3.6.1 Niobium Ground Plane

We begin with an oxidized silicon wafer. The manufacturer of these wafers produces these wafers in a clean environment, and they are double sealed to ensure they are not contaminated with particulates in transit. We have therefore found that the best strategy in our working environment is to do as little to the wafers as possible before depositing the first layer of metal, rather than trying to do a solvent rinse of any kind. When this thesis work was first started, standard operating procedure was to mount a wafer in an uncontrolled environment (as mentioned in Section 3.2.3) and then blow the surface of the wafer with compressed, dry nitrogen. It quickly became clear that this situation was poorly suited to fabricating devices with large active areas with any appreciable yield. With the installation of the flow hood, our yield improved significantly. Our yield was further improved after realizing the nitrogen blowing step was actually adding a significant amount of particulate contamination to the wafer surface, rather than clearing it off. In retrospect, it should have been obvious that a regularly maintained HEPA or ULPA filter would need to be installed at the output of the gun to prevent such a situation. Our deposition process proceeds as follows:

1. Put on a face mask, head covering, and eye covering (goggles or glasses).

2. Clean critical surfaces and tools inside the flow hood with isopropyl alcohol and a cleanroom wipe

3. Wipe off outer surfaces of sputter system near loadlock using isopropyl alcohol and a tekwipe

4. Vent sputter system load lock with dry nitrogen

5. Quickly transfer sample mount out of loadlock and into flow hood, then quickly close loadlock door to keep contamination out

6. Retrieve fresh oxidized silicon wafer from wafer boat

7. Center Wafer on sample mount, polished side up, and mount in place with aluminum ring

8. Holding the sample mount (and sample) upside down to minimize contamination, quickly transfer sample from flowhood into load lock of sputter system. Close loadlock and pump down as quickly as possible.

9. Follow standard pumpdown procedure and transfer sample into main chamber

10. Use argon ion mill to prepare substrate surface for deposition (improves metal adhesion to the oxide surface). Mill for approximately 15 seconds (removes roughly 7–10 nm of oxide).

11. Deposit 40 nm of niobium following the recipe shown in Table 2.

12. Transfer wafer into loadlock

13. Vent, following standard procedure

14. Remove wafer from mount, load into plastic wafer carrier. Double seal wafer carrier in plastic bags for transit to WCAM.

After transferring the wafer into the WCAM, it is time to pattern our ground plane features as follows:

Kurt Lesker Sputter System	
Argon Pressure	3.9–5 mTorr
Target Size	3 inch
Fixed Parameter	Power
Cleaning Power	400W
Deposition Power	500W
Deposition Rate	40–50 nm/min

Table 2: Niobium Deposition Parameters: The argon pressure is adjusted periodically to maintain a slight compressive stress in the niobium films[50]. The ideal pressure drifts over time, and changes based on how much material has been sputtered off of the niobium target.

1. Clean wafer of any contamination it collected while being handled in the non-clean environment of our labs by running it through a spin rinse dryer (SRD).
2. Spin coat the wafer with SPR-955 CM.7, spinning at 3000 rpm
3. Prebake the wafer at 100 °C for 90 seconds
4. Expose ground plane pattern in Body 8 stepper.
5. Post bake at 110 °C for 90 seconds
6. Develop in MF-CD26 developer for 60 seconds
7. Rinse developer off in DI water beaker for 60 seconds
8. Rinse wafer under flowing DI water for 30 seconds

9. Bake wafer at 125 °C for 180 seconds to reflow PR, creating sloped sidewalls
10. Examine wafer under microscope to verify pattern transferred correctly

After the lithography is complete, the ground plane needs to be etched. This process is performed in the Unaxis 790 RIE tool. The procedure for this process is as follows:

1. Preclean chamber with 10 minute, 500W O_2 Plasma
2. Preseed chamber with 5 minute run of plasma recipe shown in Table 3
3. Vent chamber, load sample, pump down
4. Run plasma recipe in Table 3 for 50 seconds
5. Vent chamber, retrieve sample, and check under microscope. Etched regions should appear blue.
6. Pump down sample chamber
7. Post clean chamber with 5 minute, 500W O_2 Plasma

After etching the ground plane, it is necessary to very thoroughly strip all of the leftover PR and any other organic byproducts which have been left behind by previous processing. This is generally done in the following way:

1. Preheat beaker full of Microposit Remover 1165[3] to 75 °C
2. Perform coarse strip of PR residue with flowing acetone (this helps conserve 1165 by getting the bulk of the easily removable PR off ahead of time)

[3]Specialized resist stripper which mostly consists of n-methyl-pyrrolidinone (NMP)

Unaxis 790		
Chamber Pressure		40 mTorr
Process Gas Flow	SF_6	15 sccm
	O_2	20 sccm
RF Power		150W
Etch Rate		90 nm/min

Table 3: Niobium Ground Etch Parameters

3. Without allowing the wafer to dry at all, submerge it in the beaker of hot 1165. Soak in hot 1165 for 30 minutes.

4. Quickly transfer wafer into beaker full of room temperature acetone without allowing it to dry at all in the process.

5. Sonicate wafer in beaker full of acetone for 15 minutes

6. Quickly transfer wafer into beaker full of de-ionized (DI) water, sonicate for 10 more minutes

7. Remove wafer, quickly place under ample supply of flowing DI water. Rinse in this way for 2 minutes

8. Quickly load wafer into SRD[4]

9. Unload, examine under microscope to check for any particulate contamination of solvent residue (which appears as discolored streaks on the wafer in an optical

[4]Spin rinse dryer

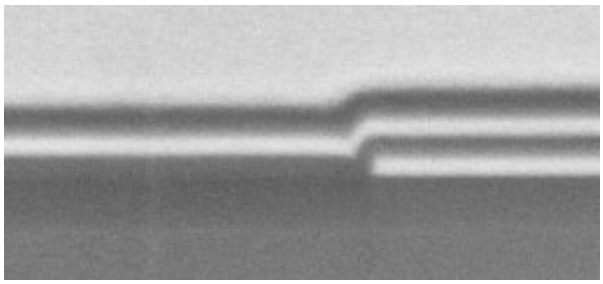

Figure 3.7: Cross Section of an SCDLD: The lowest bright layer is the ground plane of our detector. This layer is etched with the sloped sidewall process to improve the step-coverage of the detector layer which is the next bright color layer. The dark layers around these two are silicon nitride. On top of everything is a platinum layer which is only there to aid in imaging the device. Thanks to Edward Leonard who took this image on a Zeiss Focused Ion Beam-Scanning Electron Microscope.

microscope)

It is now time to deposit the dielectric layer which separates the ground plane from the detector wire(s). This is performed in the PT-70 as follows:

1. Heat the sample platen in the deposition chamber to the deposition temperature (250 °C)
2. Run plasma preclean process (O_2 and CF_4) for 10 minutes
3. Preseed by running deposition recipe (shown in Table 4) on empty chamber for 10 minutes
4. Vent deposition chamber. While waiting for chamber to finish venting, run wafer through SRD to clear any particulates that may have collected since its last cleaning

5. Load sample onto sample platen in deposition chamber, pump down chamber to base pressure

6. Run deposition recipe in Table 4 for 20 minutes

7. Vent, remove sample– set sample on a metal surface to cool before putting it back in plastic carrier

8. Run post clean plasma (same as preclean)

9. Meanwhile, examine wafer under microscope. In particular, make sure there are no obvious pinholes in the film. There should be no streaks in the color of the film either— if there is, solvent residue was trapped under the film and the yield for this wafer is likely to be very low.

PT-70		
Chamber Pressure		400 mTorr
Sample Temperature		250 °C
Process Gas Flow	N_2	750 sccm
	2% Silane (in N_2)	83.3 sccm
	5% Ammonia (in N_2)	200 sccm
RF Power		100W
Deposition Rate		7 nm/min

Table 4: Silicon Nitride Deposition Parameters

We do not perform any lithography on this dielectric at this point. Doing so only adds unnecessary complexity to the process and more opportunities for solvent residue

or particulate contamination to cause issues later on. At this point, if a multi-wafer lot is being run, it is advisable to split the lot into multiple parts as the chances of success on a single run drop drastically from here on out. Typically, we begin a run with 4 wafers and at this point start processing them individually.

After finishing the silicon nitride deposition, the wafer should be run through the SRD, loaded into the wafer carrier, and then double bagged for transfer back to the deposition system in our lab. We then deposit another 40 nm of niobium following the procedure previously outlined. This is the detector layer. We then transfer the wafer back to the WCAM for more processing.

The lithography process for this layer is slightly modified:

1. Clean wafer of any contamination it collected while being handled in the non-clean environment of our labs by running it through a spin rinse dryer (SRD).

2. Spin coat a test wafer which also has a niobium layer on top with the photoresist (PR) SPR-955 CM.7, spinning at 5500 rpm

3. Prebake the test wafer at 100 °C for 90 seconds

4. Using a fine, clean cloth and isopropyl alcohol, wipe off the wafer stage in the stepper to remove particles which can be detrimental to the focus performance of the stepper.

5. Perform a focus exposure matrix test[5] on the test wafer using the detector pattern in Body 8 stepper.

[5]This test automatically varies the focus offset and exposure dose from die to die across a wafer, allowing the user to determine which conditions are optimal at that moment for that particular pattern exposure (different parts of the mask often have different optimal focus offsets)

6. Post bake the test wafer at 110 °C for 90 seconds
7. Develop in MF-CD26 developer for 60 seconds
8. Rinse developer off in DI water beaker for 60 seconds
9. Rinse wafer under flowing DI water for 30 seconds
10. Carefully inspect the test wafer under a microscope and determine which exposure/focus offset combination yielded the best pattern.
11. Spin coat the real wafer with SPR-955 CM.7, spinning at 5500 rpm
12. Prebake the real wafer at 100 °C for 90 seconds
13. Expose detector pattern in the stepper, using the optimal focus and exposure parameters found previously.
14. Post bake at 110 °C for 90 seconds
15. Develop in MF-CD26 developer for 60 seconds
16. Rinse developer off in DI water beaker for 60 seconds
17. Rinse wafer under flowing DI water for 30 seconds
18. Examine wafer under microscope to verify pattern transferred correctly.

After this is complete, the detector pattern needs to be etched into the metal. This step is critical to the success or failure of a wafer. The recipe used in etching the niobium ground plane is a standard recipe used in many processes in our group for etching niobium. However, in this work, we found that etching wires with a small

cross section (w <2 µm) this etch process severly degrades the measured critical current density of even moderately long wires (longer than approximately 1 mm). This was test by making single layer test wafers in pairs which had modified detector patterns as shown in Figure 3.8. These wafers were processed in paralell under identical conditions except for the detector etch. In one wafer, the detector was etched using the SF_6 process while the other was ion milled using the Kauffman source.

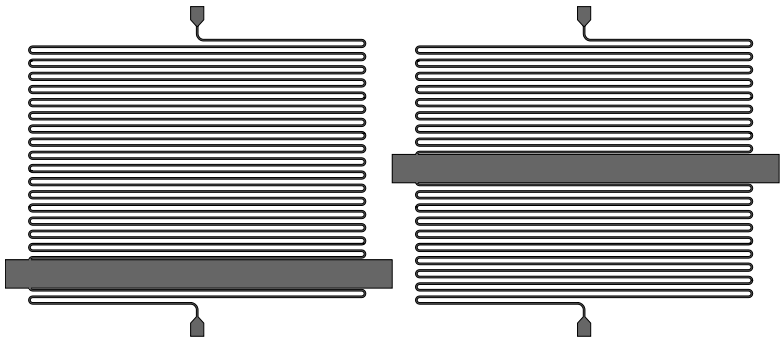

Figure 3.8: Critical Current Density Test: The wide bar in the middle is generated by modifying the stepper lithography program to purposefully not expose a band across each die. By moving this band around, we can split each detector into two shorter wires which are both shorted to ground on one side. This allows us to make measurements of the critical current density of many different wire lengths without having to purchase a special made mask with this type of test structure.

The results, summarized in Figures 3.9 and 3.10, clearly indicate that the SF_6 recipe is degrading the quality of our films. This etch recipe also has the unfortunate quality that it etches the silicon nitride under the detector at an extremely high rate (nearly twice as fast as the niobium itself). In response to these factors, we developed a chlorine based etch recipe, summarized in Table 5. The tool this etch is performed in is also

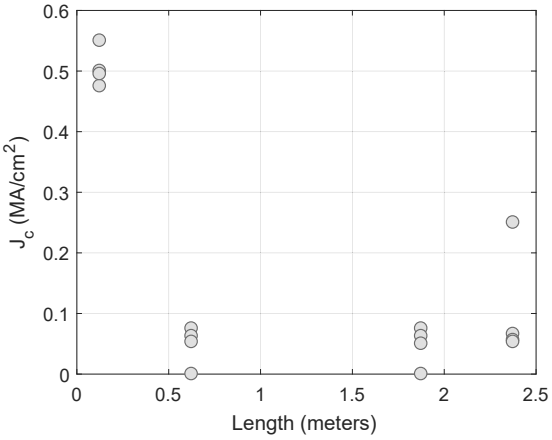

Figure 3.9: Critical Current Density vs. Length Ion Mill

loadlocked, which should cut down on exposure to ionized oxygen during the etch process. The procedure for performing this etch is:

1. Preclean etch chamber and carrier wafer with 10 minute oxygen plasma
2. Preseed etch chamber and carrier wafer with 10 minute run of etch recipe with no sample
3. Mount sample wafer onto 6 inch carrier wafer using a tiny amount of santovac between them
4. Run etch recipe shown in Table 5
5. Remove carrier wafer, warm to 50 °C and then sample wafer from carrier wafer. Rinse with water in case any volatile chlorine byproducts remain on the wafer

This etch recipe worked well sometimes, but occasionally the etch chamber would become contaminated by another user's process, and our measured critical current densities would be suppressed across every wafer we ran through the tool. We attempted to

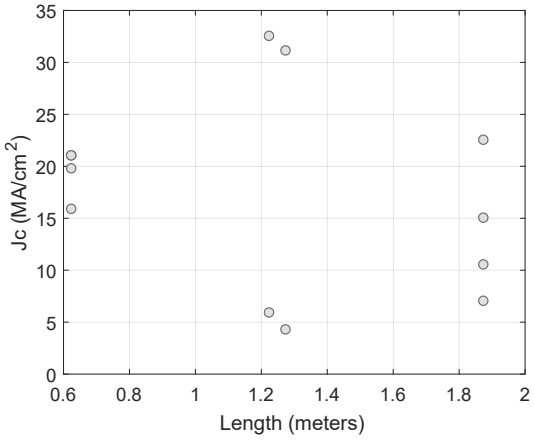

Figure 3.10: Critical Current Density vs. Length Ion Mill

PT-770		
Chamber Pressure		40 mTorr
Process Gas Flow	BCl_3	15 sccm
	Cl_2	20 sccm
	Ar	20 sccm
RIE Power		50W
ICP Power		300W
Etch Rate		17 nm/min

Table 5: Niobium Detector Chlorine Etch Parameters

use plasma spectroscopy to detect what contamination was present that was degrading our films, but when we compared the spectra of etches which yielded good devices versus bad we weren't able to see any significant differences.

Due to the unreliability of this etch, we focused on just using the Kauffman Ion

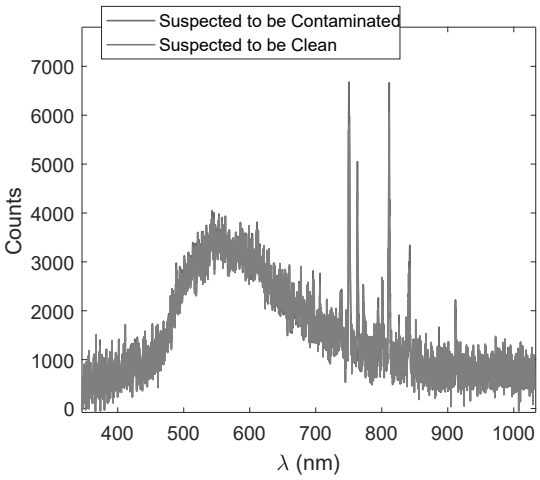

Figure 3.11: Niobium Etch BCl3+Cl2+Ar Plasma Spectroscopy

Source to etch our patterns. The recipe for this etch is shown in Table 6.

Kauffman Ion Source	
Argon Pressure	.2 mTorr
Beam Voltage	600 V
Accelerator Voltage	120 V
Beam Current	46 mA
Neutralizer Current	51 mA
Etch Rate	24–30 nm/min

Table 6: Niobium Ion Mill Etch Parameters

After etching the detector layer, it is critical that as much of the remaining photoresist residue as possible is stripped. This can be espeically difficult to do after ion milling. The procedure to do this is:

1. Preheat beaker full of Microposit Remover 1165[6] to 80 °C
2. Perform coarse strip of PR residue with flowing acetone (this helps conserve 1165 by getting the bulk of the easily removable PR off ahead of time)
3. Without allowing the wafer to dry at all, submerge it in the beaker of hot 1165. Soak in hot 1165 for 3 hours. Meanwhile, preheat shared 1165 tank (which is built into a chemical bench) to maximum temperature of 75 °C.
4. Transfer wafer into shared 1165. Leave it there overnight.
5. The next day, preheat a beaker of acetone to 75 °C
6. Quickly transfer wafer into the beaker of acetone without allowing it to dry at all in the process.
7. Soak in the hot acetone for at least 30 minutes.
8. Sonicate wafer in beaker full of acetone for 30 minutes
9. Quickly transfer wafer into beaker full of de-ionized (DI) water, sonicate for 10 more minutes
10. Remove wafer, quickly place under ample supply of flowing DI water. Rinse in this way for 2 minutes
11. Quickly load wafer into SRD[7]

[6]Specialized resist stripper which mostly consists of n-methyl-pyrrolidinone (NMP)

[7]Spin rinse dryer

12. Unload, examine under microscope to check for any particulate contamination of solvent residue (which appears as discolored streaks on the wafer in an optical microscope).

After this heavily crosslinked resist is stripped, it is time to deposit the encapsulation layer. The procedure is similar to the procedure for depositing the wiring dielectric as described above, but slightly modified:

1. Heat the sample platen in the deposition chamber to the deposition temperature (250 °C)
2. Run plasma preclean process (O_2 and CF_4) for 10 minutes
3. Preseed by running deposition recipe (shown in Table 4) on empty chamber for 10 minutes
4. Vent deposition chamber. Turn off the sample platen heater. Allow the sample platen temperature to fall below at least 130 °C. When it is close to this temperature, run the wafer through the SRD one final time.
5. Load sample onto sample platen in deposition chamber, pump down chamber to base pressure
6. Run deposition recipe in Table 4 for 20 minutes
7. Vent, remove sample– set sample on a metal surface to cool before putting it back in plastic carrier
8. Run post clean plasma (same as preclean)

9. Meanwhile, examine wafer under microscope. In particular, make sure there are no obvious pinholes in the film. Solvent/PR residue isn't great at this point, but it shouldn't significantly affect the yield. It may, however, cause devices to have non-uniform propagation velocities.

In order to access the detector and ground plane, it is necessary to etch vias (holes) through both the encapsulation layer that was just deposited as well as the wiring dielectric layer which was deposited several steps ago. First, perform lithography as follows:

1. Spin coat the wafer with SPR-955 CM.7, spinning at 3000 rpm
2. Prebake the wafer at 100 °C for 90 seconds
3. Expose via etch pattern in Body 8 stepper.
4. Post bake at 110 °C for 90 seconds
5. Develop in MF-CD26 developer for 60 seconds
6. Rinse developer off in DI water beaker for 60 seconds
7. Rinse wafer under flowing DI water for 30 seconds
8. Examine wafer under microscope to verify pattern transferred correctly

This will generate a pattern with holes connecting to both the detector layer and the ground layer. Normally this would be unadvisable because there are different thicknesses of silicon nitride between the top surface and those two layers. However, our recipe for etching silicon nitride (shown in Table 7) has extremely good selectivity between silicon

nitride and niobium; that is, it etches silicon nitride at approximately 125 nm/min and niobium at only 5 nm/min. This means we can tolerate etching the detector part of the niobium slightly longer than necessary in order to etch the ground bond pads through the extra 100 nm of nitride. The procedure for this etch is given below:

1. Preclean chamber with 10 minute, 500W O_2 Plasma
2. Preseed chamber with 5 minute run of plasma recipe shown in Table 7
3. Vent chamber, load sample, pump down
4. Run plasma recipe in Table 7 for 120 seconds
5. Vent chamber, retrieve sample, and check under microscope. Etched regions should appear silver.
6. Pump down sample chamber
7. Post clean chamber with 5 minute, 500W O_2 Plasma
8. Probe bond pads which should be electrically connected. Verify that the measured resistance is correct (roughly $20\,\Omega$ for ground plane pads within the same die, roughly $10\,\mathrm{M}\Omega$ for pads on either side of a detector)

At this point, we've completed all of the processing steps for the wafer. It is useful to probe all of the detectors on the wafer as described in Section 4.2. After this, the wafer can be diced into separate dies and fully characterized as described in Chapter 4.

PT-790		
Chamber Pressure		100 mTorr
Process Gas Flow	CHF_3	50 sccm
	O_2	20 sccm
RF Power		150W
Etch Rate		125 nm/min

Table 7: Silicon Nitride Etch Parameters