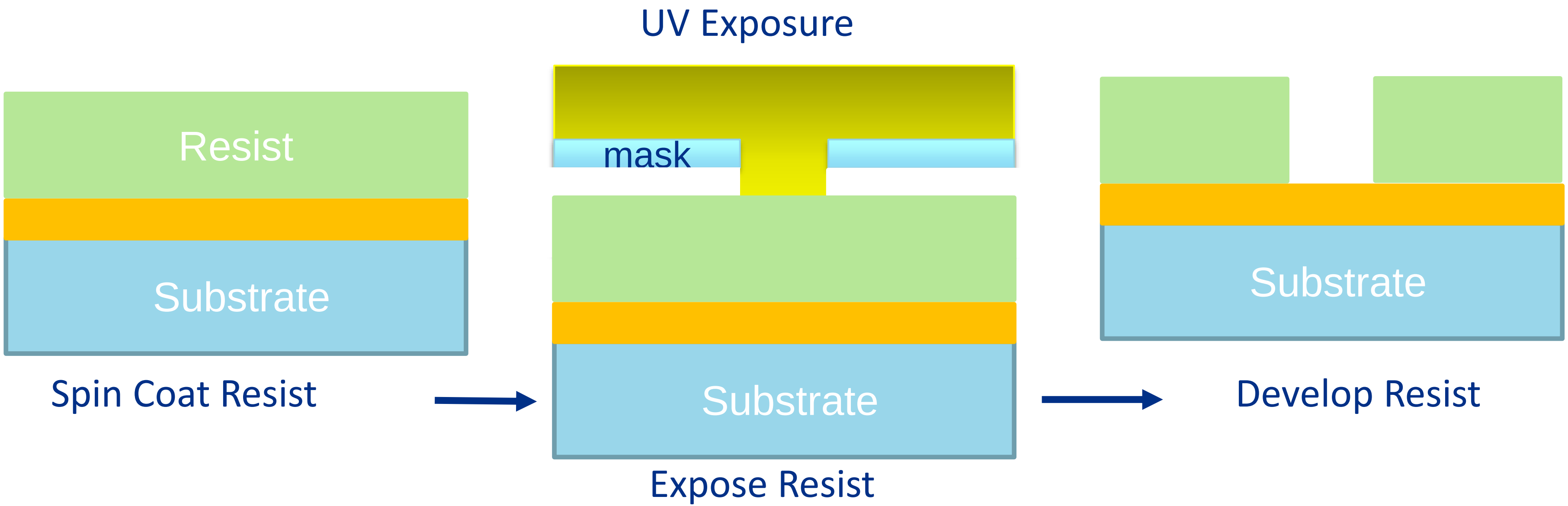
Pattern transfer (next step after lithography)

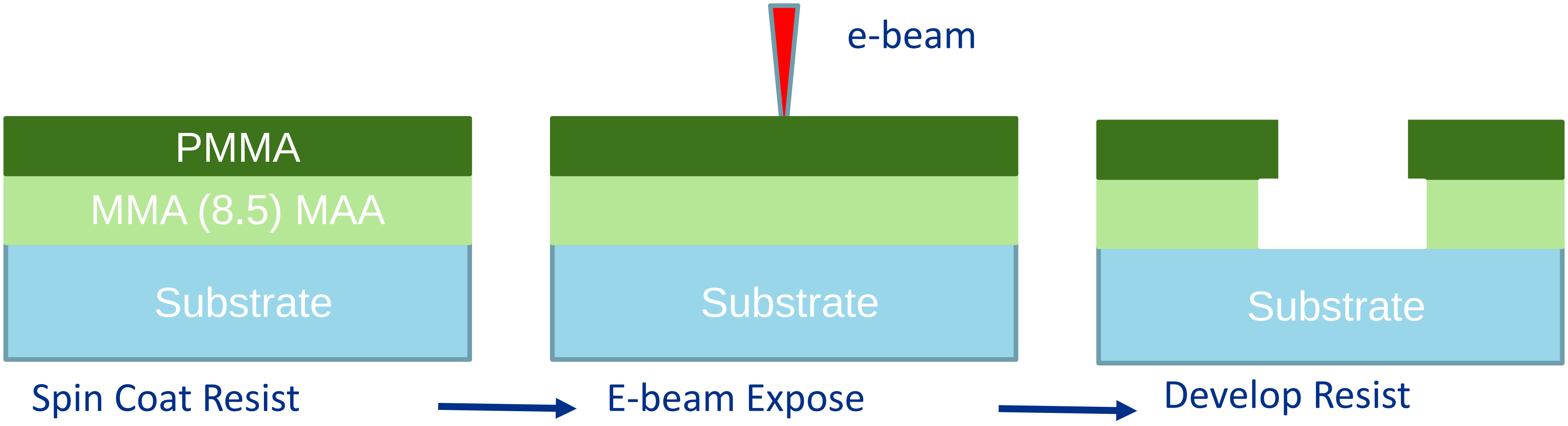


Photolithography



- Process used to transfer a pattern from a photomask to the surface of a substrate (Nowadays maskless aligners are widely available as an alternative)
- Formation of images with visible or ultraviolet radiation in a photoresist
- No limitation of substrate (Si, glass, metal, plastic...)
- For R&D, it is the most widely used lithography system, but with ~1μm feature size, so only for *micro*-fabrication.

Electron Beam Lithography



- Electron beam has a wavelength so small that diffraction is insignificant.
- Tool is just like an SEM with on-off capability controlled by a “beam blanker”.
- Accurate positioning (alignment): “see” the substrate first, then expose.
- Beam spot diameter of 2nm can be achieved, at typical acceleration voltage of >20keV.
- Interaction of electrons and resist leads to beam spreading
- But typical resolution ~15nm (>> beam diameter), limited by proximity effect and lateral diffusion of secondary electrons.
- Direct write technique (no mask)

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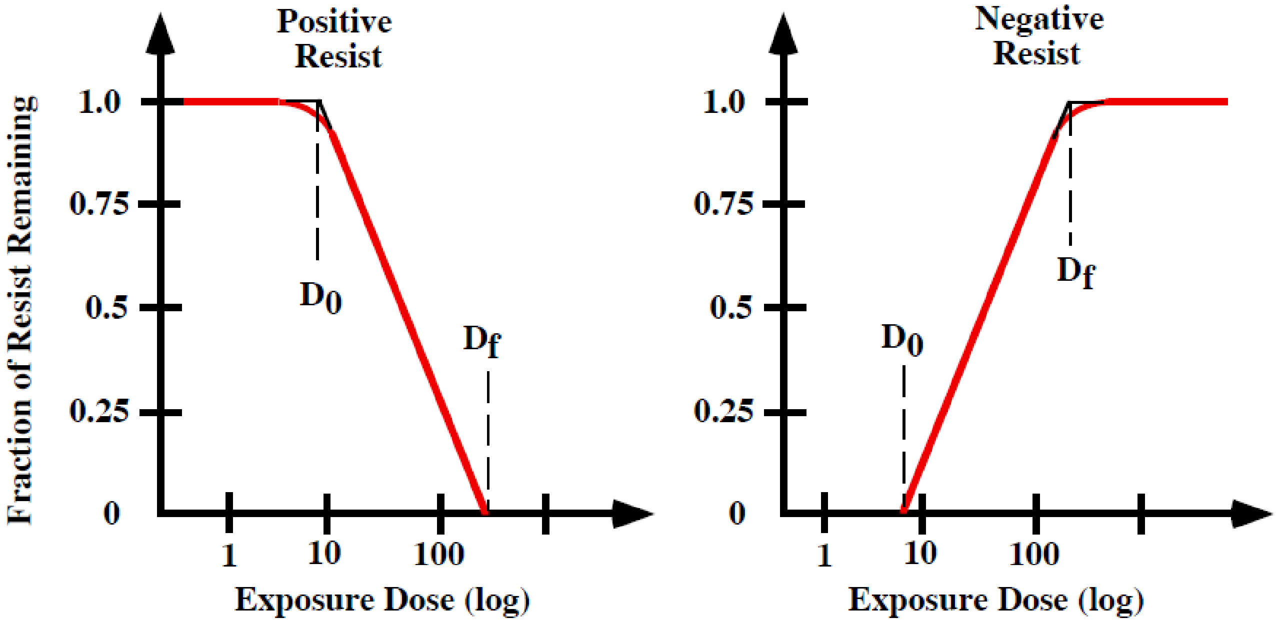
Resist Contrast & Sensitivity

Contrast γ is defined as:

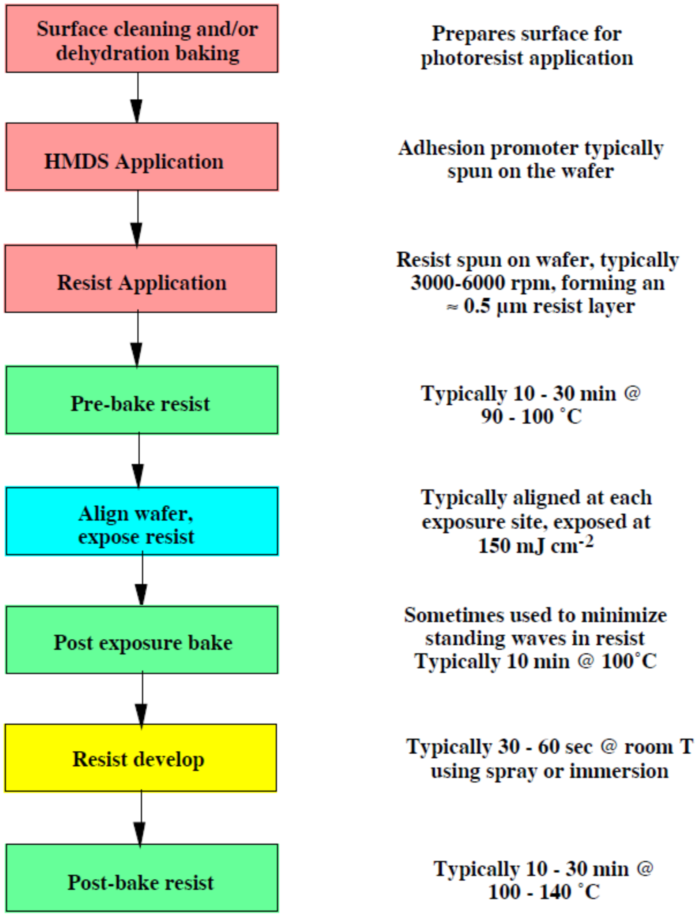
$$\gamma = \frac{1}{\log_{10} \frac{D_f}{D_0}}$$

- γ and D_f are not intrinsic properties of the resist - they depend on process conditions (developer, development time, baking time, λ, substrate…).

D_f is Sensitivity.



Typical Process Flow for Photolithography

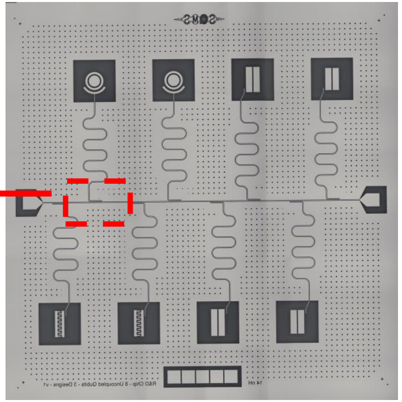


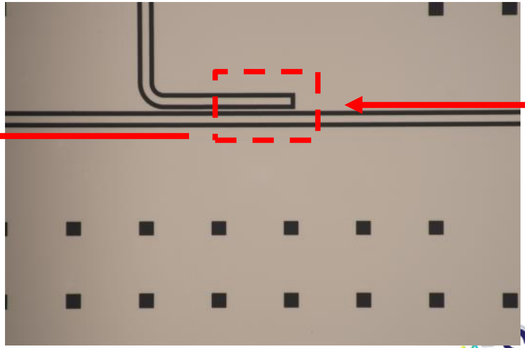
Process Flow to Define Qubit Circuitry on Sapphire

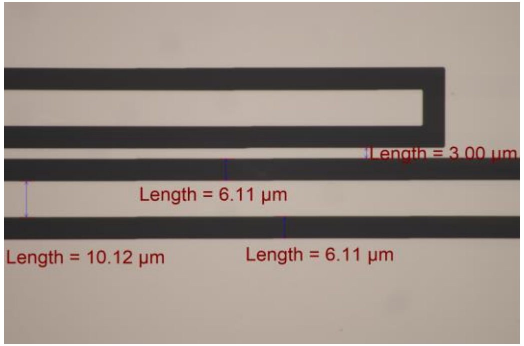
Substrate Preparation
- Solvent Clean
- RCA SC-1 Clean

Film Deposition
- Sputter Nb film
- Deposit 5-10 nm encapsulation layer
  - Ta & Al encapsulation in situ (sputter)
  - TiN encapsulation ex situ (ALD)

Patterning Film
- Photolithography
- Dry Etch







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Subtractive Process: Etching

Etching is done either in "dry" or "wet" methods:

- Wet etching uses liquid etchants with wafers immersed in etchant solution.
- Wet etch is cheap and simple, but hard to control (not reproducible), not popular for ***nanofabrication*** for pattern transfer purpose.
- Dry etch uses gas phase etchants in plasma, both chemical and physical (sputtering process).
- Dry plasma etch works for many dielectric materials and some metals (Al, Ti, Cr, Ta, W…).
- For other metals, ion milling (Ar^+) can be used, but with low etching selectivity. (as a result, for metals that cannot be dry-etched, it is better to pattern them using liftoff)

Etching is consisted of 3 processes:

- Mass transport of reactants (through a boundary layer) to the surface to be etched.
- Reaction between reactants and the film to be etched at the surface.
- Mass transport of reaction products from the surface through the surface boundary layer.

Figures of merit: etch rate, etch rate uniformity, selectivity, and anisotropy.

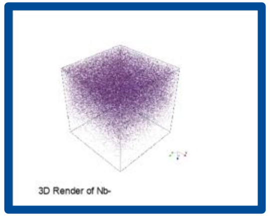
Plasma-Therm ICP RIE

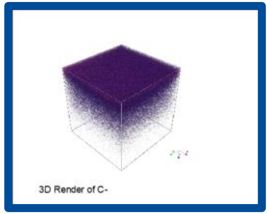
Advantages/Disadvantages of Dry Etching

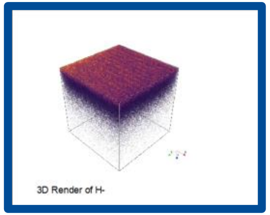
Dry etching advantages

- Eliminates handling of dangerous acids and solvents
- Uses small amounts of chemicals
- Isotropic or anisotropic/vertical etch profiles
- Directional etching without using the crystal orientation of Si
- Faithful pattern transfer into underlying layers (little feature size loss)
- High resolution and cleanliness
- Less undercutting
- Better process control

Fl-based Dry Etch Chemistry
(Nb and Ta encapsulated Nb Films – 1st Round)







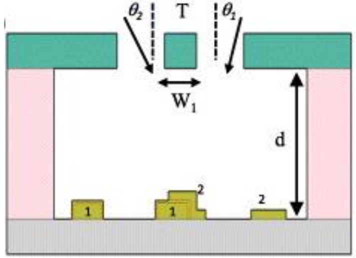
Dry etching disadvantages:

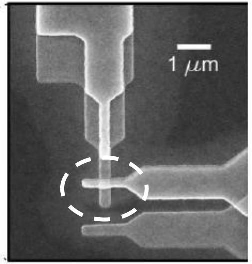
- Some gases are quite toxic and corrosive.
- Re-deposition of non-volatile compound on wafers.

Liftoff Process to Fabricate Submicron Al/AlOx/Al Junctions

Shadow evaporation with tilt, "Dolan bridge" – Single pattern step, single pumpdown

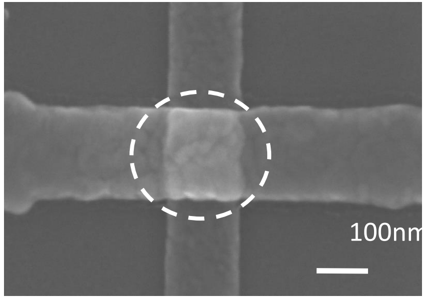
1) Pattern, deposit 1st layer, oxidize, tilt, deposit 2nd layer





Overlap junction - 2 patterns, pumpdowns

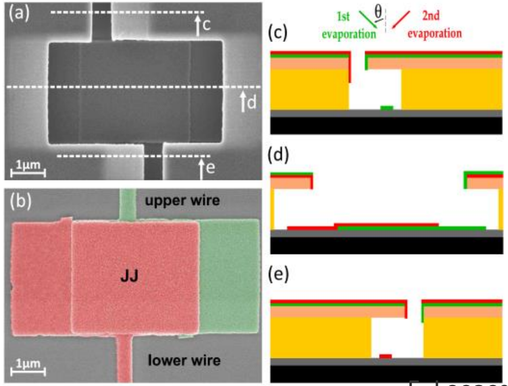
1) Pattern
   pumpdown
   LO BE
2) Pattern
   pump down
   mill BE & oxidize
   LO TE

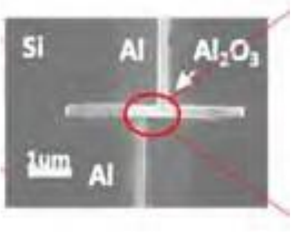


X. Wu, et. al APL (2017)

M. Steffen et. al, PRL97, 050502 (2006)

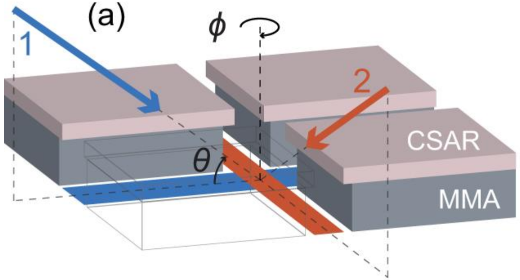
"Bridgeless" – shadow evaporatioin high aspect tilt

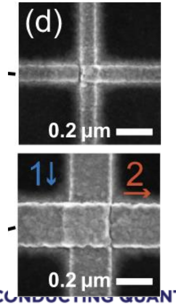




F. Lecocq et al Nanotechnology (2011)

"Manhattan style" – shadow evaporation Use rotation between the two depostions and oxidation



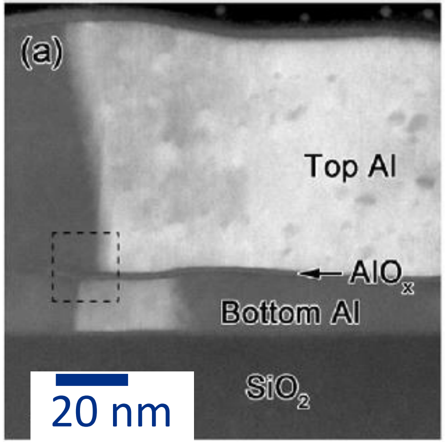


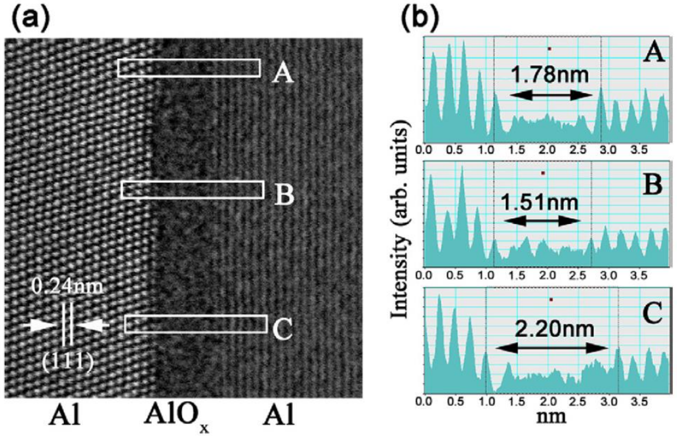
J M Kreikebaum *et al* 2020 *Supercond. Sci. Technol.* **33** 06LT02

Josephson junctions - sensitive to atomic-level defects

- Aluminum oxidation is conformal, ~ 1.8 nm thick Al/AlOx/Al
- Thickness variations with exponential dependence of current
  - Less than 10% of total barrier active
  - Strong inhomogeneity of tunnel current across junction
- Amorphous materials are lossy – make junctions small

Cross-section TEM

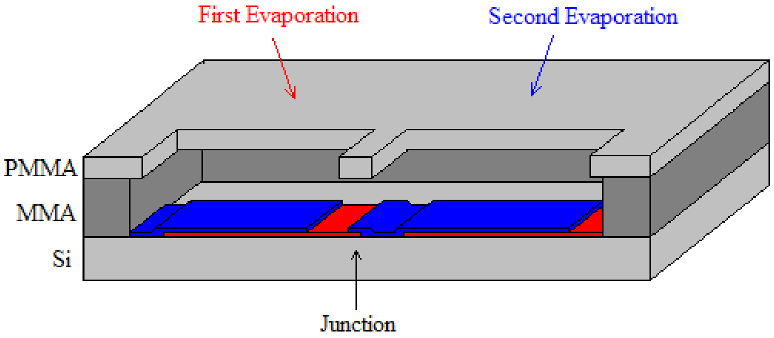




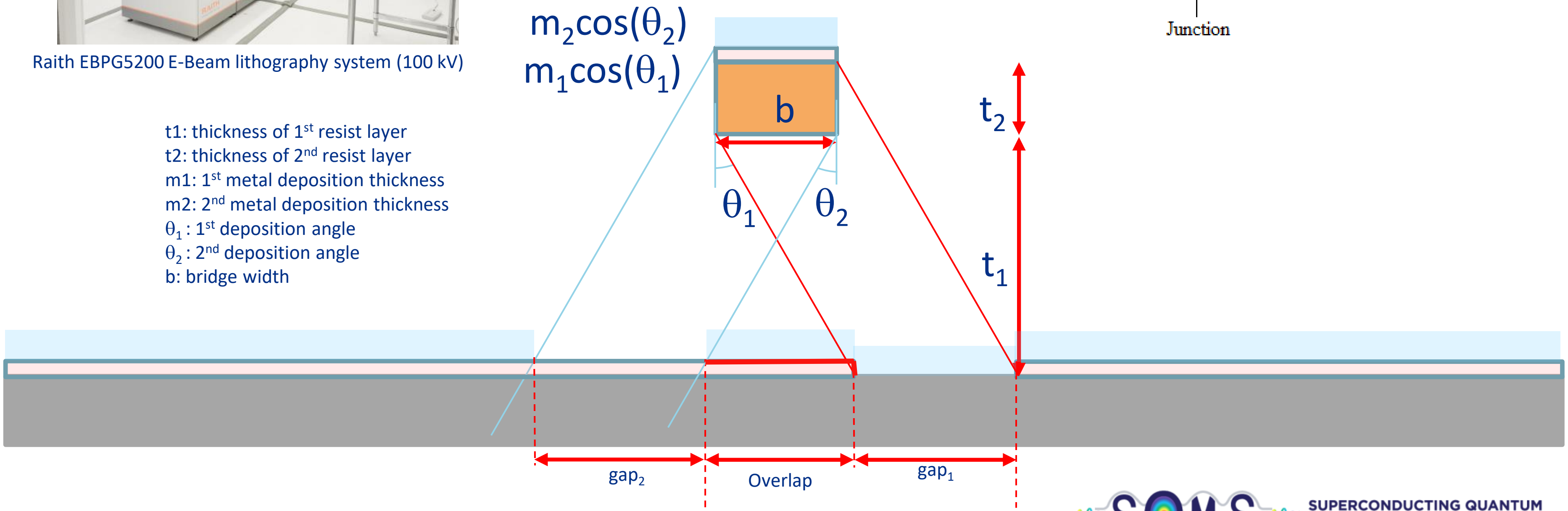
Zeng, J. Phys. D: Appl. Phys. **48** (2015) 395308

Dolan Bridge Double Angle Shadow Evaporation

Raith EBPG5200 E-Beam lithography system (100 kV)



t1: thickness of 1st resist layer
t2: thickness of 2nd resist layer
m1: 1st metal deposition thickness
m2: 2nd metal deposition thickness
θ_1 : 1st deposition angle
θ_2 : 2nd deposition angle
b: bridge width

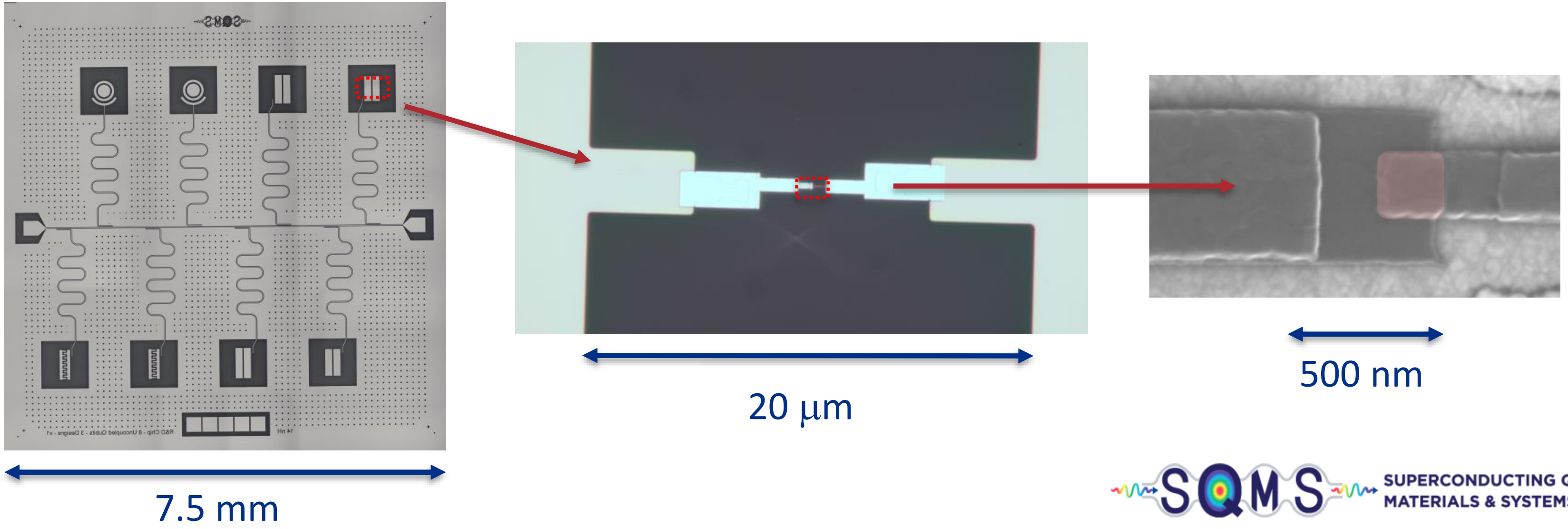


SUPERCONDUCTING QUANTUM MATERIALS & SYSTEMS CENTER

Josephson Junction Deposition at FNAL

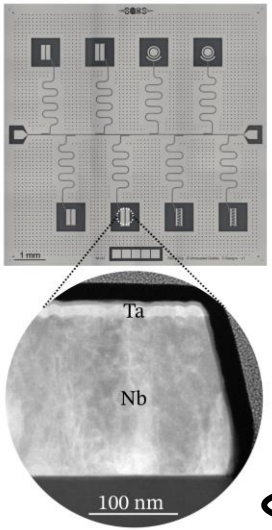
- Al/AlOx/Al Junctions are deposited at +22 / -22 degree angles relative to the normal of the substrate.
- 2'15" (45"/45"/45" at +60/0/-60 degree) Ar ion milling to remove oxide on Nb.
- Bottom/Top electrode thicknesses are 40 nm/90 nm.
- The Oxidation is 20 mBar for 12 minutes (Ar/O2 (85/15) mixture)
- Typical Junction area is approximately 200 nm x 200 nm

Plassys MEB550S



SUPERCONDUCTING QUANTUM MATERIALS & SYSTEMS CENTER

SQMS is at the Forefront of Qubit Coherence



Top published transmon qubit coherence

Group	Best T_1 (μs)	Freq. (GHz)	Substrate	Primary Material	Publication Year
Yu	503	3.8-4.7	Sapphire	Ta, dry etch	2022
SQMS	**451**	**4.5-5**	**Silicon**	**Ta/Nb, dry etch**	**2023**
Houck	360	3.1-5.5	Sapphire	Ta, wet etch	2021
IBM	340	~4	Silicon	Nb, dry etch	2022
IBM	234	3.808	Silicon	Al, dry etch	2021
SQMS	**198**	**4.5-5**	**Sapphire**	**Ta/Nb, dry etch**	**2023**

arXiv:2304.13257v2