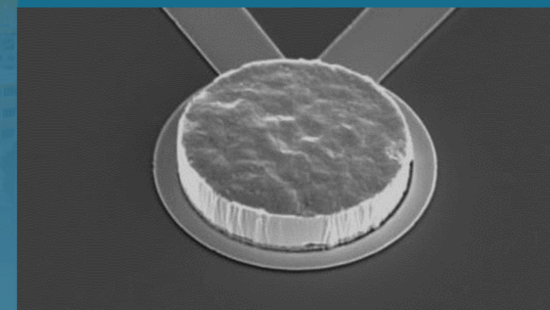
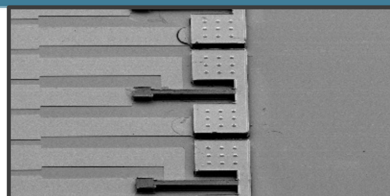
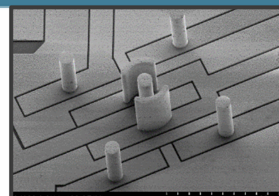
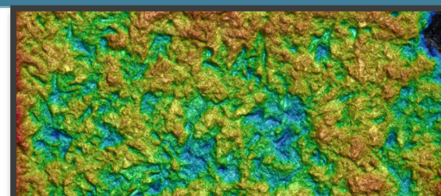
This paper describes objective technical results and analysis. Any subjective views or opinions that might be expressed in the paper do not necessarily represent the views of the U.S. Department of Energy or the United States Government.

SAND2023-04246C

In-Situ Analysis of Chronopotentiometry Data to Predict Electrodeposition Rates



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[1] Sandia National Laboratories

243rd ECS Meeting – Boston, MA

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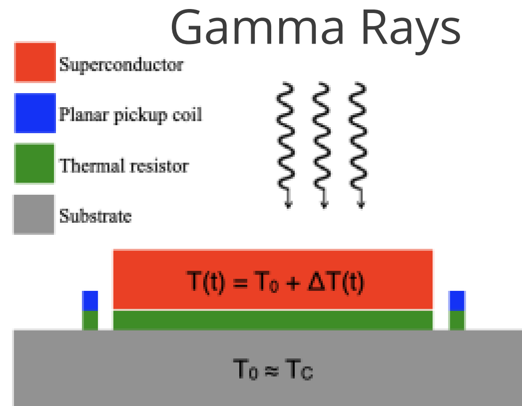
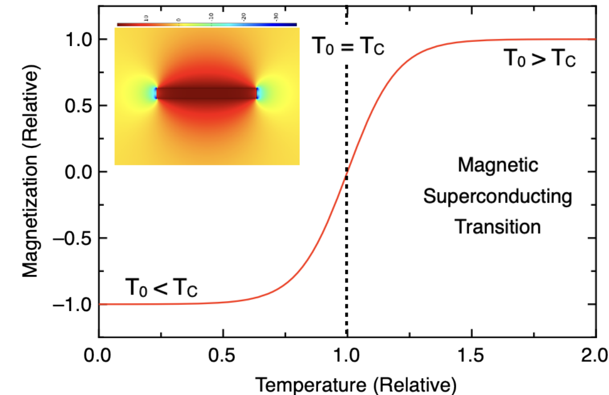
Meissner-Effect Transition-Edge-Sensor (ME-TES)

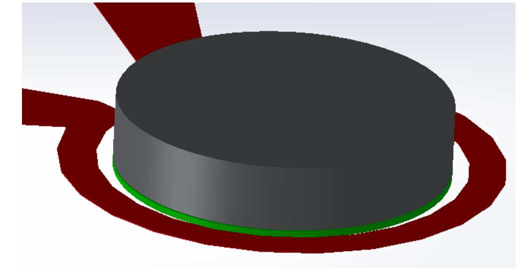
- Operating temp below superconducting transition temp, expulsion of magnetic flux from tin
- Operating temp above superconducting transition temp, tin acts normally
- In between, change in temp corresponds to change in flux

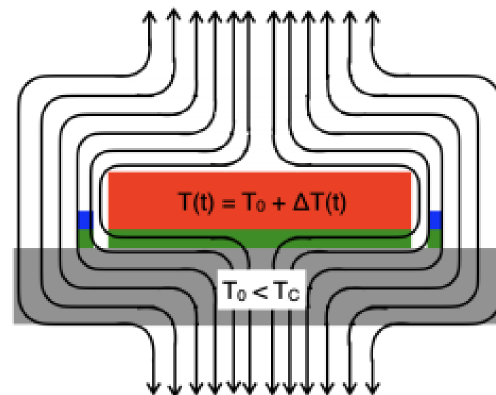
Tin
Niobium
Silicon Nitride
Silicon

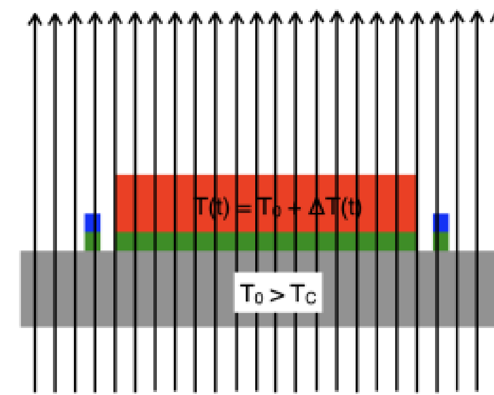
Gamma Rays









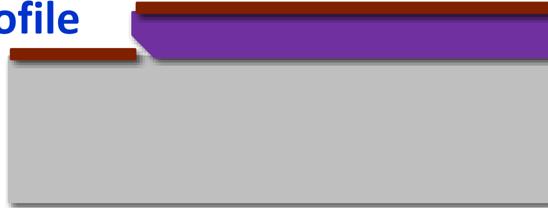


Lift-off, etch, and test wafer microfabrication

Niobium metal deposition to pattern coils

Photoresist with re-entrant profile

silicon substrate 6”

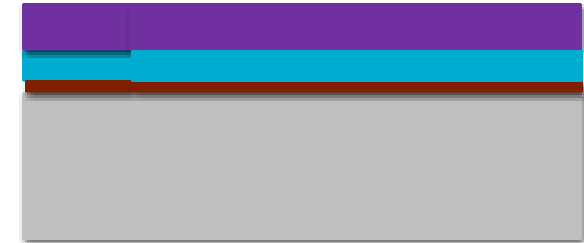


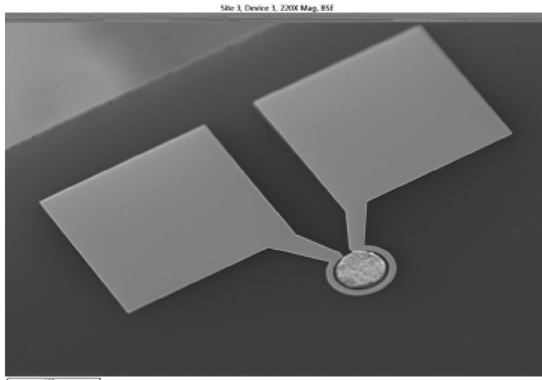
Photoresist

Silicon oxide

Niobium metal deposition

silicon substrate 6”



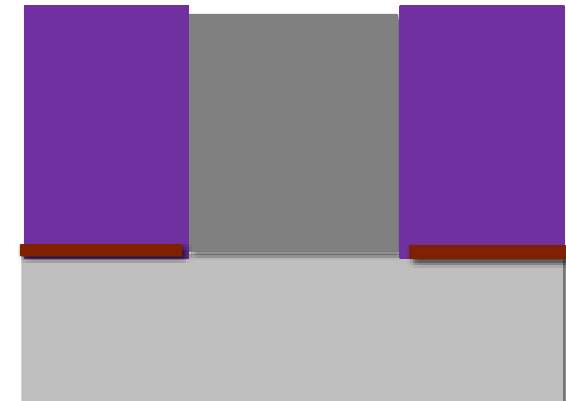


Tin metal deposition

Photoresist with straight sidewall

Niobium metal

Silicon substrate 6”



Objective

- Possible variables
  - Device diameter – 5-1000 microns
  - Mask layout
  - Wafer processing steps
  - Resistance of underlayers
  - Plating method
    - Chronopotentiometry or chronoamperometry
    - Pulse or DC
  - Current density, real or estimated
  - Depletion of chemistry
  - Total area

- Objective: consistent plating
  - Plating tin is not hard, but we have a fast rate with a small area where it is easy to over or undershoot

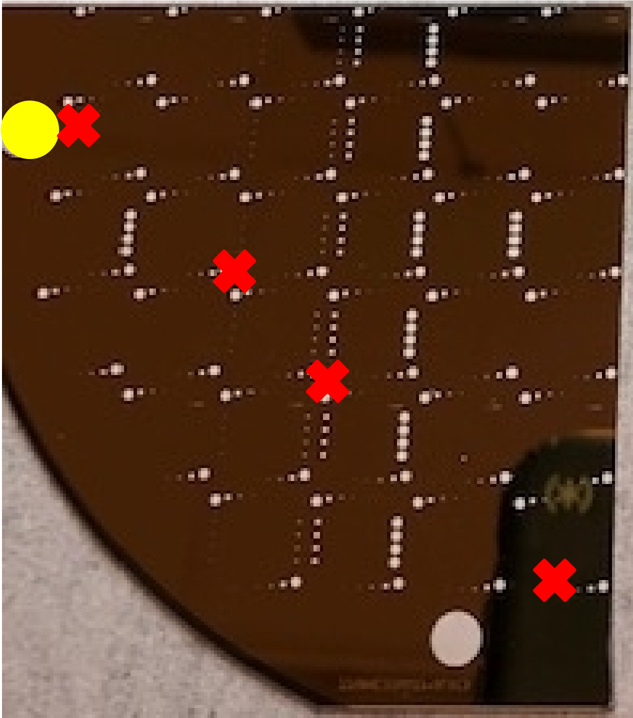
Optimized Electroplating Parameters[1]

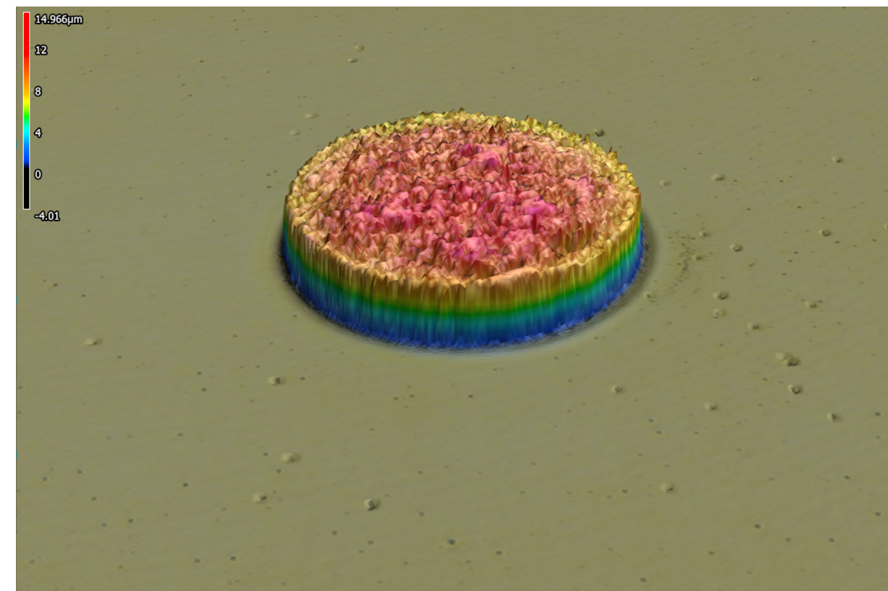
- Plate 8-12 microns
- Biologic SP-300 potentiostat
- Commercial Solderon Tin/Silver Plating Bath with no silver ions
- Limit device size to only 50 micron diameter
- 80 mA/cm^2 current density DC
- Constant current

(1) Arrington, C.L. **Optimizing Tin Electroplating for MEMS Meissner-Effect Transition-Edge Sensors**. In *243rd ECS Meeting with SOFC-XVII, Boston, MA, 2023*.

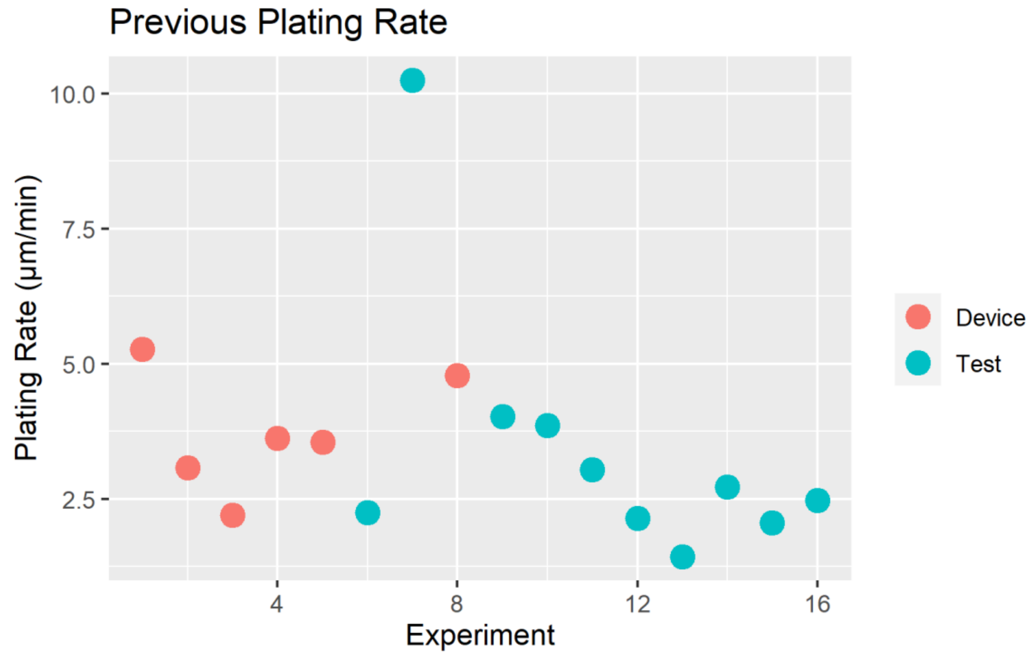
Experimental

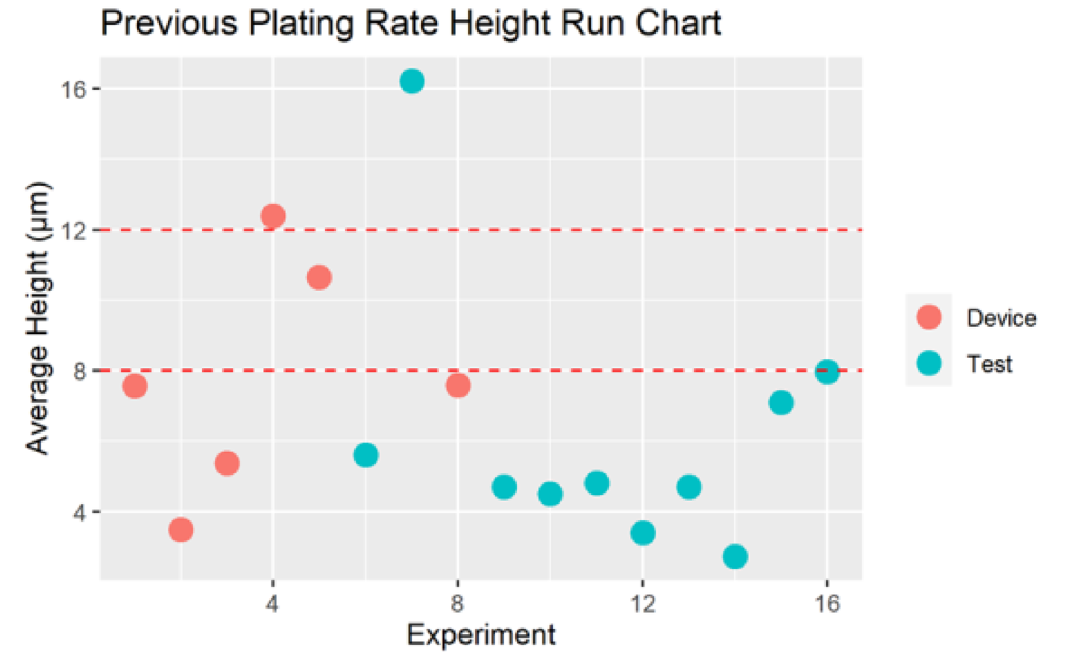
- Plate one quarter with ~750 devices
- Confocal microscopy to measure height and surface roughness
- Average of 4 locations at different distances from contact
- Right circular cylinder geometry desired
- Estimate rate on test wafer and commit device wafer





Early Frustration



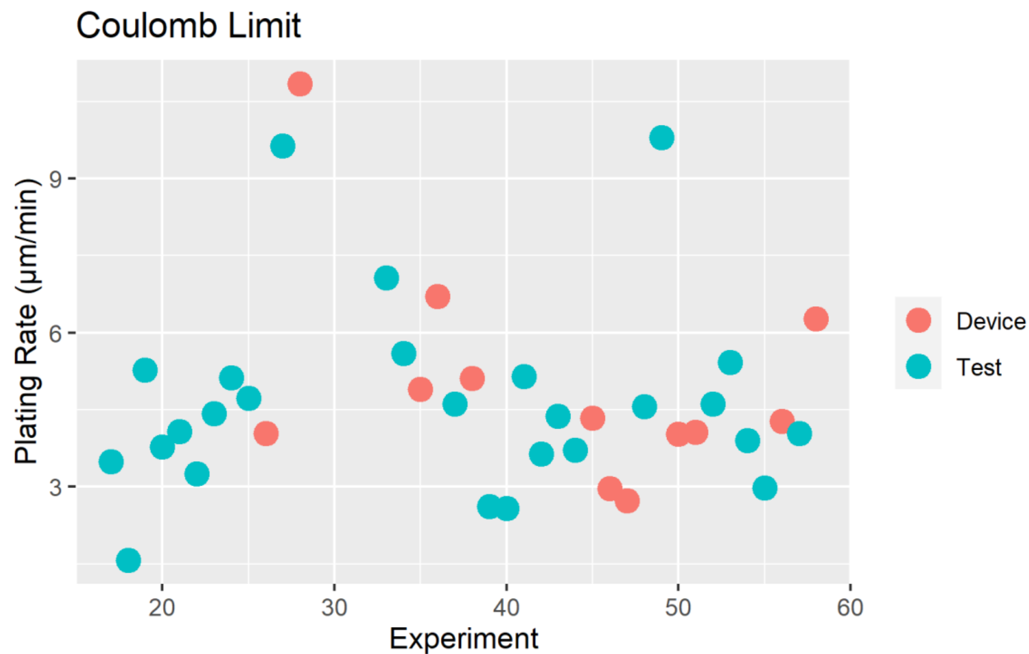


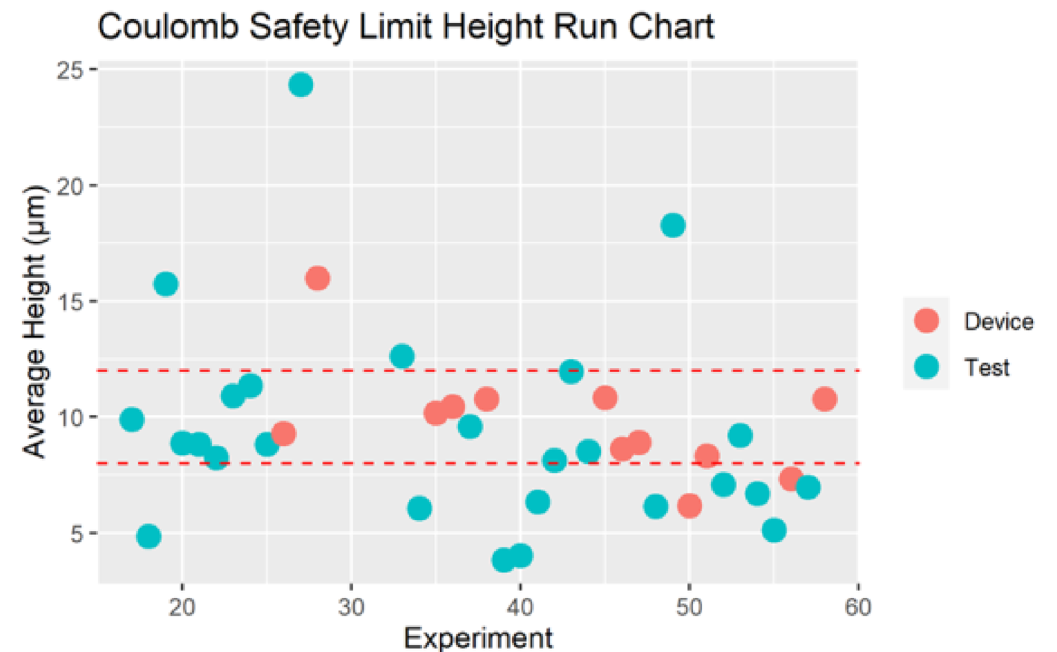
- Trouble dialing in a plating rate
- Plating rates from 1-10 µm/min. Should the deposition be 1 minute or 4? Will we end up with 3 microns or 16?
- 2-step plating process not ideal as it brings crystal structure into question

Coulomb Safety Limit

$$\frac{(Area * Plating\ Height * Density)}{Atomic\ Weight} * 2\,e^{-} * 96485\ C\ mol^{-1}$$

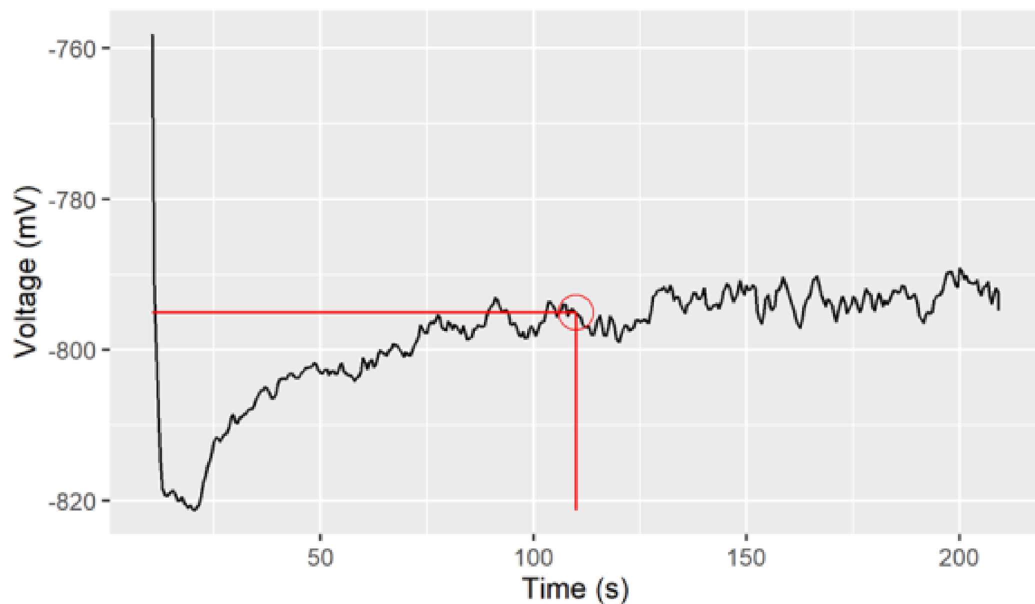
- EC-Lab software includes a charge transfer safety limit
- Added in a Ag/AgCl/KCl (sat'd) reference electrode
- This was just as erratic

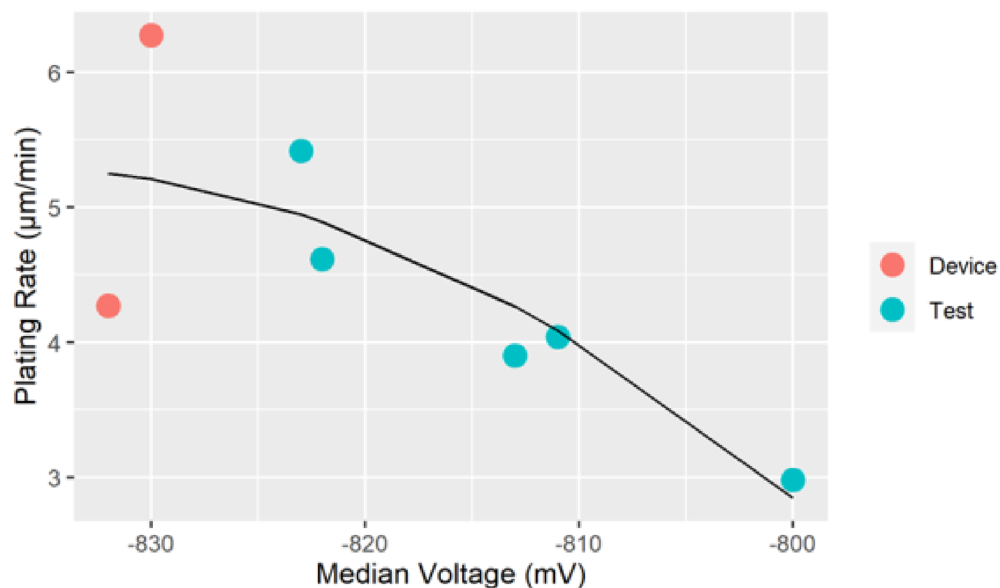




Finding something useful – Median Voltage

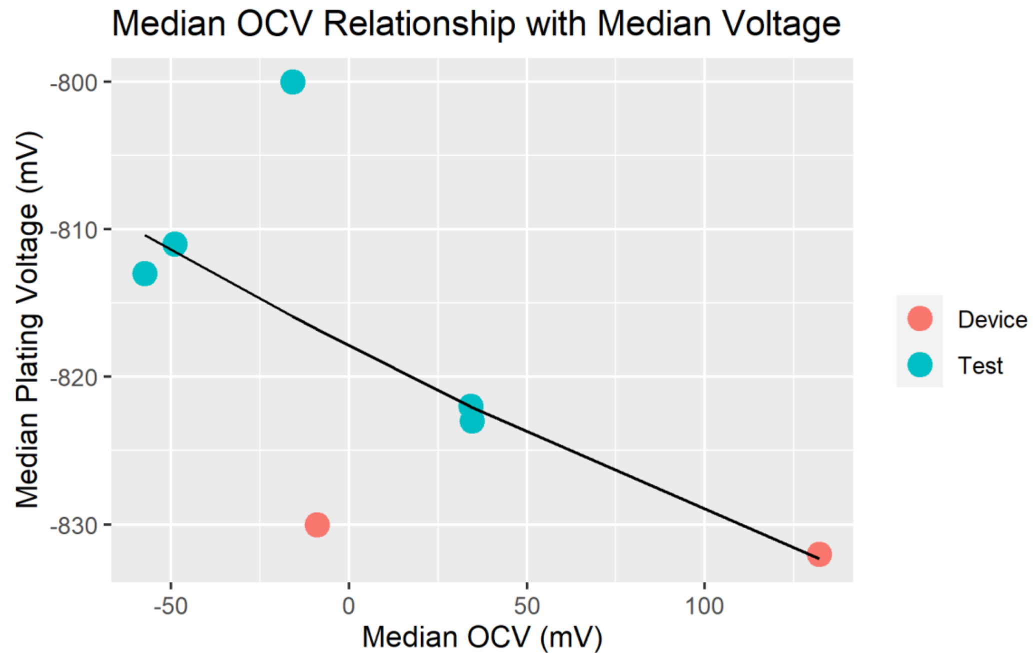
- Spend time with the data
- Noticed the relationship between median voltage and plating rate at constant current
- But what is the middle?
- Need leading, not lagging indicator

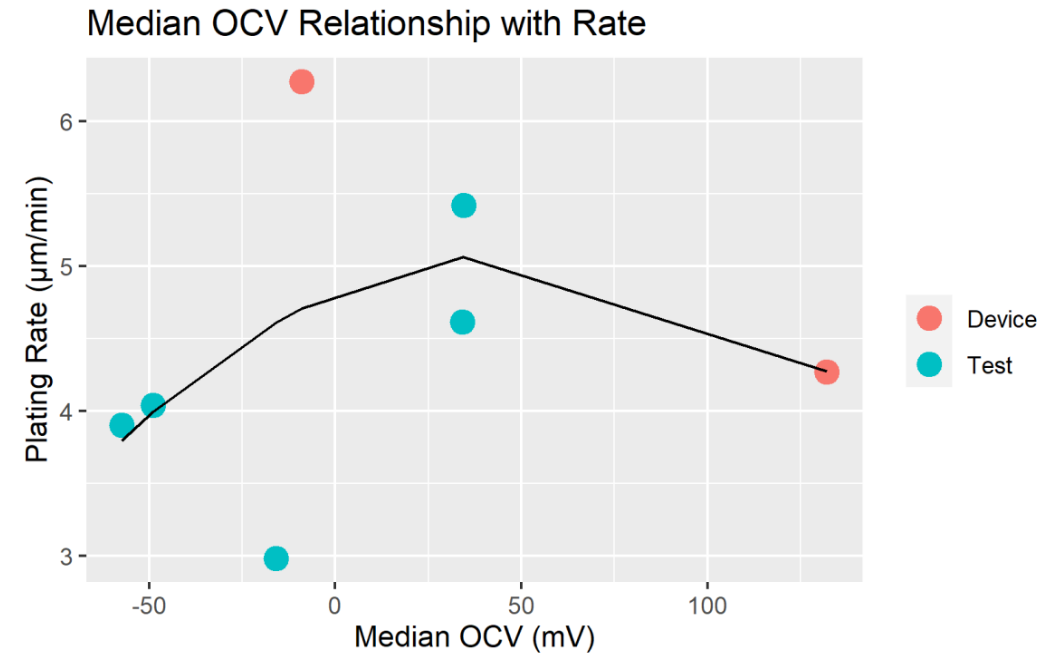




OCV to Median Voltage

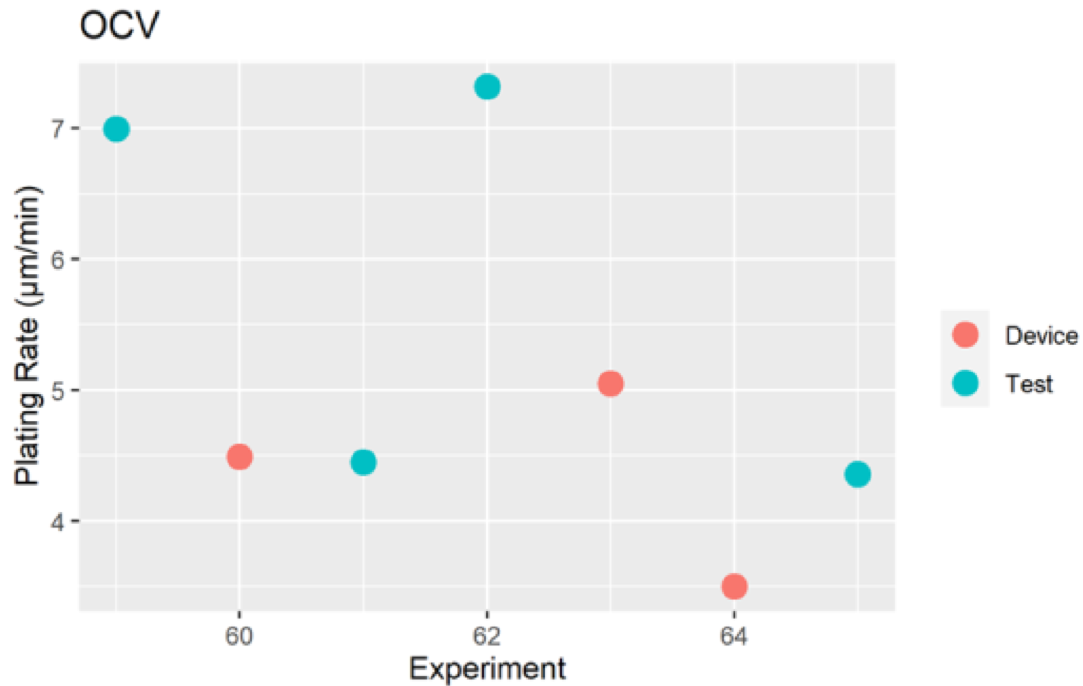
- Some relationship with OCV, median plating voltage, and plating rate

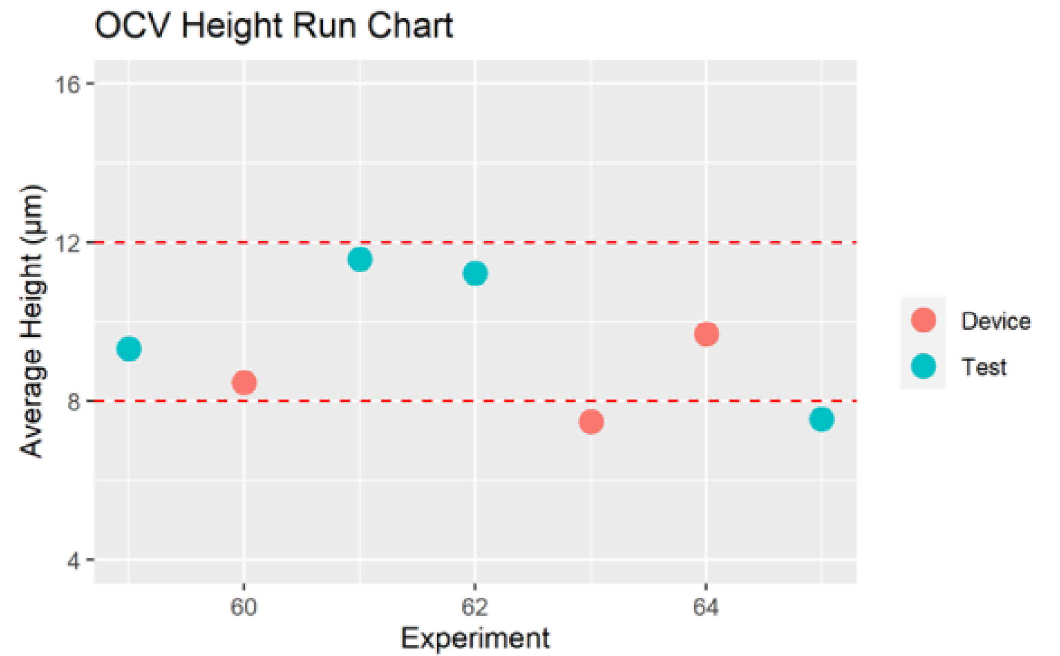




OCV

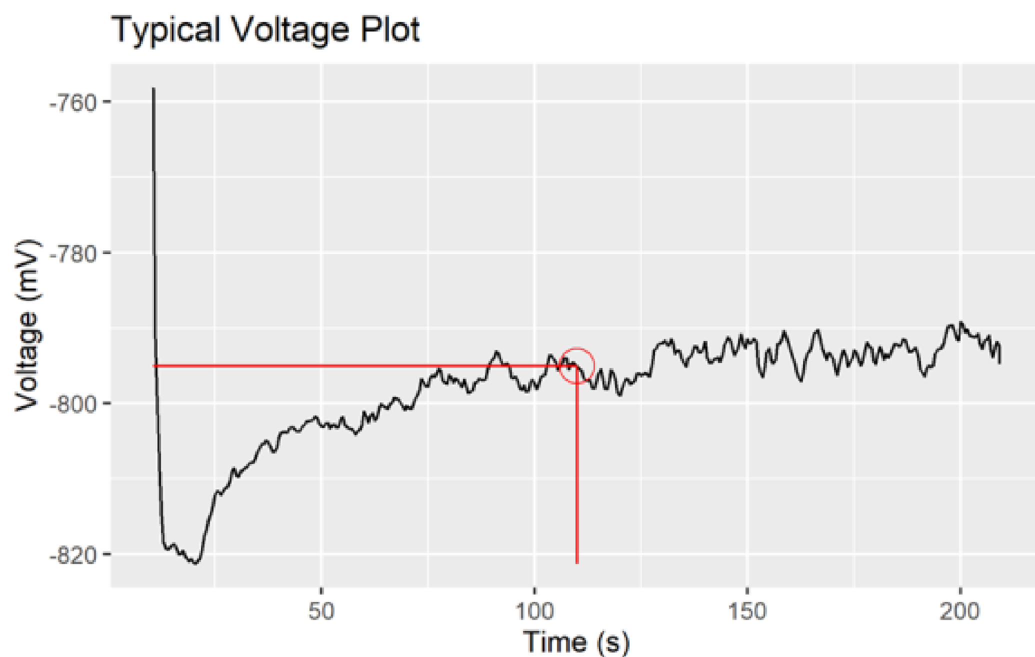
- Even with rates ranging from 3.5 to 7 microns per minute, we still achieved average heights between 7.5 and 11.5 microns
- Improved results, but these relationships were not very strong





Change Experiment Length During Processing

- Software allows changing ending time on the fly
- How much data is enough?



Devices
SP-300 - virtual
Experiment
Safety/Adv. Settings
Cell Characteristics
External Devices
Parameters Settings
1 - OCV
2 - CP

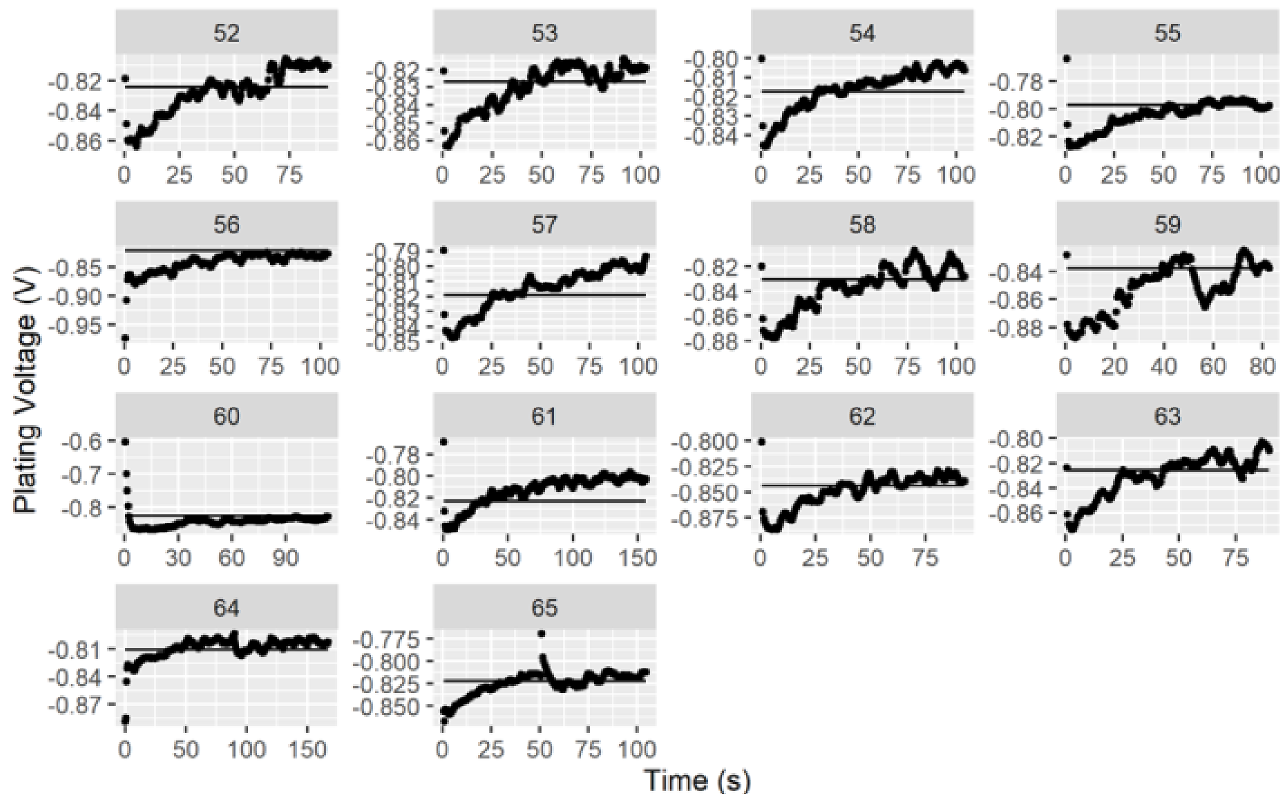
Turn to OCV between techniques
0

Apply I_s = -17.600 mA vs. <None>
for t_s = 0 h 3 mn 47.000 0 s
Limits E_{we} < E_M = pass V
|ΔQ| > ΔQ_M = 1.110 mA.h
Record <Ewe>
every dE_s = 0.0 mV
or dt_s = 0.500 0 s average on 2500 points
E Range = -1 V; 1 V
Resolution = 50 µV
I Range = 100 mA
Bandwidth = 8

Go back to sequence N_s' = 0 (9999 ends technique)
for n_c = 0 time(s) (0 for next sequence)

How much data is enough?

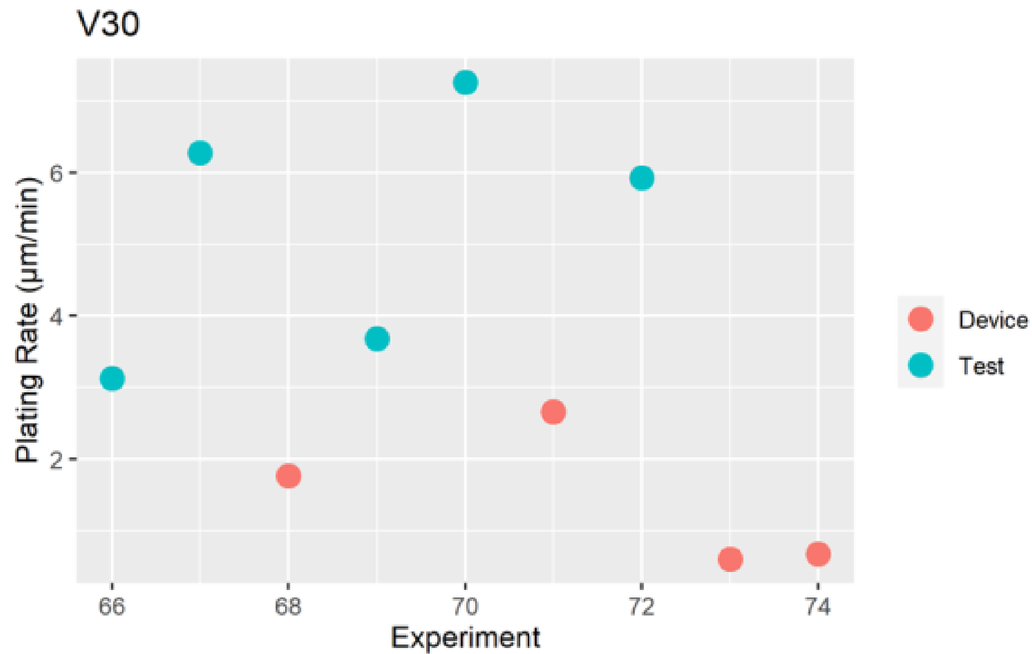
- Work backwards from previous experiments to determine what voltage would have given the correct results
- Shortest experiments were 60 seconds
- Average time for correct voltage at 37 seconds, mode around 30 seconds

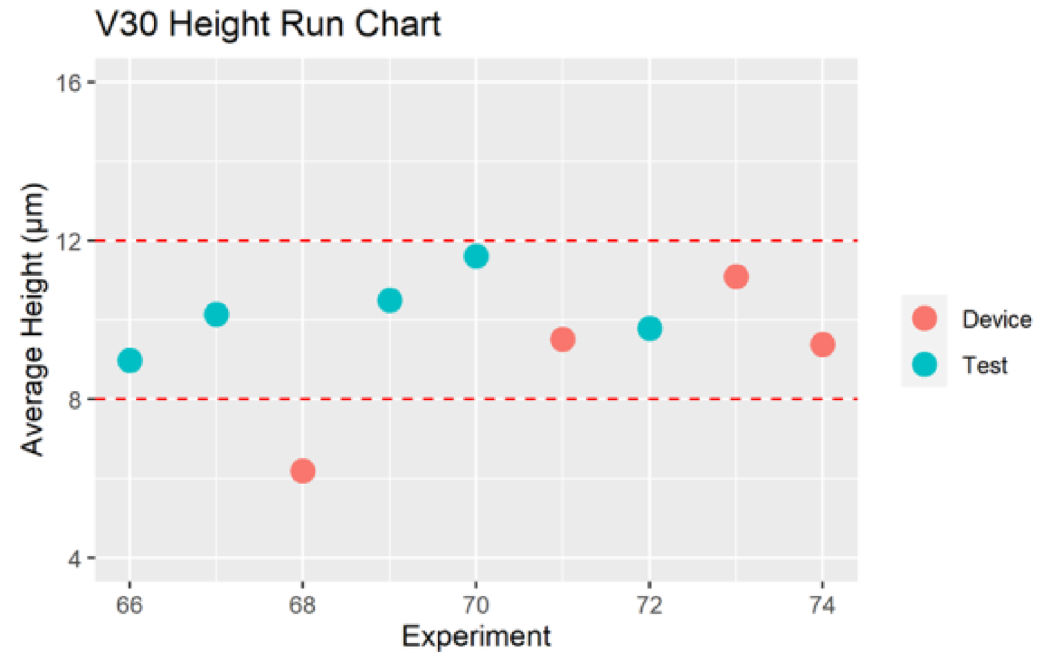


Experiment	Voltage Estimate Time (s)
52	30
53	34
54	22
55	39
56	62.5
57	24
58	31.5
59	38.5
60	95
61	23.5
62	32
63	24
64	34.5
65	28.5
Avg	**37.1**

Voltage @ 30 seconds (V30)

- Change the experiment time while running
- Only one outlier – trust the process
  - 3.5 minute plate instead of 5
- Despite large changes in plating rate, final height still within range

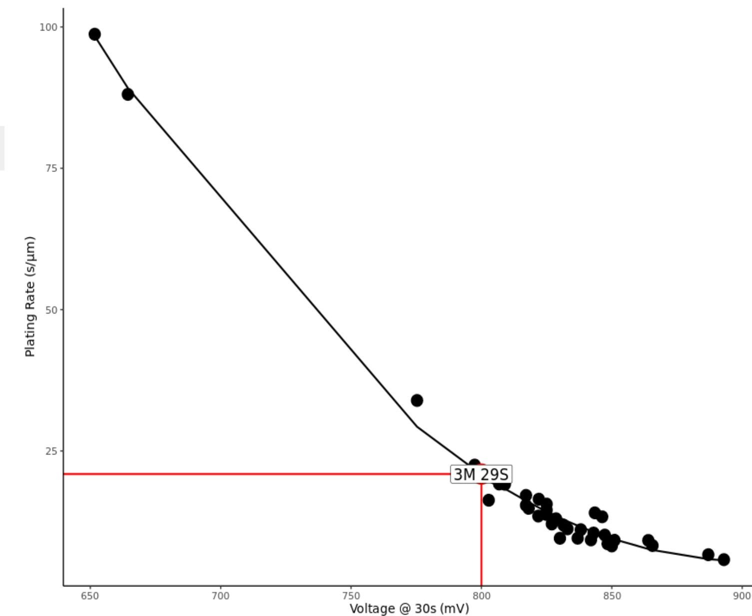




Graphical User Interface (GUI)

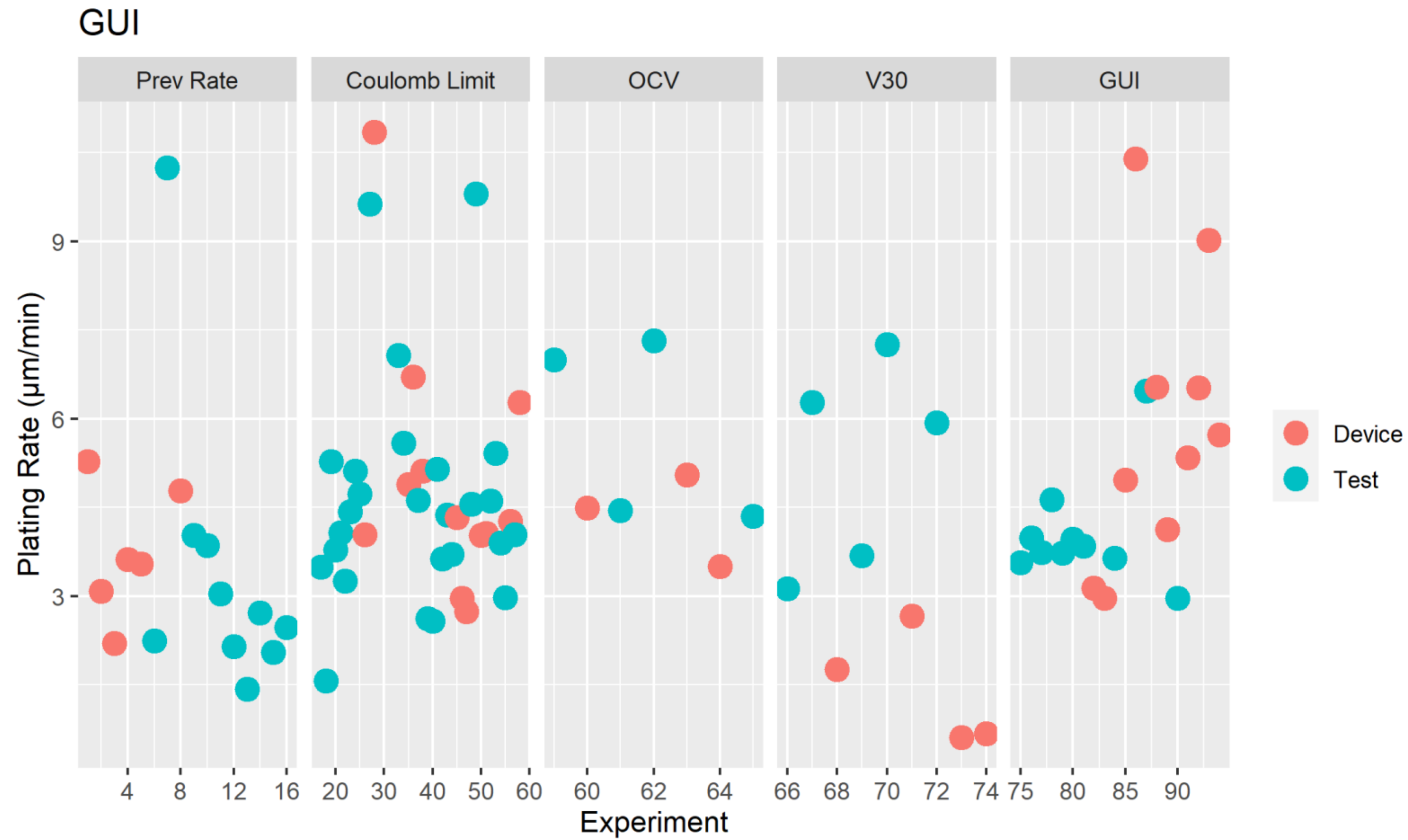
- User interface to improve usability and update with new data
- Internal Posit Connect server to host R Shiny application
- Still want some control while continuing research

Plating Voltage	875	
Rate	11.73592	
Thickness	10	
Time	117.3592	
	1	minutes
	57	seconds
End Time	127	
	2	minutes
	7	seconds



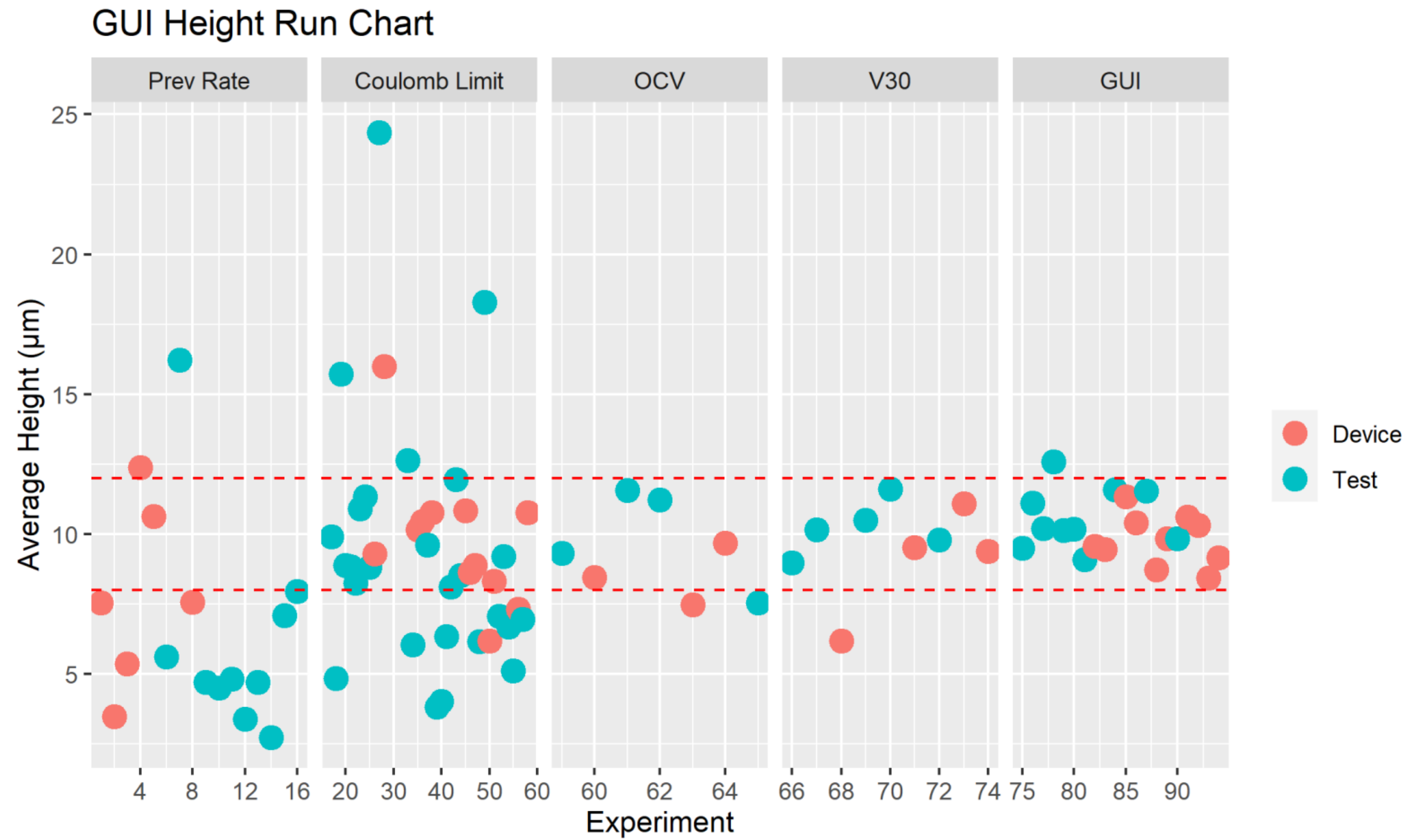
GUI

- Plating rate still variable between 3 and 10 microns/minute



GUI

- Final height range between 8.4 and 12.6 over 20 experiments



Future Work and Conclusions

- Reduced standard deviation of plating height from ? To ?

- Biologic Developer’s Software
- Dull the blade of the knife – cool chemistry

Questions?

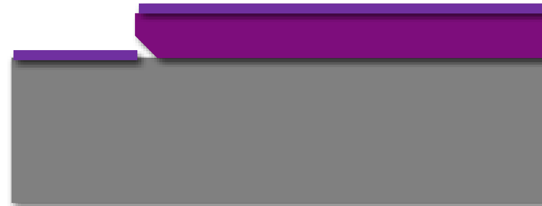
Backup slides

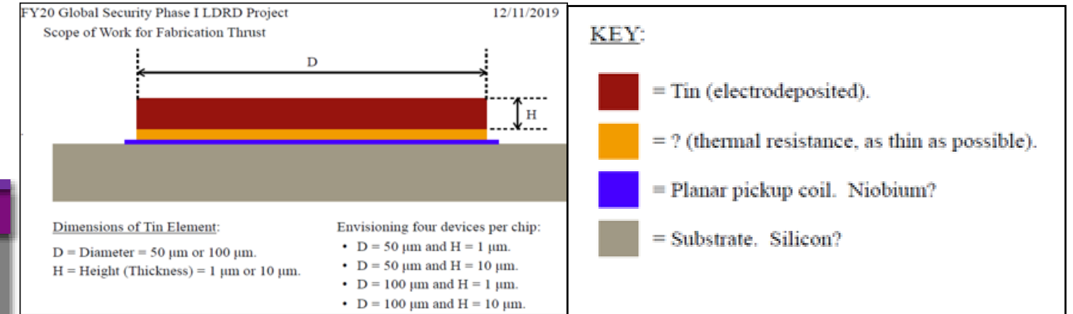
“Lift off” and electroplating Calorimeter process

Niobium metal deposition to pattern coils

Photoresist with re-entrant profile

silicon substrate 4”





Tin metal deposition

Photoresist with straight sidewall

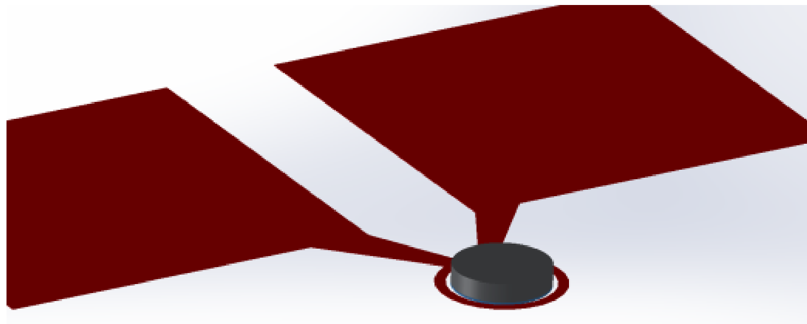
Niobium metal

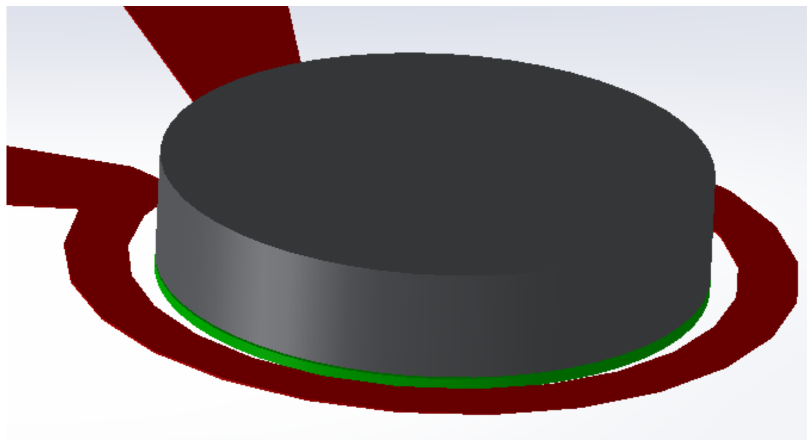
Silicon substrate 4”

CAD layout and fabrication

- 2 chrome photomasks ordered and delivered to SNL
- Layer 0 lithography complete to verify lift off process Ti/Au
- Microscopy pics below
- SEM pics coming Friday afternoon

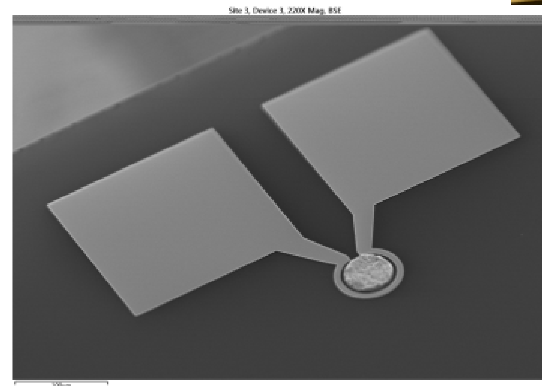
3D CAD view showing superconducting ring and bonds pads (red), Silicon nitride (green), and Sn plating (grey)





Layer 0 lithography images Ti/Au lift off

Lithography



Wafer/die after Ti/Au deposition