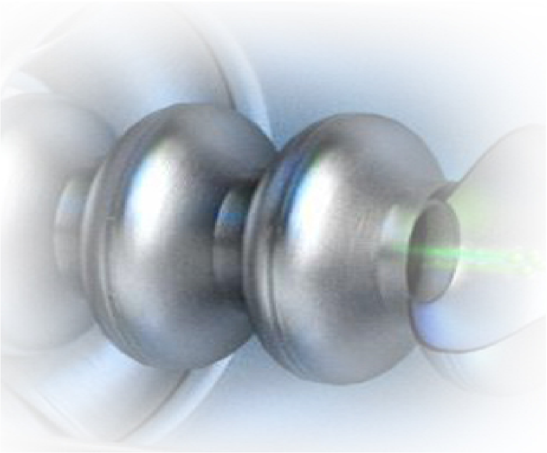
Multilayer Development At JLab



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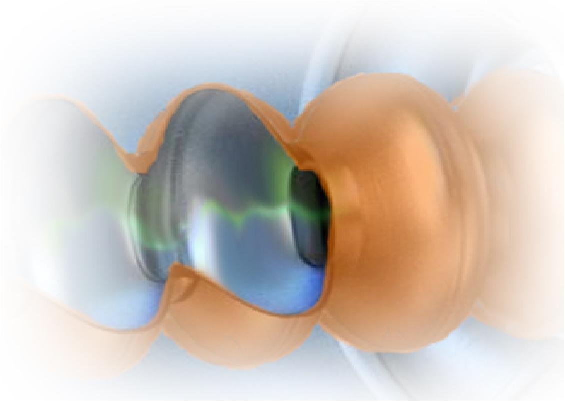

Jefferson Lab

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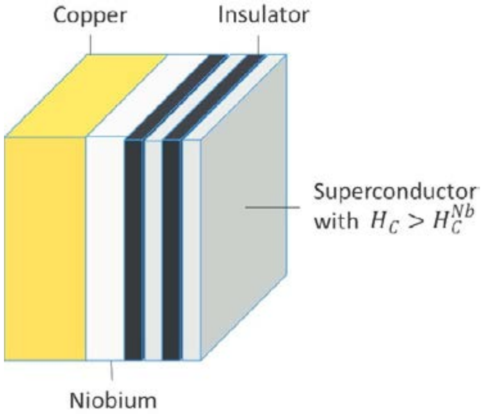
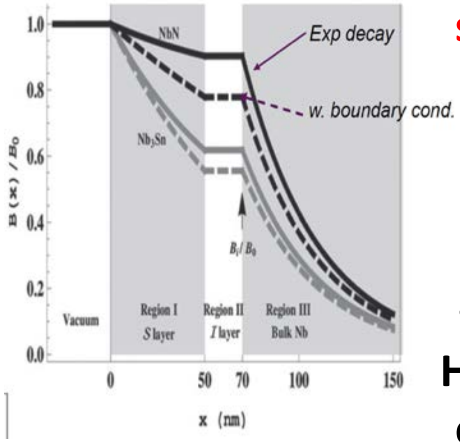
JSA

OUTLINE

- ☐ Introduction
- ☐ Materials choice
- ☐ Experimental Setup
- ☐ NbTiN based SIS structures
- ☐ RF Results
- ☐ Magnetometry Results
- ☐ Nb_3Sn based SIS
- ☐ Conclusion & Future Work

SRF Application beyond Nb: SIS Multilayers



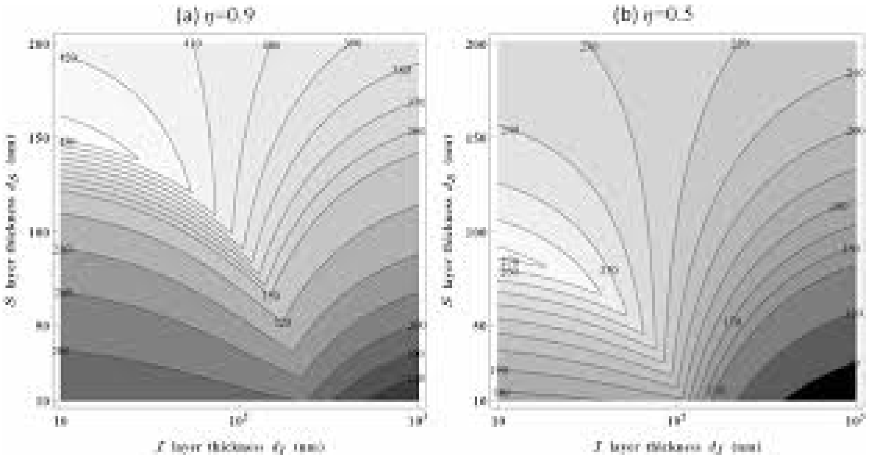


Taking advantage of the high –T_c superconductors with much higher H_c without being penalized by their lower H_{c1}…

Alex Gurevich, Appl. Phys. Lett. 88, 012511 (2006)
Alex Gurevich, AIP ADVANCES 5, 017112 (2015)
T. Kubo, Applied Physics Letters 104, 032603 (2014)

Multilayer coating of SC cavities: alternating SC and insulating layers with d < λ

Higher T_c thin layers provide magnetic screening of the Nb SC cavity (bulk or thick film) without vortex penetration



- ☐ Strong increase of H_{fp} in films allows using RF fields > H_c of Nb, but lower than those at which flux penetration in grain boundaries may become a problem=> no transition, no vortex in the layer
- ☐ High H_{fp} ,applied field is damped by each layer
- ☐ Insulating layer prevents Josephson coupling between layers
- ☐ Applied field, i.e. accelerating field can be increased without high field dissipation
- ☐ SC layers with higher T_c, Δ (Nb_3Sn, NbN, etc.) => Strong reduction of R_{BCS} (ie high Q_0)

Possibility to move operation from 2K to 4.2K

Candidate Superconductors

Material	Critical Temp. T_c [K]	Normal-state resistivity ρ_n [μΩcm]	Critical Field $\mu_0H_c(0)$ [mT]	Lower Critical field $\mu_0H_{c1}(0)$ [mT]	Upper Critical field $\mu_0H_{c2}(0)$ [mT]	Superheating Field $\mu_0H_{SH}(0)$ [mT]	Penetration depth $\lambda(0)$ [nm]	Coherence length ξ [nm]	Gap Δ [meV]	Type
Nb	**9.23**	**2**	**200**	**180**	**400**	**219**	**40**	**28**	**1.5**	**II**
Pb	7.2		80	N/A	N/A		48			I
NbN	**17.1**	**70**	**230**	**20**	**15000**	**214**	**200-350**	**<5**	**2.6**	**II, B1**
NbTiN	**17.3**	**35**		**30**	**17000**		**150-450**	**<5**	**2.8**	**II, B1**
Nb_3Sn	**18.3**	**20**	**540**	**50**	**30000**	**425**	**80-100**	**<5**	**3.1**	**II, A15**
V_3Si	17	4	720	72	24500		179	3	2.5	II, A15
Mo_3Re	15	10-30	430	30	3500	170	140			II, A15
MgB_2	**39**	**0.1-10**	**430**	**30**	**3500**	**170**	**140**	**5**	**2.3/7.2**	**II- 2 gaps**
~~YBCO~~	~~93~~		~~1400~~	~~10~~	~~100000~~	~~1050~~	~~150~~	~~0.03/2~~	~~20~~	~~d-wave~~
2H-$NbSe_2$	7.1	68	120	13	2680-15000	95	100-160	8-10		II- 2 gaps
Pnictides	**30-55**		**500-900**	**30**	**>50000**	**756**	**200**	**2**	**10-20**	**s/d-wave**

Dielectrics – requirements & candidates

Requirements:

- ☐ Good dielectric
- ☐ Lattice mismatch of S & I materials
- ☐ Sharp interfaces
- ☐ No intermixing between S & I layers
- ☐ Preserve layer material quality when depositing the subsequent layer so individual properties (superconducting or insulating) are not degraded
- ☐ Preserve underlying bulk superconductor

Superconductor

Nb_3Sn 5.327Å
NbTiN 4.35 Å

Insulator

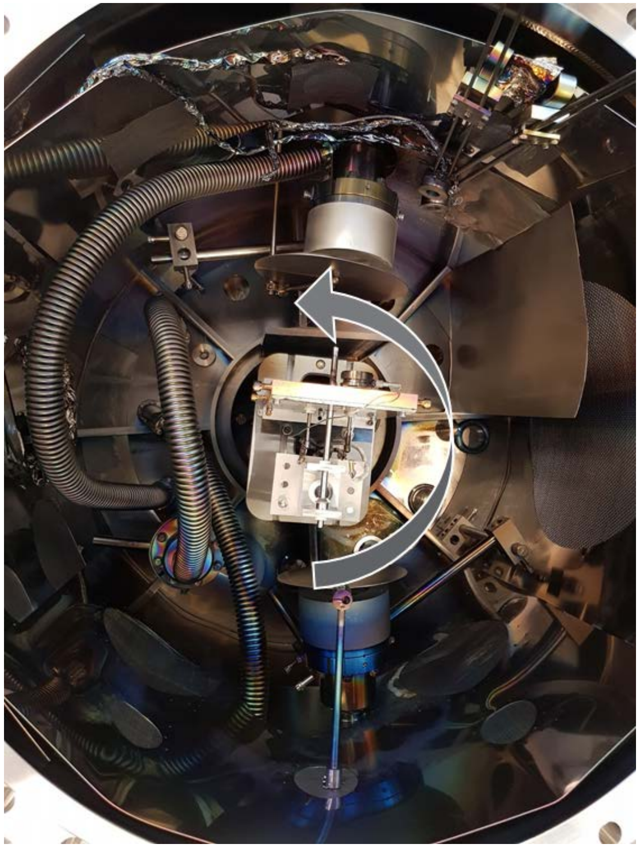
AlN: a= 4.08 Å, n=2.16
Zr_3N_4:a= 5.899 Å (dielectric), n=3.64
ZrN : a= 4.5675 Å cubic (metallic behavior)
Al_2O_3: amorphous, α-Al2O3 , a= 4.78 Å, n=1.77
MgO: highly reactive, a= 4.05 Å, n=1.73

Other considerations for materials choice for SIS Structures

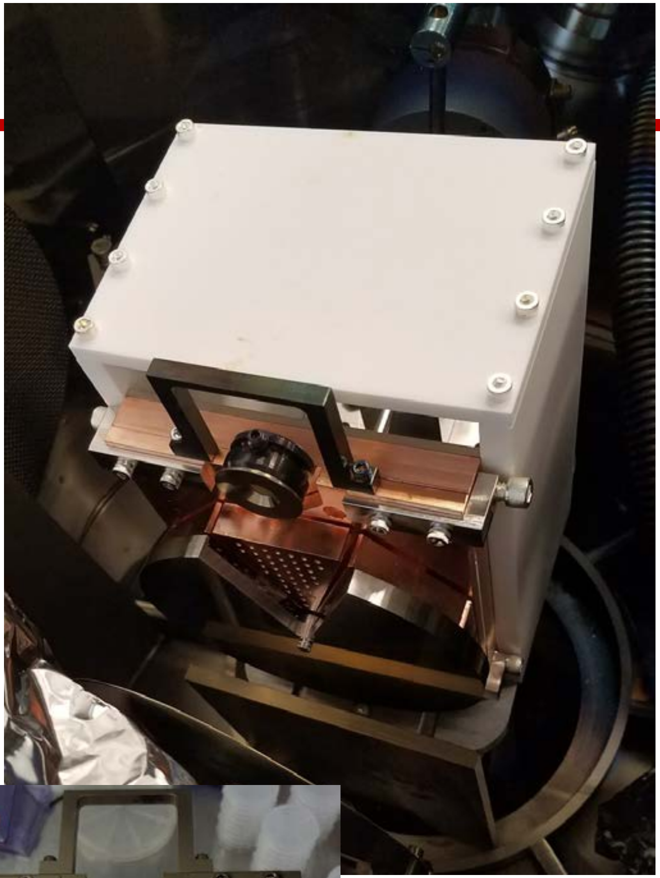
- ☐ Adequate template for each layer: amorphous or poly-crystalline affect growth thus properties of layer material
- ☐ Crystal phases achieved as function of coating temperature, …
- ☐ Stability in temperature
- ☐ Reactivity between S & I layer compounds
- ☐ Chemical inertness (transition metal nitrides)
- ☐ Achievable roughness
- ☐ Deposition methods: -achievable or required temperature
  - -hyperthermal ions versus substrate T
  - -conformality, scalability to large 3D structures
- ☐ Continuous, immiscible and low-strained interfaces S-I AND I-S.
- ☐ Existing chemistry for removal/etching?

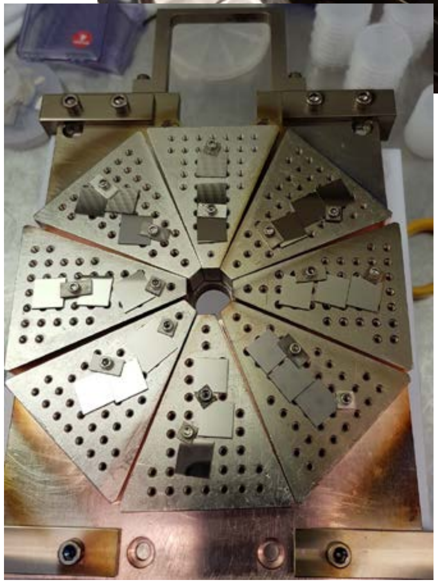
UHV DEPOSITION SYSTEM

Samples heated up to 600 °C
Base pressure 10^{-10} Torr
3 magnetron sources
DC, RF and HiPIMS capable



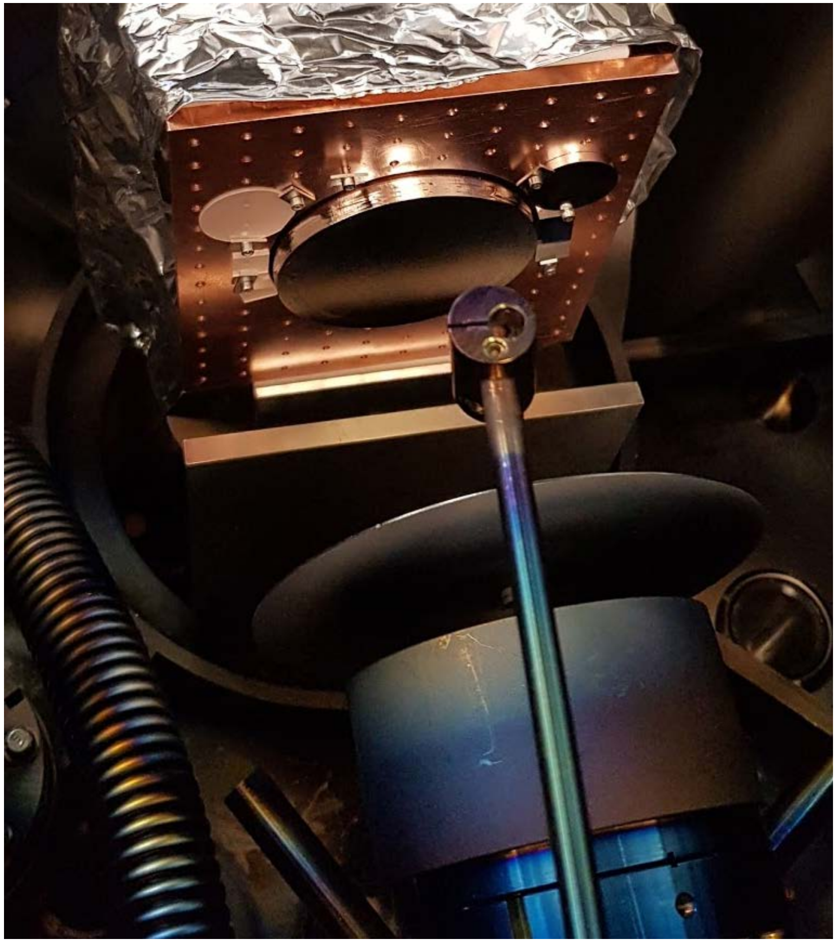
Central stage mounted on differentially pumped CF system to position samples in front of each magnetron source

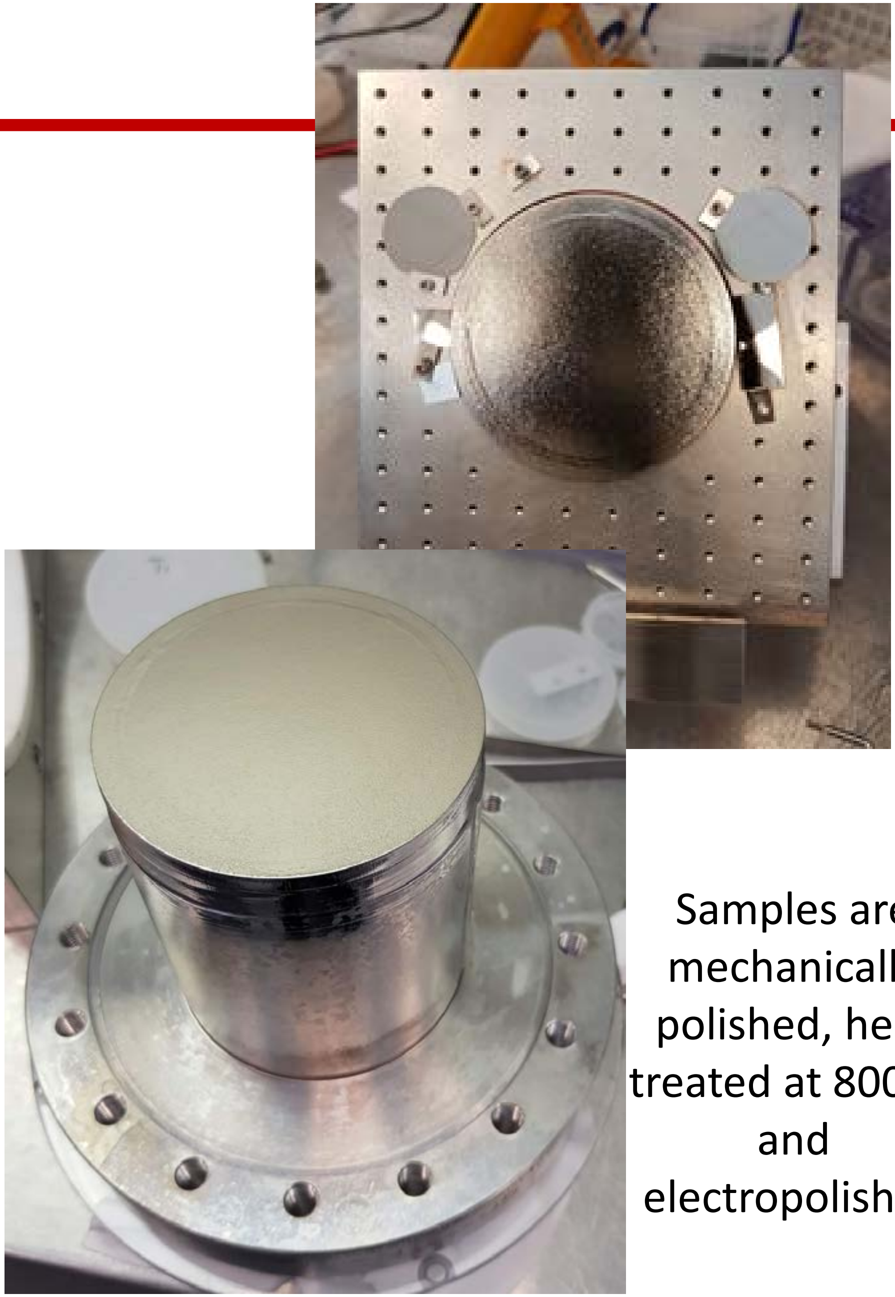




UHV DEPOSITION SYSTEM

QPR Sample Coating Setup





Samples are mechanically polished, heat treated at 800 °C and electropolished

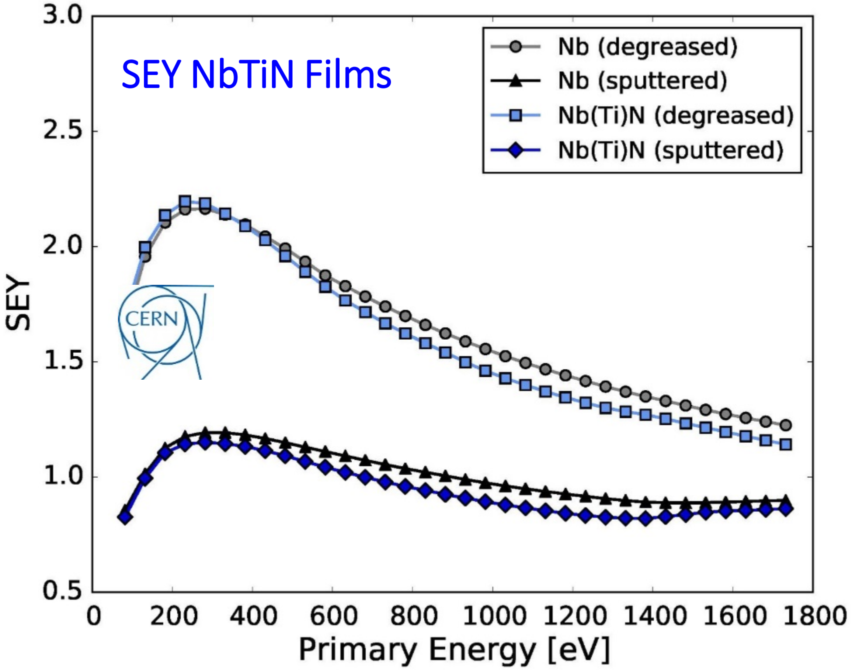
SIS Multilayers: Materials Choice

NbTiN

Good superconductor, bulk Tc~17.3 K

δ-phase for T_c> 16 K

Typically more stable and metallic behavior than NbN



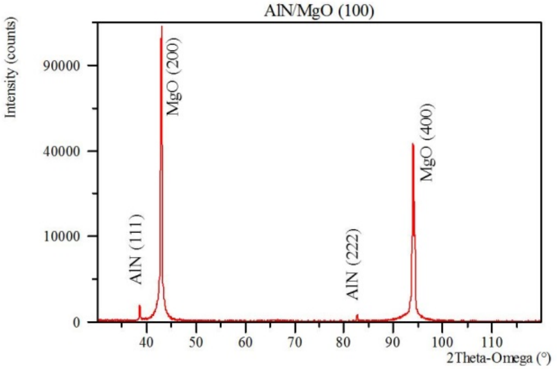
Measurements at room temperature

Max. SEY =2.2 ± 0.1 comparable to EP Nb

After sputtering away ~ 3 nm, SEY down to 1.15

AlN

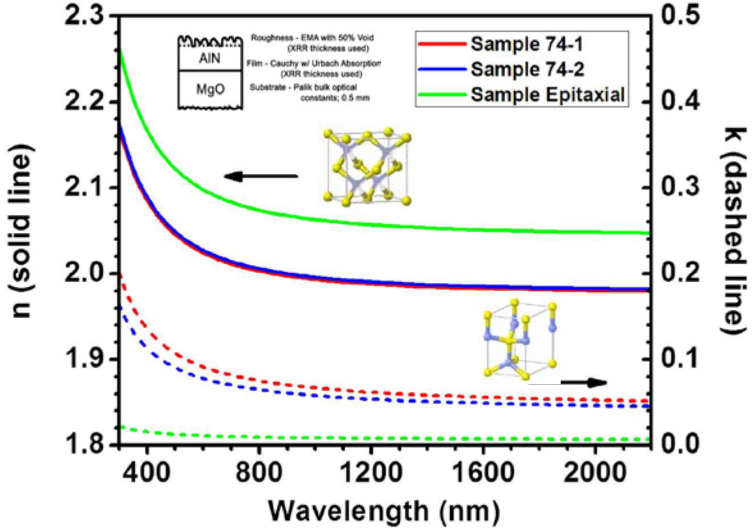
Good Dielectric Behavior



Good quality AlN are readily produced at 600 and 450°C by dc-reactive magnetron sputtering with N_2/Ar ~33%.
The films exhibit the cubic structure (single crystal) at 600 °C and the hexagonal structure (polycrystalline) at 450 °C .

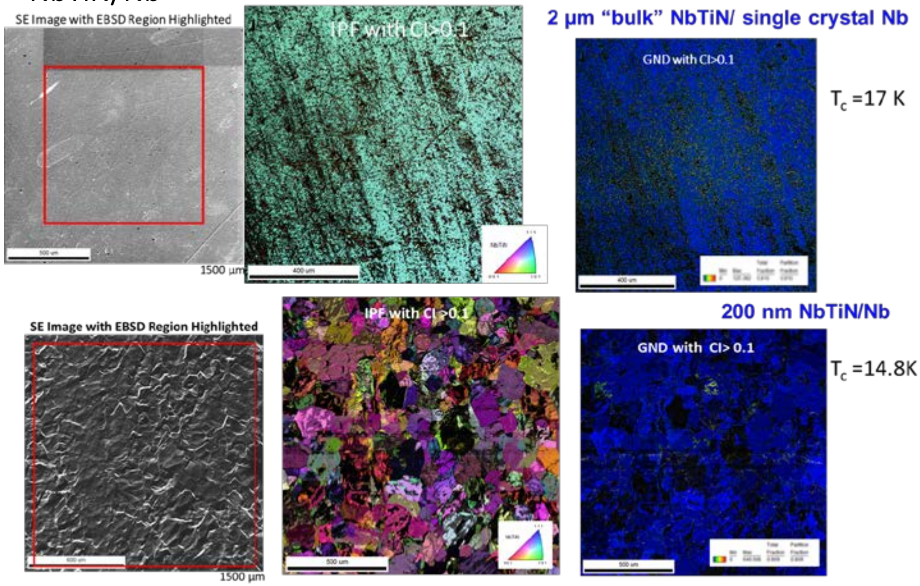
At 450 °C, 30 nm AlN films exhibit dielectric properties of polycrystalline AlN films

n in the range of 1.98- 2.15



SIS NbTiN/AlN structures on MgO & Nb surfaces

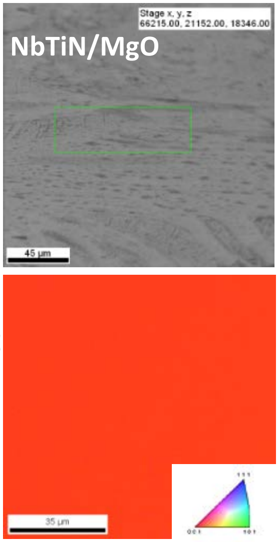
NbTiN/Nb

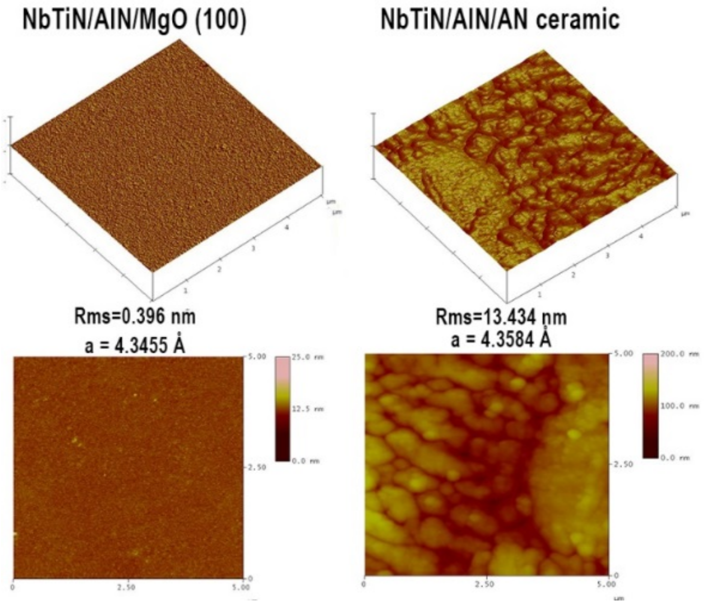


T_c =17 K

T_c =14.8K

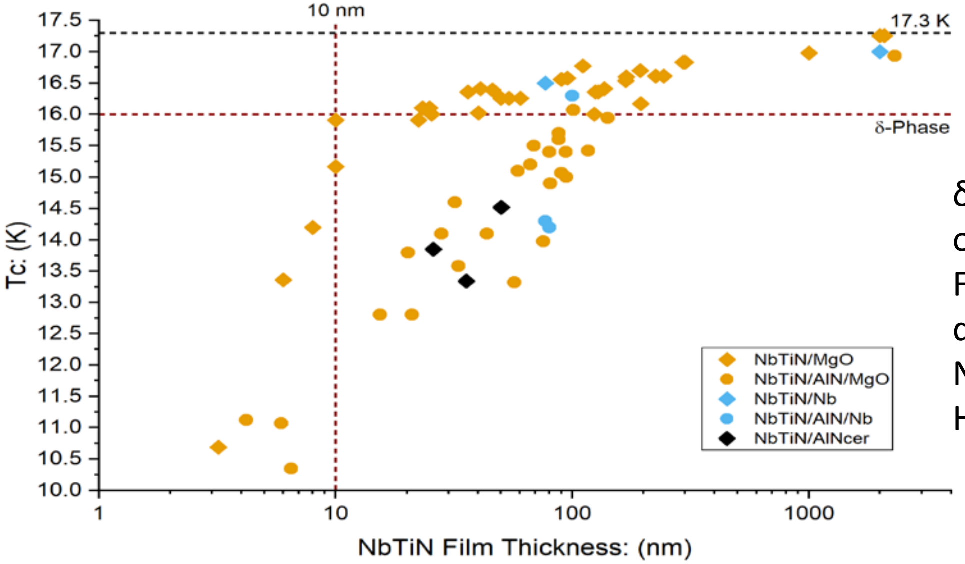
NbTiN/MgO





NbTiN/AlN/MgO (100): Rms=0.396 nm, a = 4.3455 Å

NbTiN/AlN/AN ceramic: Rms=13.434 nm, a = 4.3584 Å



δ-phase achieved for films deposited at 450°C on MgO, 10 nm and up (~ ideal case).
For other substrates, metallic or ceramic, desirable phase achieved at higher thickness
NbTiN/AlN ceramic
Have a T_c slightly depressed in comparison

Film Interface development in SIS Multilayers - Metamaterials

Metamaterial superconductor based on NbTiN (metamaterials synergistic project, DARPA-BAA funded)

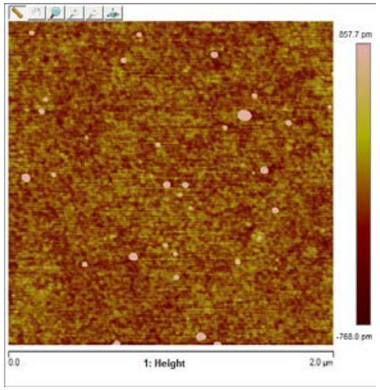
Nature Scientific Reports 6, Article number: 34140 (2016)

- Multilayer structure of NbTiN = 3 nm and AlN = 2/1.5/1 nm. Series of 16-, 8-, 4-, 2-, 1- bilayers deposited on NbTiN/MgO
- Metamaterial layered superconductor of NbTiN and AlN can enhance T_c compared to NbTiN single layer.
- Low roughness of sequential films is necessary to accomplish sharp interfaces.

V. Smolyaninova et al.

Bilayers deposited on NbTiN/MgO.

# bilayers	NbTiN [nm]	AlN [nm]
16	3	2.5
8	3.3	2.4
4	4.3	2.5
2	3.4	2



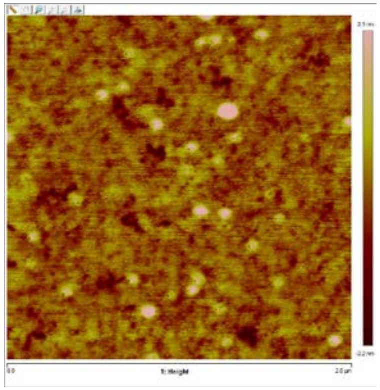
8 layers of NbTiN/AlN on NbTiN/MgO

RMS = 0.389 nm,

Ra = 0.19 nm

NbTiN = 3.3 nm,

AlN = 2.5 nm



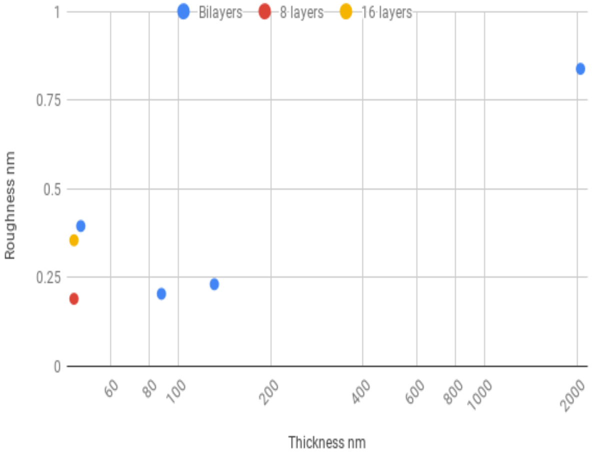
16 layers of NbTiN/AlN on NbTiN/MgO

RMS = 0.556 nm,

Ra = 0.355 nm

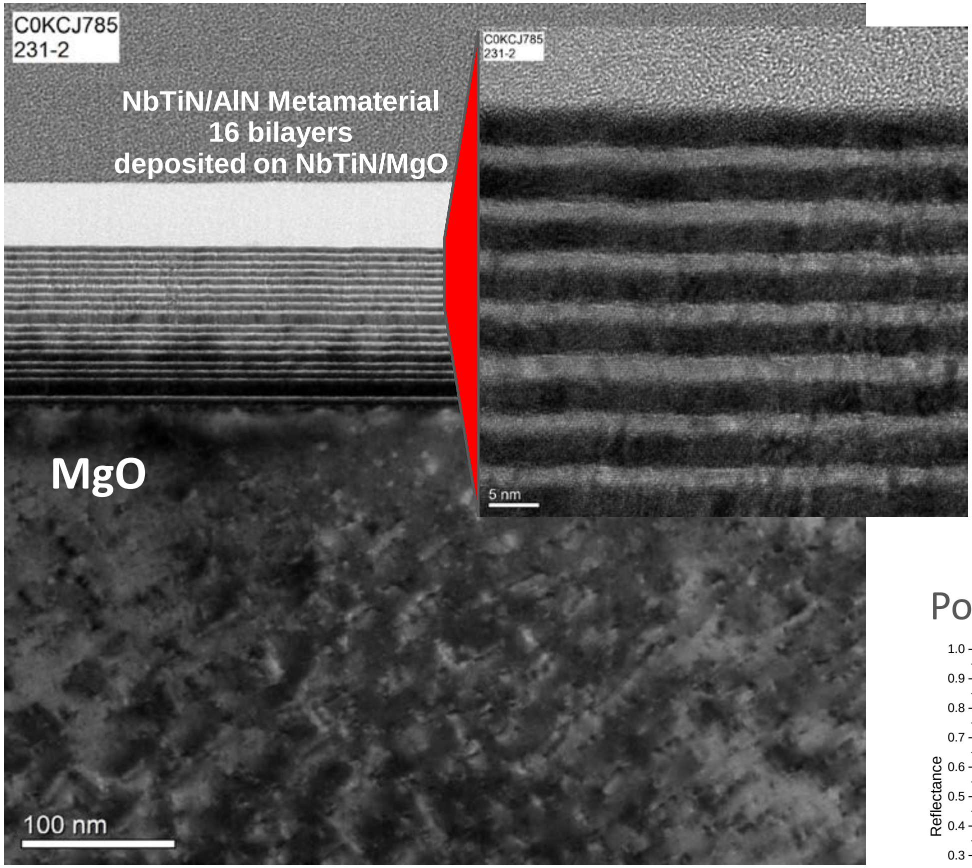
NbTiN = 3 nm,

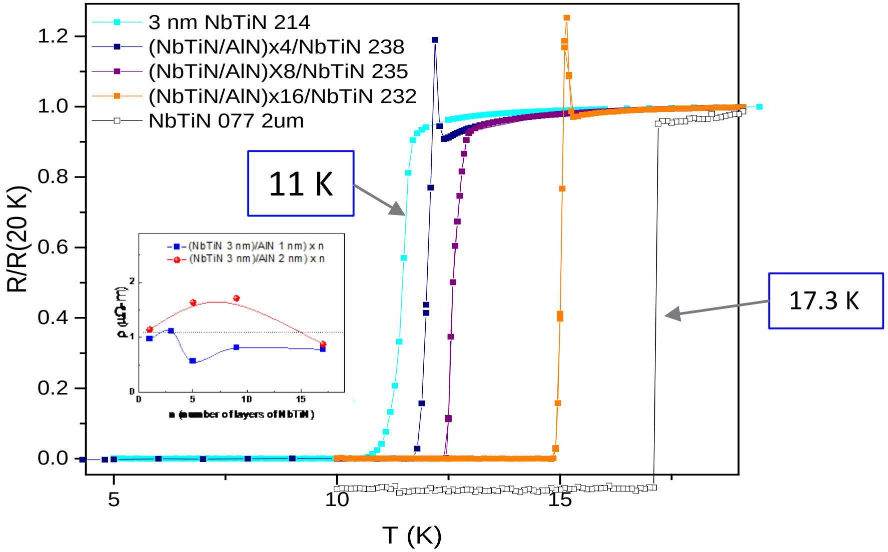
AlN = 2.4 nm



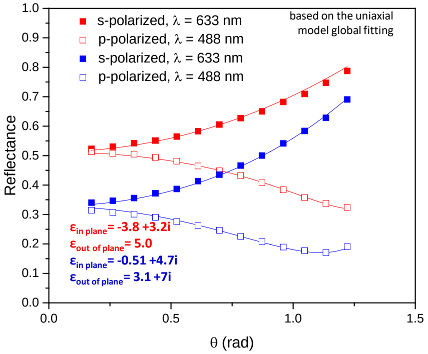
No significant increase of film roughness with additional layers.
Preserving the potential for sharp interfaces

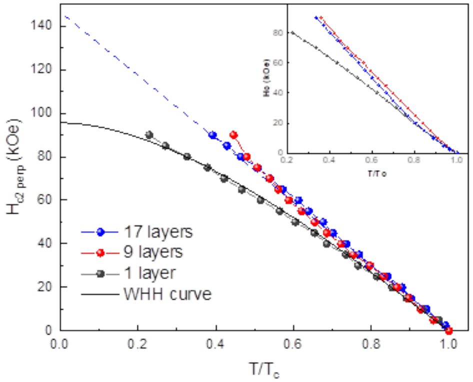
Metamaterial superconductor based on NbTiN





Polarization reflectometry



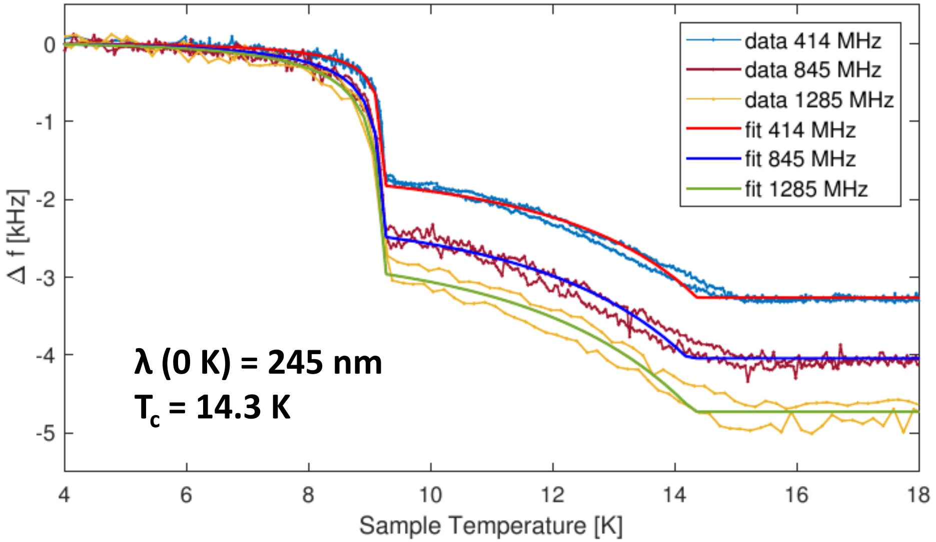


V. Smolyaninova et al.

Hyperbolic metamaterial properties demonstrated

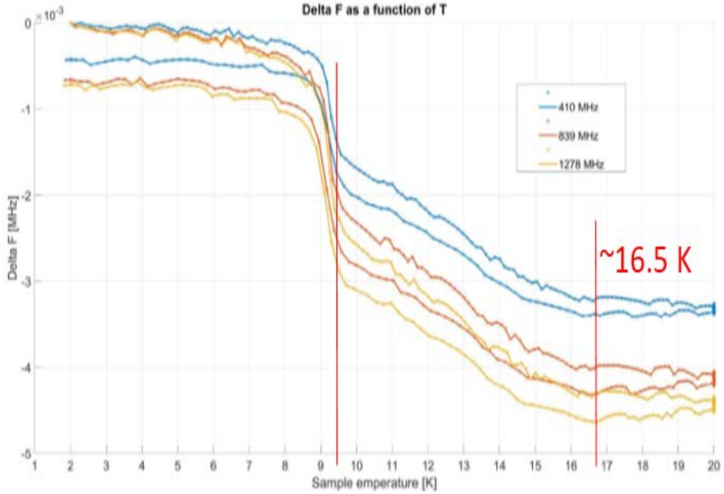
T_c enhancement with increased number of bi-layers but limited due to small coherence length

NbTiN based QPR Samples – T_c & λ



λ (0 K) = 245 nm

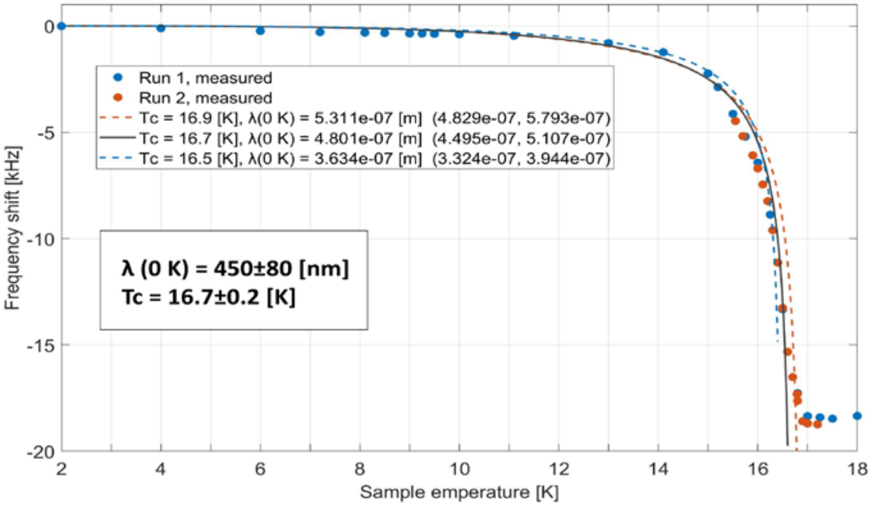
T_c = 14.3 K


Delta F as a function of T

~16.5 K

Sample Temperature (K)

Measured resonance frequency shift as a function temperature for the thick film sample (a)

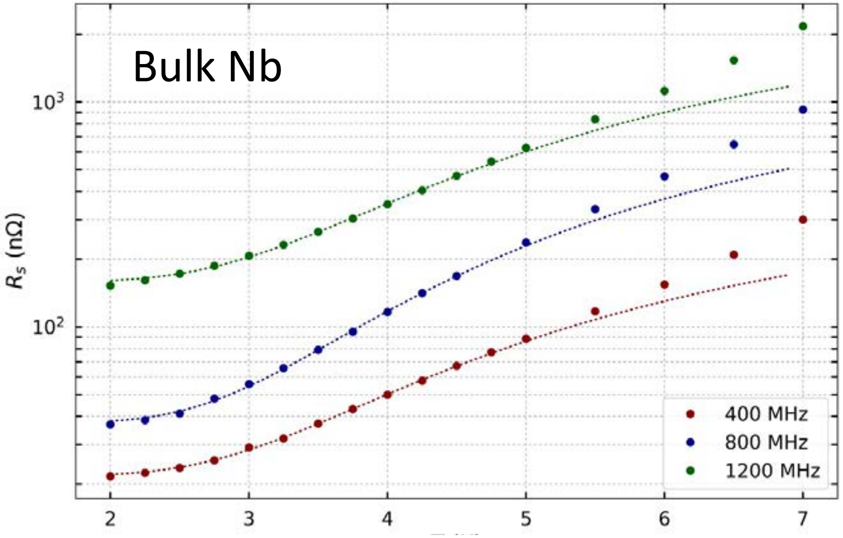


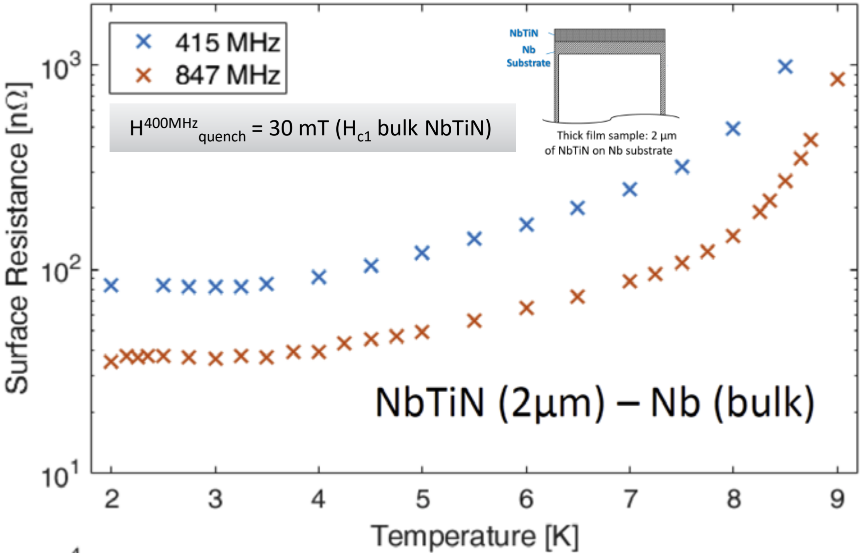
λ (0 K) = 450±80 [nm]
Tc = 16.7±0.2 [K]

QPR measurements @ HZB

S. Keckert ,D. Tikhinov

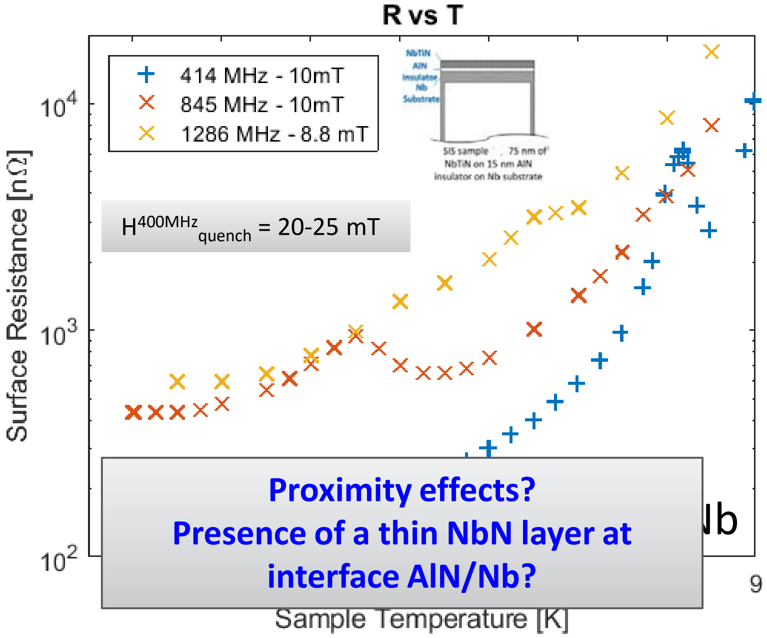
NbTiN Structures versus Bulk Nb Baseline – $R_s(T)$





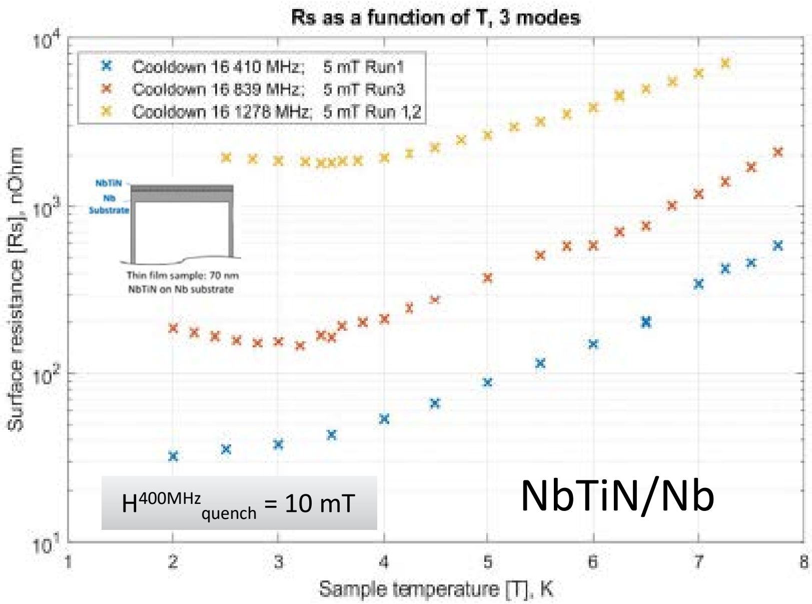
H^{400MHz}_{quench} = 30 mT (H_{c1} bulk NbTiN)

NbTiN (2µm) – Nb (bulk)



H^{400MHz}_{quench} = 20-25 mT

Proximity effects?
Presence of a thin NbN layer at interface AlN/Nb?



H^{400MHz}_{quench} = 10 mT

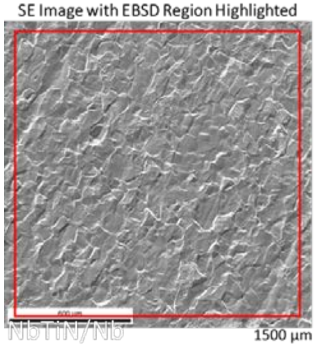
NbTiN/Nb

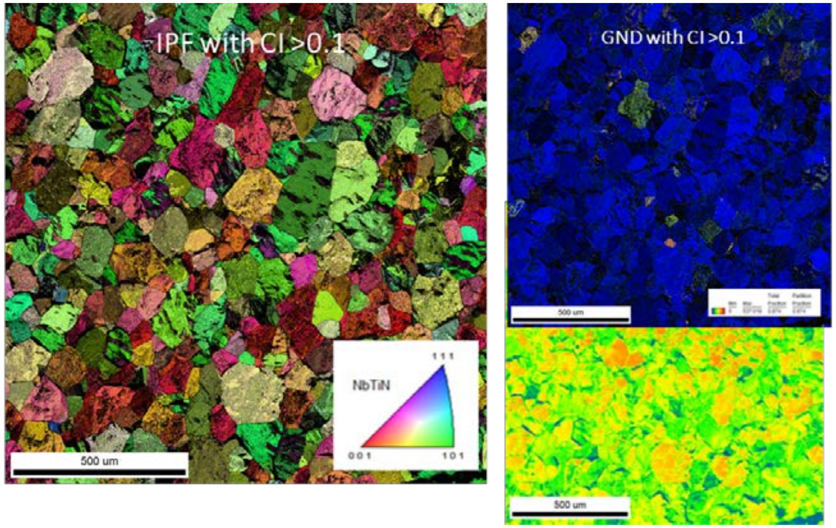
NbTiN based QPR samples structure

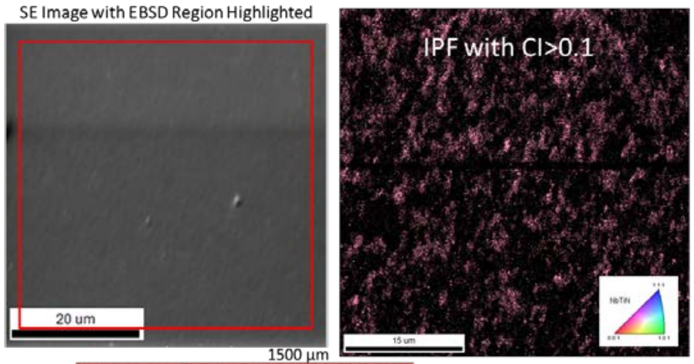
75 nm NbTiN/AlN/Nb

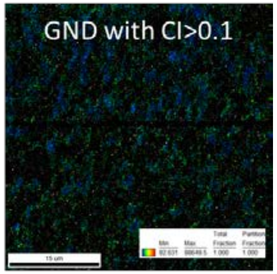
Not able to acquire EBSD map (only a few Kikuchi pattern here and there)

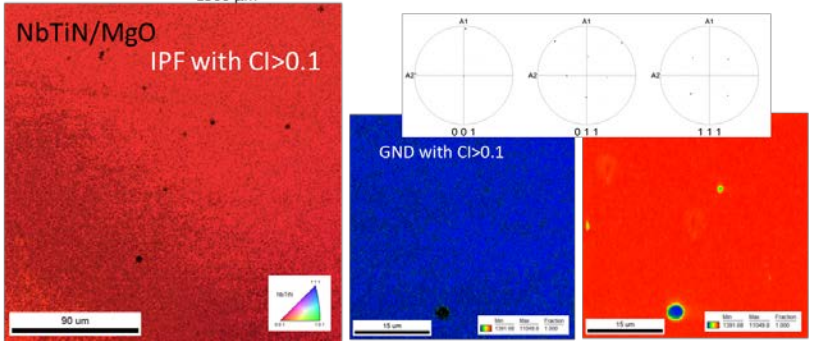
75 nm NbTiN/Nb







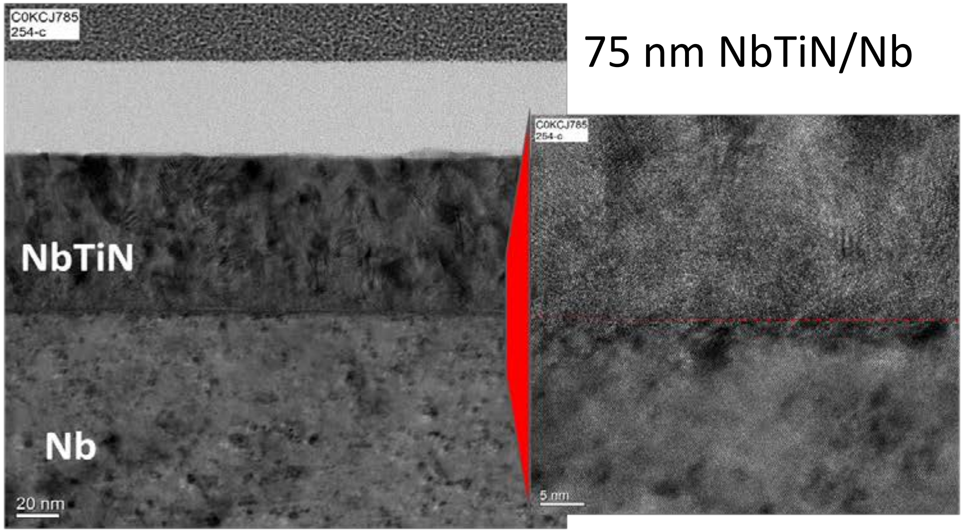


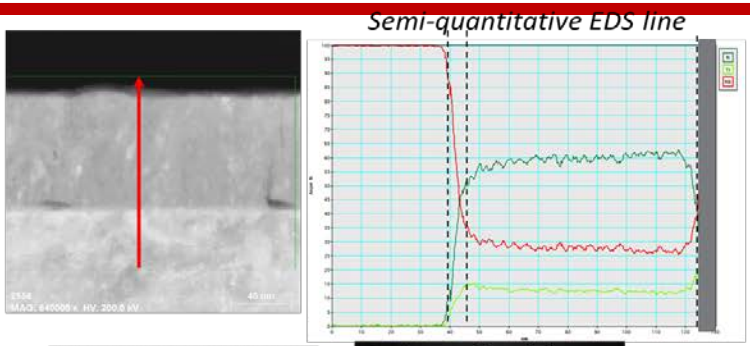


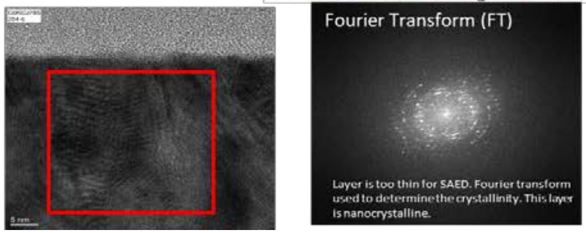
Bulk NbTiN (2µm)

Interfaces

75 nm NbTiN/Nb

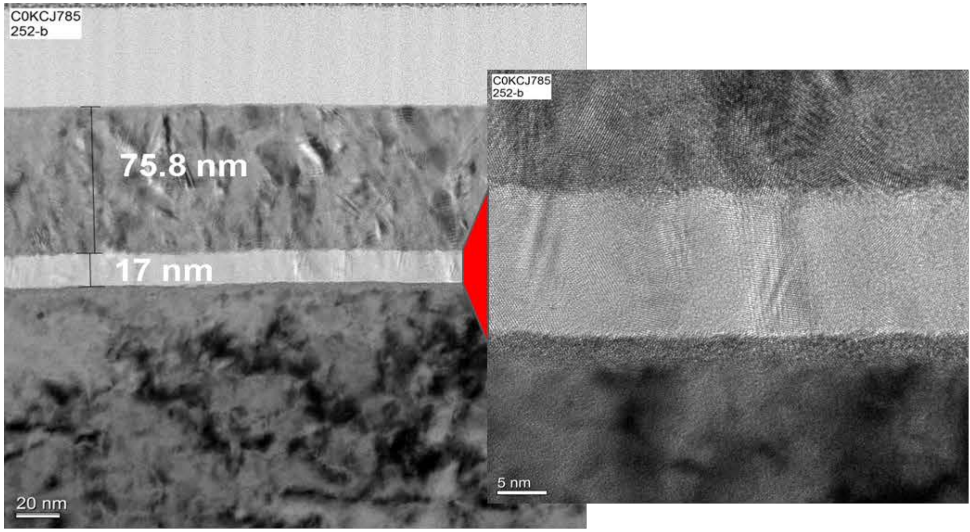


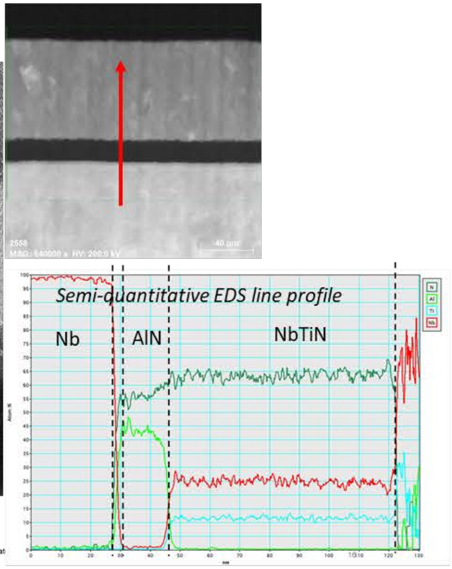




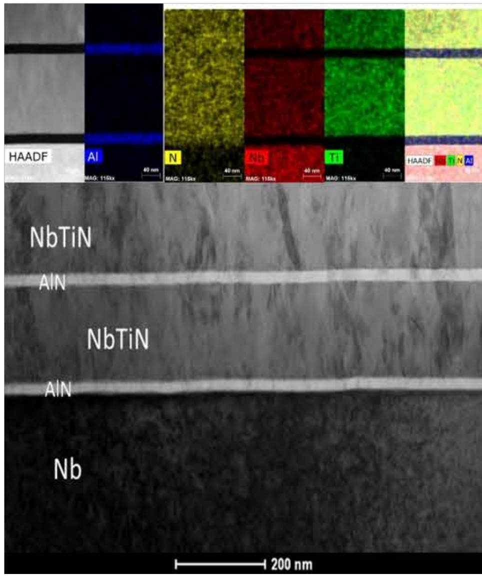
Layer is too thin for SAED. Fourier transform used to determine the crystallinity. This layer is nanocrystalline.

75 nm NbTiN/AlN/Nb

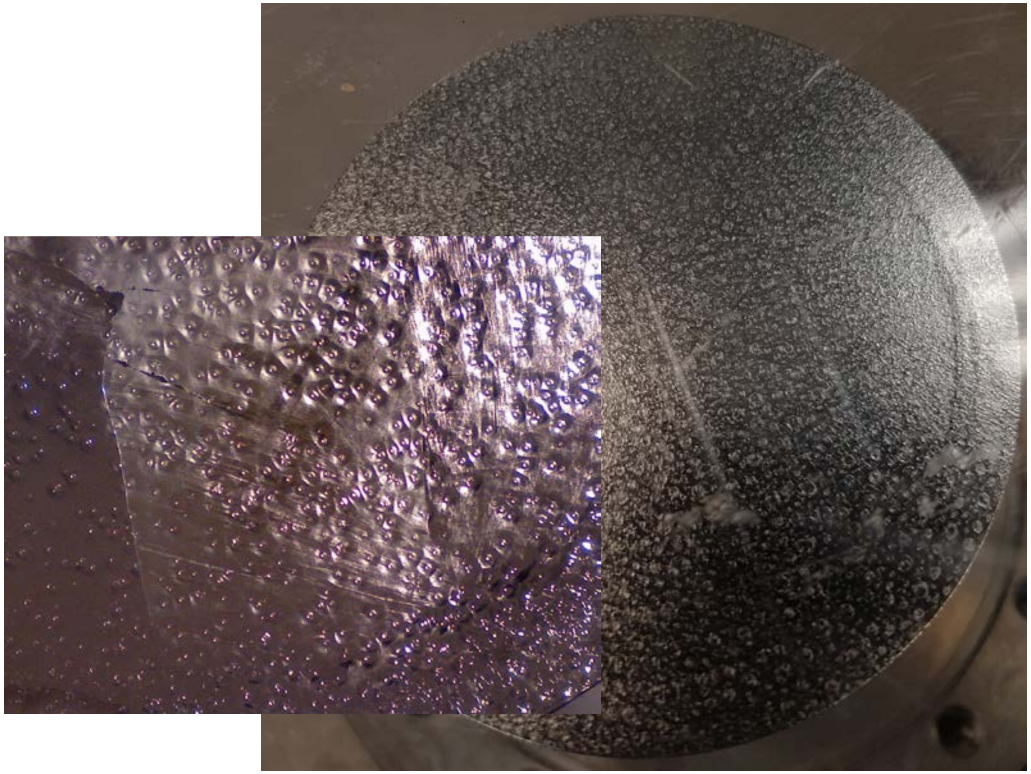




$(NbTiN/AlN)_2/Nb$



Known Issues



QPR sample after SIS removal and EP

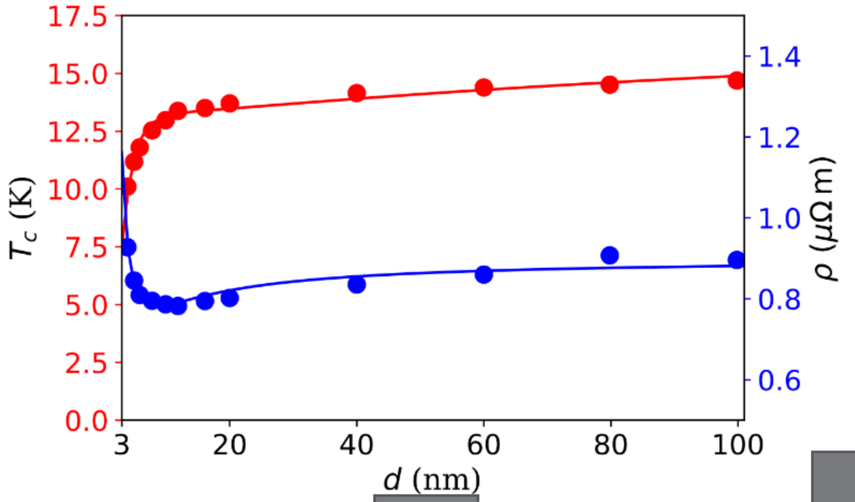
- 1st coating cycle aborted due to short in NbTi Magnetron source
- AlN removal with KOH
  (Need to establish new chemistry for alternative materials)
- Features appeared on the Nb surface, only removed with extensive EP

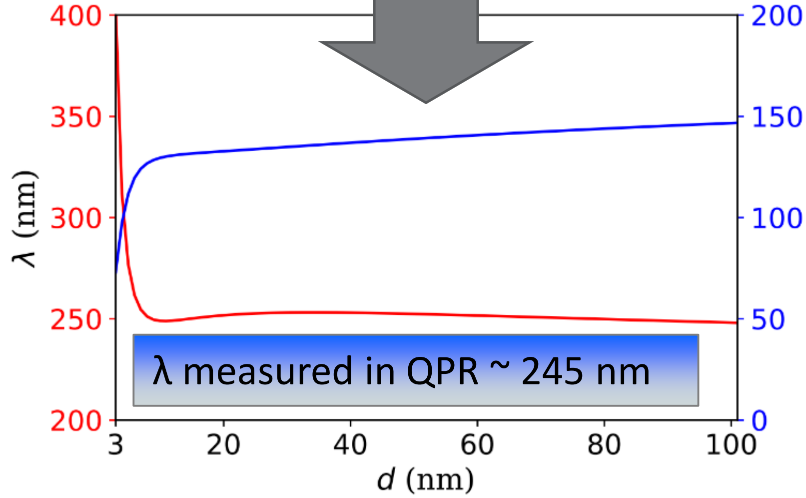
issues with the bulk Nb material quality

Potential H_{max} for NbTiN

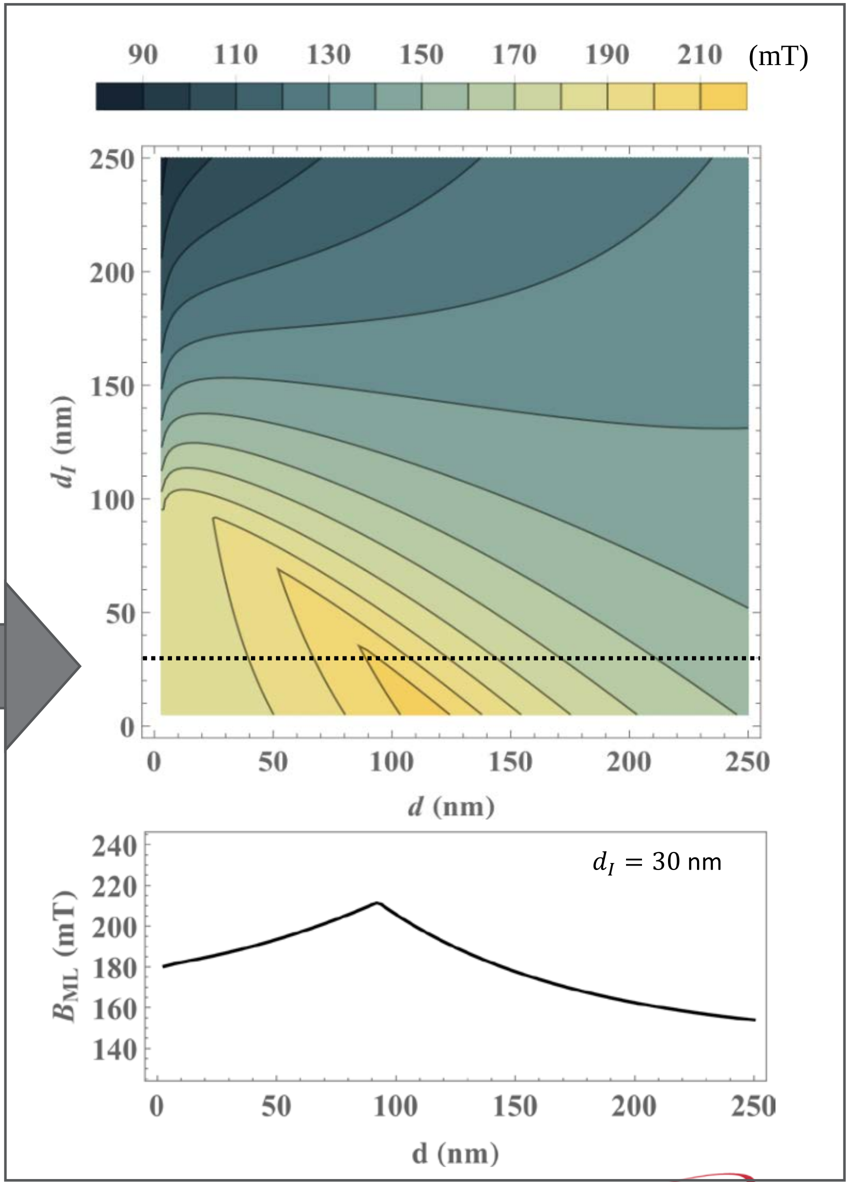
Courtesy T. KUBO

T_c and ρ from L. Zhang et al., Appl. Phys. Lett. 107, 122603 (2015)
$\Delta = 1.86\ k_B T_C$ from T. Hong et al., J. Appl. Phys. 114, 243905 (2013).
$N_0 = 1.17 \times 10^{47}\ J^{-1} m^{-3}$
from D. Hazra et al., Phys. Rev. B 97, 144518 (2018)





λ measured in QPR ~ 245 nm

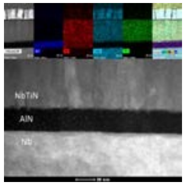


Field enhancement with NbTiN SIS Multilayered Structures

Some NbTiN/AlN SI structures exhibit **H_{fp} enhancement** compared to bulk-like NbTiN film

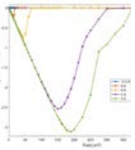
Measurement on NbTiN SIS structure and films samples

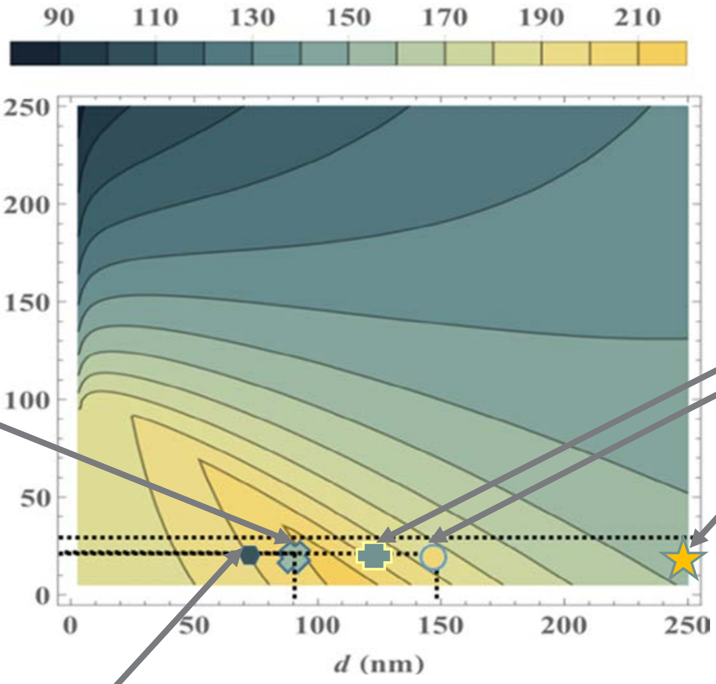
DC/SQUID



T_c=14.2 K

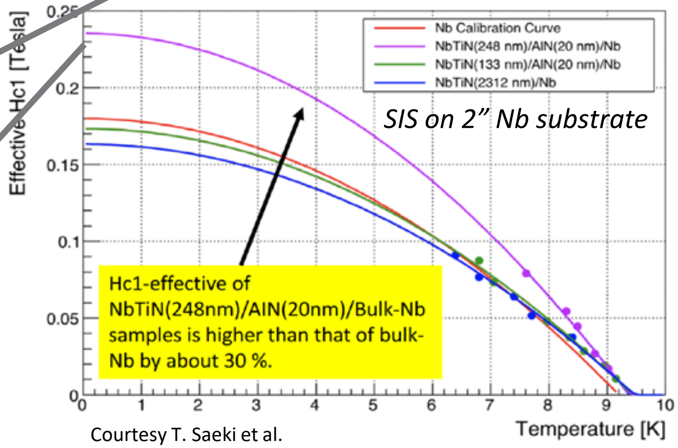
H_{fp} ~155 mT





Contour plot calculated for NbTiN [T. Kubo]

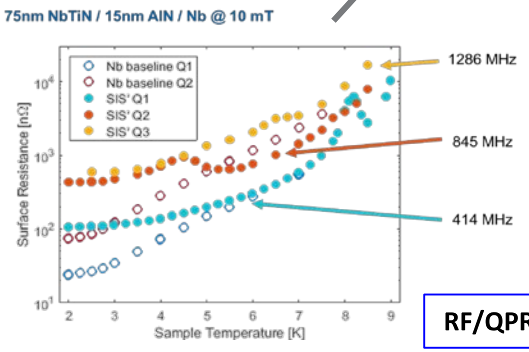
	Thickness [nm]	H_{c1} [mT]	T_c [K]
NbTiN/MgO	2000	30	17.3
NbTiN/AlN/AlN ceramic	145	135	14.8
NbTiN/AlN/MgO	148	200	16.7



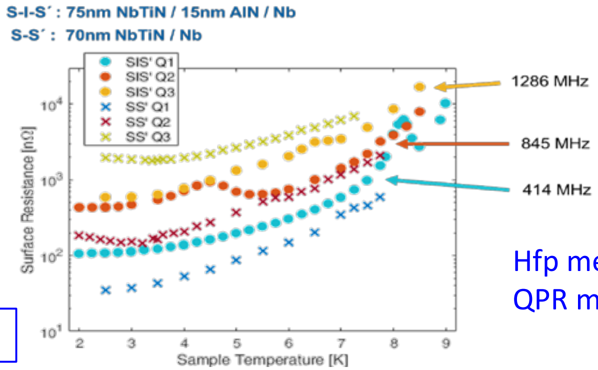
SIS on 2" Nb substrate

Hc1-effective of NbTiN(248nm)/AlN(20nm)/Bulk-Nb samples is higher than that of bulk-Nb by about 30 %.

Courtesy T. Saeki et al.



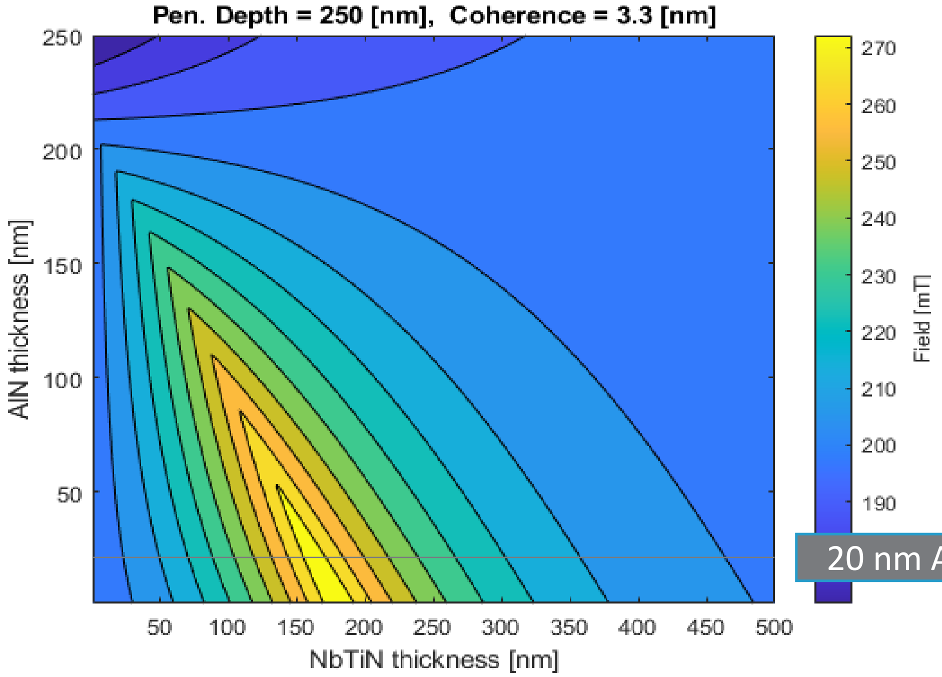
RF/QPR



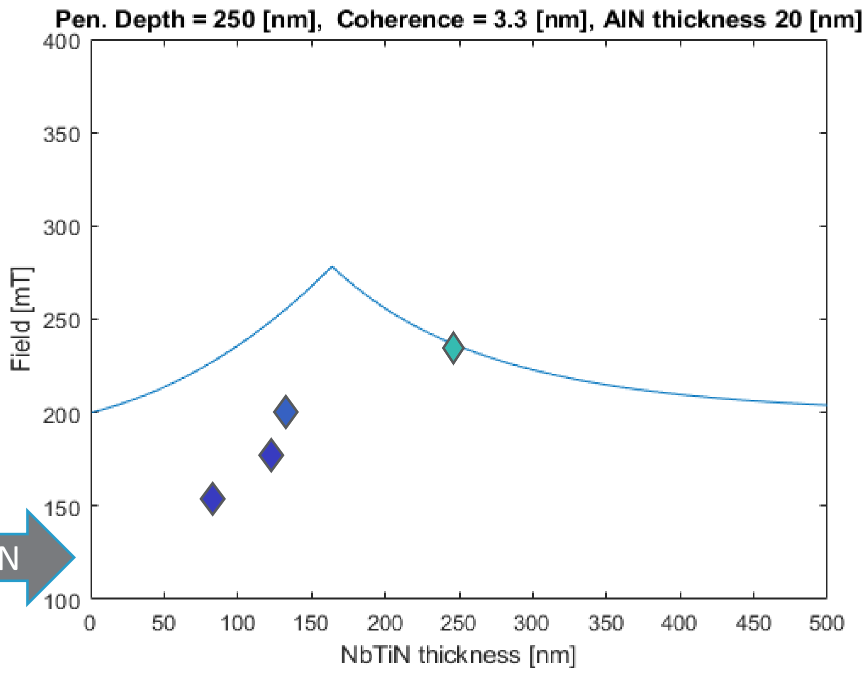
Hfp measurement by SQUID in agreement with QPR measurement.

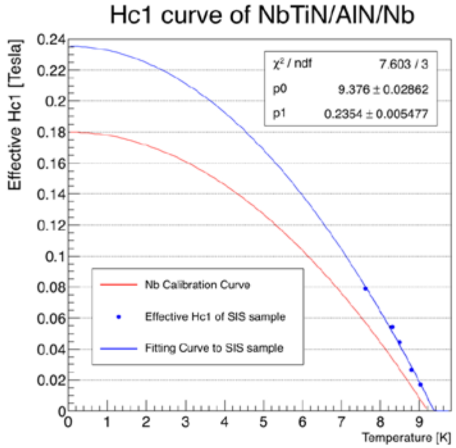
Field enhancement with NbTiN SIS Multilayered Structures

Refine the contour plot with parameters from actual films coated



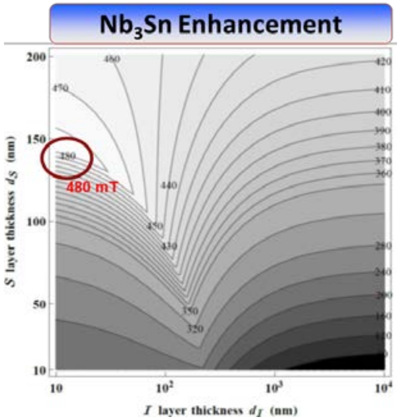
20 nm AlN

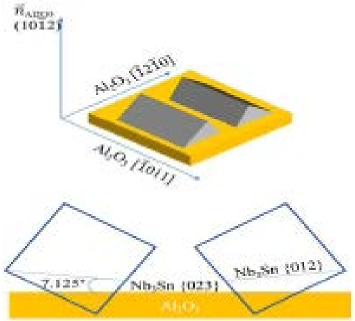


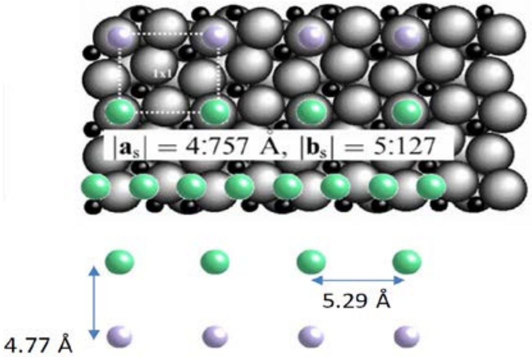


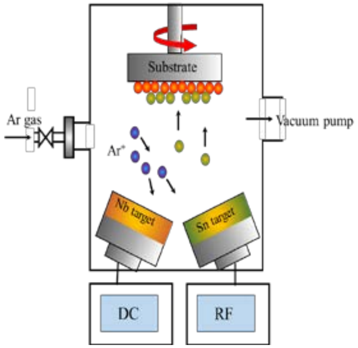
Nb_3Sn based SIS

Higher field enhancement anticipated with Nb_3Sn (5.327Å)
Potential insulator: Al_2O_3 (highly stable in temperature amorphous, α-Al_2O_3 depending on technique, parameters)



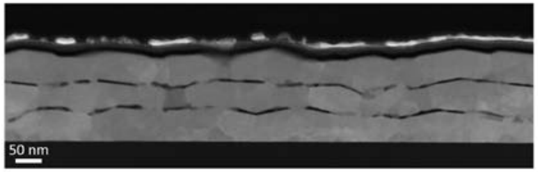




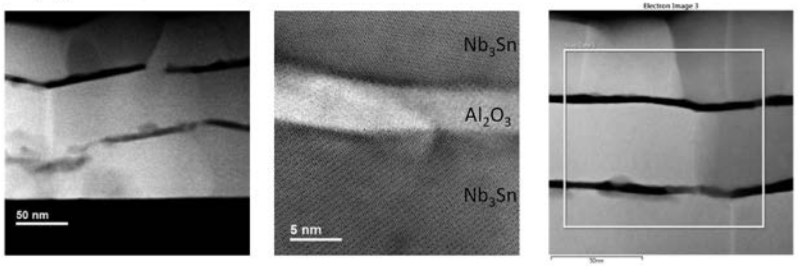


Deposition method	T_c	ΔT_c	RRR
Stoichiometric target	17.83	0.03	5.41
Co-sputtering	17.6	0.22	3.63

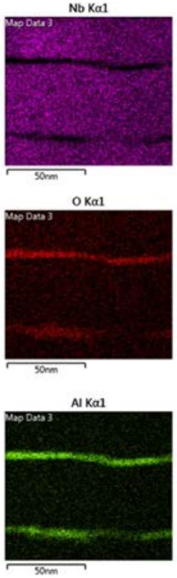
M N Sayeed et al 2020 IOP Conf. Ser.: Mater. Sci. Eng. 756 012014



Al_2O_3 present, but incomplete



Al_2O_3 layer well-confined



Development and characterization of Nb3Sn/Al2O3 superconducting multilayers for high-performance radio-frequency applications
Christopher Sundahl, TFSRF 2021

Collaborations: HZB, STFC, CEA Saclay, TRIUMF

CONCLUSION & FUTURE WORK

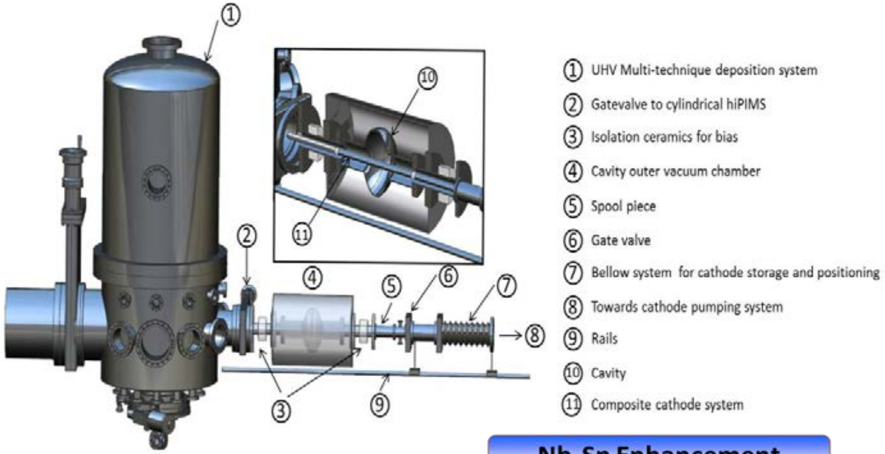
In depth analysis of RF behavior ongoing across frequencies, cooling and applied magnetic field condition
3rd harmonic measurements for first penetration field measurements ongoing (CEA Saclay, KEK, JLab)

- **Thickness and T_c on large sample are consistent with small sample studies**
- **H_{max} field values towards higher thicknesses than predicted**
- **Films and structures follow substrate topology**

- ❑ S-I-S' deposition on QPR sample with no surface weld
- ❑ Continue exploring NbTiN/AlN thickness with QPR and 3rd harmonic measurements
- ❑ Reactive HiPIMS to densify the films and get relaxed structure at earlier growth stage
- ❑ Explore other adequate dielectric (Zr_3N_4, …)
- ❑ ***Implement cavity deposition with cylindrical reactive DC-MS and HiPIMS***

- ❑ ***Develop Nb_3Sn based SIS structures with HiPIMS/ECR for high Nb incident ion energy***

SIS cavity deposition design



Nb_3Sn Enhancement