ALD-Based NbTiN studies for SIS R&D

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TESLA Technology Collaboration Meeting (Virtual)

19th-21st January 2021

Outline

- Superconductor- Insulator- Superconductor multistructures motivation and possible materials
- Atomic Layer Deposition: Thermal and Plasma
- Plasma Enhanced Atomic Layer Deposition: NbTiN thin films
- Process Optimization of PEALD NbTiN
- Effect of thermal treatment on NbTiN thin films

Motivation: S-I-S Multilayers

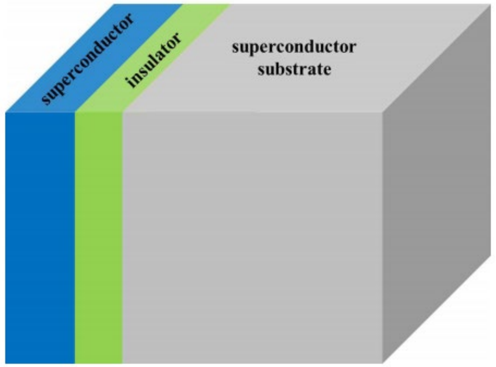
New structure proposed by A. Gurevich [1]

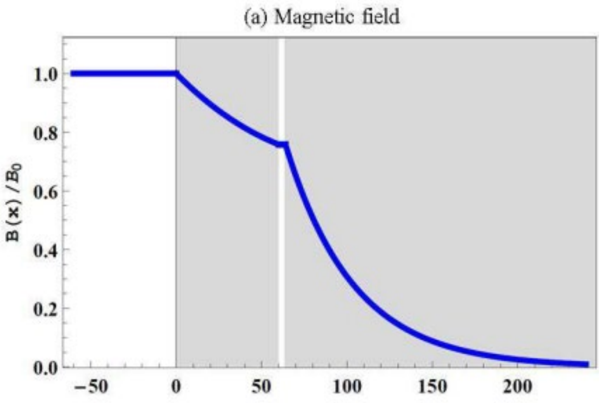
The idea is to coat the SRF cavities with alternating superconducting and insulating layers.

Requirements for the superconductor:

- Thin film thickness << λ_L
- Higher Tc and Δ
- Lower ρ_n

These multilayers provide magnetic screening of the bulk cavity and lower surface resistance which allows to increase the accelerating field and reduce the losses.



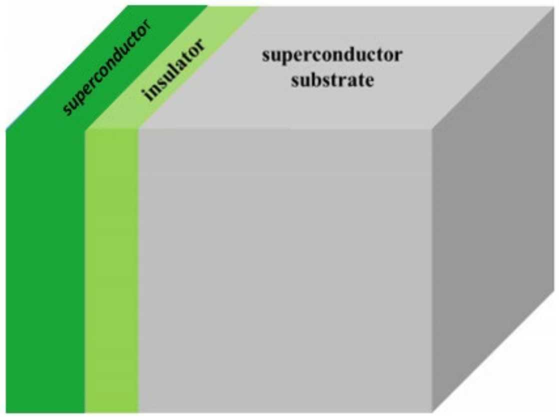


T.Kubo 2017 Supercond. Sci. Technol. [2]

Motivation: S-I-S Multilayers

SUPERCONDUCTOR
Nb compounds: Nb_3Sn , Nb nitrides (NbN and NbTiN)
Others: MgB_2

Tc [K] : Nb=9.23 ; NbN=16.2 ; NbTiN=17.23
excellent superconducting properties of NbN with the good metallic and structural properties of TiN



INSULATOR
Al_2O_3
AlN
Nb_2O_5
MgO

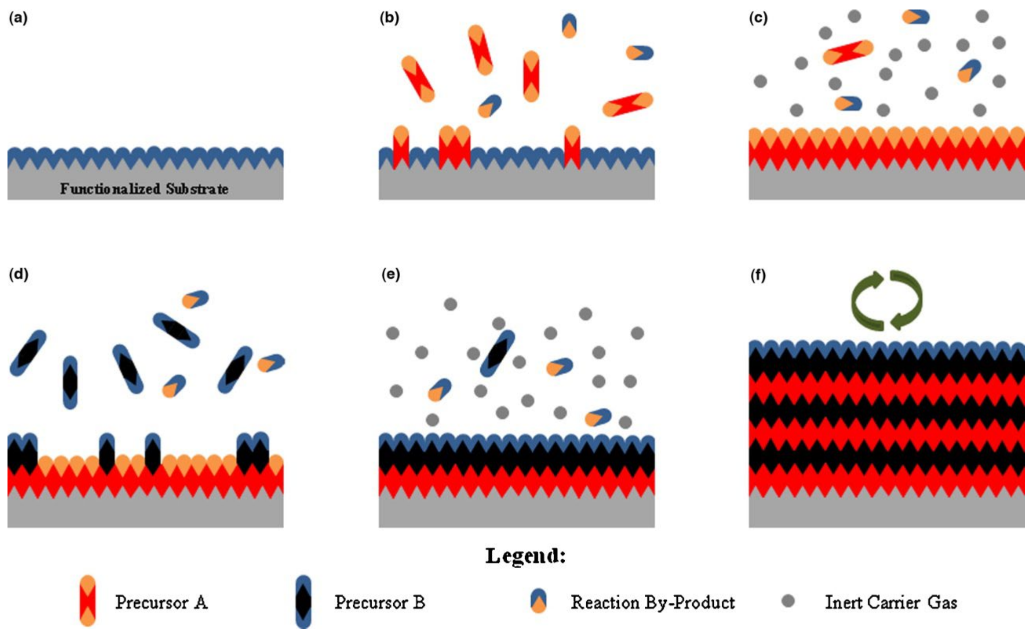
AlN as buffer layer improves the NbTiN superconducting properties

Deposition Techniques
- High power impulse magnetron sputtering
- High Temperature Chemical Vapor Deposition
- Atomic Layer deposition (ALD)

Atomic layer deposition (ALD)

ALD is a sequential technique based on the self-limiting reactions between gases and solids

- ✓ Exceptional conformality (also on high-aspect ratio structures)
- ✓ Precise thickness control (constant growth per cycle (GPC))
- ✓ Homogeneity (pinhole-free)
- ✓ Small film roughness
- x Slow process
- x Not all stoichiometries are possible – correct precursors necessary



Thermal ALD vs Plasma Enhanced ALD (PEALD)

Thermal ALD

- **Metal chloride precursors** ($NbCl_5$ and $TiCl_4$) can **contaminate** the deposited film with chlorine
- NH_3 as nitrogen source is often **insufficient** as **reductant power to obtain stoichiometric metal nitrides**
- Requires **high ALD temperaturas** (>400 °C)

Plasma Enhanced ALD

- **Highly reactive radicals** produced by a plasma source act as a coreactant
- **Low ALD deposition temperatures** (<400 °C)
- Metallorganic precursors

Supercycle ALD Approach for AlN $Nb_xTi_{1-x}N$

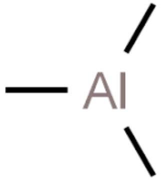
System

Arradiance GEMStar XT-P

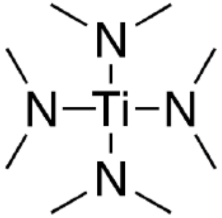
dry pump (Ebara A10s)
P_{base} = 4E-2 mbar
Ar (6.0) purge gas

Precursor

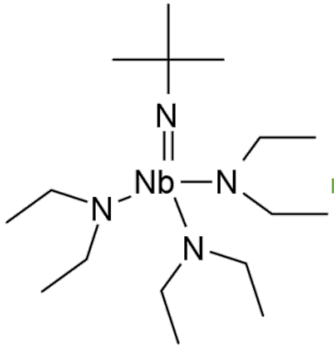
TMA



TDMAT



TBTDEN



Plasma

N_2/H_2-Plasma

Aim

Supercycle ALD

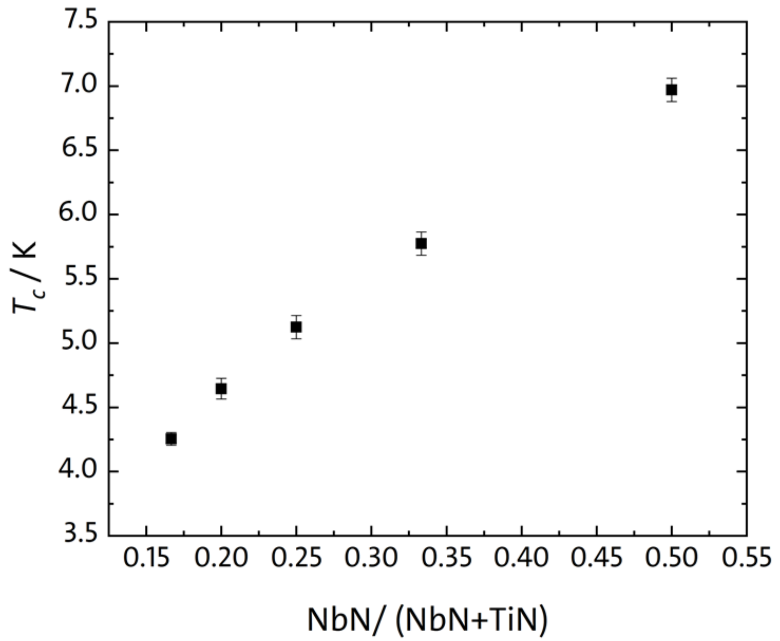
AlN

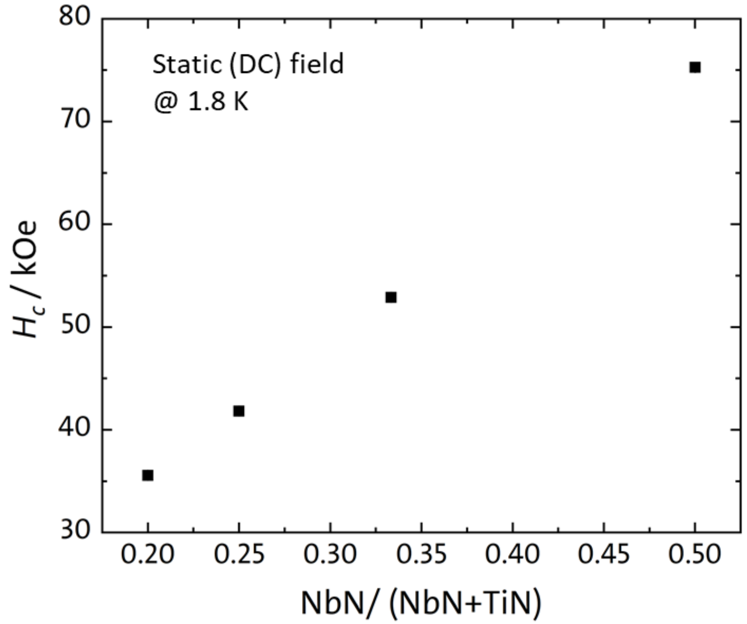
TiN

NbN

$Nb_xTi_{1-x}N$

Tuning the superconducting properties by varying ratio of NbN to TiN

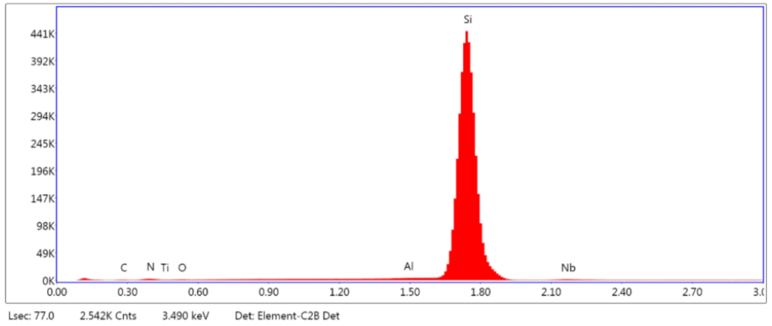




EDX Composition Analysis

TiN:NbN	At% Ti	At %Nb
5:1	0.69	0.15
4:1	0.64	0.15
3:1	0.68	0.2
2:1	0.58	0.23
1:1	0.34	0.33
1:2	0.22	0.41

Increasing Nb-content in thin films → increase in T_c and H_c



CHyN
Center for Hybrid Nanostructures

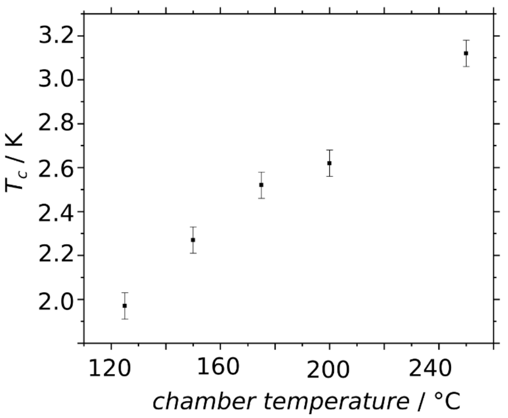
DESY.
Universität Hamburg
DER FORSCHUNG | DER LEHRE | DER BILDUNG

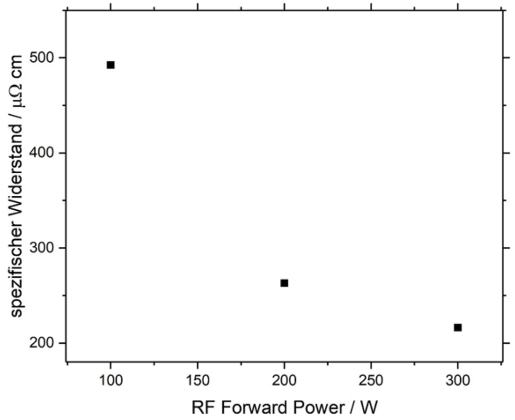
Process Optimization of individual binary processes

The deposition process has been optimized in order to improve the superconducting properties of NbTiN thin films

- Deposition Temperature
  - → @250°C Limitation of process
- Plasma Parameters
  1) RF Forward Power
  - → @300W Limitation of setup

TiN





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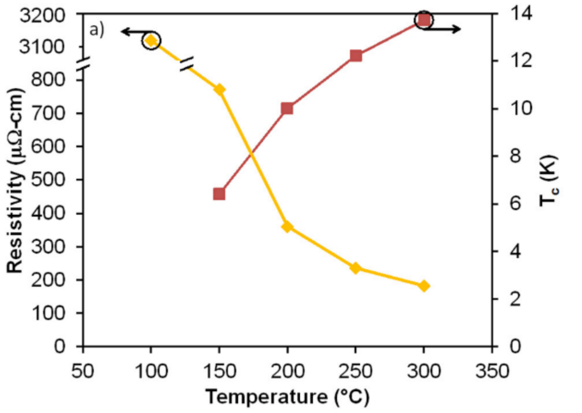
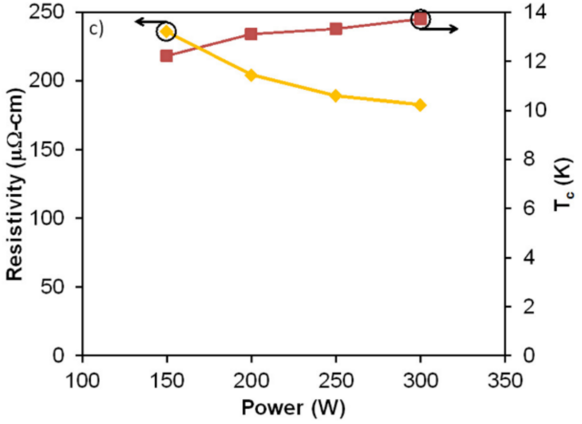
NbN

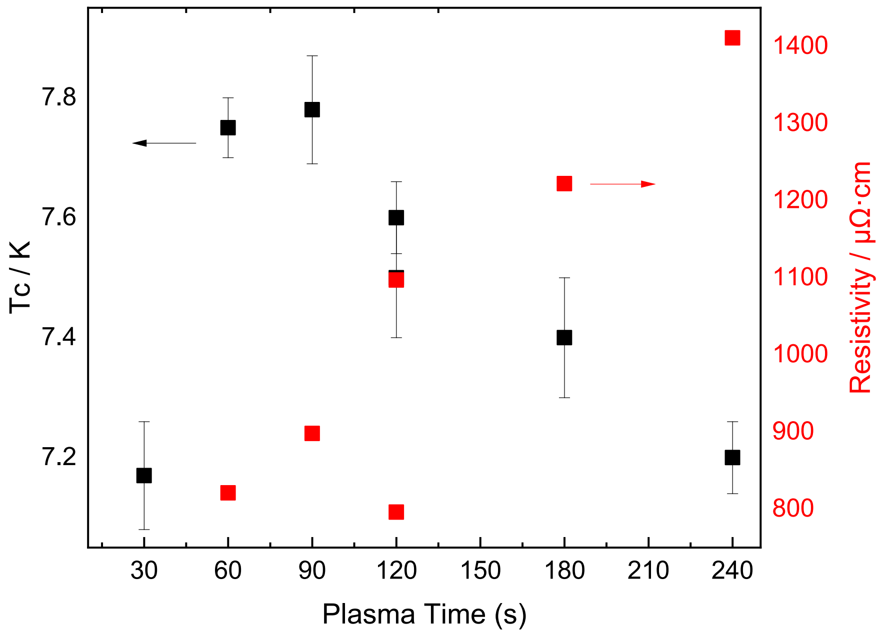




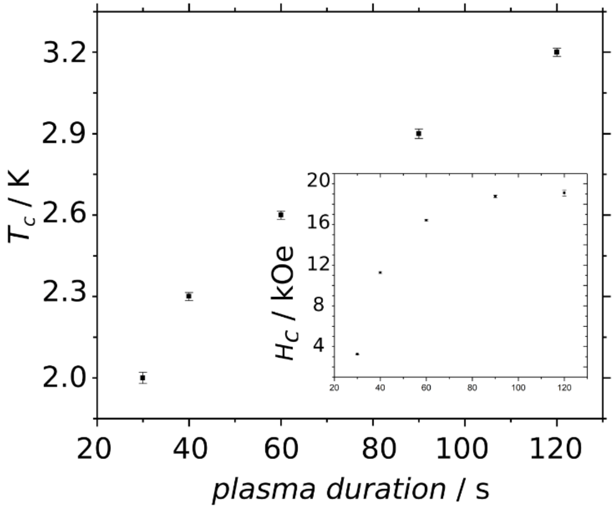
Sowa, Mark J., Yemane, Yonas, Zhang et al. [3]

Process Optimization of individual binary processes

- Plasma Parameters
  2) Plasma Dose Time



TiN



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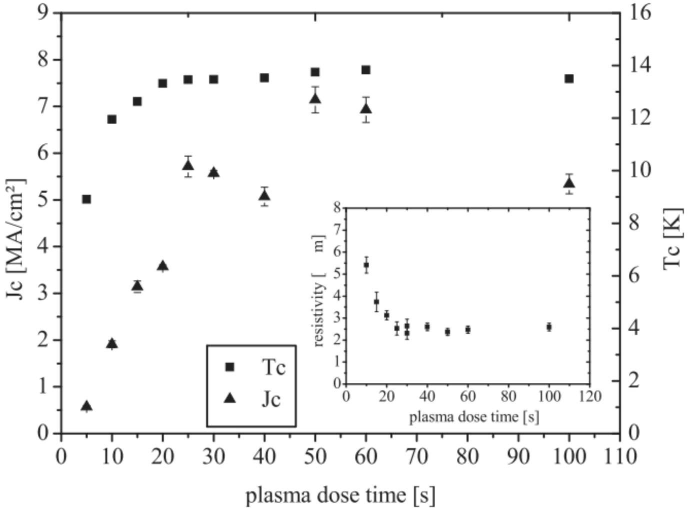
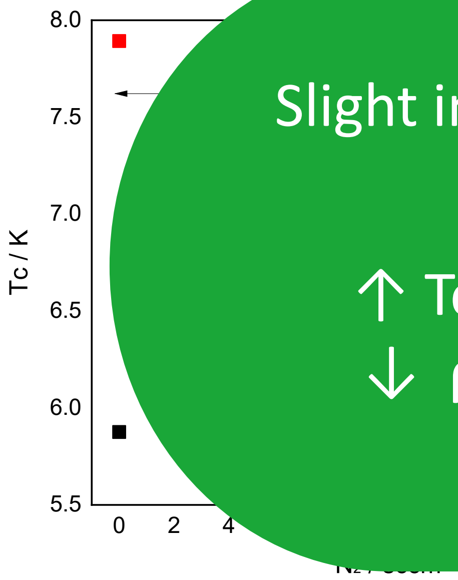
NbN



M. Ziegler, S.Linzen, S. Goerke et al. [4]

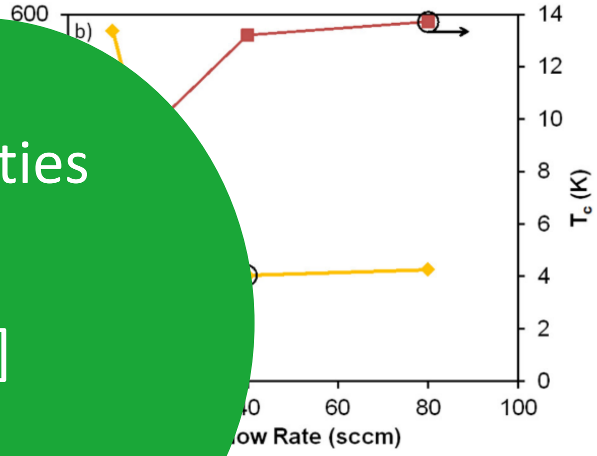
Process Optimization of individual binary processes

- Plasma Parameters
  3) Gases Flow: H_2 & $N_2 \rightarrow H_2$=45 ; N_2=12



TiN

NbN



…J., Yemane, Yonas, Zhang et al. [4]

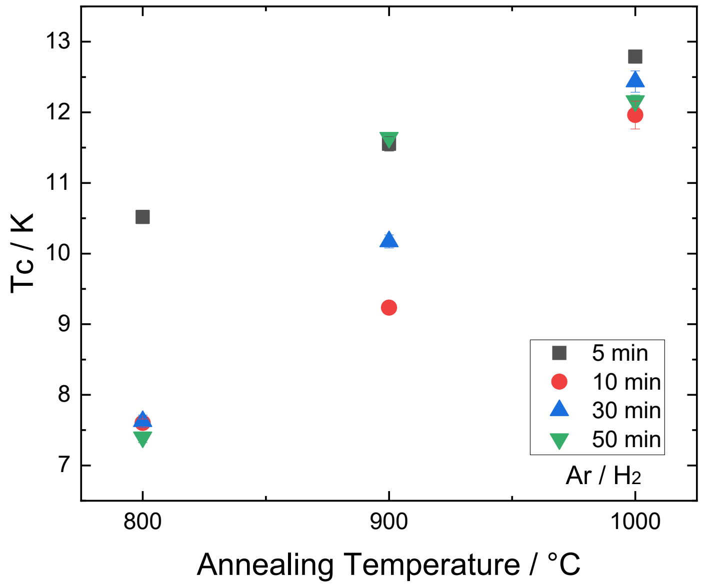
Slight improvement in the superconducting properties optimizing $Nb_{0.66}Ti_{0.33}N$ deposition

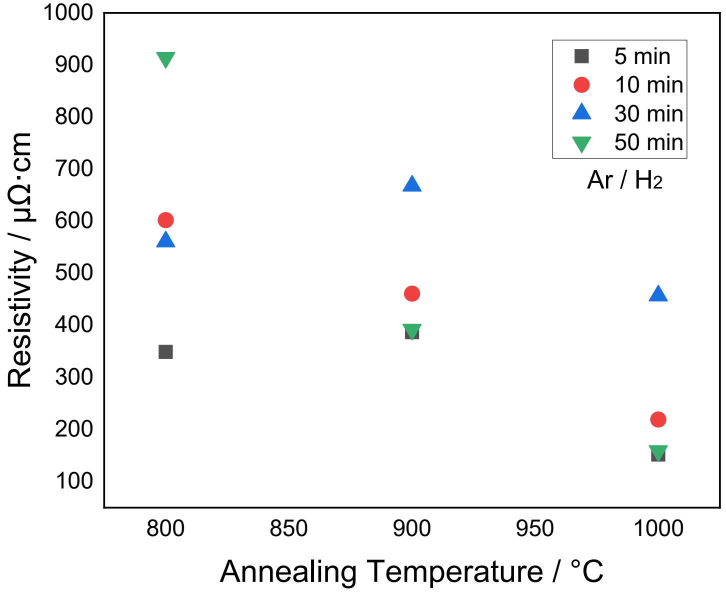
↑ Tc 10% **Before:** Tc= 7.03 [K]; ρ =1268 [μΩ·cm]
↓ ρ 30% **After:** Tc = 7.78 [K] ; ρ = 898 [μΩ·cm]

- **Pressure:** decrease the base pressure improves the film quality

Rapid Thermal Annealing

Influence of temperature and time of the annealing process in the transport properties of the $Nb_{0.66}Ti_{0.33}N$ films





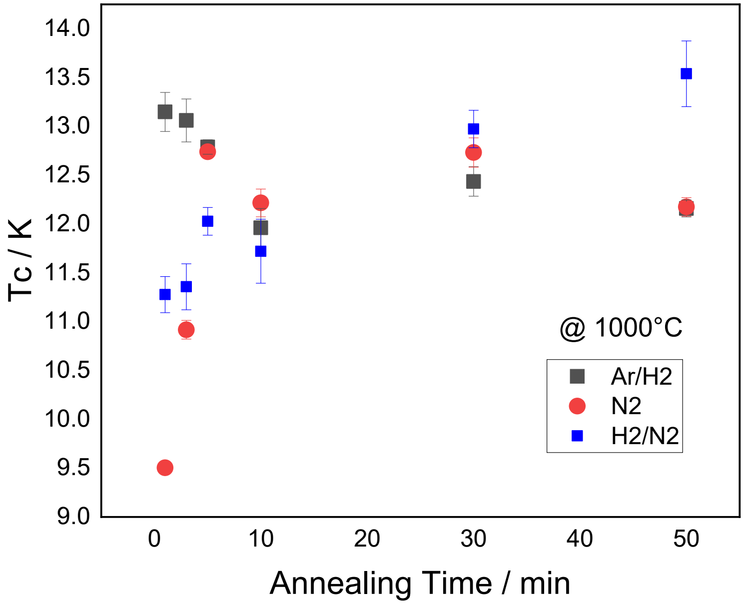
Tc and resistivity were improved by RTA.

Rapid Thermal Annealing

Influence of the gas atmosphere in the transport properties the $Nb_{0.66}Ti_{0.33}N$ films

- Different annealing atmospheres :

1. Ar/H_2 mixture (95% of Ar and 5% of H_2)
2. N_2
3. N_2/H_2 mixture (85% of N_2 and 15% of H_2)



Maximum Tc reached **Tc= 13,5 K**

- Investigation different RTA atmospheres

5 min	Resistivity / µΩ·cm	Tc
Ar/H2	152	13,05
N2	198	12,7
H2(15%)/N2(85%)	186	12,03
H2(33,3%)/N2(66,6%)	302	11

1. After the first RTA, a 2nd thermal treatment (without vacuum break) in pure N_2 atmosphere has been performed at 1000°C. The results showed that Tc has been increased.
   The best result is **Tc= 13,93 K ; ρ = 132 µΩ·cm**

Further studies are needed to establish best recipe

Summary + Next Steps

WORK UNTIL NOW

✓ PE-ALD NbTiN deposition process has been optimized for T_c and resistivity, still insufficient.

✓ Post-deposition thermal annealing has been performed to investigate the effect on the films and different temperatures, annealing times and gas atmospheres have been studied.

As deposited Tc=7.78 K $\rightarrow$ After RTA Tc=13.93K

NEXT STEPS

- Lattice characterization, using XRR/XRD/EBSD/PALS. The aim is to fully understand the effect of the RTA
- Analyze the H_2 concentration using EMGA
- SRF measurements to obtain H_{c1} and the superconducting gap Δ

References

[1]Alex Gurevich, Appl. Phys. Lett. 88, 012511 (2006)

[2]Takayuki Kubo 2017 Supercond. Sci. Technol. 30 023001

[3] Sowa, Mark J., Yemane, Yonas, Zhang et al. , Plasma-enhanced atomic layer deposition of superconducting niobium nitride, Journal of Vacuum Science & Technology A 35, 01B143 (2017)

[4] M. Ziegler, S.Linzen, S. Goerke et al. , Effects of Plasma Parameter on Morphological and Electrical Properties of Superconducting Nb-N Deposited by MO-PEALD, IEEE Transactions on Applied Superconductivity (Volume: 27, Issue: 7, Oct. 2017)

THANK YOU FOR YOUR ATTENTION!

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Backup

Material	T_c [K]	ρ_n(μΩcm)	$H_c(0)$ [mT]	$H_{c1}(0)$ [T]	$H_{c2}(0)$ [T]	H_{SH} [T]	λ(0) [nm]	Δ [meV]	ξ [nm]	Type
Nb	9.23	2	200	0.17	0.28	0.219	40	1.5	28	II
Pb	7.2		80	N/A	N/A		48			I
NbN	16.2	70	230	0.02	15	0.214	200-350	2.6	<5	II, B1 comp.
NbTiN	17.3	35		0.03			150-200		<5	II, B1 comp.
Nb_3Sn	18	20	540	0.05	30	0.425	80-100	3.1	<5	II, A15
V_3Si	17	4	720	0.072	24.5		179	2.5	<5	II, A15
Mo_3Re	15	10-30	430	0.03	3.5	0.17	140			II, A15
MgB_2	40	0.1-10	430	0.03	3.5-60	0.17	140	2.3/7.2	2.3/7.2	II- 2 gaps
2H-$NbSe_2$	7.1	68	120	0.013	2.7-15	0.095	100-160		8-10	II-2gaps
YBCO	93		1400	0.01	100	1.05	150	20	0.03/2	d-wave
Pnictides	30-55		500-900	0.03	>100	0.756	200	10-20	2	s/d wave

Supercycle ALD Approach for $Nb_xTi_{1-x}N$

Aim: Tuning the superconducting properties of the deposited thin by varying ratio of NbN to TiN