Superconducting Nb-AlOx Process Development

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Introduction

Greetings, fellow traveler! This page is part of a larger project, with the ultimate goal of creating a Josephson Junction Voltage Standard at OV. Information on this kind of standard can be found on the Josephson Junction Voltage Standard- Project page.

A silicon CMOS process is while on one hand far more useful, a lot more complex and harder to achieve good enough tolerances for that a useful chip can realistically be designed. A bipolar transistor is in many ways easier to manufacture, but good doping gradients require high temperature thermal cycling if any kind of diffusion is to be preformed. Also, there is a vast ocean of regular semiconductor devices that can be readily ordered online, they just aren't that fun.

Superconductors are strange materials. Capable of conduction at effectively zero resistance, able to hold indefinite currents, a macroscale quantum mechanical phenomenon and still not properly understood in the case of high temperature superconductivity. This is way more fun!

Superconducting tunneling junctions are especially interesting devices as they under correct biasing can act like perfect Frequency-to-Voltage converters, a property which is today used as worldwide to realize a non-physical primary standard for the Volt.

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Most basic superconducting circuits can be made with little more than a

Deposition: All preformed with E-Beam system in mor di.

- All materials in question are easily deposited with an electron beam
- Substrate movement only required for

Even though

Lithography: Hop

at OV can not only do basic deposition, but microcircuit fabrication and superconductor experimentation. I (HT) do not know of any simpler process we might use to accomplish this, the costs involved are minimal and virtually no additional equipment is required.
It will however be potato-quality and cause nanolab engineers to roll of their chairs laughing.

Stage 1: Simple Nb film deposition, measurements and basic optimization.

Fulfillment needs

- Mor Di:
  - [x] Operational Water Cooling System
  - [x] Operational Power System
    - [x] Largely Done
  - [x] Operational Helium Cooler

- [x] Has been thoroughly inspected
- [x] Has been power tested
- [x] Has been operationally verified
- [x] Has NOT had performance verification
- [ ] May need Helium Fill

- [x] Operational Control System
    - [x] Almost completely verified. Timers, setpoints and program cycles all operate as normally intended.
    - [x] Has been through many dry runs already
    - [x] Hasn't done entire Cycle

- [ ] Operational E-Beam Supply
    - [x] Not yet powered up
    - [ ] Has had multiple issues w/ either scan controls or disrupted beam optics

- [ ] Working Chamber Mechanics
    - [ ] Pneumatics issues must be investigated.

- [x] Functioning Quartz Balance

- Other Equipment
    - [ ] Multimeter
    - [ ] Repeatable Kelvin Probes
    - [ ]
- Supplies
    - >2 Pyrolytic Carbon Crucibles, : ~€180 each from kjlc
    - Pure Nb Pellets; €102.00 for 50g from KJLC
    - AlOx Pellets/shards
    - Nb Pellets, €102.00 for 50g from kjlc: https://www.lesker.com/newweb/deposition_materials/depositionmaterials_evaporationmaterials_1.cfm?pgid=nb1
    - SiO2 Pellets, €28 for 100g from kjlc: https://www.lesker.com/newweb/deposition_materials/depositionmaterials_evaporationmaterials_1.cfm?pgid=si2
    -
    - 2" Silicon Wafers: 60kr each from NTNU Nanolab Supplies

Important measurements

MUSTs

- Chamber temperatures
    - Water temperatures
    - Substrete Temperatures
- Chamber Pressure
- Deposition Rate

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- Approximate Film Thickness

Requirements

- OV

Useful knowledge

- A Characterized deposition rate curve, assuming some repeatability efforts are taken
- A full operational verification of Mor Di: All systems now both known and tested to be operational.

Stage 2: Recipe Design; Film Inspection.

After having Verified the deposition process in Stage 1, A recipe for Nb/AlOx- epitaxy can now be designed. Several test depositions are required and need careful stydying, the electron microscope may come very much in handy for this.

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Proper lithography, process control and maybe NbN- sputtering to better layer adhesion and superconducting properties.

Will probably need to access nanolab and have some backing from Timini Labs or a technician, but not an unrealistic step up.

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Stage 3: Basic Lithography

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Stage 3A: (Photo)mask Application

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Stage 3B: Development

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Stage 3C: Deposition (In case of lift-off)

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Stage 3D: Feature Extraction: Plasma Etching/Lift-Off

After the layer has been deposited and exposed photomask has been applied, the features now need to be made.

The most common and best looking result will be achieved through etching, but Nb and AlOx pretty much need Hydrofluoric acid, an extremely potent, toxic, bone seeking acid that can be found in small amounts in some drain cleansers and rust removers, but due to the inherent danger massive safety precautions are required, far more than are realistically appliccable at OV. Besides, wet etching is not a highly repeatable process, and even etch gradients are difficult to achieve as well as hard to maintain, good chemical process control is rather important.

Reacitve Plasma Etching is another technique that is frequently used today in semiconductor processes. One fills a chamber with a low pressure inert gas, and applies a strong RF electric field between two electrodes, one of which acting as a pedestal for the wafer. The resulting plasma once ignited will be full of decomposed radicals from the feed gas, and if one uses a fluorocarbon feed gas this will decompose into among other things, free fluorine ions which are extremely reactive and will readily etch nearly any surface. If this gas is emptied through a readily reactive filter, the fluorine atoms will react with the surface of the filter and become inert again, if a filtering system is properly implemented no dangerous gasses will be released (though the gasses will probably have a very high greenhouse potential, FC gasses are known for this)

This kind of a plasma etch is the first of two practical implementations for use here at OV, and is doable within reason although still quite labour intensive. A dedicated chamber must be built with a high voltage feedthrough, a load lock or sample door,

There is an even easier way, although it is a tad messy. Lift-Off is exactly what the name implies. You deposit material on top of the photomask, and once done, you dissolve away the mask, destroying the film on top in the process. Some light brushing may be required.

Lift-Off does have it's issues: the photomask must be inside the vacuum chamber where deposition occurs and may be exposed to high heat, if the deposited film along the rims of the photomask is too thick and is elastic it may rip and tear unevenly, and may even cause partial or full delamination of the desired film patterns on the substrate.

Stage 4: Cleaning for next deposition

In the case of etching there is merely photo-mask, maybe containing some degraded etched material around edges, to be removed, typically through a wash in some reasonably aggressive solvent.

If however a lift-off step has instead been preformed, both cleaning and potentially surface treatment need to be preformed. Lifting off material will inevitably cause imperfect edges as the deposited material has just been ripped off. If the material is ceramic or a glass, there will probably be some small brittle edges remaining, but ears are generally not to be expected. This is sadly not the case for metals and other plastically deforming materials, as these will often leave long ears and bad edges. If there is a good mask aspect ratio and low deposition isotropy these ears will be far thinner than the bulk material, yielding a good weak point for proper detachement. For thicker edges this may be less doable.

Stage 4: Junction Testing.

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Method for preparing soldered pad connections to Nb- Pads with indium solder & Low temp soldering iron:

https://www.sciencedirect.com/science/article/pii/001122759592884U/pdf?md5=b061acc3d6cb04d9d46e2f3943ea614b&pid=1-s2.0-001122759592884U-main.pdf&_valck=1

Links:

- KJLC Notes on AlOx deposition https://www.lesker.com/newweb/deposition_materials/deposition-materials-notes.cfm?pgid=al6