Surface engineering by Atomic Layer Deposition for SRF cavities

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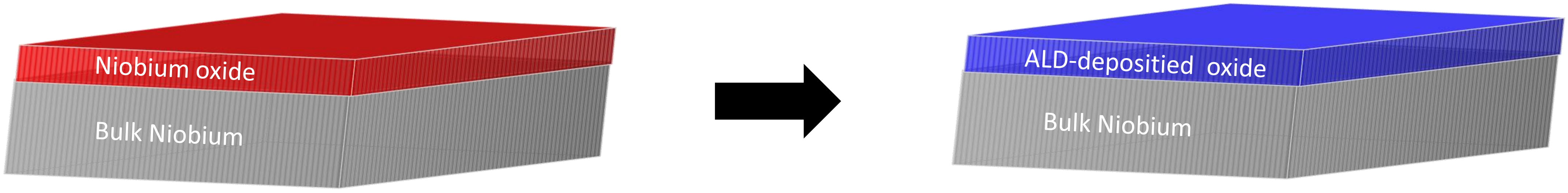
Outline

- At CEA, we are trying to improve the performances of niobium cavities by tayloring their inner surface using the technique of atomic layer deposition:

I. Changing the nature of oxide layer and studying their impact on high and low field performances.



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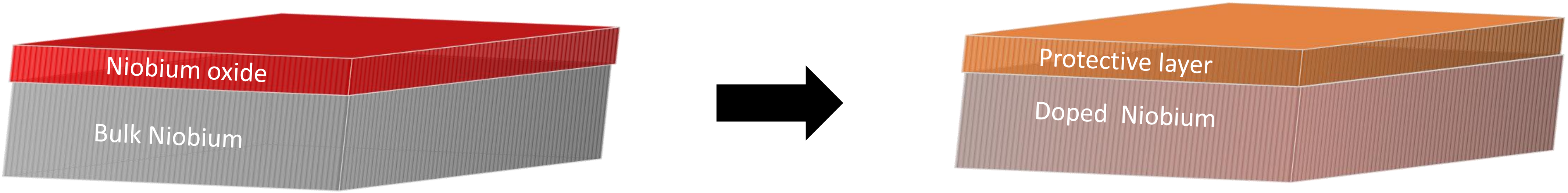

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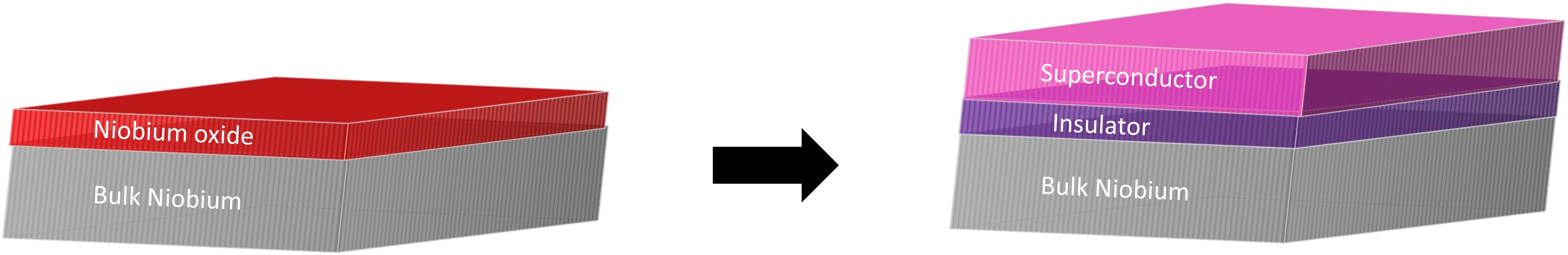
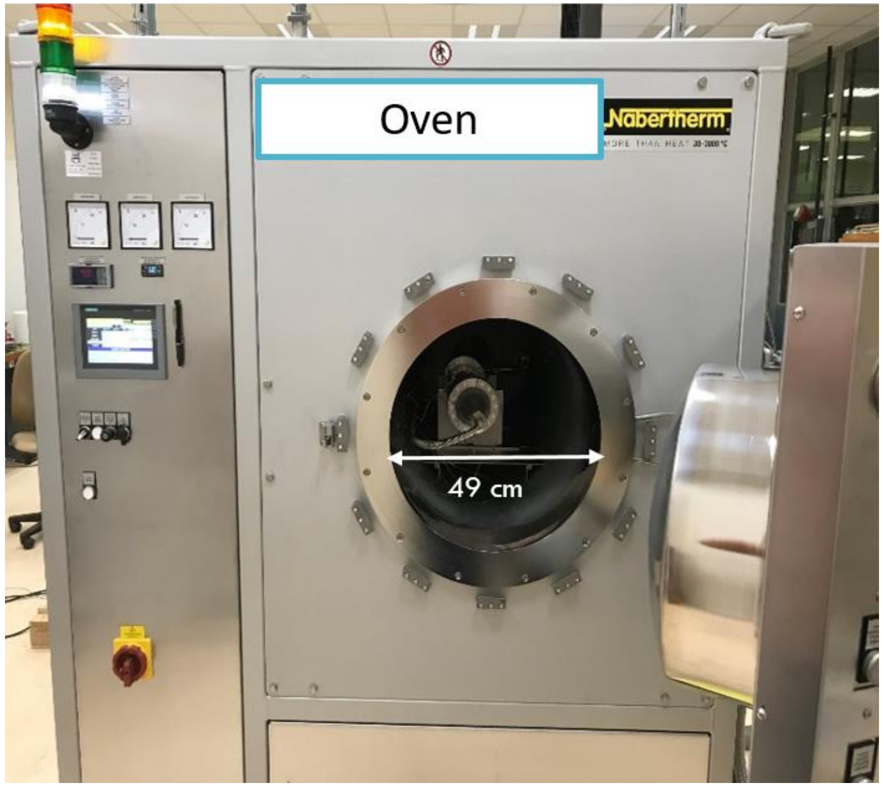
I. Changing the nature of oxide layer and studying their impact on high and low field performances.
II. Doping Niobium cavities.
III. Using a multilayer structure to screen the magnetic field seen by Niobium.

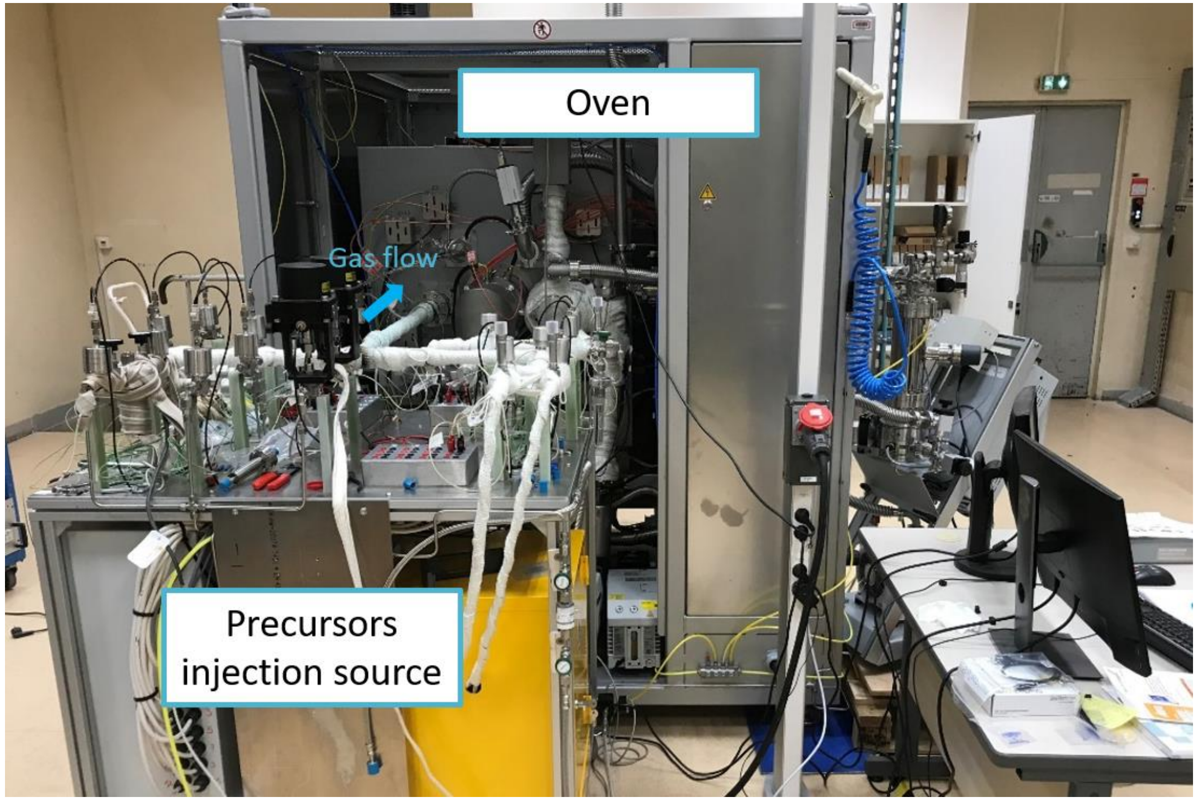


ALD system for cavity coating at CEA

Front



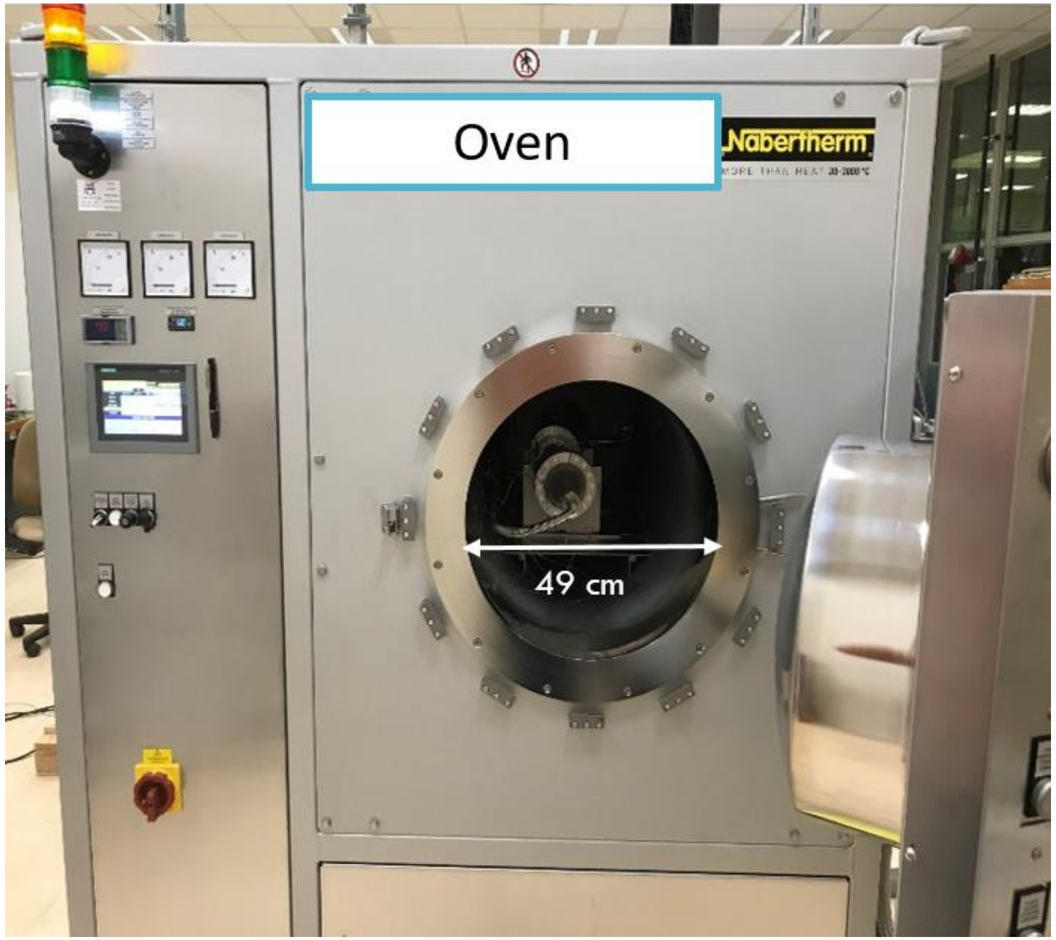
Back

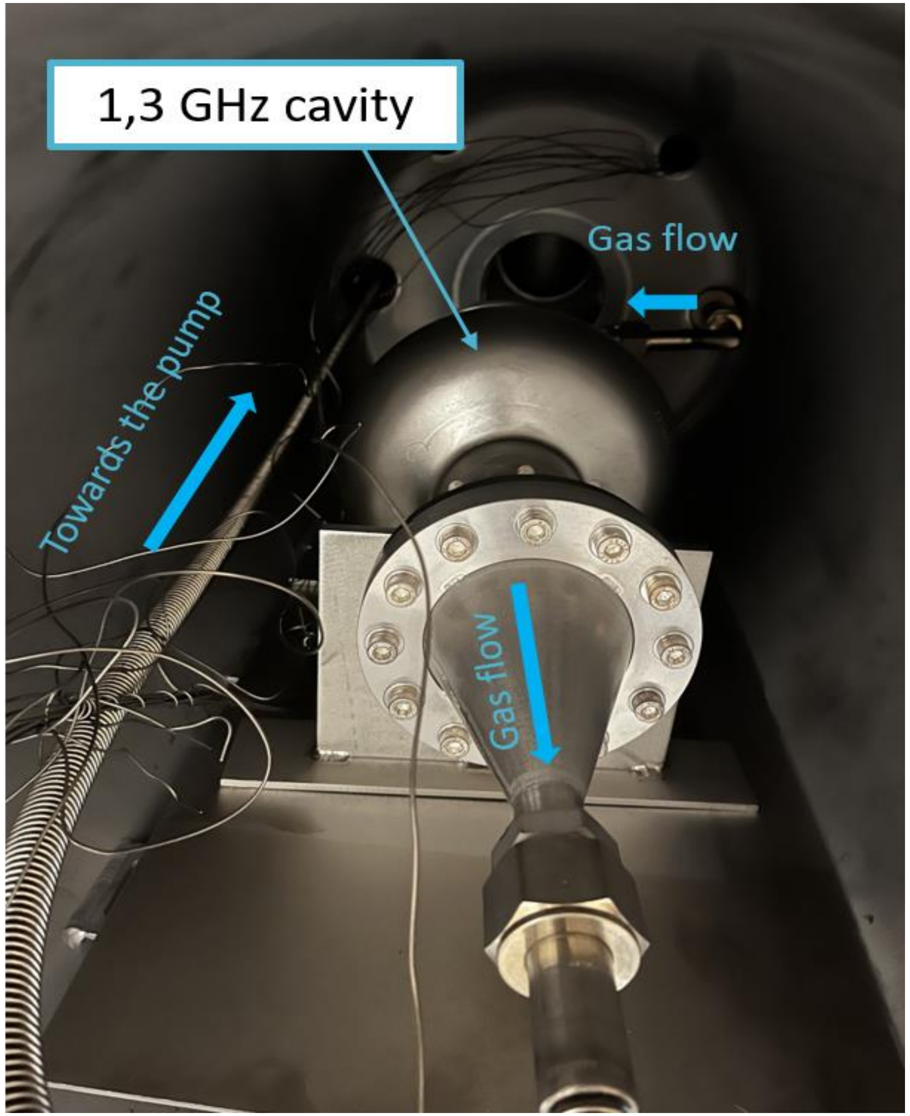


- <u>High vacuum oven</u>:
  - 650°C – 10^{-6} mbar / 900°C 1bar N_2
  - Volume retort: Φ = 49 cm, L= 110 cm (1.3, 0.7 GHz cavities)
- <u>ALD system</u>:
  - 9 precursor lines (2 gases, 2 liquids, 4 solids, 1 Ultra high temp).
  - RGA synthesis monitoring.

- <u>Interface and control:</u>
  - Labview program of ALD system and Oven.
  - Automatic synthesis parameter control (overnight dep.) and monitoring.

ALD system for cavity coating at CEA





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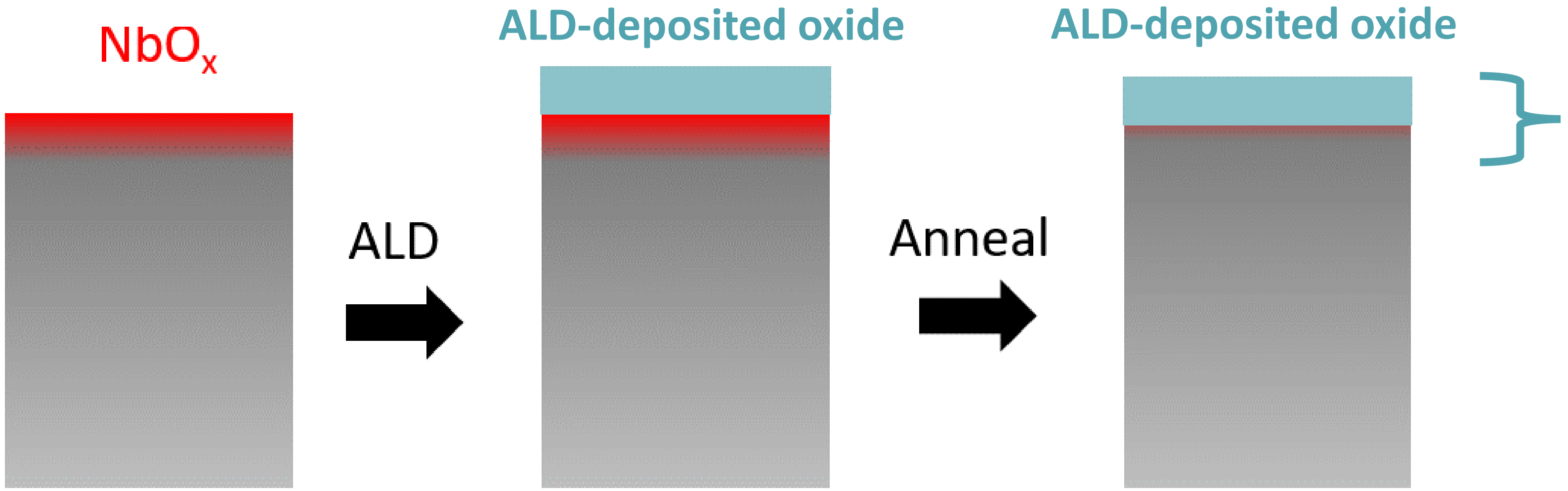
Part I:

Enhancement of niobium superconductivity through the use of ALD-oxides

To replace niobium native oxides with ALD-deposited protective layer [1]

1) Deposit ~ 10 nm oxide layer by ALD (Al_2O_3 , Y_2O_3 and MgO) onto Niobium.
2) Perfom a subsequent thermal treatement to dissolve niobium native oxide underneath (vacuum levels 10^{-6} mbar)

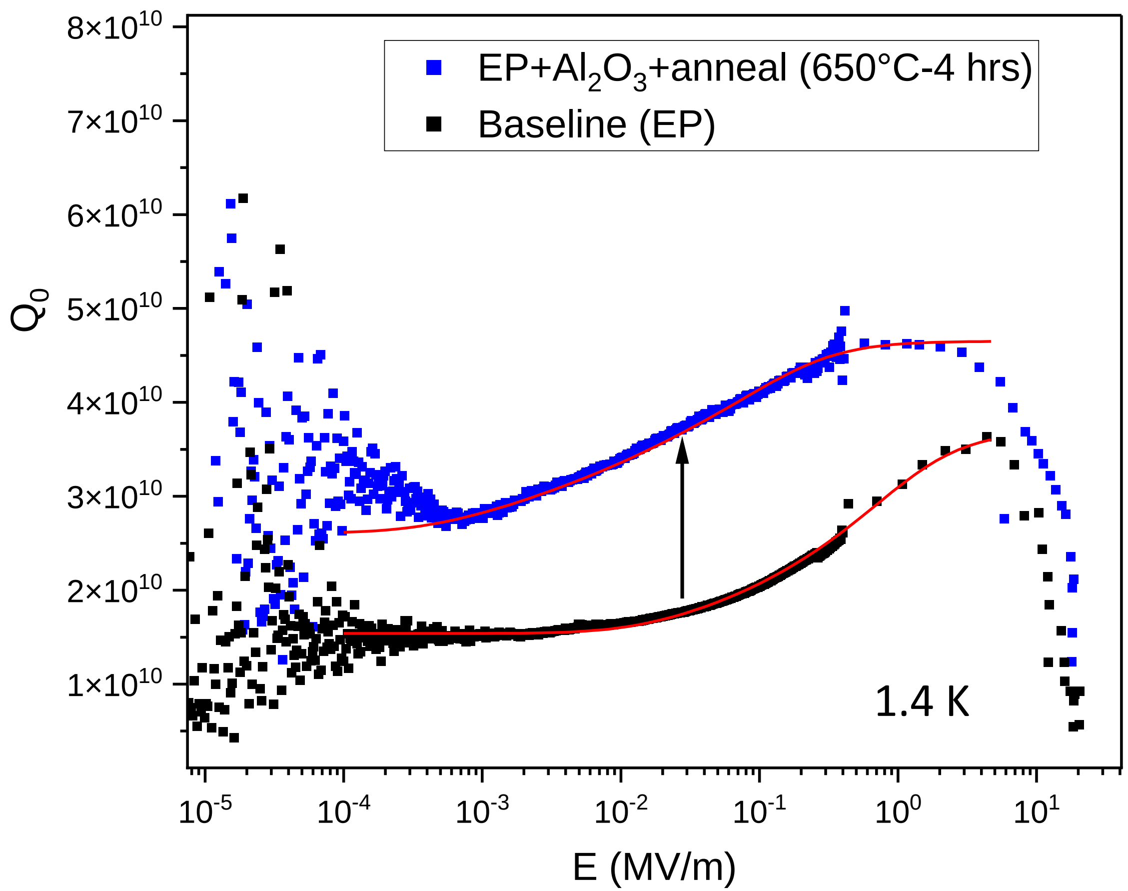


The oxide layer must be **thermally stable** and **have low dielectric** losses.

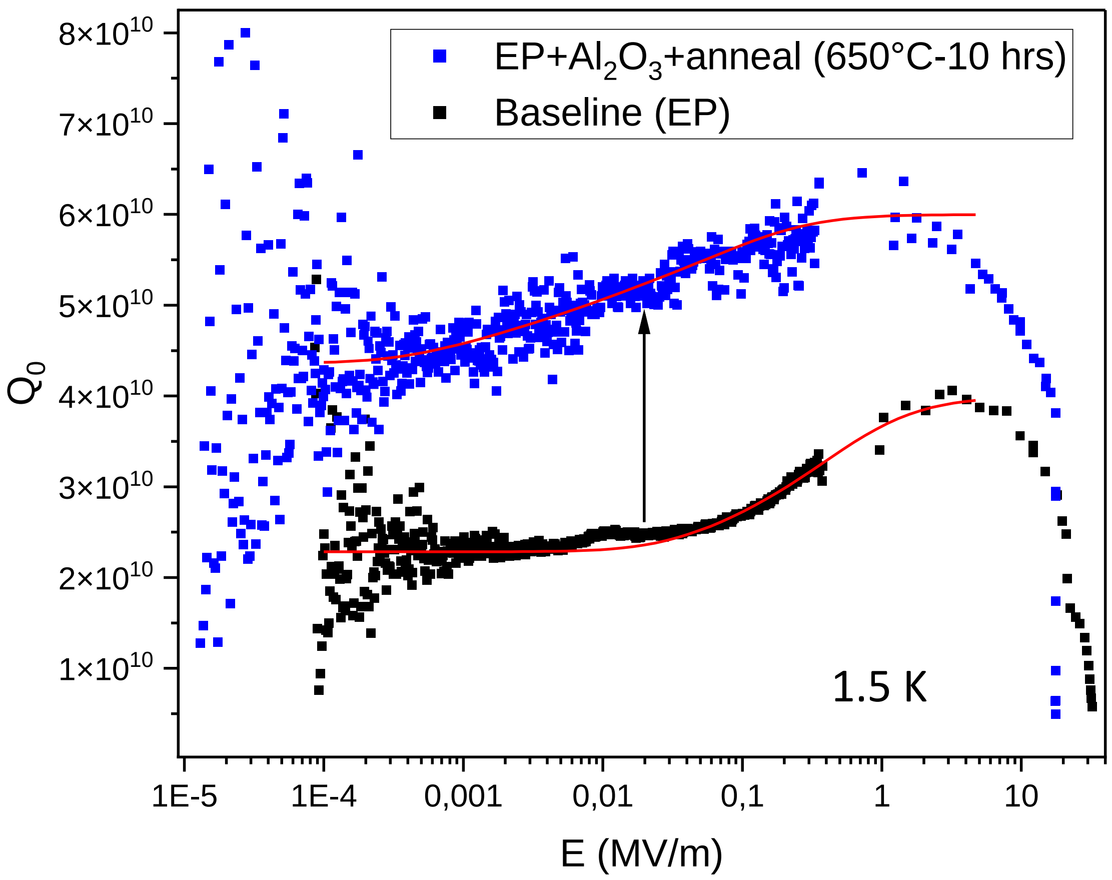
[1] T. Proslier et al . Improvement and protection of niobium surface superconductivity by atomic layer deposition and heat treatment. Applied Physics Letters, 93(19):192504, November 2008

Low field behaviour of the Al_2O_3 coated 1.3 GHz Nb cavities

First experiment



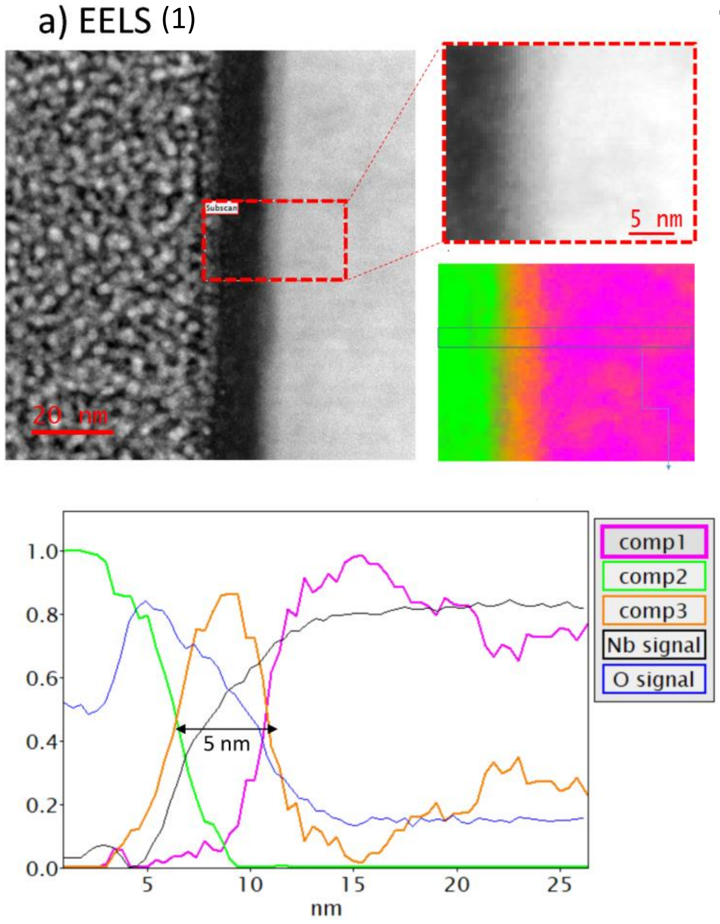
Second experiment

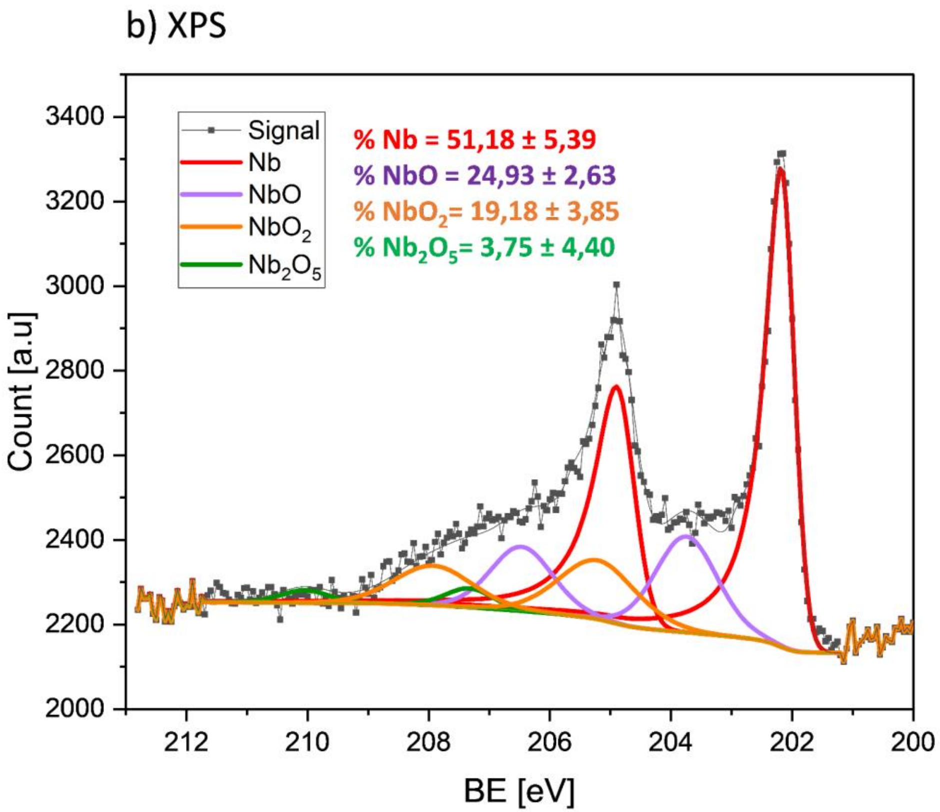


- The 10 nm Al_2O_3 film + annealing significantly improves the quality factors of the Nb cavity in the low field regime.

Chemical analysis of the Al_2O_3-Nb interface

As deposited





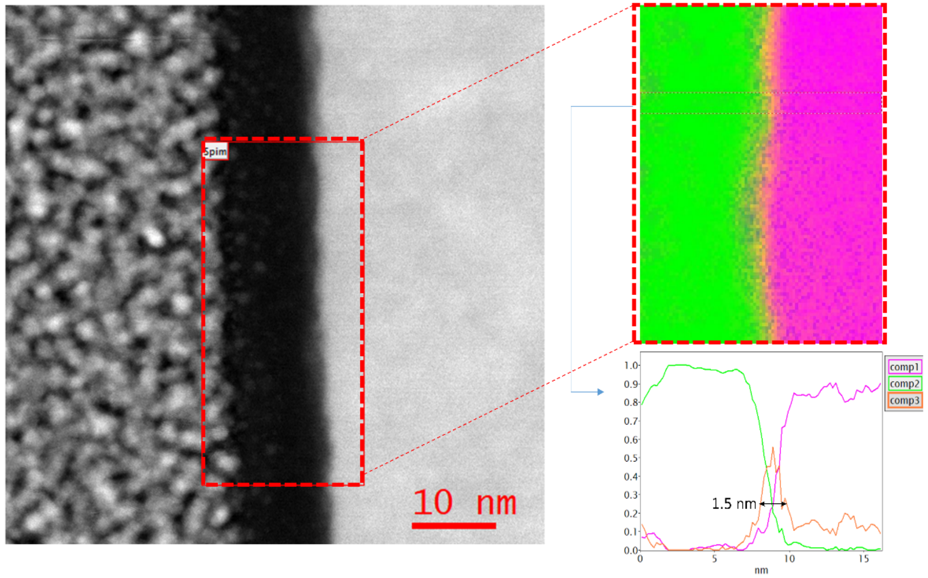
[1] Sarra Bira and Yasmine Kalboussi PhD Thesis

- We find a 5 nm thickness of NbO_x at the interface between the Al_2O_3 and Nb

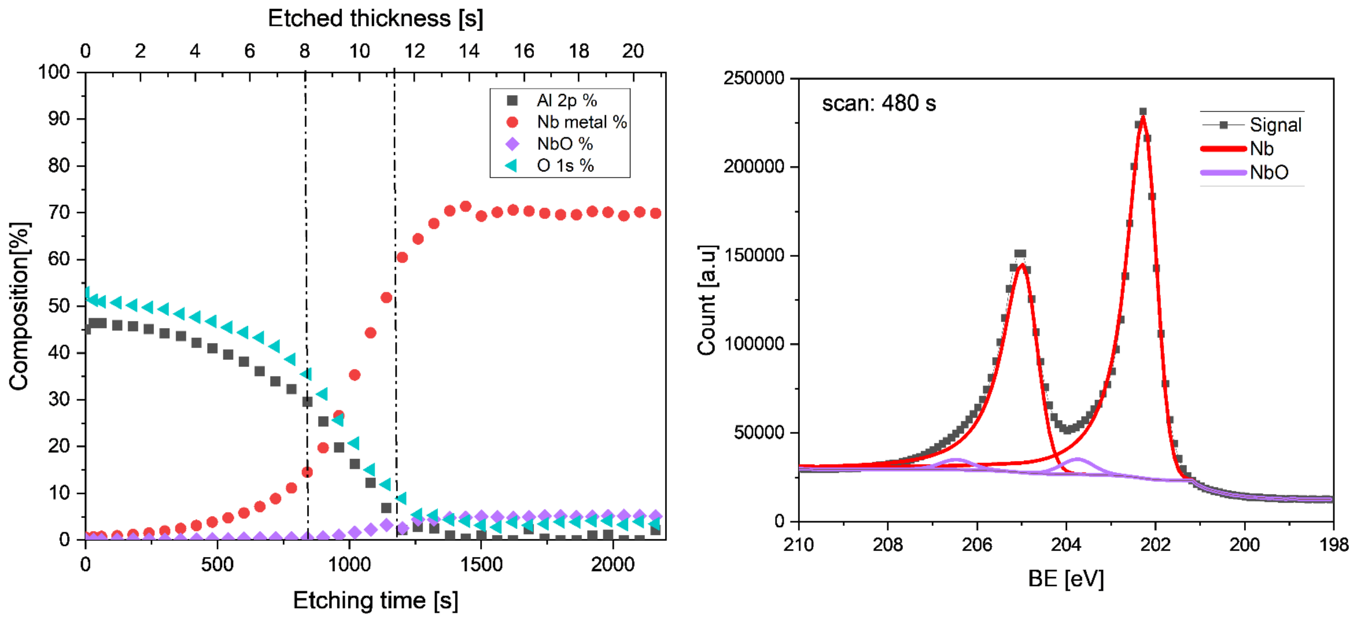
Chemical analysis of the Al_2O_3-Nb interface

After annealing 650°C-4 Hours

a) EELS (1)



b) XPS



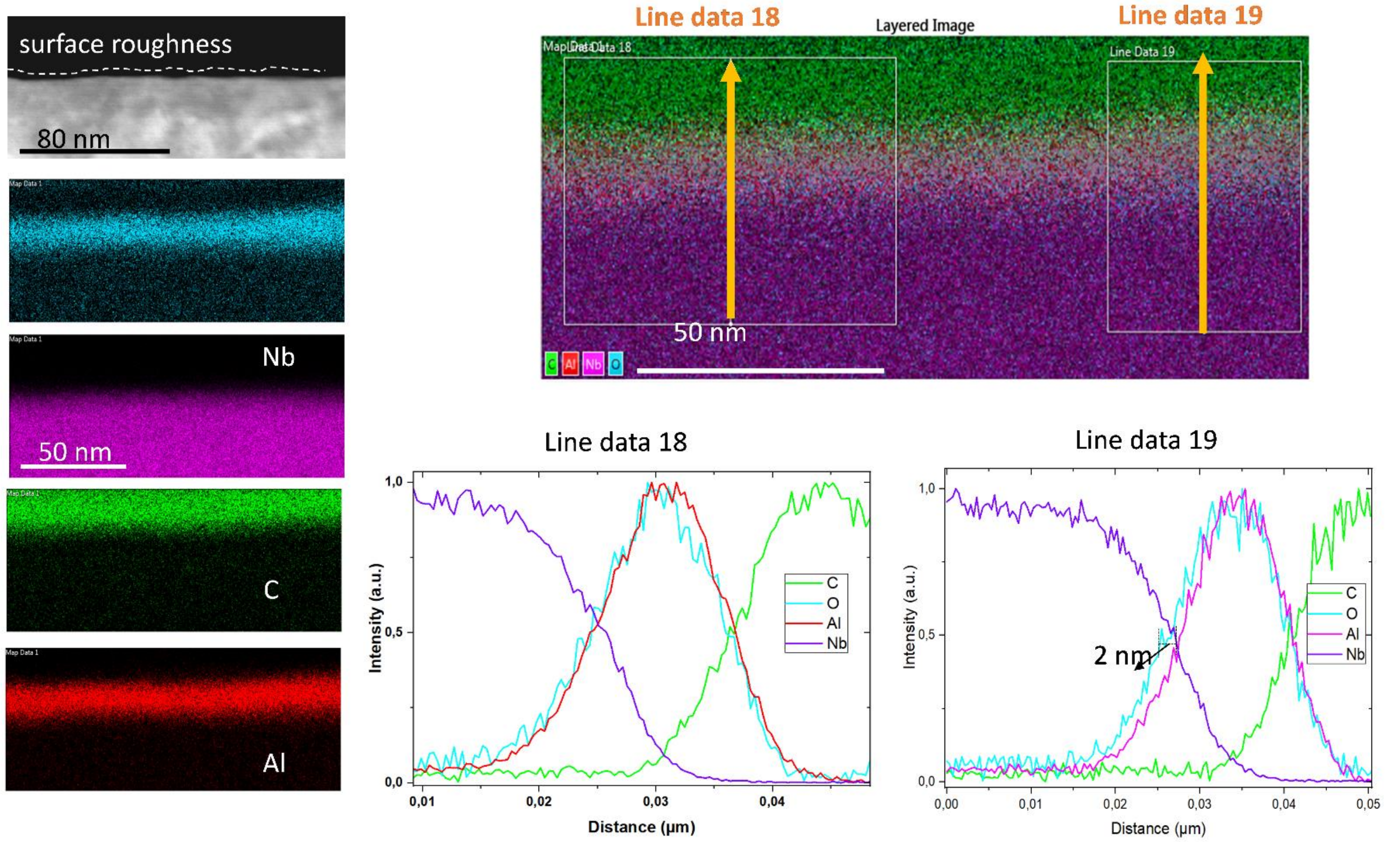
[1] Sarra Bira and Yasmine Kalboussi PhD Thesis

- After the annealing at 650°C- 4 hours, the thickness of NbO_x at the interface between the Al_2O_3 and Nb is reduced to 2 nm.

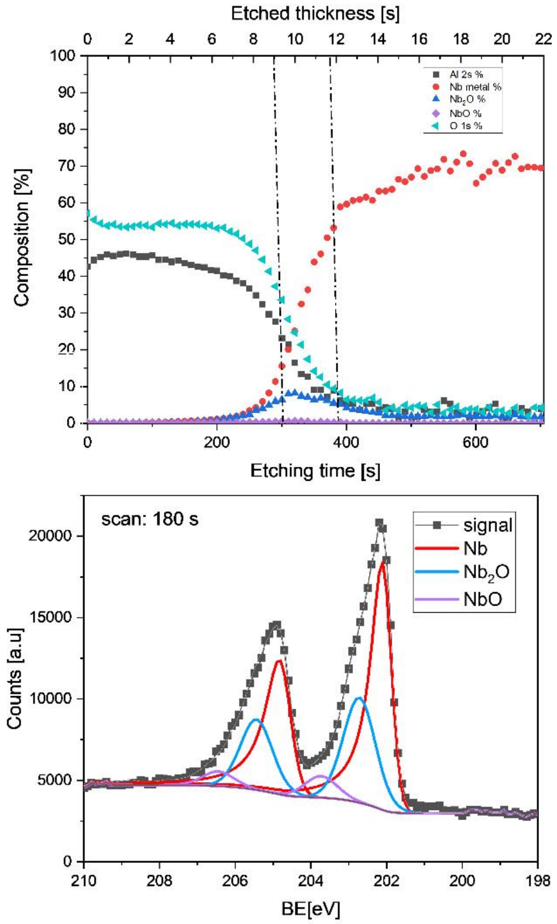
Chemical analysis of the Al_2O_3-Nb interface

After annealing 650°C-10 Hours

a) EDX



b) XPS

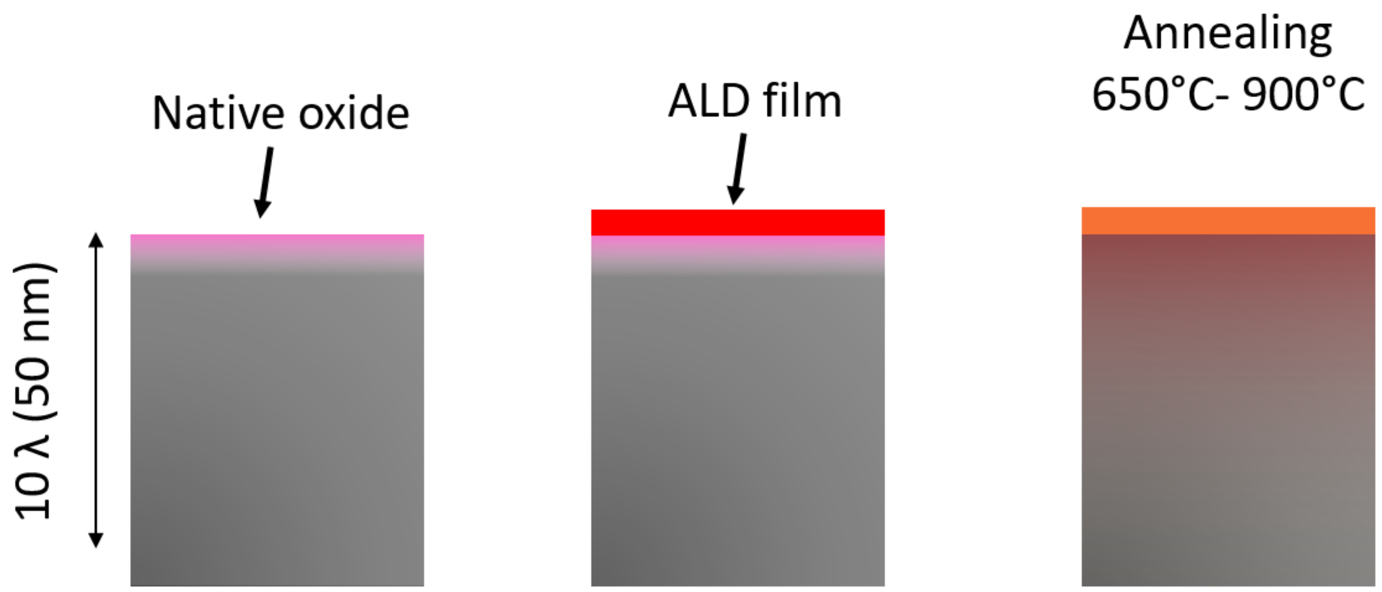


- After the annealing at 650°C- 10 hours, we witness further dissolution of NbO_x .

Part II:

Doping SRF cavities

ALD approach for doping cavities



ALD synthesis: NbN, TiN, ZrN, AlN, MgO, Al_2O_3, Y_2O_3 …

1) Well controled and uniform quantity of dopant.

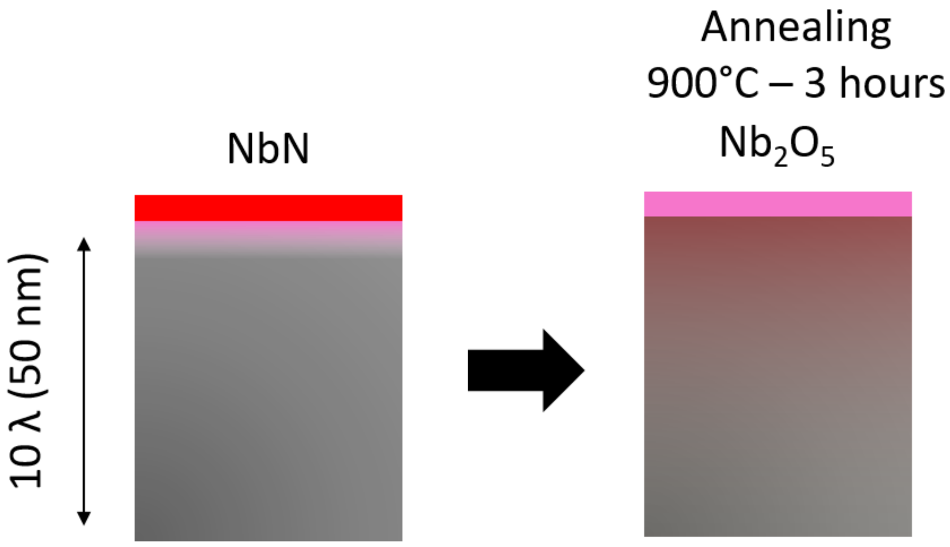
2) Induce O/N dopant in Nb but keep the metallic ions on the surface.

3) Avoid chemistry step ?

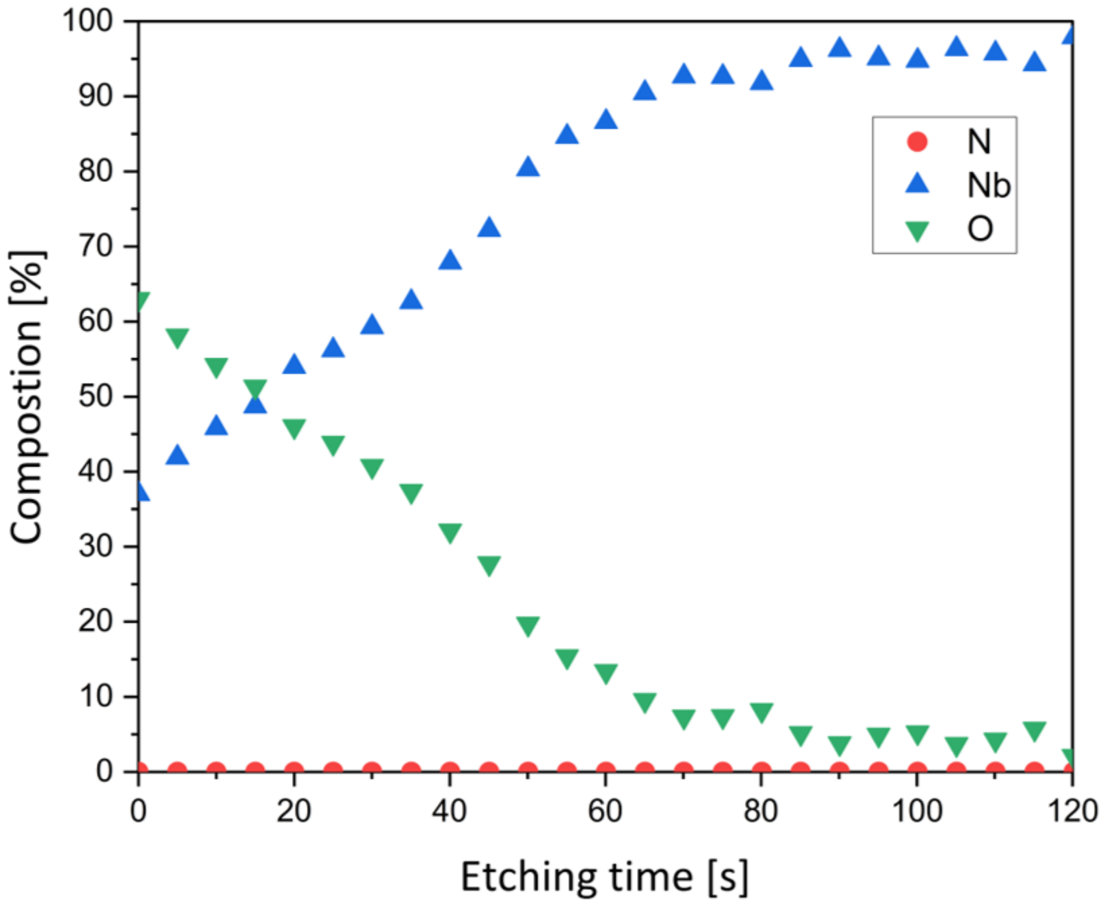
- We tested four nitrides layer: NbN, TiN, ZrN and AlN

Niobium nitride

- 5 nm of NbN + annealing 900°C- 3 Hrs - UHV



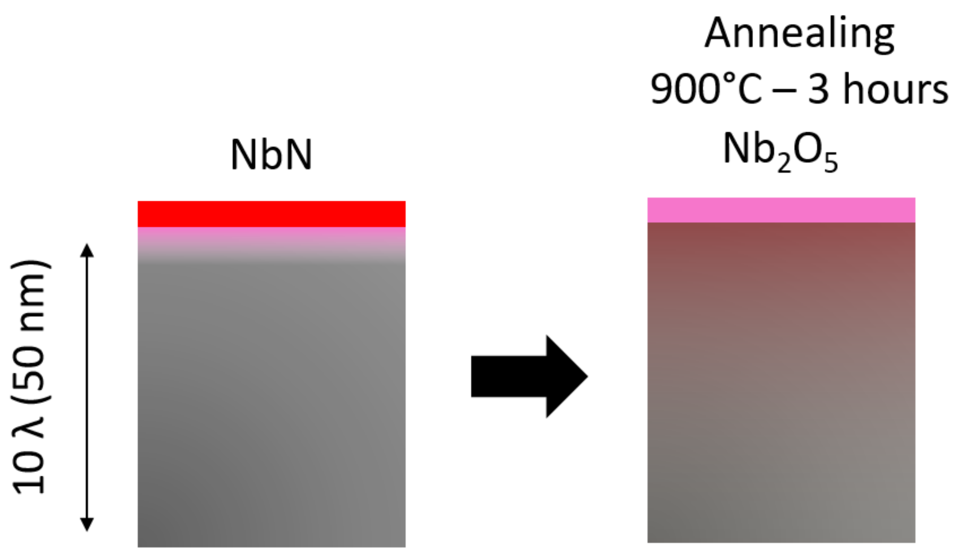
<u>XPS Profiling</u>

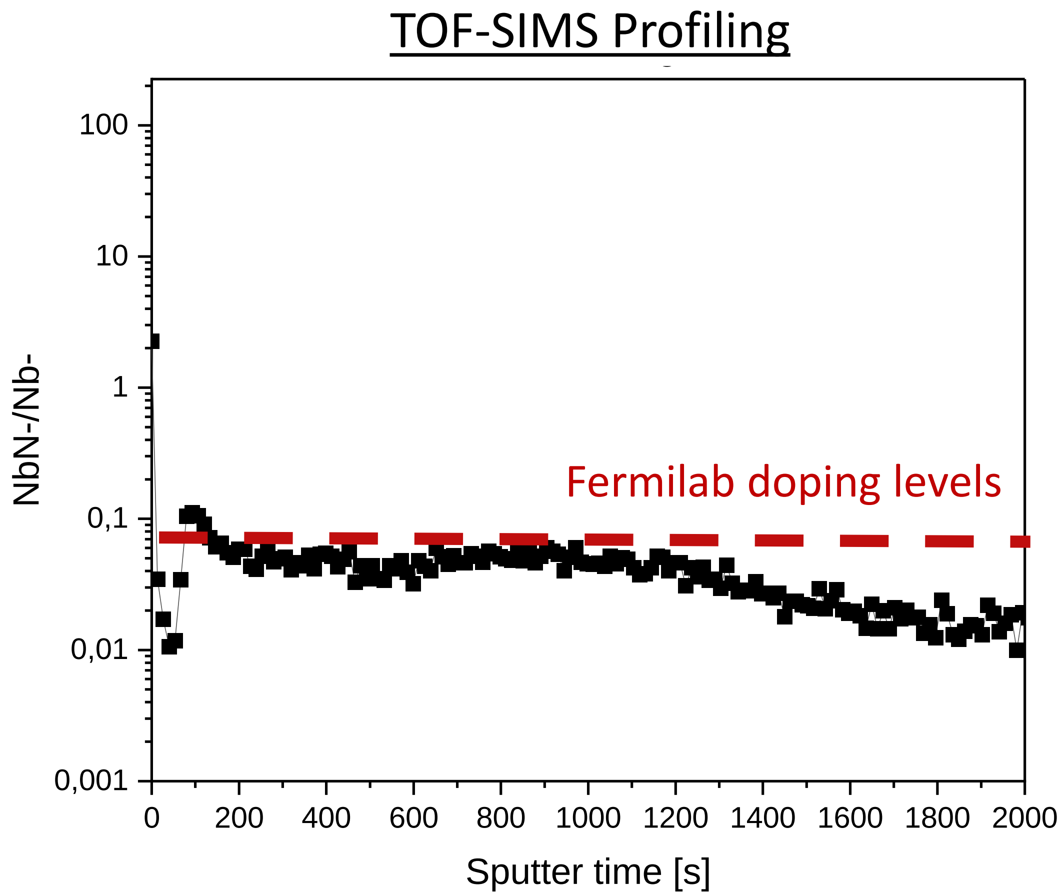


- No nitrogen detected by XPS at the surface.

Niobium nitride

- 5 nm of NbN + annealing 900°C- 3 Hrs - UHV

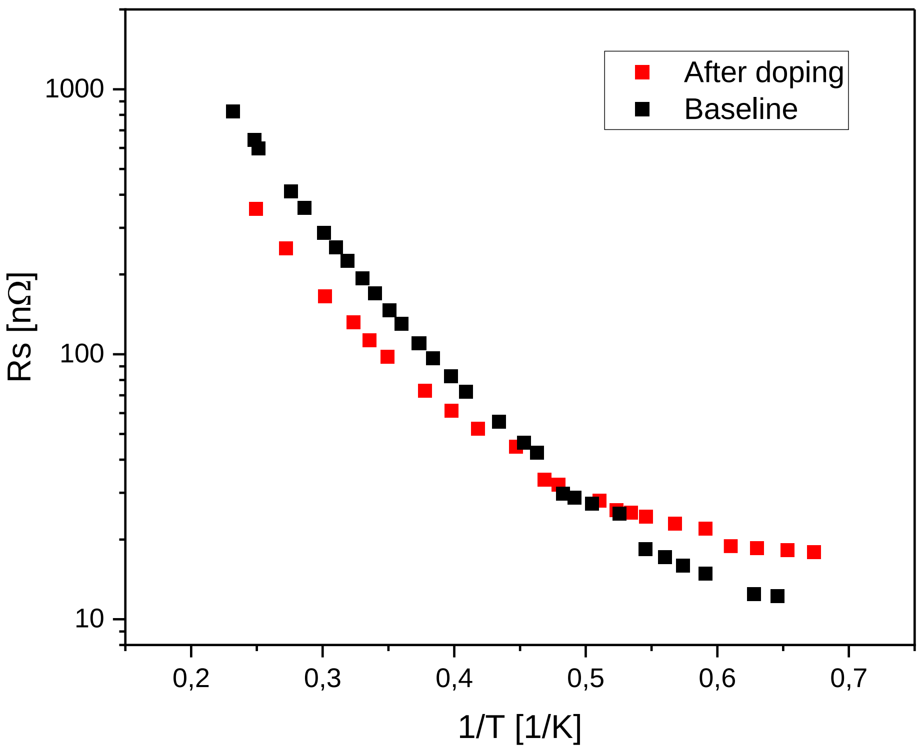


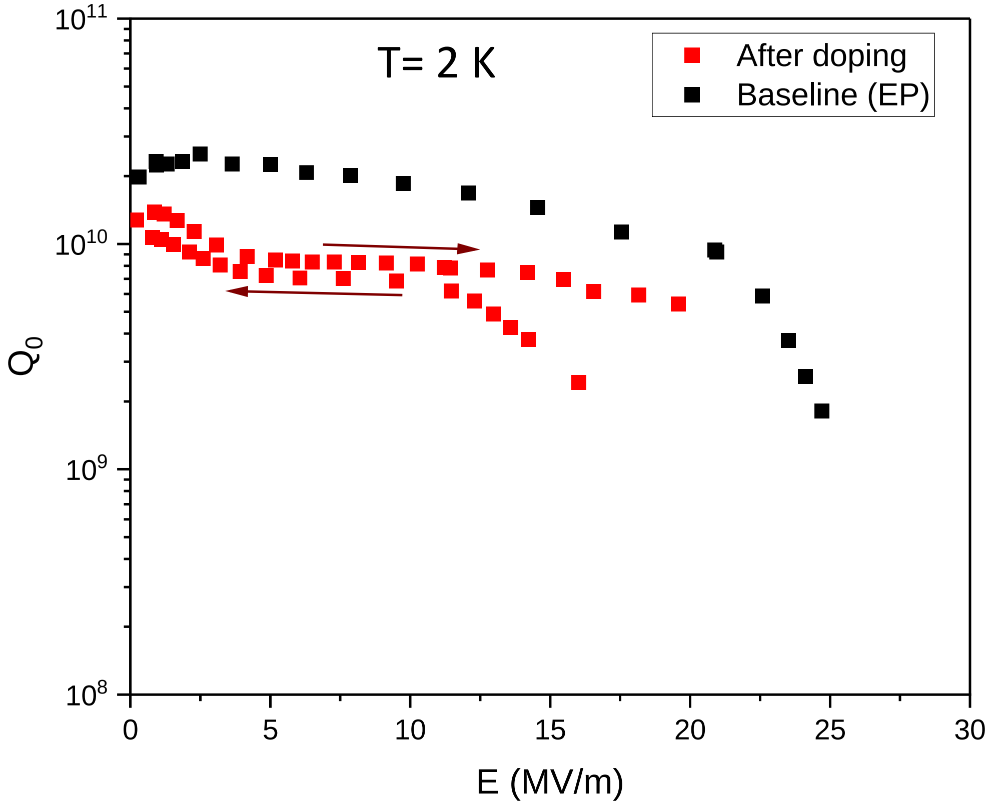


- Doping levels comparable to observed at Fermilab without electropolishing .

First test on 1.3 GHz Nb cavity

- The cavity was coated with 5 nm of NbN + annealing at 900°C-3 hours.
- No electro-polishing have been preformed.

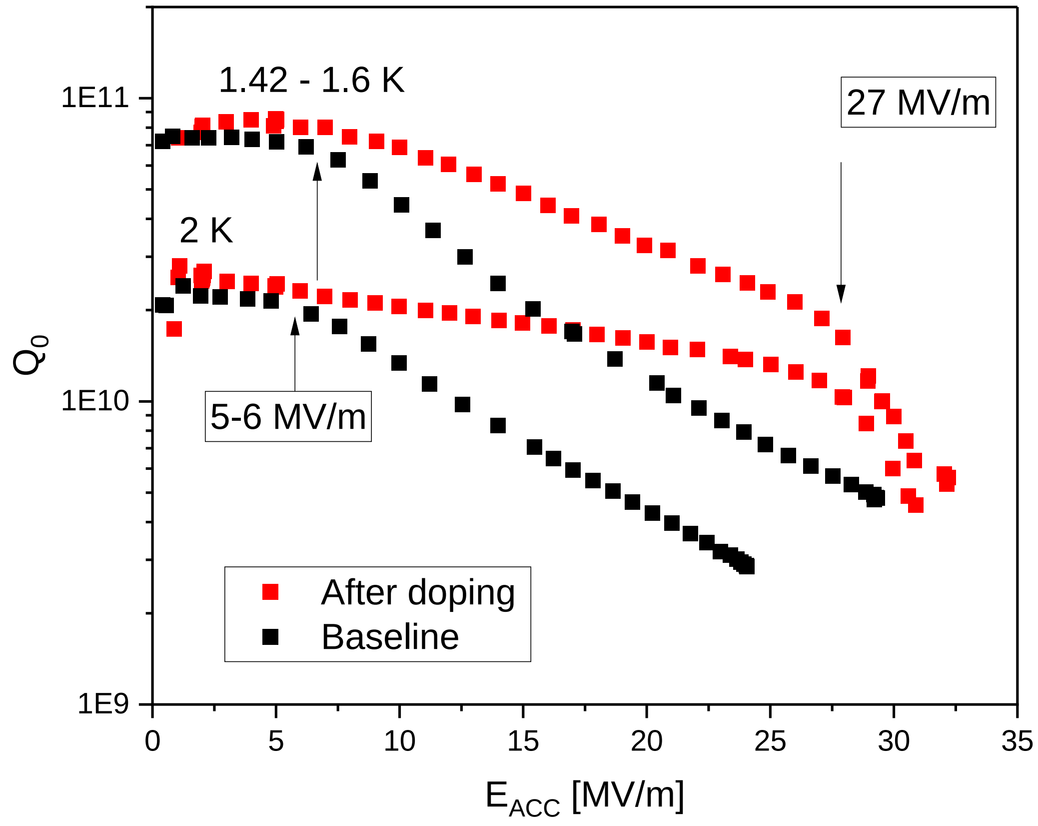




- The R_{BCS} is lowered but the residual resistance increased.
- The quality factor is higher at 4.2 K but lower at 2 K .

Second test on 1.3 GHz Niobium cavity

- The cavity was coated with 5 nm of NbN + annealing at 900°C-3 hours.
- No electro-polishing have been preformed.



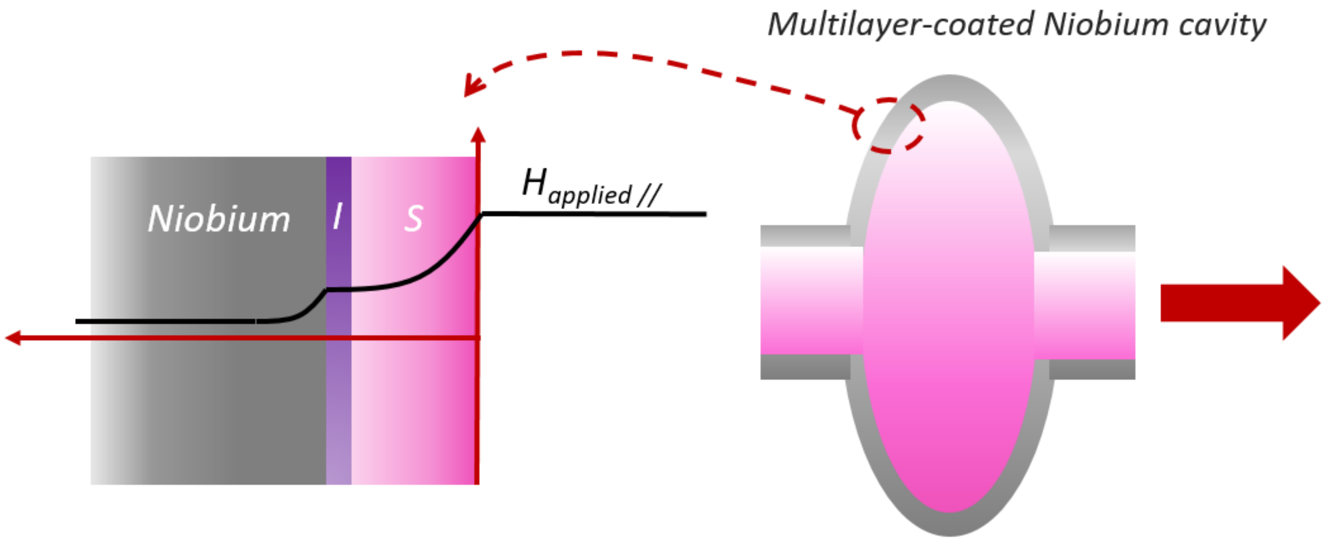
Thermal treatment and RF testing by Fermilab

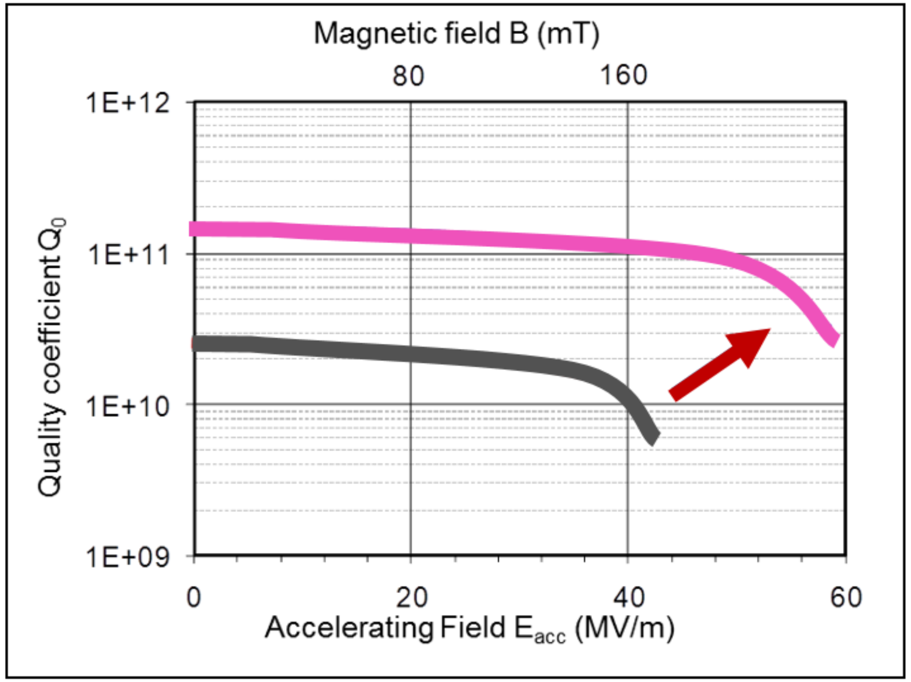
➢ The second test shows improvement over the baseline but not the typical doping performances.

Part III:

ALD-depositied multilayer to improve the superconducting performances of SRF cavities

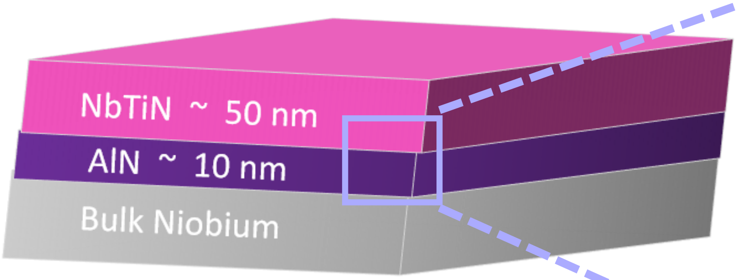
Multilayer structure





- A theoretical approach proposed by A. Gurevich (2006) to improve RF cavities through depositing a superconducting multilayer to screen the magnetic field.
- The thickness of the superconductor must be lower than its penetration depth.
- The superconducting layer must have higher T_c than Nb.

NbTiN – AlN bilayer



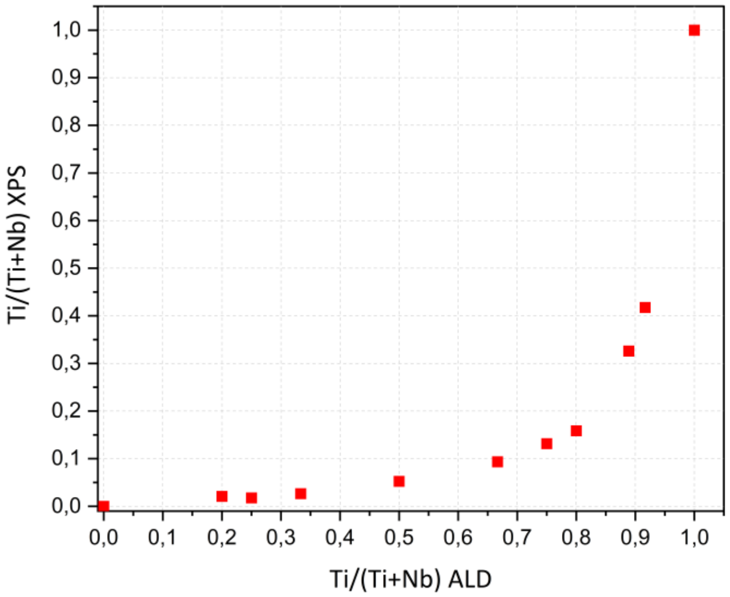
- NbTiN has good superconducting performances (T_c = 17 K) and a low resisitivity.
- AlN is a good dielectric layer and has a good chemical stability.

Chemistry: Thermal ALD @ 450°C

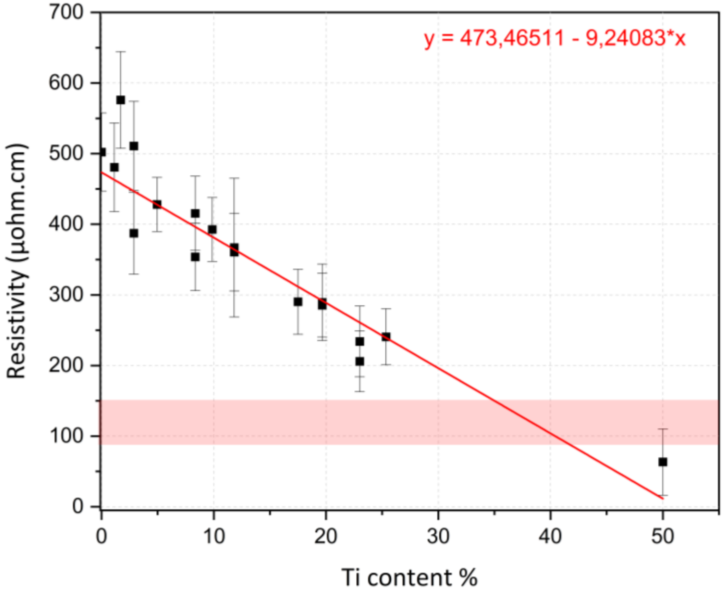
- AlN was deposited using $AlCl_3 + NH_3$
- NbTiN was deposited using a combinaison of TiN and NbN cycles

$$n\,(TiCl_4 + NH_3) + m\,(NbCl_5 + NH_3) = Nb_{1-x}Ti_xN$$

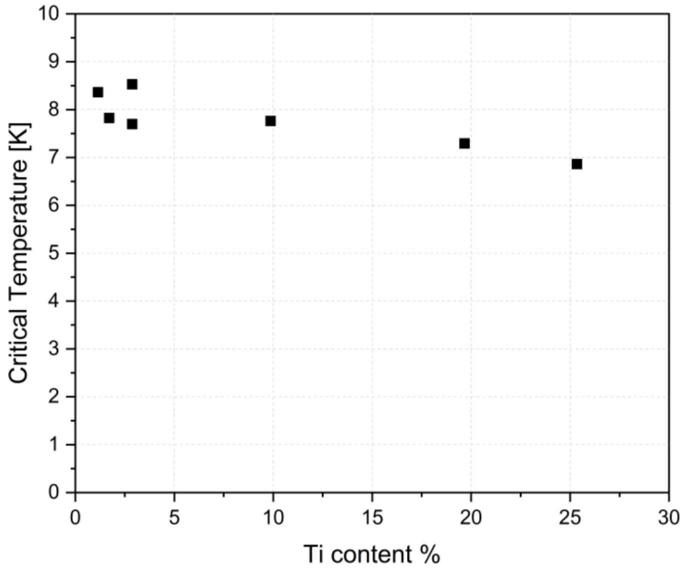
Chemical composition



Resistivity of NbTiN films

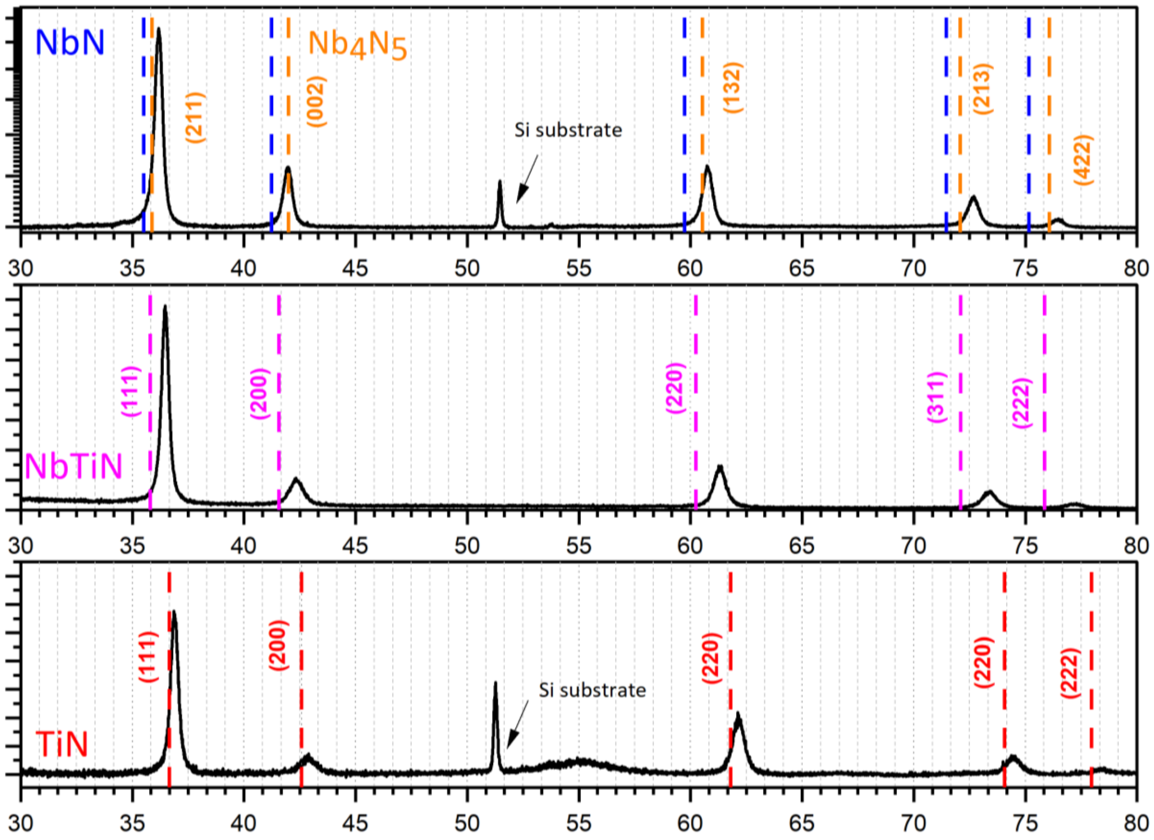


Critical temperature of NbTiN films

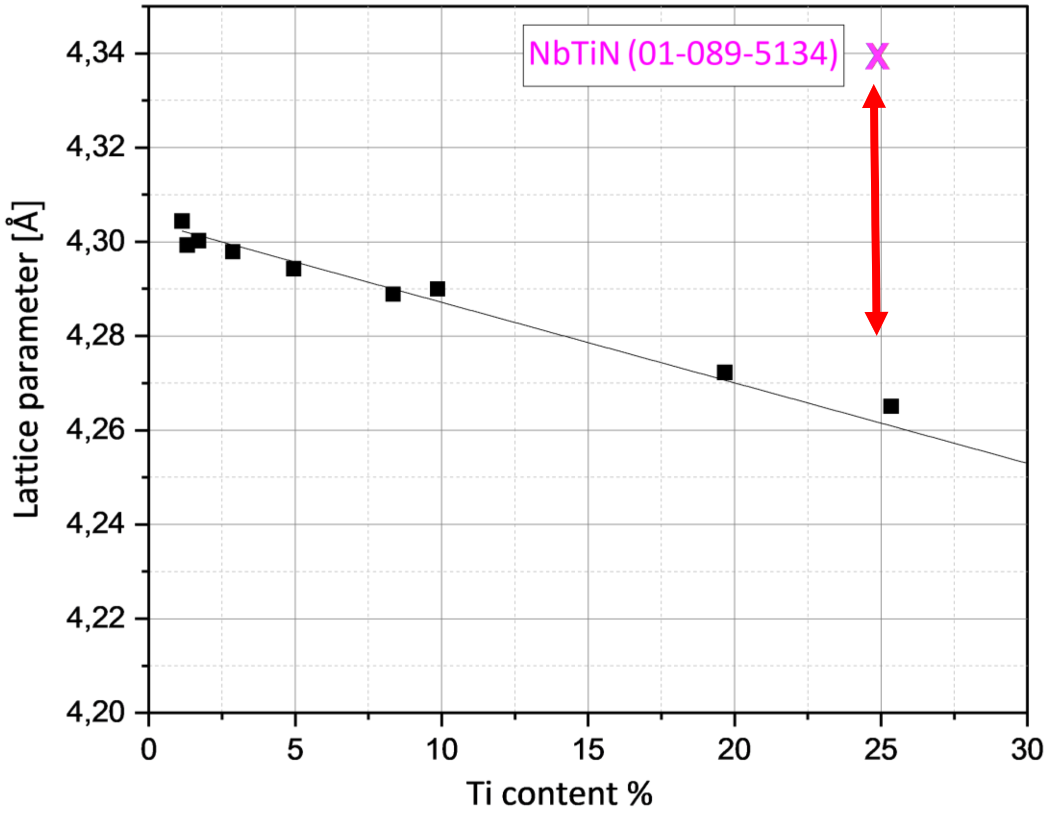


NbTiN film cristalline structures

GIXRD patterns of ALD films



DIFFRAX and INSP

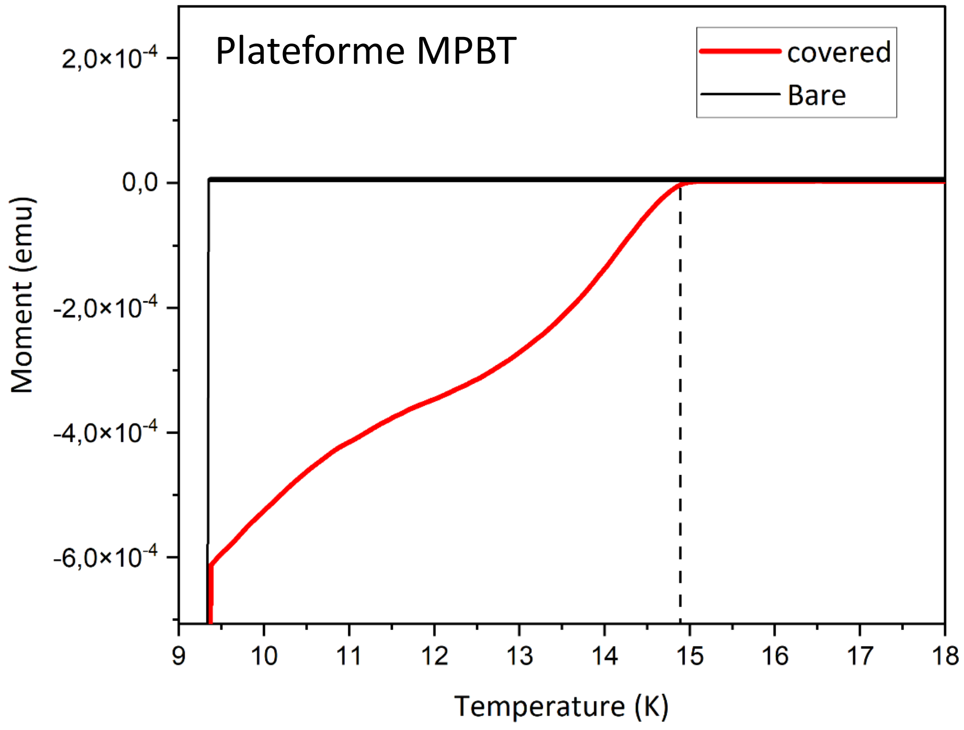


- ➢ NbTiN films are a combination of TiN and Nb_4N_5 which results in Nitrogen rich NbTiN films with smaller lattice constants than reported.

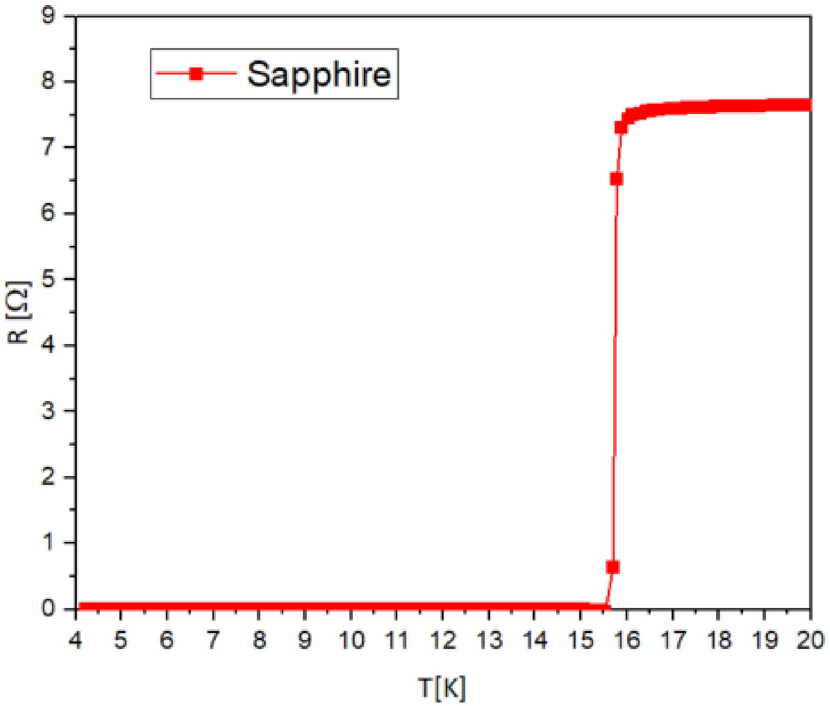
To enhance the superconducting performances of NbTiN films, several thermal treatments have been tested. The best results on Nb coated samples were obtained with:

- A first ramp of 6 °C/ minute up to 800°C
- A second ramp of 18°C/minute up to 900°C

NbTiN (45 nm) –AlN (10 nm) – Niobium



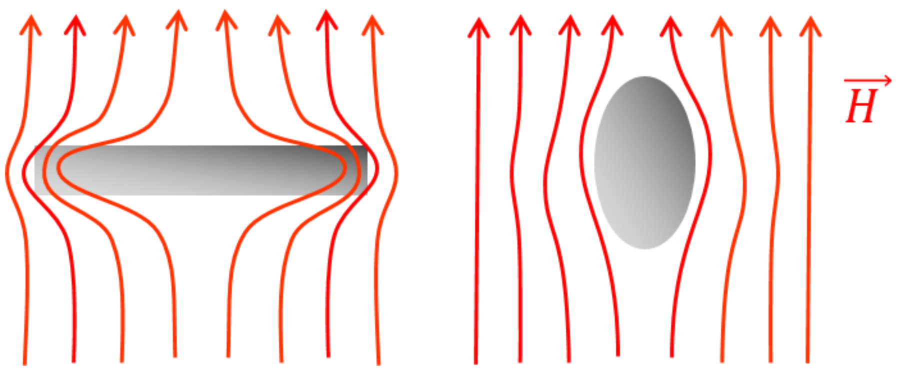
NbTiN (45 nm) –AlN (10 nm) – Sapphire



- T_c is similar on Niobium and Sapphire substrate.

Critical field enhancement on Niobium ellipsoid

- The Niobium ellipsoid was coated and annealed with the optimized NbTiN-AlN bilayer recipe.



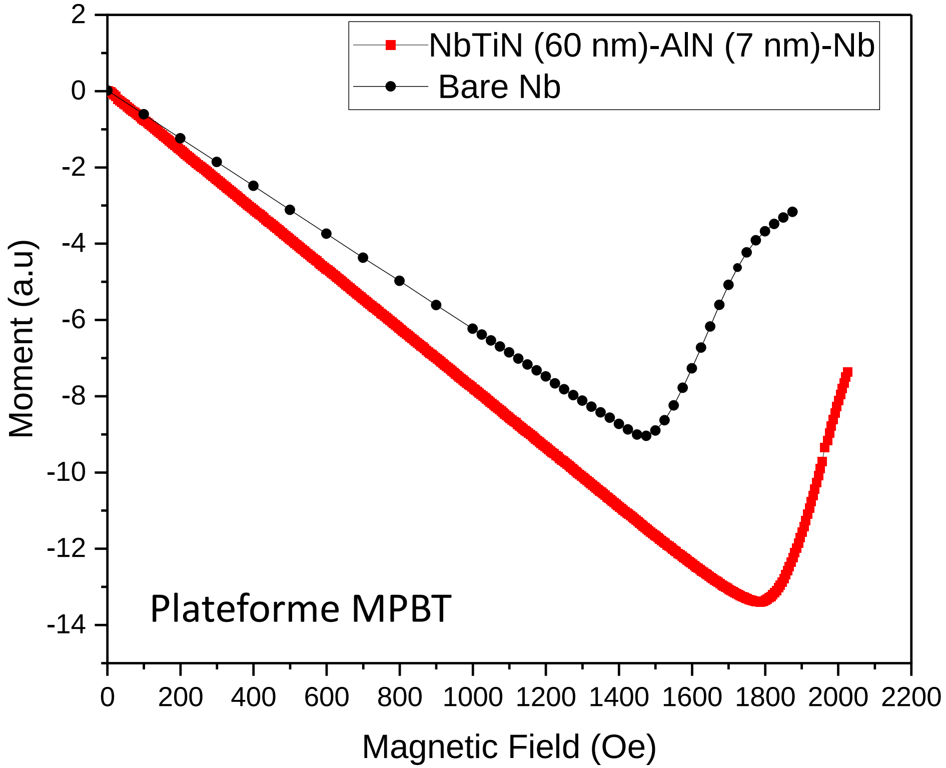
Demagnetisation factor N=0.13

$$H_{equator} = \frac{H_{applied}}{1-N}$$

Before

After

University of Victoria



- The first vortex penetration field is enhanced by 30 mT after bilayer coating.

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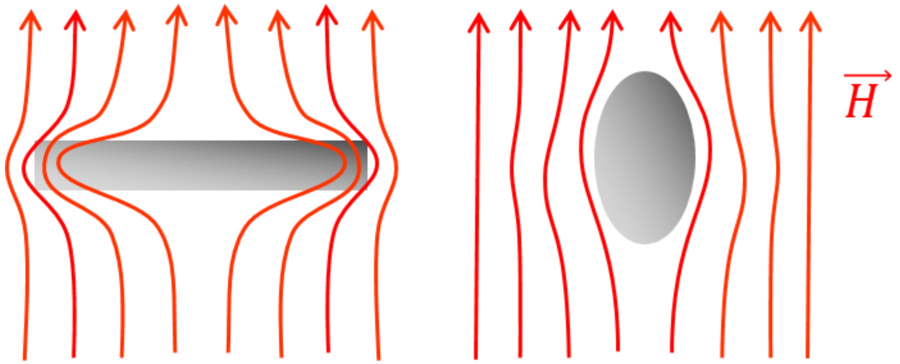

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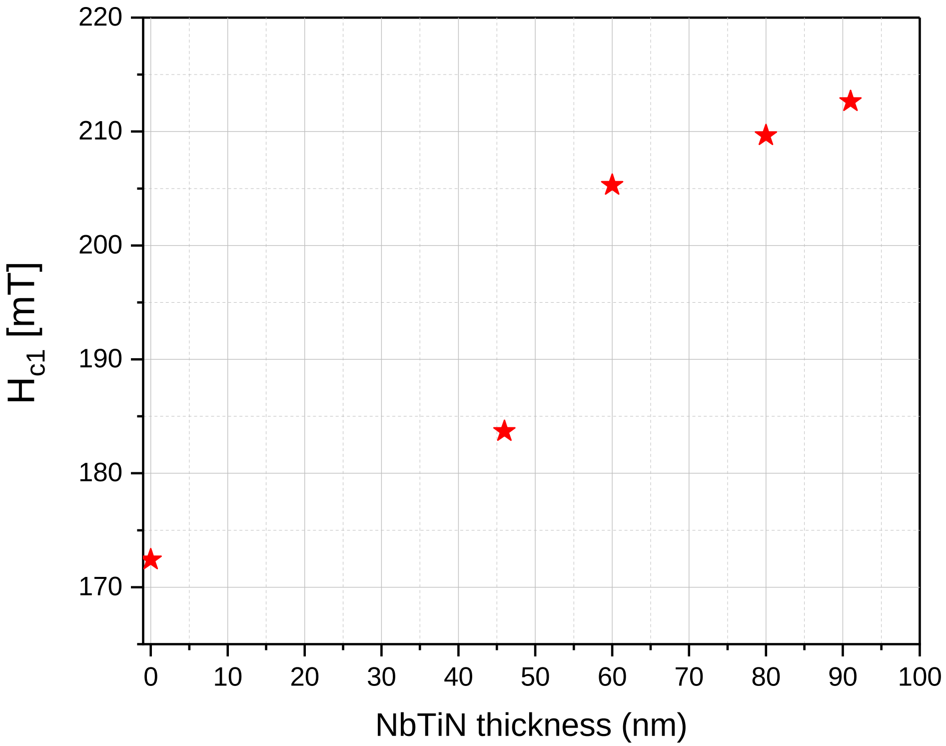
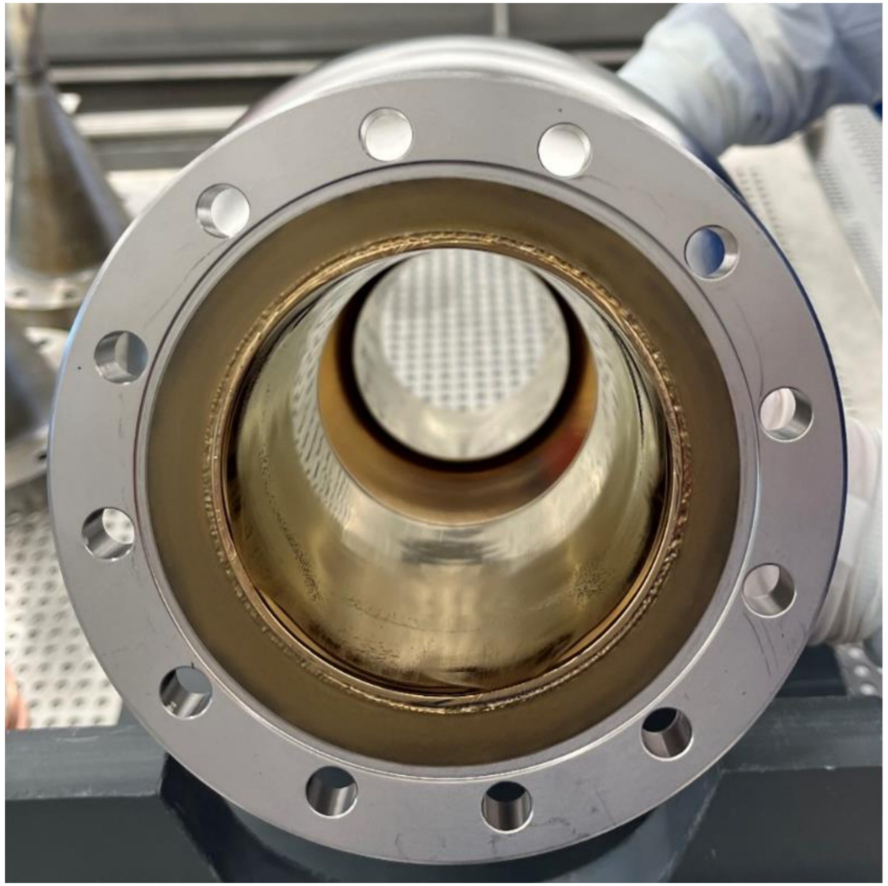


- The first vortex penetration field is enhanced by 30 mT after bilayer coating.

First RF tests of NbTiN-AlN bilayer on 1.3 GHz Nb cavity

- The Niobium cavity was coated with the optimized AlN- NbTiN bilayer recipe .



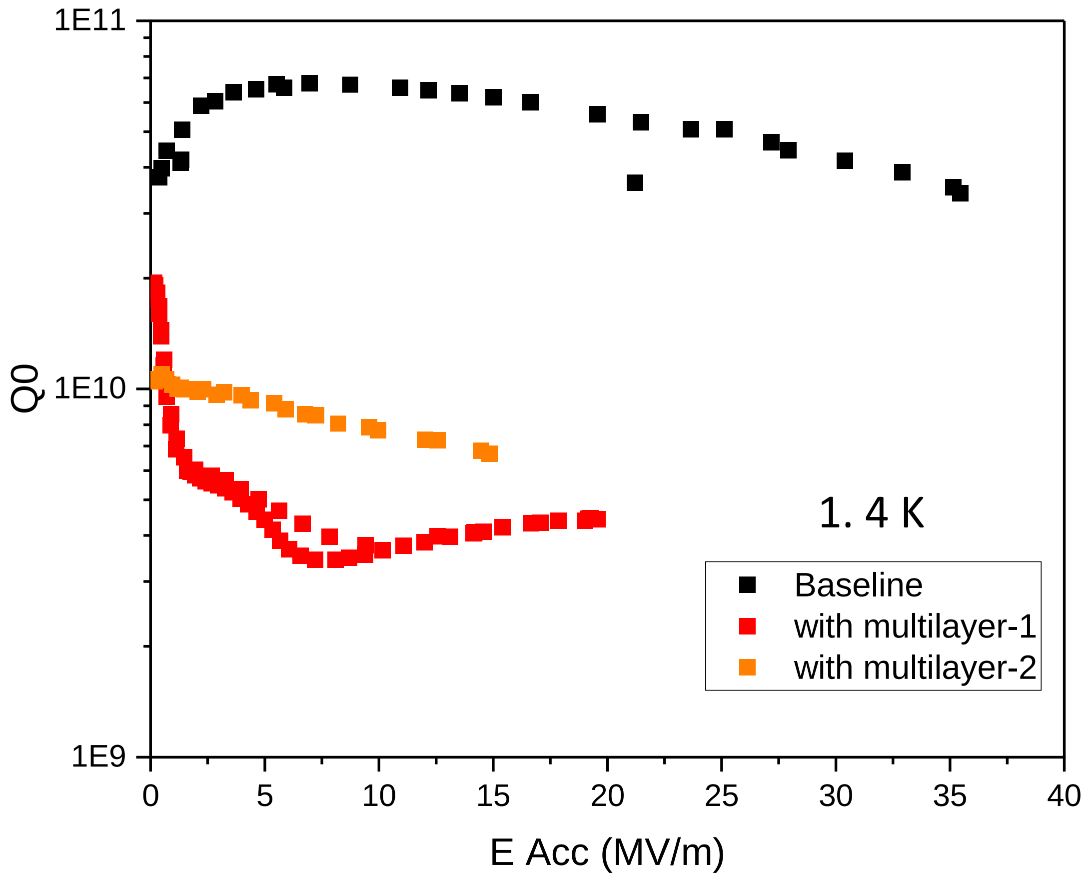
- Coating had a bright golden and uniform colour.
- The cavity was annealed @ 900°C.
- Vacuum degradation during the annealing step on the first test.
- ($P>10^{-5}$ mbar)

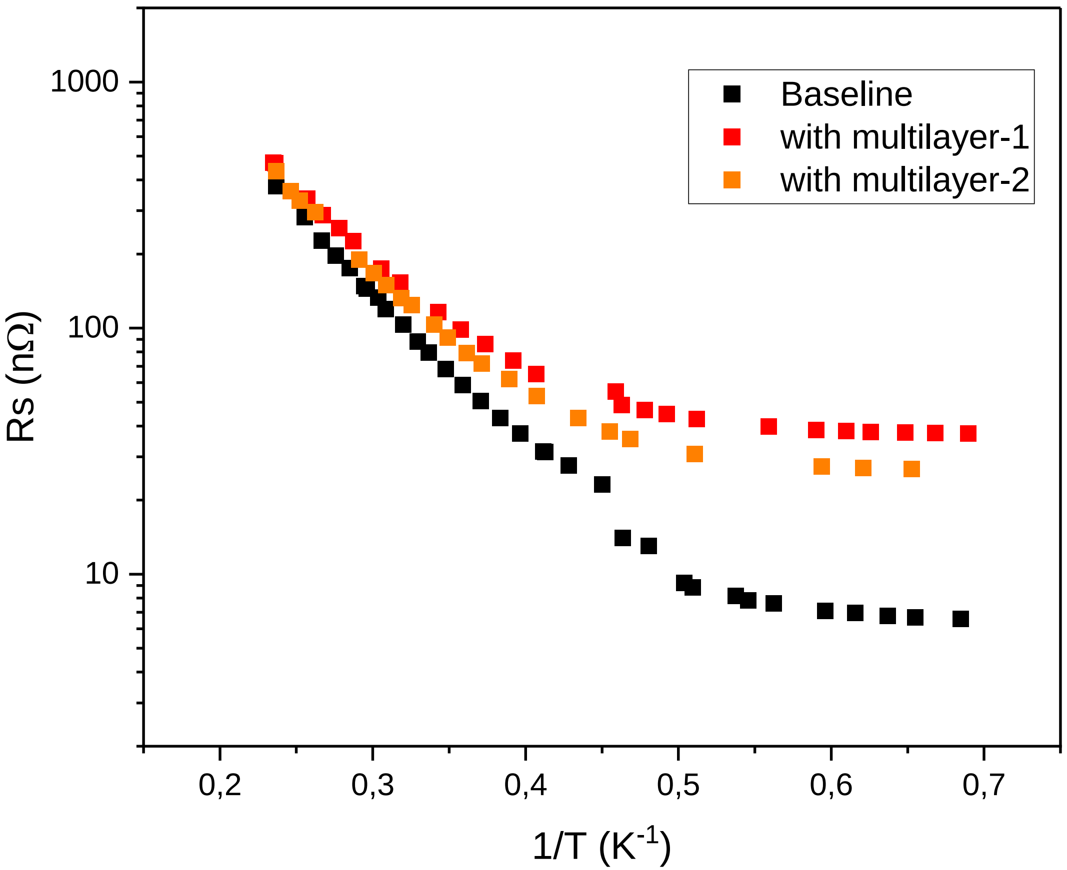
- Observed delamination in the beam tubes after annealing.

➢ A degassing step is necessary.

First RF tests of NbTiN-AlN bilayer on 1.3 GHz Nb cavity

- The Niobium cavity was coated with AlN (7 nm) – NbTiN (50 nm) bilayer .





➢ More investigations are ongoing (Q_0 vs T) …

Thermal treatment multilayer 2: IJCLAB

Summary

- ✓ Deposit uniformly thin films of Al_2O_3 and reduce drastically niobium native oxides by thermal treatment.
- ✓ Reproducible improvement of the Q_0 under low Fields -> Ongoing studies of new protective layers, deposition parameters and thermal treatments.
- ✓ Preliminary results with N-doping using ALD-deposited NbN films as dopant source on 1,3 GHz cavities without chemistry -> Ongoing optimisation of thermal treatment.
- ✓ Optimisation of AlN/NbTiN chemistry and structure via ALD + Post annealing treatments.
- ✓ Increase of first penetration field as a function of NbTiN thickness by SQUID.
- ✓ We manage to deposit uniformly thin films of AlN and NbTiN and first tests of S-I-S structure on 1.3 GHz Nb cavity.

For more details

Yasmine Kalboussi. Nano hetero-structures for improving performances of superconductors under high fields. Materials Science [cond-mat.mtrl-sci]. Université Paris-Saclay, 2023. English. ⟨NNT : 2023UPASP029⟩ ⟨tel-04116992⟩