# Manipulation of Nb carbide during Mid-T bake : Impact of baking configuration and plasma treatment.*

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**PhD student**

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# Thanks to :


- All the department, groups and people that help with this experiment.

- MAVERICS Team, IJCLAB
- SRF team, TRIUMF
- Vide et Surface plateform, IJCLAB
- Supratech plateform, IJCLAB
- International Research Laboratory (IRL) beetween TRIUMF and CNRS
- ANR-21-ESRE-0049
- Ile de France region

# Outline

1- Background and Motivations

2- Type of carbide formed during baking with TRIUMF furnace

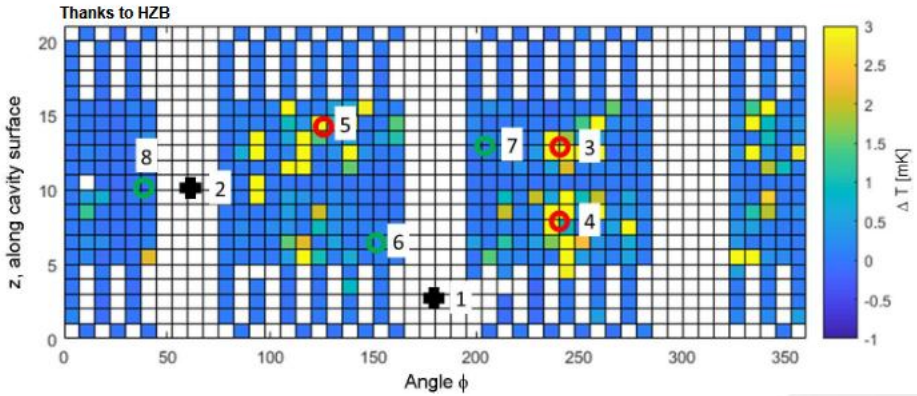
3- The origin of carbide formation

4- Limited this formation inside the cavity

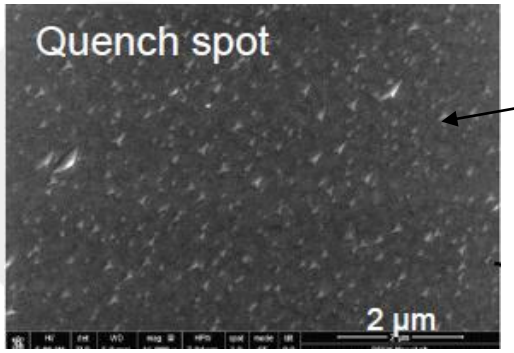
5- Summary and future outlook

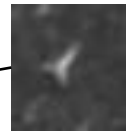
# Carbide (Nb-C) issues

Cut a cavity and look



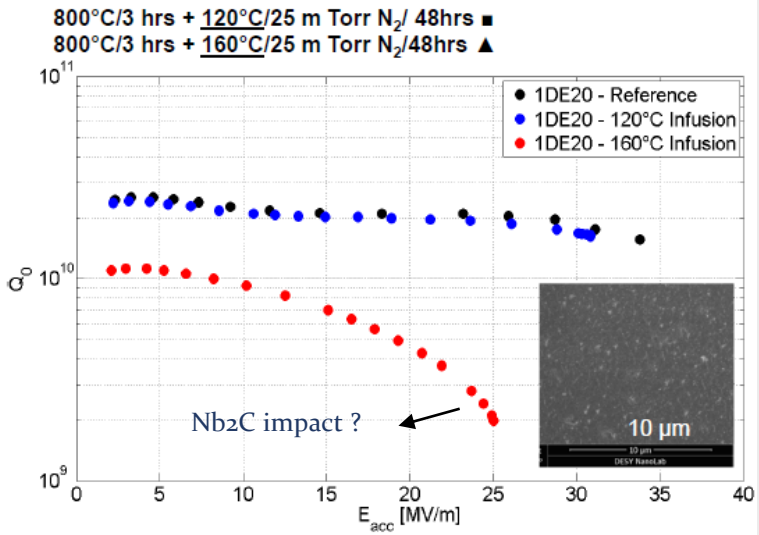

M. Wenskat | TTC Meeting @ CERN 2020



Carbide ($Nb_2C$)

Heating zone => due to the formation of carbides on the surface




## Do carbides degrade cavity performance ?

- **SEM:** carbon rich precipitates for N-infused samples at 120 °C and 160 °C
- **XPS:** more Nb-C content for infusion @ 160 °C, no Nb-N bonding observed
- **SIMS:** more C for 160 °C infusion, no NbN identified

**Higher carbide content for N-infusion at higher T could be related to failed cavity treatment**

A. Dangwal Pandey, SRF 2019, Dresden 30th June –5th July 2019

# Goal/Issues

Manipulation of Nb carbide during Mid-T bake : Impact of baking configuration and plasma treatment

**Determine the type of carbide formed during baking under UHV ($Nb_2C$, NbC) ?**

**The origin of carbon causing these carbides is still not identified:**

- **from residual gas during heat treatment**
- **from carbon layer**
- **from the bulk**

**How can we reduce this formation to minimize its impact on cavity results?**

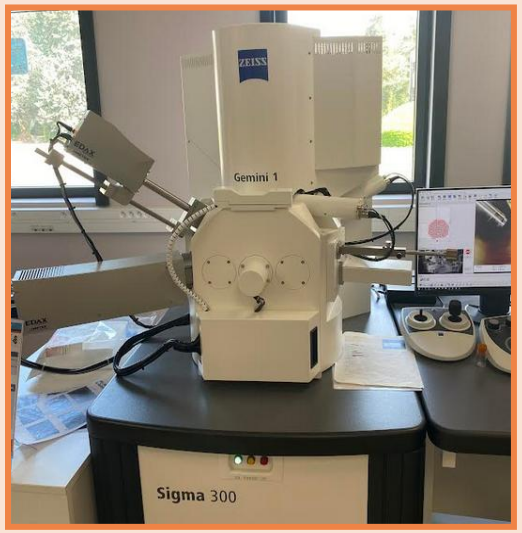
# Goal

Manipulation of Nb carbide during Mid-T bake : Impact of baking configuration and plasma treatment

**Determine the type of carbide formed during baking under UHV ($Nb_2C$, NbC) ?**

**TRIUMF induction furnace**


**XPS at IJCLab**




**SEM at IJCLab**

# Goal

Manipulation of Nb carbide during Mid-T bake : Impact of baking configuration and plasma treatment

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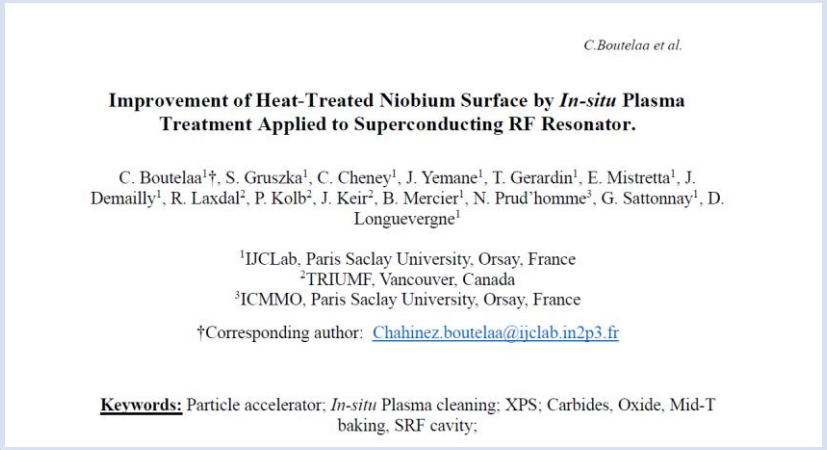
- from residual gas during heat treatment
- from carbon layer
- from the bulk



C.Boutelaa et al.

**Improvement of Heat-Treated Niobium Surface by *In-situ* Plasma Treatment Applied to Superconducting RF Resonator.**

C. Boutelaa[1]†, S. Gruszka[1], C. Cheney[1], J. Yemane[1], T. Gerardin[1], E. Mistretta[1], J. Demailly[1], R. Laxdal[2], P. Kolb[2], J. Keir[2], B. Mercier[1], N. Prud'homme[3], G. Sattonnay[1], D. Longuevergne[1]

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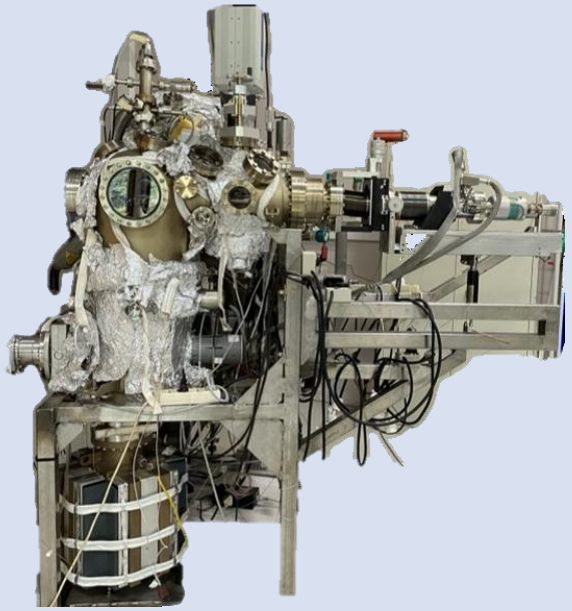
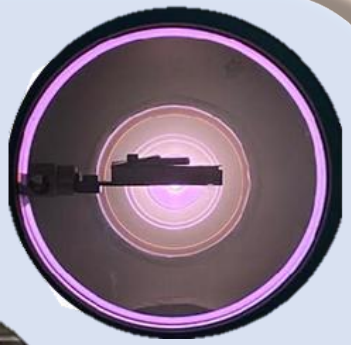
**Keywords:** Particle accelerator; *In-situ* Plasma cleaning; XPS; Carbides, Oxide, Mid-T baking, SRF cavity;





plasma chamber

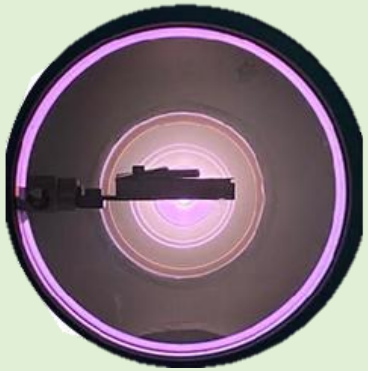
XPS and Heat chamber

XPS, heat and plasma set up at IJCLab

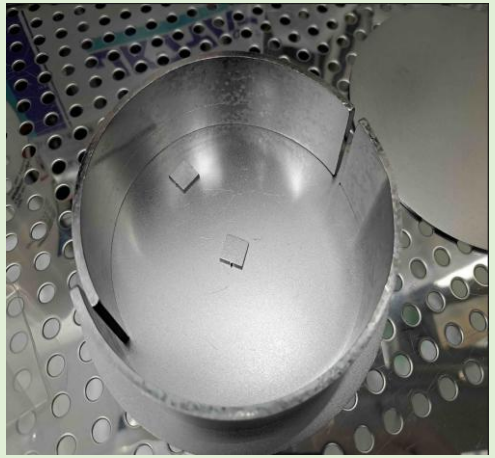
# Goal

Manipulation of Nb carbide during Mid-T bake : Impact of baking configuration and plasma treatment

**How can we reduce this formation to minimize its impact on cavity results?**



**Plasma cleaning after heat treatment**



**Nb box**

**Furnace at Supratech**

# Outline

1- Background and Motivations

**2- Type of carbide formed during baking with TRIUMF furnace**

3- The origin of carbide formation

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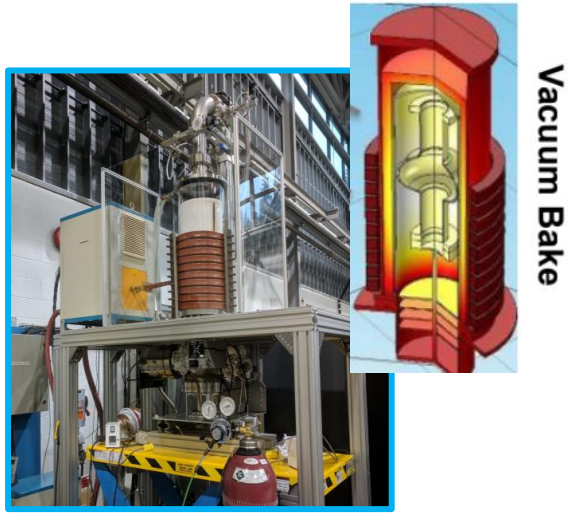
5- Summary and future outlook

# Nb sample preparation at TRIUMF

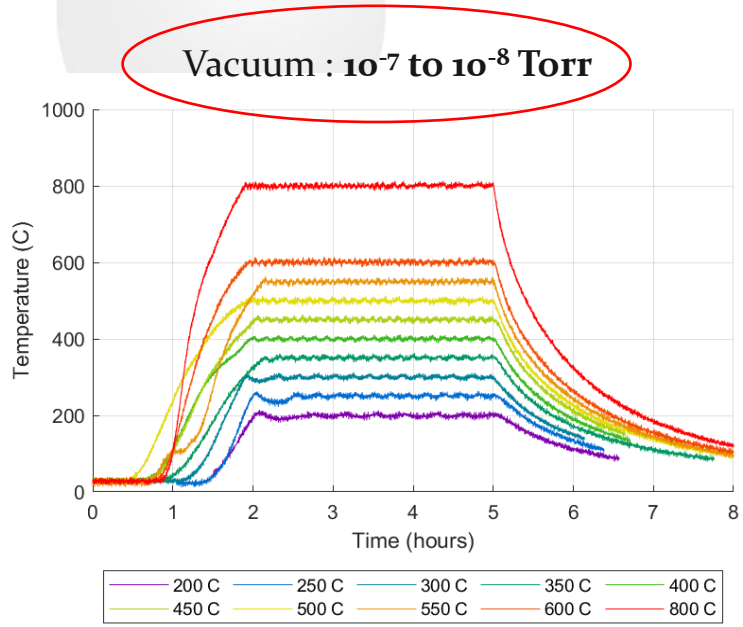
Aims at TRIUMF :

**Determine the type of carbide formed during baking under UHV ($Nb_2C$, NbC) ?**

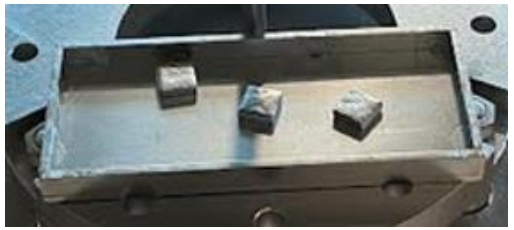



Heat treatments in the TRIUMF induction furnace.

- Niobium samples waterjet cut out of RRR fine grain sheet
- Bulk BCP for 100$\mu m$ (on all samples)
- 800°C 3hrs (on all samples)
- 10$\mu m$ follow up BCP
- heat treatments (RT – 900 °C), venting with $N_2$.

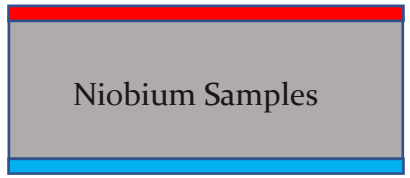



Heat treatment : 100°C to 900°C (3 hrs)



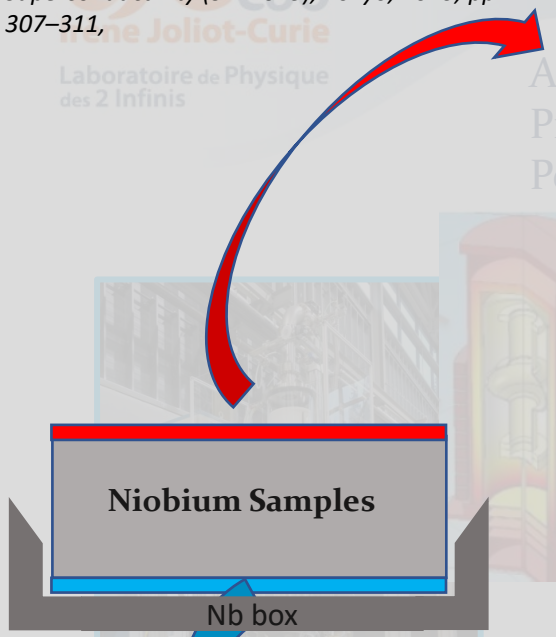
Niobium Samples box.

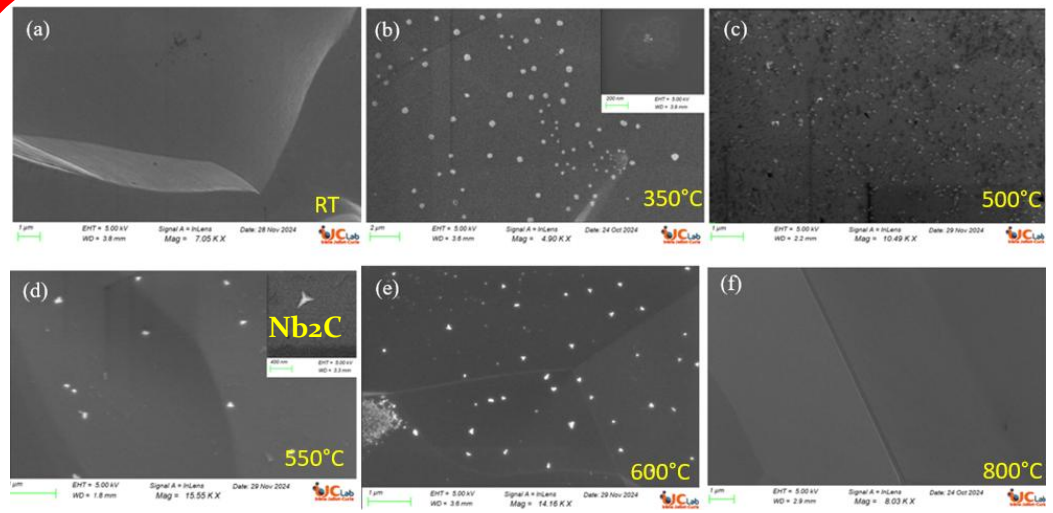
Niobium face to the residual gaz

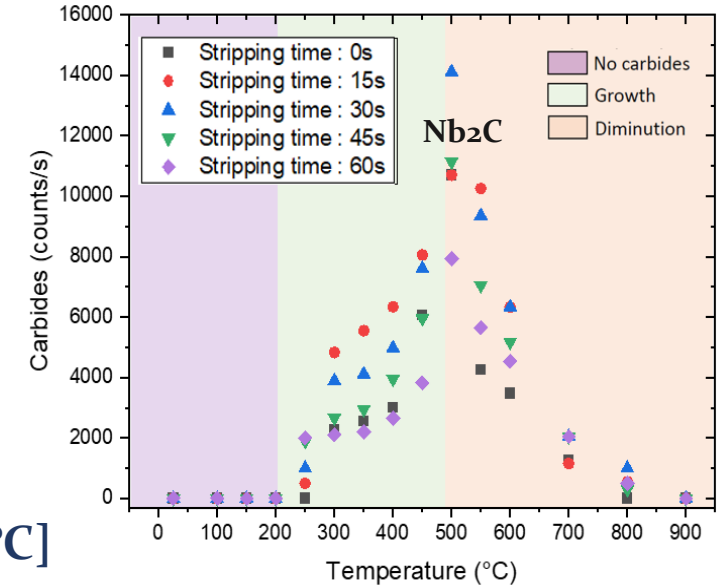



Niobium face to the Nb Box

*C. Boutelaa and al., "Study of niobium surface under ultra high vacuum after heat treatments for SRF cavities," in Proc. 22nd Int. Conf. RF Superconductivity (SRF2025), Tokyo, 2025, pp. 307–311,*

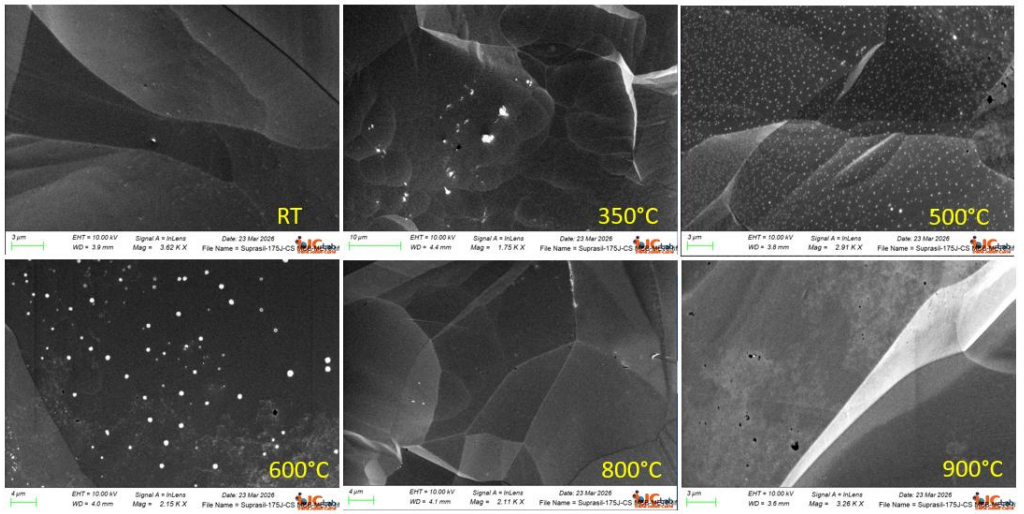


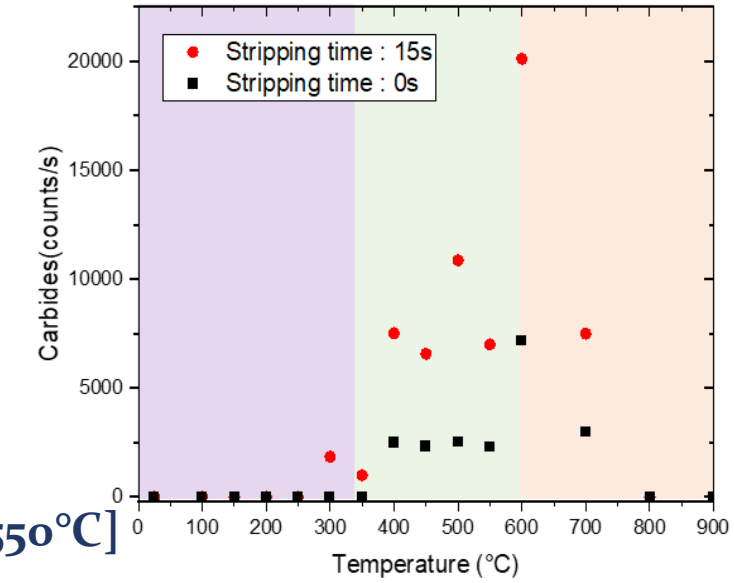




Evolution of carbide by XPS

- **Formation of Nb2C [450°C ; 600°C]**
- **Worst case is for 500°C**




Evolution of carbide by XPS

- **Saturation of carbide formation [400°C ; 550°C]**
- **Worst case is for 600°C**

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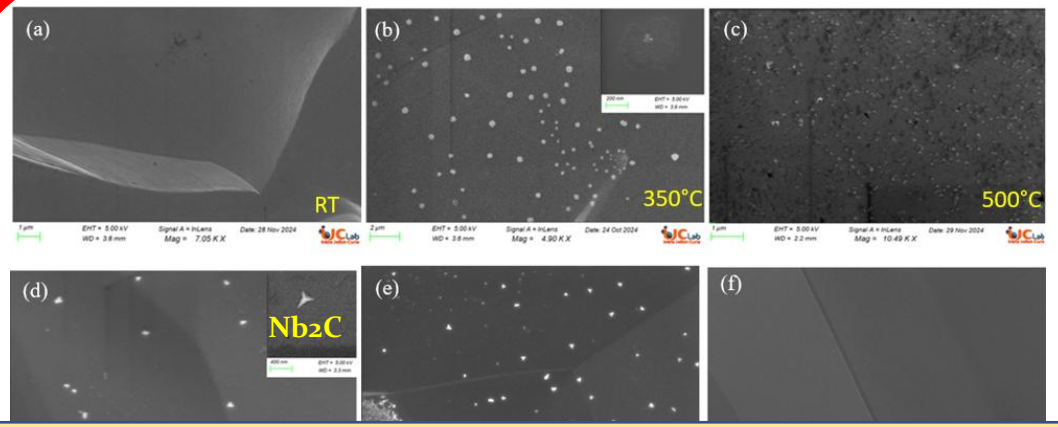
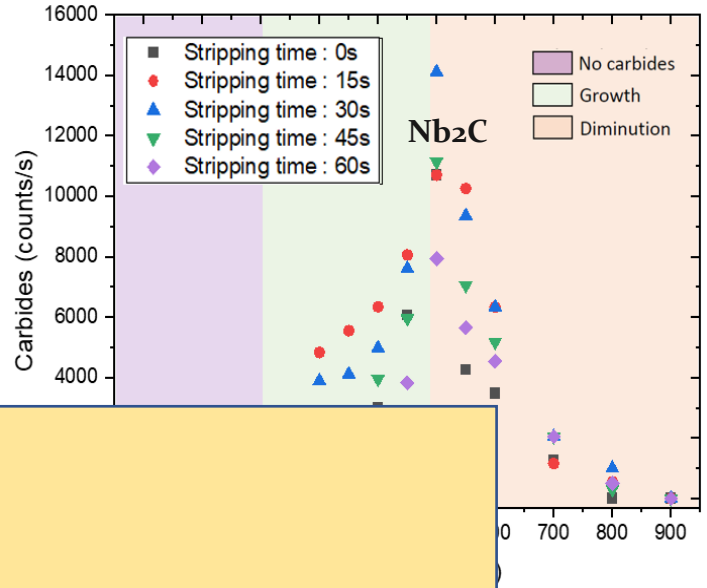
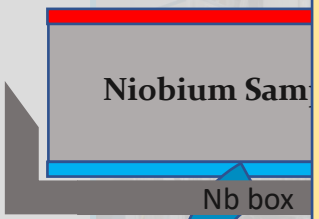
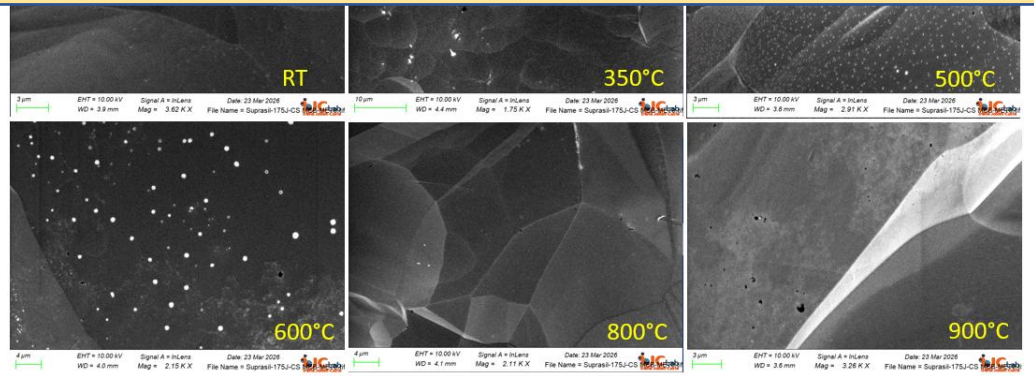
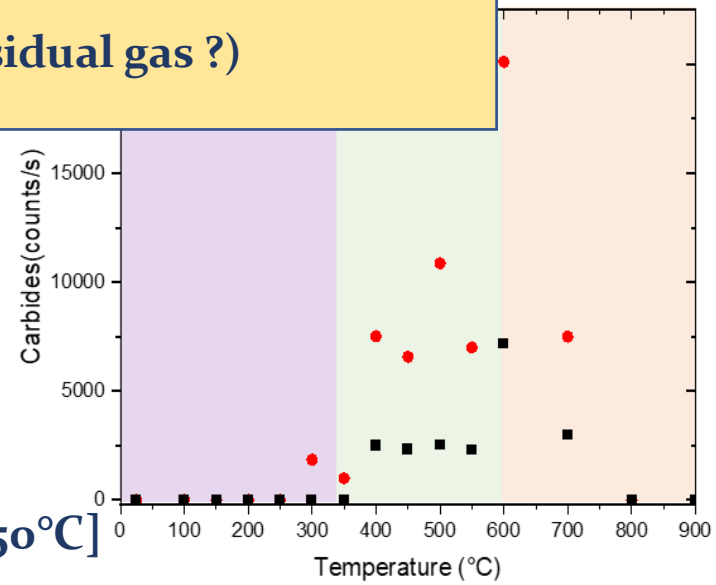



- See the Nb2C by SEM/XPS
- See the NbC by XPS
- Why the structure is not the same ( vacuum face vs Nb box face ) ?
- Interaction with vacuum : 500°C and interaction with Nb box : 600°C !
- The origin of carbon causing these carbides ( essentially the residual gas ?)

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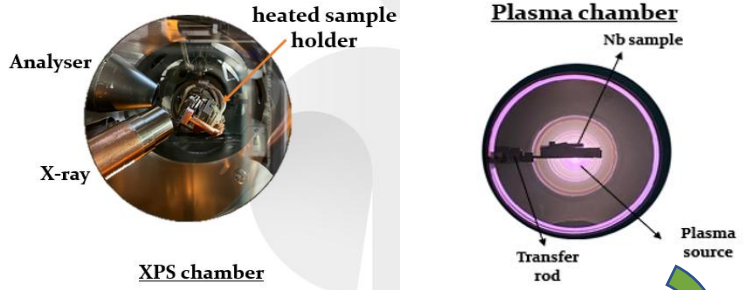
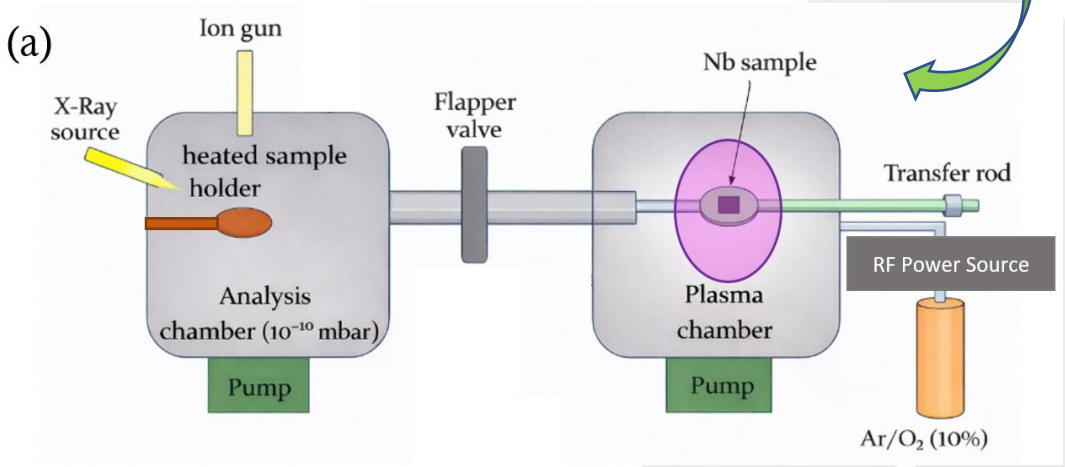
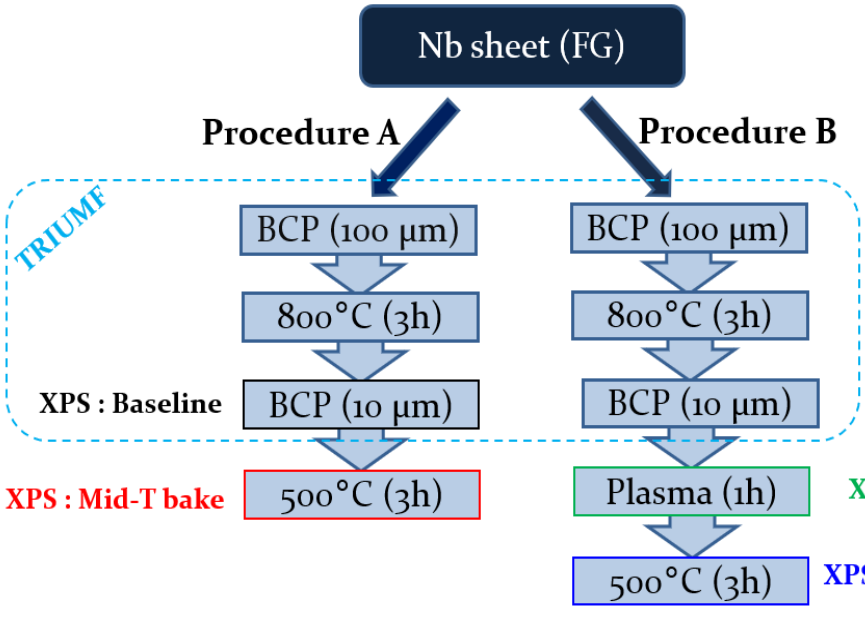
# The origin of carbon causing these carbides ?

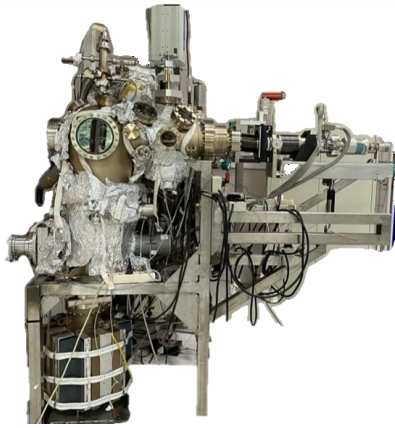


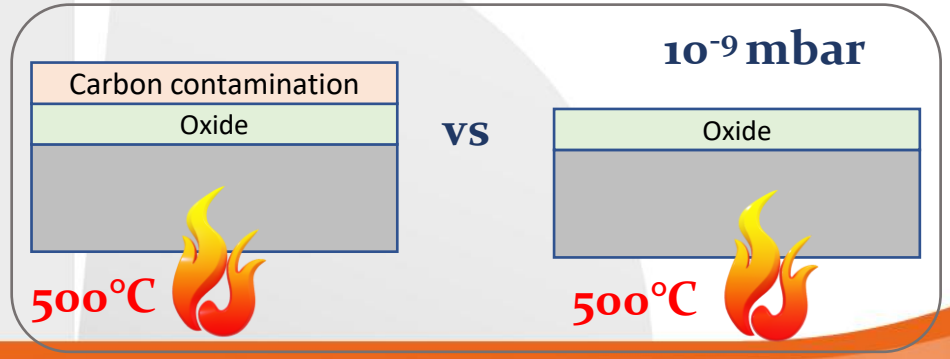

*In-situ equipement for XPS, heat and plasma cleaning*

- 500°C (worst case) for 3h at $10^{-9}$ mbar
  Ar/O2 plasma for 1h

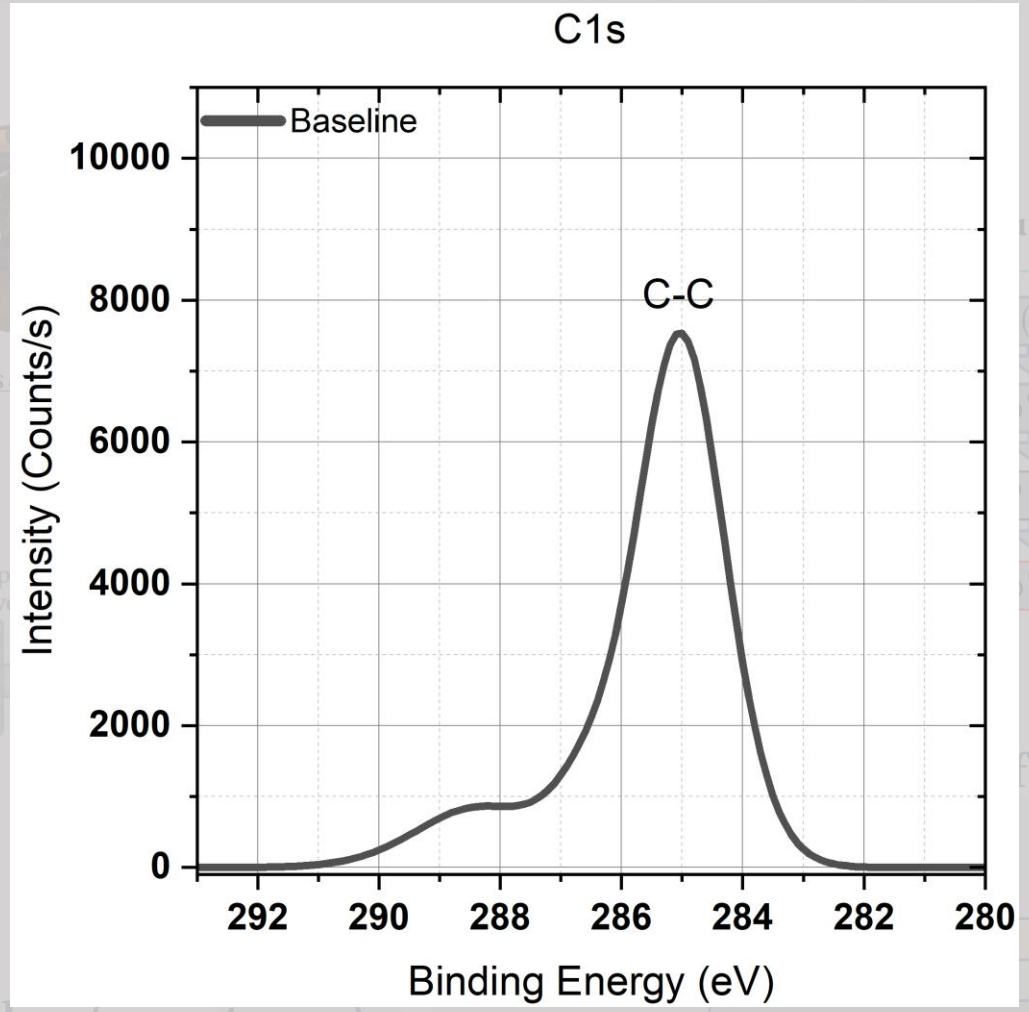


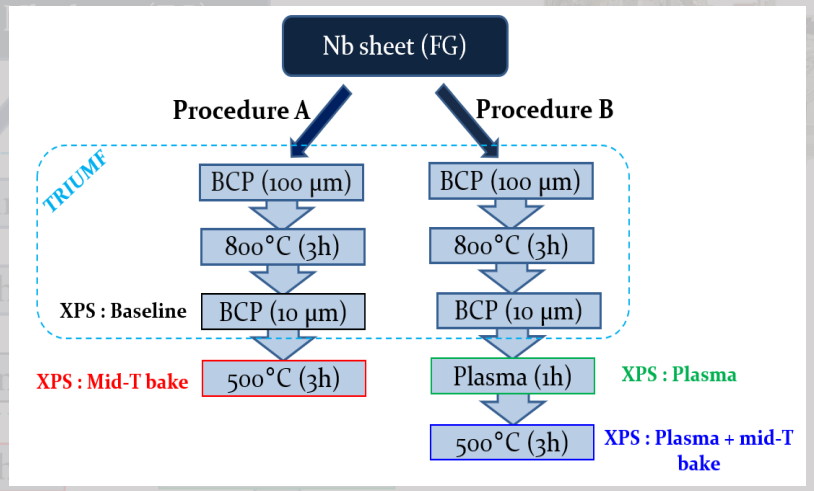



## Quantification of carbide for Mid-T vs for Plasma + Mid-T ?



*C. Boutelaa and al.,"Insitu XPS study of mid-T bake study with plasma treatment on Nb surface", in Proc. Int. Conf. IPAC26, 2026*




**In-situ Mid-T bake study by XPS with Plasma (Ar/O2)**

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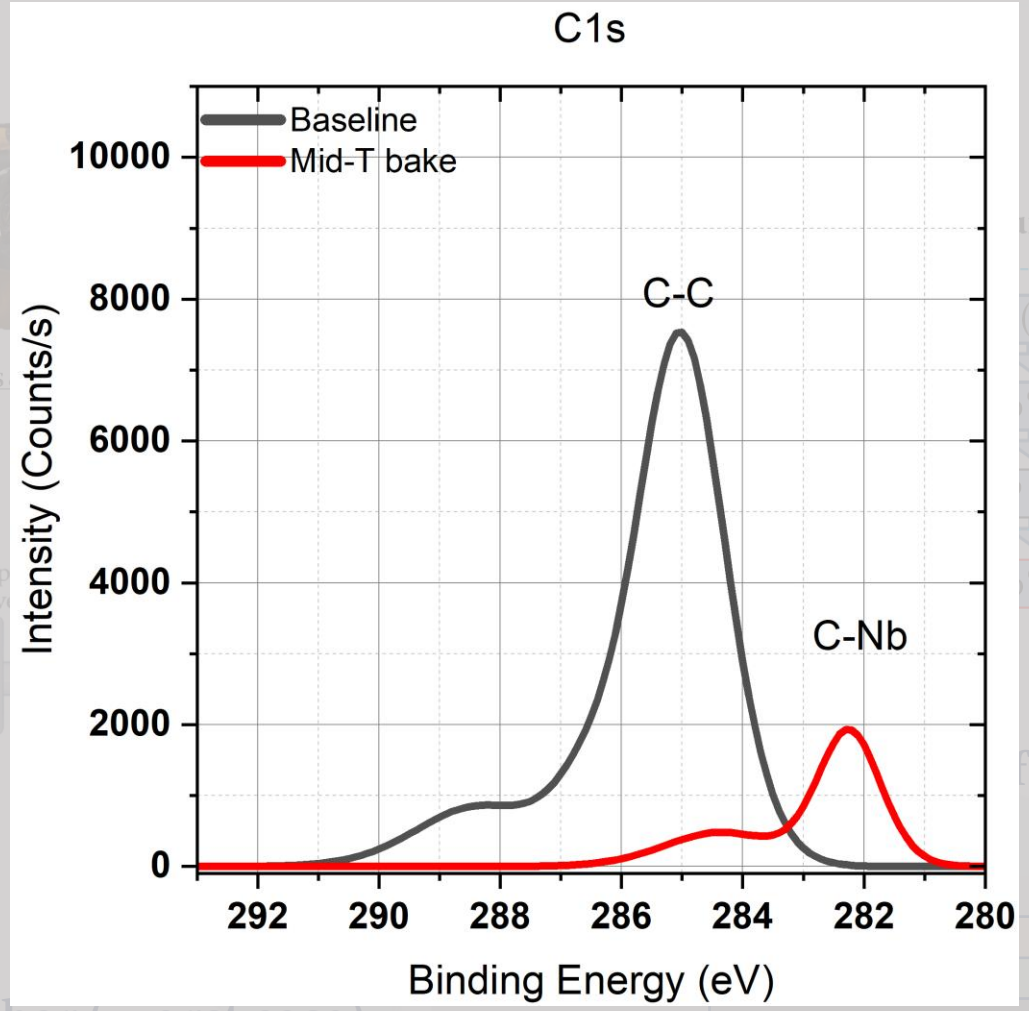


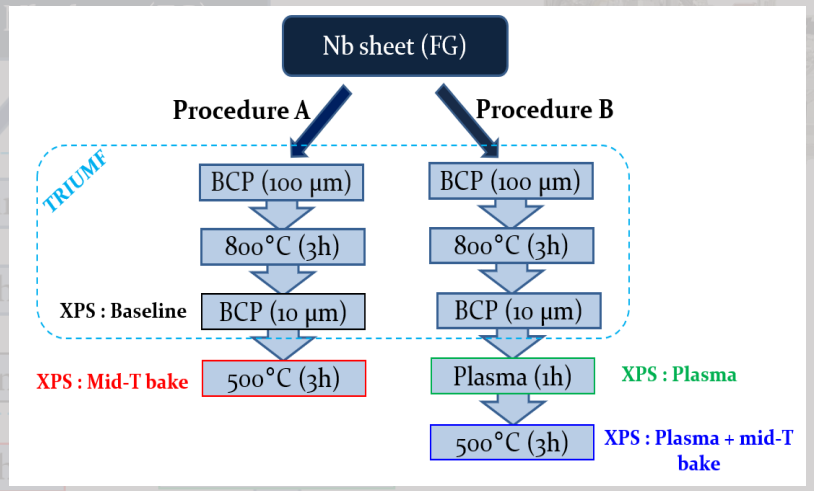


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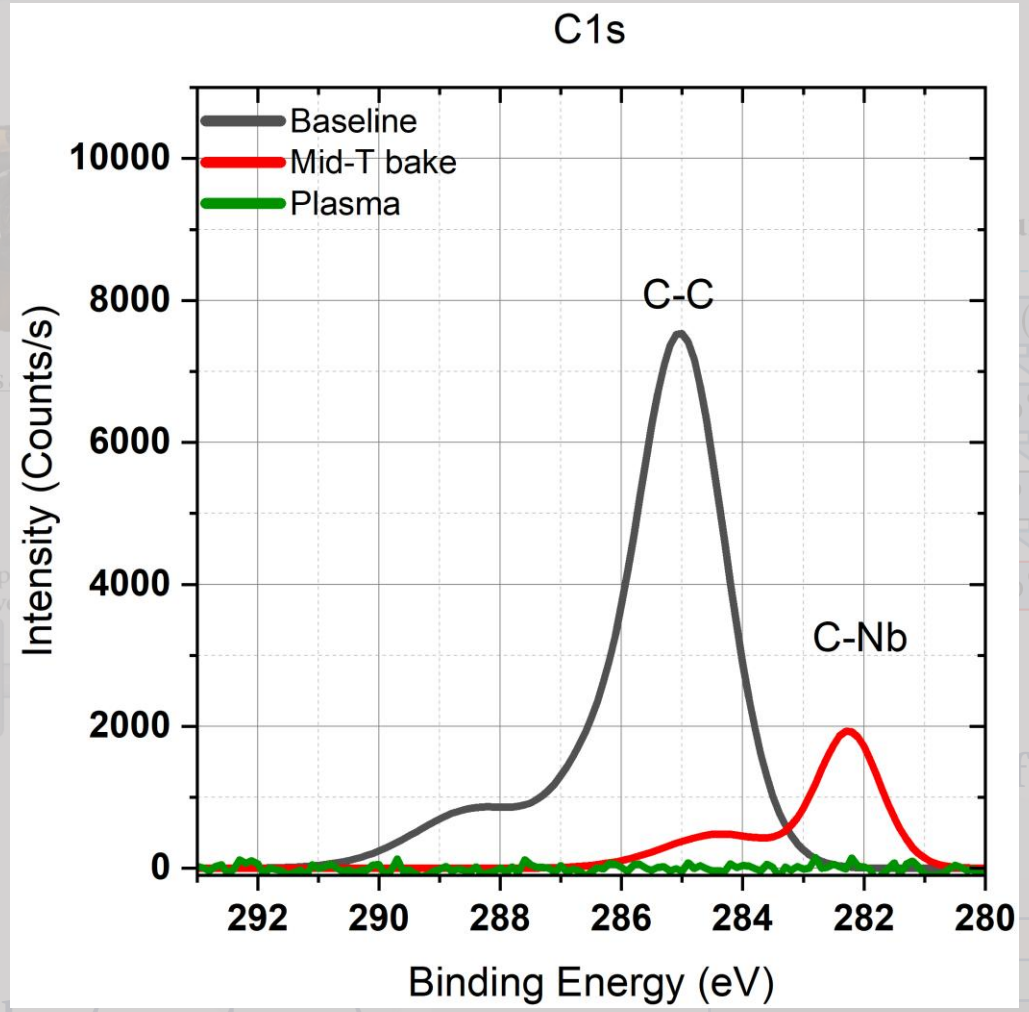


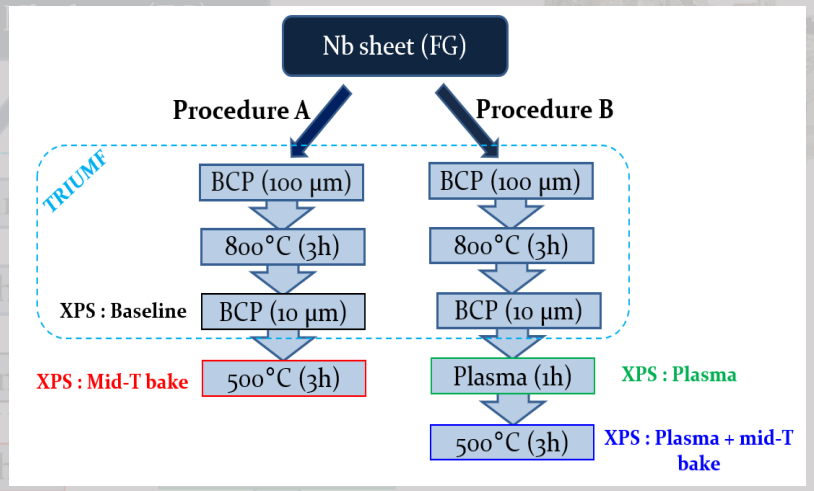


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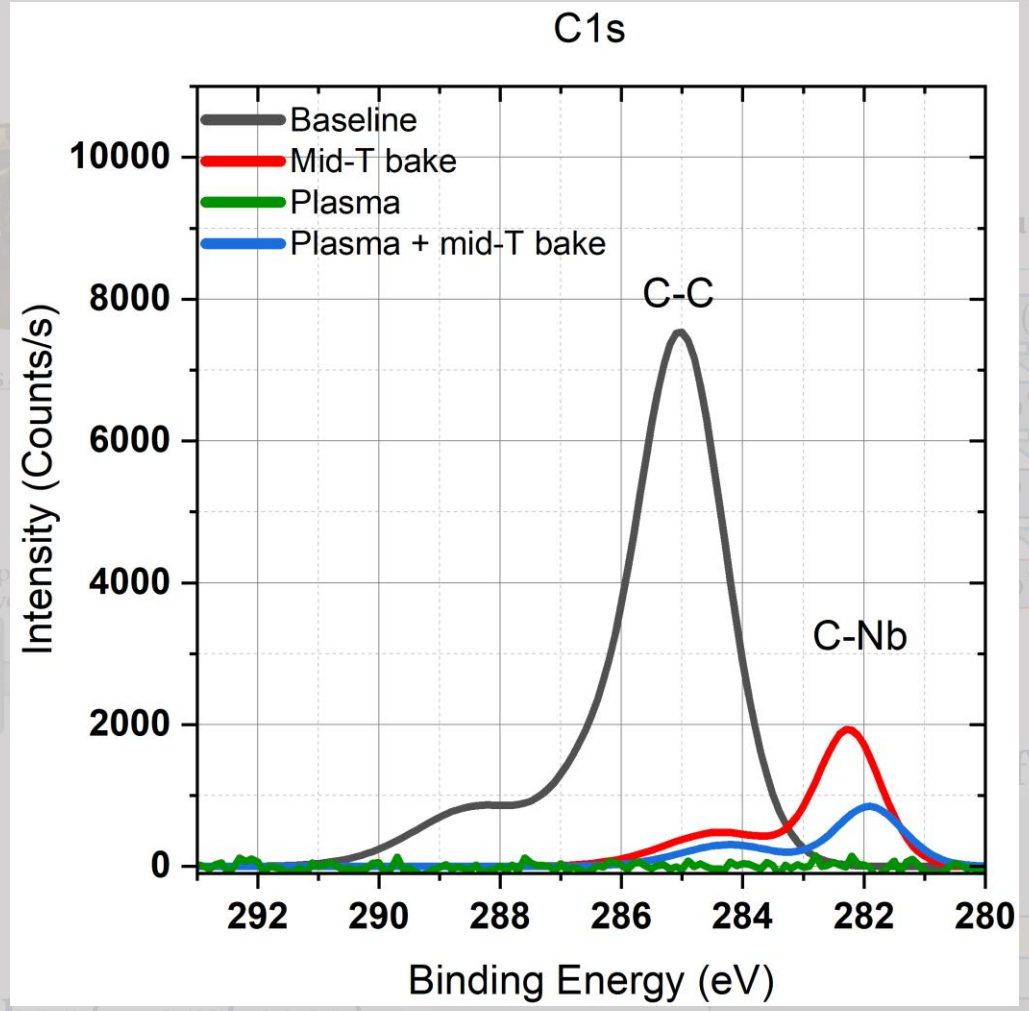
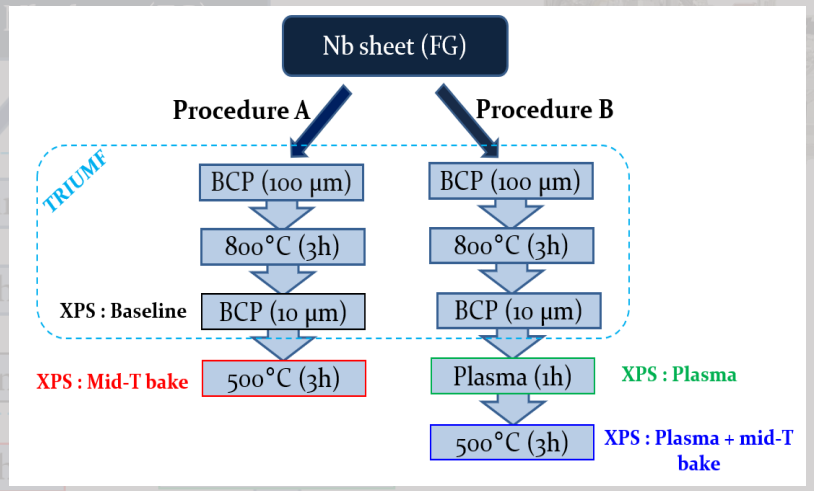


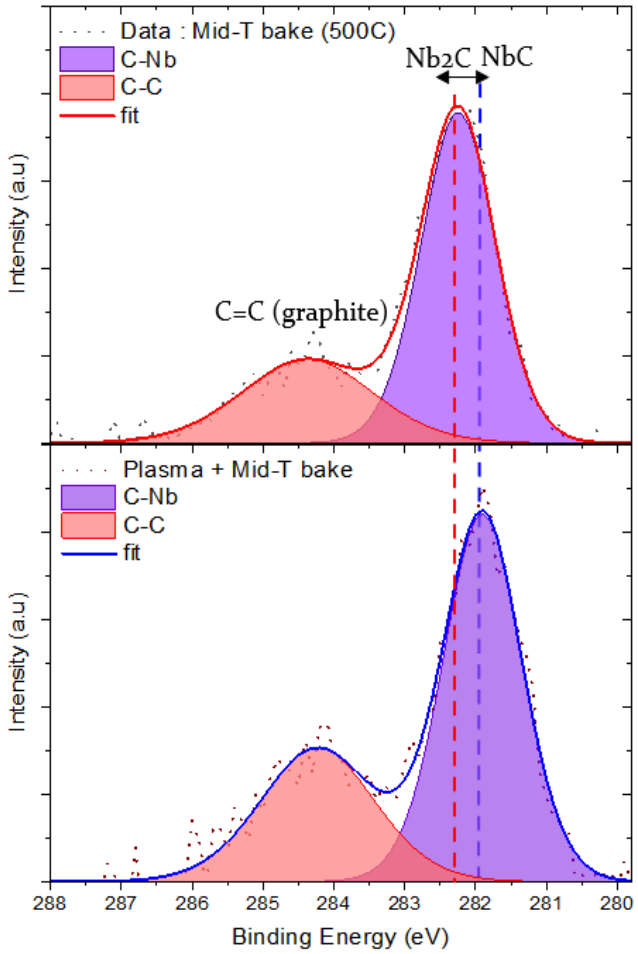



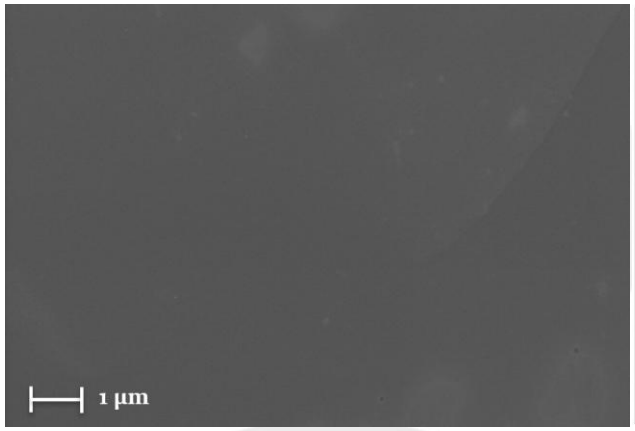

- **Plasma :**

## In-situ Mid-T bake study by XPS with Plasma (Ar/O2)

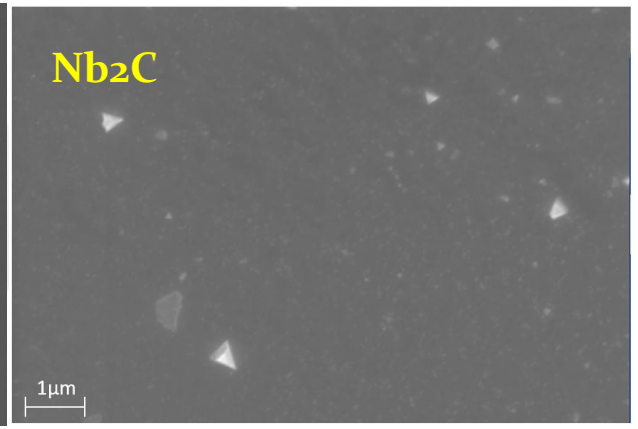
- **Remove the contamination layer.**
- **Carbide formation reduced by 52% after baking.**

# C1s fitting : Mid-T vs Plasma + Mid-T

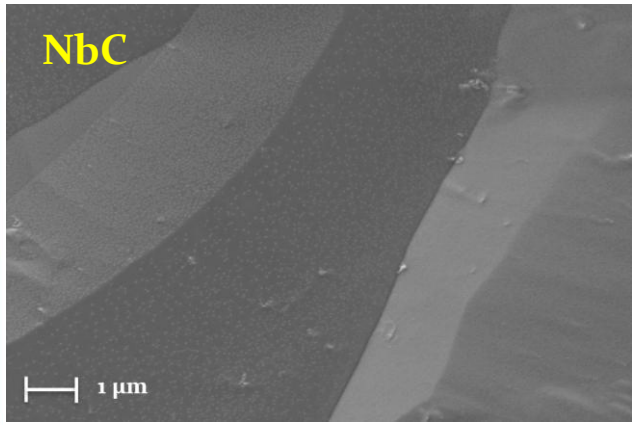



Baseline




Mid-T

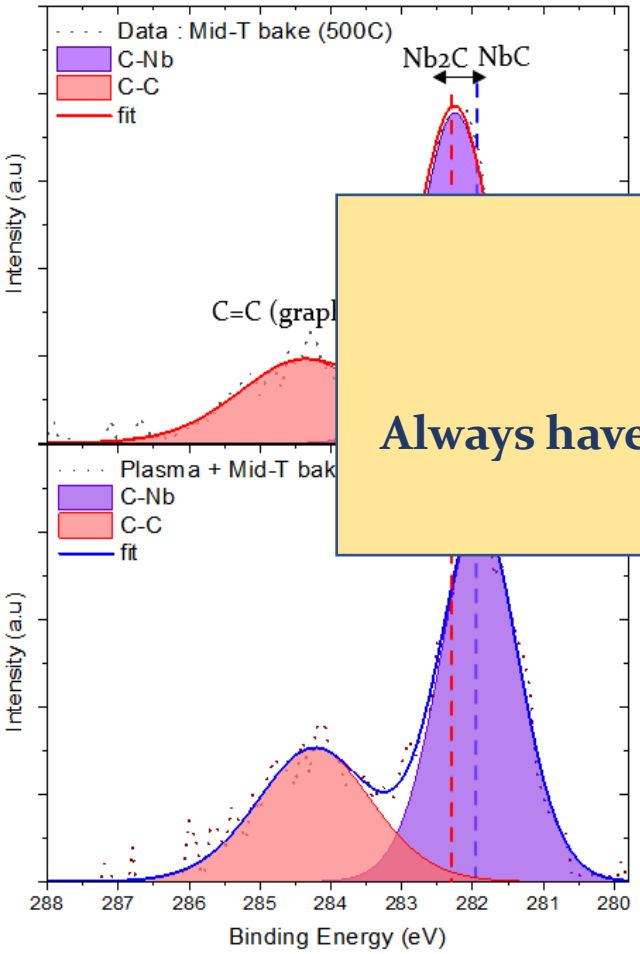


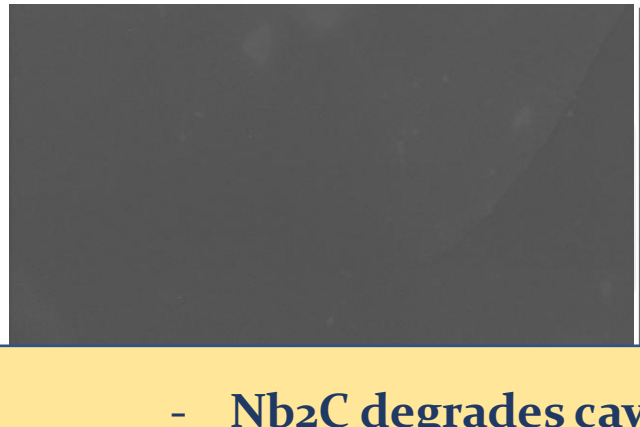

Plasma + Mid-T

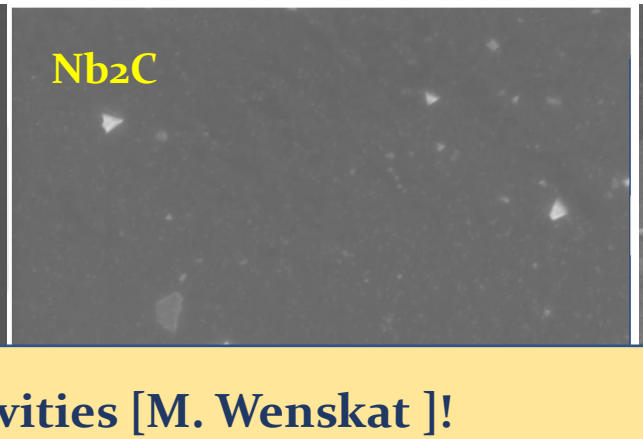
**<u>The origin of carbon causing these carbides is still not identified</u>:**

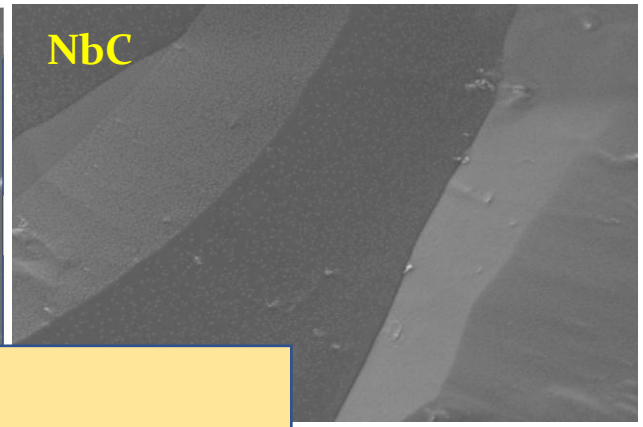
- **52 % of carbide formation is due to the carbon layer. The rest comes from the residual gas and the bulk.**
- **Classic 500°C bake, formed Nb2C phase.**
- **Plasma treatment before baking allowed the NbC phase on the surface .**

# C1s fitting : Mid-T vs Plasma + Mid-T






- Nb2C degrades cavities [M. Wenskat ]!
- NbC is a superconducting material (Tc=11K)

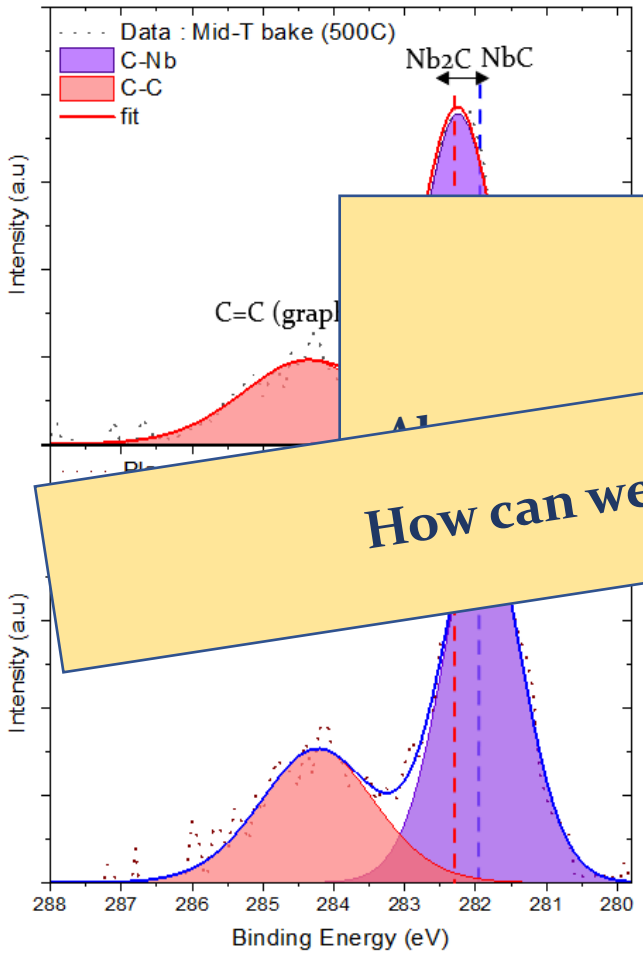
**Always have Carbides for all medium temperature baking ( with good vacuum, with plasma, without carbon layer )!!!**

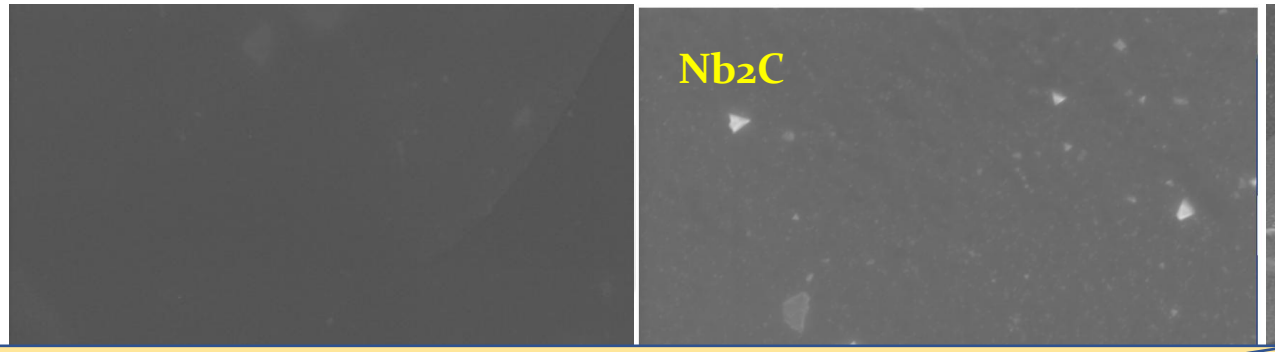
**na + Mid-T**

**:**

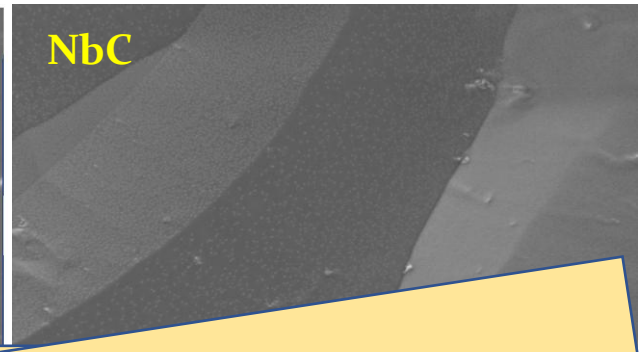
- **52 % of carbide formation is due to the carbon layer. The rest comes from the residual gas and the bulk.**
- **Classic 500°C bake, formed Nb2C phase.**
- **Plasma treatment before baking allowed the NbC phase on the surface .**

# C1s fitting : Mid-T vs Plasma + Mid-T






- Nb2C degrades cavities [M. W...
- NbC is a super...

**How can we reduce this formation to minimize its impact on cavity results?**

...Mid-T

...aking ( with good vacuum, ...ithout carbon layer )!!!

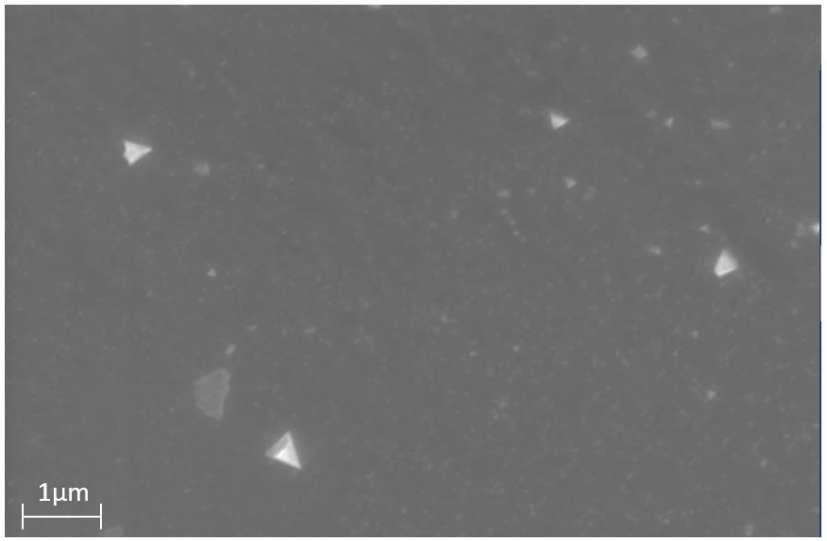
...:

52 % of carbide formation is due to the carbon layer. The rest comes from the residual gas and the bulk.

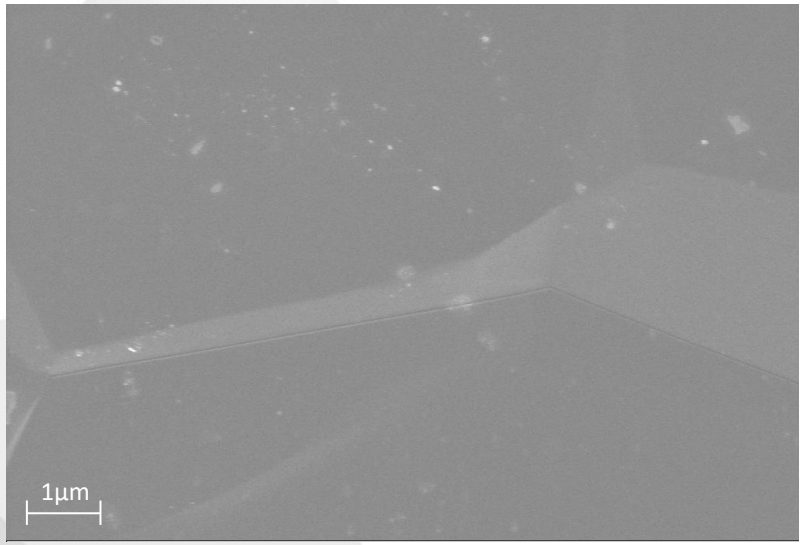
- Classic 500°C bake, formed Nb2C phase.

- Plasma treatment before baking allowed the NbC phase on the surface .

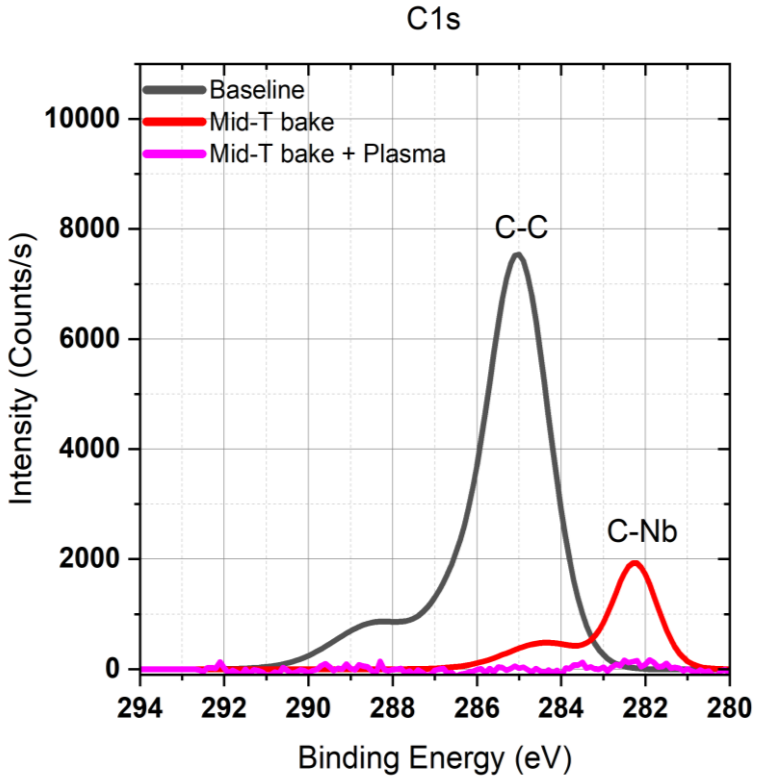
# 1) Plasma after Mid-T




Mid-T




Mid-T + Plasma



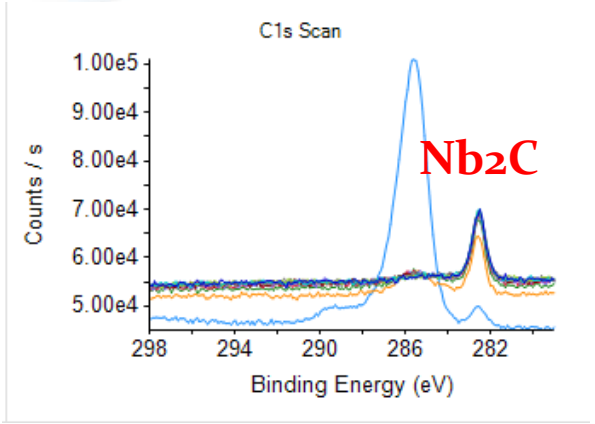

In-situ Plasma with XPS

**Ar/O2 plasma can remove the Nb2C at the extreme surface !**

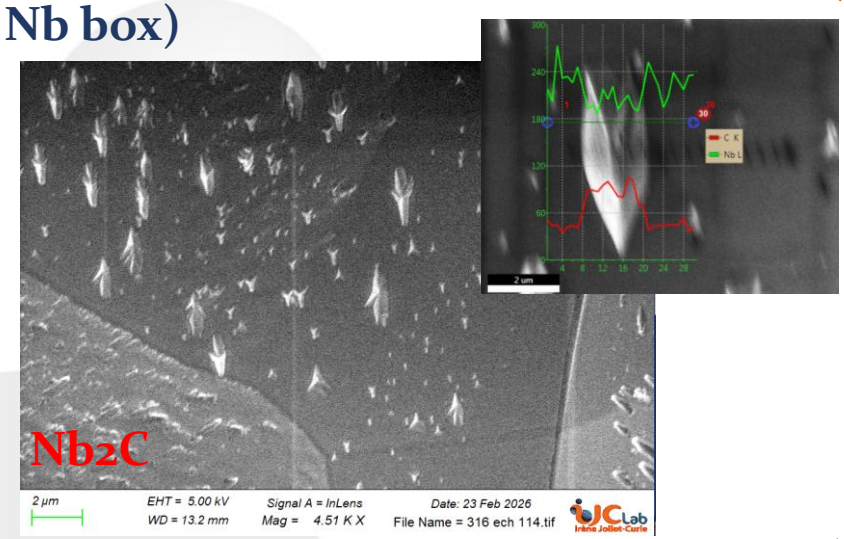
# 2) Nb box during the baking ?

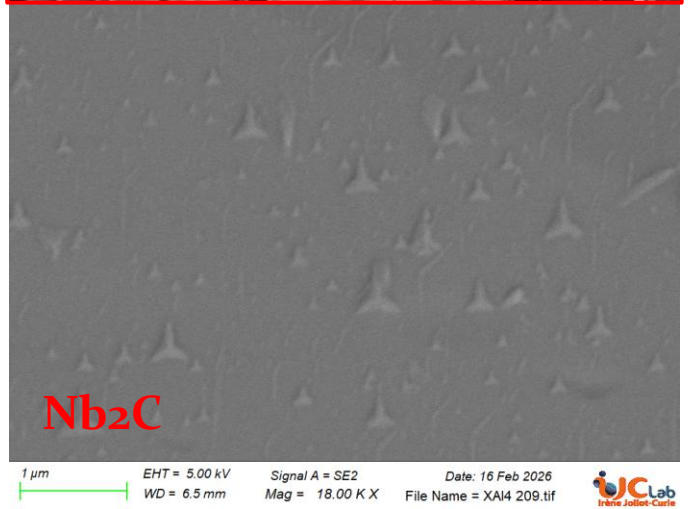
**SUPRTACEH furnace : 900°C for 3hrs ( $10^{-6}$ mbar)**

## ( With Nb box)



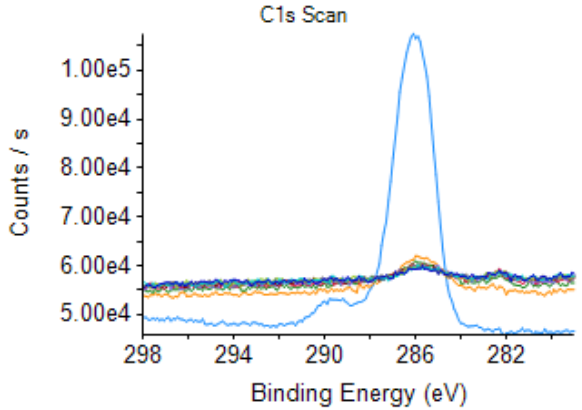

**Carbides ($Nb_2C$) formation !**



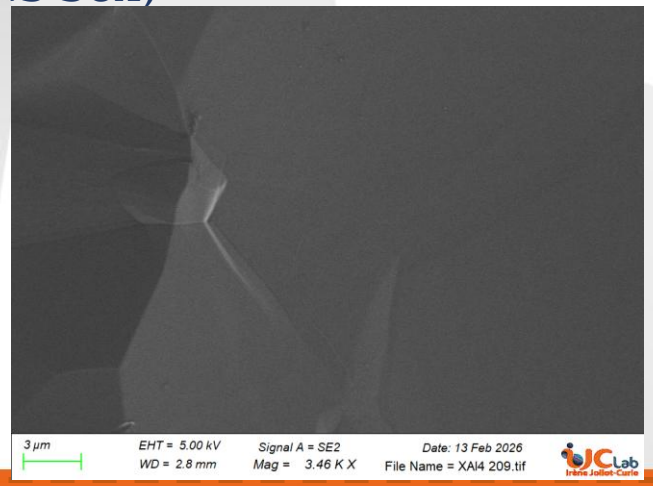




**(SEM picture of Nb box)**

## ( Without Nb box)



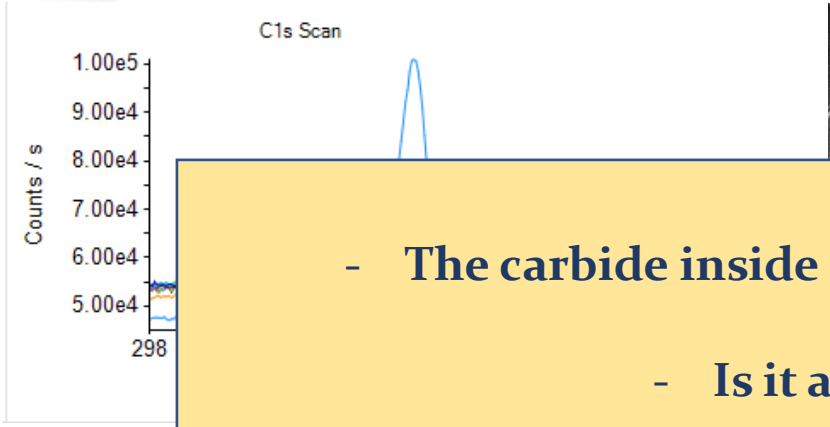

**No Carbide formation !**

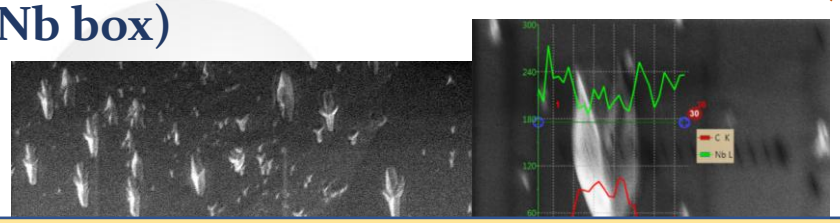


# 2) Nb box during the baking ?

**SUPRTACEH furnace : 900°C for 3hrs ( $10^{-6}$ mbar)**

**( With Nb box)**

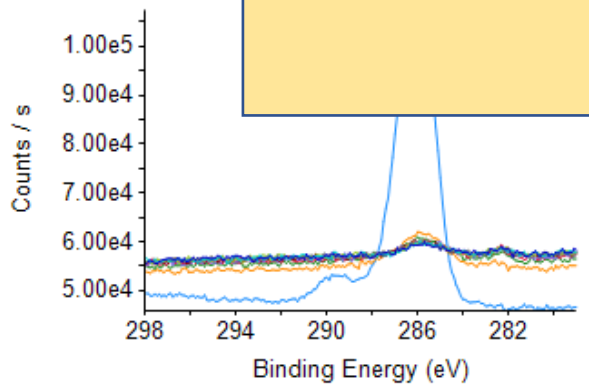


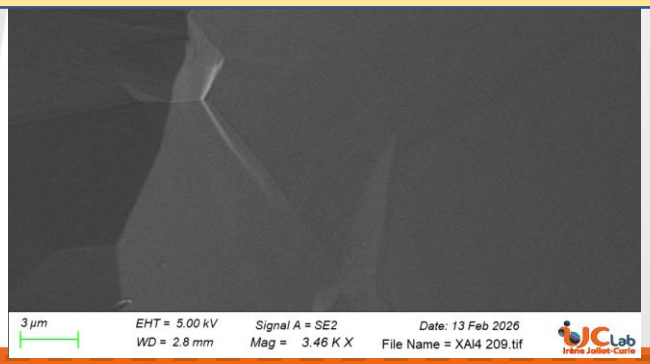


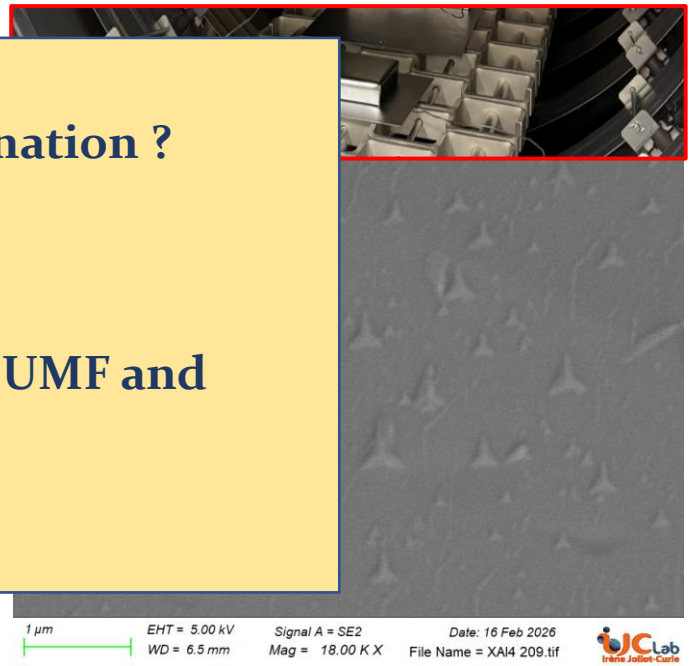
**Carbide**

- **The carbide inside the Nb box can release Carbon contamination ?**
- **Is it always a good idea to use Nb caps ?**
- **We didn't find carbide formation for any 900C bake with TRIUMF and SURPATECH furnace.**




**No Carbide formation !**




**(SEM picture of Nb box)**

# Summary

Manipulation of Nb carbide during Mid-T bake : Impact of baking configuration and plasma treatment

**Determine the type of carbide formed during baking under UHV ($Nb_2C$, NbC) ?**

- $Nb_2C$ is observed between 450°C to 600°C
- No formation for 900°C
- NbC formation for Plasma + Mid-T

**The origin of carbon causing these carbides ?**

- Carbon contamination layer : 52 %
- From residual gas during heat treatment /bulk : 48 %

**How can we reduce this formation to minimize its impact on cavity results ?**

- Plasma cleaning before baking ( $Nb_2C$ to NbC)
- Plasma cleaning after baking ( Remove $Nb_2C$ and NbC at extreme surface)

**Outlook**

The first SEY ( secondary electron yields) study on Nb samples at 7K :

⇒ Mid-T samples (TRUIMF)
⇒ Low Temperature baking
⇒ Plasma treatment

# Thank you for your attention !

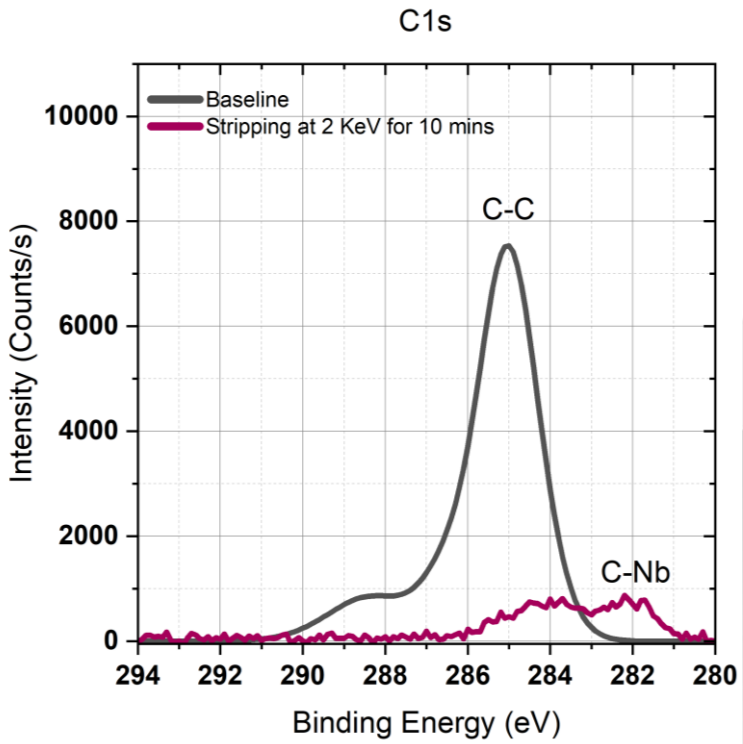
# Merci à vous !

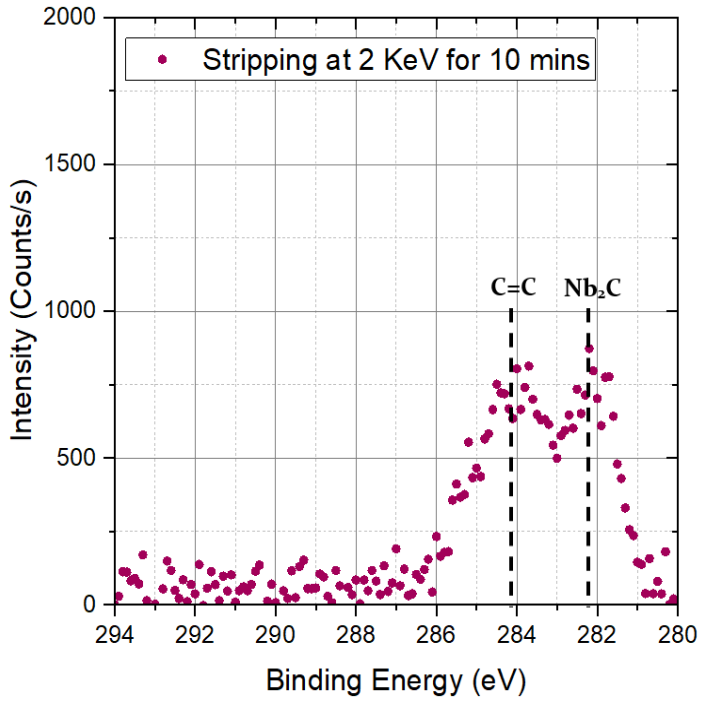
Chahinez.boutelaa@ijclab.in2p3.fr

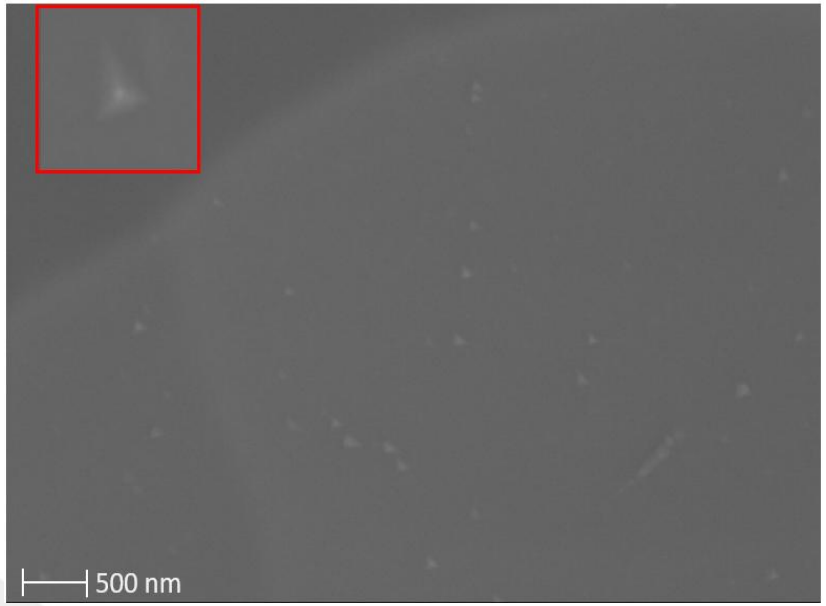
# Thanks to :


- All the department, groups and people that help with this experiment.

- MAVERICS Team, IJCLAB
- SRF team, TRIUMF
- Vide et Surface plateform, IJCLAB
- International Research Laboratory (IRL) beetween TRIUMF and CNRS
- ANR-21-ESRE-0049

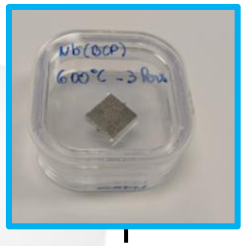


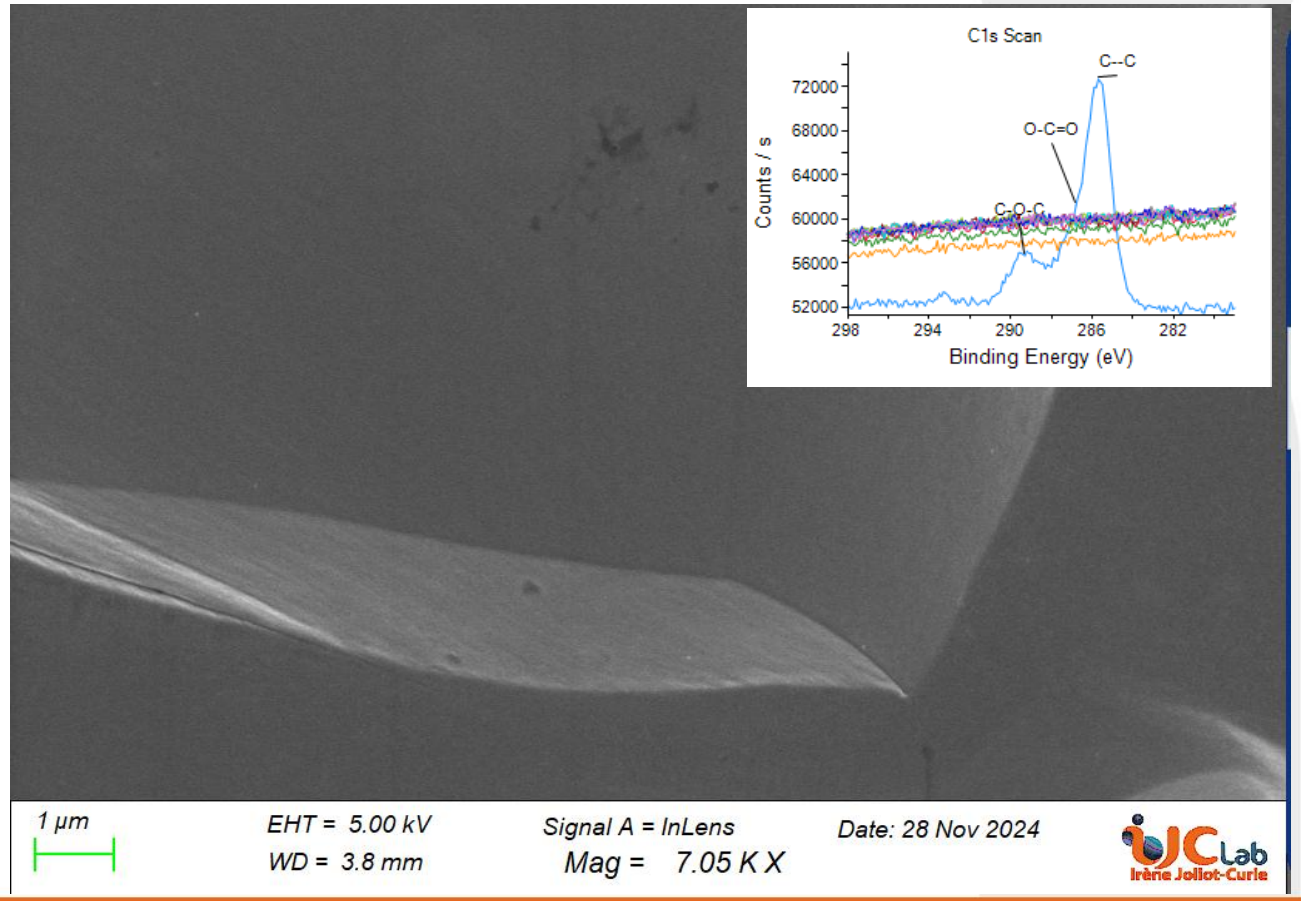




*In-situ* comparison of the C1s peak XPS before and after abrasion at 2 KeV for 10 mins (a) with $Ar^+$ bombardment by ion gun; Positions peaks XPS for abrasion at 2 KeV for 10 mins (b); *Ex-situ* SEM pictures after abrasion at 2 KeV (c).

# Same sample ( 1 year ago Vs Now)

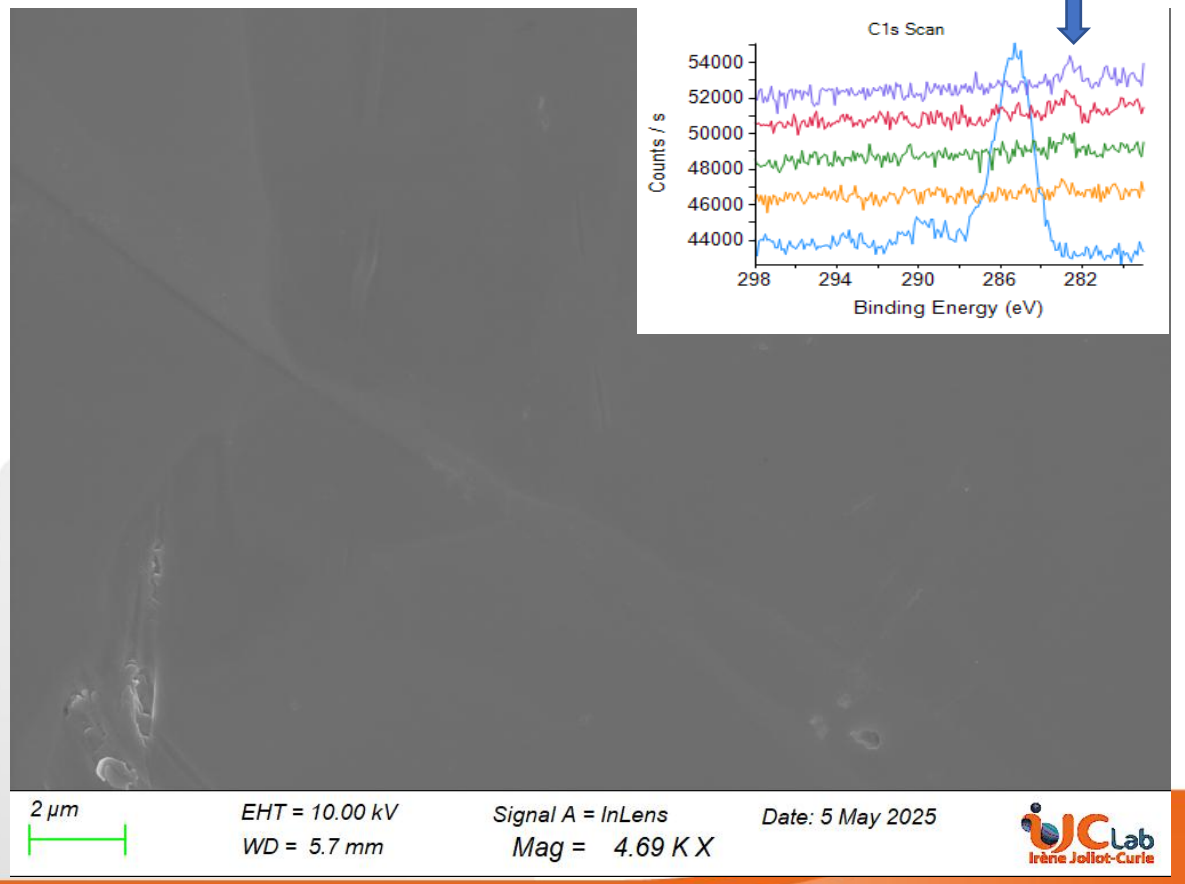


## Nb-RT-BCP-2024




## Nb-RT-BCP-2025

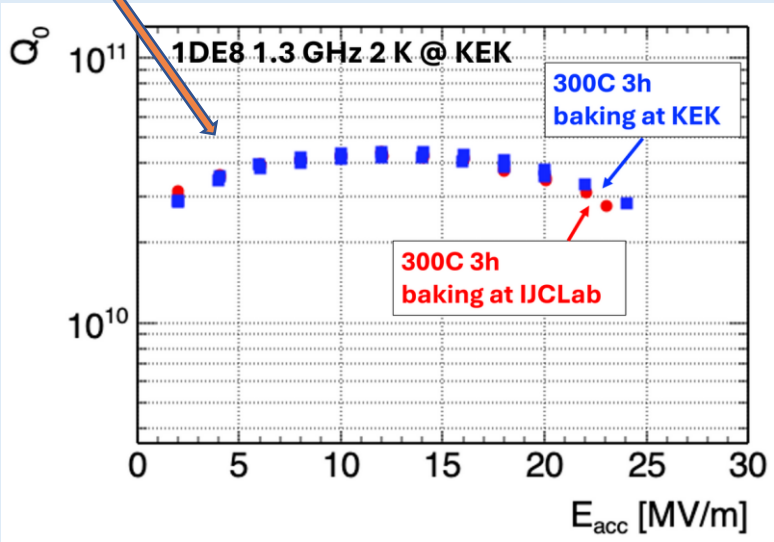
## Nb-C

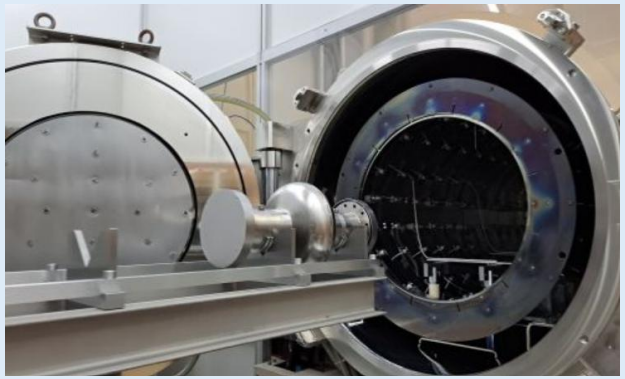


# Contamination study of mid–T Bake with KEK furnace

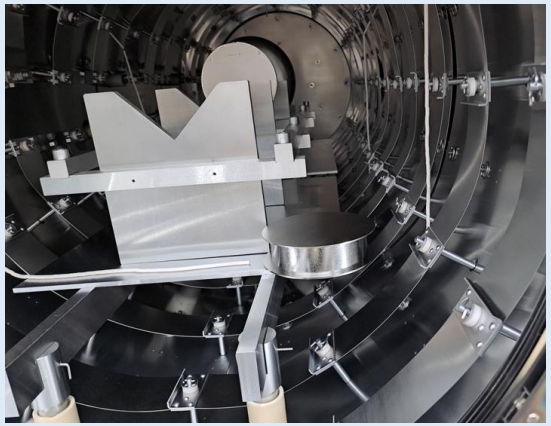
Aims at KEK :
Perform heat treatments with KEK furnace
( 300°C-3h) for elliptical cavities ( 1.3 GHz)+ Nb sample => Analysis at IJCLab ( XPS and SEM)

300°C-3h



KEK furnace + Cavity + Nb sample



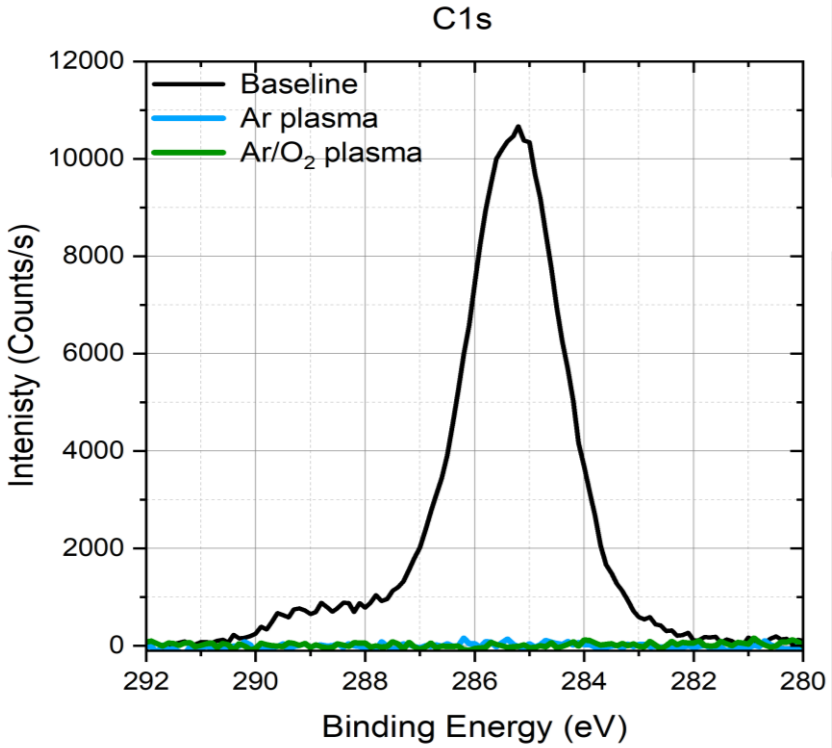
Cavity preparation :
1) bulk EP at CEA
2) 600C annealing at IJCLab
3) flash EP at CEA
4) Test at CEA (2024)
5) 300C 3h baking at IJCLab
6) Test at KEK in 2024
7) 800C annealing to reset mid-T without flash EP at KEK
8) Test at KEK in 2025
9) 300C 3h baking at KEK
10) Test at KEK in 2025 ( same results as 6)

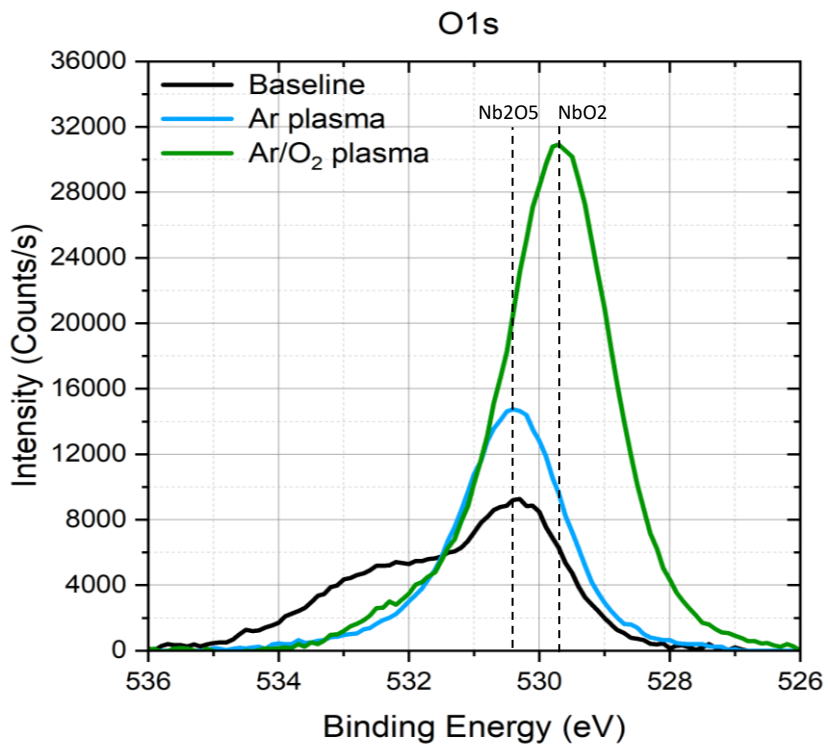
Mid-T bake conditions at KEK :
- Cavity and samples were annealed at 300°C 3h with :

Before (26°C): <3.2e-6 Pa = <3.2e-8 mbar
During (300°C): 8.2e-5 Pa
After (35°C): 2.5e-6 Pa

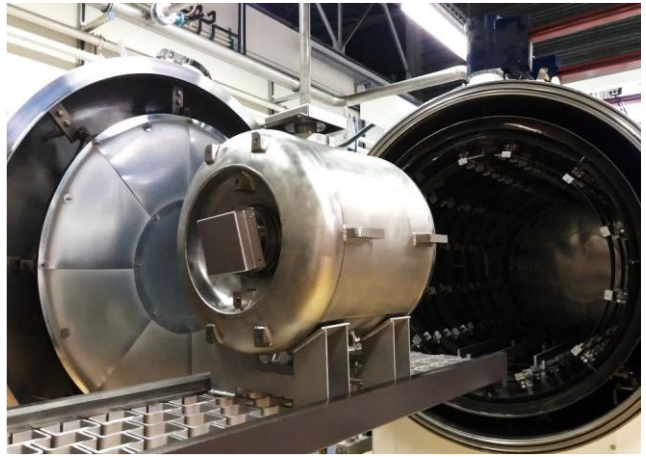
- Purge with Argon at 50°C

# Ar plasma treatment by XPS



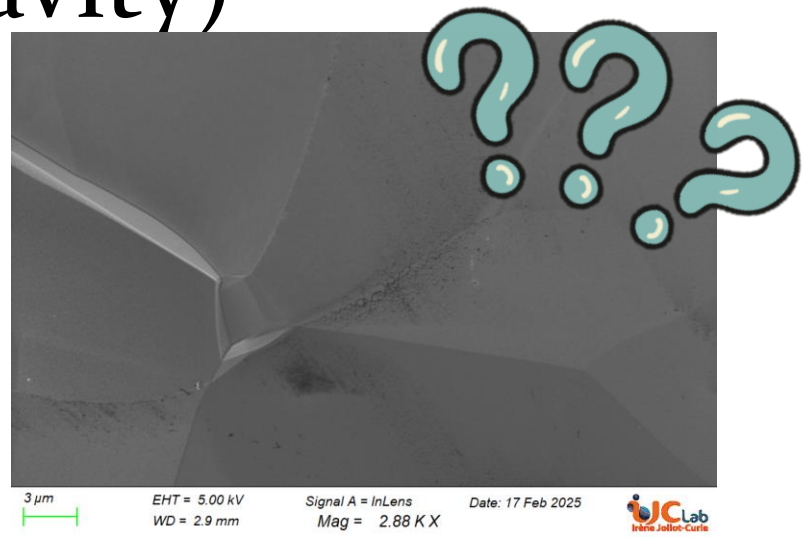
# 600°C for 10h with supratech furnace (Nb sample + cavity)



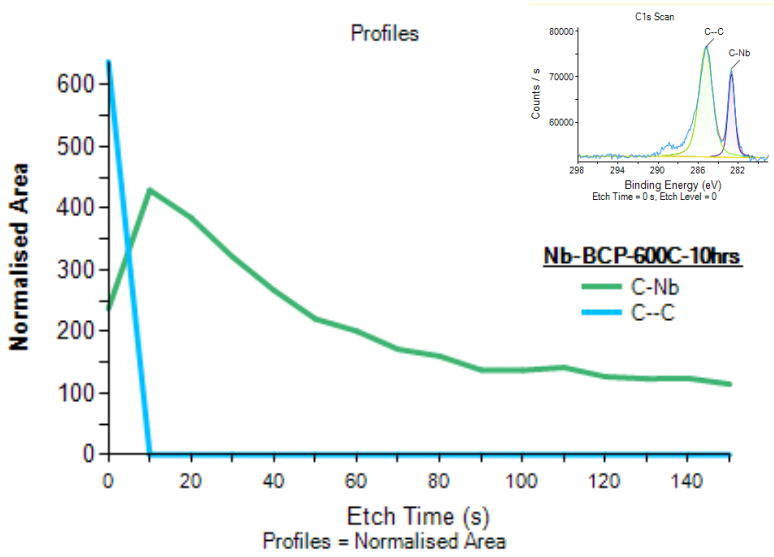
**600°C for 10h with Supratech furnace**

**Nb sample**

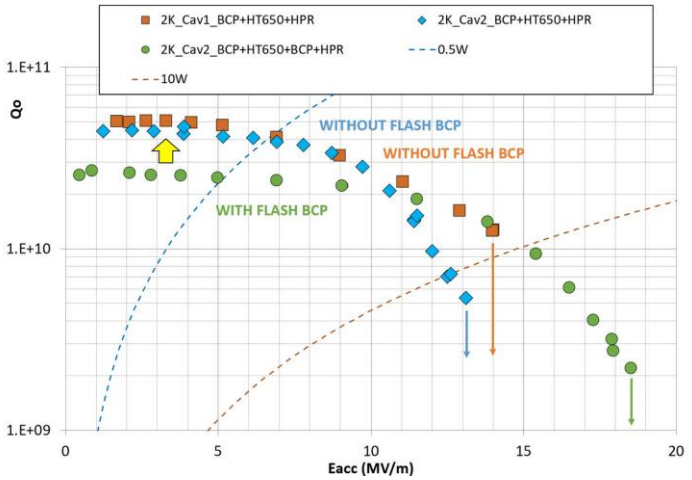
**MYRRHA Simple Spoke Cavity**




SEM pictures of Nb samples at 600°C during 10h.




Carbides depth profile for 600°C.




D.Longuvergne,JT-RTV-2025

Discussion:

- **Best results at IJClab for 650°C 10hrs.**

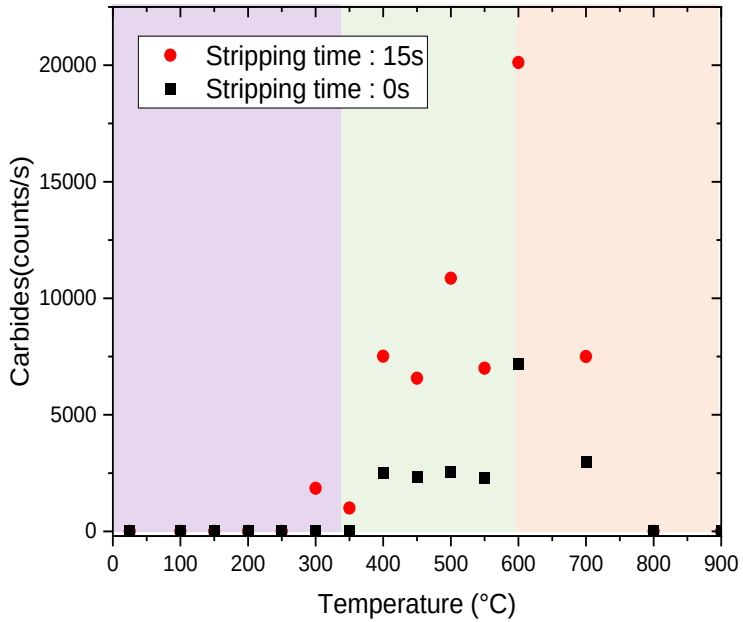
**Questions :**

- **SEM results confirm good Qo test, but we observe Nb-C by XPS !**
- **Issues only with the structure of carbides?**

*C. Boutelaa, D.longuergne (IJCLab), G.Sattonnay (IJCLab)*

# TRIUMF bake : Carbide analysis

**Surface in contact to the Nb box**




**Surface in interaction with the vacuum**