Atomic Layer deposited thin coatings for Secondary Electron Emission yield optimization

Mathieu LAFARIE
PhD student 2nd year
ONERA/DPHY/CSE
CEA/DRF/IRFU/DAMC/LIDC2

Yasmine KALBOUSSI
PhD 2023
Researcher
CEA/DRF/IRFU/DAMC/LIDC2

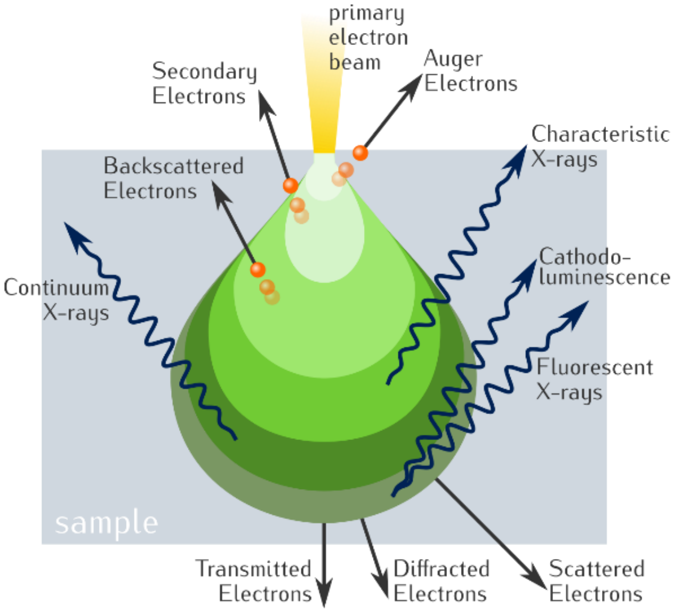
Thesis director: Mohamed BELHAJ ONERA
Thomas PROSLIER CEA

Support : ONERA/CEA

Baptiste Delatte[1], Claire Antoine[1], Diana Dragoe[2], Jocelyne Leroy[3], Sandrine Tusseau-Nenez[4], Fréderic Miserque[5], Sarra Bira[6], David Longuevergne[6], Y. Zheng, L. Maurice[1], E. Cenni[1], Q. Bertrand[1], P. Sahuquet[1], E. Fayette[1], G. Jullien[1],Christophe Inguimbert[7], Mohamed Belhaj[7],Thomas Proslier[1].
[1]IRFU, [2]ICMMO, [3]IRAMIS, [4]Polytechnique, [5]DEN, [6]IJClab, [7]ONERA.

The problems induced by electronic emission yield

Electron – matter interactions



Electron – matter interactions

Peter W. Hawkes, John C. H. Spence, Springer Handbook of Microscopy, Springer Nature Switzerland AG 2019

SEEY = secondary electron emission yield => number of secondary electrons emitted by a surface for each incident electron of a given energy

TEEY = Total electron emission yield => number of secondary and backscattered electrons emitted by a surface for each incident electron of a given energy.

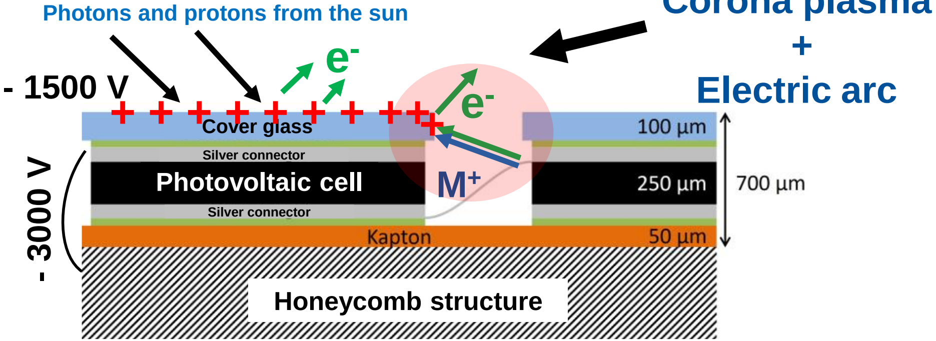
Secondary electron= low-energy electron (<50 eV) resulting from the inelastic interaction between a primary or backscattered electron and an electron in the electron cloud of one or more atoms

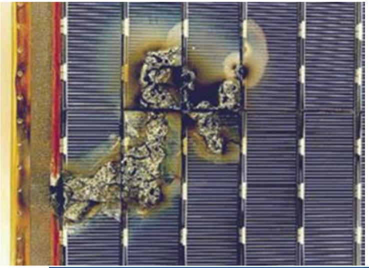
Backscattered electron= high-energy electron (>50 eV) resulting from the elastic interaction between a primary electron and the nucleus of an atom

The challenge of electronic emission yield

Electrostatic discharge on orbiting satellites

Potential difference between dielectric and conductive areas



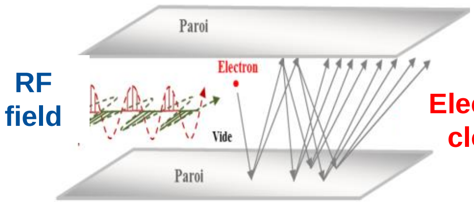


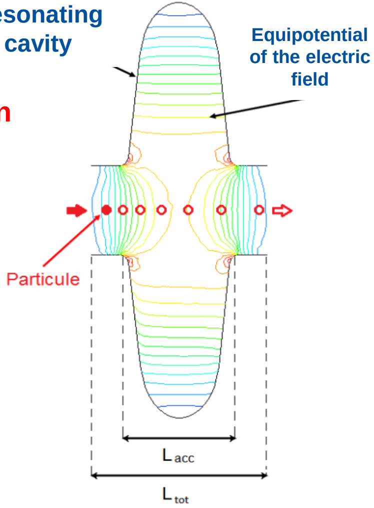
EURECA satellite solar array sustained arc, damage Credits: ESA

Multipactor Effect in RF components

Synchronisation between electronic emission and RF field

Exponential dependency on TEEY





Solution :

To use Atomic Layer Deposited coatings with tunable TEEY and electrical conductivity

Atomic Layer Deposition : principes

L'Atomic Layer Deposition (ALD) est une technique de dépôt chimique en phase gazeuse basée sur des réactions séquentielles gaz-surface auto-saturantes.

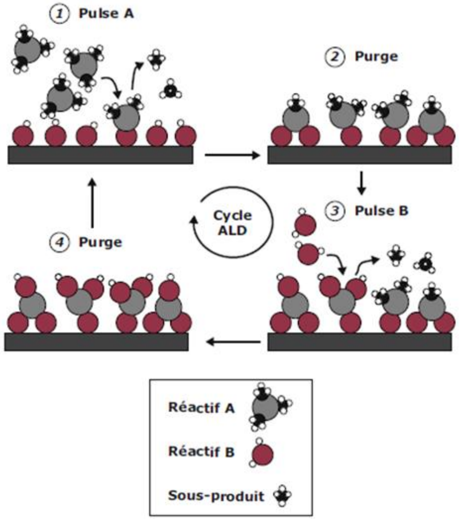


Schéma réactionnel d'un cycle ALD

Avantages

Grande variétés de matériaux déposables

Revêtements uniforme même sur surfaces complexes

Grandes gammes d'épaisseurs possibles

Contrôle relativement simple du processus

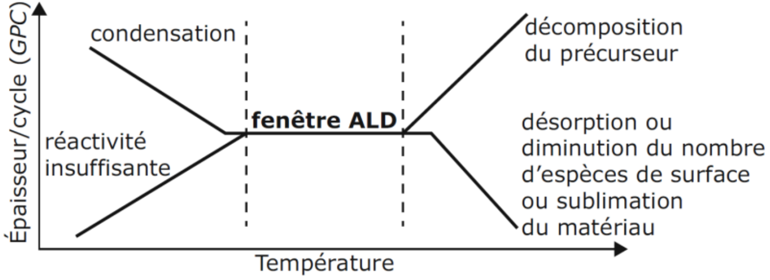
Très bonne répétabilité

Inconvénients

Temps de dépôt potentiellement très long

Propriétés des revêtements variables en fonction du nombre de cycles ALD

Nécessité de traiter des réactifs et des produits de réaction dangereux



Relation entre le taux de croissance par cycle et la température

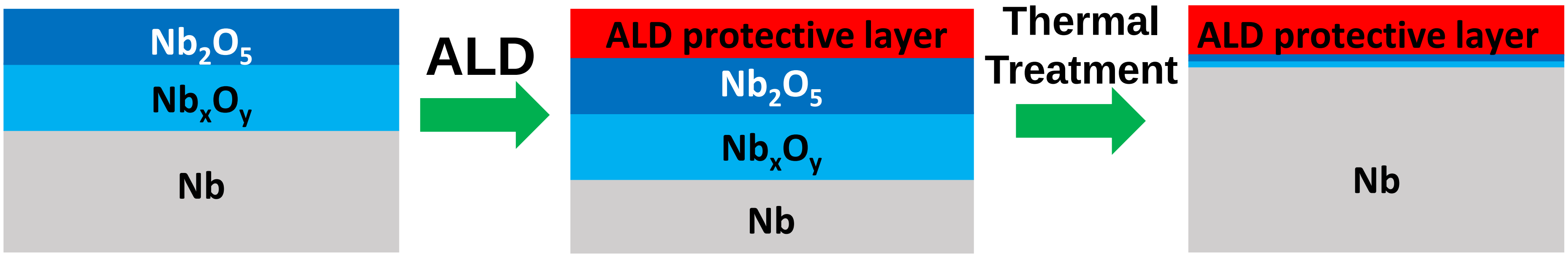
Al_2O_3 / TiN coatings for Multipacting mitigation
1- Thickness dependence

Niobium native oxyde supression

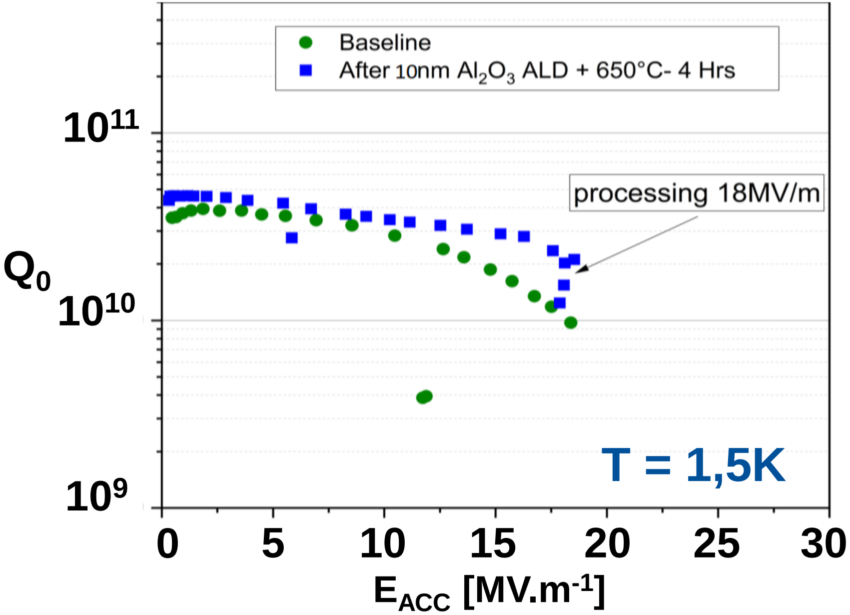
- Niobium oxidizes naturally in air
- Oxidized niobium contains impurities (two level systems) that absorb a part of the RF power
  - Leads to a diminution of quality factor
  - Limits applicable RF intensity in cavities
  - Also problematic for Q-bits application

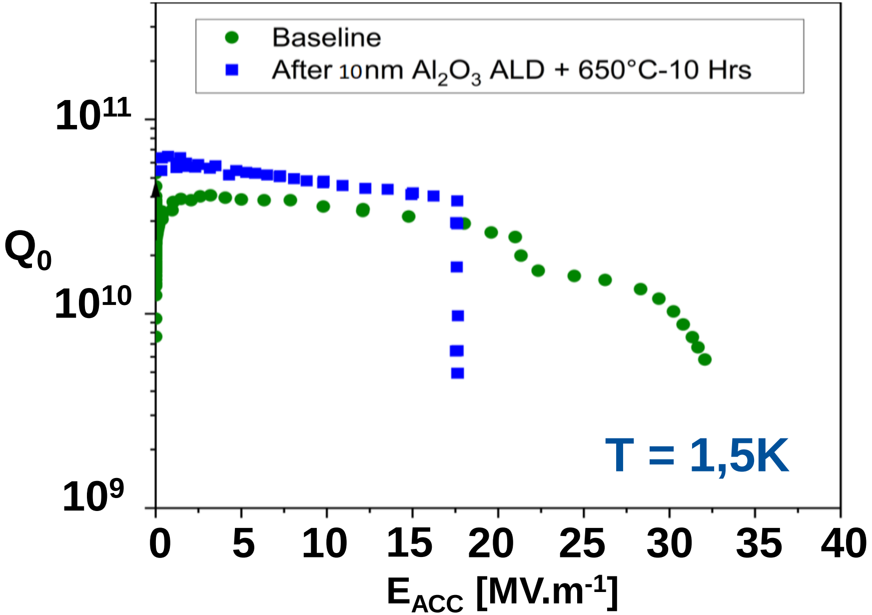
Y. Kalboussi presentation

A possible solution is to coat the oxydized niobium with a protective, low TEEY layer and then to thermal treat the coated cavity to reduced the niobium oxydes with a controled oxygen diffusion in the Nb bulk



RF test on Al_2O_3 coated Niobium cavities

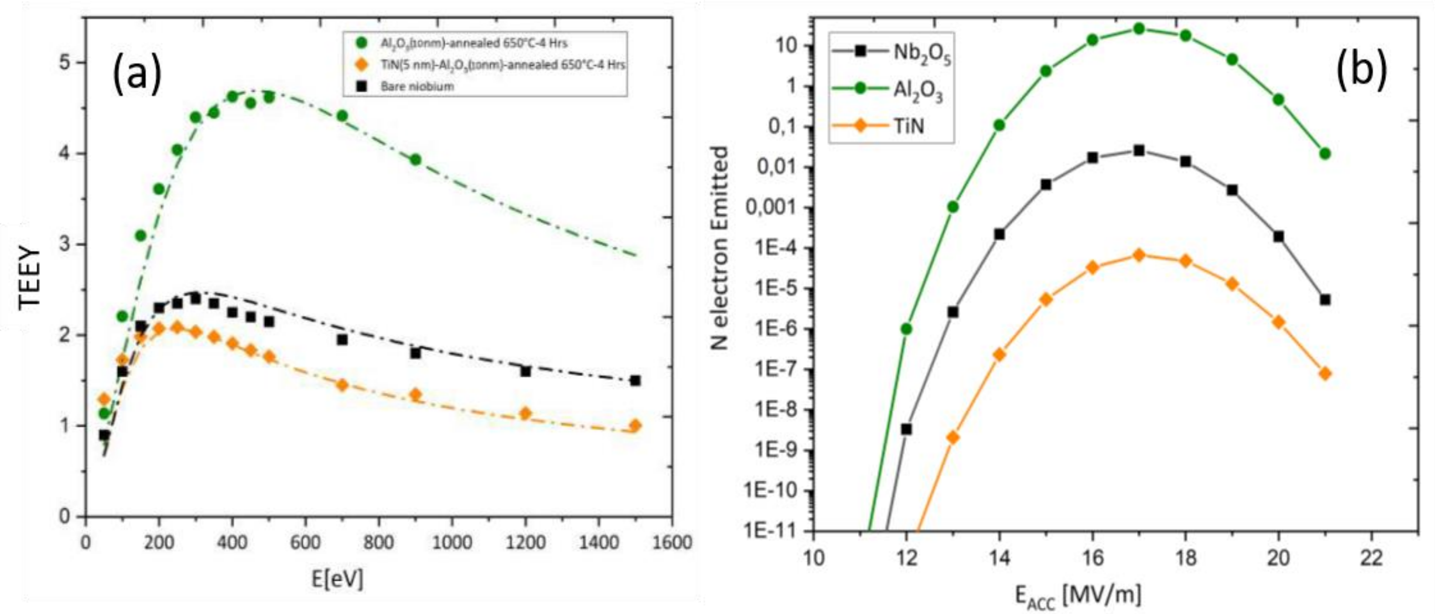




Effects of a 10nm Al_2O_3 coating on the cavity surface :

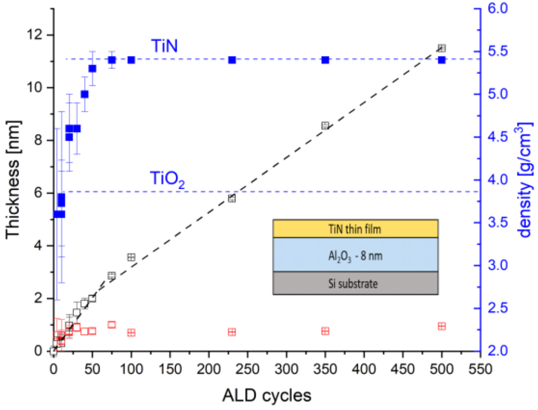
- **Improvement in quality factor for high fields**
- **Multipacting barrier at 18 MV.m^{-1}**

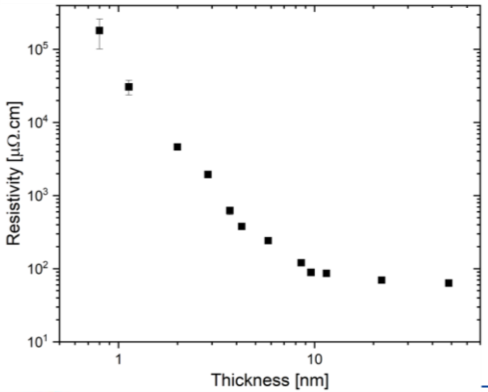
What material choice? TiN

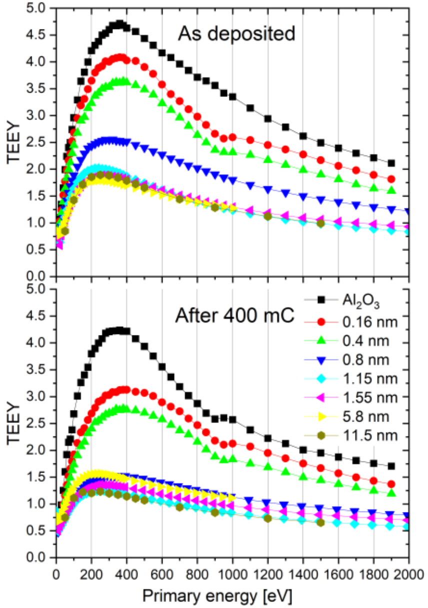


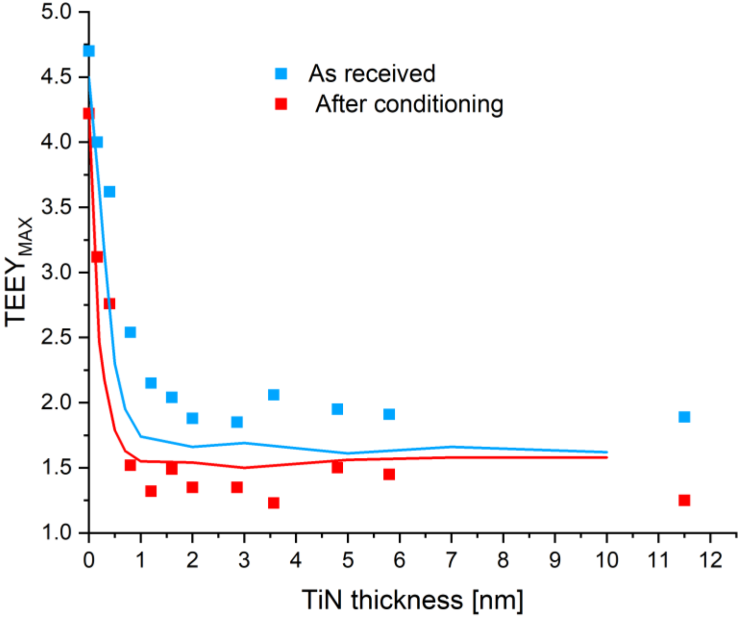
- Al_2O_3 is the cause for the multipacting barrier encountered.
- TEEY and simulation indicate that TiN is a good candidate.

Multipacting: TiN on coupons.









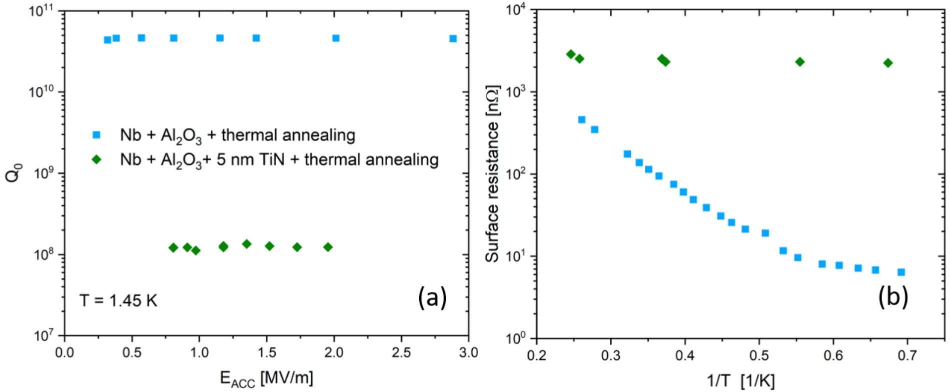
- TEEY measurements and simulations done at ONERA
- TiN initial growth rate up to 50 cy is faster (0.4 Å/cy) -> nucleation regime
- ALD thickness control -> TEEY and conductivity tuning.

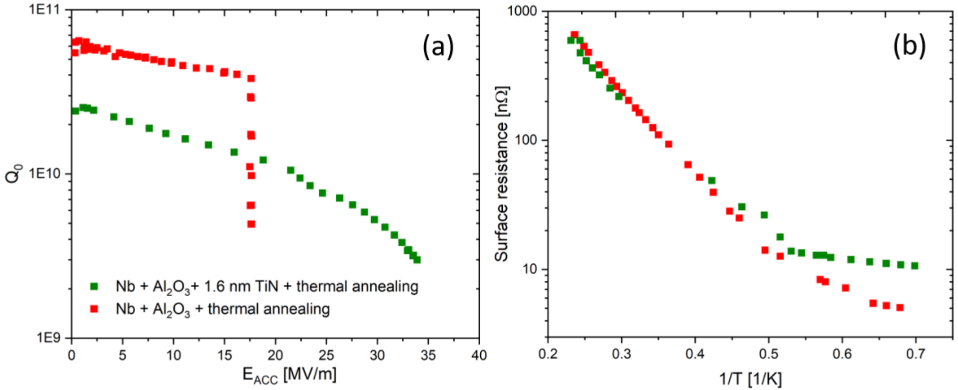
RÉPUBLIQUE FRANÇAISE
Liberté
Égalité
Fraternité

ONERA
THE FRENCH AEROSPACE LAB

cea

Multipacting: TiN on Nb SRF cavities.

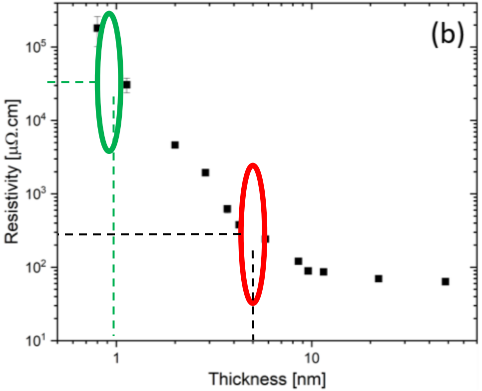


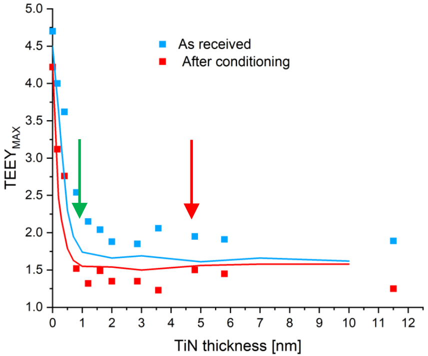


- **Metallic layer d << δ_{TiN}**

$$R_s = R_{s0} + R_{TiN} \qquad R_{TiN} = \mu_0^2\omega^2\lambda^2\sigma_{TiN} d$$

- d_{TiN} = 1.6 nm, ρ=3.10^4 µΩ.cm -> Rs ~ 2 nΩ





- **Reduced dissipation -> increase Q by 2 orders of magnitude**
- **Recover the E_{MAX} before ALD coatings**
- **Thickness control by ALD -> tuning of conductivity and TEEY**

A. Gurevich, T. Kubo. 2017, Phys. Rev. B, Vol. 96, p. 184515

Multilayered ZnO/MgO coatings

2- Chemical composition control

Multilayred ZnMgO coatings : introduction

- **ZnO :**
  - **- SEEY = 2**

    Xiangping Zhu et al., *Theoretical and experimental investigation of secondary electron emission characteristics of ALD-ZnO conductive films*, J. Appl. Phys. 128, 065102 (2020)

  - **- Conductivity = 7,1 10^3 $\Omega^{-1}.m^{-1}$**

    W.J. Jeong et al., *Preparation and characteristic of ZnO thin film with high and low resistivity for an application of solar cell,* Thin Solid Films 506 – 507 (2006) *180 – 183*

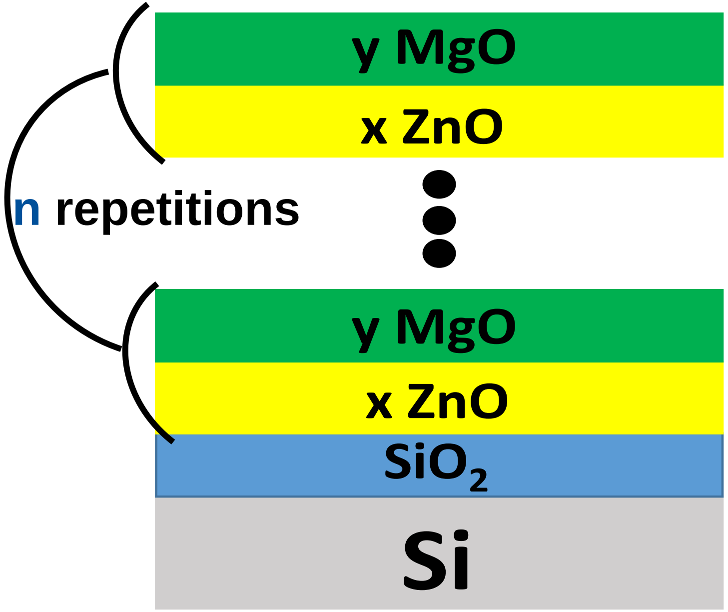
- **MgO :**
  - **- SEEY = 6,2**

    J. Guo, et al., *Theoretical and experimental investigation of secondary electron emission characteristics of MgO coating produced by atomic layer deposition* ,Ceramics International 46 (2020) 8352–8357

  - **- Conductivity = 10^{-15} $\Omega^{-1}.m^{-1}$**

    H. KATHREIN and F. FREUN,*Electrical conductivity of magnesium oxide single crystal below 1200 K*, J. Phys. Chem. Solids Vol 44. No. 3. pp 177-186. 1983

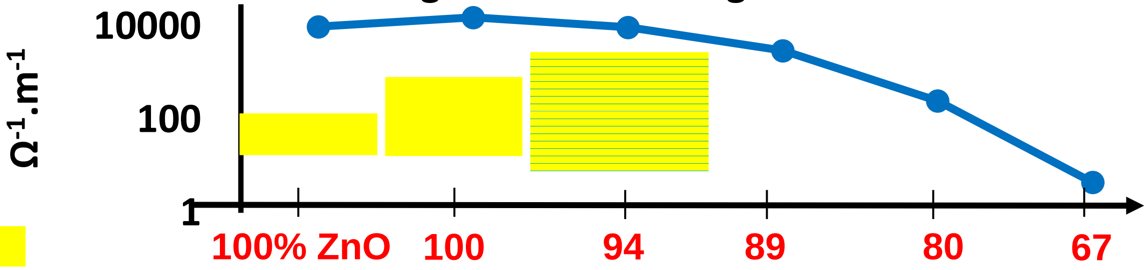
- **Common properties :**
  - **Metallic oxydes**
  - **Chemically stable**
  - **good transparency in the visible range**
  - **Similar Growth Per Cycle**



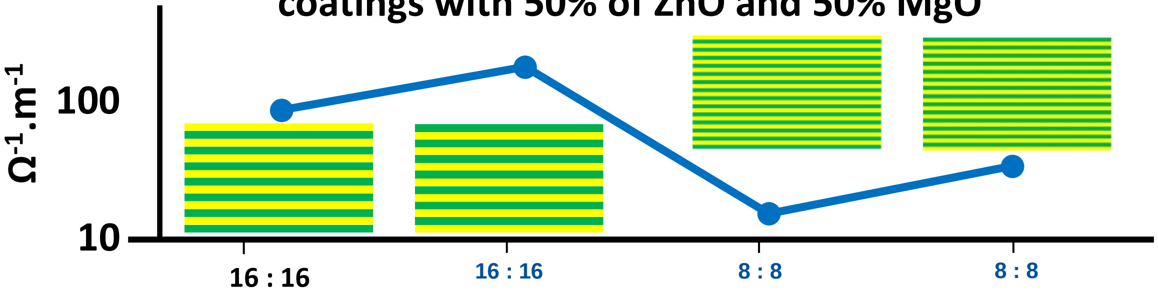
schematic diagram of a multilayer coating
x ZnO / y MgO x n

Multilayered ZnMgO coatings : conductivity

Electrical conductivity of multilayered ZnMgO coatings with decreasing ZnO content

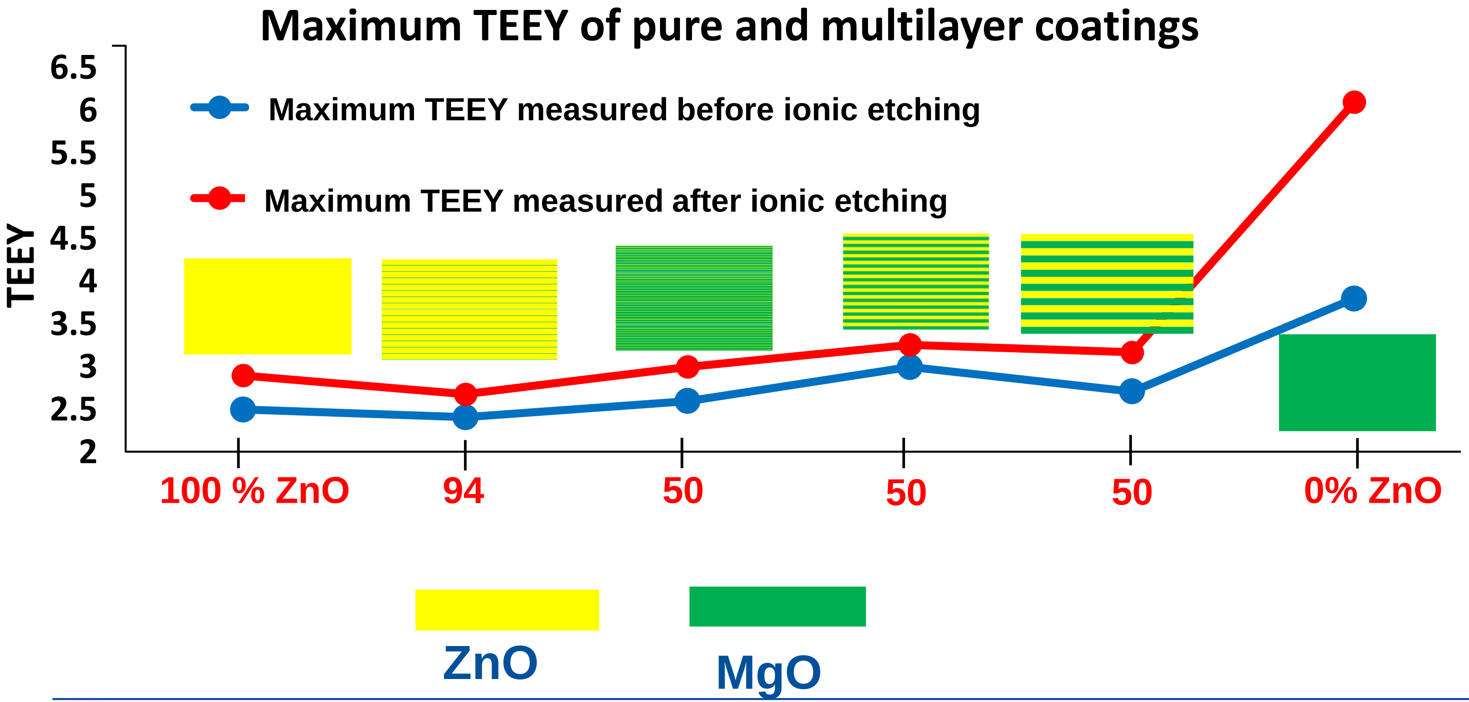


Electrical conductivity of multilayered ZnMgO coatings with 50% of ZnO and 50% MgO



The chemical composition and stacking structure of materials can modulate the coating conductivity

Multilayred ZnMgO coatings : TEEY



The chemical composition and stacking structure of materials can modulate the TEEY

New materials : why? How?

Why ?

- Extend the modulation range of TEEY and electrical conductivity of coatings

How ?

- Select a new material with a lower TEEY and more conducting than ZnO

New material: TiC

TiC : Carbide

Synthesis : TMA + $TiCl_4$

Jinjuan Xiang et al, *Investigation of TiAlC by Atomic Layer Deposition as N Type Work Function Metal for FinFET*, 2015 ECS J. Solid State Sci. Technol. 4 P441

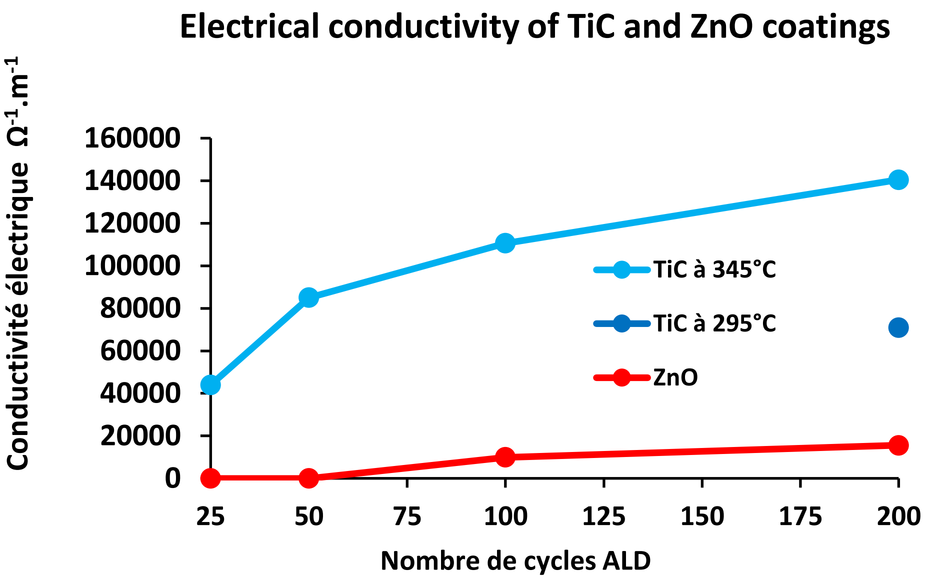
SEEY = 0,9 (CVD thin film)

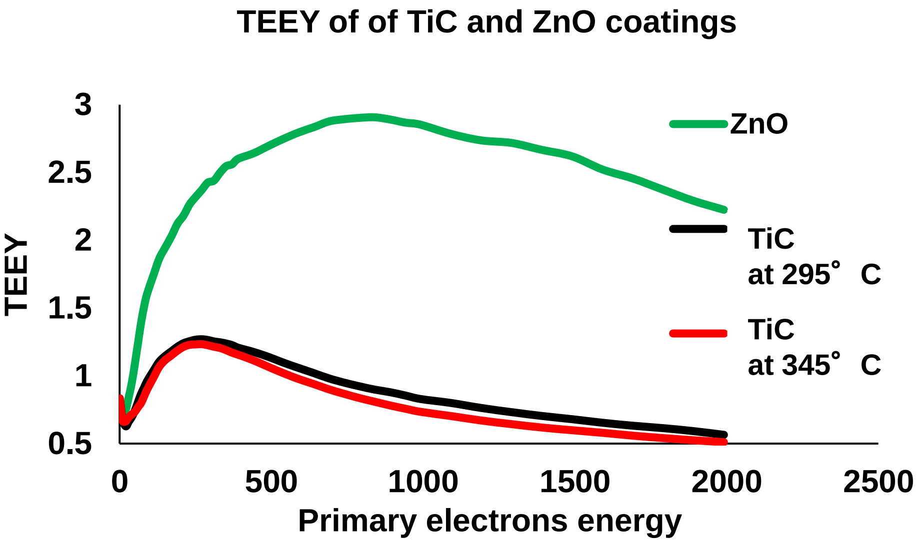
Emilio Franconi, *SECONDARY ELECTRON YIELD OF GRAPHITE AND TiC COATINGS*, FUSION TECHNOLOGY VOL. 6 SEPTEMBER 1984

Electrical conductiviyt= $10^5\ \Omega^{-1}.m^{-1}$ (400° C, 20nm)

J. Xiang et al., *Investigation of thermal atomic layer deposited TiAlX (X = N or C) film as metal gate*,Solid-State Electronics 122 (2016) 64–69

TiC : electrical conductivity and TEEY





TEEY and electrical condctivity looks good

but

TMA starts decomposing at 345° C and TiC films unstable in air when grown at 290° C

New material: MgF_2

Synthesis : $Mg(thd)_2$ + TaF_5

Tero Pilvi et al., *Atomic Layer Deposition of MgF2 Thin Films Using TaF5 as a Novel Fluorine Source*, Chem. Mater. 2008, 20, 5023–5028

SEEY = 6,5

I. Krainsky et al., *SECONDARY ELECTRON EMISSION YIELDS*, NASA. Lewis Research Center Spacecraft Charging Technol., 1980

Conductivité électrique : Pas de données expérimentales ou théoriques dans la littérature

No risk to oxidize the TiC

Transparency > 90% between 1000 and 200 nm (as for bulk)

New materials: MgF_2

3 precursors evaluated: $Mg(thd)_2$; $Mg(Cp)_2$; $Mg(EtCp)_2$

	$Mg(EtCp)_2$		$Mg(Cp)_2$
Température de dépôt (° C)	300	345	345
$\%_{at}$ Mg	26,2	23	13
$\%_{at}$ F	45,4	43	26,6
$\%_{at}$ C	17	23	33,4
$\%_{at}$ O	9,4	9	23
$\%_{at}$ Ta	2	2	4
Ratio F/Mg	1,7	1,87	2,04

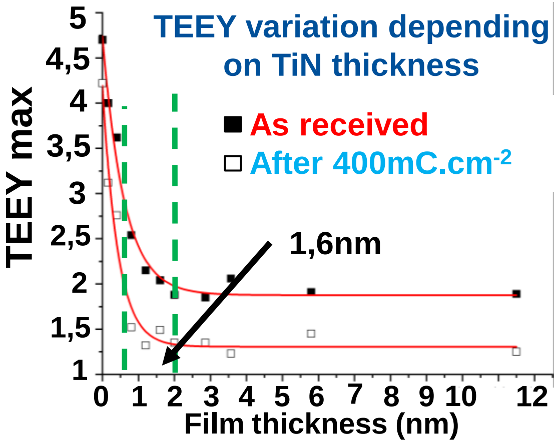
Can grow MgF2 but:
Too much Ta impurities.

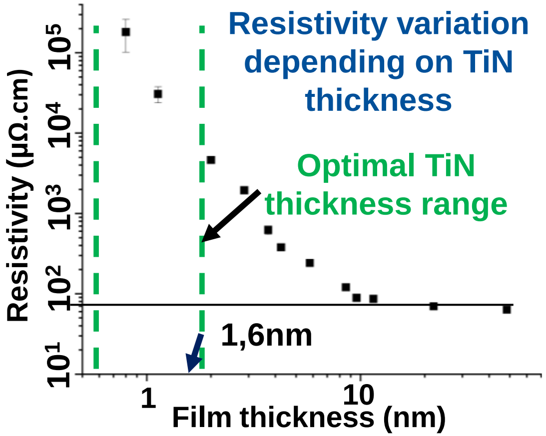
$Mg(EtCp)_2$ and $Mg(Cp)_2$ start decomposing at 345° C

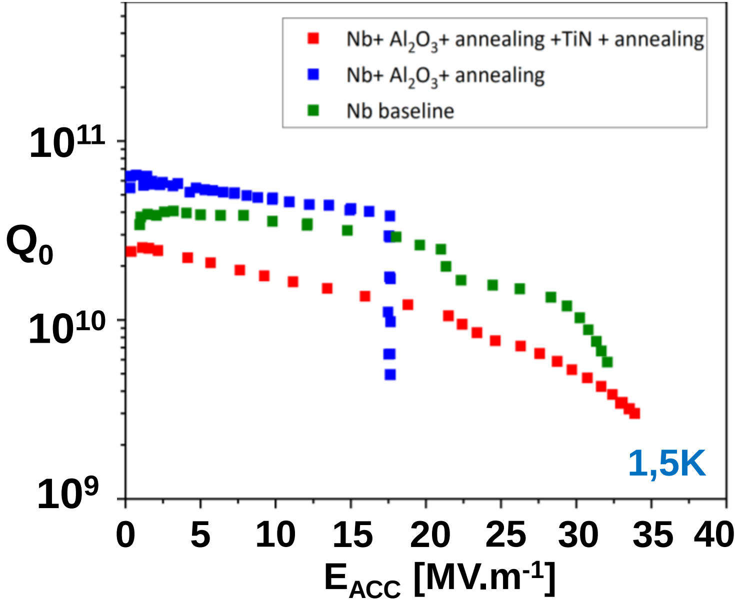
Conclusion

- **Tuning the TEEY and the electrical conductivity by controlling the thickness by ALD.**
- **Successful application to SRF cavities.**
- **Tuning the TEEY and the electrical conductivity by controlling the chemical composition by ALD: Multilayer coatings based on ZnO and MgO**
- **On going work on TiC and MgF_2.**

Effect of 40 ALD TiN cycles on Al_2O_3







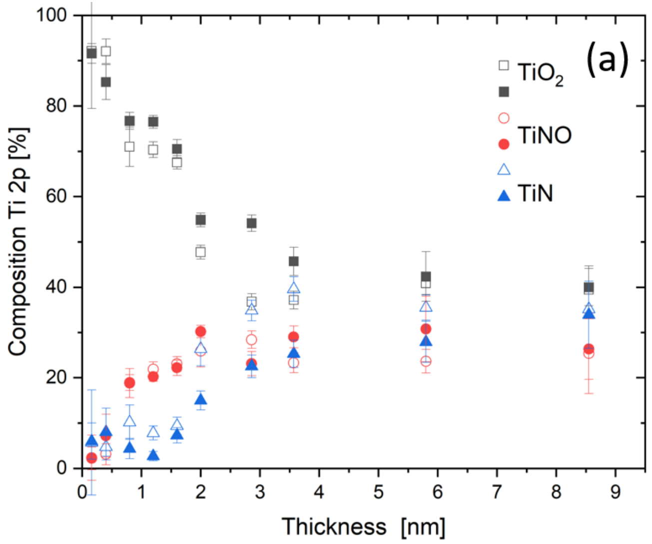
Parameters for exponential d_0 fits $y = y_1.e^{-d/d0}+y_0$			
	y_1	d_0 [nm]	y_0
As deposited	2.8 ± 0.1	0.60 ± 0.06	1.87 ± 0.06
After conditioning	2.9 ± 0.2	0.42 ± 0.06	1.30 ± 0.07

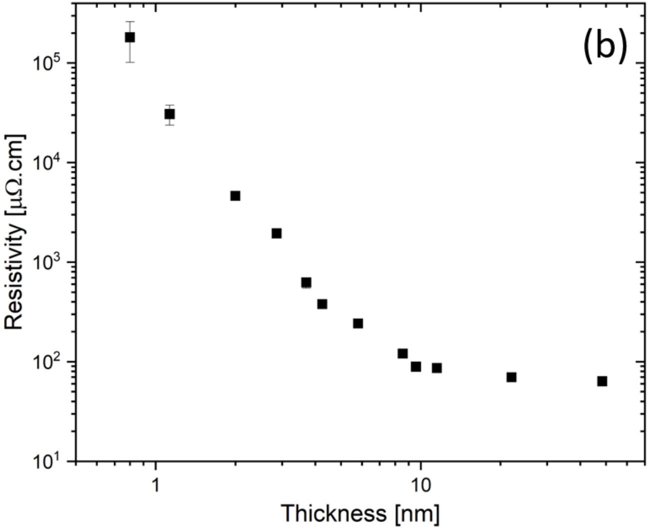
Effects of a 1,6nm TiN coating on the cavity surface :

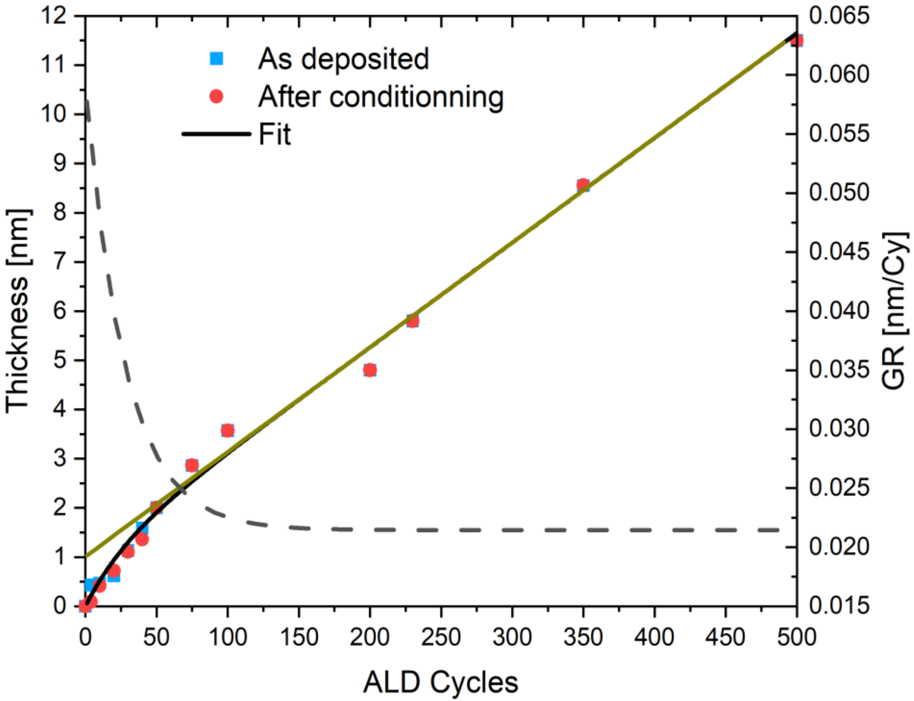
- No Multipacting barrier
- Acceptable reduction of the quality factor for some particle accelerators.

➡ **Can TEEY and electrical conductivity be modulated according to chemical composition and coating structure?**

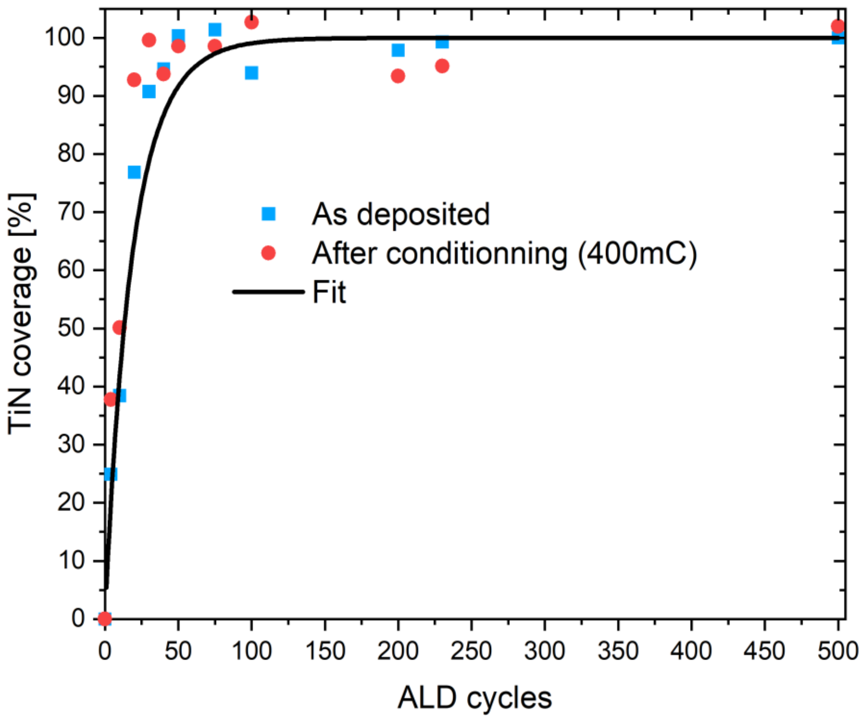
RÉPUBLIQUE FRANÇAISE
Liberté Égalité Fraternité
ONERA
THE FRENCH AEROSPACE LAB
cea


(a)
TiO$_2$
TiNO
TiN
Composition Ti 2p [%]
Thickness [nm]
100
80
60
40
20
0
0
1
2
3
4
5
6
7
8
9


(b)
Resistivity [μΩ.cm]
Thickness [nm]
10^5
10^4
10^3
10^2
10^1
1
10



As deposited
After conditionning
Fit
Thickness [nm]
ALD Cycles
GR [nm/Cy]



As deposited
After conditionning (400mC)
Fit
TiN coverage [%]
ALD cycles