Surface engineering by Atomic Layer Deposition for SRF cavities

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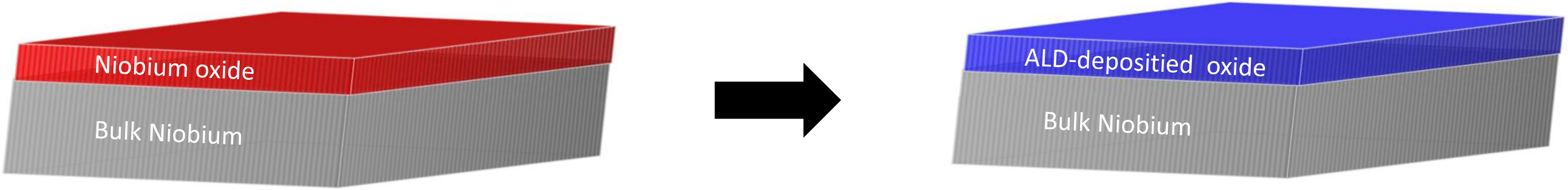
WEIBA01- SRF conference 2023

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 fied performances.



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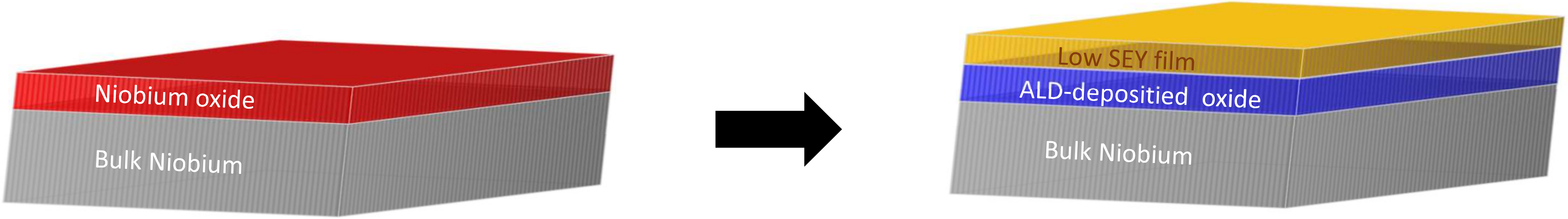

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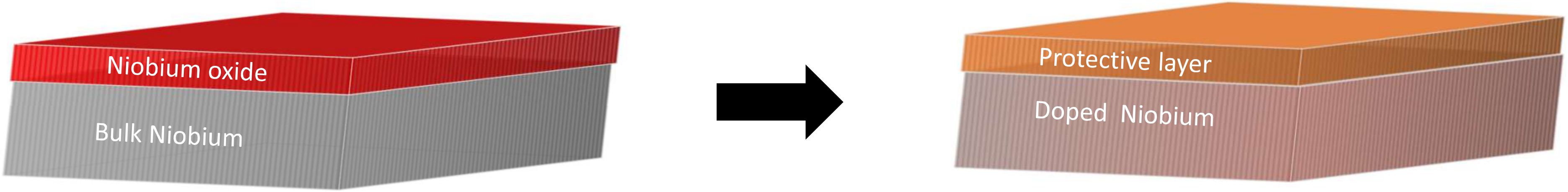
III. Doping Niobium cavities.



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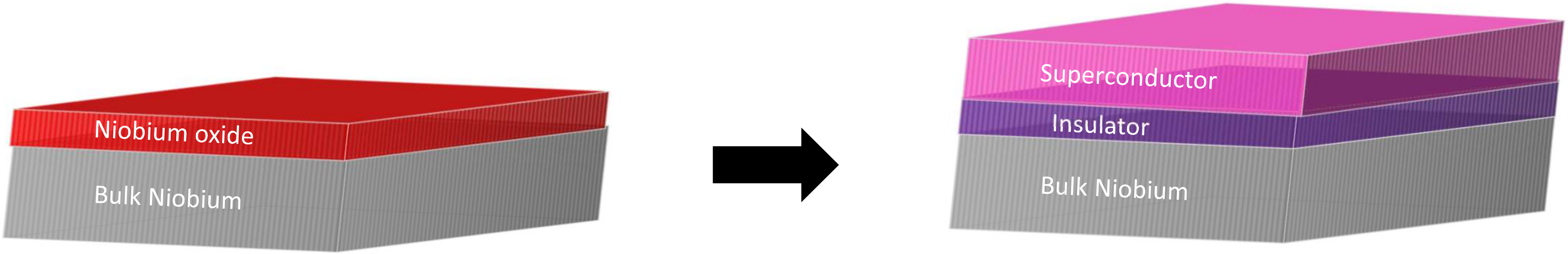
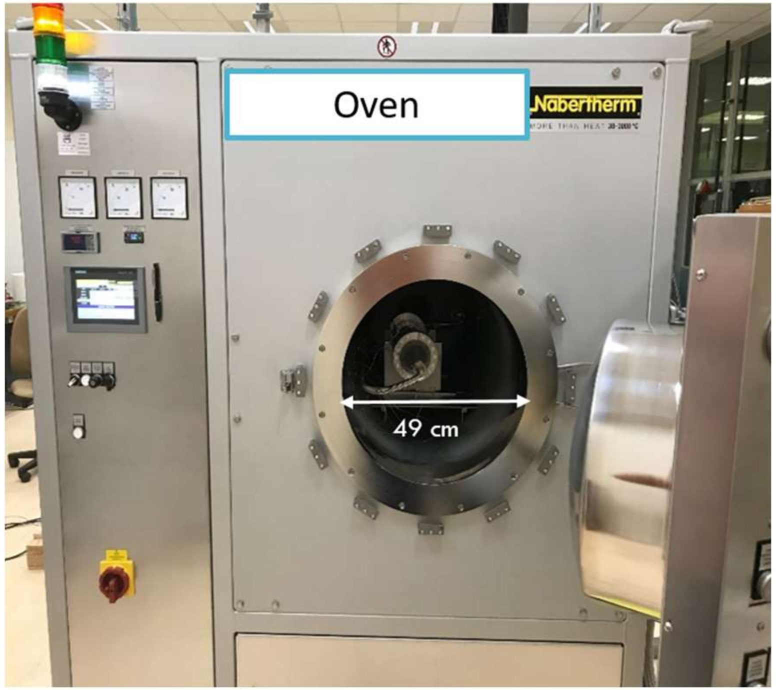
III. Doping Niobium cavities.
IV. Using a multilayer structure to screen the magnetic field seen by Niobium.

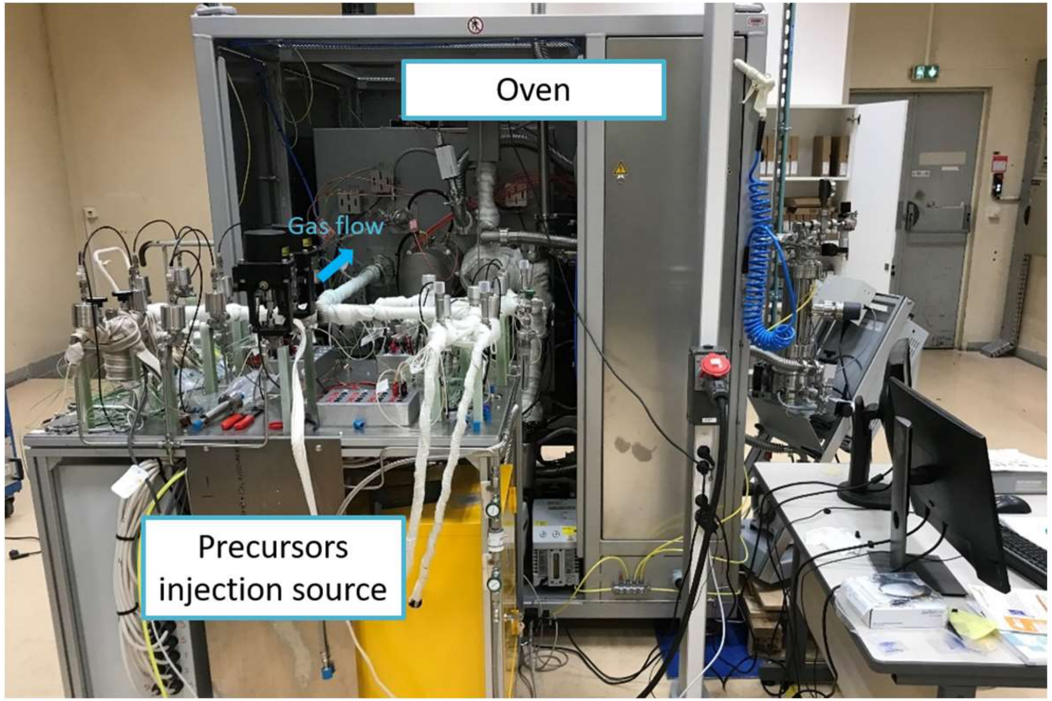


New ALD system for cavity coating at CEA

Front

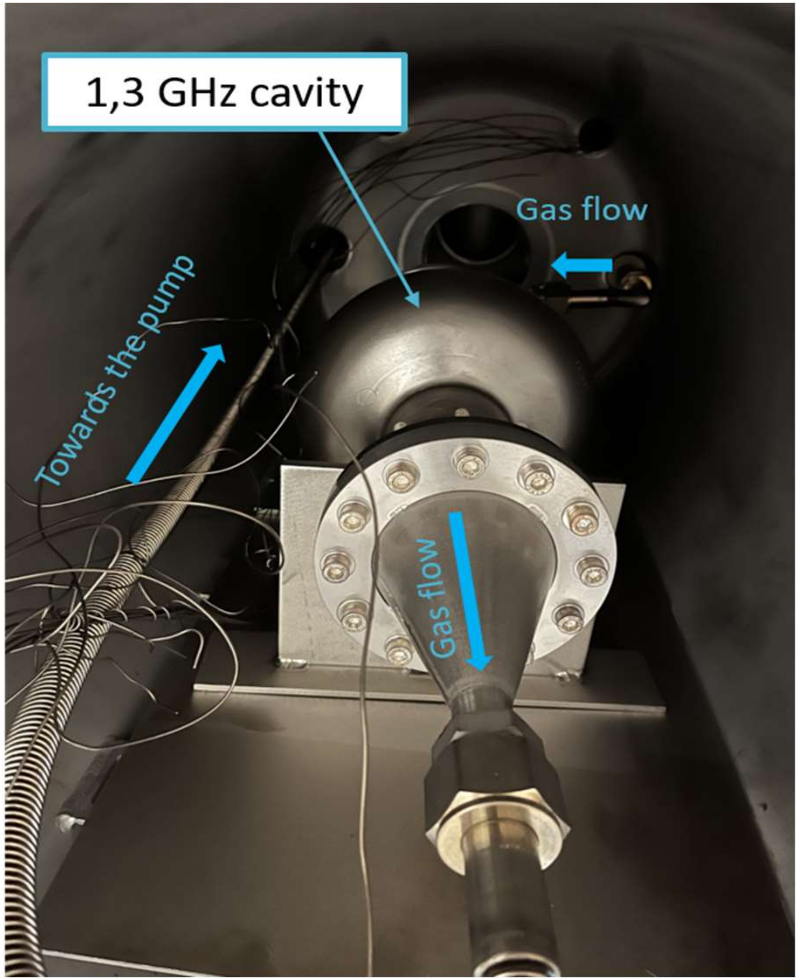


Back



- High vacuum oven:
  - 650°C – 10^{-6} mbar / 900°C 1bar N_2
  - Volume retort: Φ = 49 cm, L= 110 cm (1.3, 0.7 GHz cavities)
- ALD system:
  - 9 precursor lines (2 gases, 2 liquids, 4 solids, 1 Ultra high temp).
  - RGA synthesis monitoring.
- Interface and control:
  - Labview program of ALD system and Oven.
  - Automatic synthesis parameter control (overnight dep.) and monitoring.

New ALD system for cavity coating at CEA



- <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)

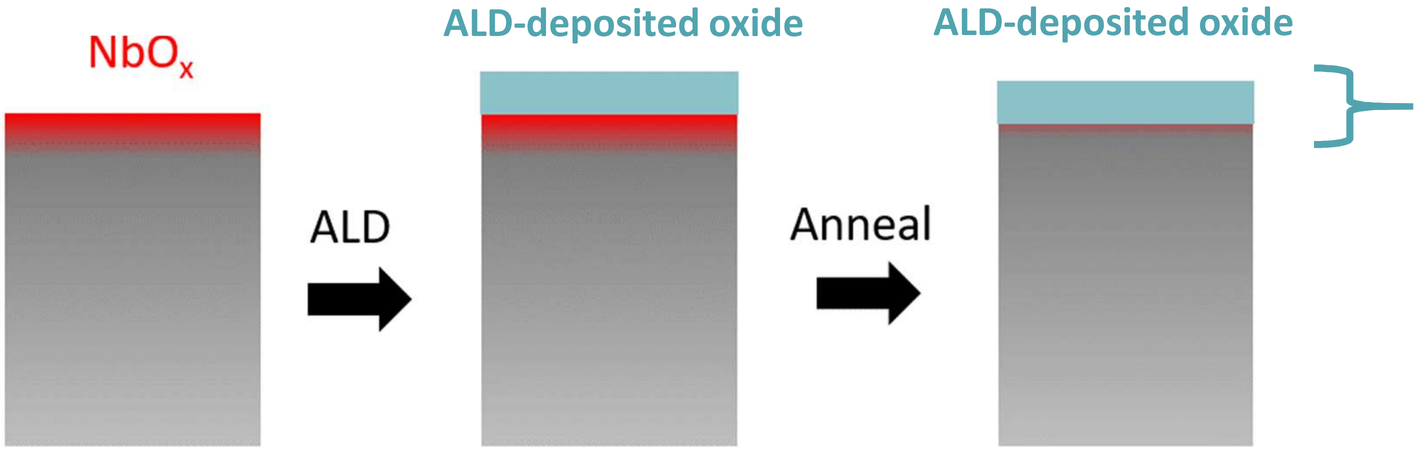
Part I:

Enhancement of niobium superconductivity through the use of ALD-oxides

Replacing niobium native oxides with ALD-deposited oxide layer

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)

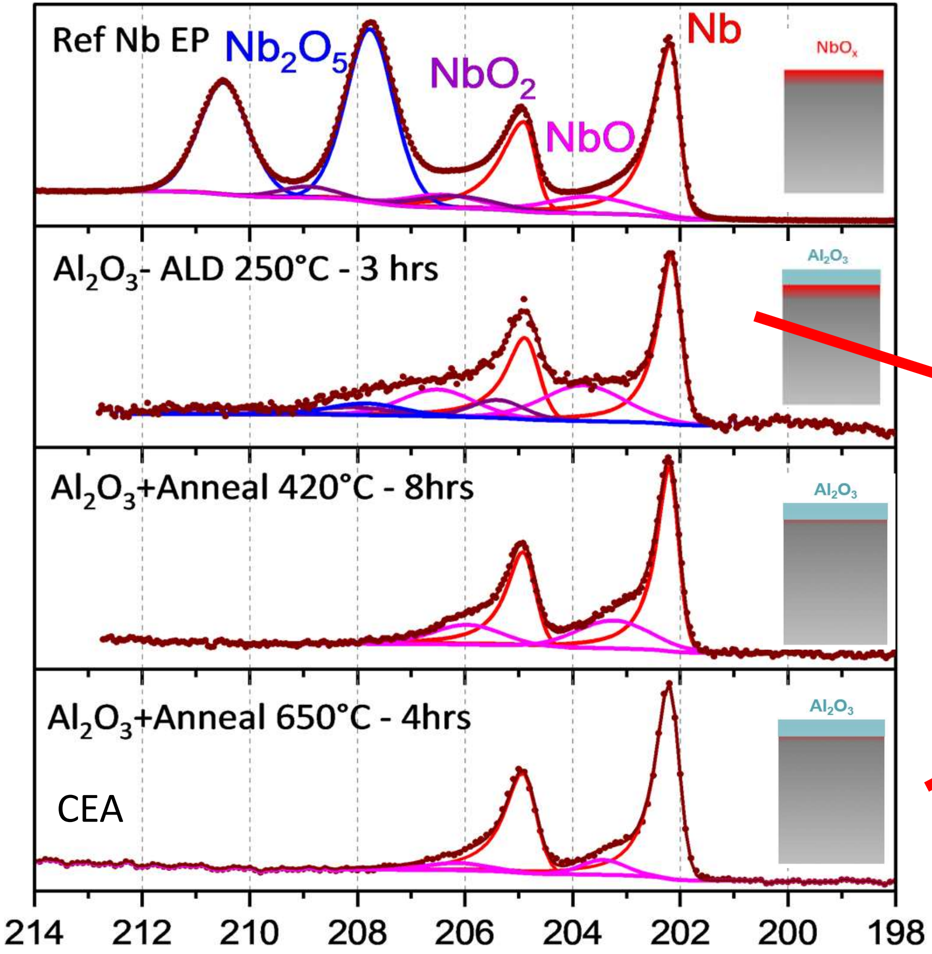


The oxide layer must be **thermally stable** and **have low dielectric** losses.

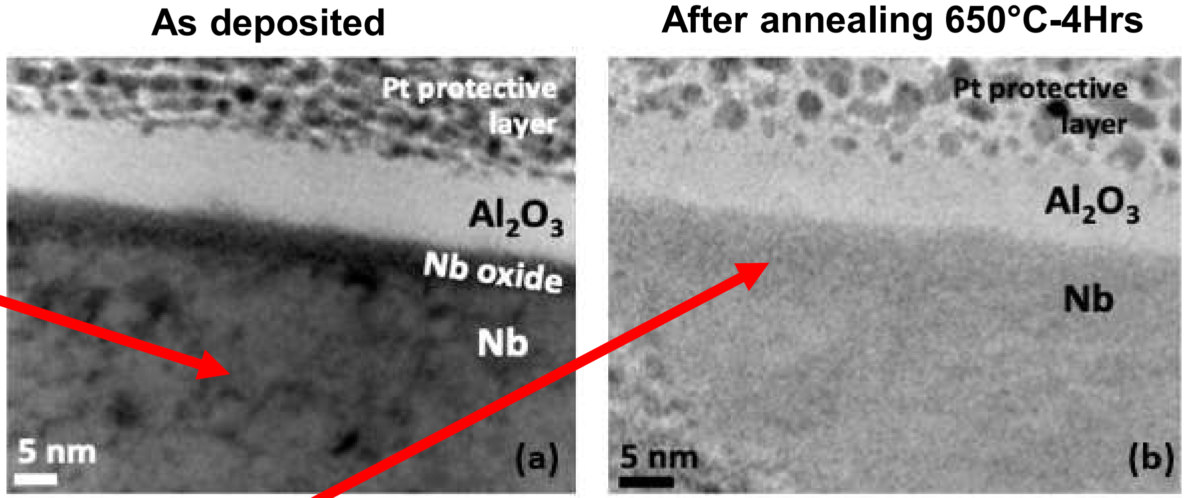
[1] T. Proslier et al . Improvement and protection of niobium surface superconductivity by atomic layer deposition and heat treatment. Applied Physics Letters, 93(19):192504, November 2008

Thermal stability of niobium oxides Vs Al_2O_3

XPS analysis of the Nb 3d core-level spectra



Transmission electron microscopy images



Plateforme MOSAIC[1]

- The10 nm film of Al_2O_3 coating is intact after the annealing at 650°C- 4 hours.

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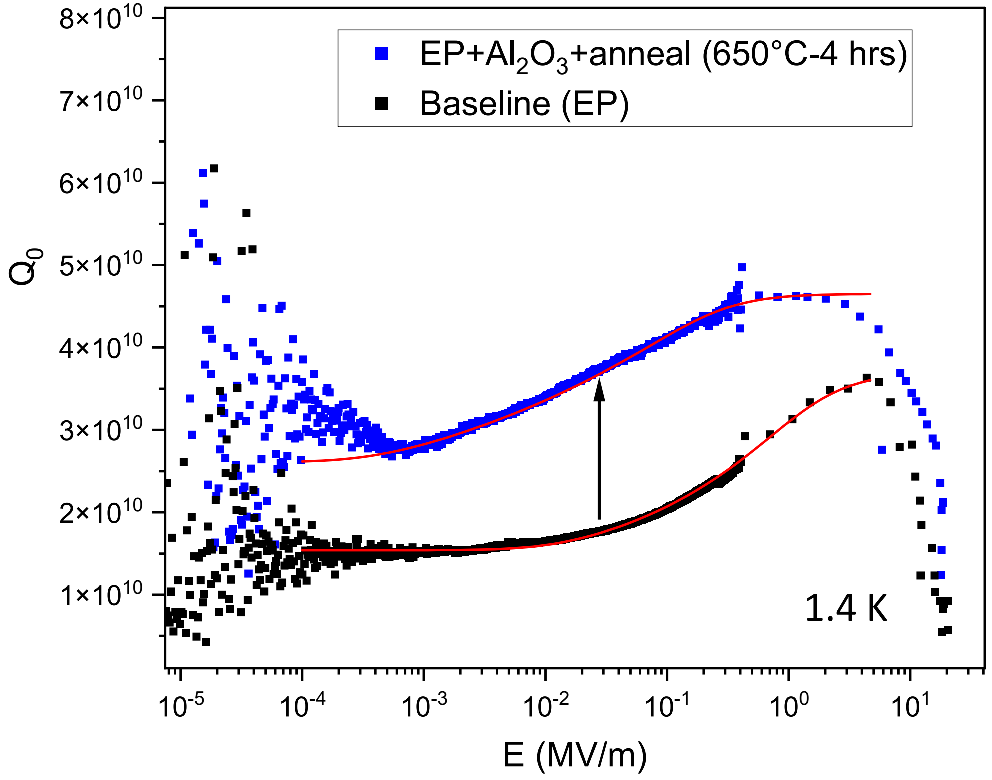
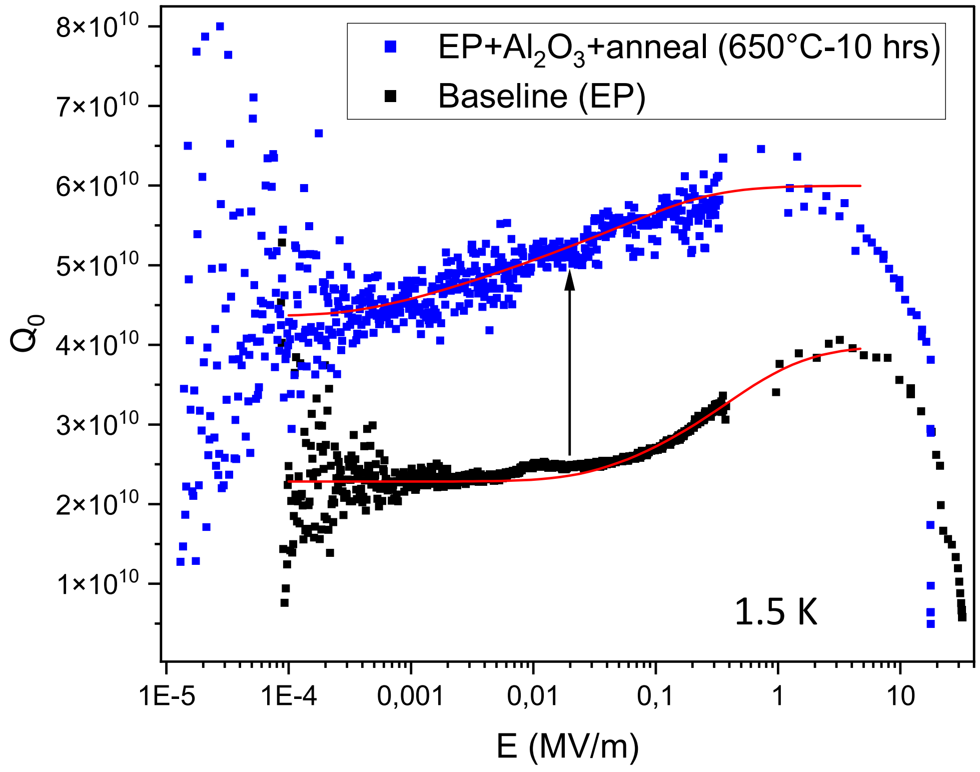
[1] *Sarra Bira and Yasmine Kalboussi PhD Thesis.*

Low field behaviour of the Al_2O_3 coated 1.3 GHz Nb cavities

First experiment



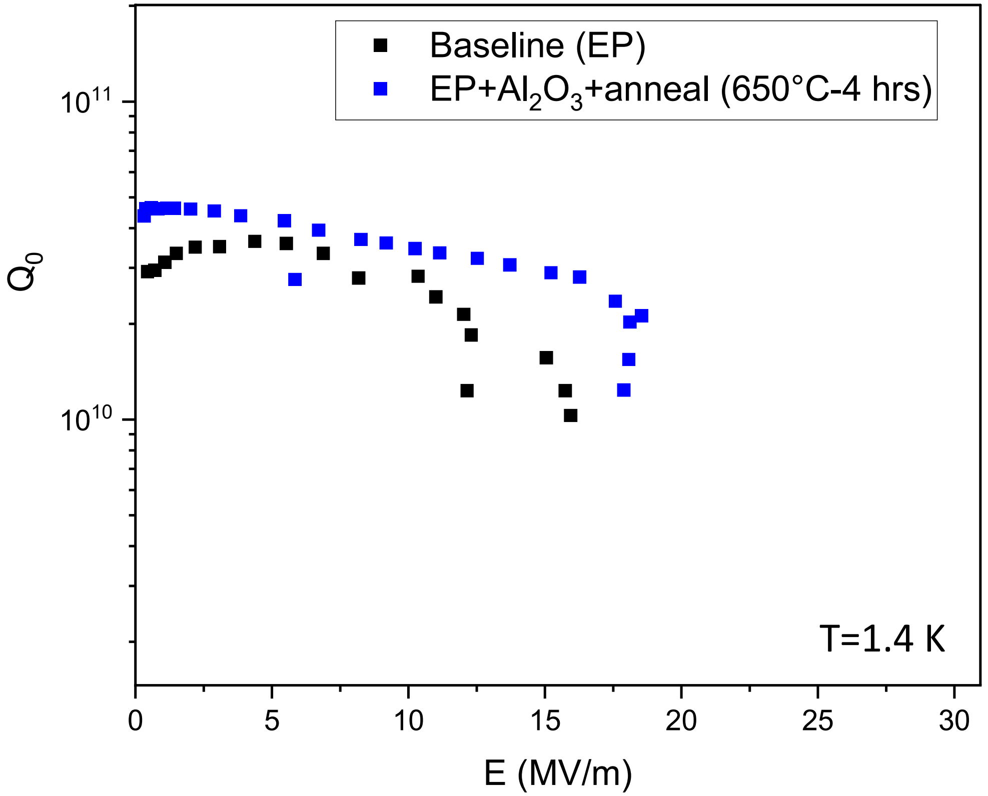
Second experiment



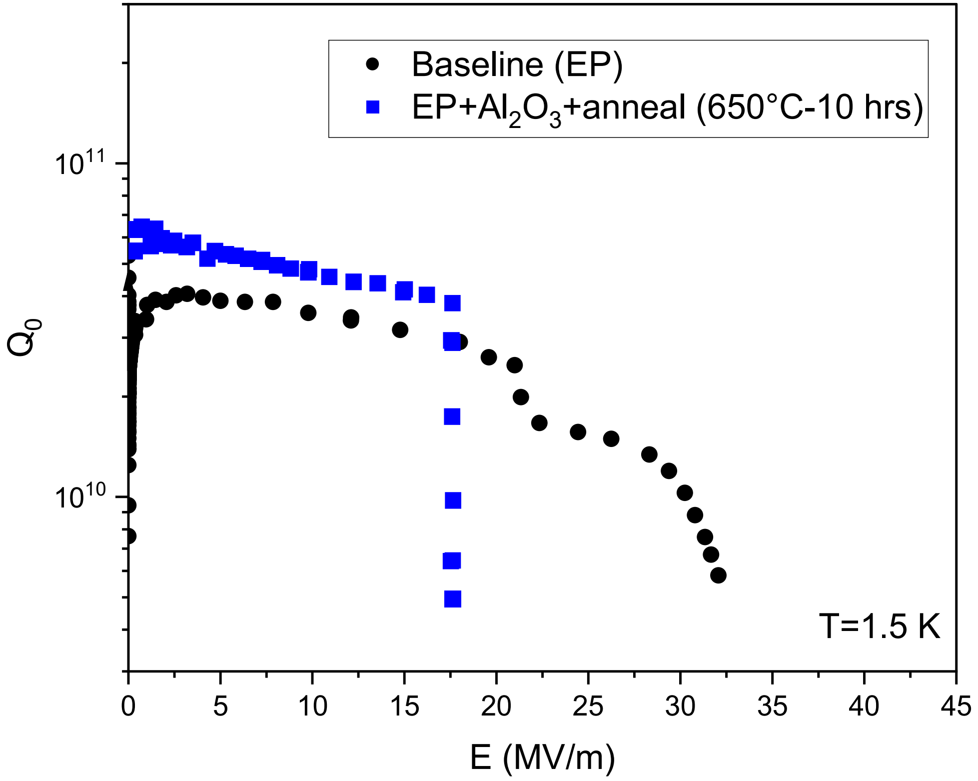
- The 10 nm Al_2O_3 film + annealing significantly improves the quality factors of the Nb cavity in the low field regime.

RF tests on the Al_2O_3 coated 1.3 GHz Nb cavities

First experiment



Second experiment

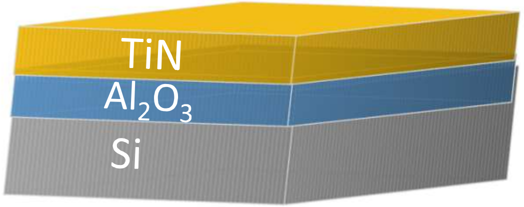


- The 10 nm Al_2O_3 coating improves the quality factors of the niobium cavity.
- The presence of multipacting barriers at 18 MV/m.

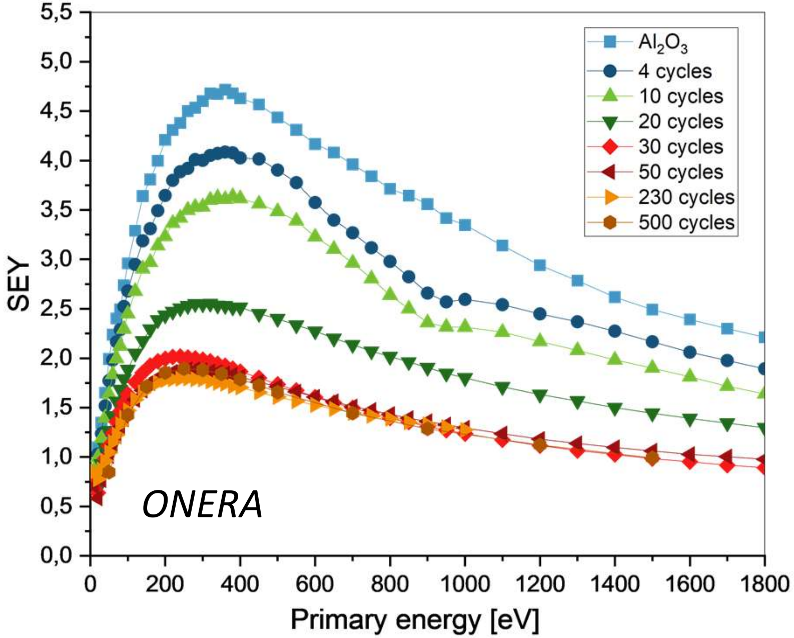
Part II:

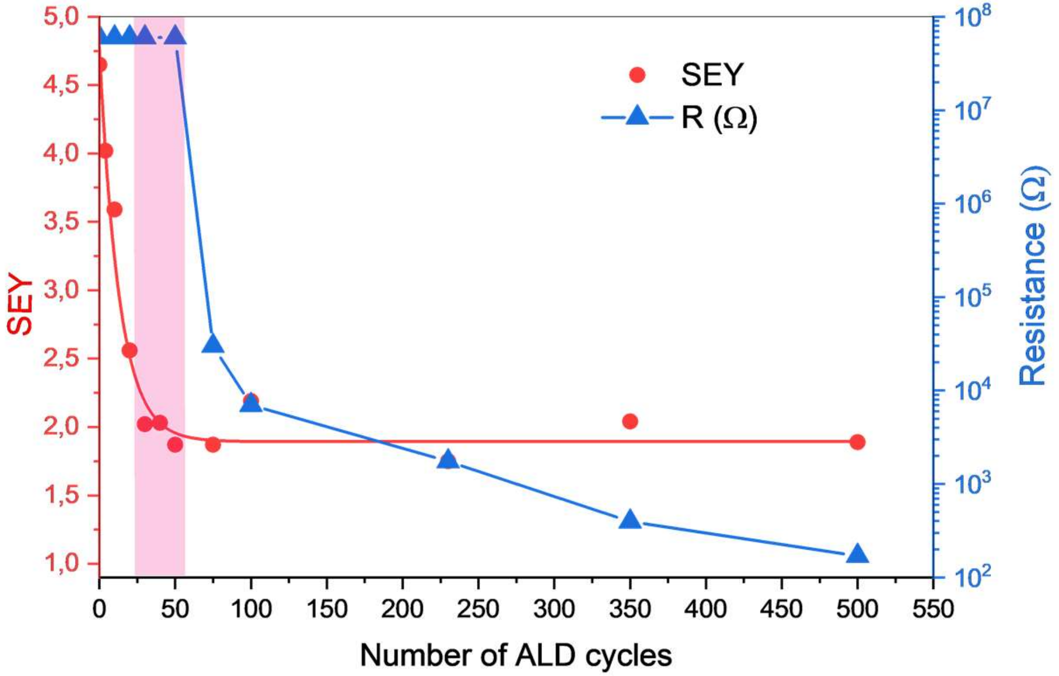
Multipacting mitigation in RF cavities

SEY measurements on the TiN- Al_2O_3 samples



- We tested increasingly thicker TiN films (by increasing the number of TiN ALD cycles) deposited on 10 nm of Al_2O_3 .



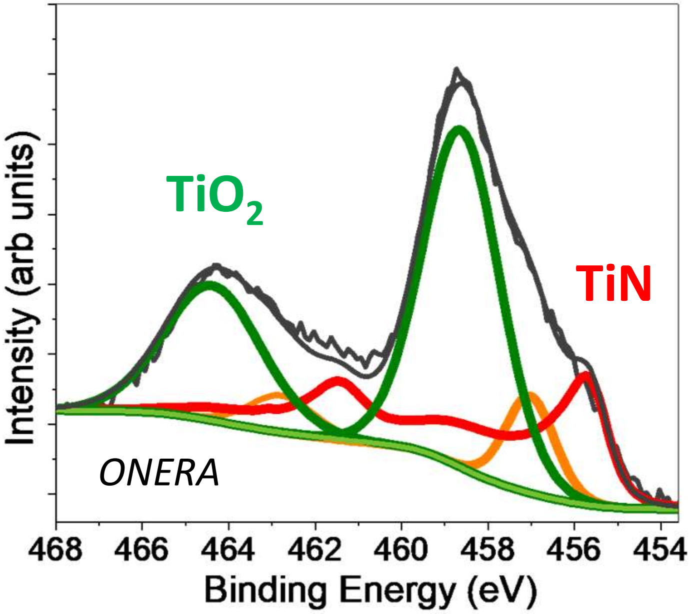


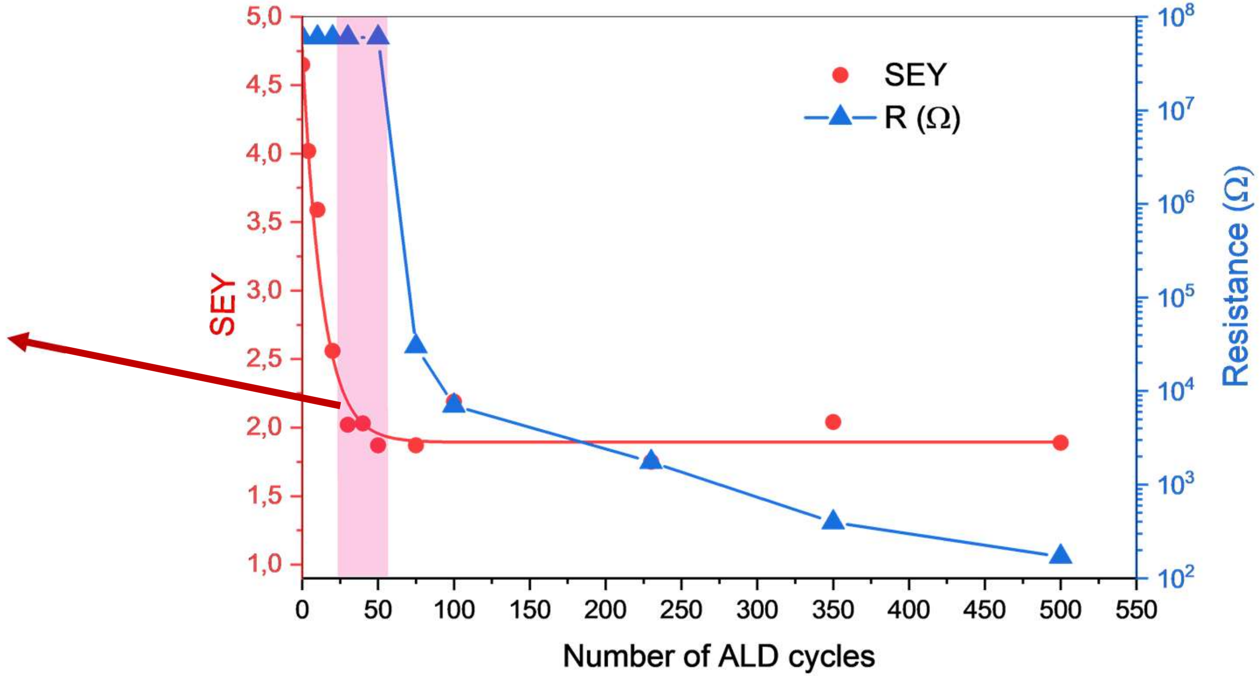
- ➢ There is a window of 30 - 50 cycles where the SEY is low and the TiN film resistance is high.

SEY measurements on the TiN- Al_2O_3 samples

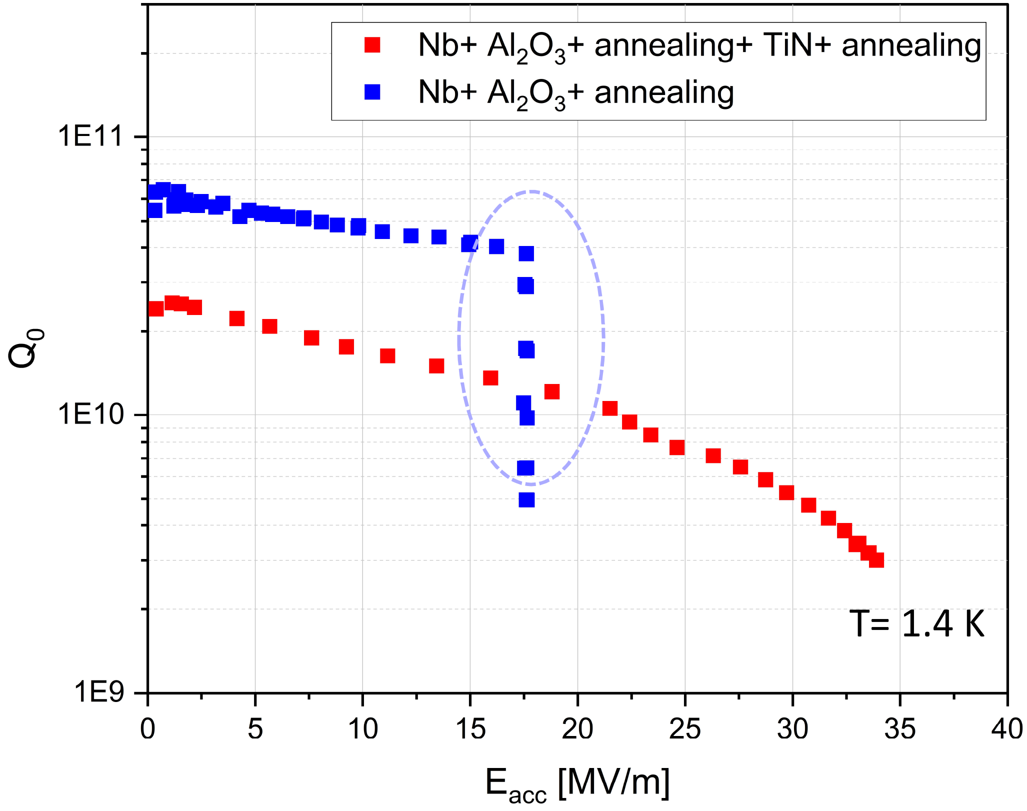
- The 30 - 50 cycles of TiN corresponds to thickness of 1 to 2 nm.
- XPS revealed a strong presence of TiO_2 in these ultra thin TiN films.

XPS of the 40-cycles TiN film





RF test of the TiN thin film on 1.3 GHz Niobium cavitiy



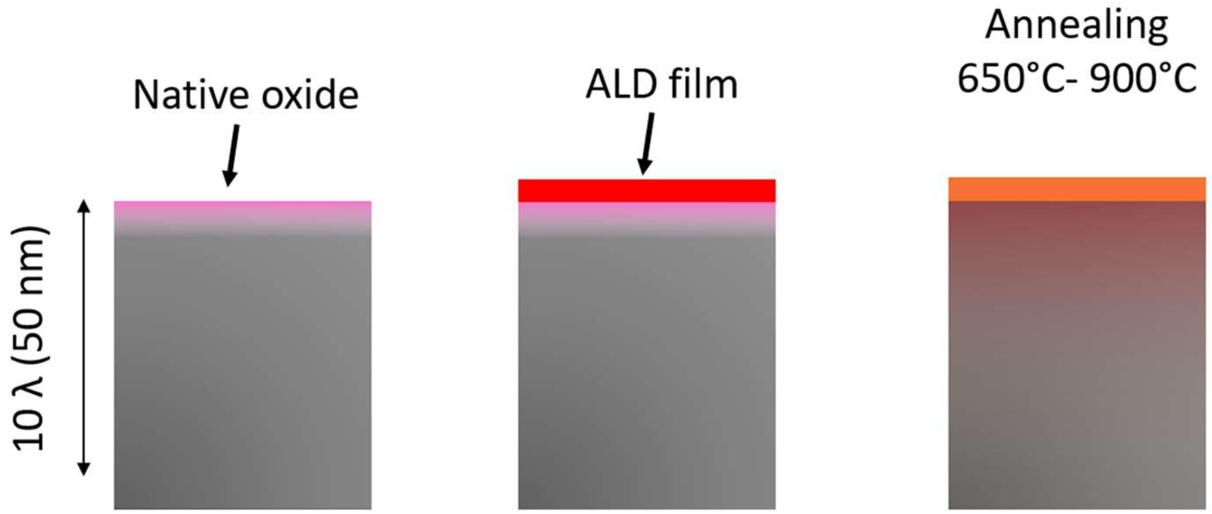
- ➢ The quality factor is lower but the 1.5 nm (40 cycles) thick TiN film is effective as a multipacting mitigation layer.

Part III:

Doping SRF cavities

Doping for SRF cavities

ALD approach for doping cavities



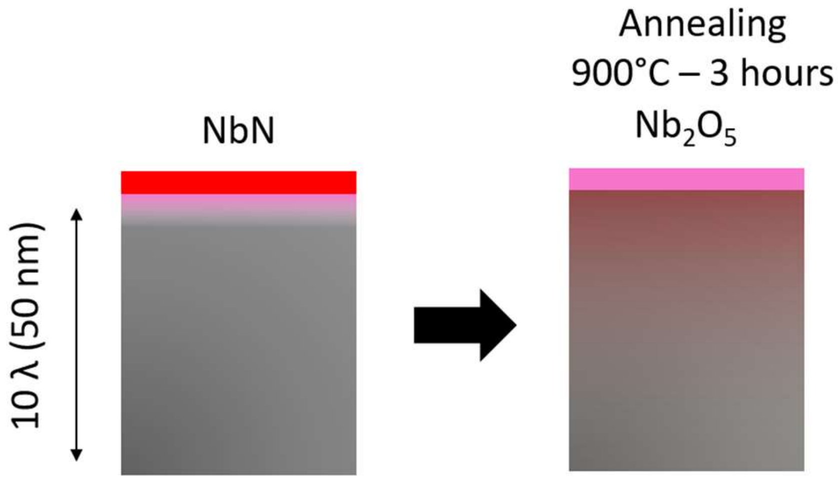
ALD synthesis: NbN, TiN, ZrN, AlN, MgO, Al_2O_3, Y_2O_3 …

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 ?

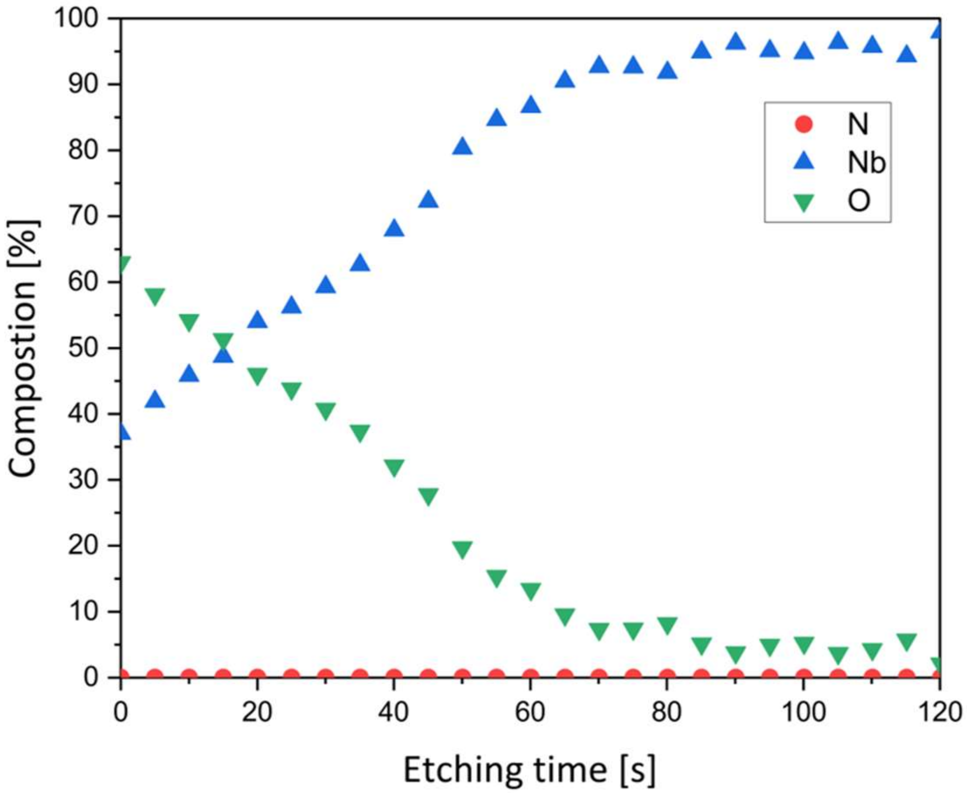
- We tested four nitrides layer: NbN, TiN, ZrN and AlN

Niobium nitride

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



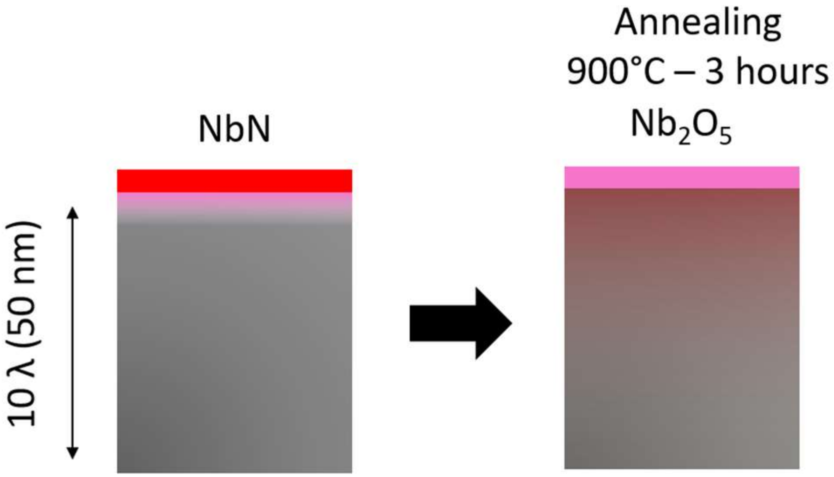
XPS Profiling



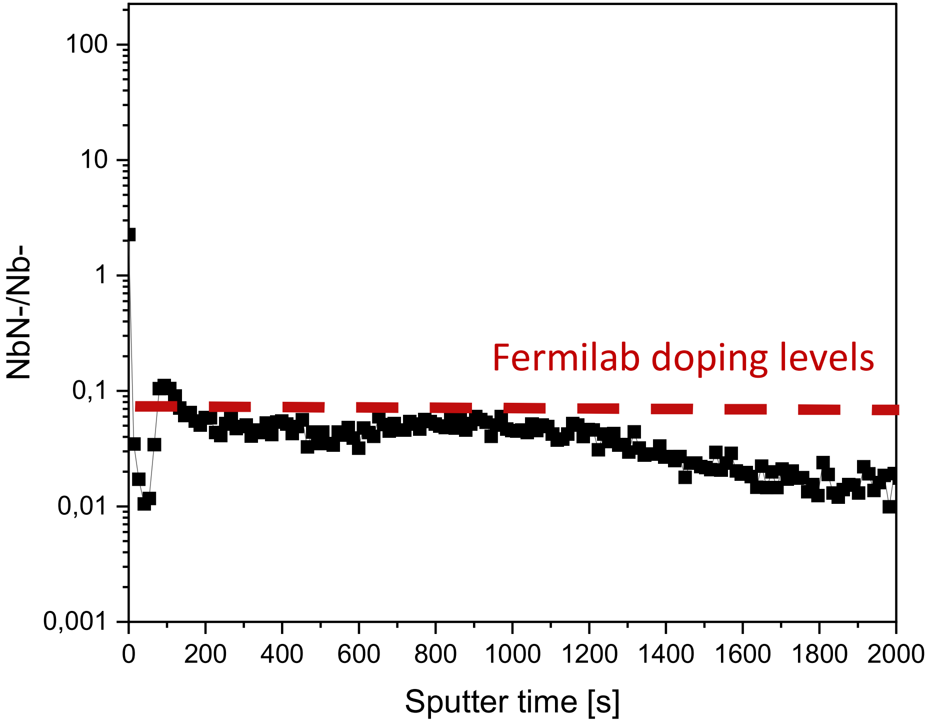
- No nitrogen detected by XPS at the surface.

Niobium nitride

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



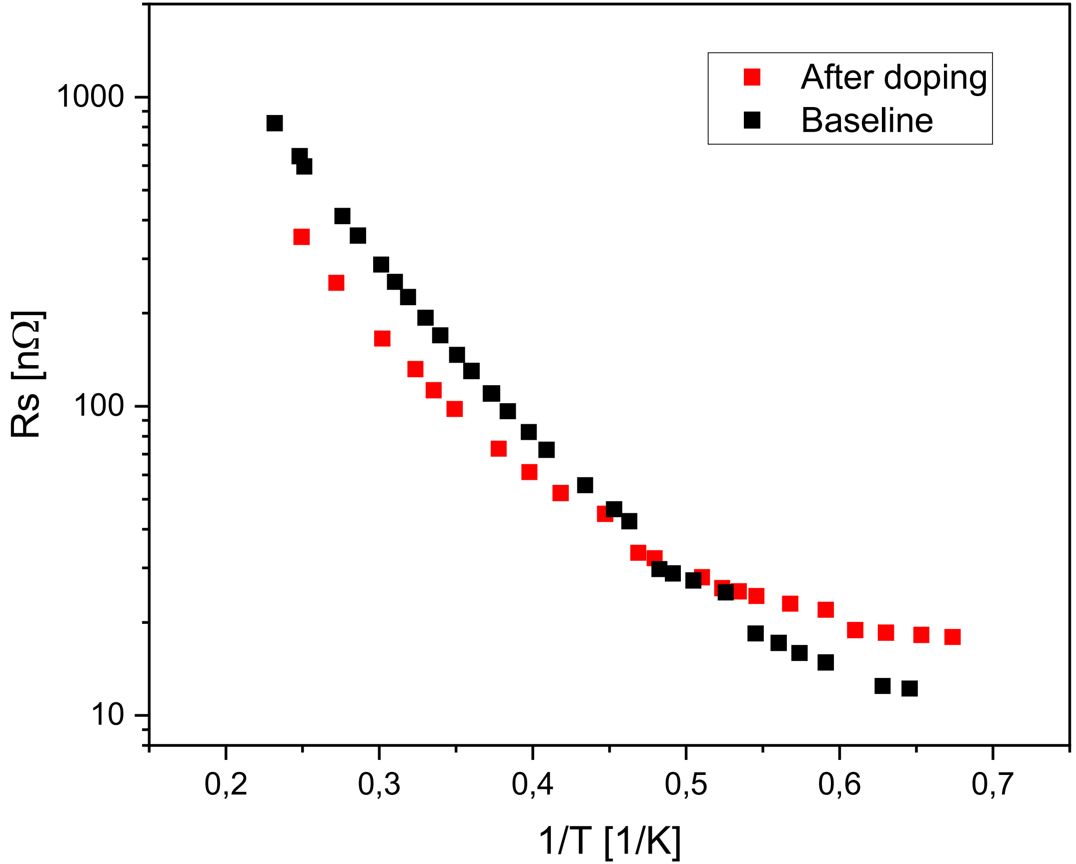
TOF-SIMS Profiling



- Doping levels comparable to observed at Fermilab without electropolishing .

Test on 1.3 GHz Nb cavity

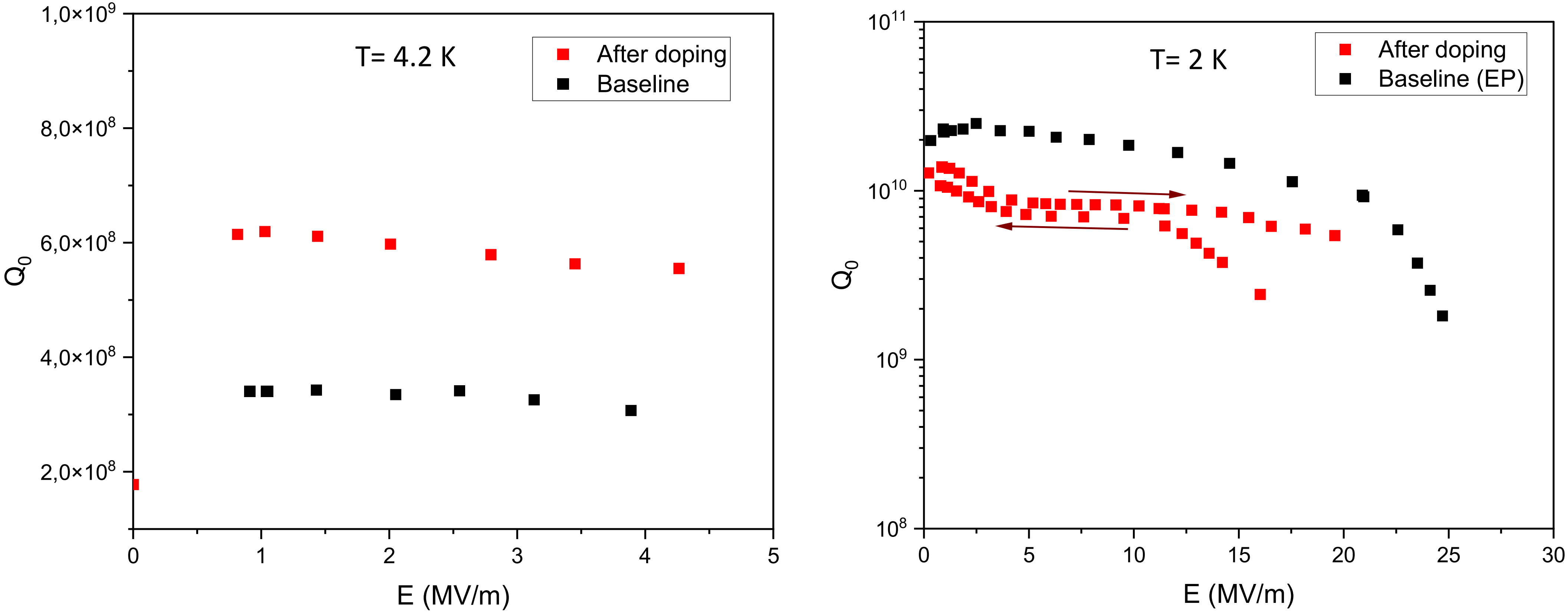
- The cavity was coated with 5 nm of NbN + annealing at 900°C-3 hours in vacuum.



➢ The R_{BCS} is lowered but the residual resistance increased.

Test on 1.3 GHz Niobium cavity

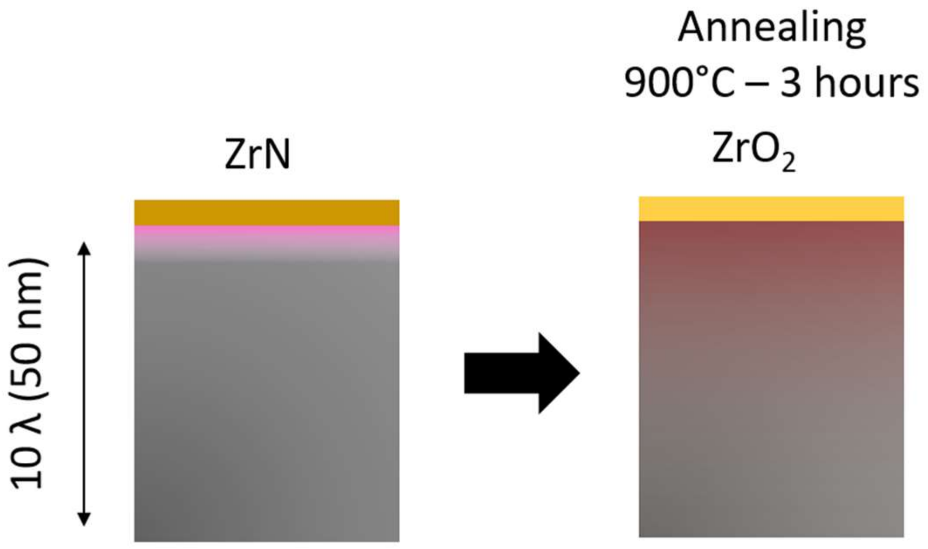
- The cavity was coated with 5 nm of NbN + annealing at 900°C-3 hours.
- No electro-polishing have been preformed.



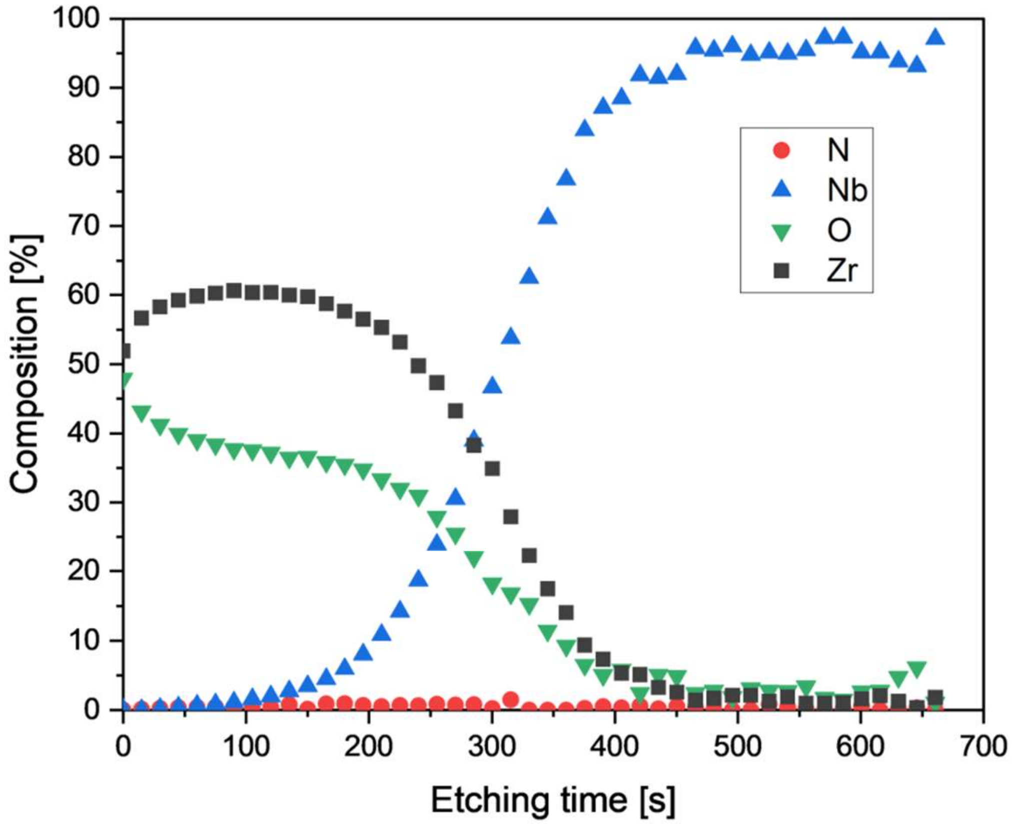
➢ The quality factor is higher at 4.2 K but lower at 2 K .

Zirconium nitride

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



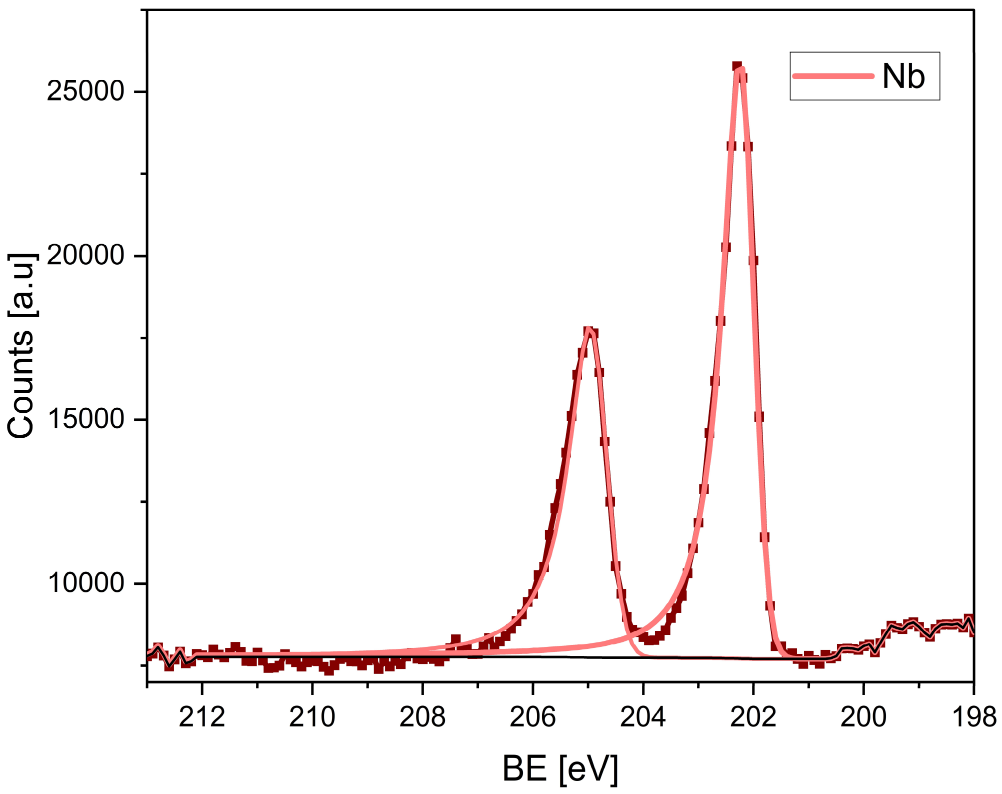
XPS Profiling

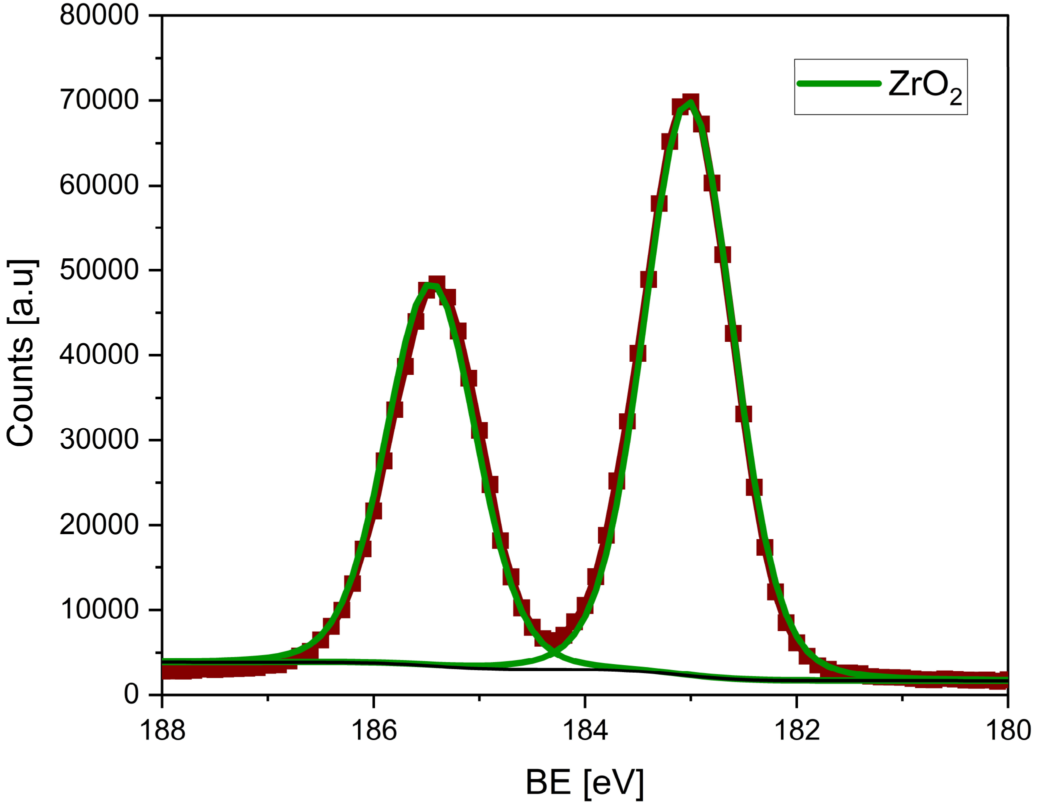


- No nitrogen detected by XPS at the surface.

Zirconium nitride

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



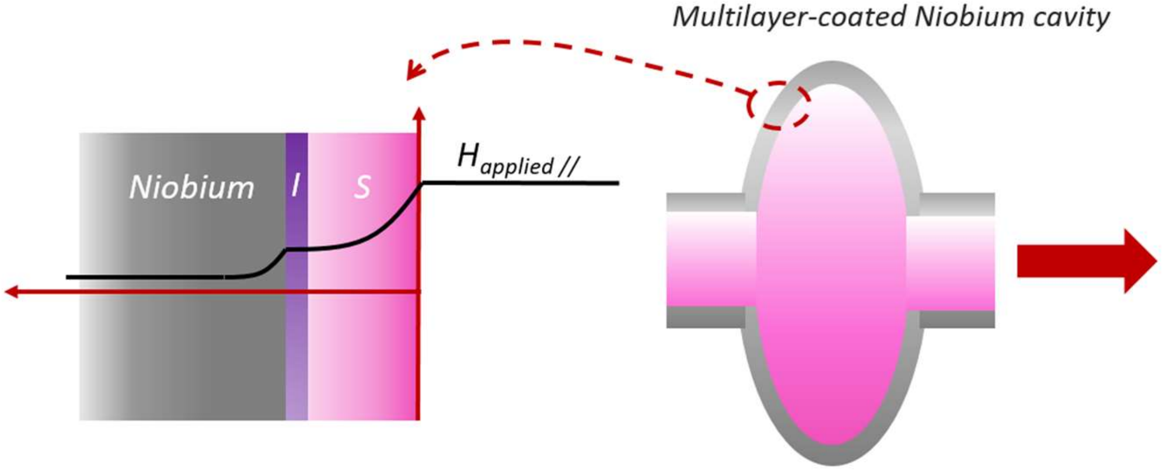


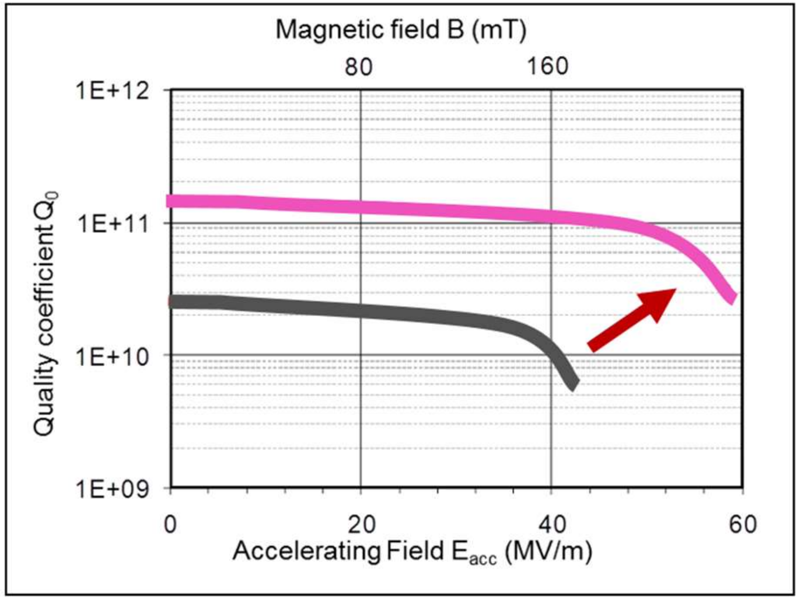
- The Nb is well passivated with a ZrO_2 layer.

Part IV:

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

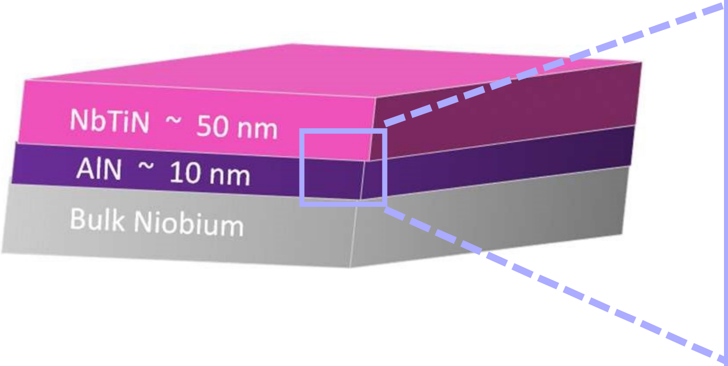
Multilayer structure





- 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



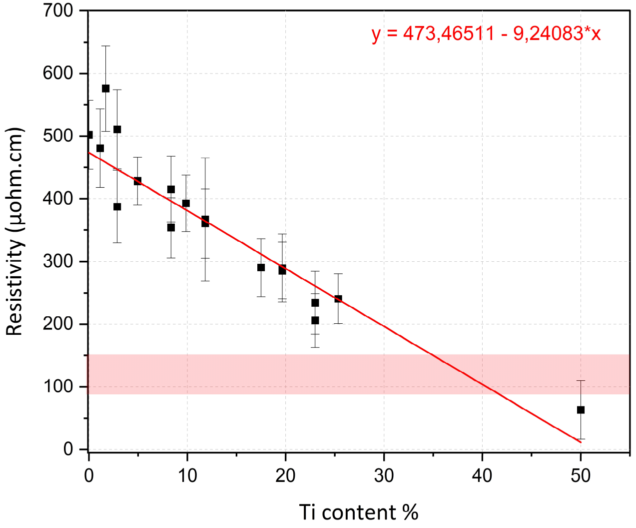
- NbTiN has good superconducting performances (T_c = 17 K) and a low resisitivity.
- AlN is a good dielectric layer and has a good chemical stability.

Chemistry: Thermal ALD @ 450°C

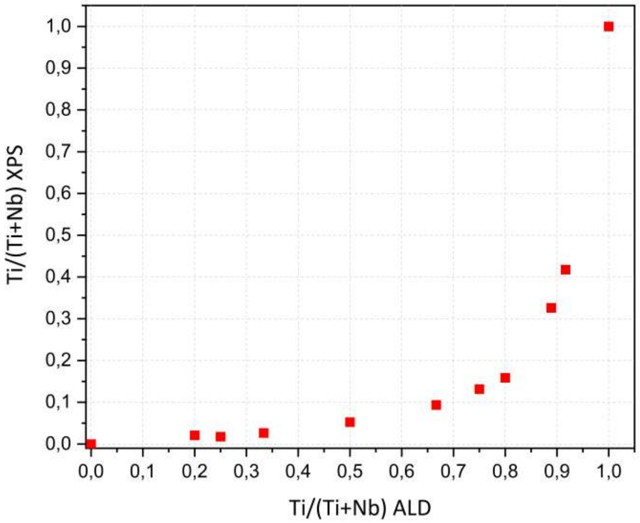
- AlN was deposited using $AlCl_3 + NH_3$
- NbTiN was deposited using a combinaison of TiN and NbN cycles

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

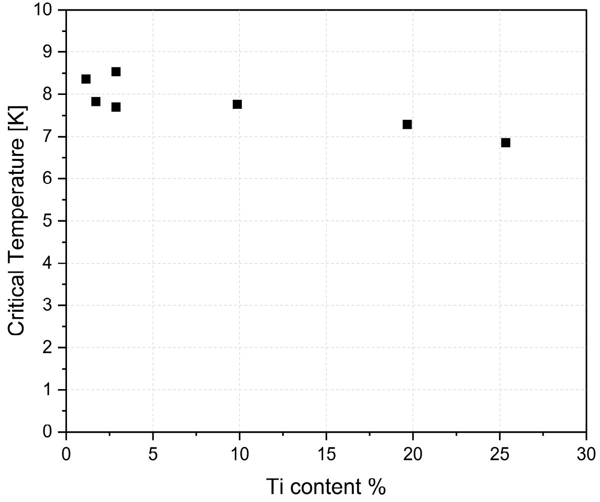
Resistivity of NbTiN films



Chemical composition

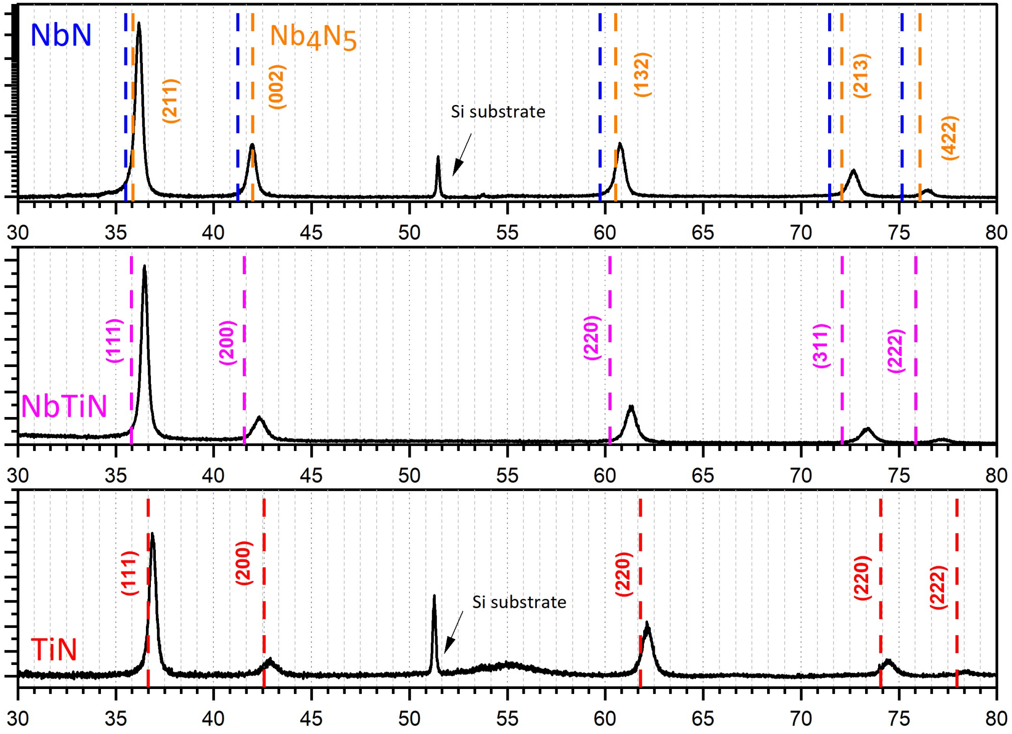


Critical temperature of NbTiN films



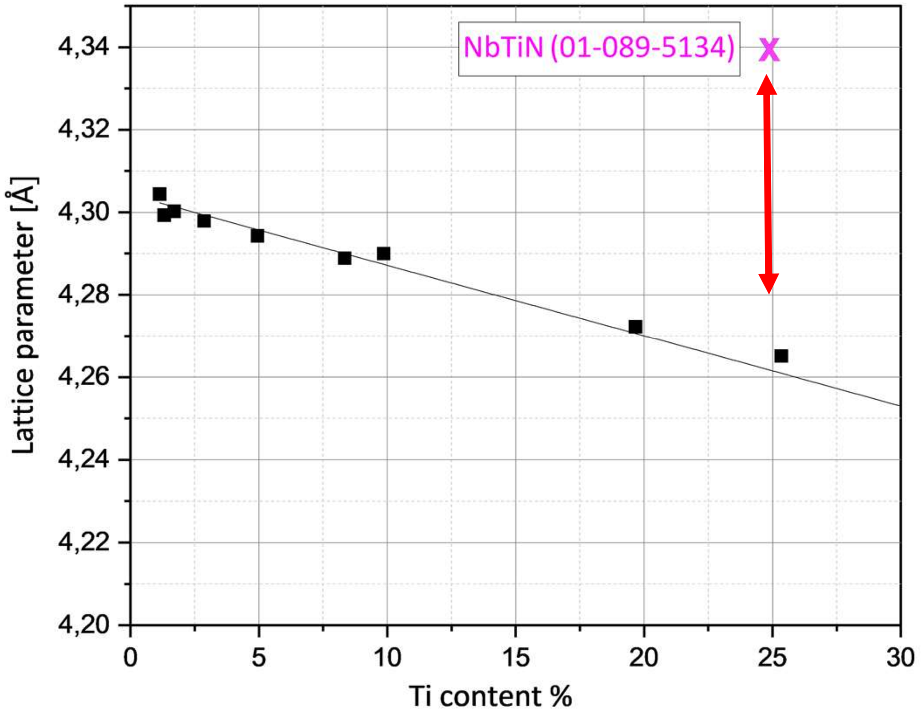
NbTiN film cristalline structures

GIXRD patterns of ALD films



DIFFRAX and INSP

2θ [degree]



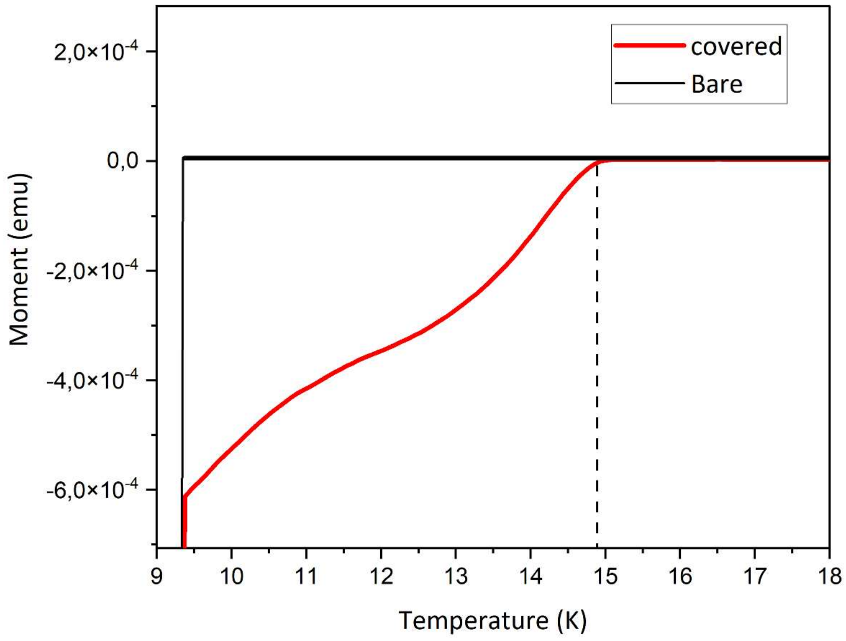
- NbTiN films are a combination of TiN and Nb_4N_5 which results in Nitrogen rich NbTiN films with smaller lattice constants than reported.

Testing thermal treatements on NbTiN-AlN bilayers

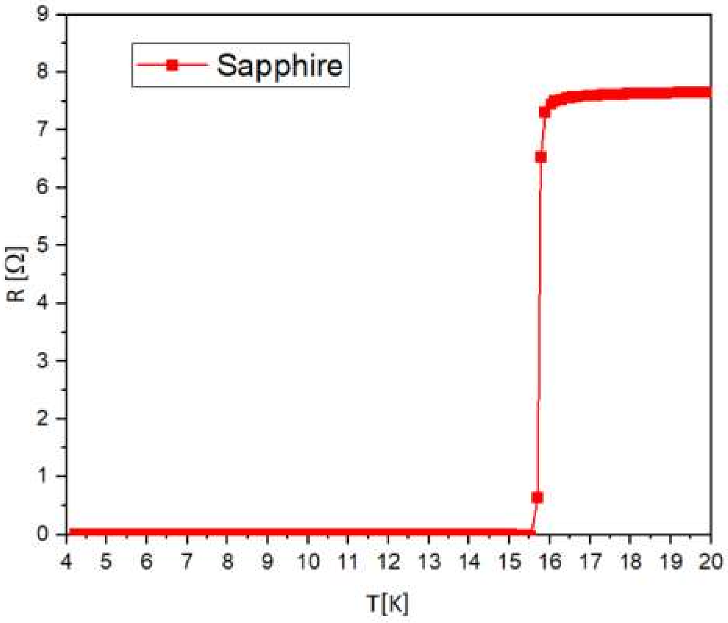
To enhance the superconducting performances of NbTiN films, several thermal treatments have been tested. The best results on Nb coated samples were obtained with:

- 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

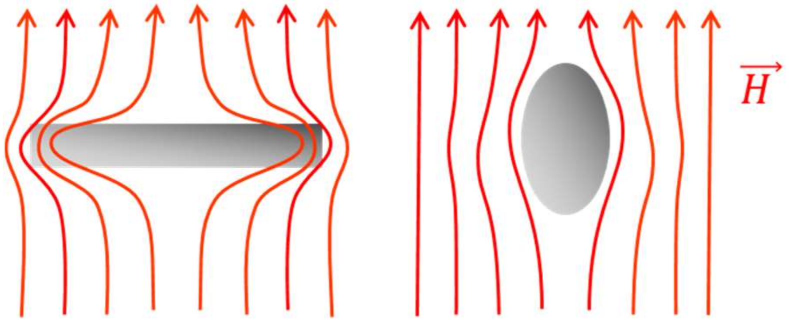


Plateforme MPBT

- T_c is similar on Niobium and Sapphire substrate.

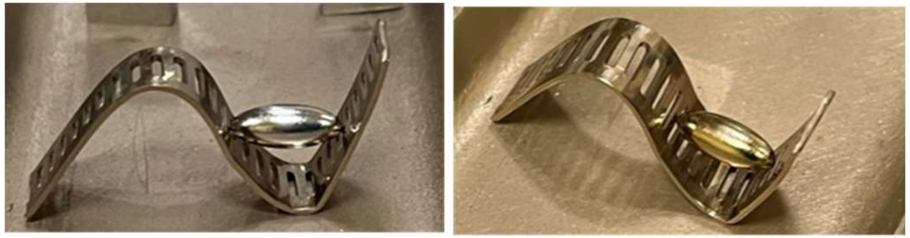
Critical field enhancement on Niobium ellipsoid

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



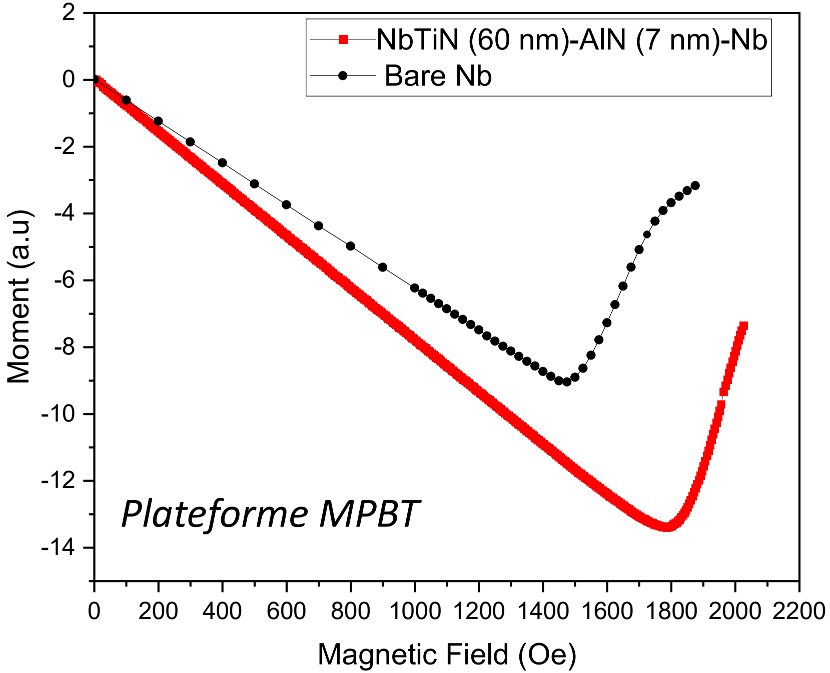
Demagnetisation factor N=0.13

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



Before After

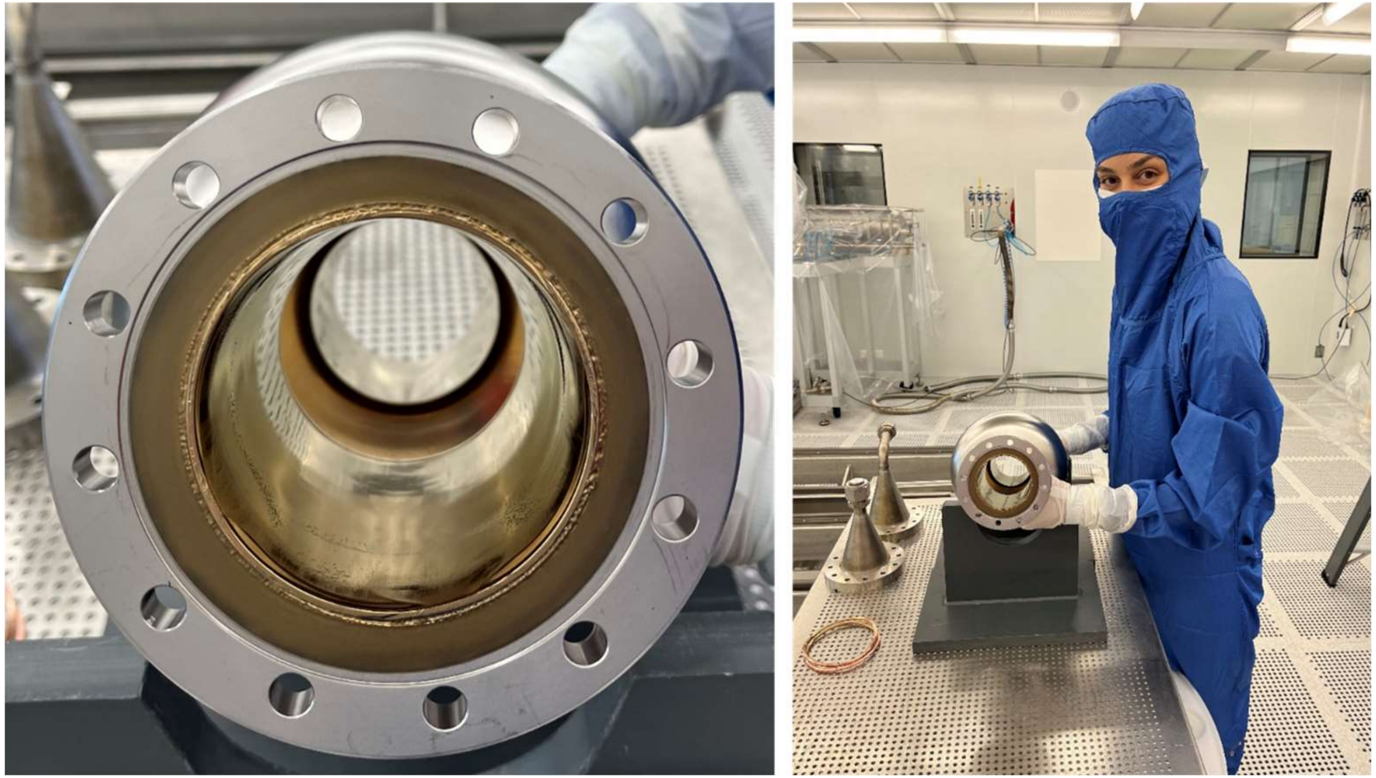
University of Victoria



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

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 .



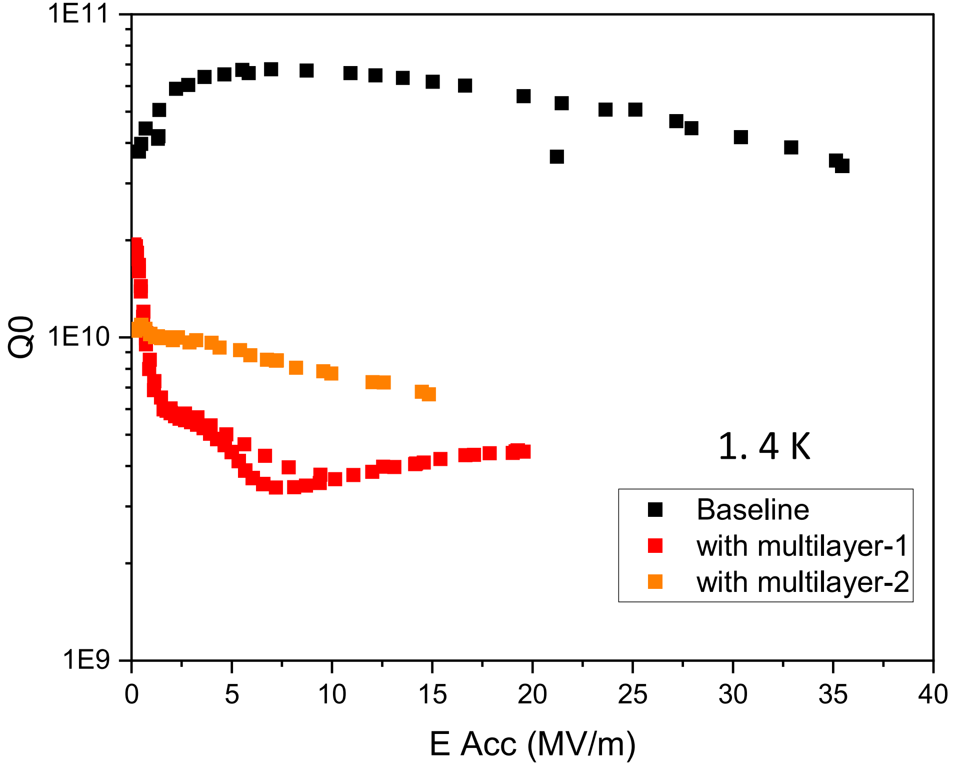
- Coating had a bright golden and uniform colour.
- The cavity was annealed @ 900°C.
- Vacuum degradation during the annealing step on the first test. ($P>10^{-5}$ mbar)

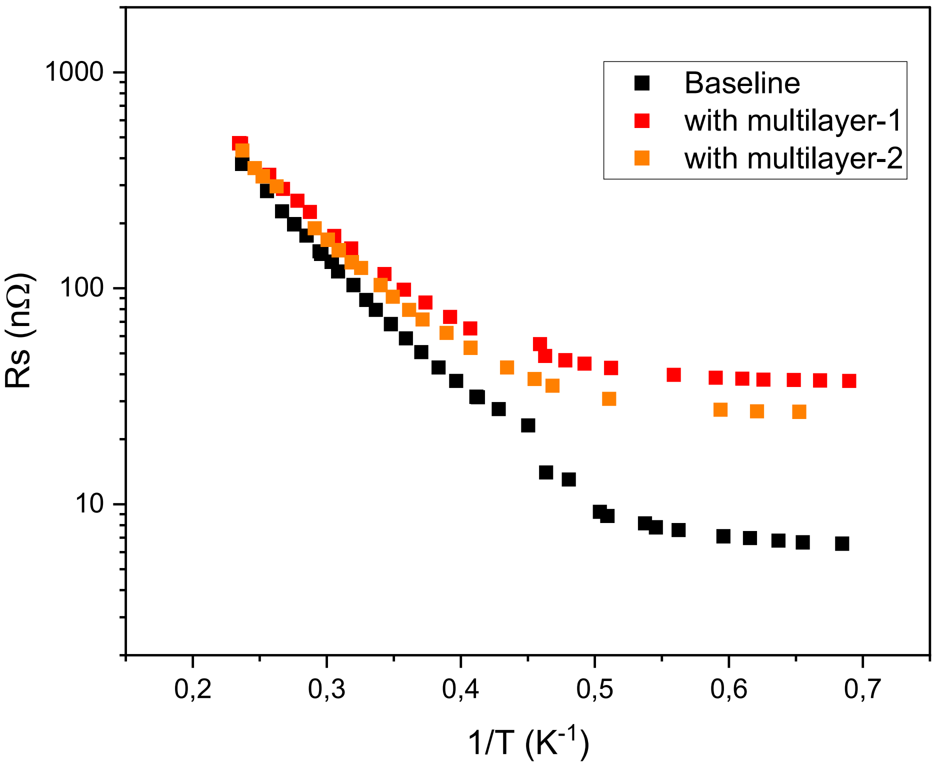
- Observed delamination in the beam tubes after annealing.

➢ 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 .





Thermal treatment multilayer 2: IJClab

➢ More investigations are ongoing (Q_0 vs T) …

Summary

- ✓ We manage to deposit uniformly a thin film of Alumina and reduce drastically niobium native oxides.
- ✓ Significant improvement of the Q_0 under low Fields.
- ✓ Proof of multipacting mitigation in SRF cavity using ALD-deposited TiN film.
- ✓ Interesting results with N-doping using ALD-deposited NbN films as dopant source.
- ✓ 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⟩

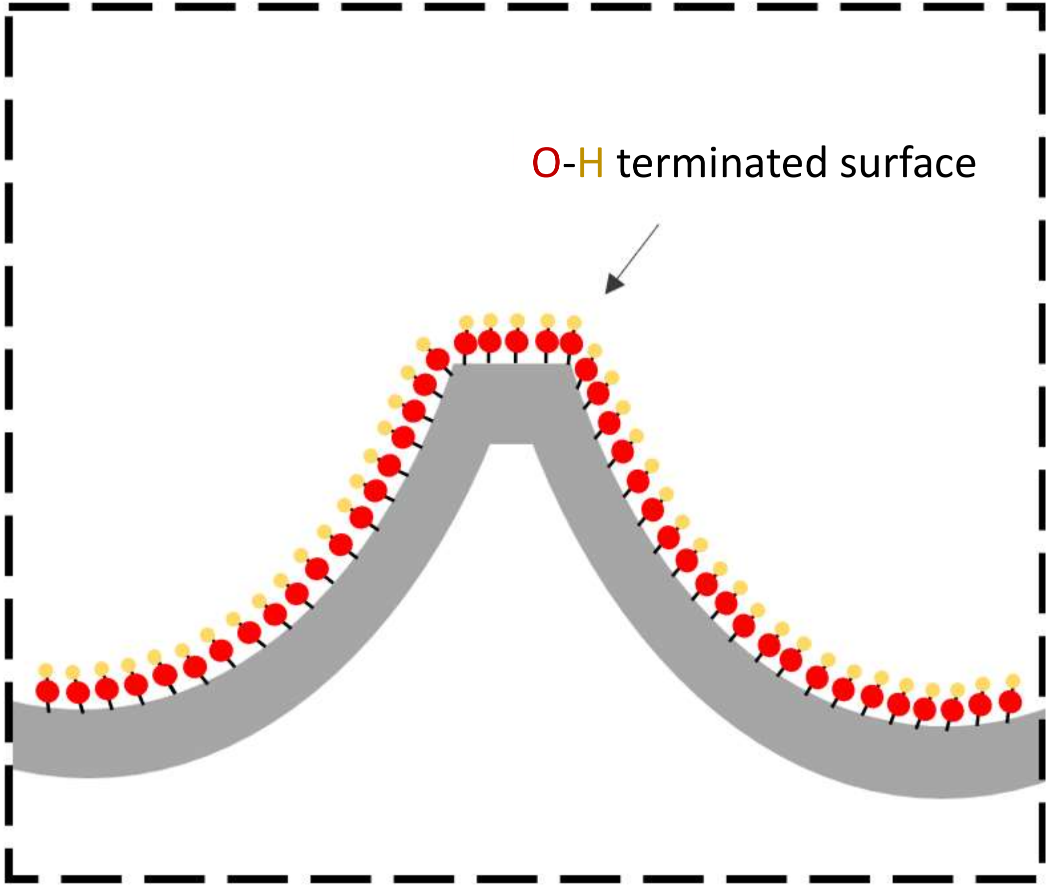
Thank you for your attention

Questions ?

Ackowledgements: RF measurements – M. Baudrier, L. Maurice; Cavity preparation: G. Jullien, F. Eozenou; Technical assistance: G. Monnereau, T. Vacher.

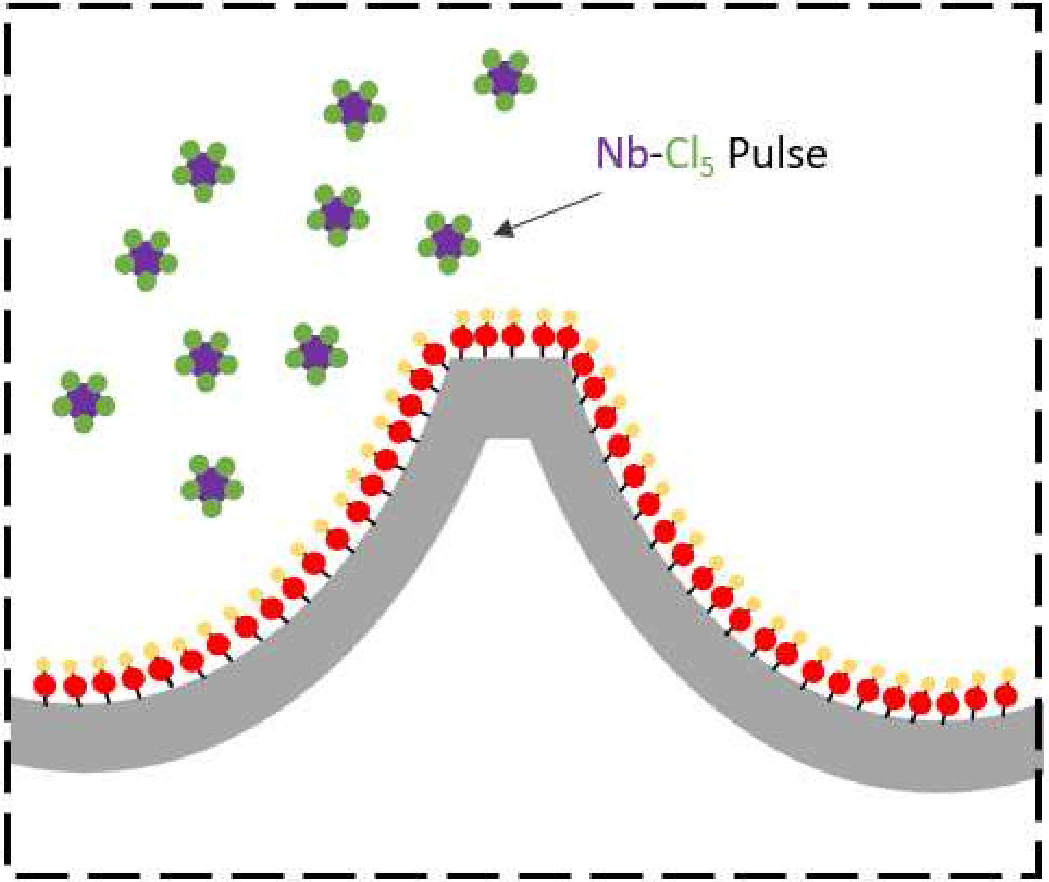
Back up

Atomic layer deposition



An ALD cycle is composed of four steps:

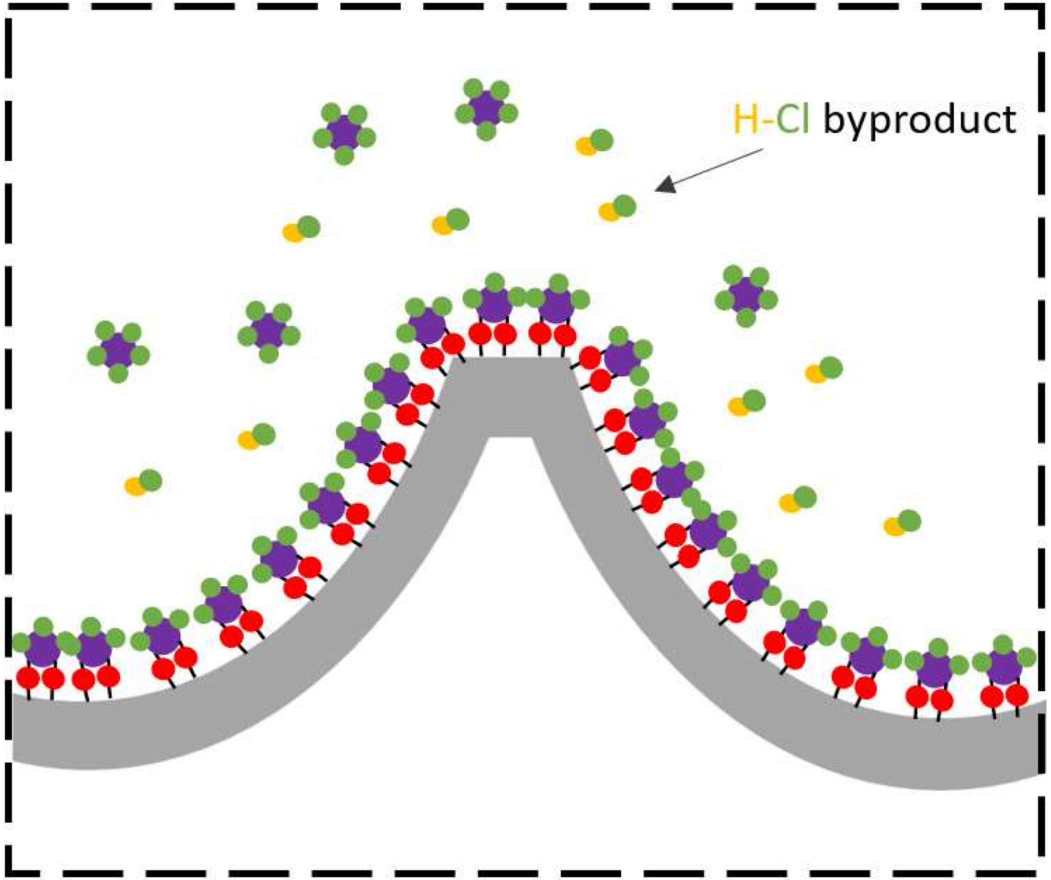
Atomic layer deposition



An ALD cycle is composed of four steps

1. Pulse of precursor 1, enabling the first gas surface reaction.

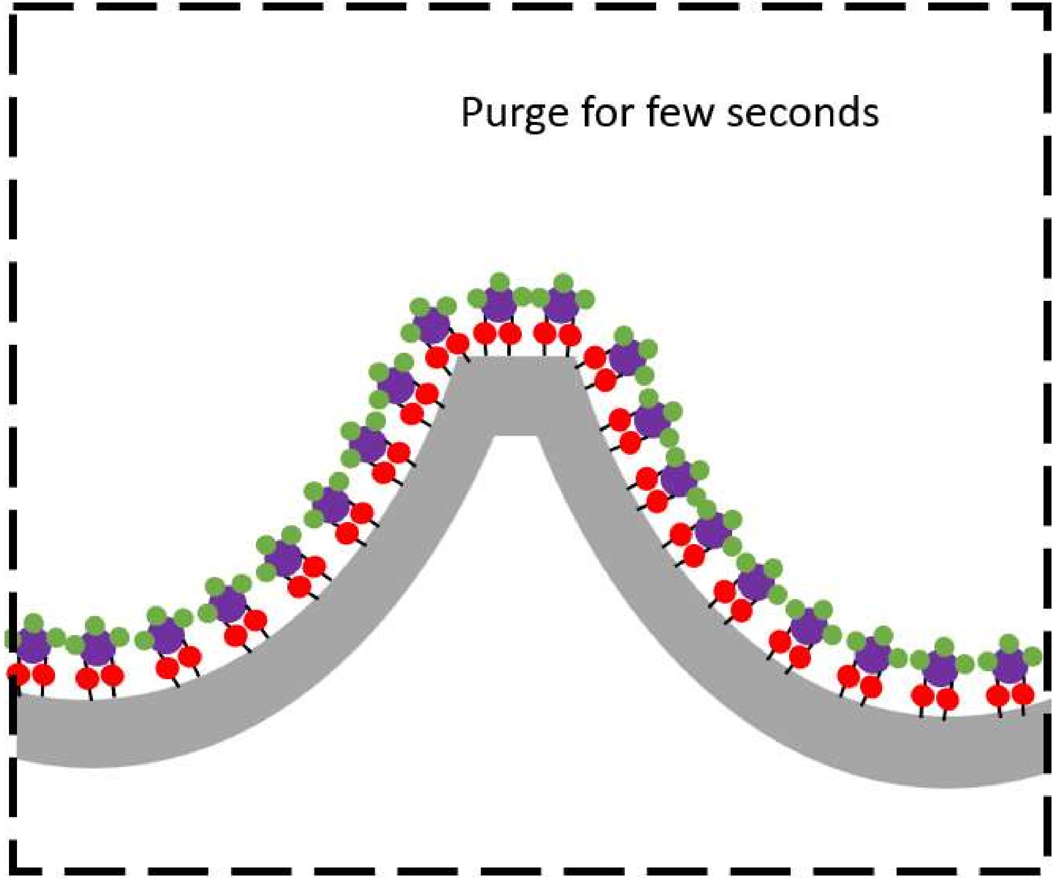
Atomic layer deposition



An ALD cycle is composed of four steps

1. Pulse of precursor 1, enabling the first gas surface reaction.

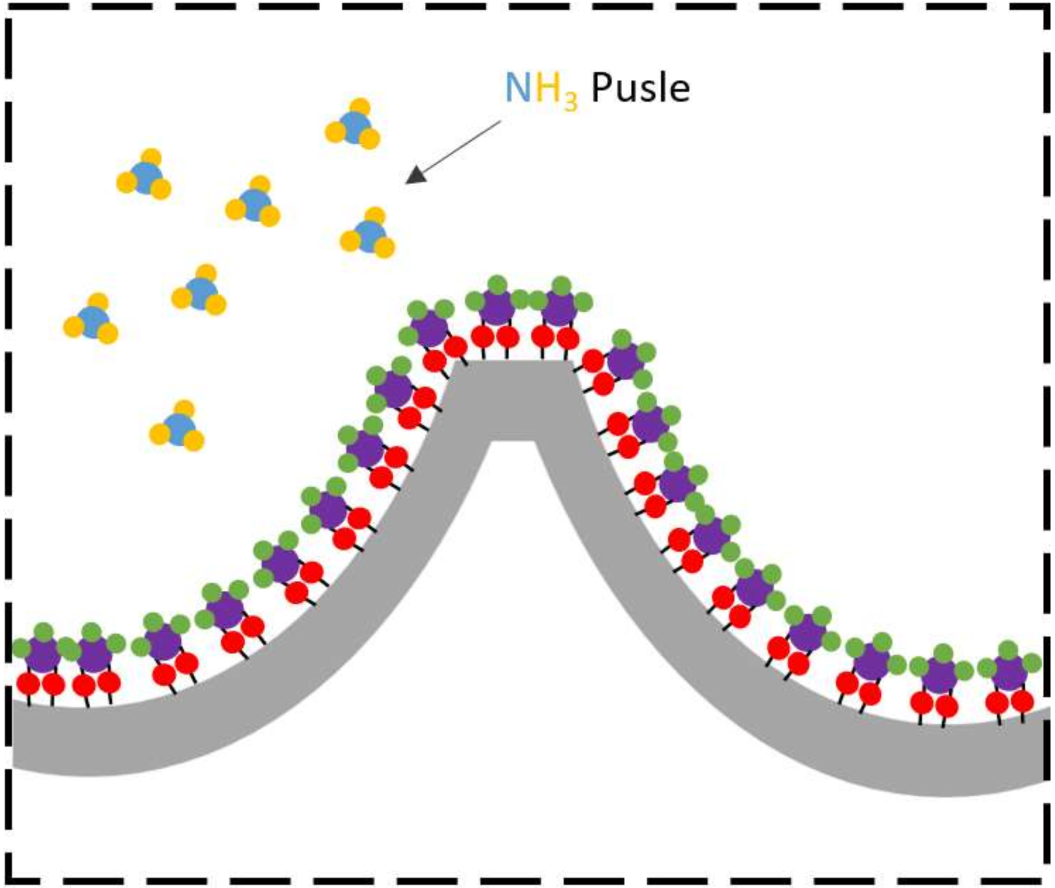
Atomic layer deposition



An ALD cycle is composed of four steps

1. Pulse of precursor 1, enabling the first gas surface reaction.
2. Purge of precursor 1, enabling the evacuation of the excess of precursor 1 and the by-products.

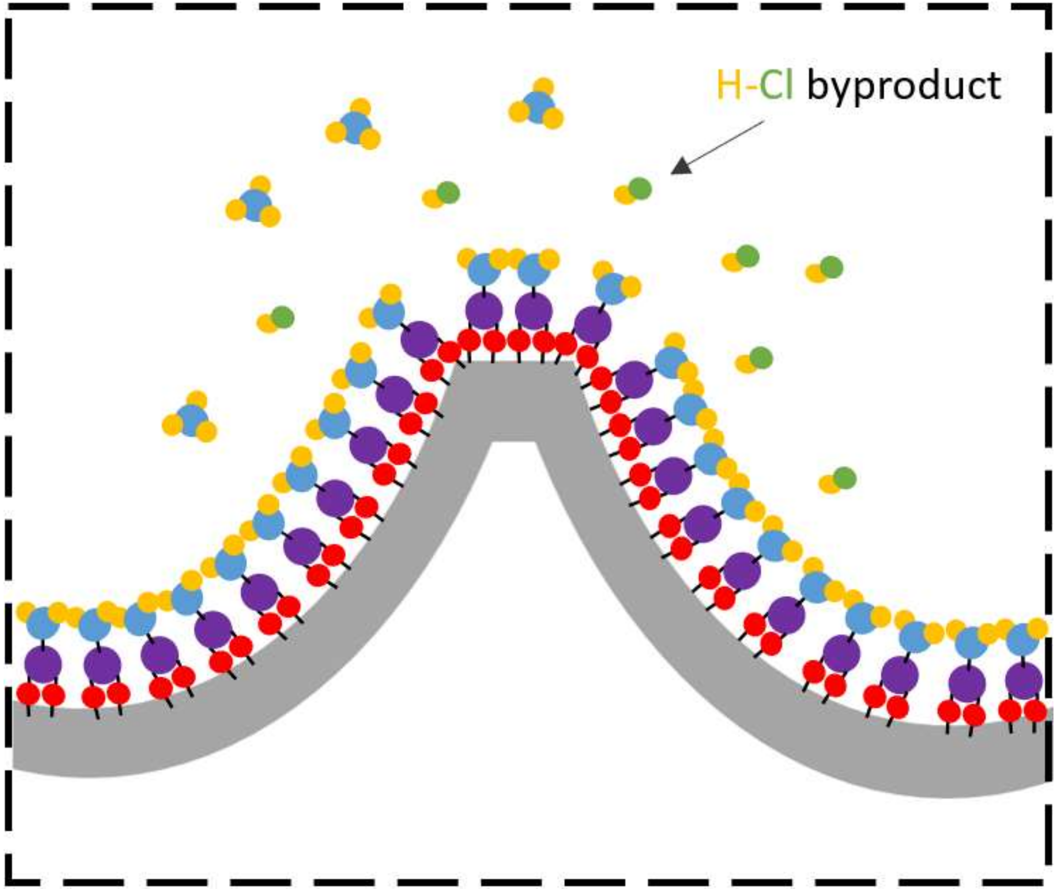
Atomic layer deposition



An ALD cycle is composed of four steps

1. Pulse of precursor 1, enabling the first gas surface reaction.
2. Purge of precursor 1, enabling the evacuation of the excess of precursor 1 and the by-products.
3. Pulse of precursor 2, enabling the second gas-surface reaction.

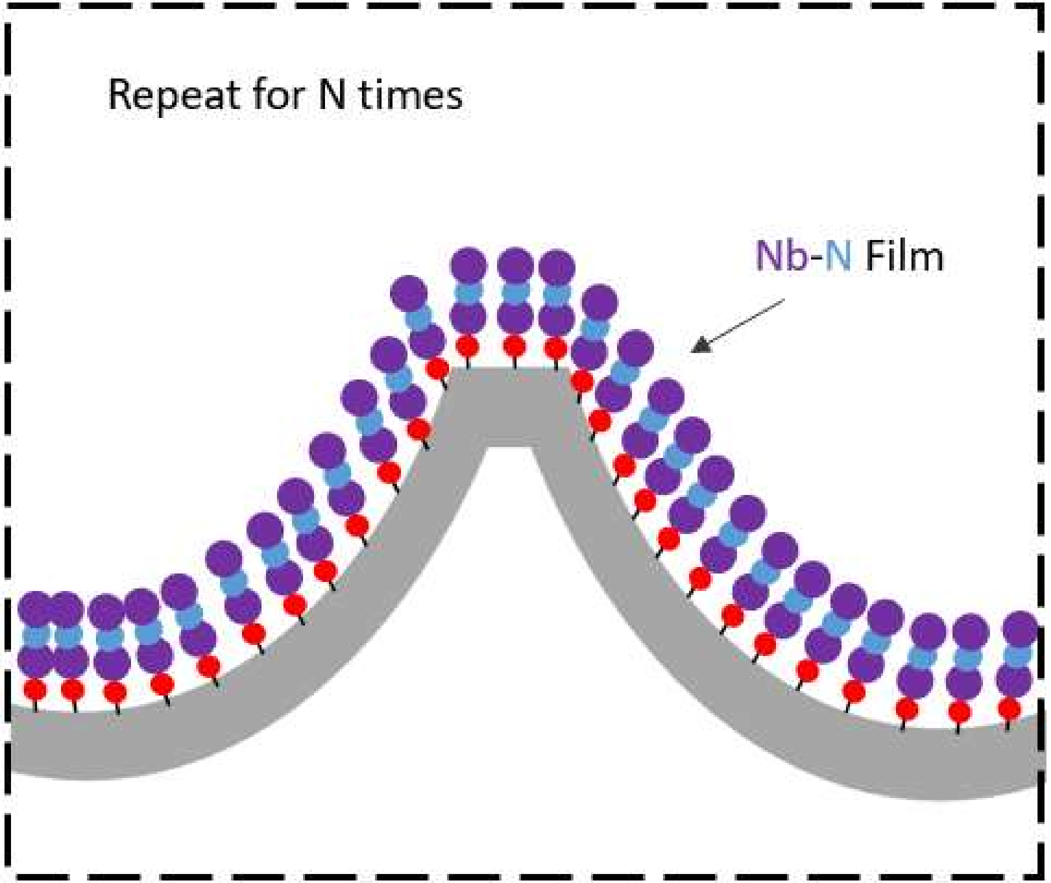
Atomic layer deposition



An ALD cycle is composed of four steps

1. Pulse of precursor 1, enabling the first gas surface reaction.
2. Purge of precursor 1, enabling the evacuation of the excess of precursor 1 and the by-products.
3. Pulse of precursor 2, enabling the second gas-surface reaction.

Atomic layer deposition



An ALD cycle is composed of four steps

1. Pulse of precursor 1, enabling the first gas surface reaction.
2. Purge of precursor 1, enabling the evacuation of the excess of precursor 1 and the by-products.
3. Pulse of precursor 2, enabling the second gas-surface reaction.
4. Purge of precursor 2, enabling the evacuation of the excess of precursor 2 and the by-products.

One monolayer of the film is deposited after each cycle.