HiPIMS Nb based thin films deposited on copper

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Why HiPIMS?

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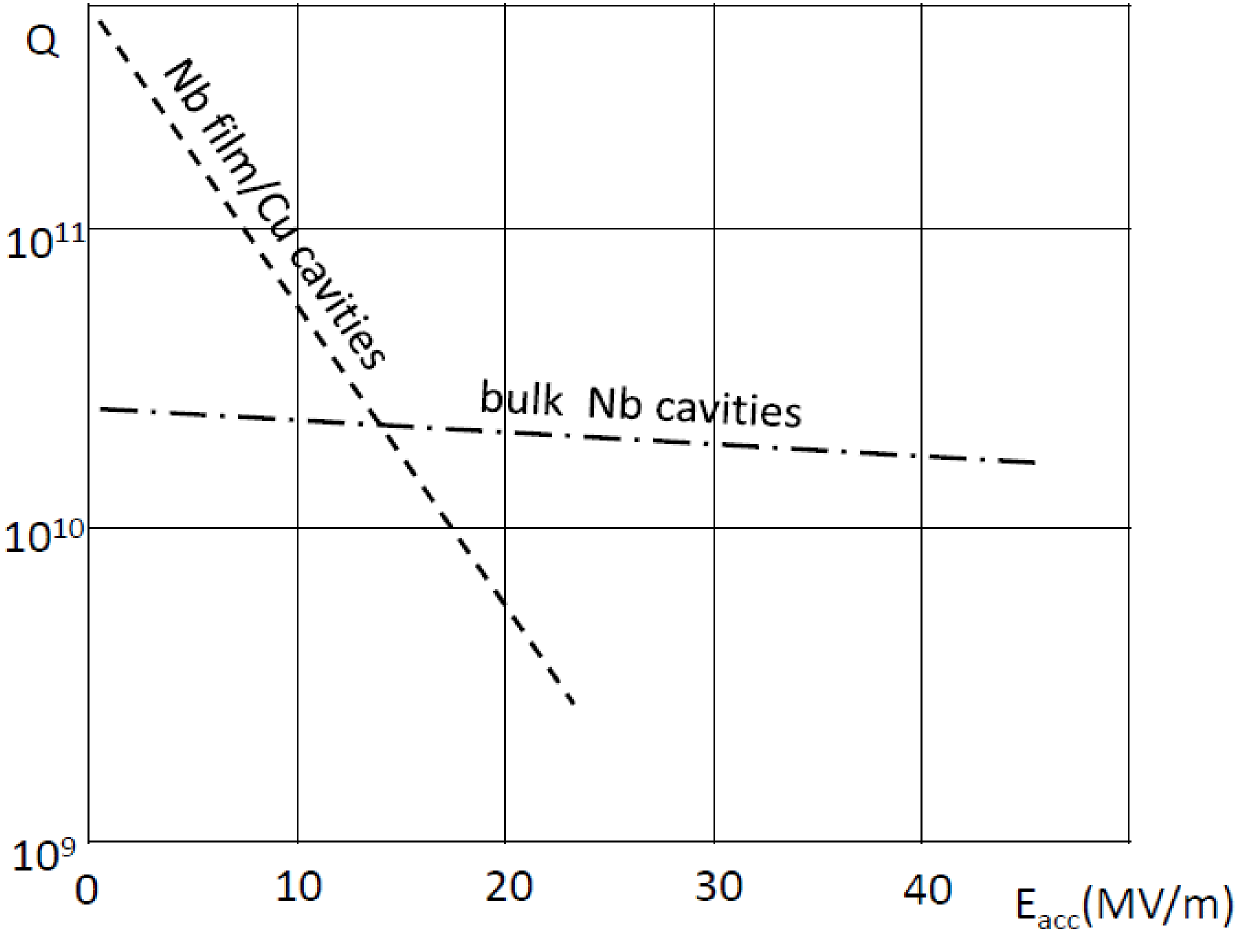
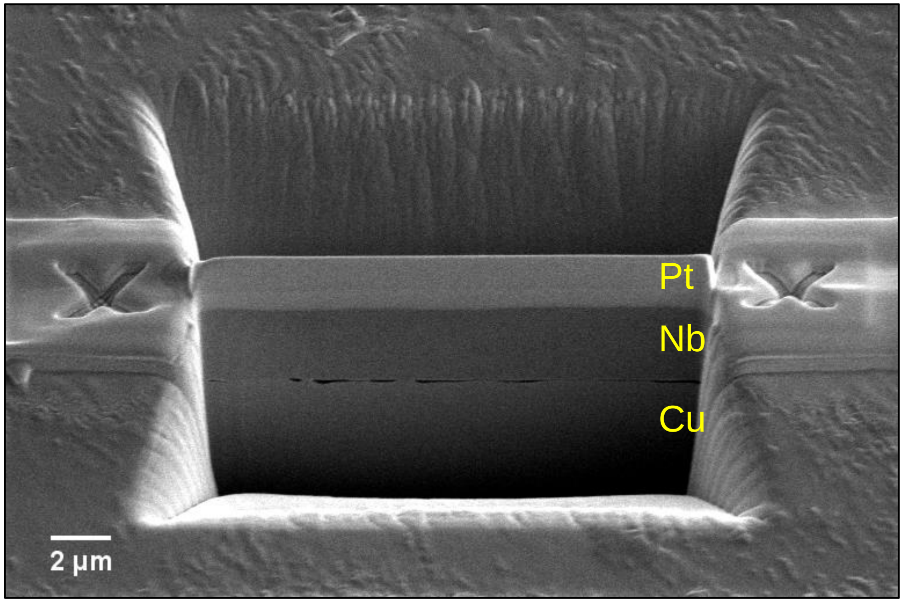

Q
10^{11}
10^{10}
10^{9}
0
10
20
30
40
E_{acc}(MV/m)
Nb film/Cu cavities
bulk Nb cavities

Why HiPIMS?

DC MS Nb



☒ Voided Interface

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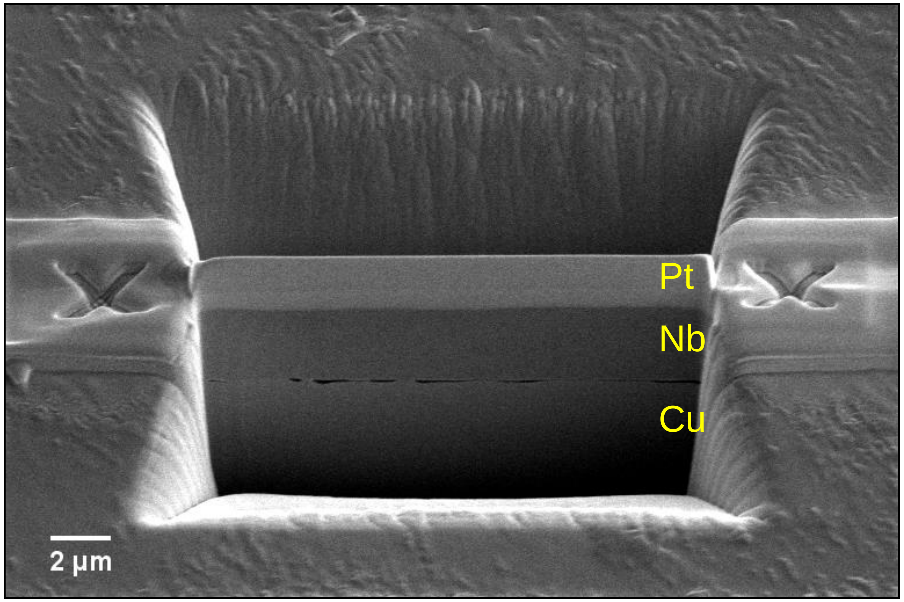

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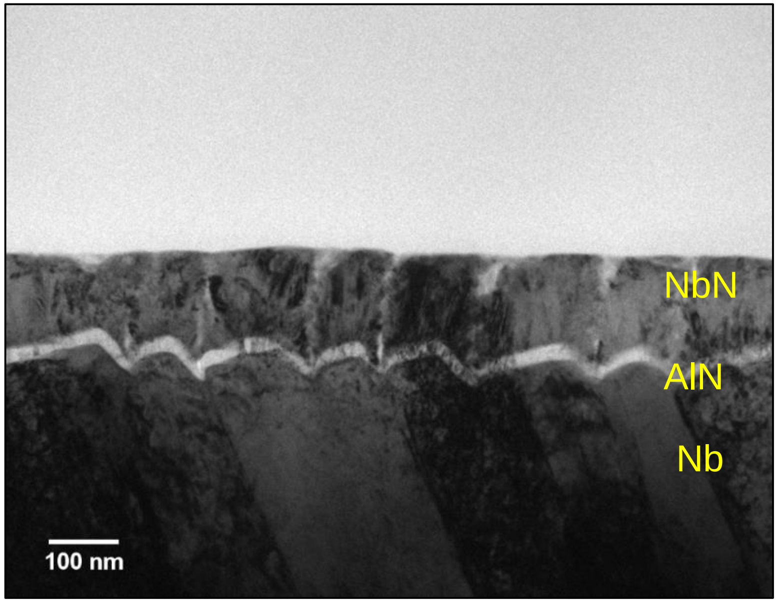
DC MS Multilayer

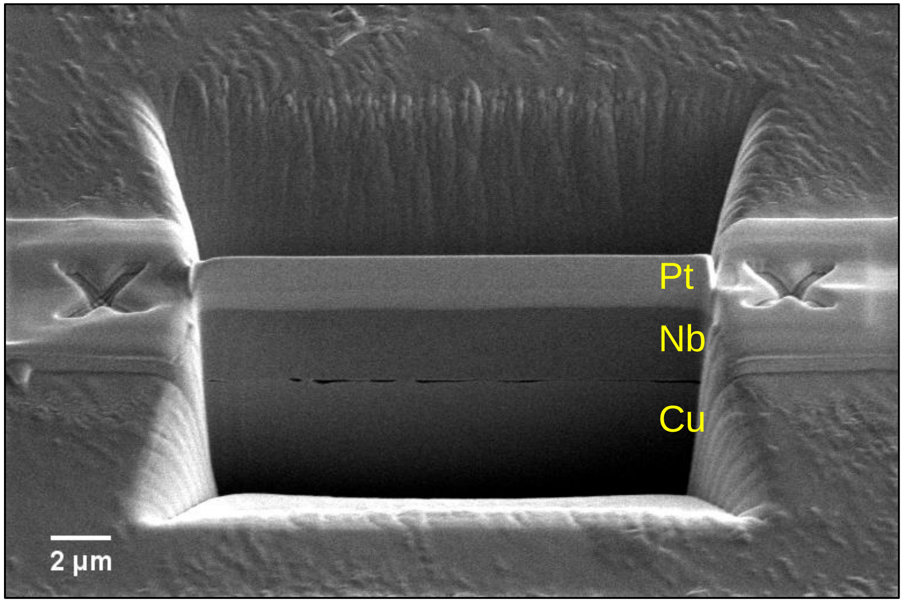


☒ Large Surface Roughness

Courtesy: Ying Li

Why HiPIMS?

DC MS Nb



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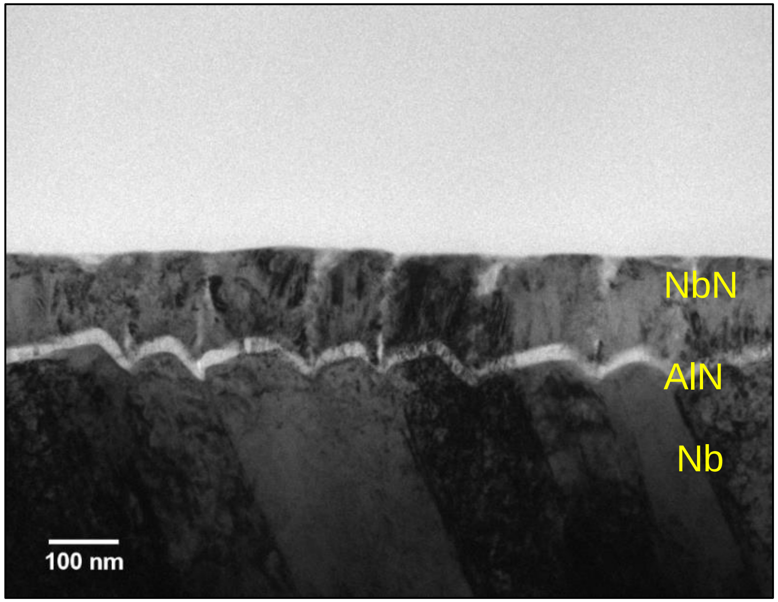


✗ Large Surface Roughness

✓

HiPIMS Promises:

- Excellent adhesion
- Superior density
- Decreased roughness
- And more

Courtesy: Ying Li

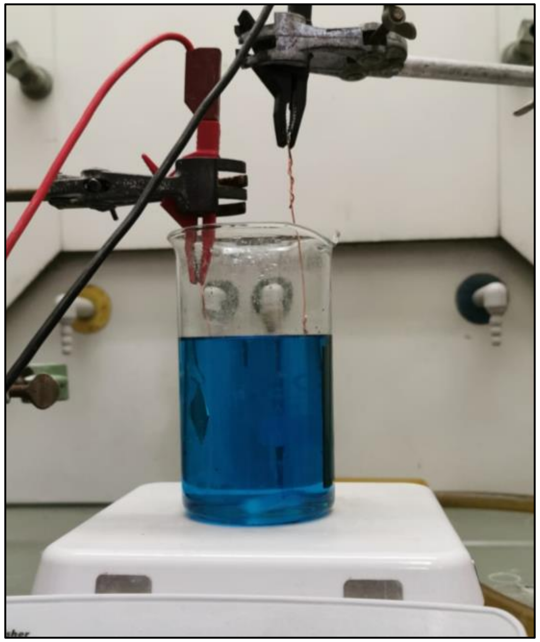
Substrate Preparation

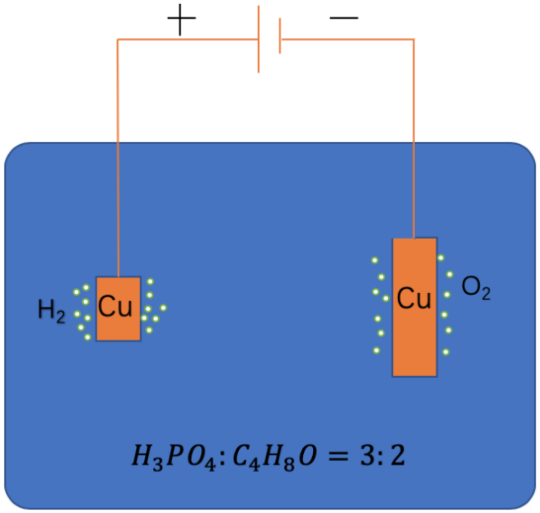
“Substrate is Key”

Substrate Preparation

Cu substrate preparation recipe:

- Mechanical polishing
- Electrolyte polishing
  - Including activation and passivation
  - $S_q = 2.58 \pm 0.26\text{nm}$
- Degreasing – Lupo 44
- MF Plasma etching





$H_3PO_4 : C_4H_8O = 3 : 2$

Substrate Preparation

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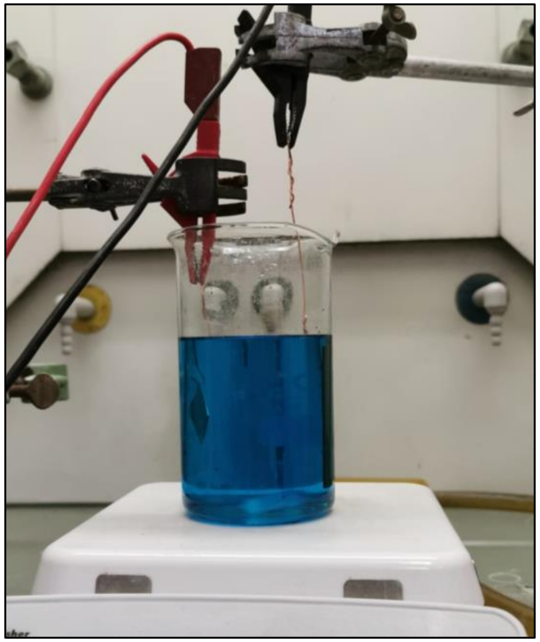
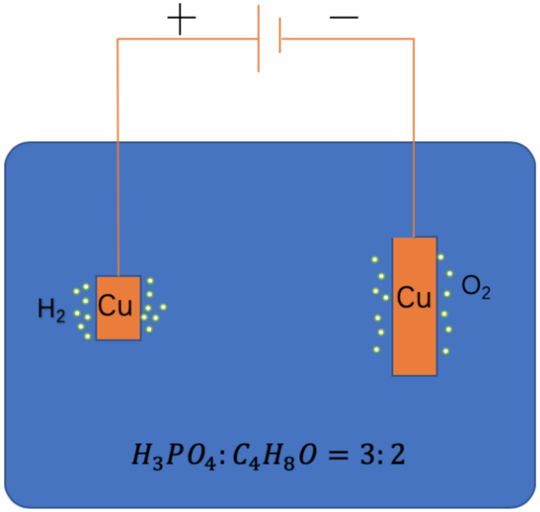
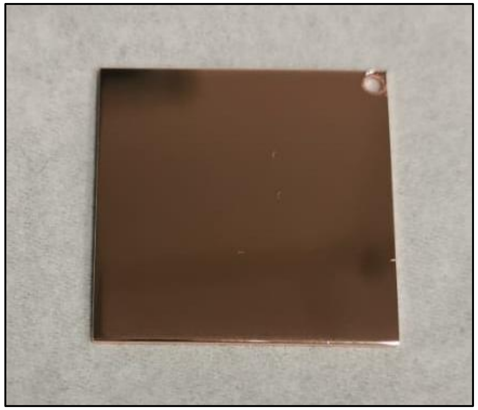
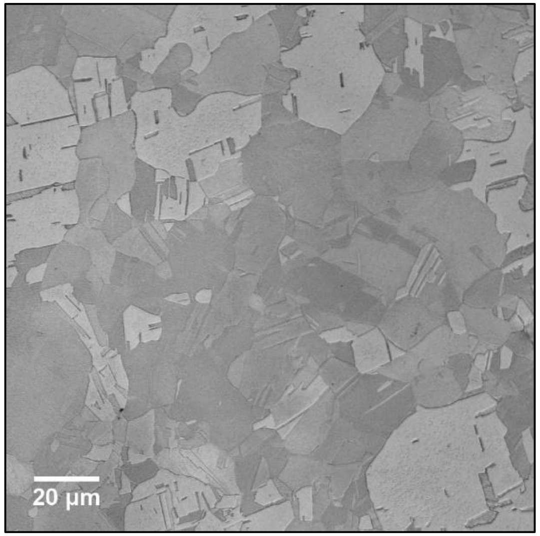
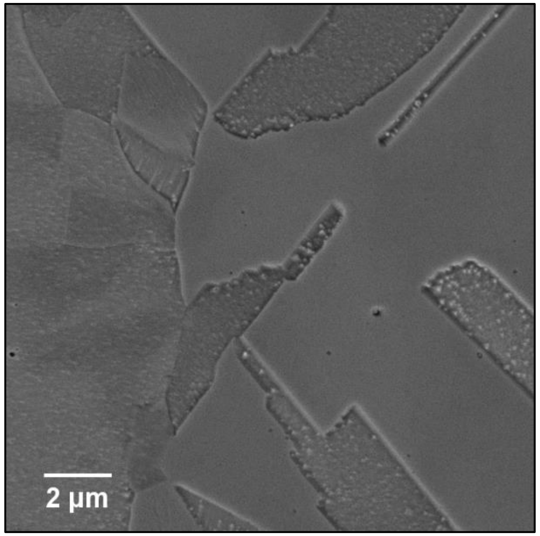






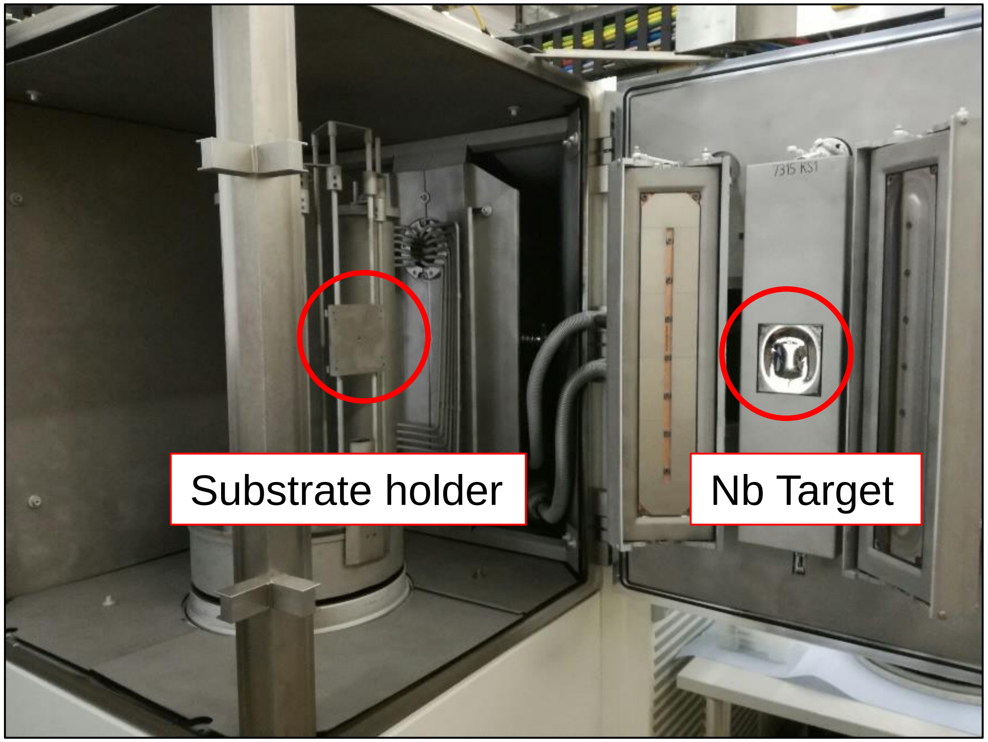




Coating Setup

Coating Setup

- Commercial high-volume, fully automated coating tool (CemeCon CC800)
  - Deposition on small flat samples (25 mm x 25 mm Cu) and QPR sample
- Base pressure of 5x10^{-7} mbar following 6 h 280°C bake
- Indirect heating of samples
- DC Power supply connected to Capacitor bank for HiPIMS operation.
- HiPIMS parameter range:
  - Pulse length: 0-200µs (20µs step)
  - Frequency: 0-2000Hz (100Hz step)



HiPIMS Nb Deposition

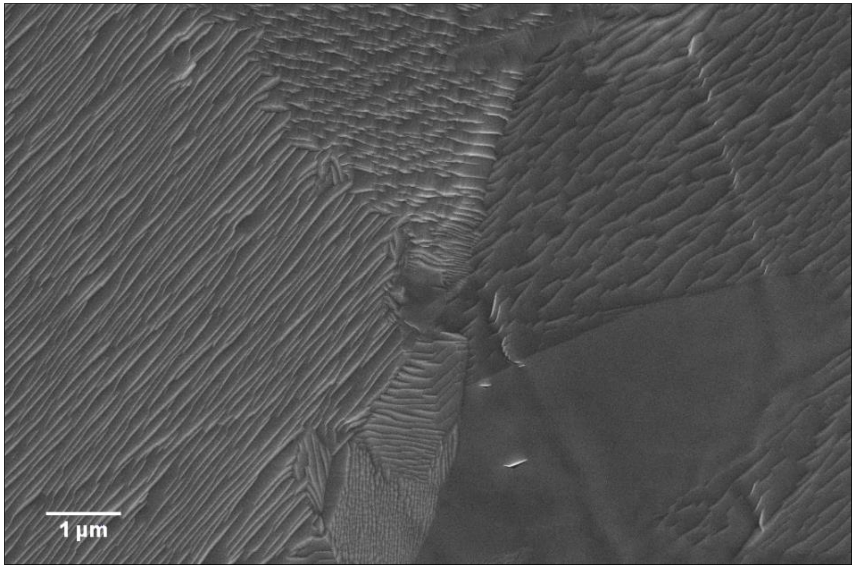
HiPIMS Nb Deposition

Deposition Parameters:

1. Cathode Power	300 to 600W
2. Deposition Pressure	800 to 1800mPa
3. Pulse Length	80 to 200µs
4. Frequency	800 to 2000Hz
5. Substrate Bias	0 to 250V
6. Substrate Temperature	120 to 290ºC
7. Film Thickness	1 to 10µm
8. Low Duty Cycle	2-4%

General Observations

- Surface topography similar for all samples.
    - Except for high substrate bias
    - Multiple grain structures present.



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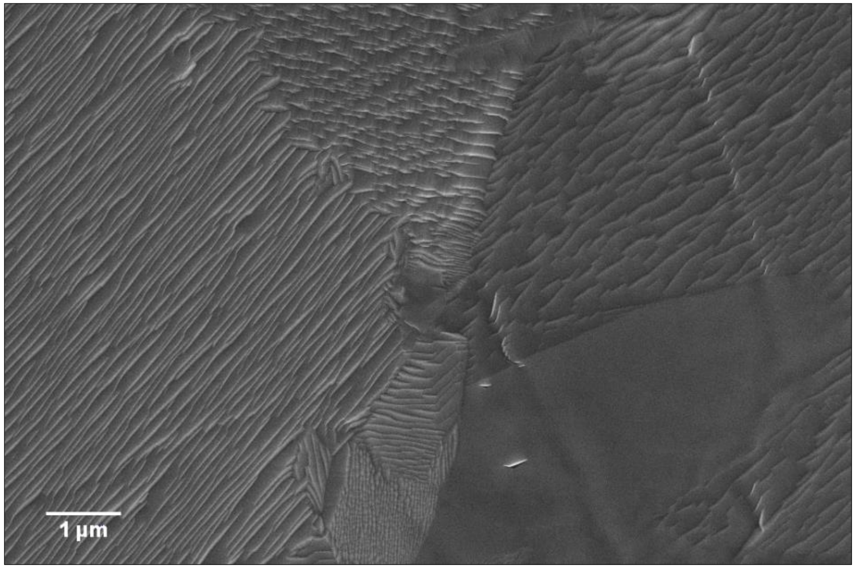
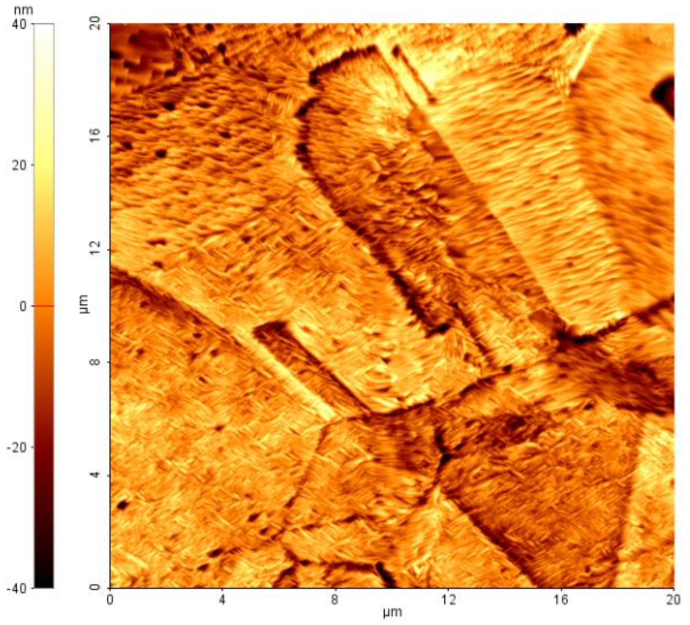
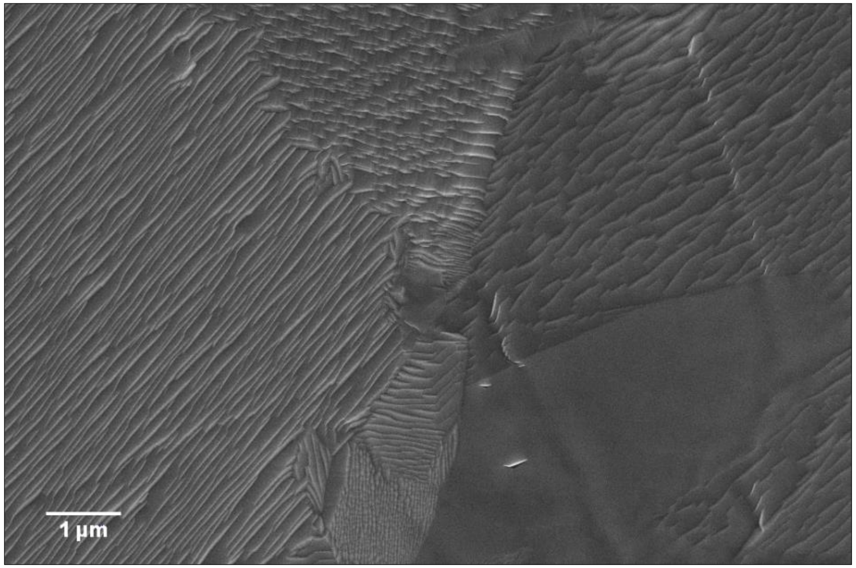
- Reduced surface roughness:
  - 11.19nm (HiPIMS) vs. 19.38nm (DC MS)

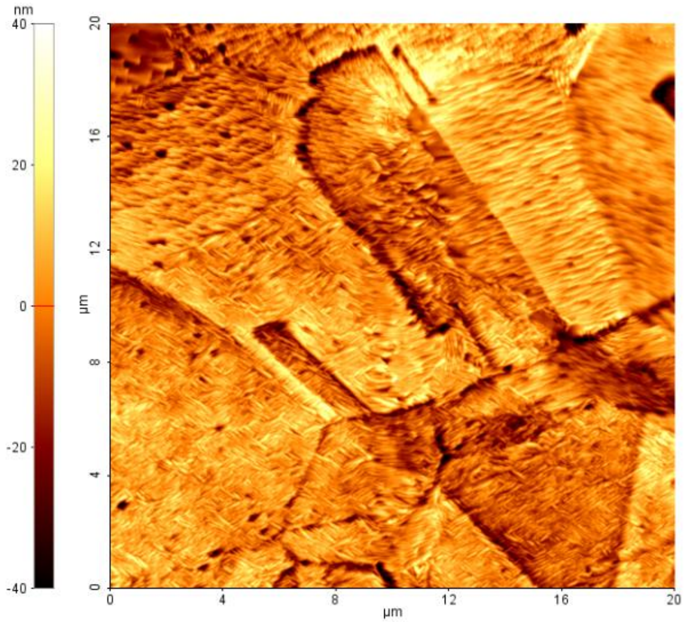


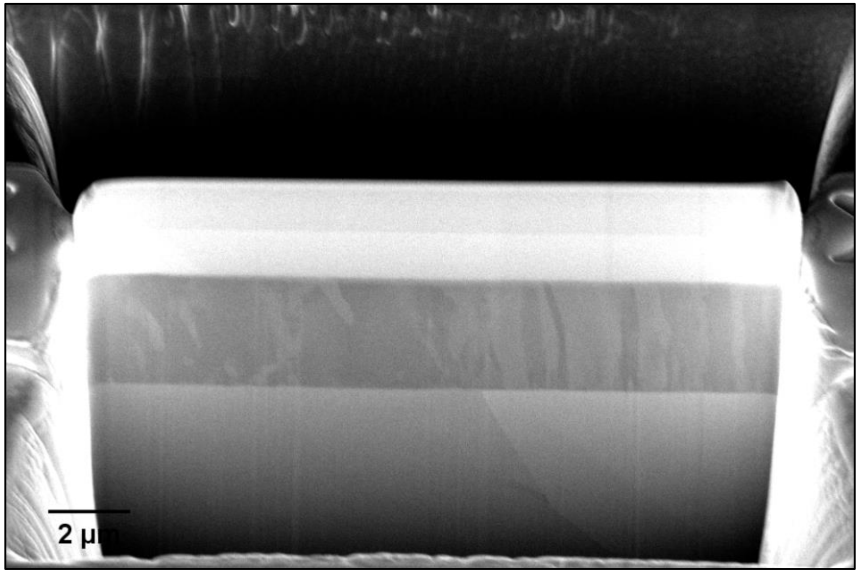


General Observations

- Surface topography similar for all samples.
  - Except for high substrate bias
  - Multiple grain structures present.
- Reduced surface roughness:
  - 11.19nm (HiPIMS) vs. 19.38nm (DC MS)
- Oxygen content generally 0% (EDX)
- Interface significantly improved. Zero voids



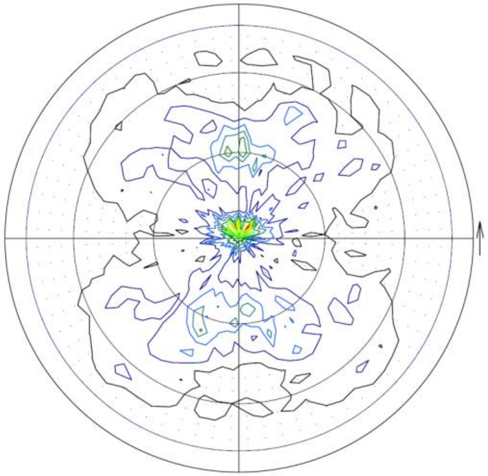


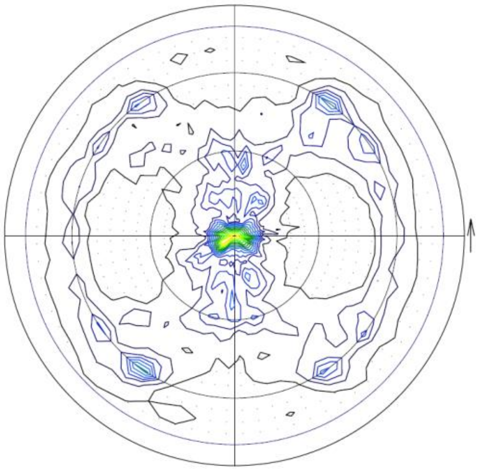


General Observations

- Pole figure measurements indicate Nb (110) / Cu (100) for all deposition conditions.
    - (Well established. See Spradlin et al, 2011)

Cu (100)





Nb (110)

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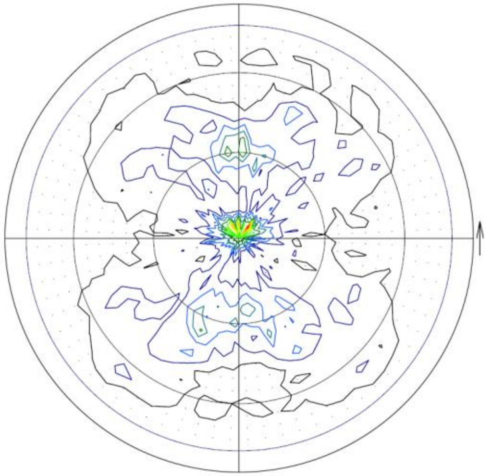
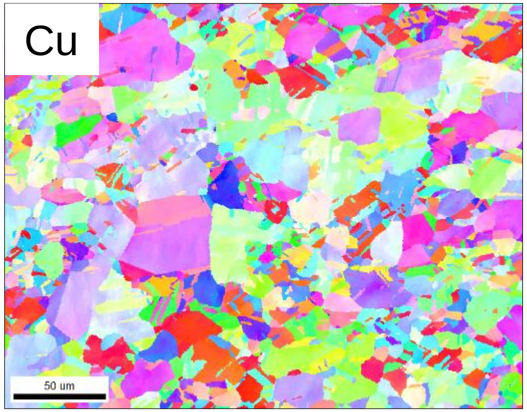
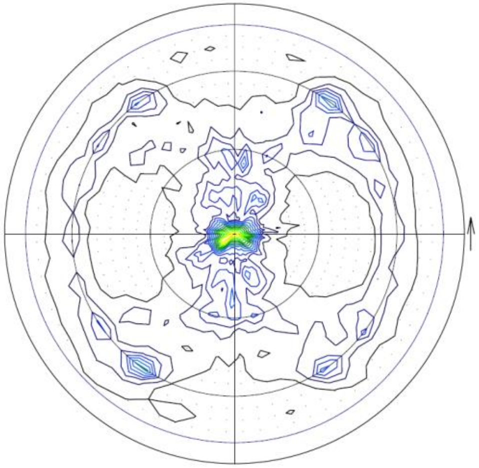
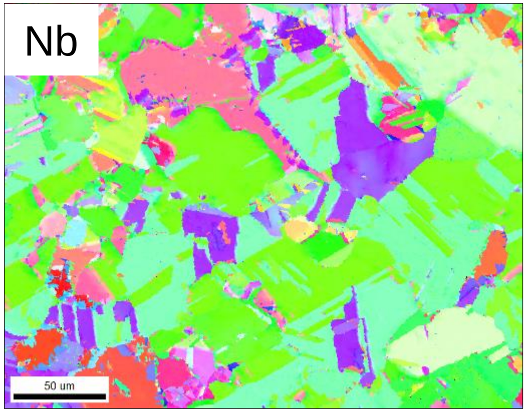
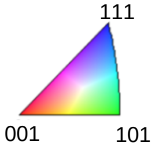
- EBSD indicates polycrystalline Nb film with no significant heteroepitaxial growth











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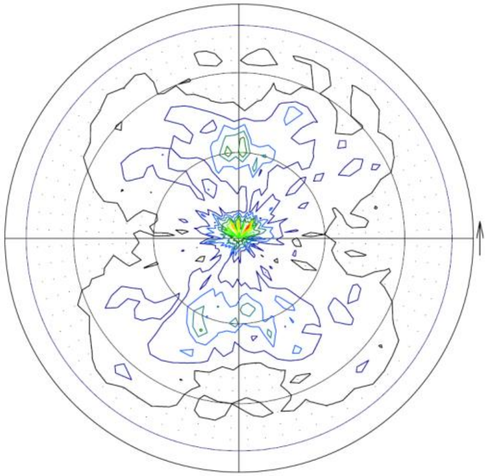
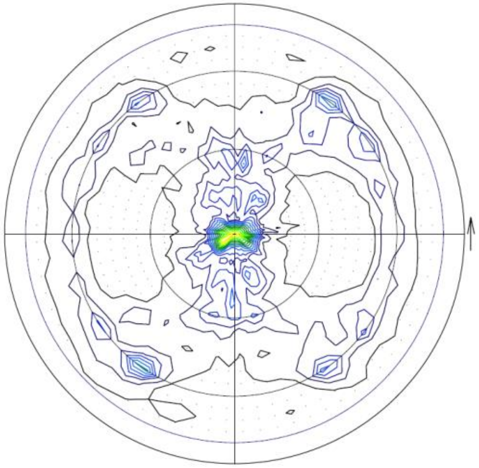
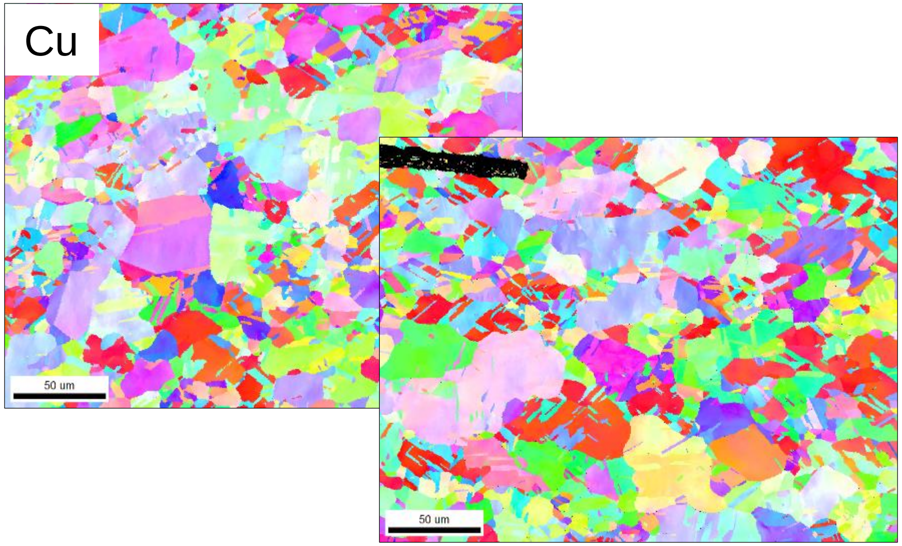


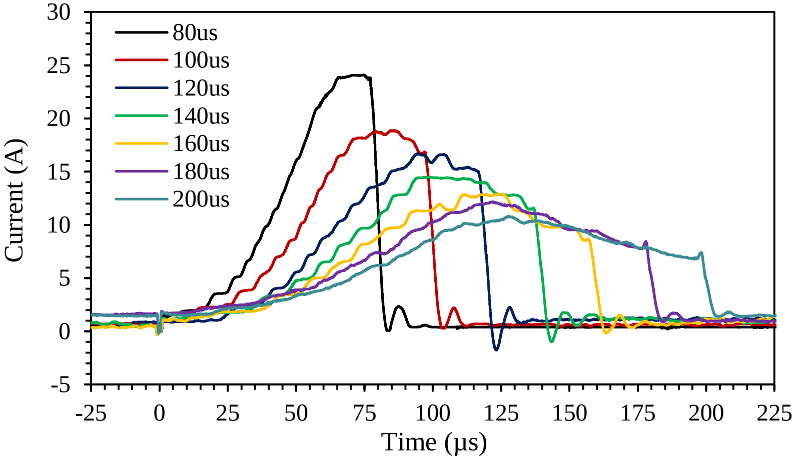


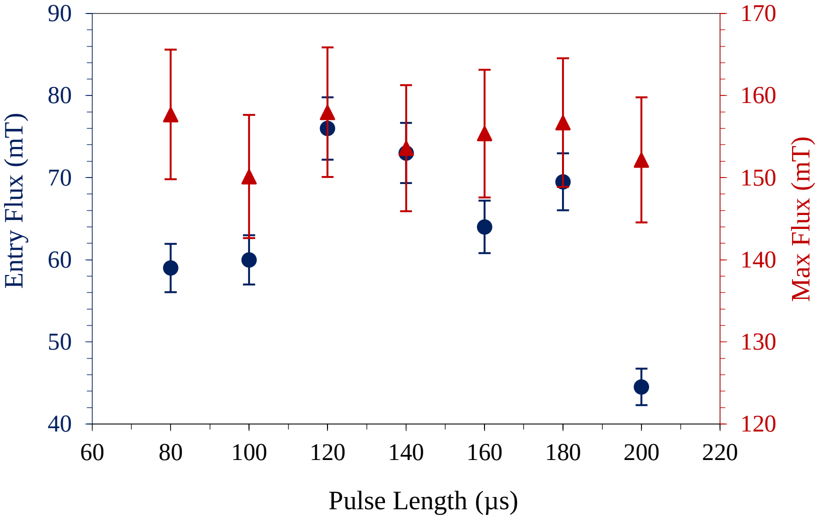
Nb (110)



Duty Cycle Effects

Pulse Length





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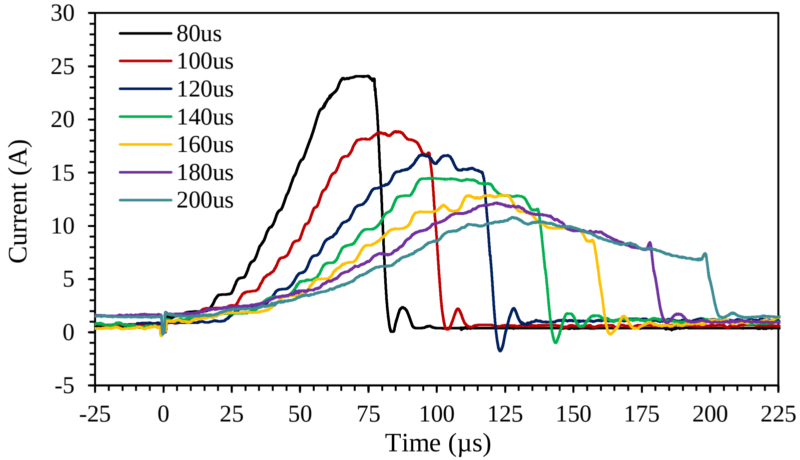
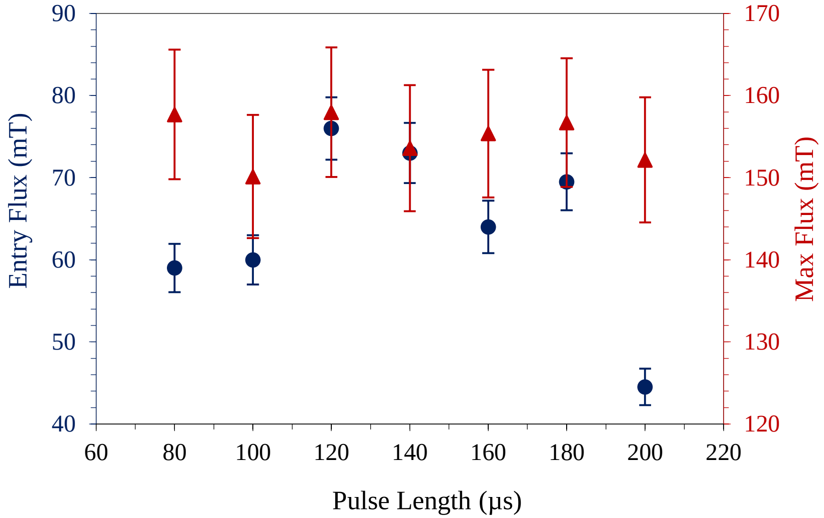
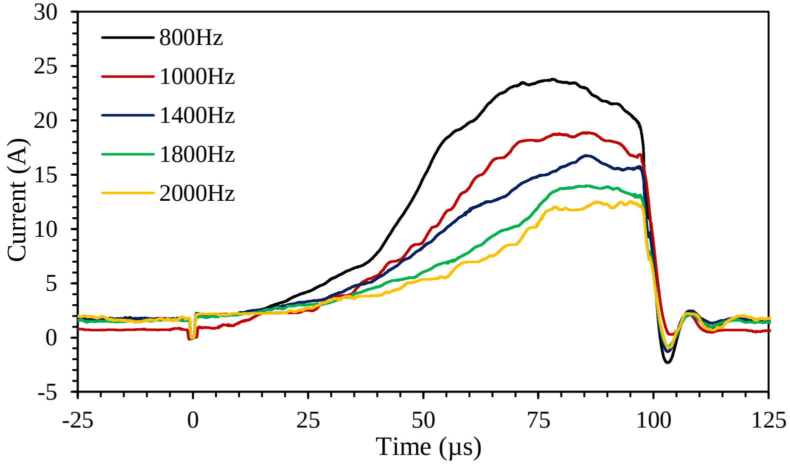
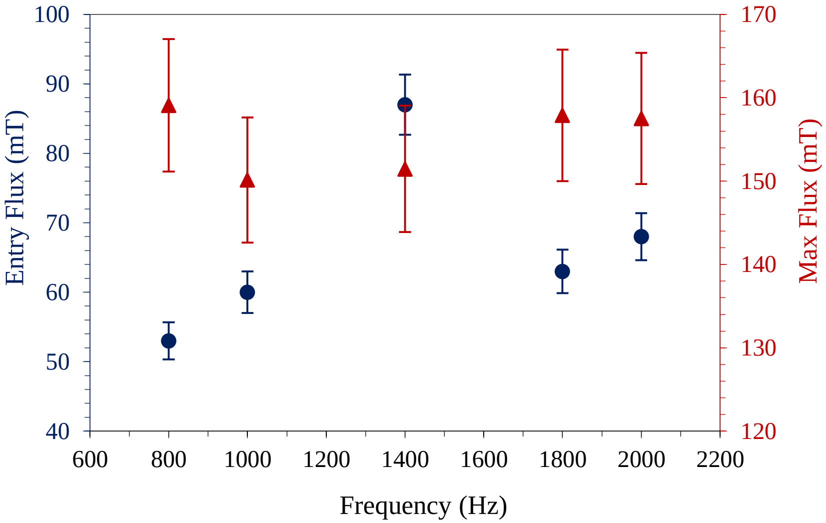




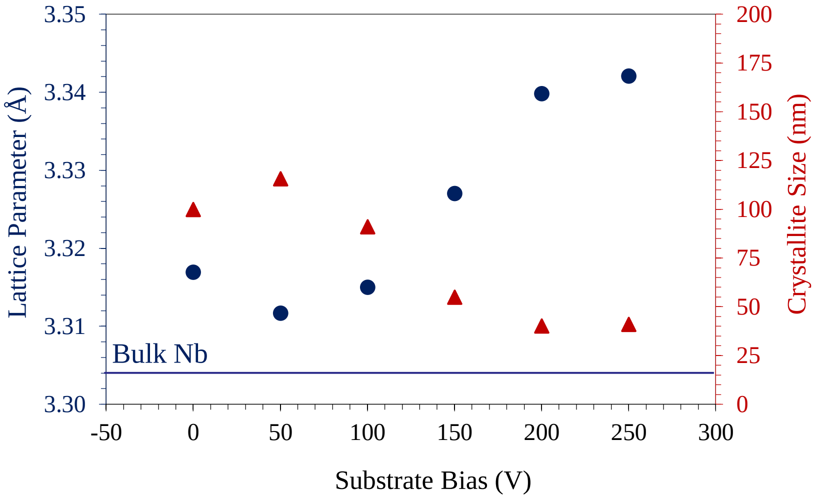
Frequency





Substrate Bias

- Duty cycle held at 10% (100µs, 1000Hz).
- Lattice parameter closest to bulk at low bias levels.



Substrate Bias

- Duty cycle held at 10% (100µs, 1000Hz).
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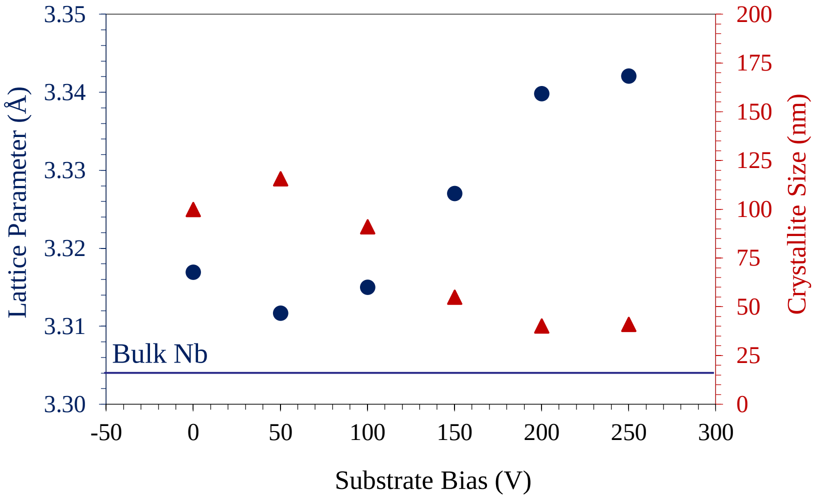
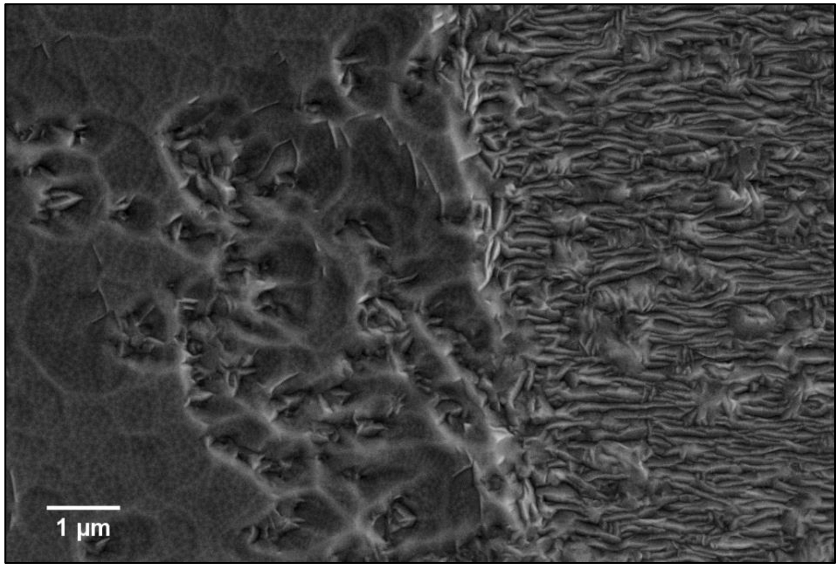
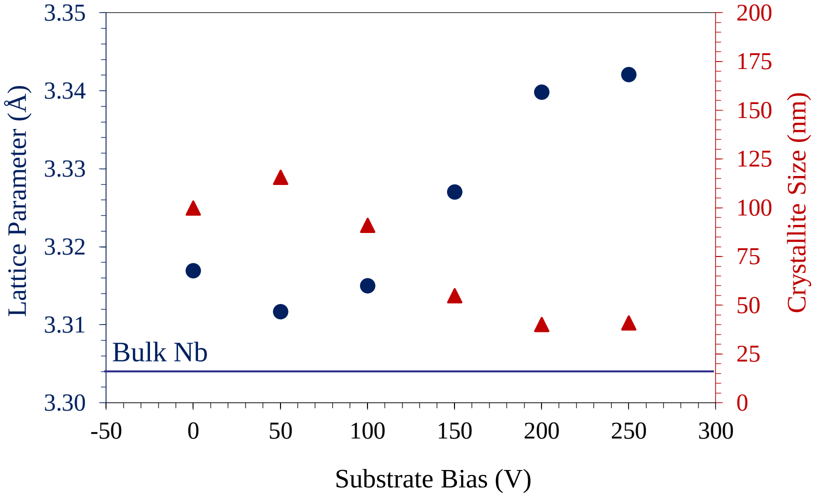
- Film surface damage at bias above 150V.

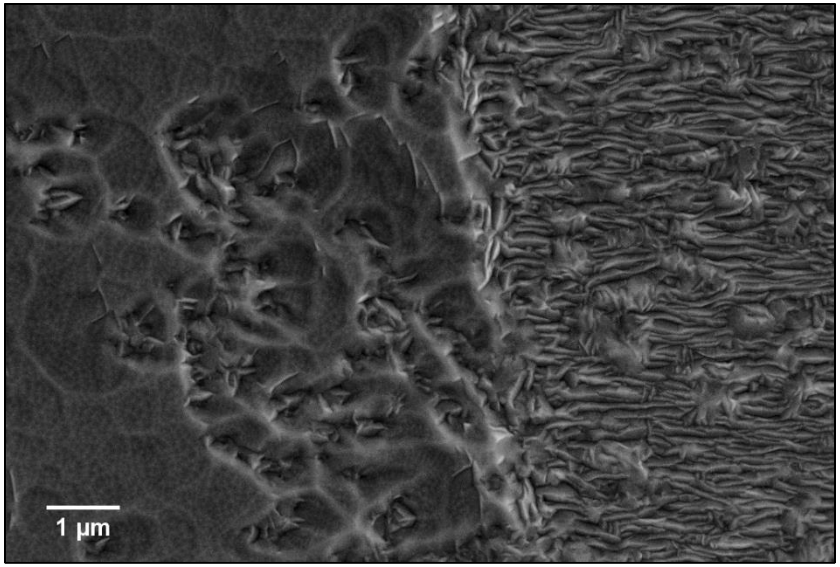


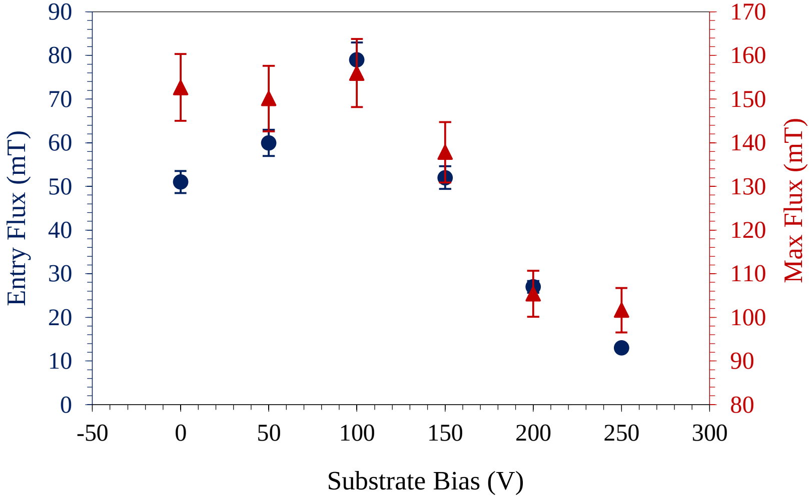


Substrate Bias

- Duty cycle held at 10% (100µs, 1000Hz).
- Lattice parameter closest to bulk at low bias levels.
- Film surface damage at bias above 150V.
- Entry flux reaches a maximum at 100V. Max flux decreases above 100V.
- Tc increases up to 9.75K at high substrate bias.

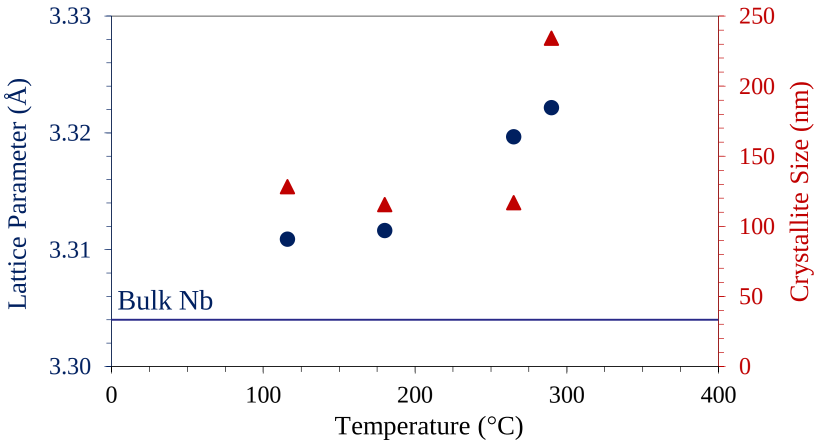


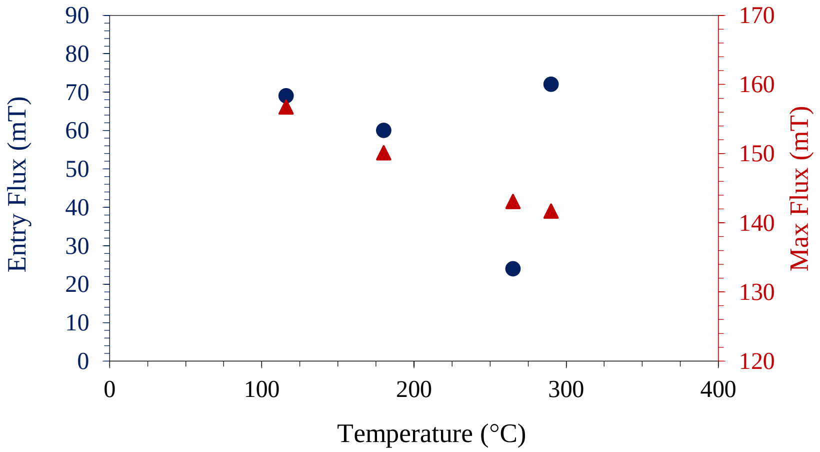




Substrate Temp + Film Thickness

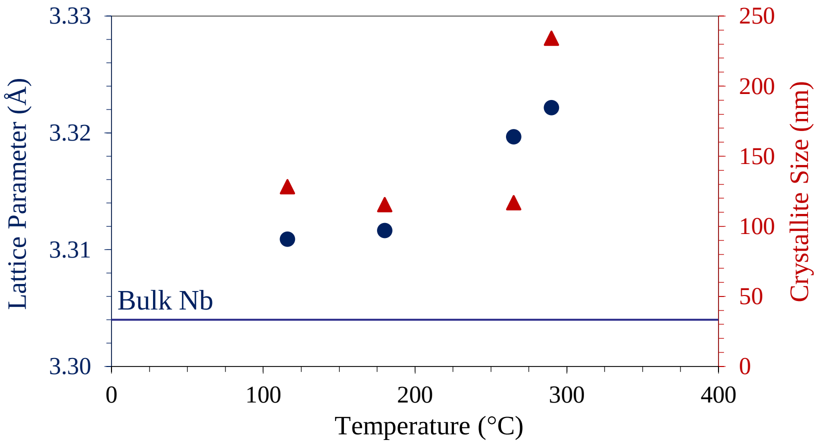
- Duty cycle held at 10% (100µs, 1000Hz) for temp investigation and 12% for thickness investigation (120µs, 1000Hz).
- Increase in film Tc to 9.65K at high Temperature

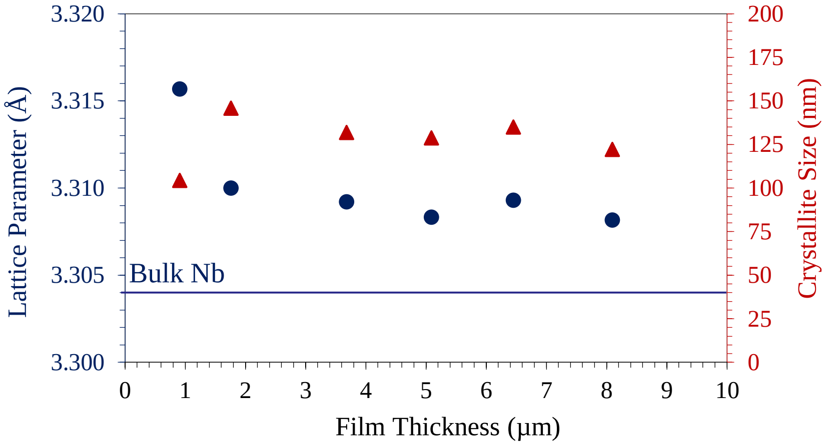


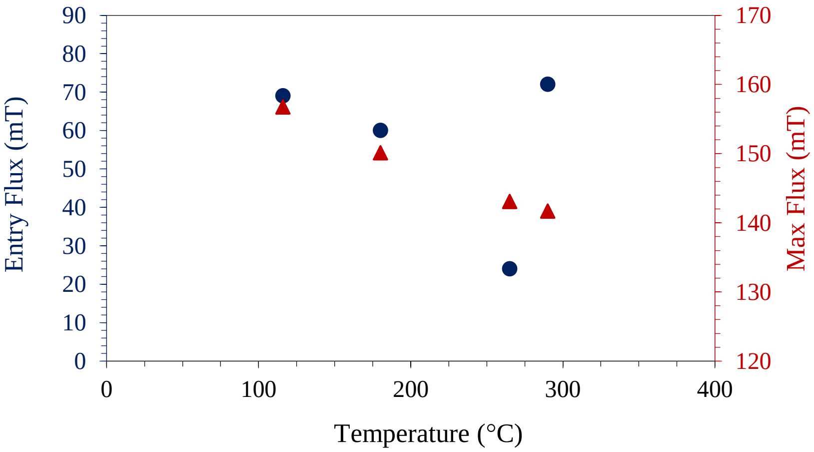


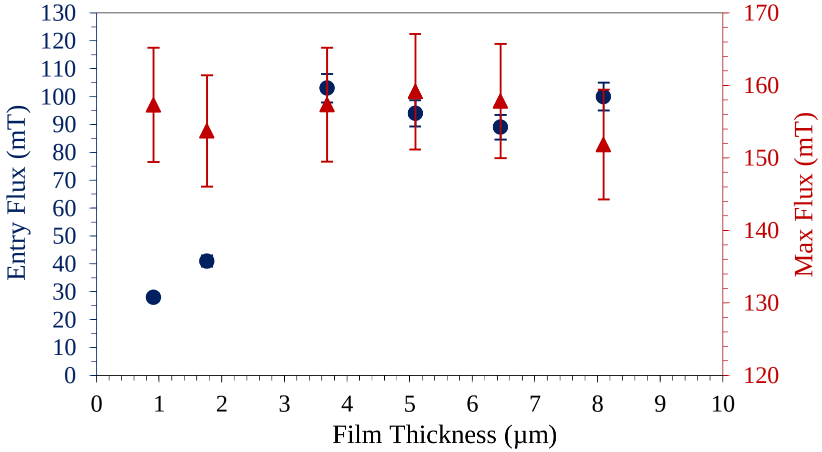
Substrate Temp + Film Thickness

- Duty cycle held at 10% (100µs, 1000Hz) for temp investigation and 12% for thickness investigation (120µs, 1000Hz).
- Increase in film Tc to 9.65K at high Temperature
- Stable, high entry flux above 3.68µm





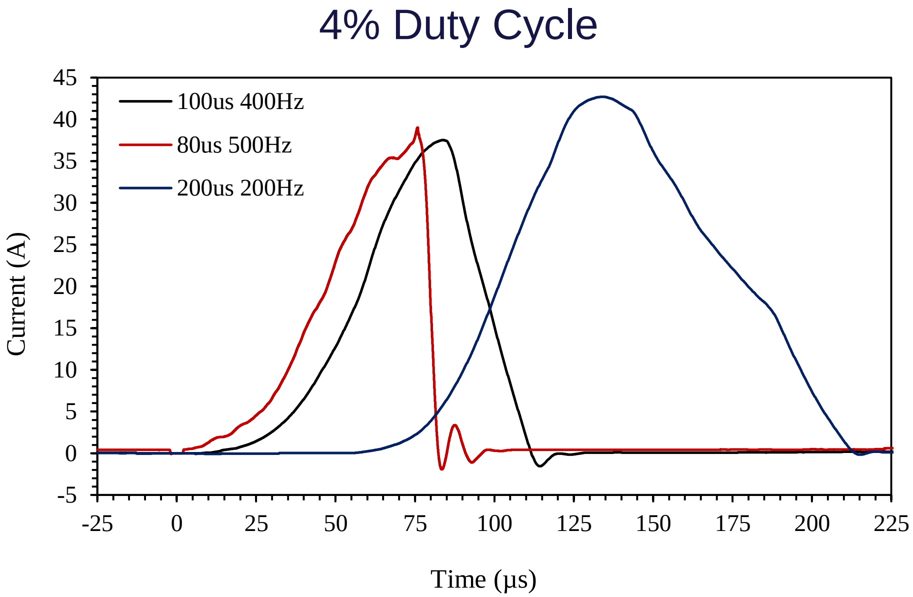




Low Duty Cycle

- Significant increase in the Max current = 55A!
    - DCMS 1.5A (constant) for ref.

- Sharper increase in current at low duty cycle



Low Duty Cycle

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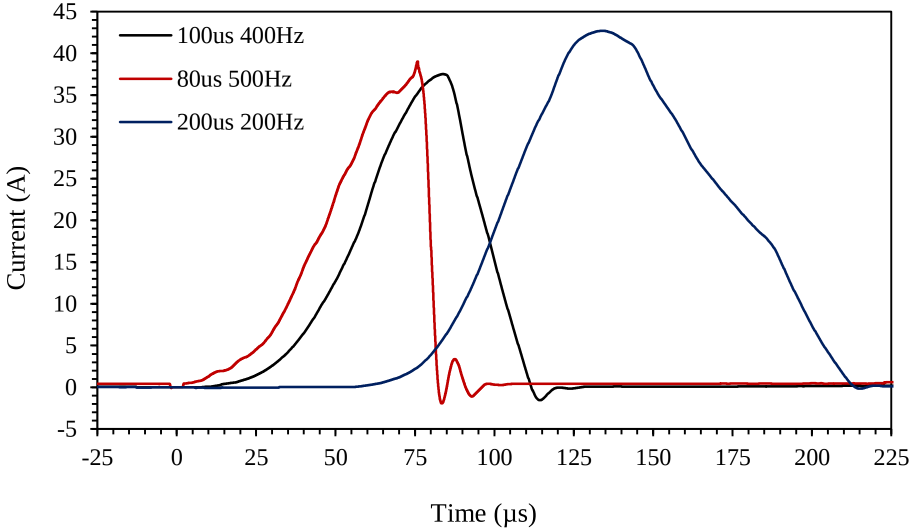
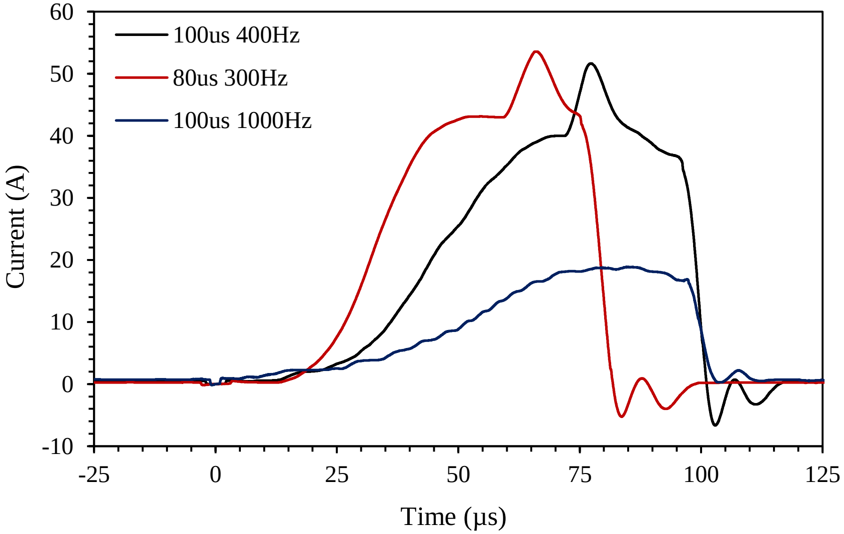
- Sharper increase in current at low duty cycle
- Appearance of self-sputtering “2nd” peak.

4% Duty Cycle

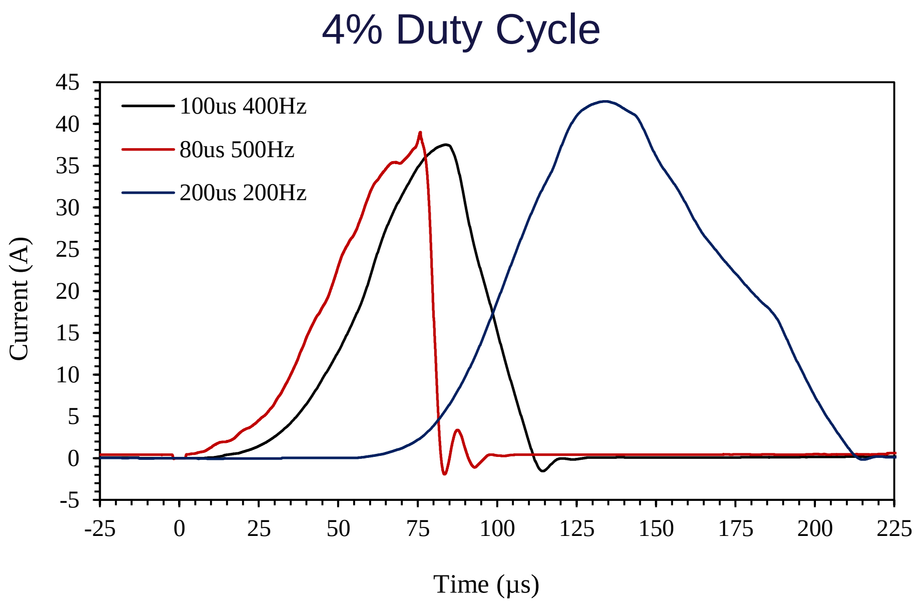


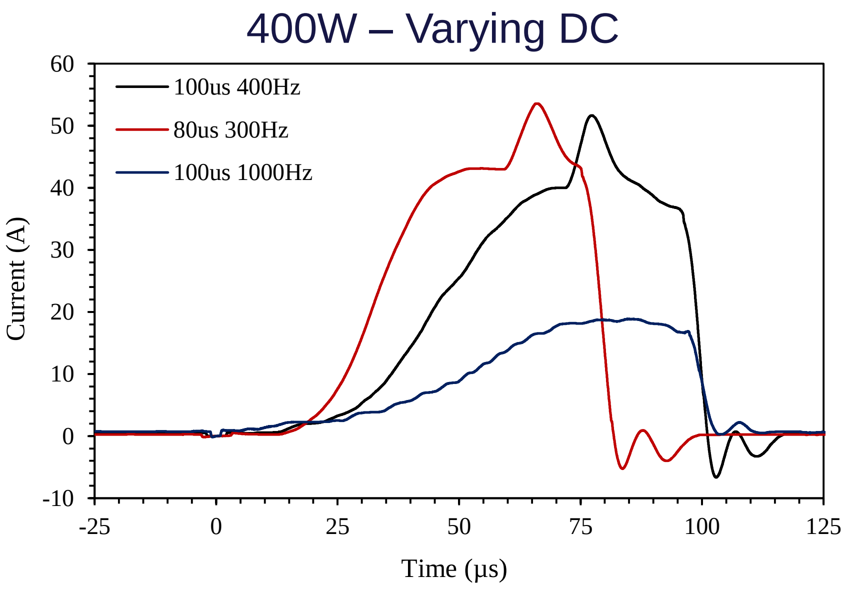
400W – Varying DC

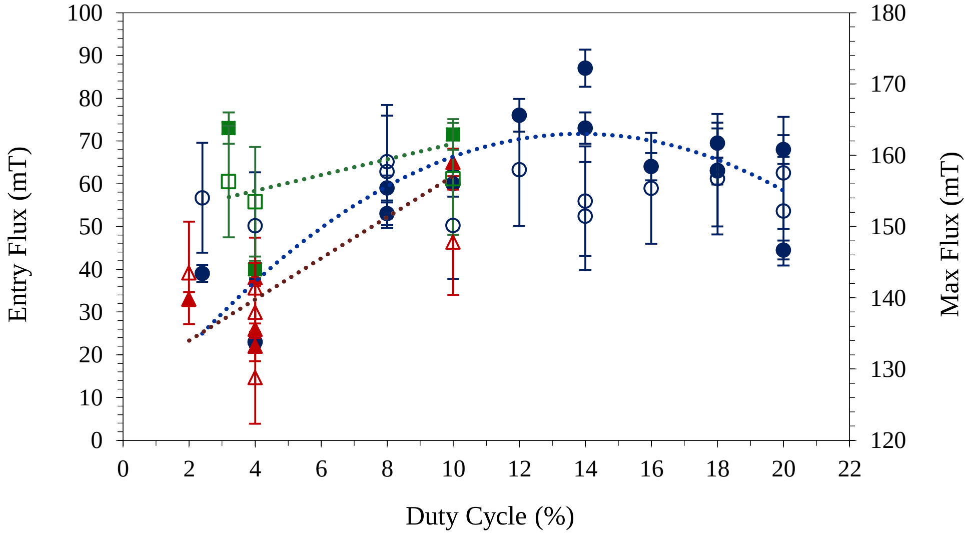


Low Duty Cycle

- Significant increase in the Max current = 55A!
    - DCMS 1.5A (constant) for ref.
- Sharper increase in current at low duty cycle
- Appearance of self-sputtering “2nd” peak.
- CERN HiPIMS at 1% DC for ref.
- Superconducting performance reaches max at medium duty cycle values


4% Duty Cycle


400W – Varying DC



Summary of HiPIMS Nb Results:

- Surface topography similar for all samples. Damage at bias >150V
- Reduced surface roughness: 9.5 ± 3.06nm vs. 19.38 ± 4.33nm
  - Increases with increased cathode power, substrate bias and film thickness

- Oxygen content <5% in all samples. Typically 0% (EDX)

- Second peak in LDC
  - Onset of self-sputtering, no runaway

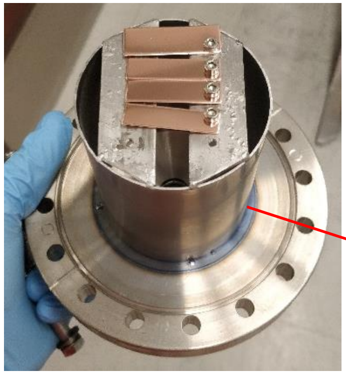
- Increased T_c at high bias and temperature (up to 9.75K). Typically = 9.3K
  - Due to increased film stresses.

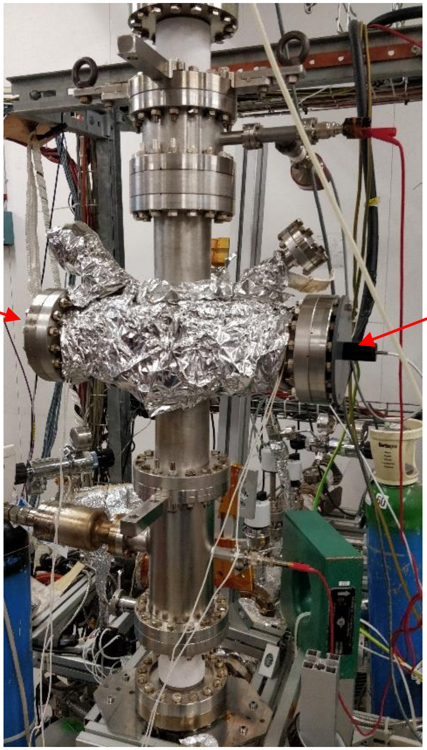
- Significant maximums in entry flux and max flux.
  - Higher cathode power and pressure
  - Mid-range duty cycle (12/14%)
  - Intermediate substrate bias (50-100V)
  - Lower substrate temperature
  - Film thickness near 3.5µm

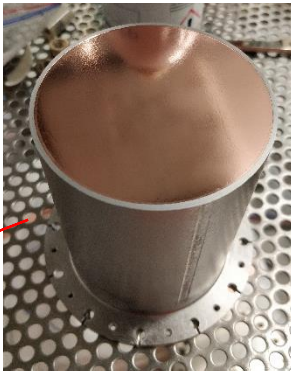
QPR HiPIMS Nb – CERN

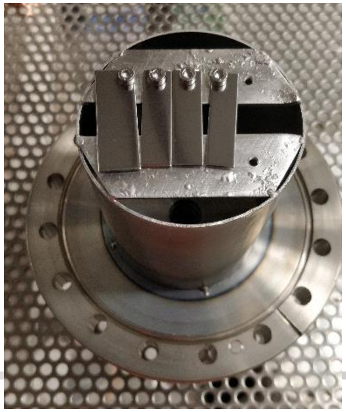
Thanks to V. Garcia (INFN) and G. Rosaz (CERN)

QPR HiPIMS Nb - CERN



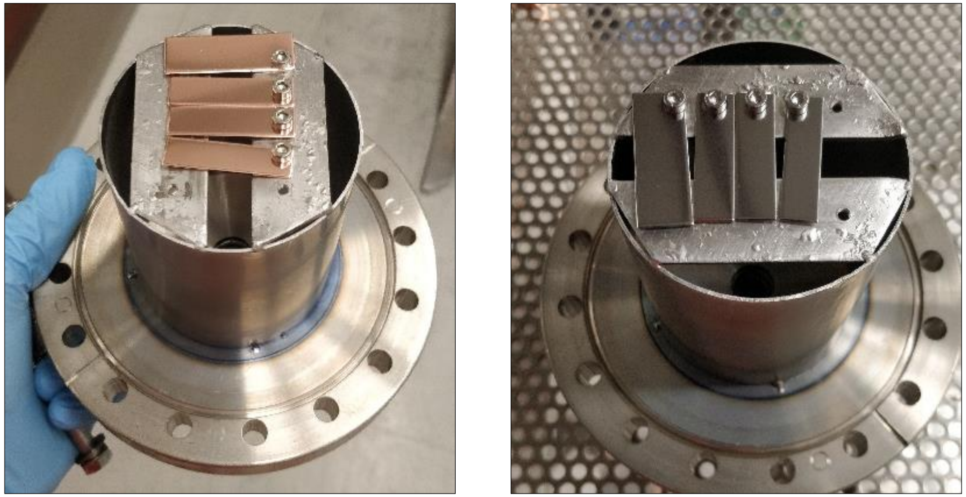


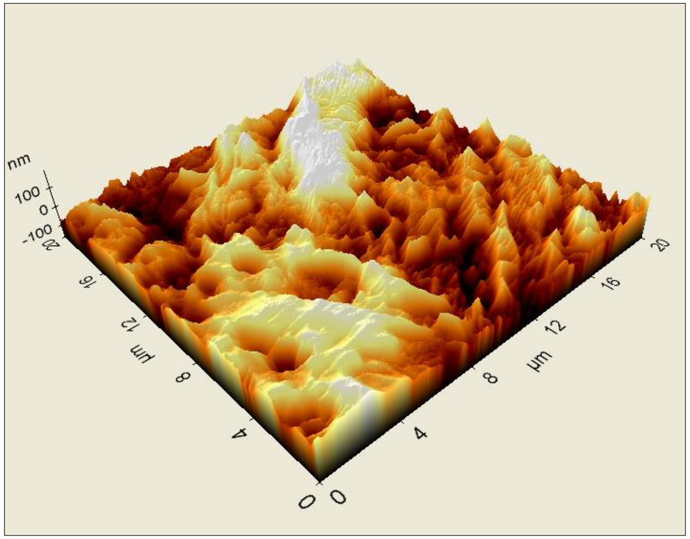


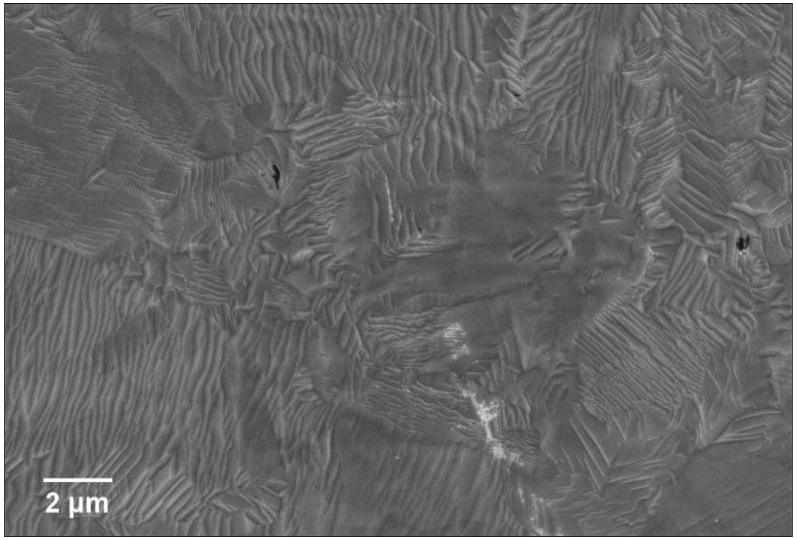


QPR HiPIMS Nb - CERN

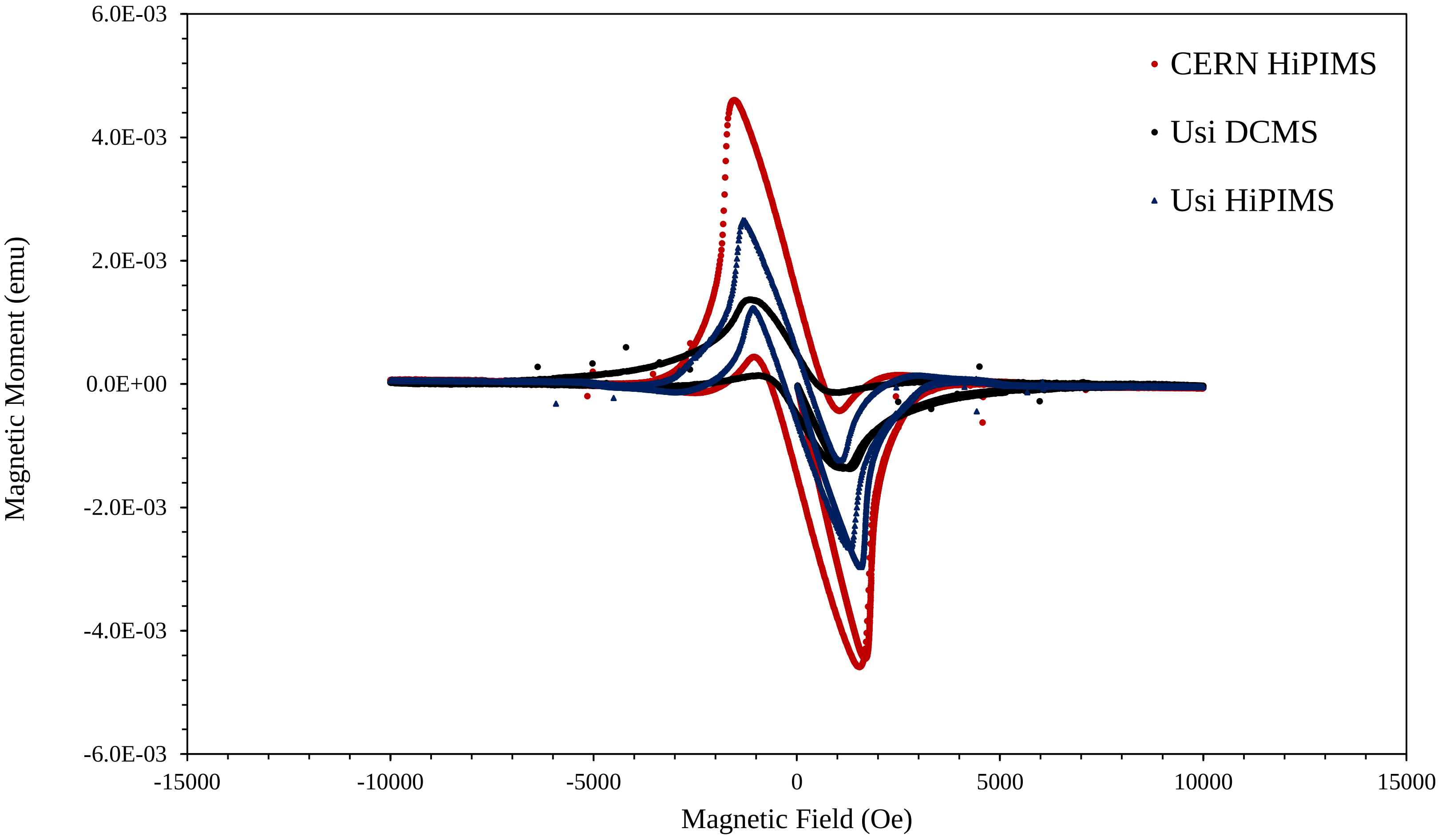
- Cu treated by SUBU
- Film thickness of 7.3µm – XRF
- Surface roughness S_q = 50.05 ± 2.24 nm
- Average crystallite size: 76 nm
- SC Properties: B_{en}: 505 Oe, T_c = 9.2 K







Magnetisation Loops



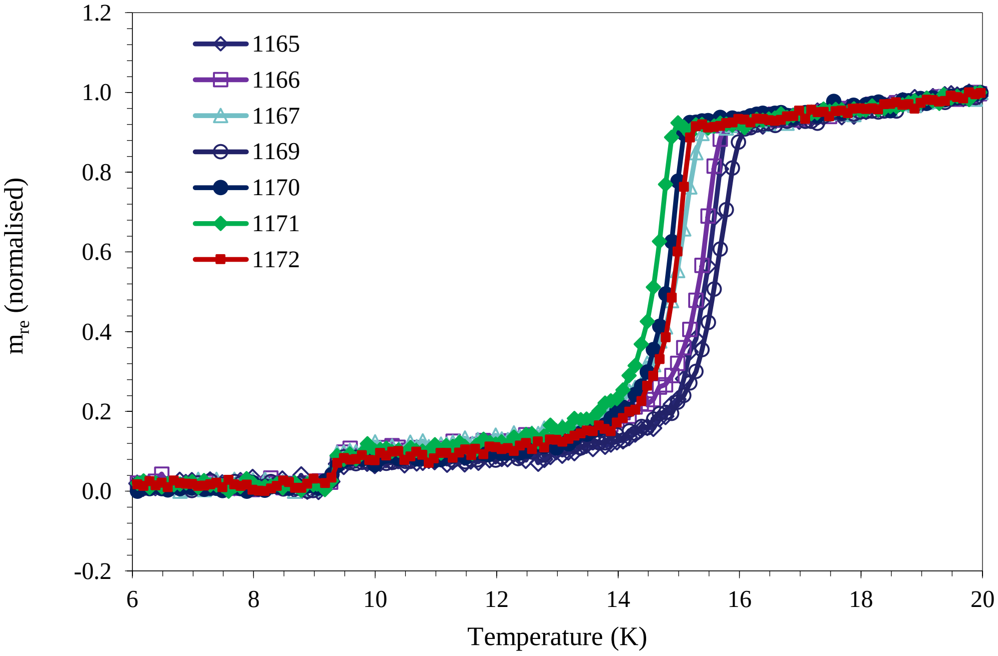
HiPIMS Nb Application

HiPIMS Nb SIS films

- Two series of HiPIMS Nb SIS samples produced (Thickness varied from 100nm to 250nm)
  1. Sample 790 recipe (Highest Ben, low Tc-11.7K)
  2. Sample 899 recipe (Highest Tc-16.1K), low Ben)

HiPIMS Nb SIS films

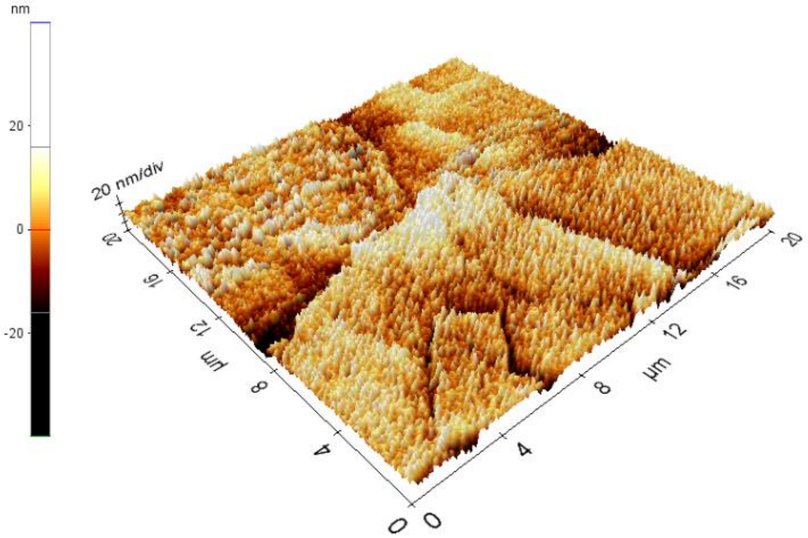
- Two series of HiPIMS Nb SIS samples produced (Thickness varied from 100nm to 250nm)
  1. Sample 790 recipe (Highest Ben, low Tc-11.7K)
  2. Sample 899 recipe (Highest Tc-16.1K), low Ben)
- Distinct differences observed.
  - All samples coated with 790 recipe display higher Ben (>600 Oe) NbN recipe influence
- Increased Tc with thickness
  - Max Tc equal to best single film value – 16.1K
  - Sample 790 recipe films show higher transition temperature in SIS films



Sample	Entry Flux (Oe)	Transition Temp (NbN) (K)
1165 (200nm)	390	15.9
1166 (150nm)	380	15.8
1167(100nm)	340	15.5
1169 (250nm)	330	16.1
1170 (200nm)	610	15.2
1171 (150nm)	615	15
1172 (250nm)	590	15.3

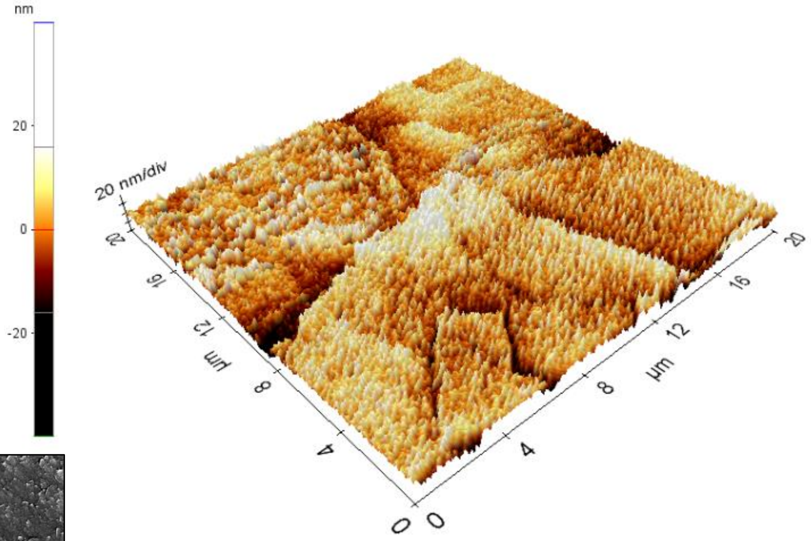
HiPIMS Nb SIS films

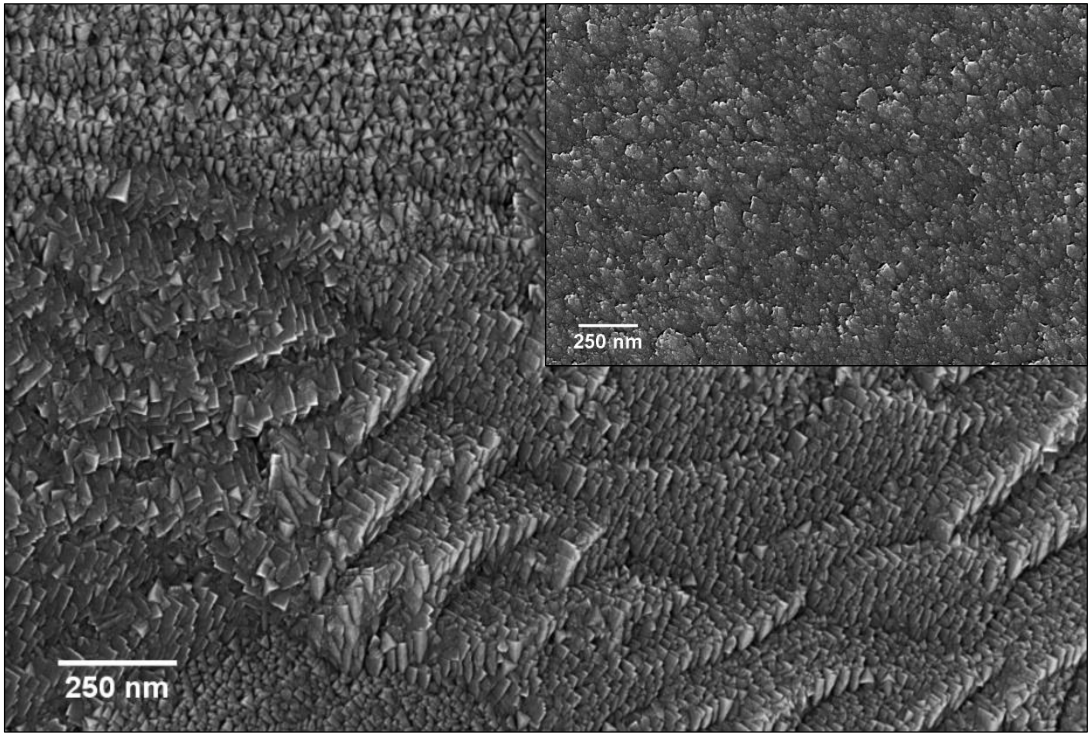
- Surface roughness of HiPIMS Nb SIS films reduced



HiPIMS Nb SIS films

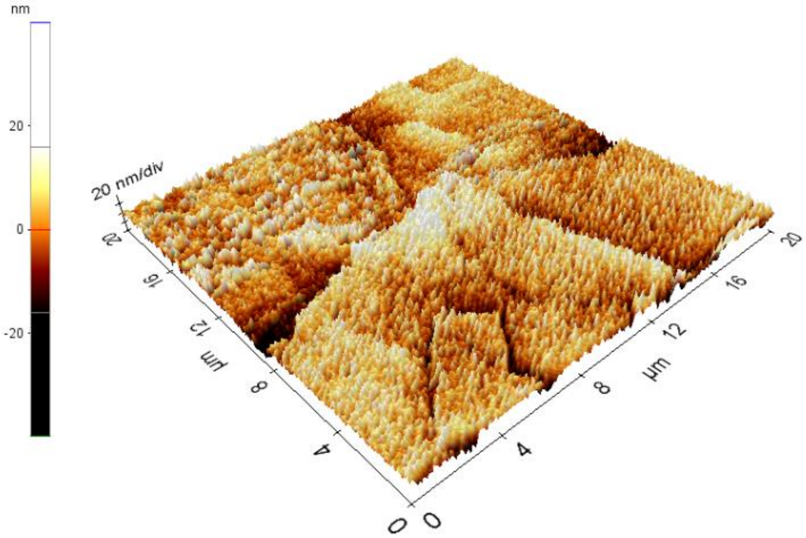
- Surface roughness of HiPIMS Nb SIS films reduced
- Microstructure of films changed compared to single layer films

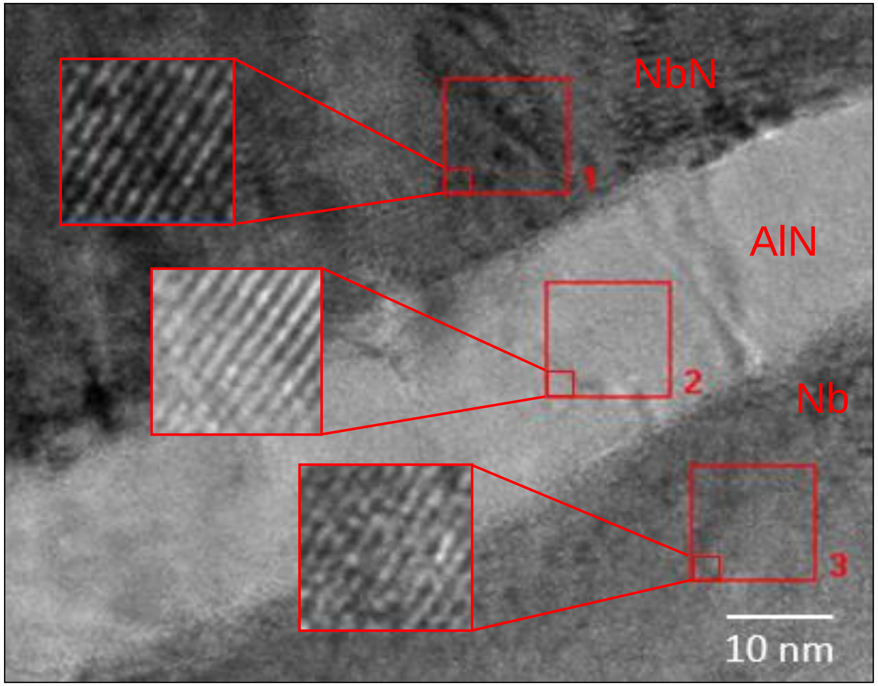




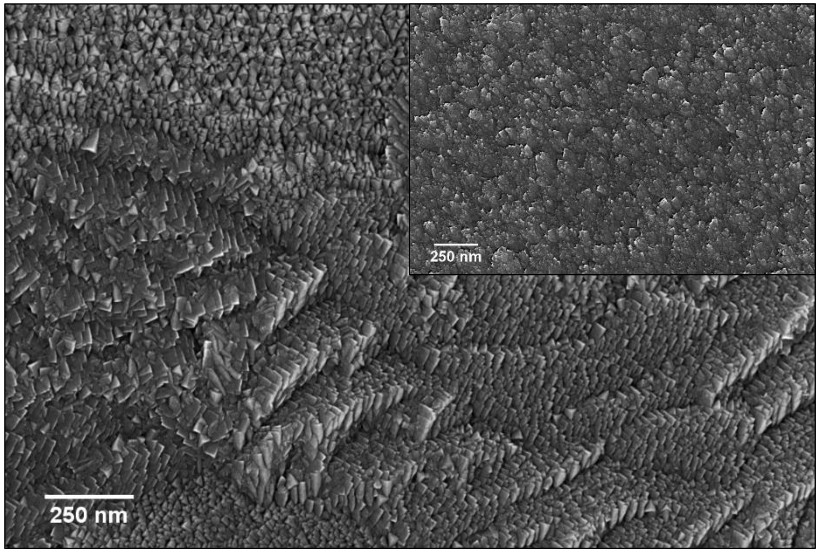
HiPIMS Nb SIS films

- Surface roughness of HiPIMS Nb SIS films reduced
- Microstructure of films changed compared to single layer films
- HRTEM shows lattice matching (heteroepitaxial growth)





Courtesy: Ying Li



Thank You!

Extra Slides

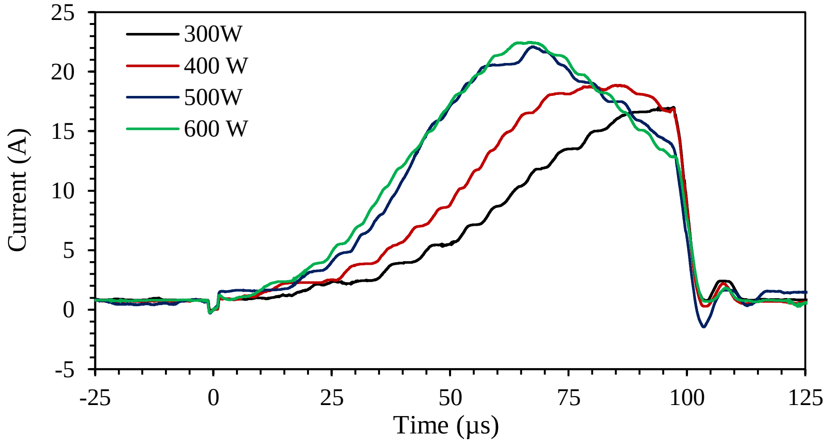
Cathode Power

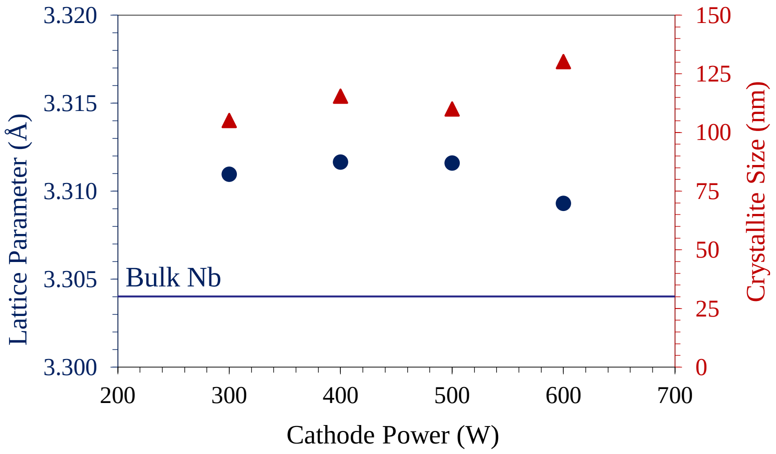
- Current increases with increasing power. Rate of increase is also greater. Peak position shifts earlier.

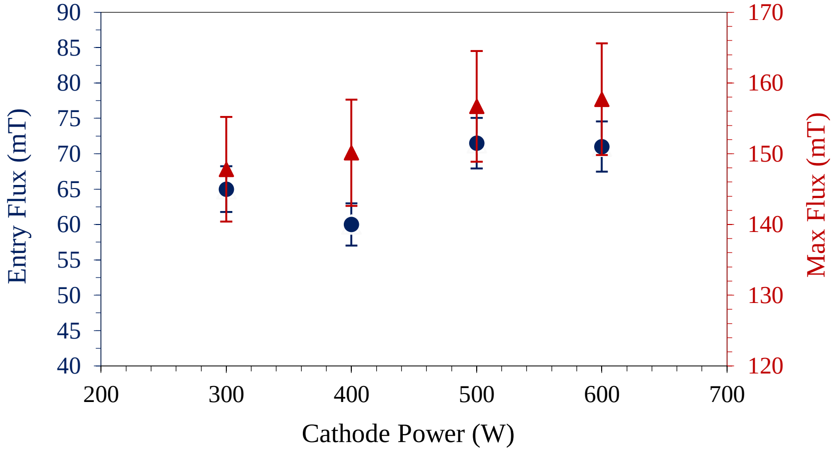
- Corresponding increase in deposition rate.

- Lattice parameters are relatively equal. Slight decrease at high power.

- SC Measurements completed with VSM. Entry flux and Max flux show an increase with increasing cathode power.







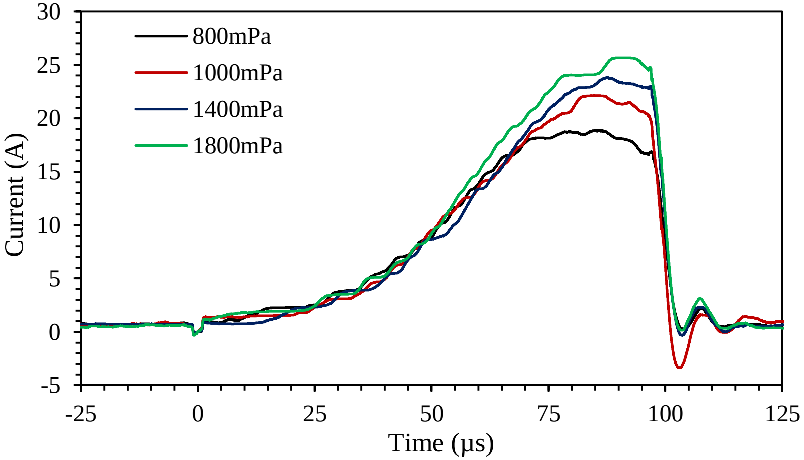
Deposition Pressure

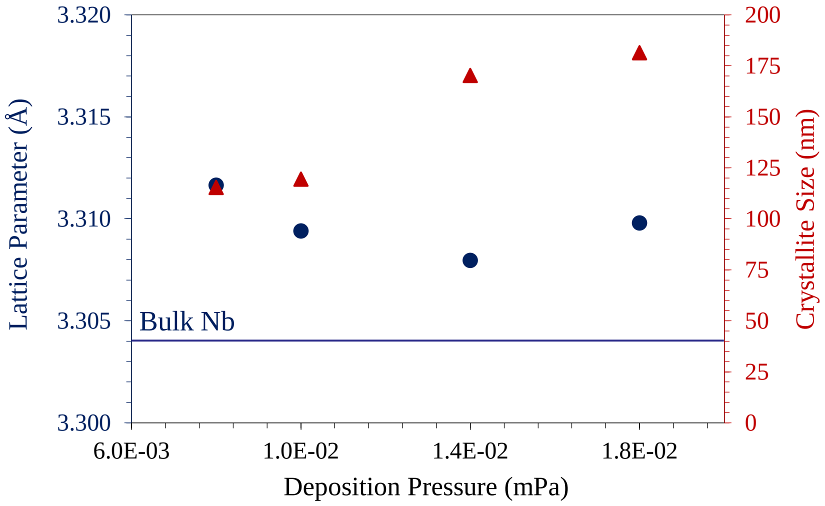
- Current increases with increasing pressure. Rate of increase is similar. Peak position shifts later.

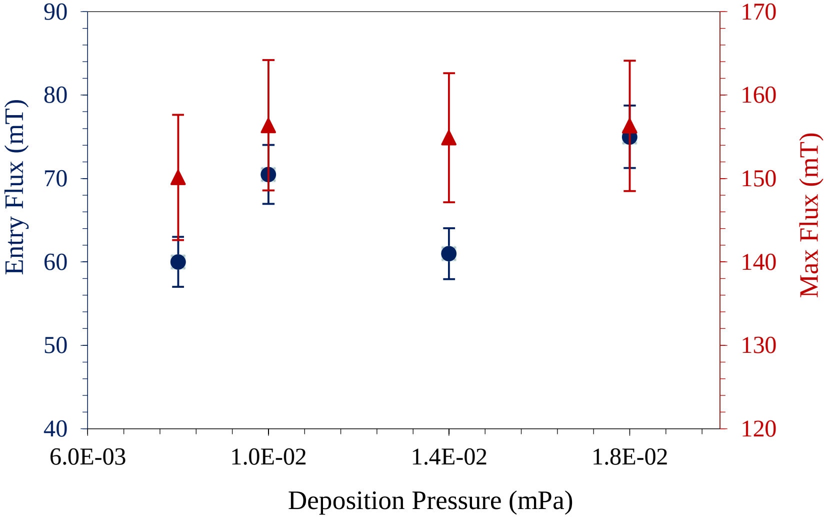
- Deposition rate remains the same.

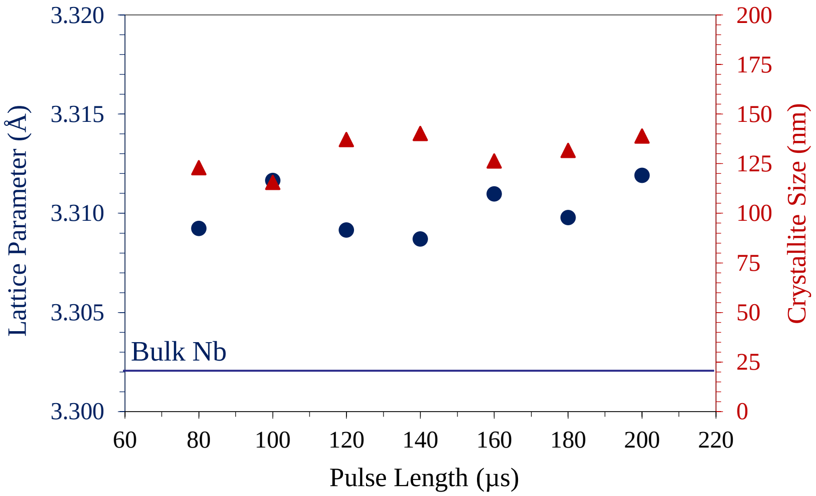
- Lattice parameters approach bulk at intermediate values. Crystallite sizes increase with increasing pressure.

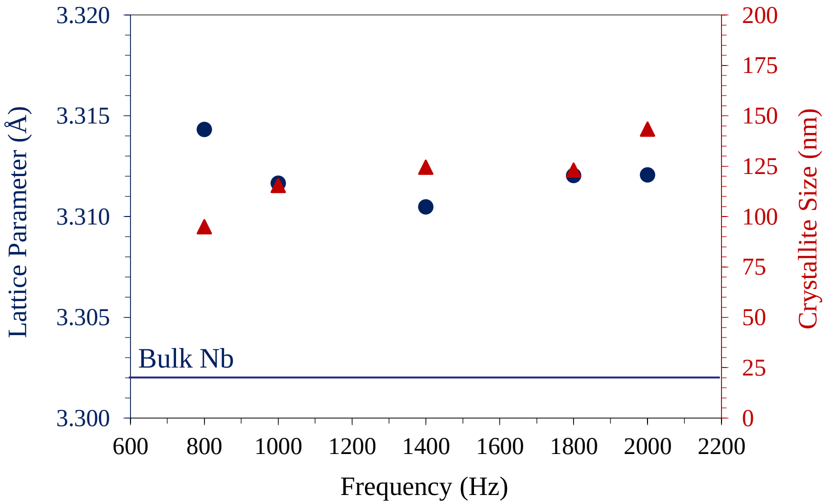
- Entry flux value at 1.4E-02 presumed incorrect. Remeasure to be completed.








Lattice Parameter (Å)
3.320
3.315
3.310
3.305
3.300
Crystallite Size (nm)
200
175
150
125
100
75
50
25
0
Bulk Nb
60
80
100
120
140
160
180
200
220
Pulse Length (µs)


Lattice Parameter (Å)
3.320
3.315
3.310
3.305
3.300
Crystallite Size (nm)
200
175
150
125
100
75
50
25
0
Bulk Nb
600
800
1000
1200
1400
1600
1800
2000
2200
Frequency (Hz)