The Development of HiPIMS Multilayer SIS film coatings on Copper for SRF Applications

9th international thin film workshop, 15-18 March 2021

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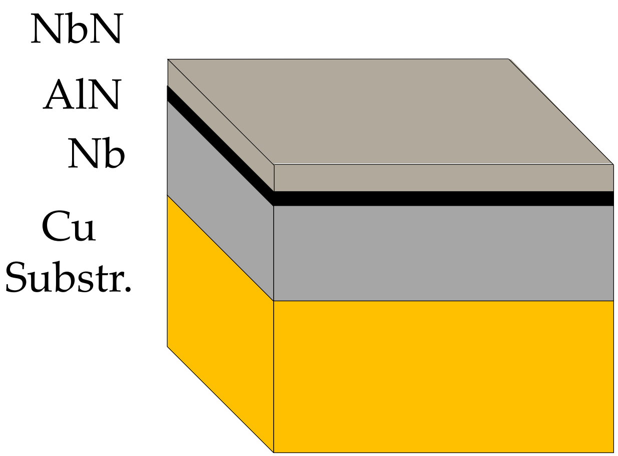
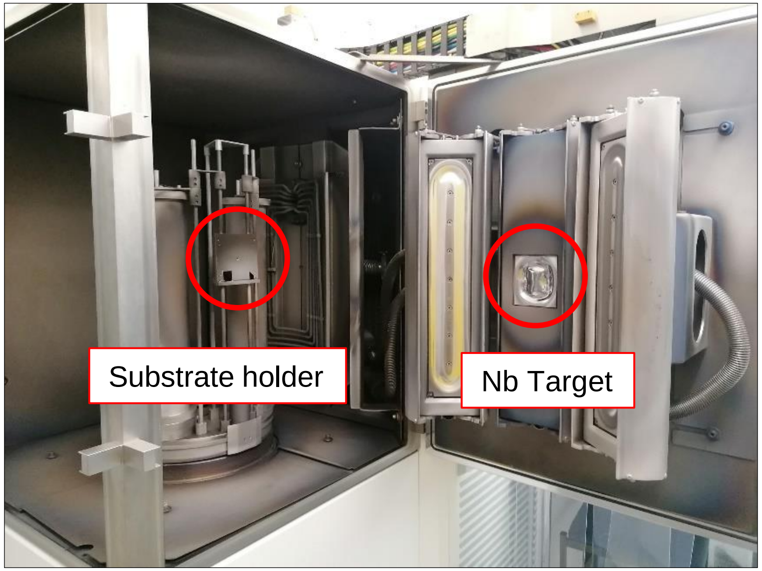
Experimental Outline

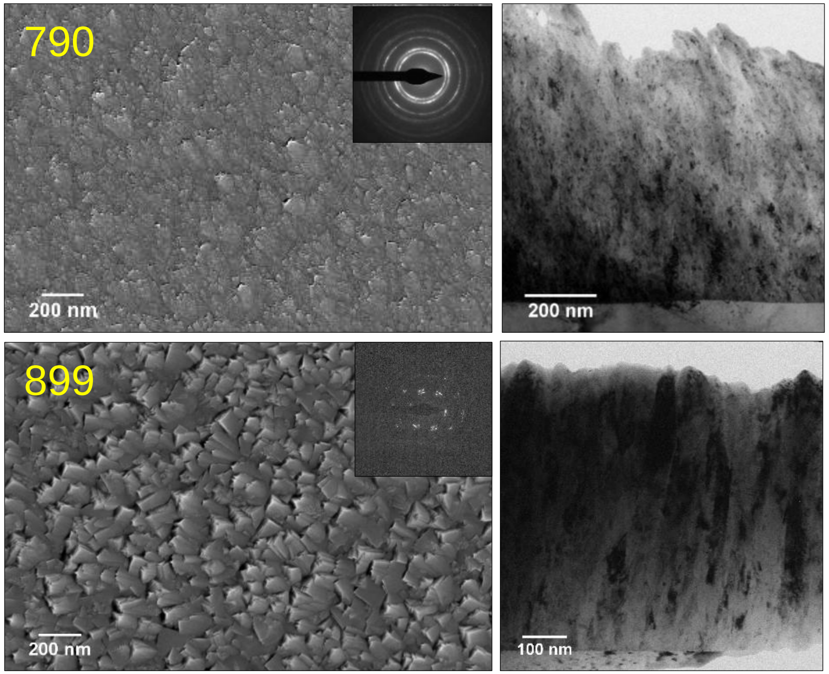
1. DC MS NbN optimisation
   - DC MS based SIS films
2. HiPIMS Nb optimisation
   - HiPIMS Nb + DC MS NbN SIS films
3. HiPIMS NbN optimisation
   - HiPIMS SIS films





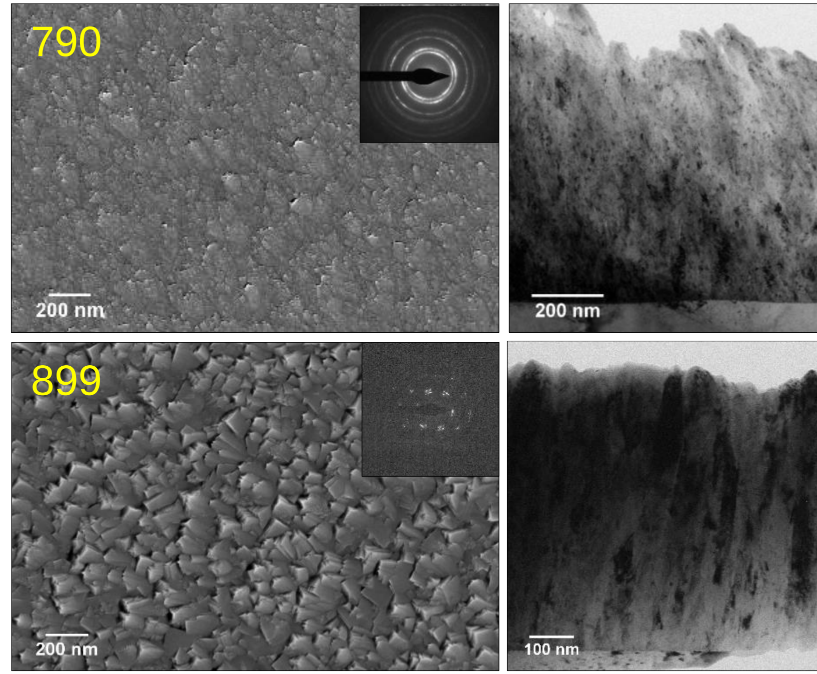
DC MS SIS films

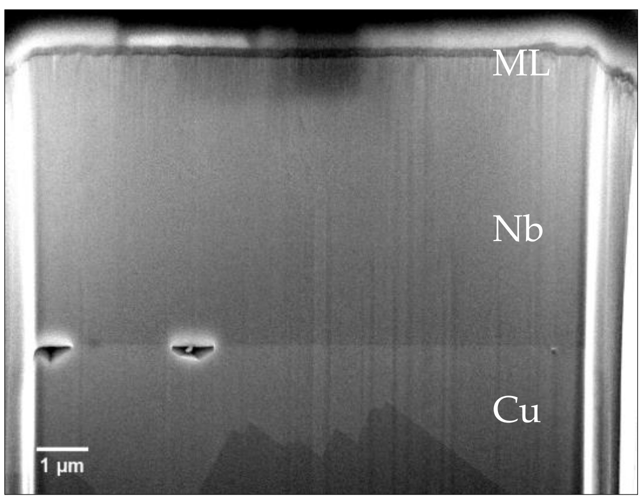
- Optimised DC MS NbN recipe
  - Highest Hen = 13.0 mT (790)
  - Highest Tc = 16.1 K (899)

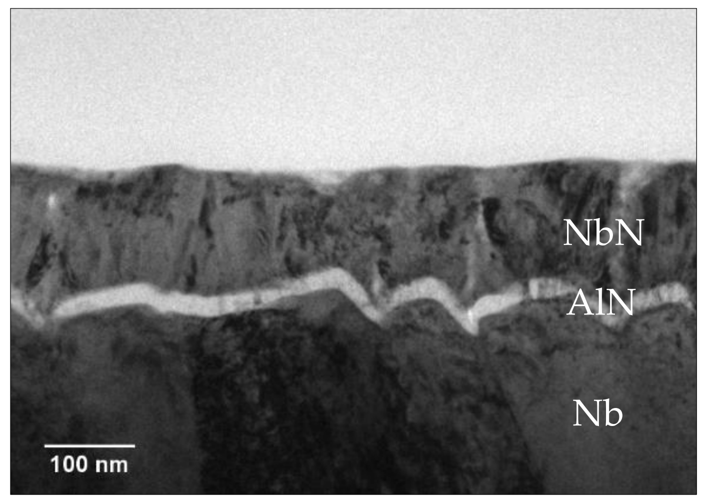


DC MS SIS films

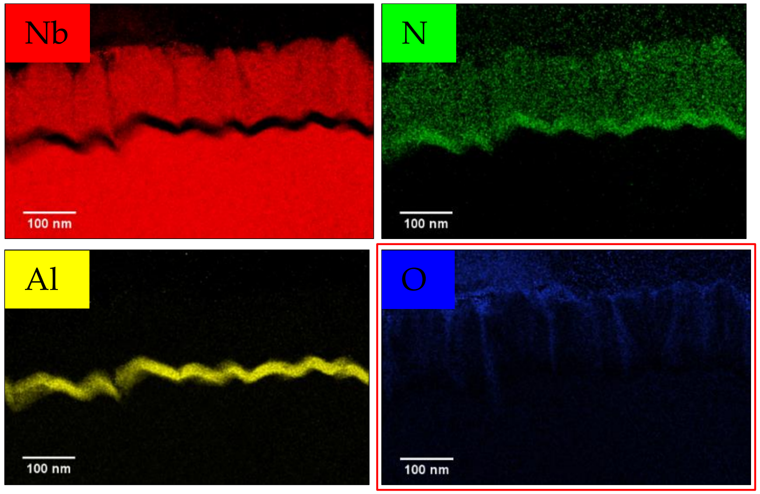
- Optimised DC MS NbN recipe
  - Highest Hen = 13.0 mT (790)
  - Highest Tc = 16.1 K (899)
- DC MS SIS film
  - Table rotated during coating
- Poor interface adhesion
- Rough Nb layer



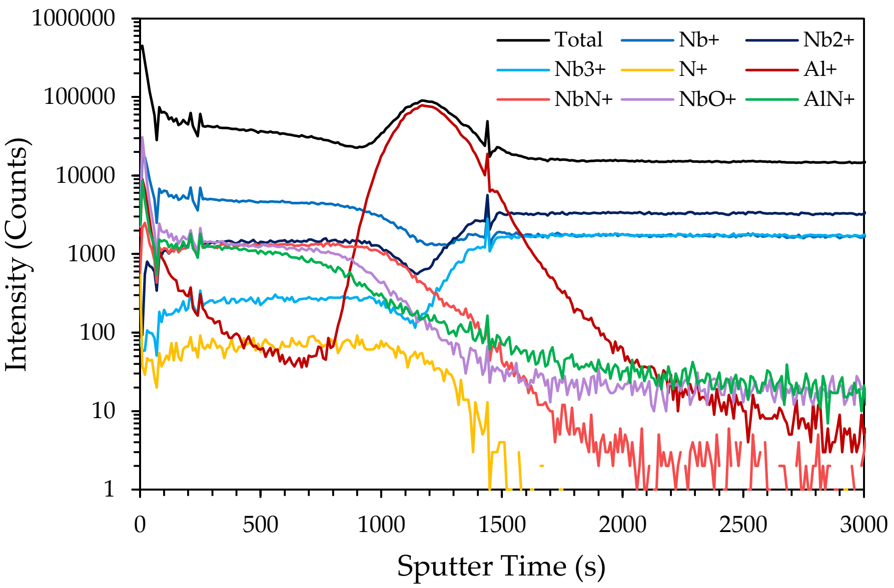




DC MS SIS films

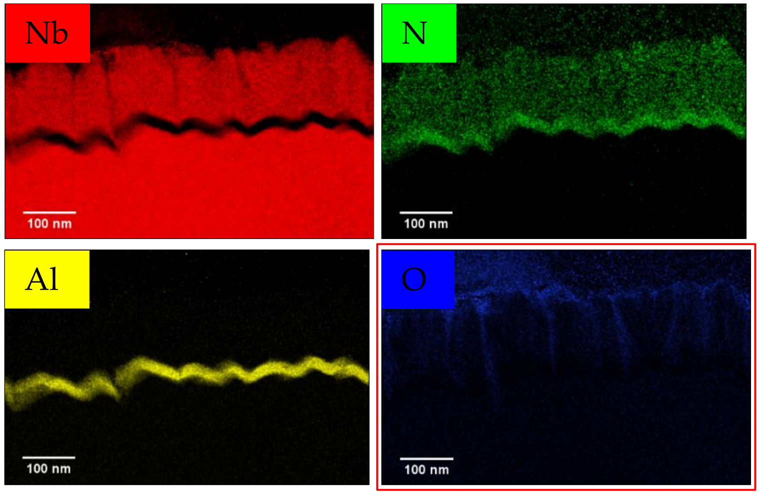


Intergranular oxygen



SIMS Courtesy of Ulrike Koch (Laboratory for Analytical Chemistry, University of Siegen)

DC MS SIS films



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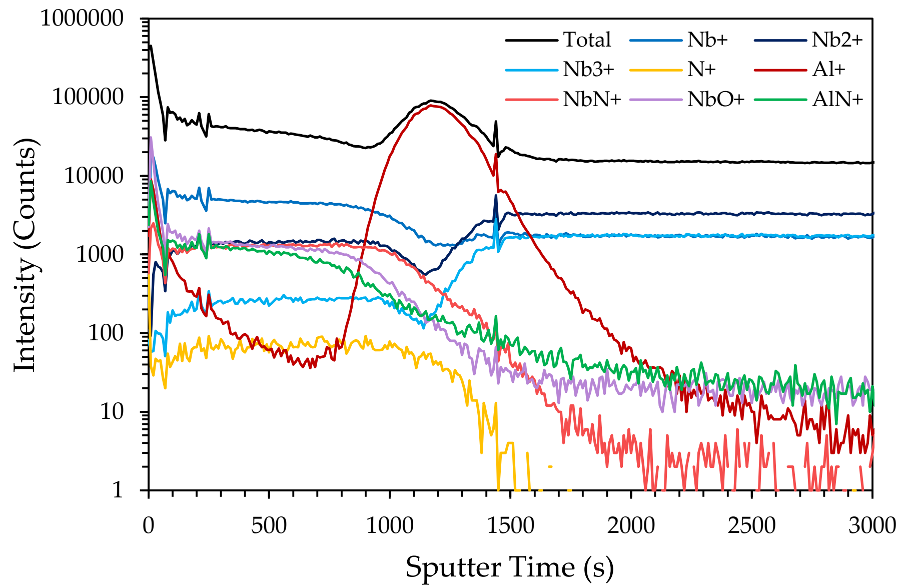

Sample (NbN)	H_{en} (mT)	Nb T_c (K)	NbN T_c (K)
SIS-1 (120)	64.5	9.4	14.7
SIS-2 (118)	14.5	9.2	14.5
SIS-3 (78)	24.0	9.2	14.0
SIS-4 (147)	26.5	9.4	14.9
Nb	52.0	9.4	15.2

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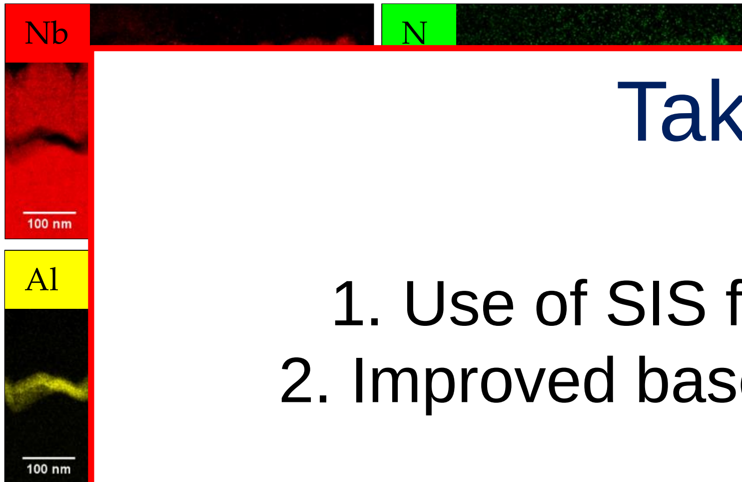
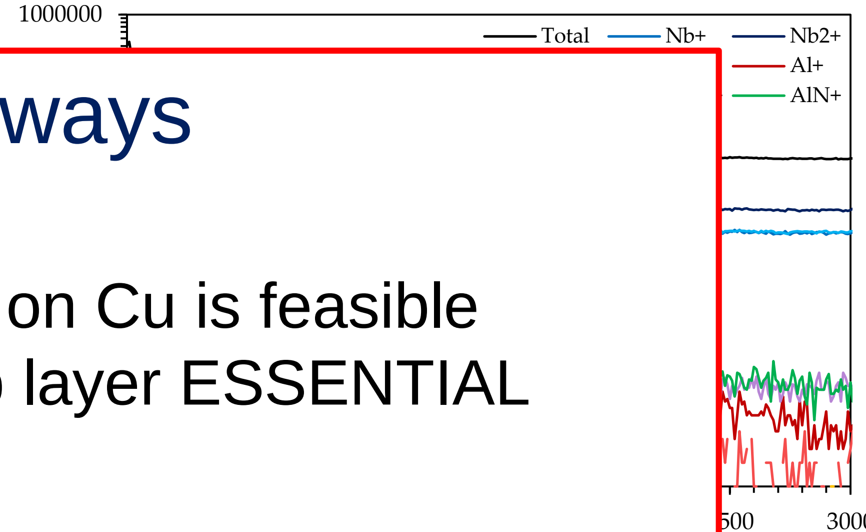
DC MS SIS films





Take aways

1. Use of SIS films on Cu is feasible
2. Improved base Nb layer ESSENTIAL

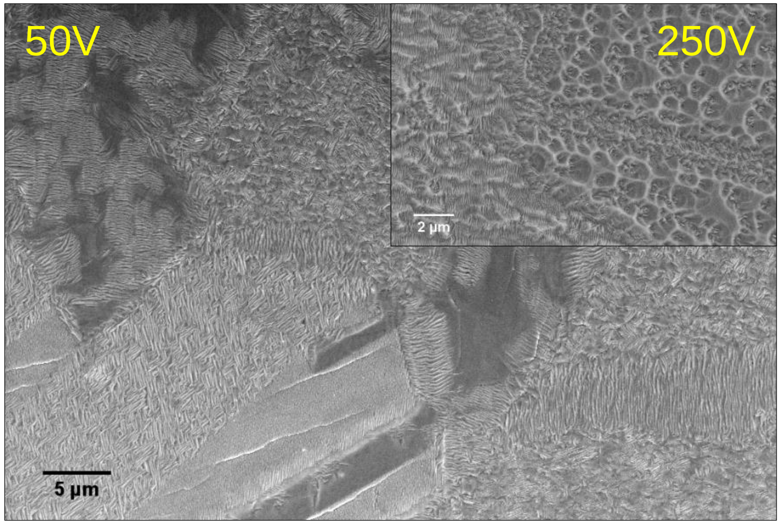
HiPIMS Nb study

Shameless plug: See "Leith *et al, Superconducting NbN thin films for use in superconducting radio frequency cavities, 2021*" for more details

SIMS Courtesy of Ulrike Koch (Laboratory for Analytical Chemistry, University of Siegen)

HiPIMS Nb

- Surface topography similar for all samples.
  - Damage at high substrate bias (> 150 V)



HiPIMS Nb

- Surface topography similar for all samples.
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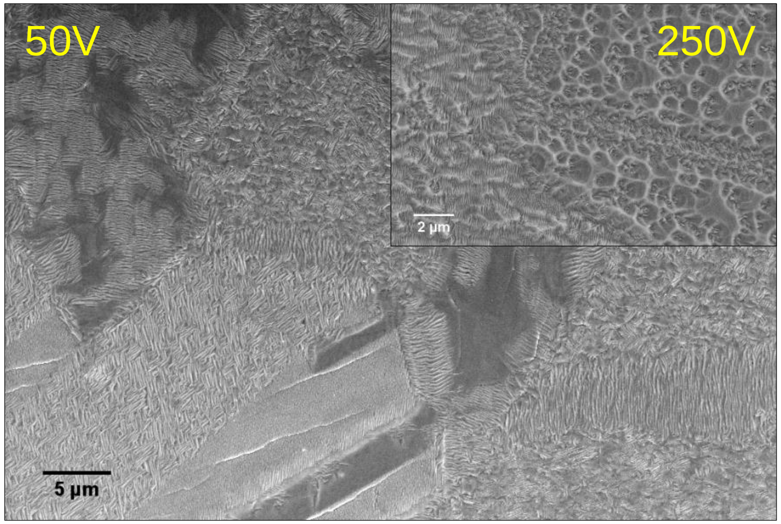
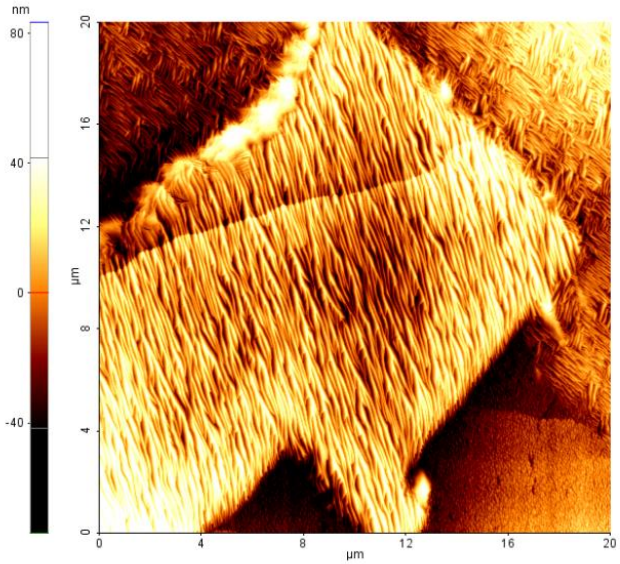
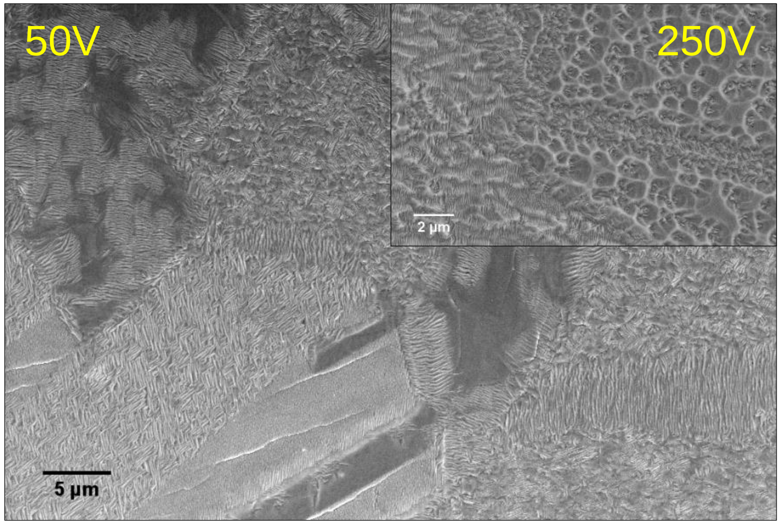
- Reduced average surface roughness:
  - Large GB influence

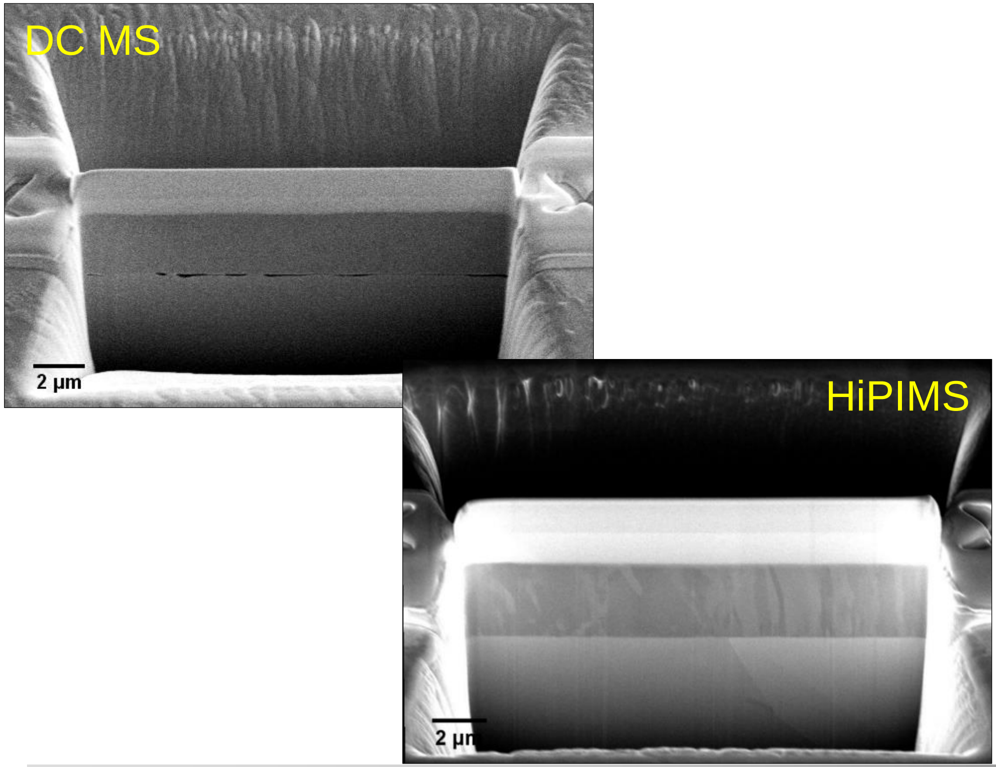


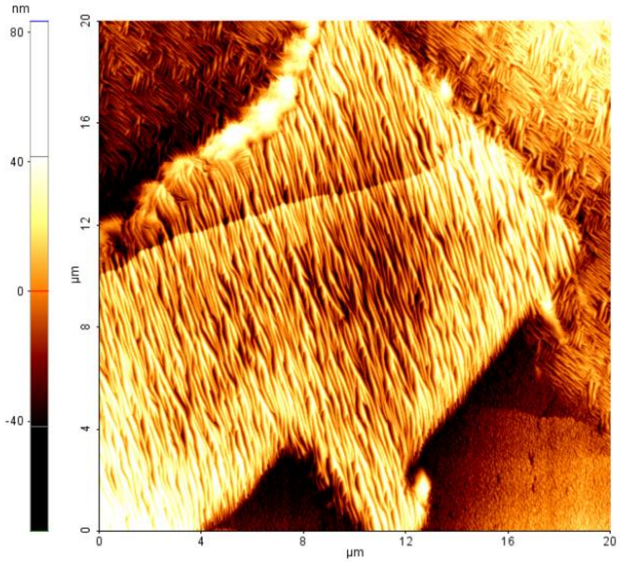


HiPIMS Nb

- Surface topography similar for all samples.
  - Damage at high substrate bias (> 150 V)
- Reduced average surface roughness:
  - Large GB influence
- Interface significantly improved.
  - 85% reduction in interfacial voids



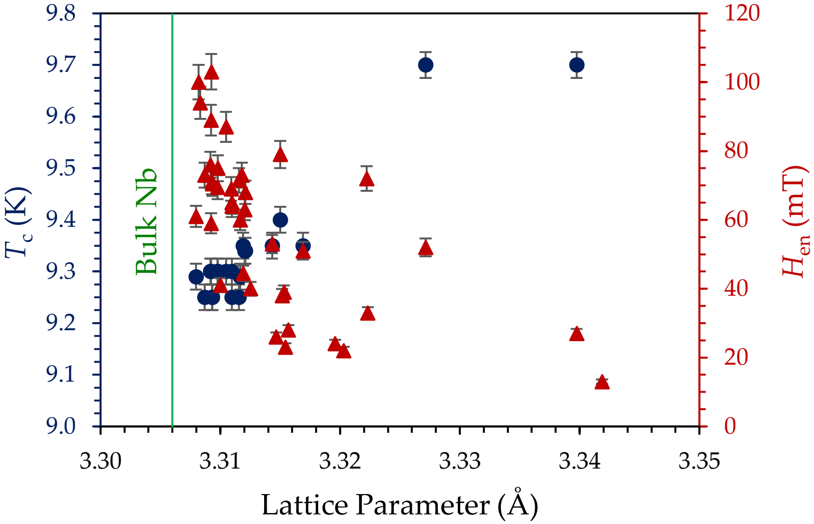




HiPIMS Nb-Conclusions

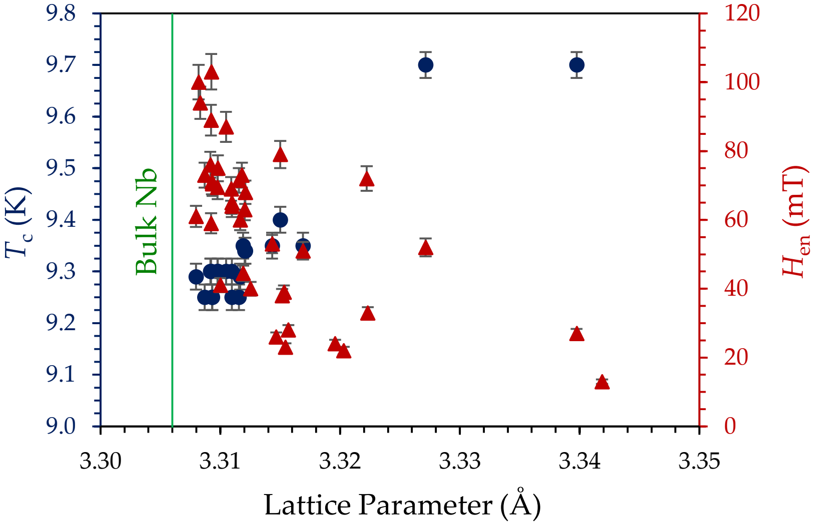
- Bulk-like films perform better
  - Lower stress, bulk-like T_c, improved H_{en}

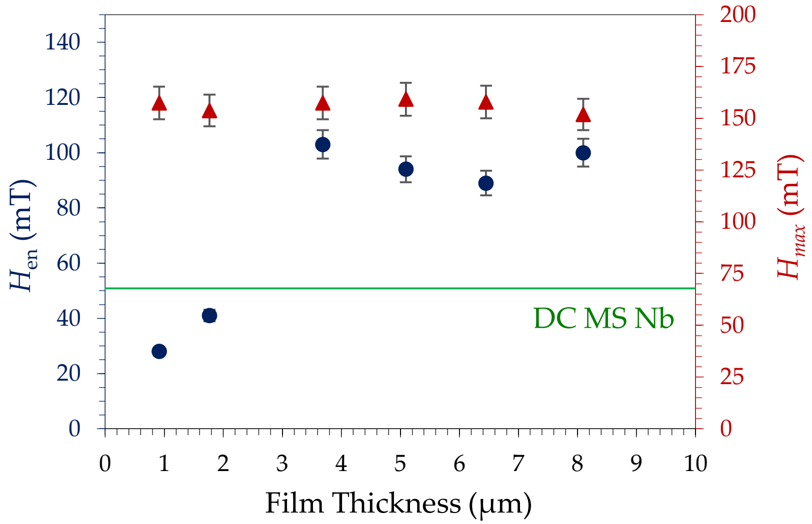
- Duty Cycle displays a relatively small influence
  - Specific maximums still observed

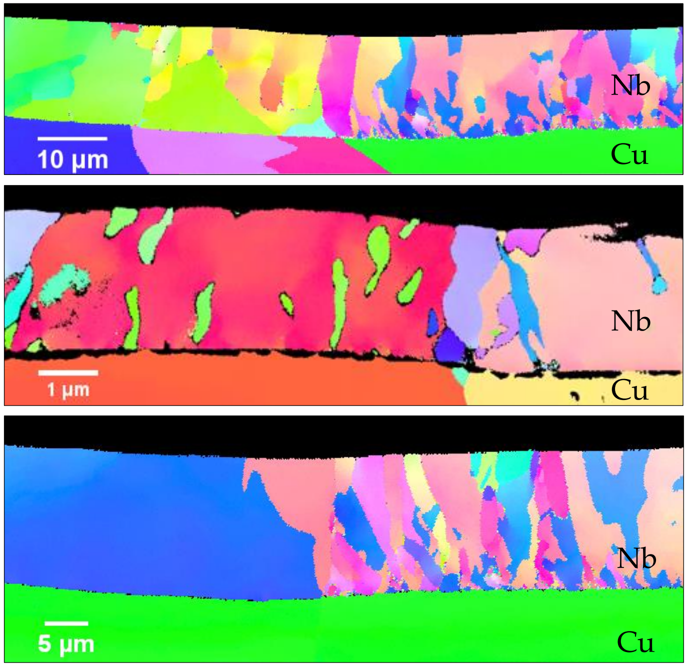


HiPIMS Nb-Conclusions

- Bulk-like films perform better
  - Lower stress, bulk-like T_c, improved H_{en}
- Duty Cycle displays a relatively small influence
  - Specific maximums still observed
- Film Thickness influences
  - “Transition zone”





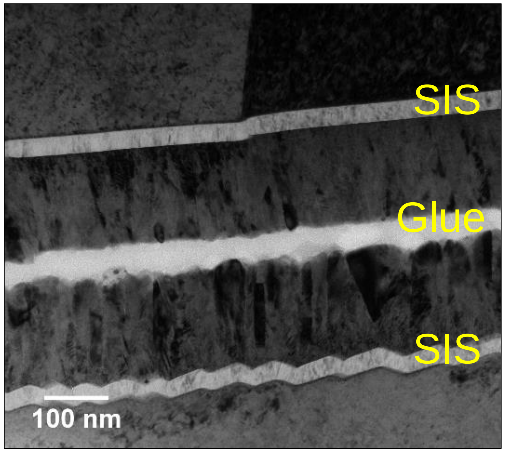


Thank you to G. Rosaz for XRF measurements

HiPIMS Nb + DC MS NbN SIS

- Two SIS film series
  - 4 x Sample 899 recipe (High T_c)
  - 3 x Sample 790 recipe (High H_{en})

- HiPIMS Nb influence on AlN and NbN



HiPIMS Nb + DC MS NbN SIS

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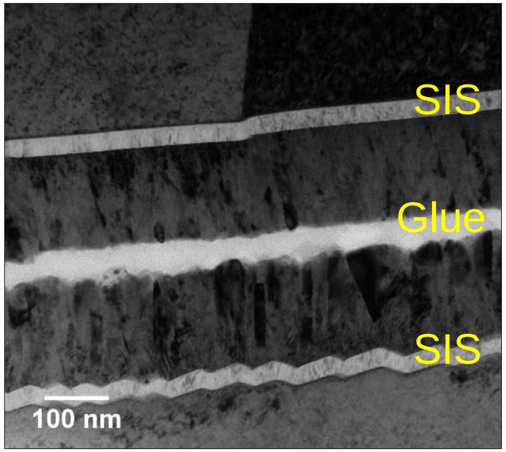
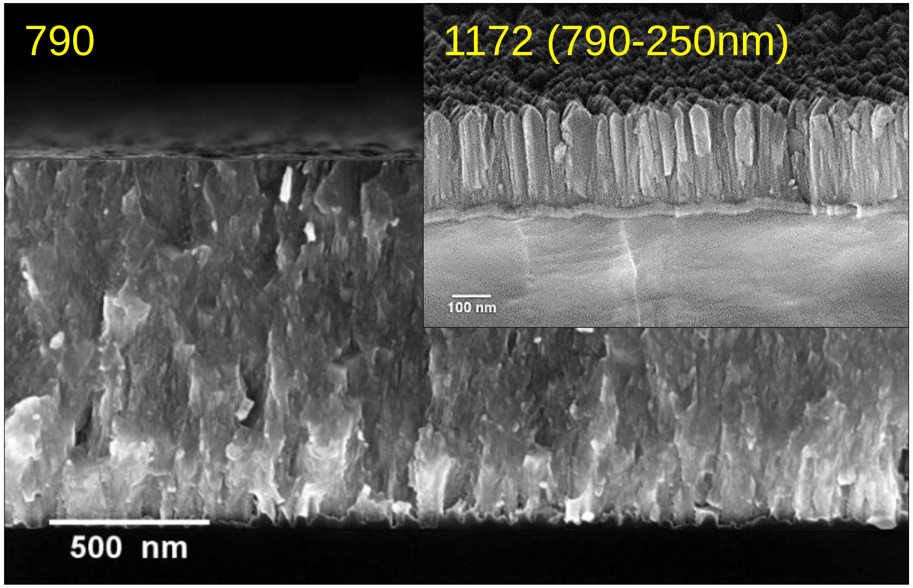
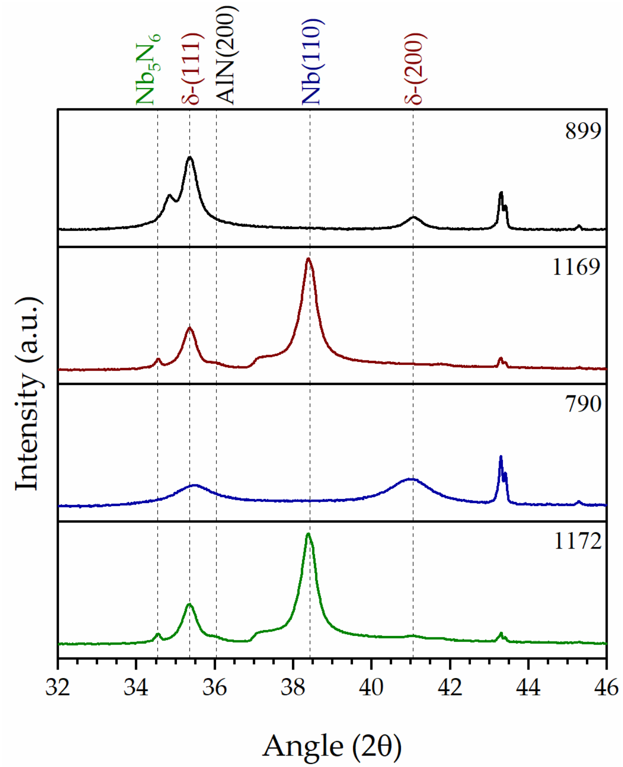
- Microstructure and orientation change due to table rotation!







HiPIMS Nb + DC MS NbN SIS

NbN Superconducting performance reliance

- (899) vs. (790)

Sample	H_{en} (mT)	Nb T_c (K)	NbN T_c (K)
1165 (207nm)	39.0	9.3	15.9
1166 (155nm)	38.0	9.3	15.8
1167 (99nm)	34.0	9.3	15.5
1169 (254nm)	33.0	9.3	16.1
1170 (181nm)	61.0	9.3	15.2
1171 (144nm)	61.5	9.4	15
1172 (246nm)	59.0	9.3	15.3
HiPIMS Nb	103.0	9.35	-

HiPIMS Nb + DC MS NbN SIS

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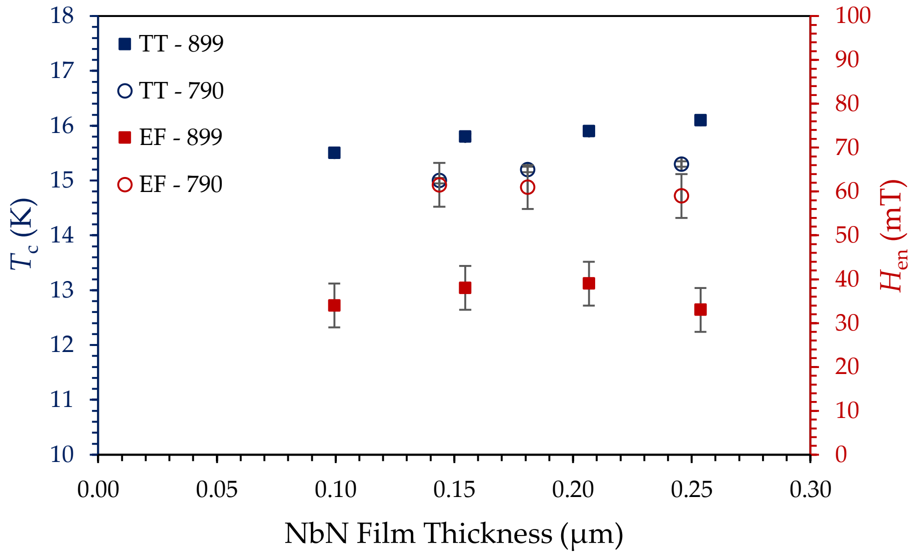
H_{en} NbN film thickness dependence



HiPIMS Nb + DC MS NbN SIS

NbN Su

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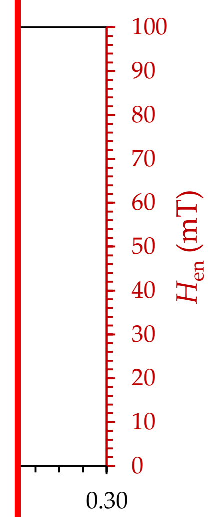
Sample
1165 (2
1166 (1
1167 (9
1169 (2
1170 (1
1171 (1
1172 (2
HiPIMS



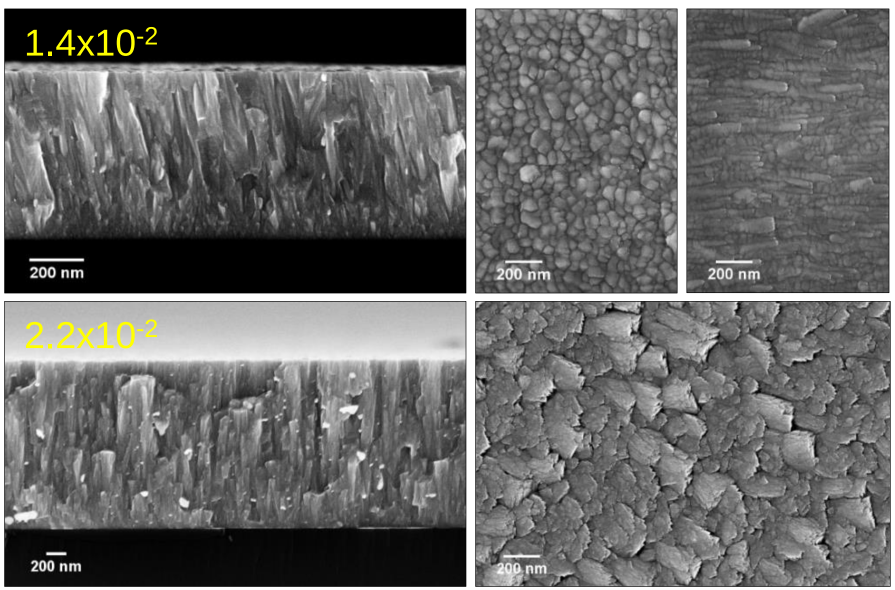
Take aways

1. Definite divide in SIS film performance based on NbN recipe – Pursue higher entry field! HiPIMS NbN Study

2. Complete rotation of table problematic. Implement a "pulse" in front of the target.

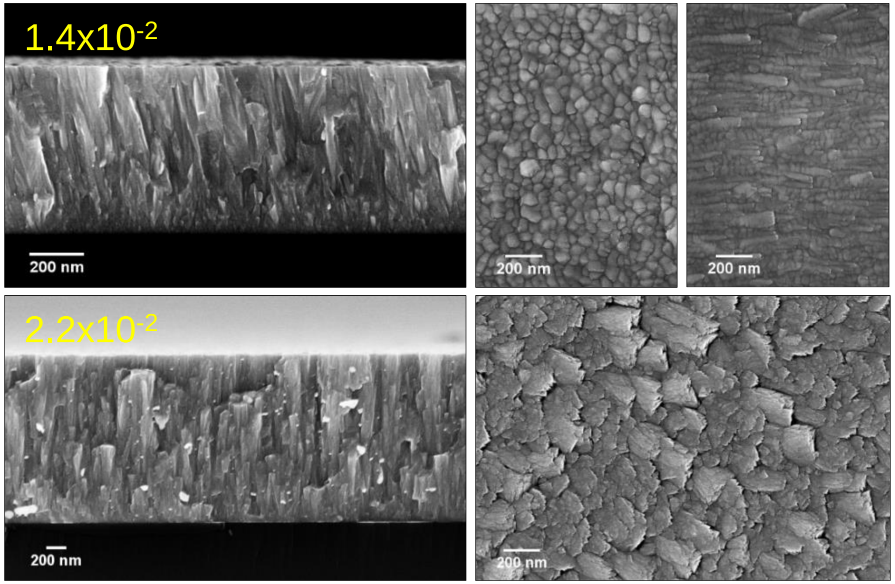
HiPIMS NbN

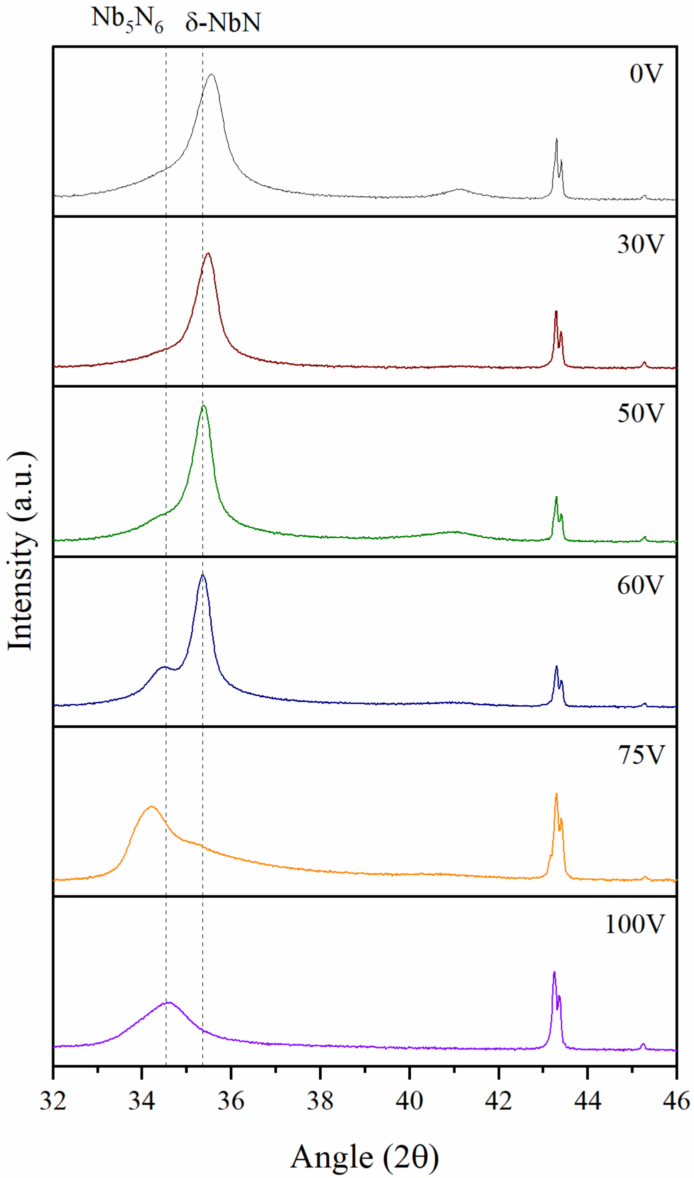
- Similar HiPIMS parameters to Nb
- Similar trends to DC MS NbN
- Improved surface roughness and density



HiPIMS NbN

- Similar HiPIMS parameters to Nb
- Similar trends to DC MS NbN
- Improved surface roughness and density
- High Bias effects
  - Change of phase (> 60 V)
  - Loss of superconductivity (> 75 V)



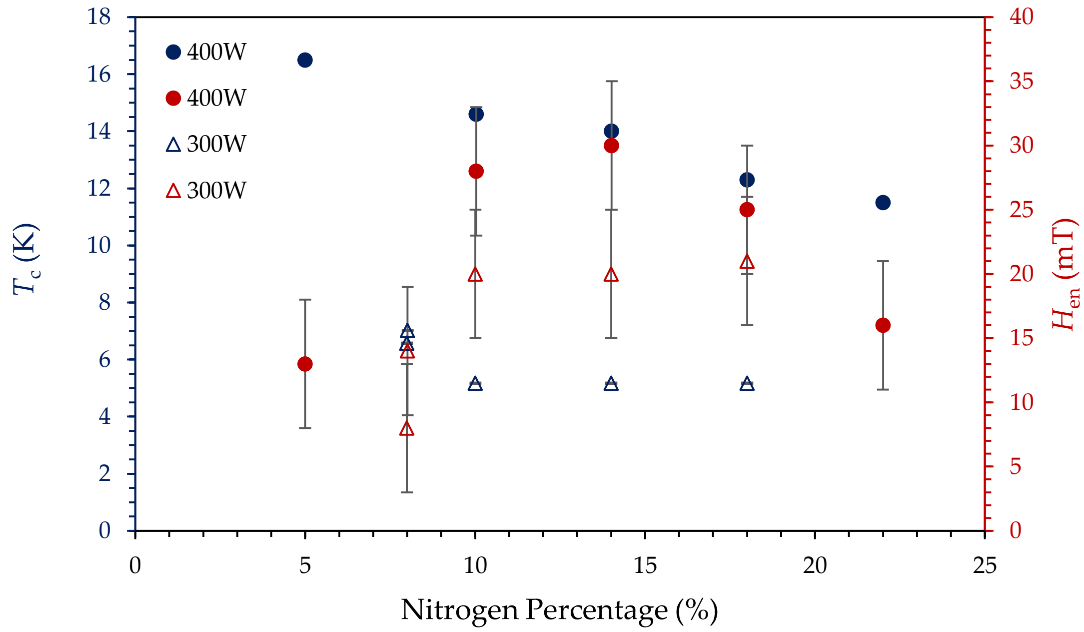


HiPIMS NbN

Significant superconductivity improvements over DC MS

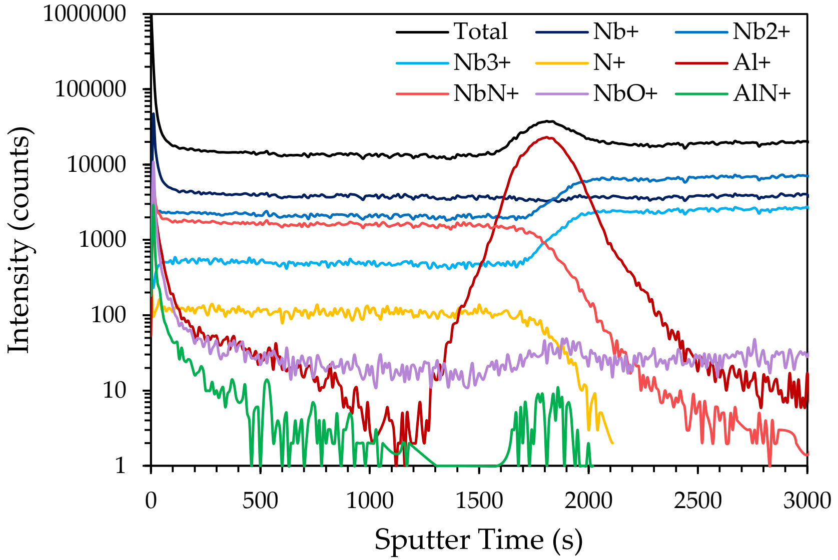
- Highest T_c = 16.5 K
- Highest H_{en} = 30 mT (DC MS = 13 mT)
- Ave H_{en} > DC MS Maximum

Interplay between p_{cath}, P_{dep} and N_2%



HiPIMS SIS Films

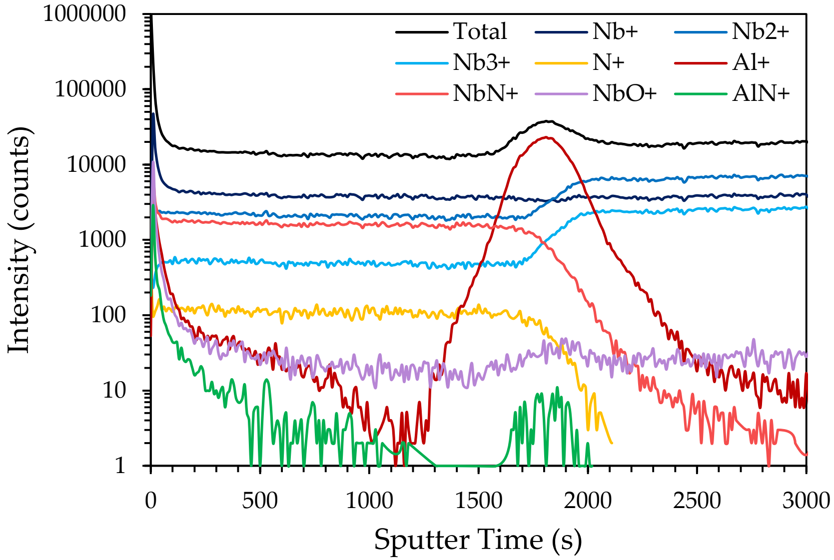
- Multiple HiPIMS SIS series
  - AlN 8 + 30 nm and NbN (100 to 200 nm)
  - Pulsing instituted for AlN + NbN
- Oxygen content reduction

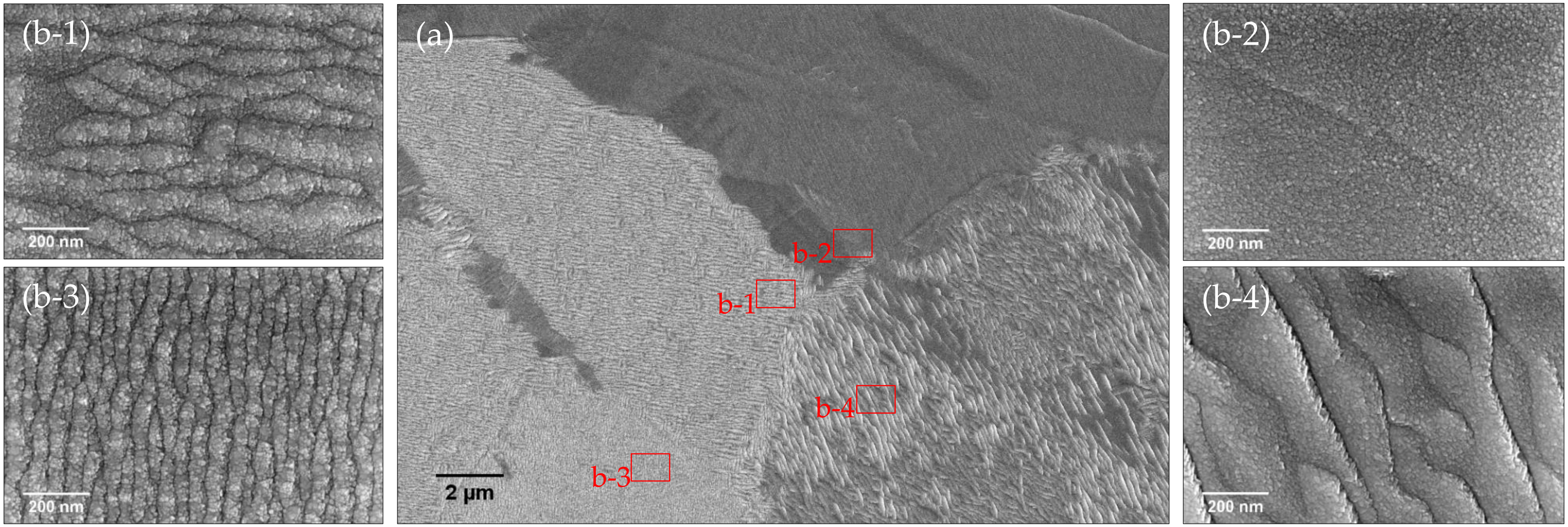


SIMS Courtesy of Ulrike Koch (Laboratory for Analytical Chemistry, University of Siegen)

HiPIMS SIS Films

- Multiple HiPIMS SIS series
  - AlN 8 + 30 nm and NbN (100 to 200 nm)
  - Pulsing instituted for AlN + NbN
- Oxygen content reduction
- SIS film morphology dominated by HiPIMS Nb
  - NbN "superimposed" on top of Nb + AlN





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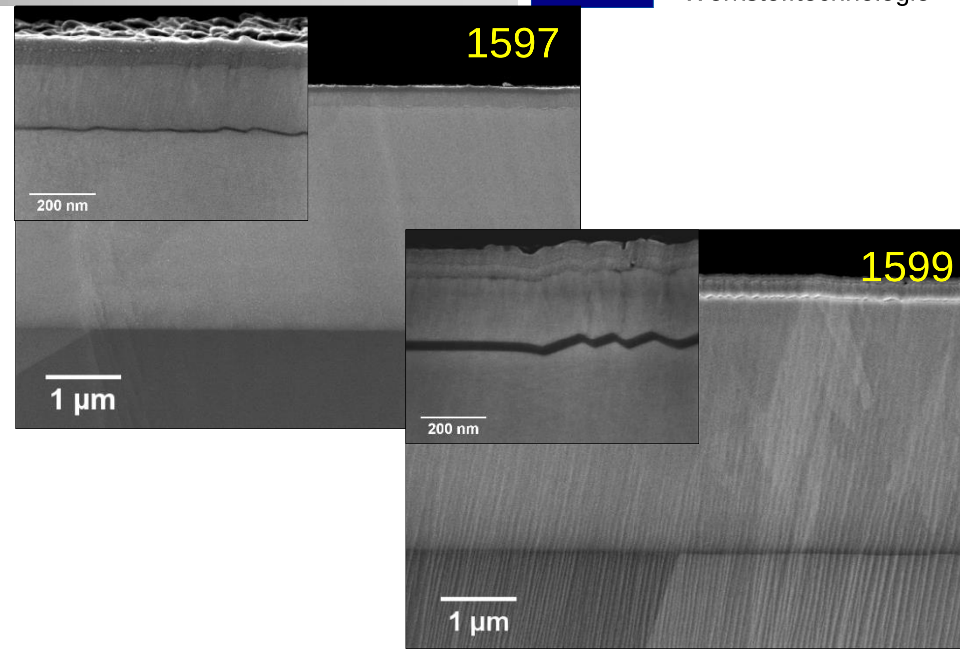
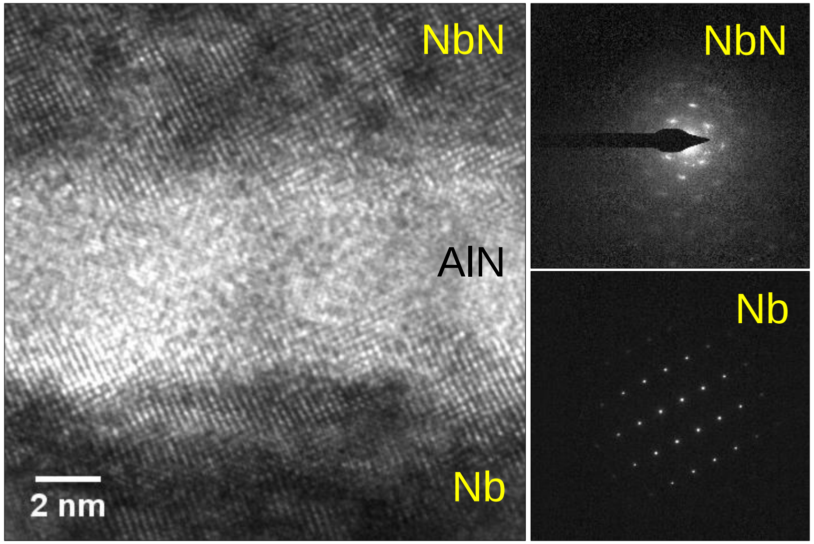
HiPIMS SIS Films

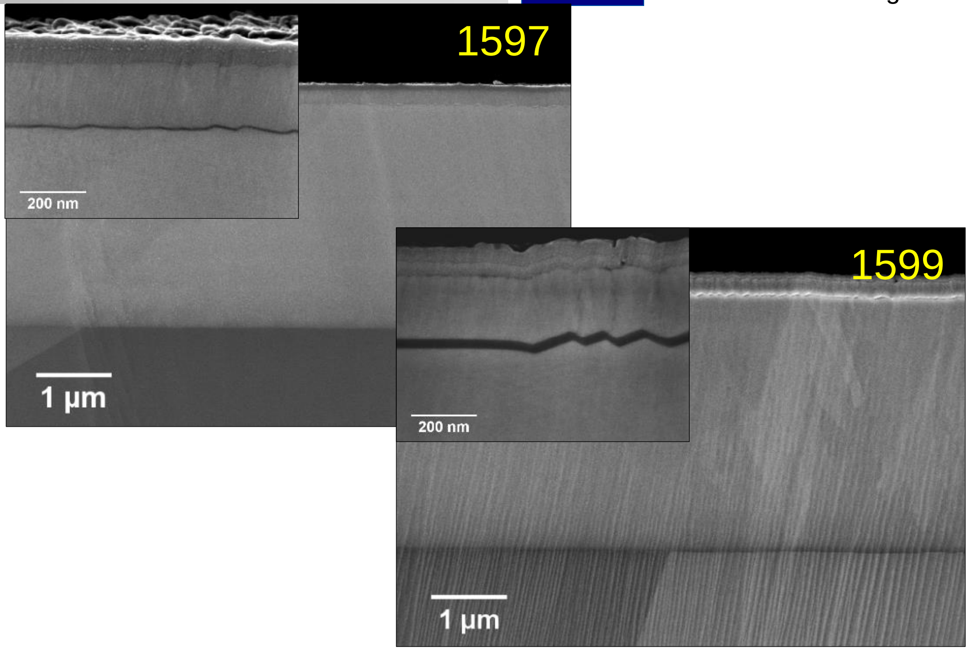
- Improved surface roughness and density



HiPIMS SIS Films

- Improved surface roughness and density
- Coherent epitaxial growth of SIS layers





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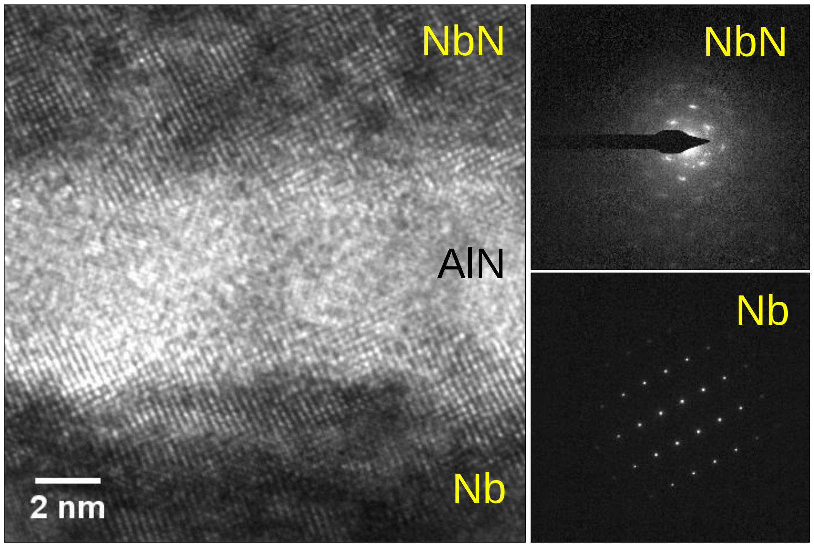
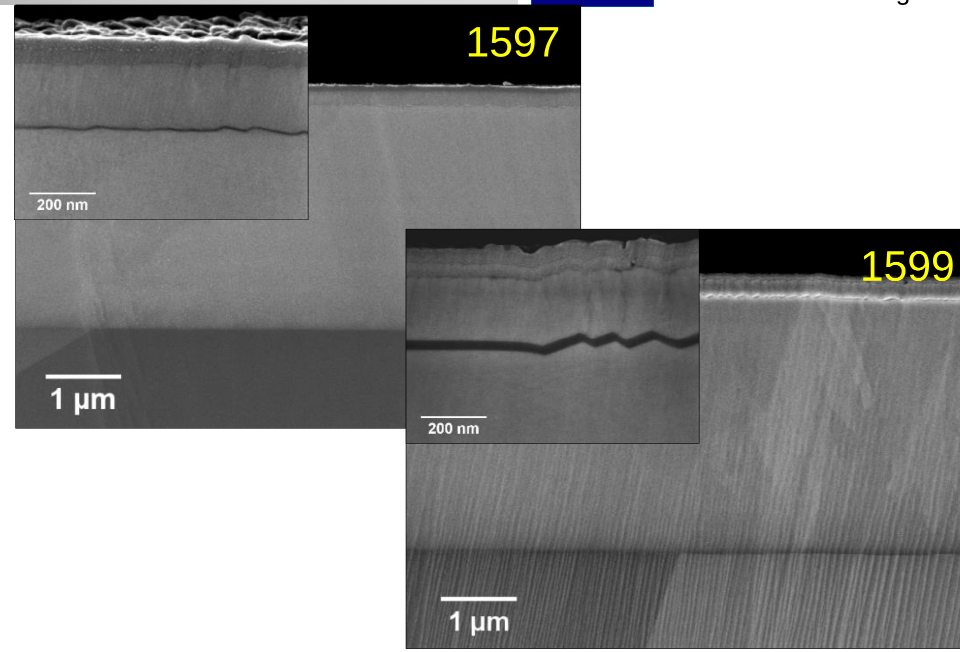
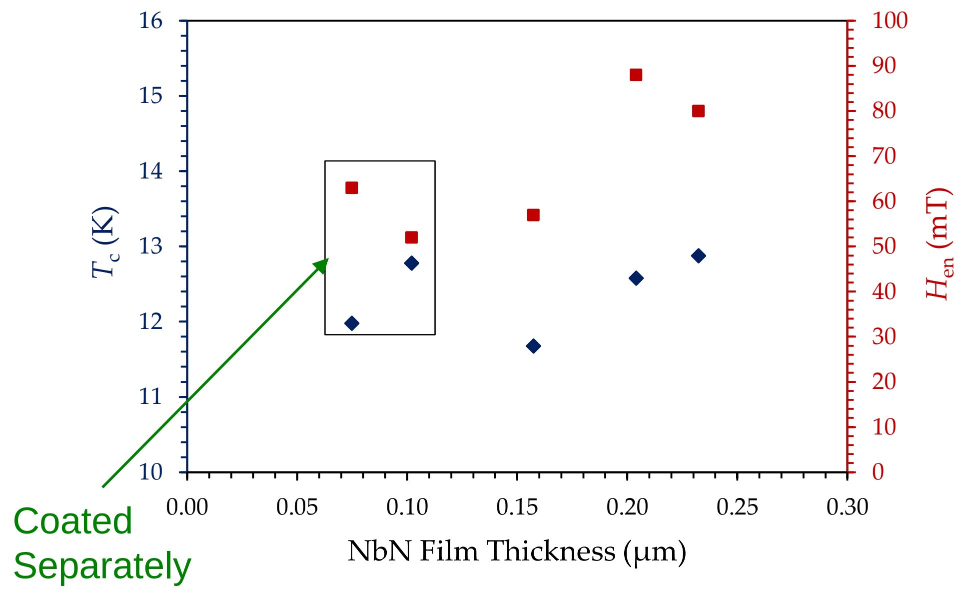


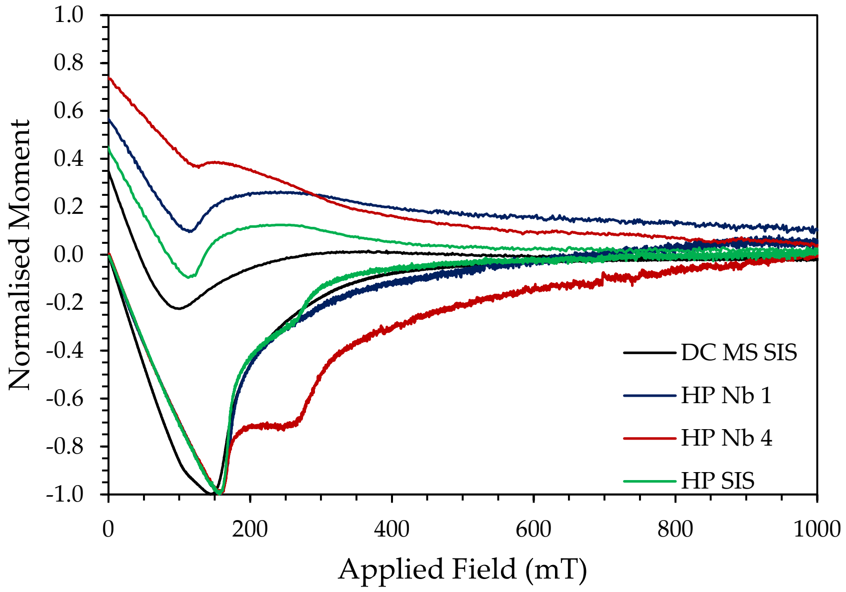


- Decreased T_c in SIS vs original
- H_{en} thickness dependence on recipe
  - Cleaner films (HiPIMS) = Thicker layer
- Increased H_{en} for HiPIMS SIS films
  - (88 mT vs. 64.5 mT)

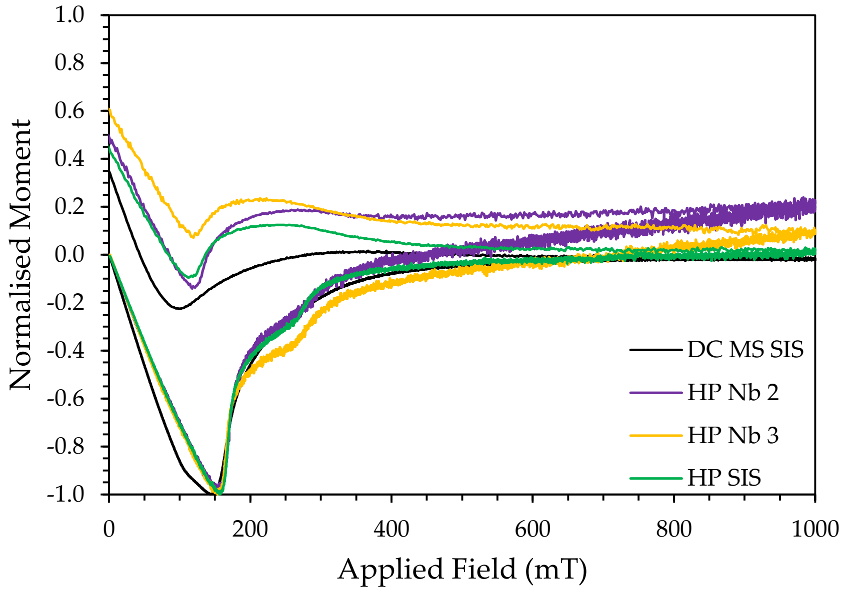


SIS film comparison

- Significant improvements offered by HiPIMS – Optimisation possible
- Best DC MS SIS film displays smallest magnetisation loop
- HiPIMS NbN shows more pronounced “dip” but earlier separation.
- Highest H_{en} samples vs. Lowest H_{tr} Measurement in oscillating field required
- Final HiPIMS SIS samples still to be tested



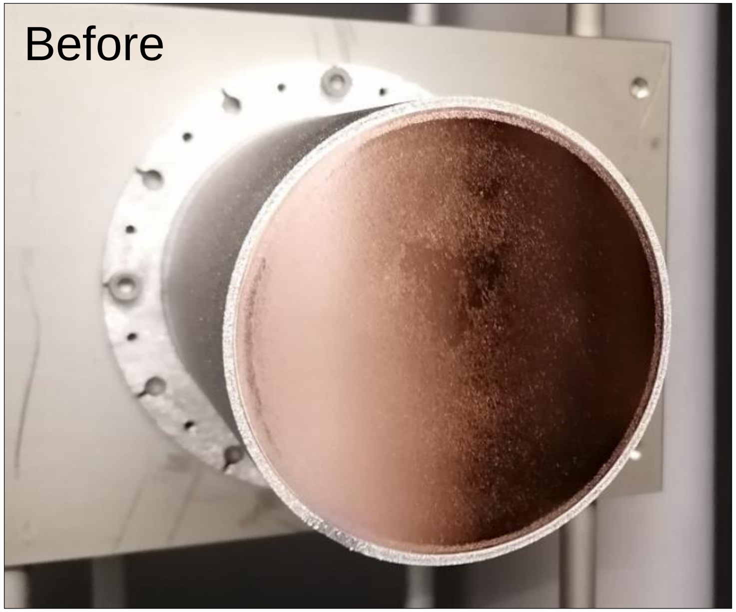
Best performing H_{en}

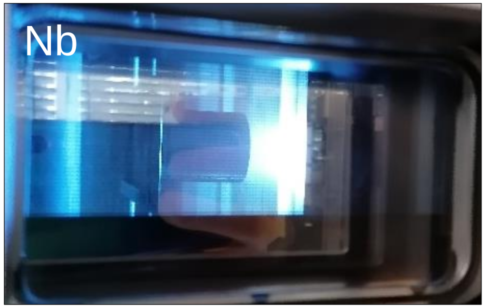


Best performing H_{tr}

*Measured in parallel field at 4.22 K

QPR SIS film Coating





QPR test results detailed by D. Tikhonov – 18/03

Thanks for your Attention!

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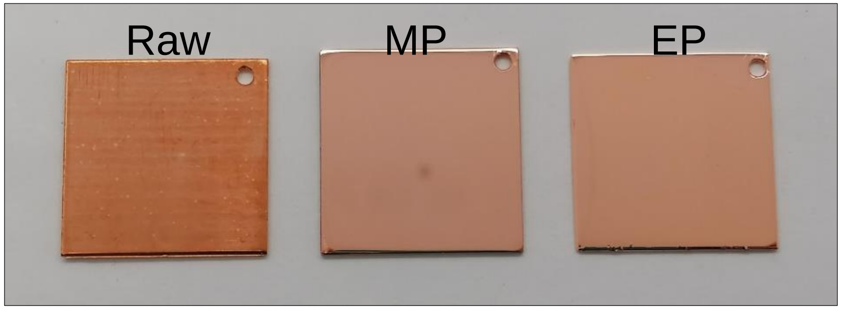
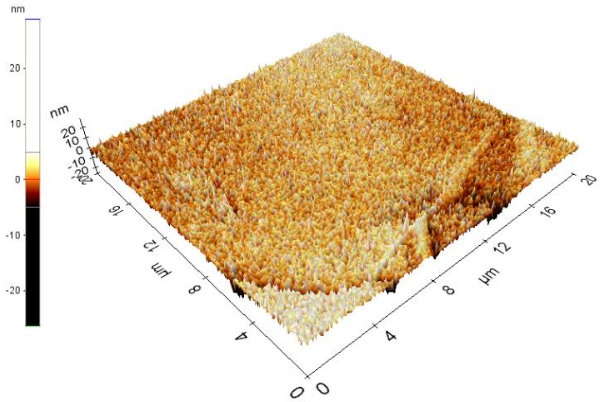
Authors would also like to acknowledge the support provided by European Union's ARIES collaboration H2020 Research and Innovation Programme under Grant Agreement no. 730871.

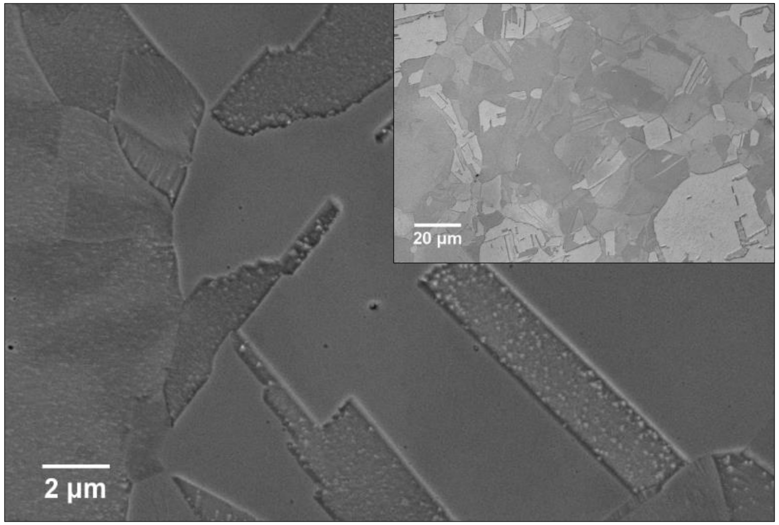
Extra Slides

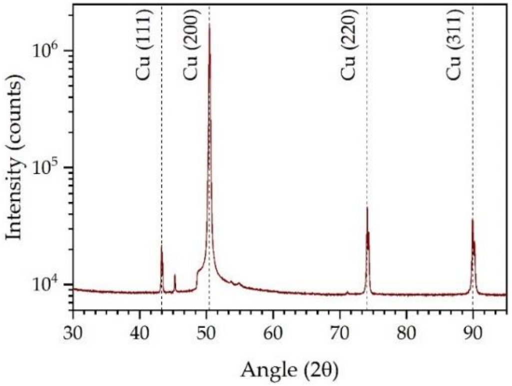
Cu sample preparation

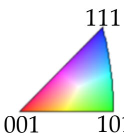
- OFHC Cu substrates (25 x 25 mm)
- MP + EP (1 hour)
  - Large step edge GB (Max 20 nm)
- S_q= 2.58 ± 0.26 nm
- Polycrystalline, (200) preferred orientation

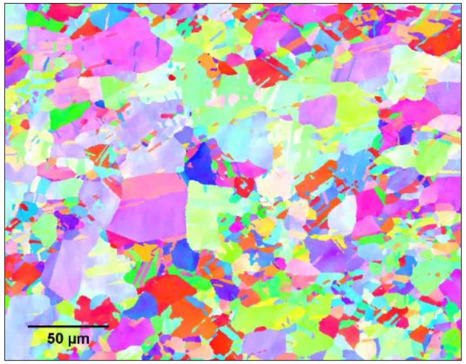










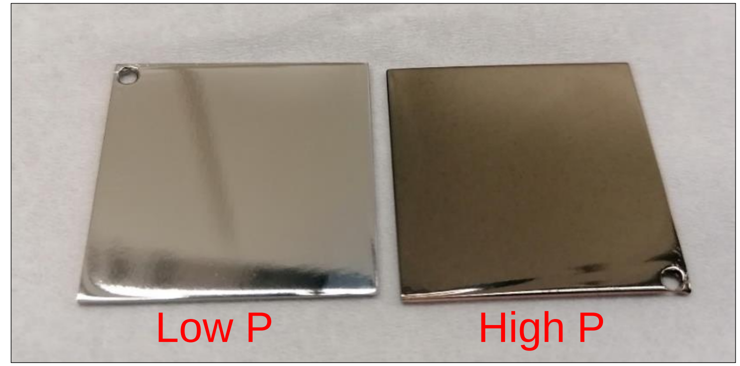


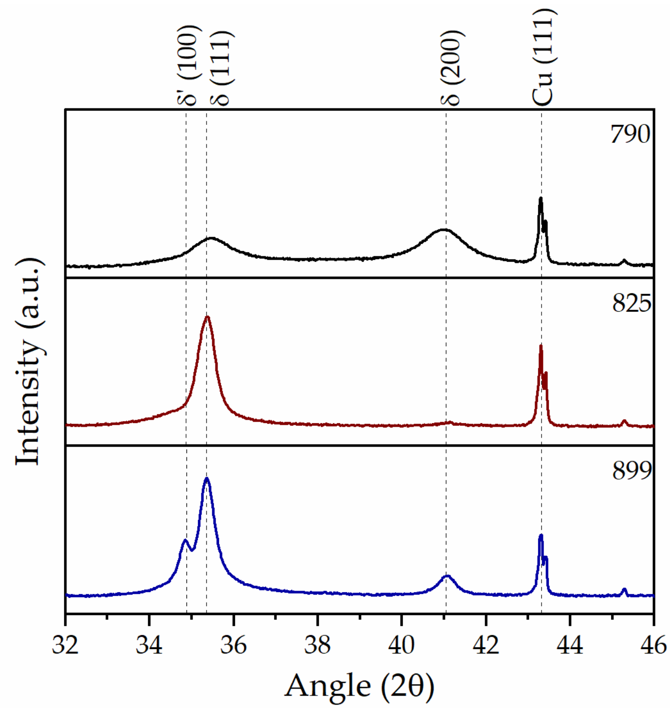
DC MS NbN optimisation

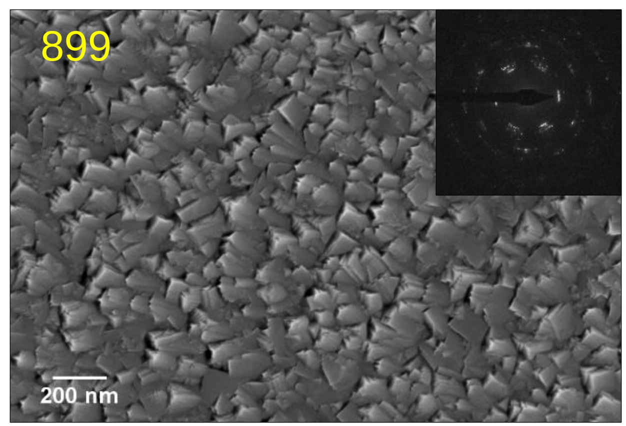
Optimised Coating:

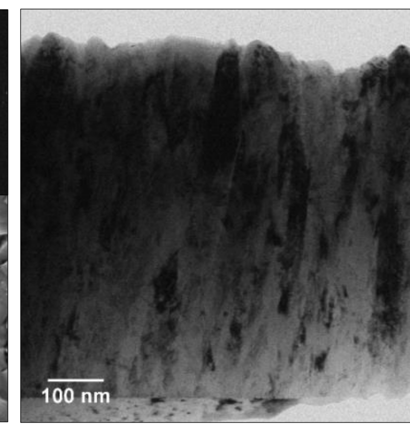
- 8% N_2
- Intermediate Pressure (1.4E-2 mbar)
- Substrate Bias (75 V)
- Increased Cathode Power

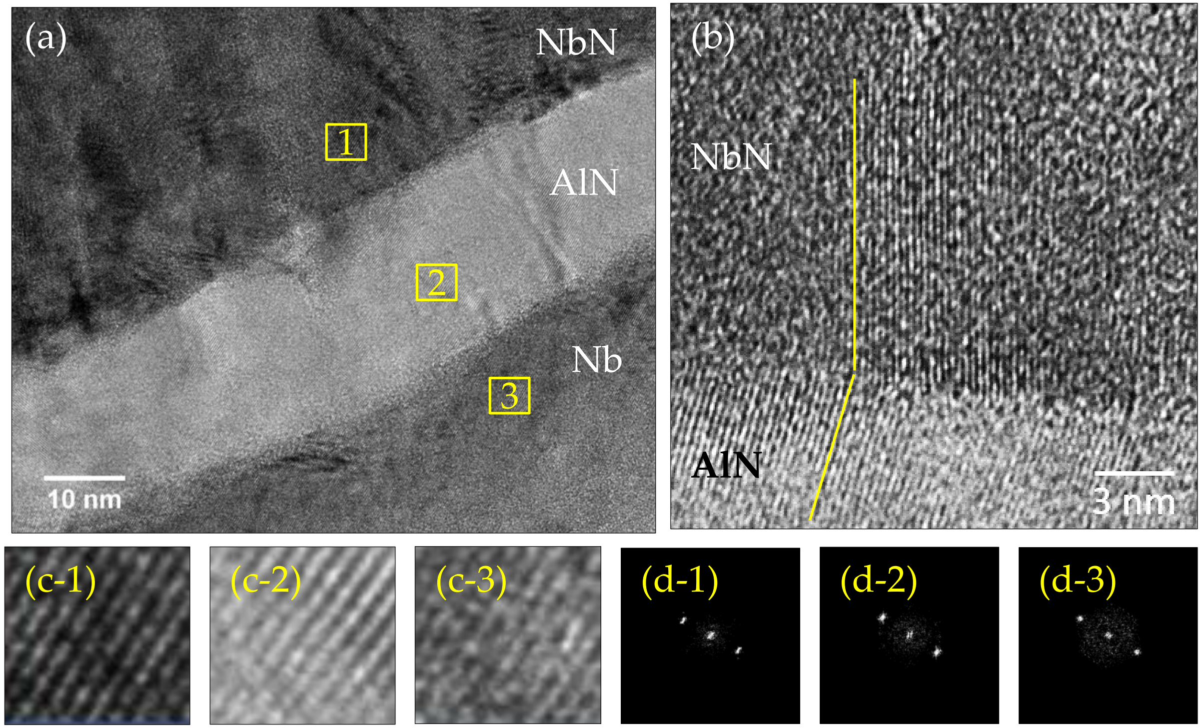
- T_c = 16.1 K, H_{en} = 5 mT
- S_q = 6.36 ± 0.42 nm
- δ-NbN(111) with small δ'-NbN

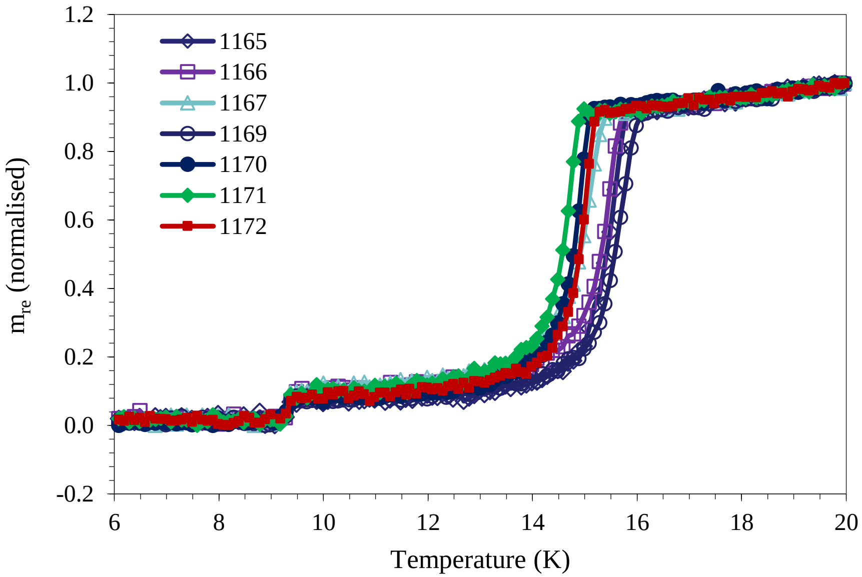









(a)
NbN
1
AlN
2
Nb
3
10 nm
(b)
NbN
AlN
3 nm
(c-1)
(c-2)
(c-3)
(d-1)
(d-2)
(d-3)

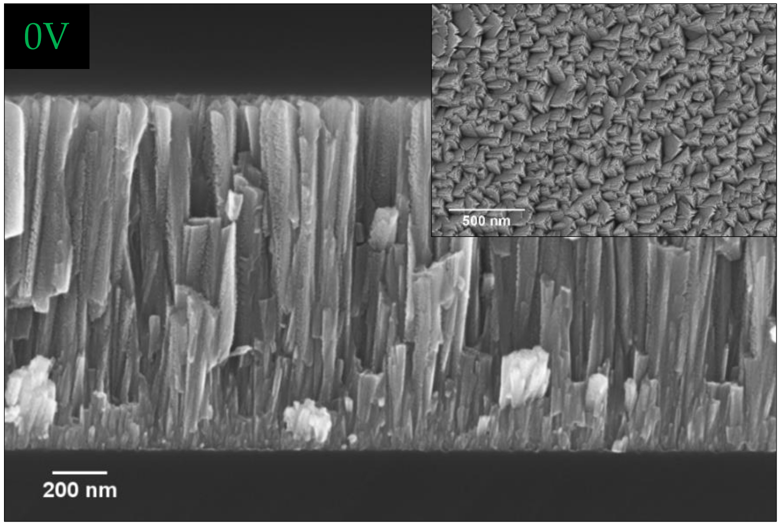

1165
1166
1167
1169
1170
1171
1172
m_{re} (normalised)
Temperature (K)
1.2
1.0
0.8
0.6
0.4
0.2
0.0
-0.2
6
8
10
12
14
16
18
20

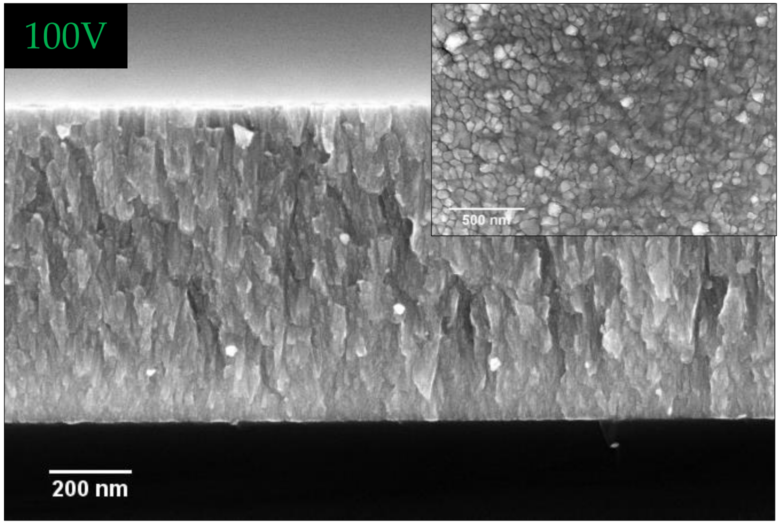
HiPIMS Nb Deposition Parameters

Parameter	Value
Cathode Power	300 to 600 W
Deposition Pressure	$8x10^{-3}$ - $1.8x10^{-2}$ mbar
HiPIMS Pulse Length	80 to 200 µs
HiPIMS Pulse Frequency	800 to 2000 Hz
Duty Cycle (Calculated)	2 to 20 %
Substrate Bias	0 to - 250 V
Substrate Temperature	115 to 290 °C
Film Thickness.	0.91 to 8.10 µm

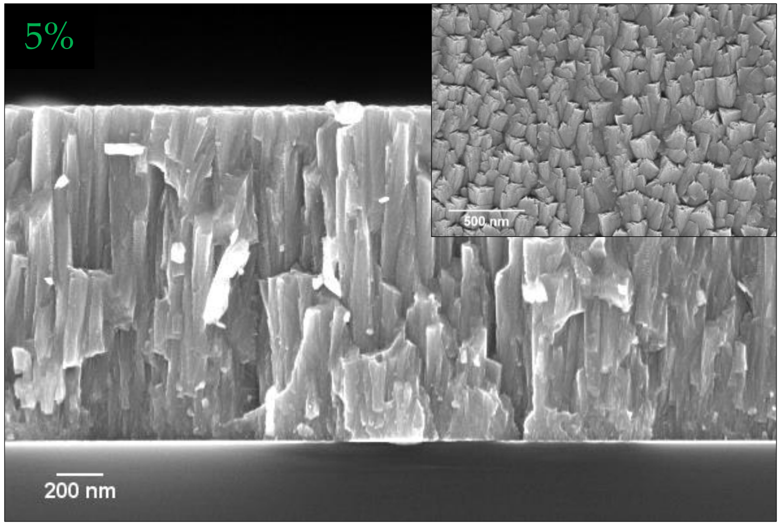
HiPIMS NbN

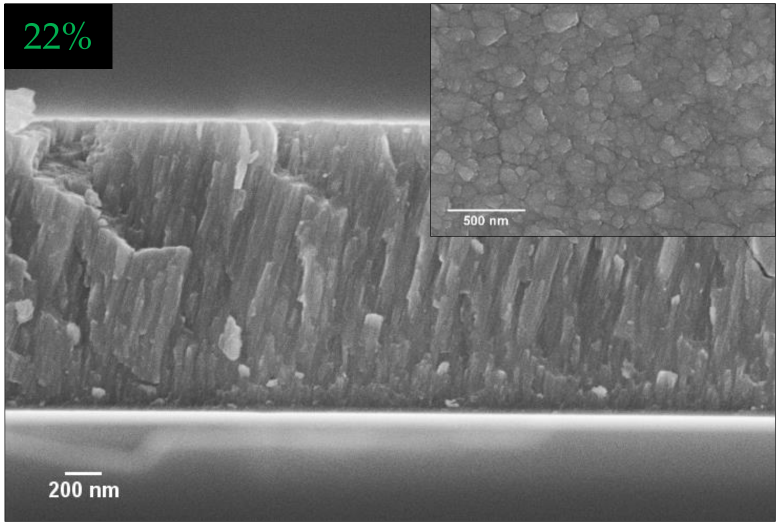
Substrate Bias



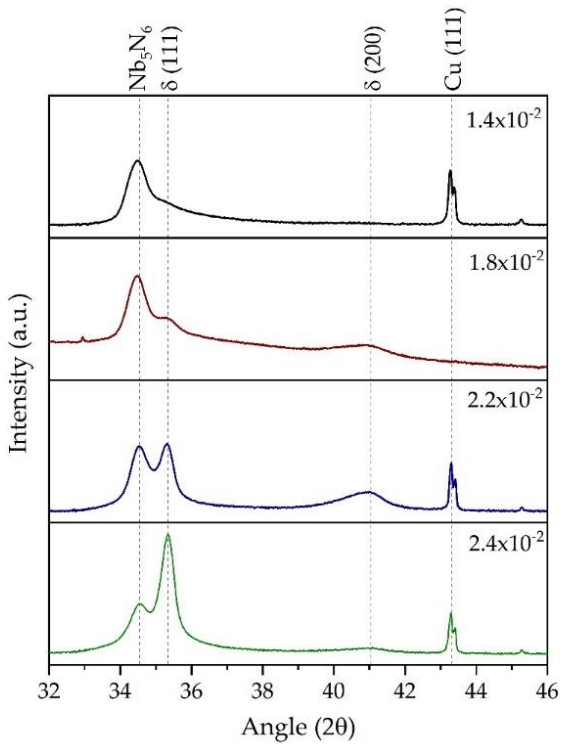


Nitrogen Percentage

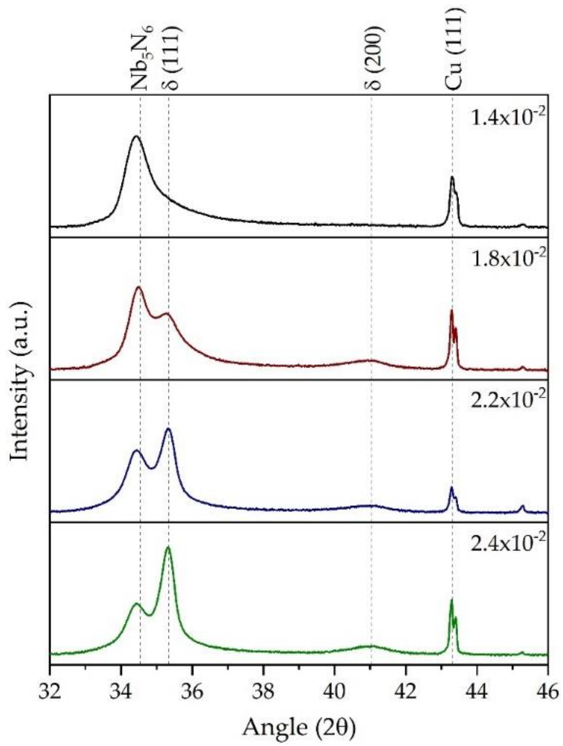




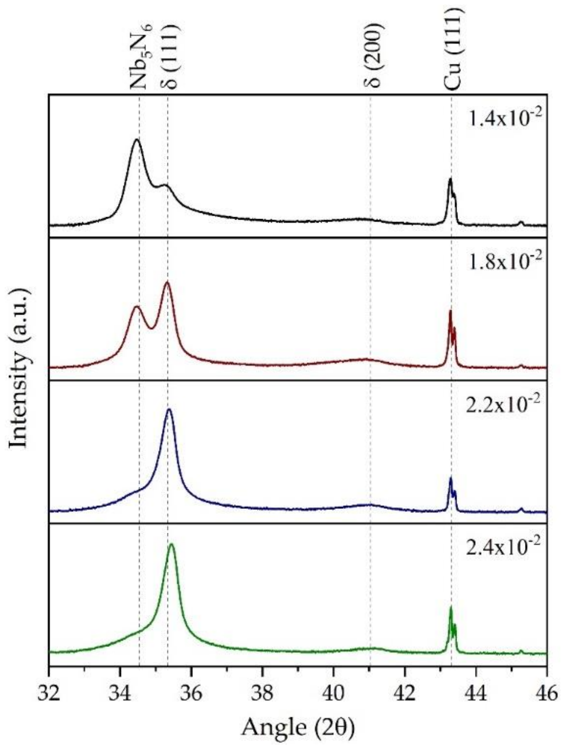
HiPIMS NbN



400W 8% N_2

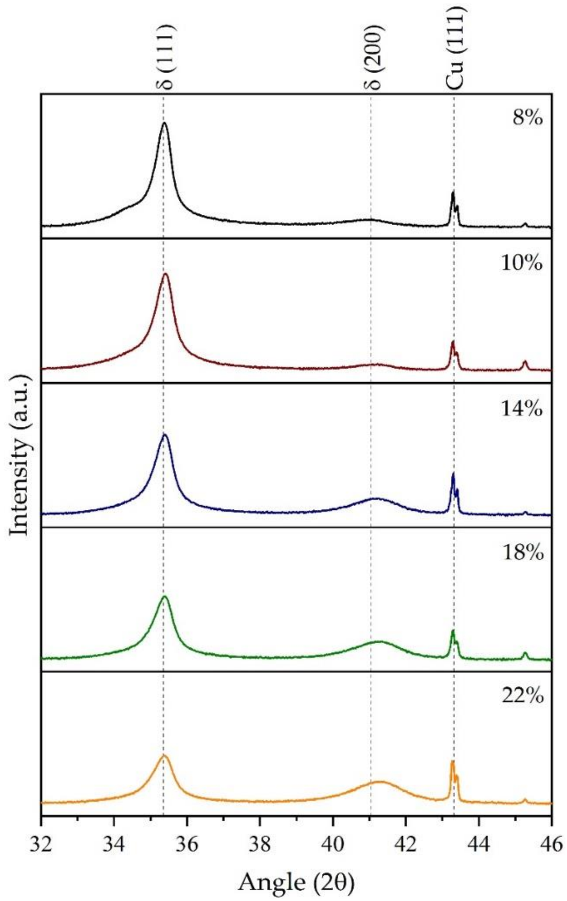


400W 10% N_2

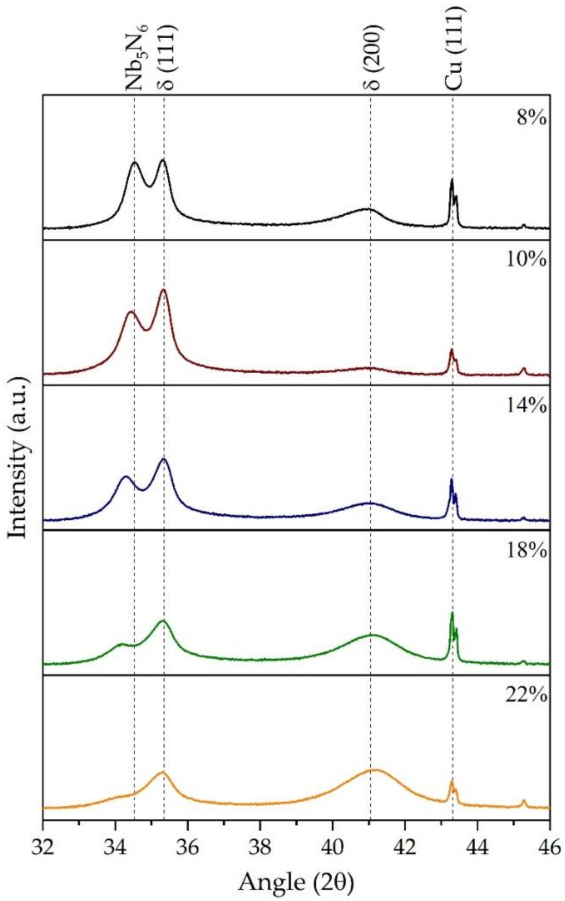


300W 8% N_2

HiPIMS NbN



300W

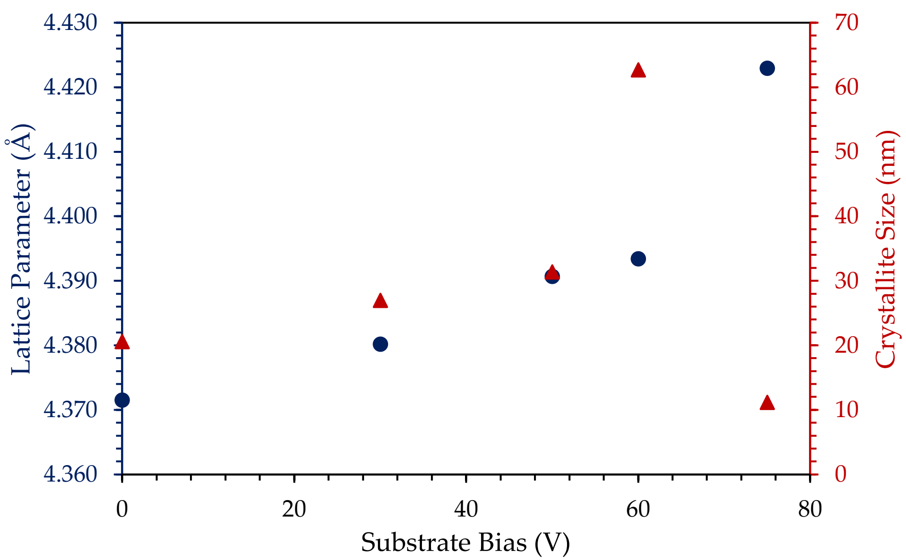


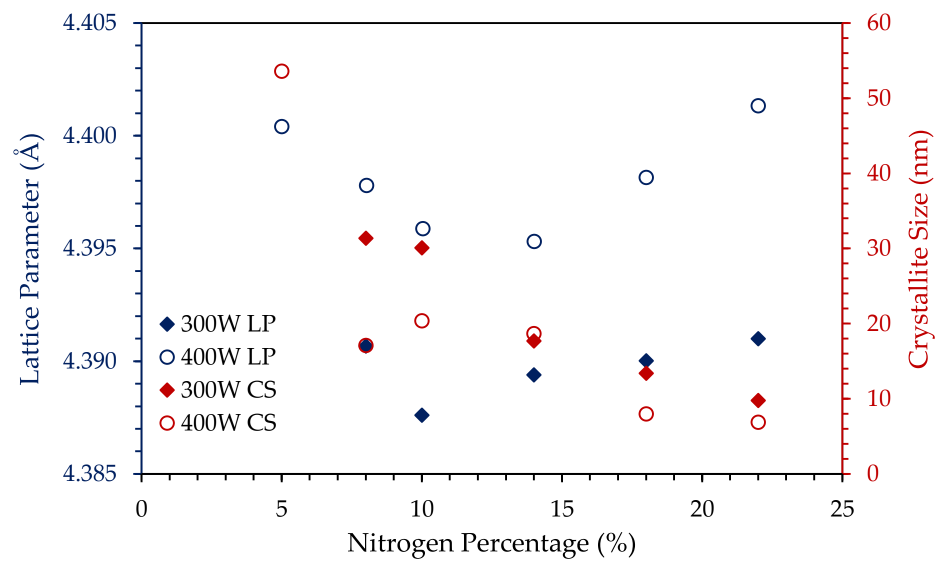
400W

HiPIMS NbN

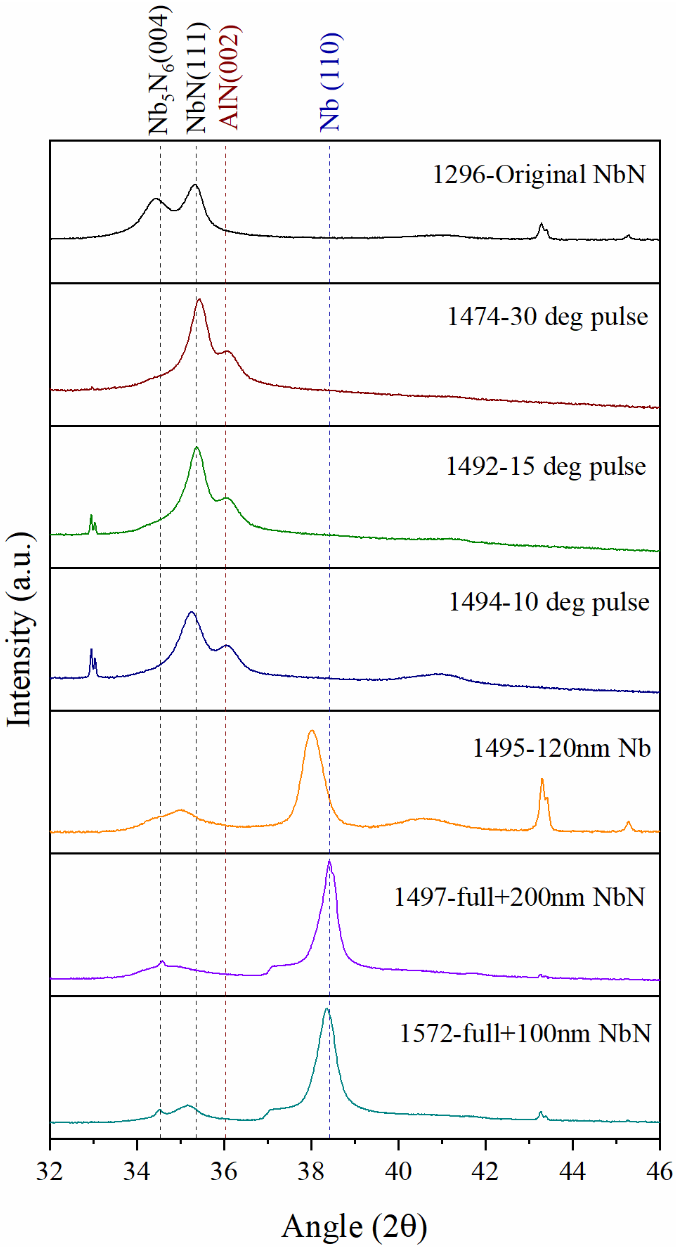
- Lattice Parameter dependent on specific deposition parameters. Increases with:
  - Increasing substrate bias
  - Decreasing Pressure
  - Increasing Cathode Power
- Reaches a specific minimum for N_2% - related to phase formation

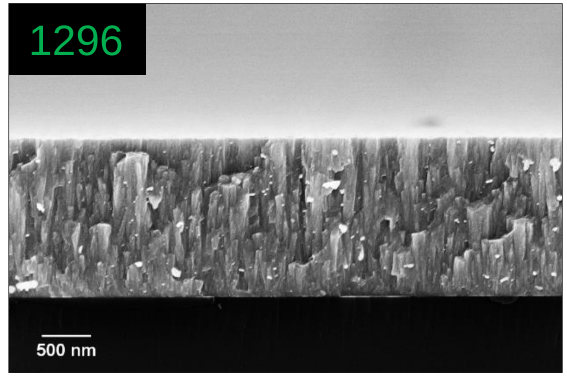
- Crystallite size increases with:
  - Decreasing cathode power
  - Decreasing N_2%
  - Increasing substrate bias. Except at high values due to phase change.
  - Increasing deposition pressure

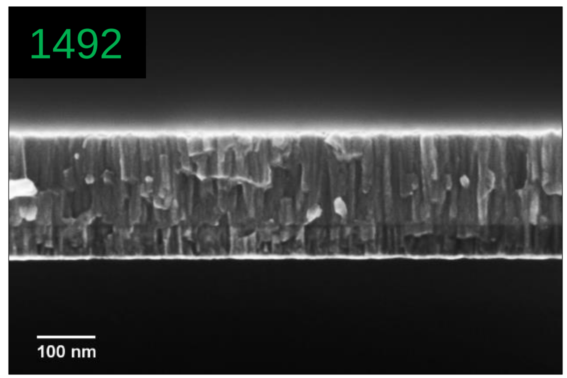


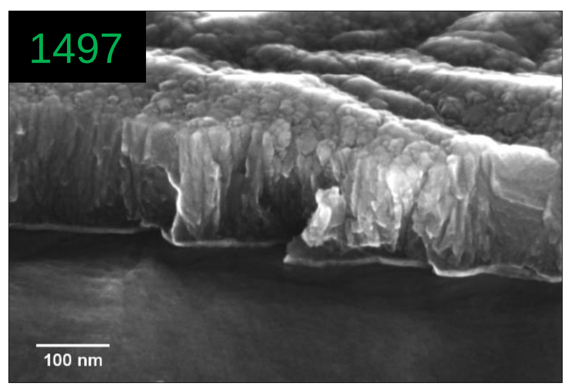


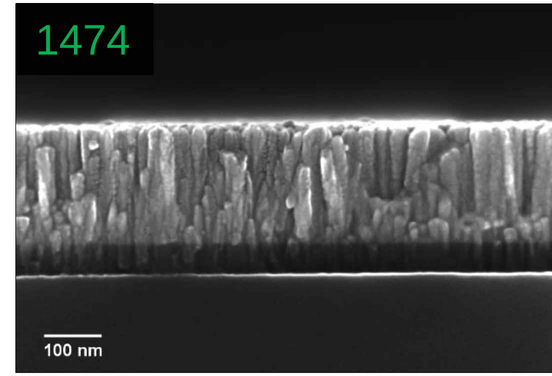
Preparatory HiPIMS SIS Coatings

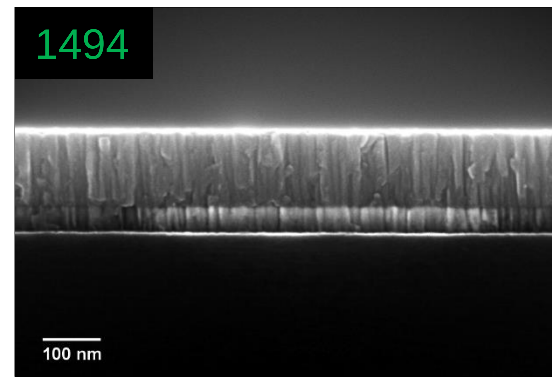


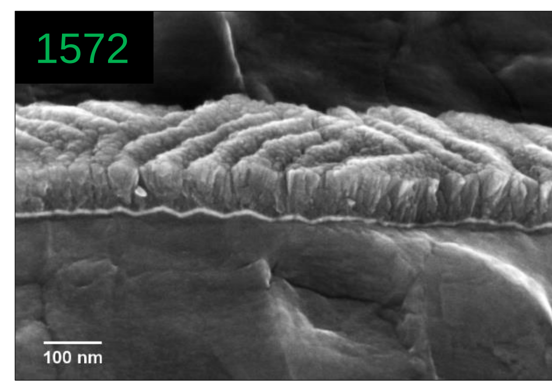




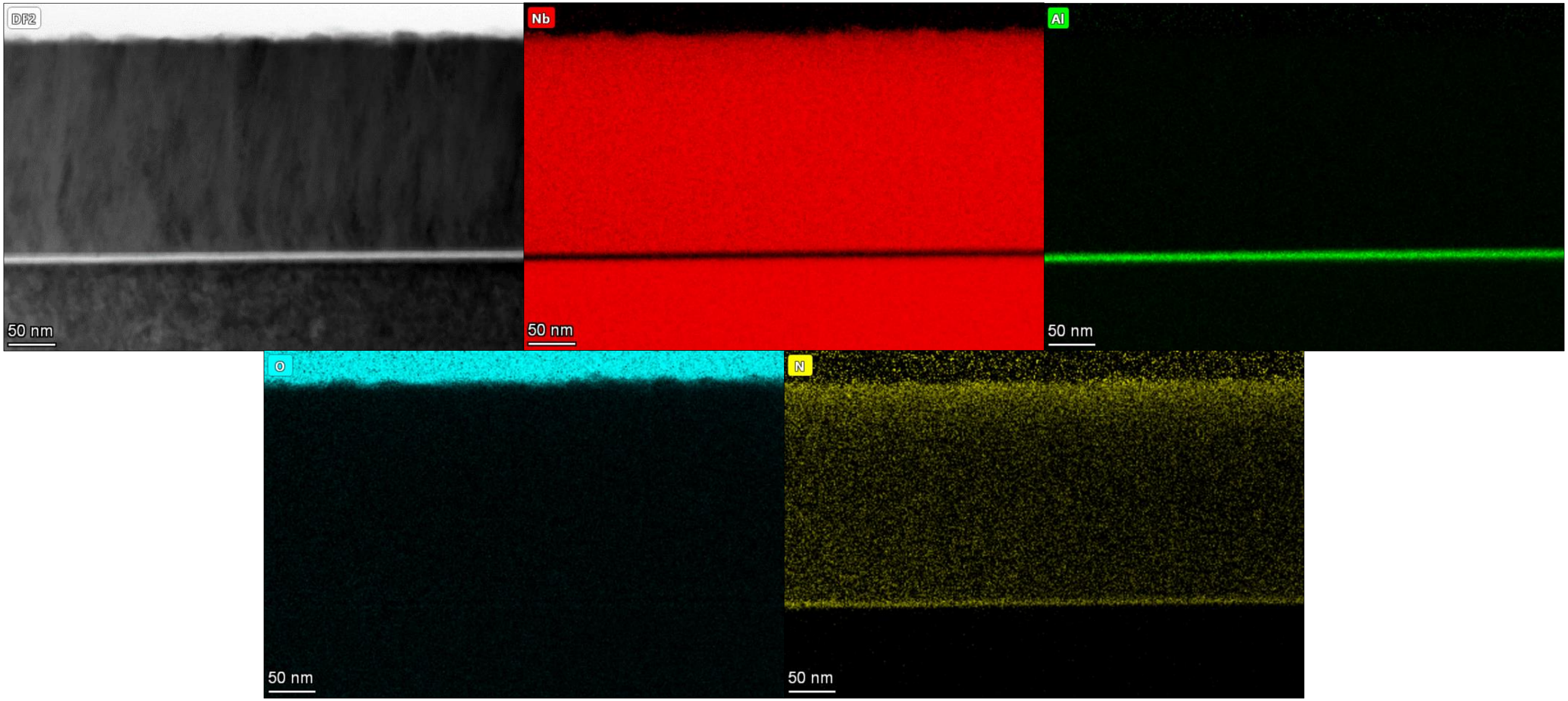








HiPIMS SIS



QPR Deposition

Recipe

- HiPIMS Nb (~3.8 µm)
  - Sample 1077 recipe
  - 400 W (1000 Hz, 120 µs)
  - 8e^{-3} mbar

- AlN (8 or 24 nm)
  - 3500 W, 6e^{-3} mbar, 100% N_2
  - 3x10º pulse

- HiPIMS NbN (180 nm)
  - Sample 1296 recipe (Highest H_{en})
  - 400 W (1000 Hz, 120 µs)
  - 2.2e^{-2} mbar, 10% N_2
  - 56x10º pulses