Nb Carbides study on Nb samples after heat treatments for SRF cavities.*

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PHD STUDENT

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CNRS, IJCLab, Orsay, France[1] TRIUMF, Vancouver, Canada[2] Western University, Ontario, Canada[3] KEK, Tokyo, Japon[4]

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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
- International Research Laboratory (IRL) beetween TRIUMF and CNRS
- ANR-21-ESRE-0049
- SRF team, KEK

Outline

1- Background and Motivations

2- Analysis of Nb samples and heat treatments with TRIUMF furnace

3- Nb carbides study for high Qo with Supratech and KEK

4- Summary and future outlook

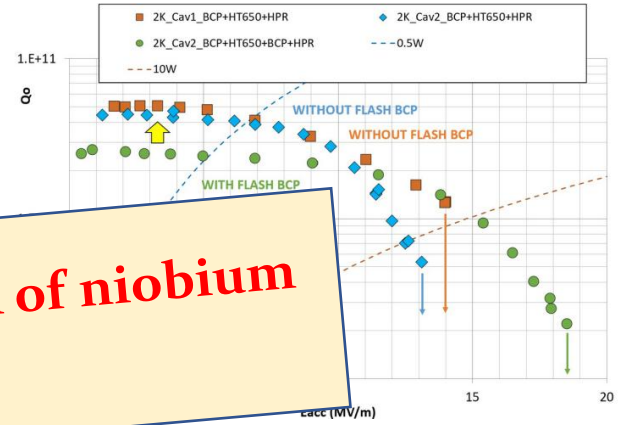
Innovative heat treatments

Infusion-like H-degassing for Spoke cavity (352MHz)

Bulk BCP 150-200µm → HPR → H-degassing → HPR

Best results at

These advanced treatments did not reveal any formation of niobium carbide during heat treatment.

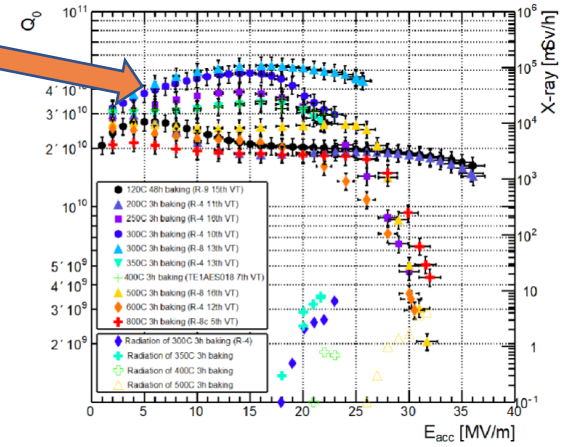


D.Longuvergne,JT-RTV-2025

Mid-T baking for elliptical cavity (1.3 GHz)

Bulk BCP 150-200µm → 800°C-3h or 650°C-10h → Flash BCP → 300°C-3h → HPR

300°C-3h



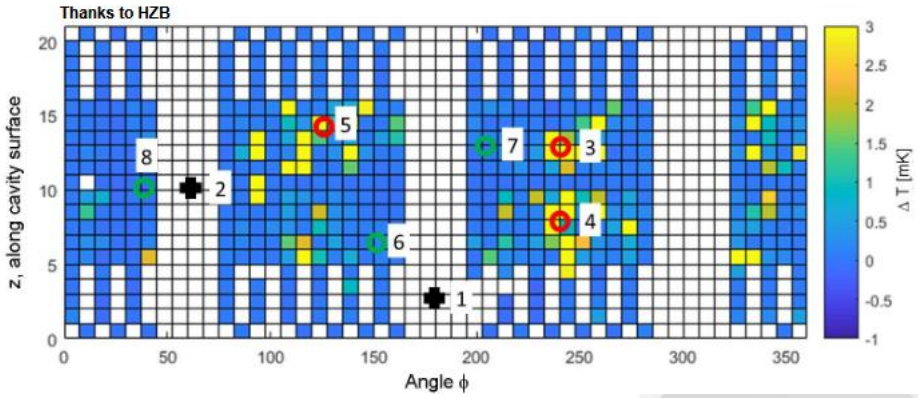
H. Ito, KEK, SRF2021

Best results at 300°C – 350°C, t = 3hrs Ref : Akira Miyazaki, Poster, SRF 2025

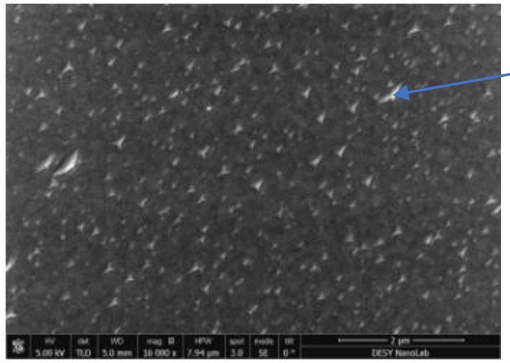
Goal

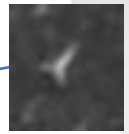
M. Wenskat | TTC Meeting @ CERN 2020

Cut a cavity and look



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





Carbide (Nb_2C)

« carbides can degrade the performance of cavities? »

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

- **from residual gas during heat treatment**
- **from chemical processes**
- **from the bulk**

What are their formation conditions?

Why Nb samples ?

It is important to study the Mid-T baking process under controlled conditions.

Study with Nb samples by **XPS, SEM** and **SIMS** at TRIUMF and IJCLab :

•Measurements: Carbides growth and Nb interactions with impurities after baking.

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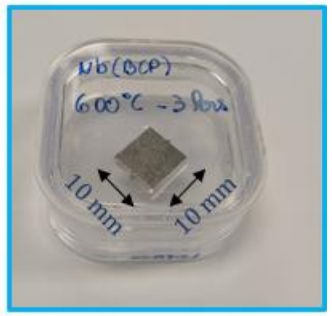
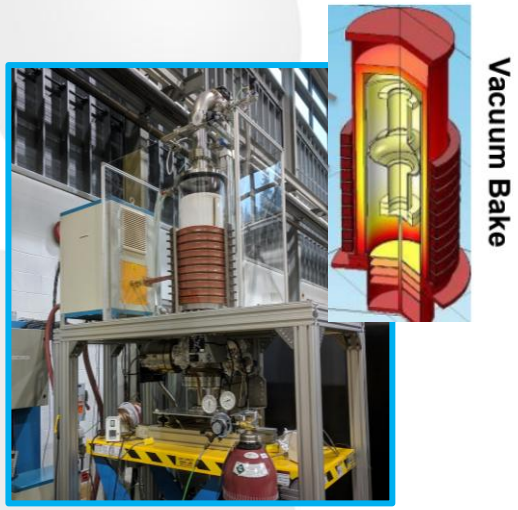
Nb samples preparation at TRIUMF

Aims at TRIUMF :
Produce Nb samples (50 samples)
Perform heat treatments with TRIUMF induction furnace => Analysis at IJCLab and Western University

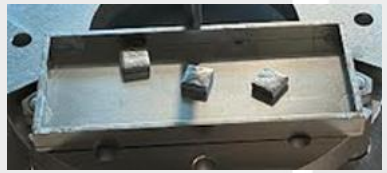
- Niobium samples waterjet cut out of RRR fine grain sheet
- Bulk BCP for 100μm (on all samples)
- 800°C 3hrs (on all samples)
- 10μm follow up BCP
- heat treatments (RT – 800 °C), venting with N_2.

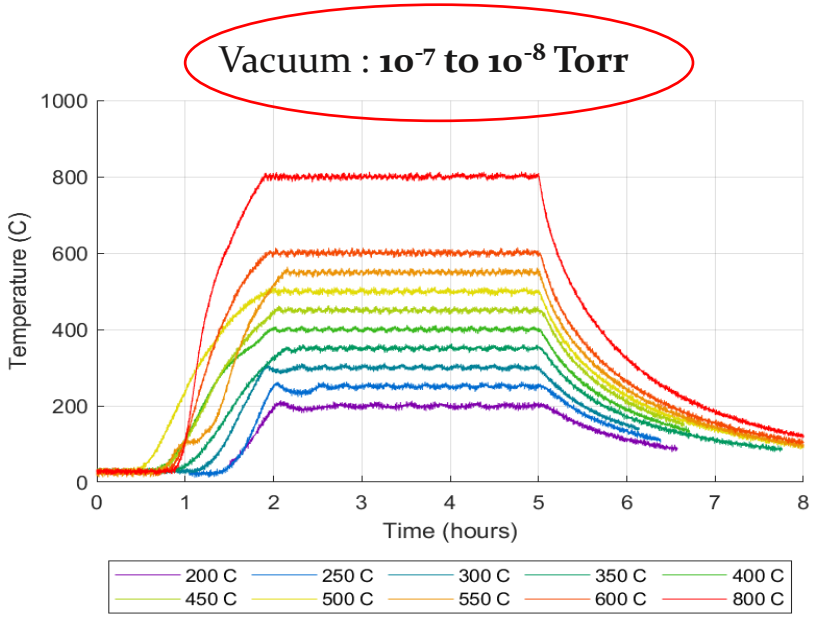


Nb sample



Heat treatments in the TRIUMF induction furnace.


Niobium Samples box.



Heat treatment : 100°C to 800°C (3 hrs)

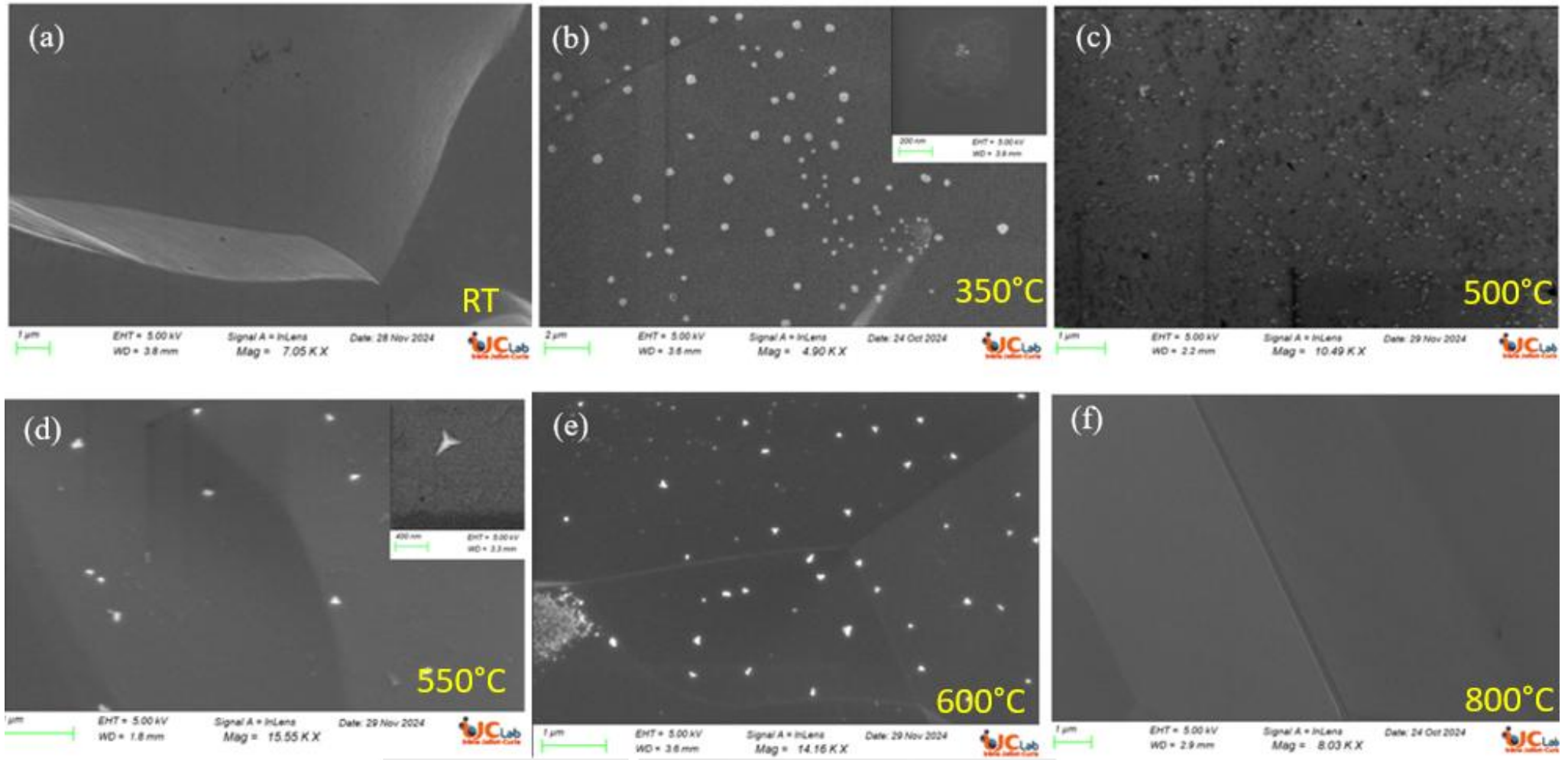
All results will be with TRIUMF samples!

C. Boutelaa, B.Laxdal (TRIUMF), P.Kolb (TRIUMF), J.Keir (TRIUMF)

SEM results

To have a preliminary study on the evolution of carbide formation on the surface [RT–800 °C]

SEM

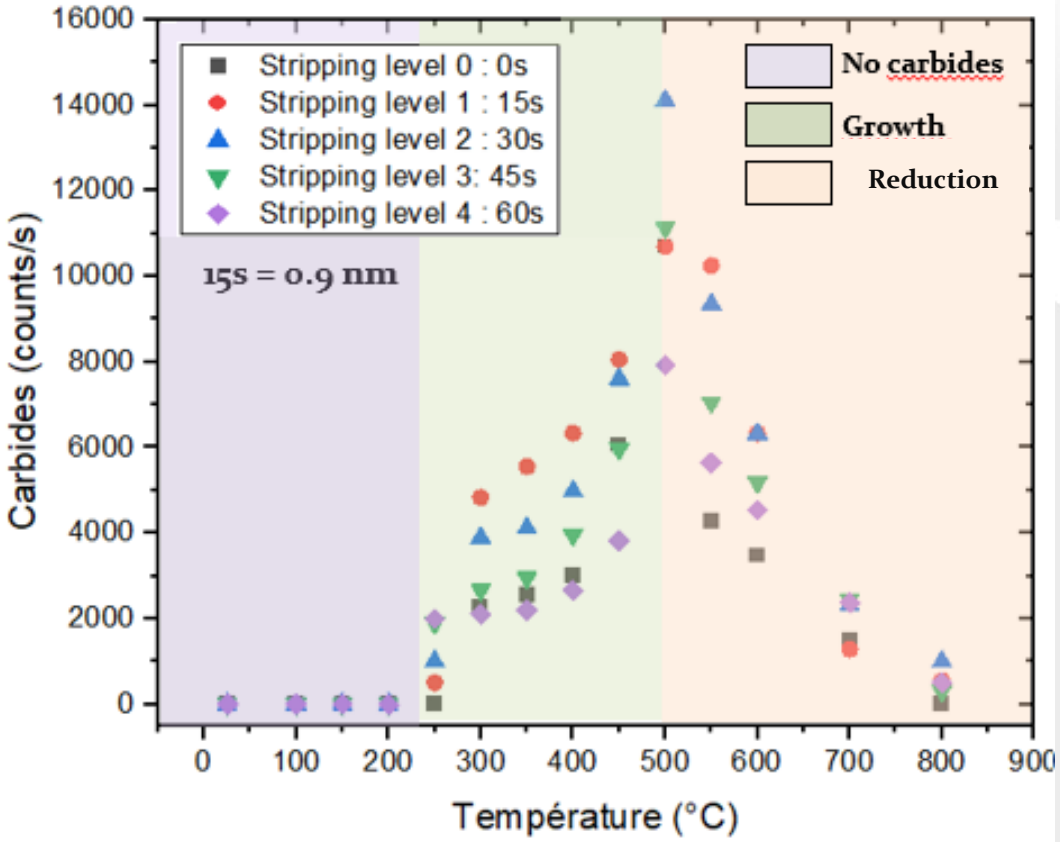


Ref : Chahinez Boutelaa, Poster, SRF conference 2025

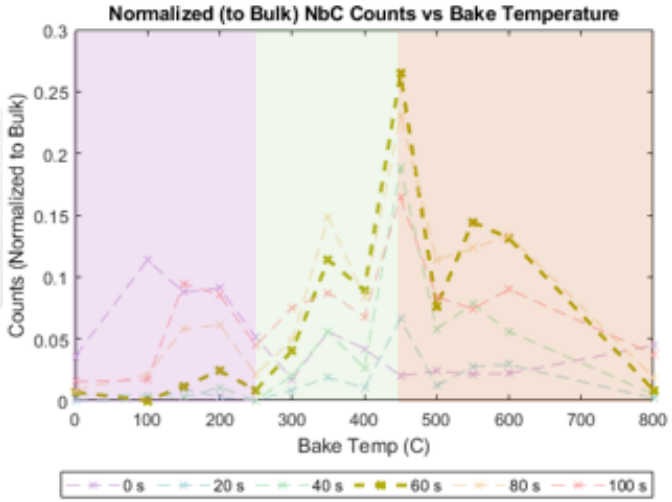
C. Boutelaa

Nb samples by XPS and SIMS

Mid-T Baking



Quantification of carbides by XPS as a function of temperature for different level of ion etching..



Quantification of carbides by SIMS as a function of temperature for different level of ion etching..

Philipp Kolb, TTC 2025, Korea

Discussion :

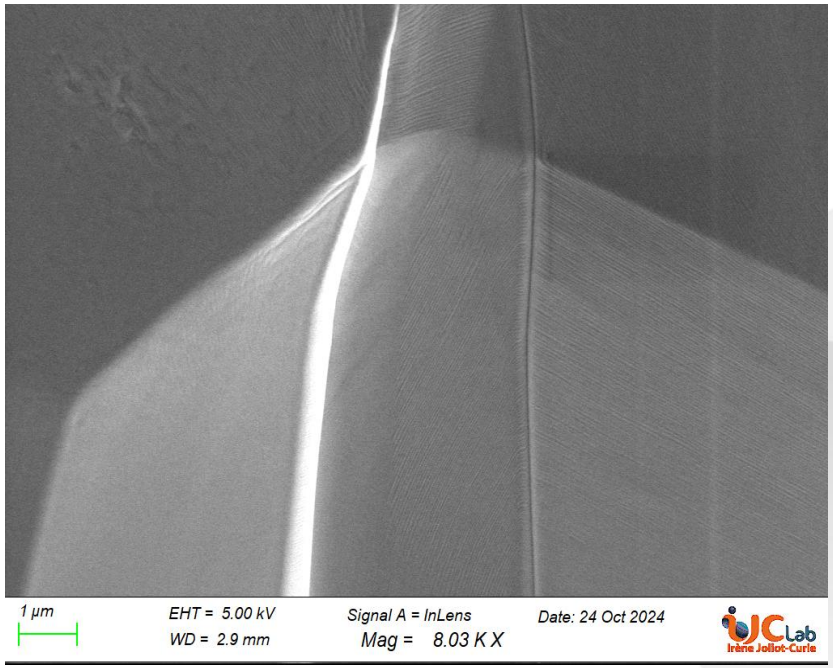
- Oxide layer protect until 250°C, increases progressively up to 500 °C.
- Reduction of carbides formation after 500°C
- We don't see carbide formation for 800°C !
- This results are confirmed by SIMS.

C. Boutelaa, B.Laxdal (TRIUMF), P.Kolb (TRIUMF), D.longuvergne (IJCLab), G.Sattonnay (IJCLab)

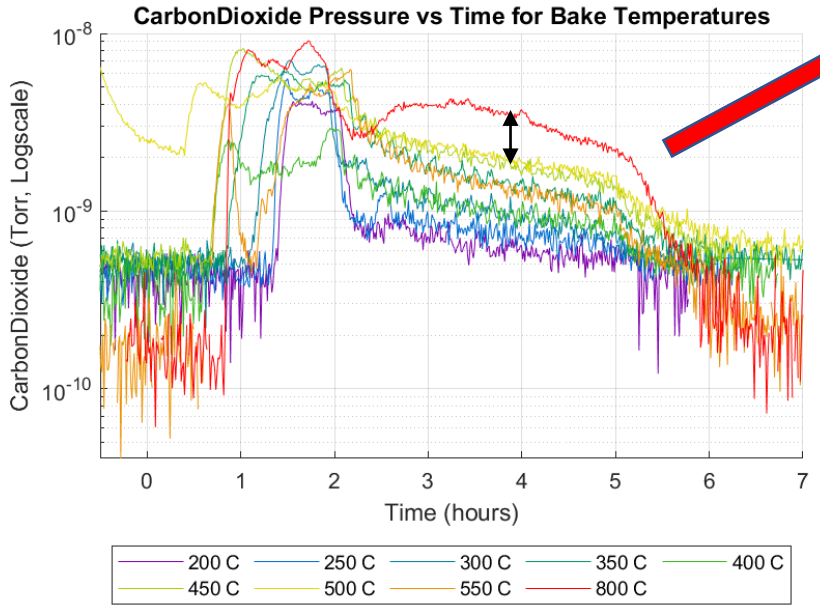
800°C with TRIUMF furnace

Why we don't have carbides for 800°C with TRIUMF furnace ?

Hypothesis: Desorption?



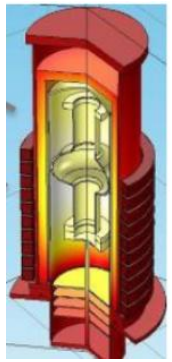
SEM pictures of Nb samples at 800°C during 3h.



Carbon dioxide pressure vs time.

"Its disappearance during the heating phase is due to the desorption of **CO or CO_2**"

A.Dacca ,Proceedings of the **1997** Workshop on RF Superconductivity, Abano Terme (Padova), Italy



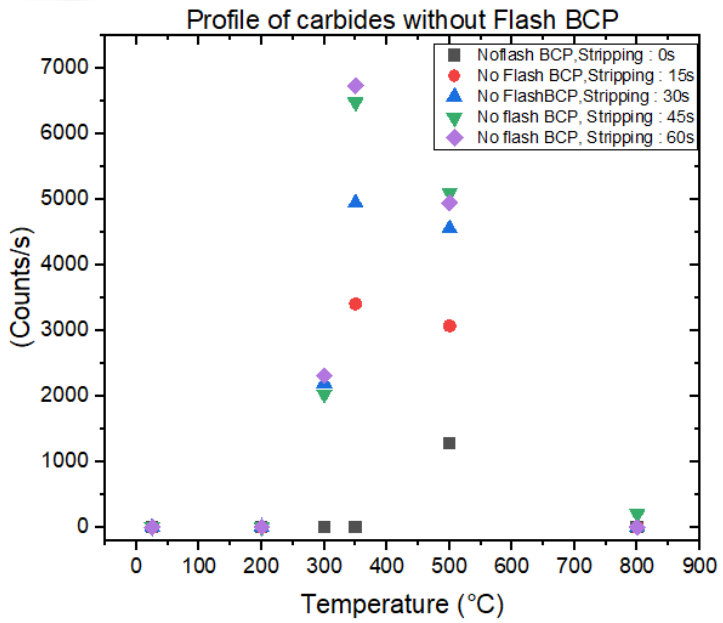
" we observe that the thermal treatment in **UHV at 1000°C** allows to obtain a surface fkee of contaminants (oxygen and carbon) "

A.Dacca ,Proceedings of the **1997** Workshop on RF Superconductivity, Abano Terme (Padova), Italy

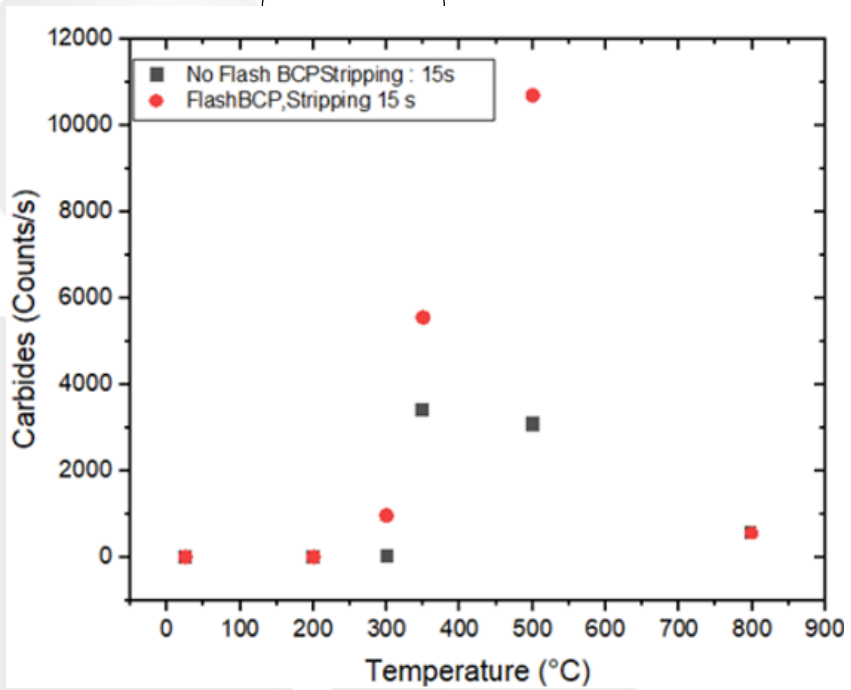
C. Boutelaa, B.Laxdal (TRIUMF), P.Kolb (TRIUMF)

No flash BCP vs Flash BCP with TRIUMF furnace

Mid-T Baking



Quantification of carbides (No flash BCP) by XPS as a function of temperature for different level of ion etching.



Quantification of carbides (No flash BCP vs flash BCP) by XPS as a function of temperature for 15s of ion etching.

Discussion :

- There are less carbides when flash BCP is skipped.

- Is that a proof that BCP is a source of carbon ?

=> Probably

« Carbon signal is weakest for an anodized surface, intermediate for a furnace cleaned surfaces and **strongest after chemical treatments**. »

Article of the 1980 on SURFACE STUDIES AND ELECTRON EMISSIONS, A. SEPTIER, Paris-Sud University

Bulk BCP 100µm → **800°C 3h** → ~~**Flash BCP**~~ → **200°C to 800°C 3h**

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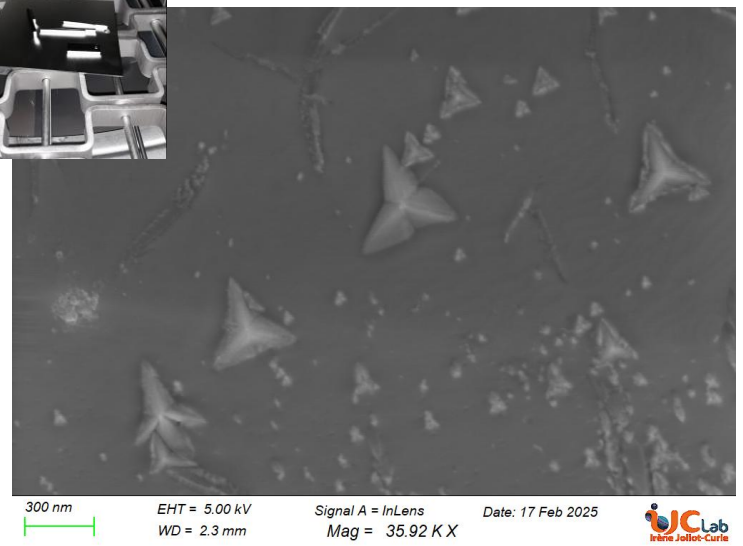
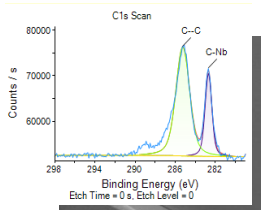
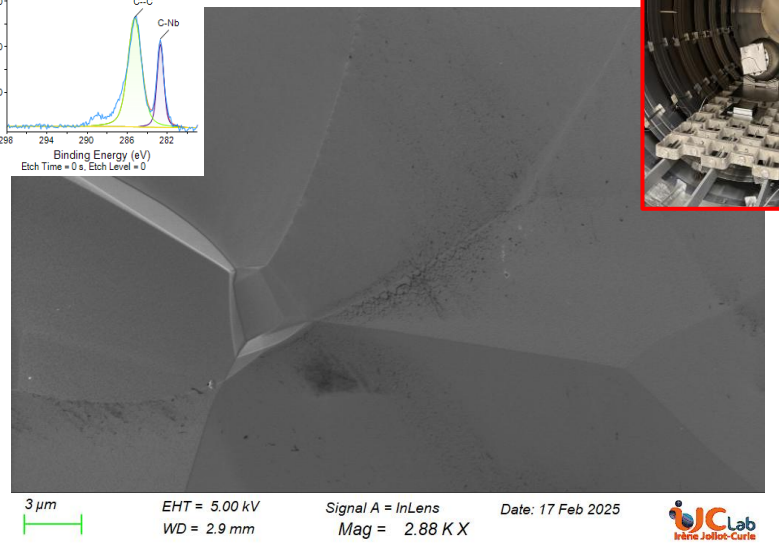
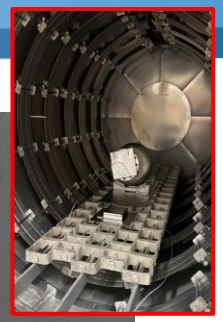
Baking with Supratech furnace



SEM pictures of Nb samples at 800°C (Supratech) during 3h.

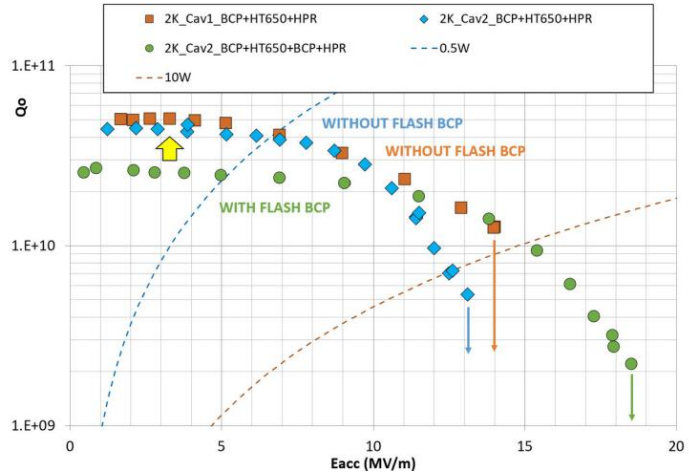






SEM pictures of Nb samples at 600°C (Supratech) during 10h.

Discussion:



Cycle :

- **Exposed directly in furnace** (not in Niobium box)
- 800°C during 3 h under vacuum (**10^{-5} mbar**)
- Cooling down to 40°C under vacuum.

Cycle :

- **Exposed in Niobium box**
- 600°C during 10 h under vacuum (**10^{-5} mbar**)
- Cooling down to 40°C under vacuum.

- **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 (Nb2C) ? => YES**

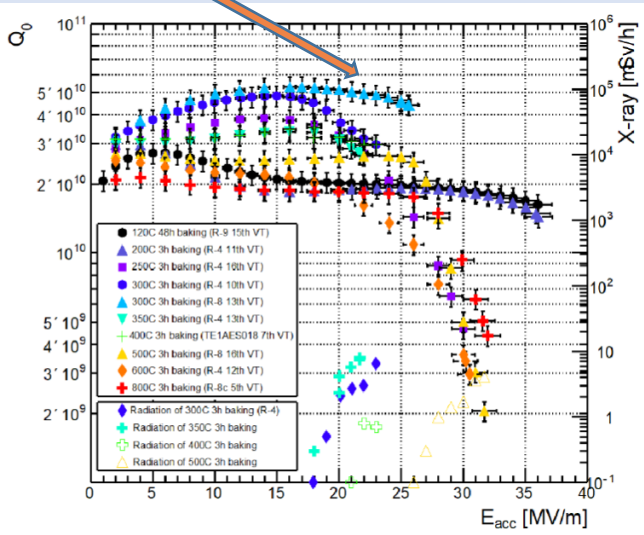
C. Boutelaa, D.longuergne (IJCLab), G.Sattonnay (IJCLab)

Contamination study of mid–T Bake with KEK furnace

- Perform heat treatments with KEK furnace (300°C-3h) for Nb samples

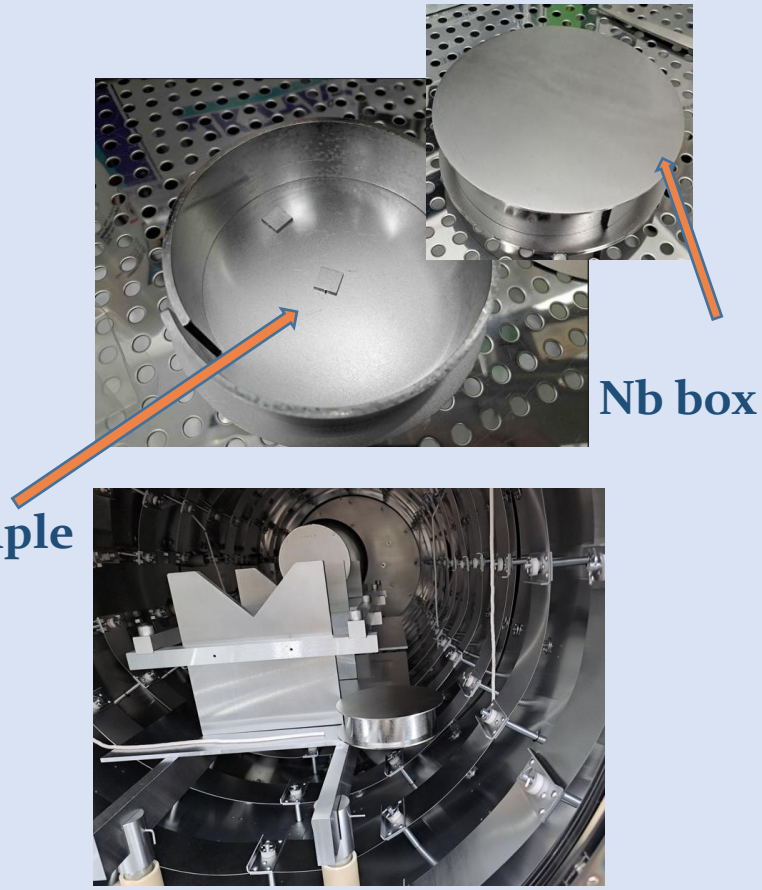
=> Analysis at IJCLab (XPS and SEM)

300°C-3h



H. Ito, KEK, SRF2021

Qualified furnace for Mid-T bake !



KEK furnace + Cavity + Nb samples

Sample preparation at TRIUMF :

- BCP bulk (100µm)
- Samples were annealed at 800°C 3h
- No flash BCP

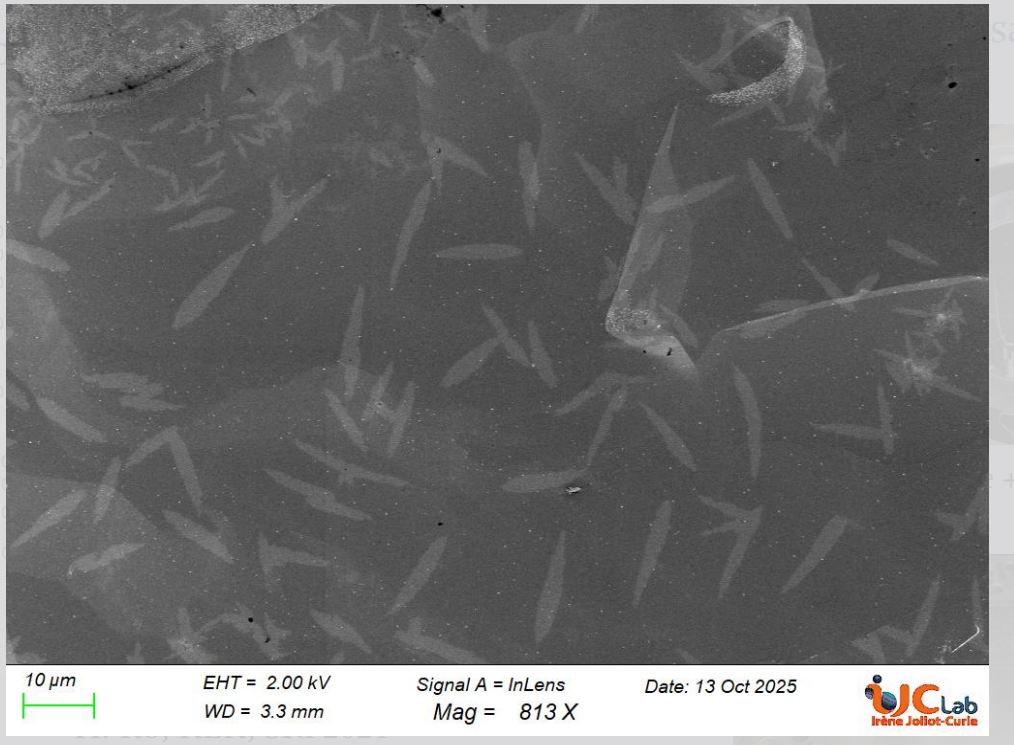
Mid-T bake conditions at KEK :

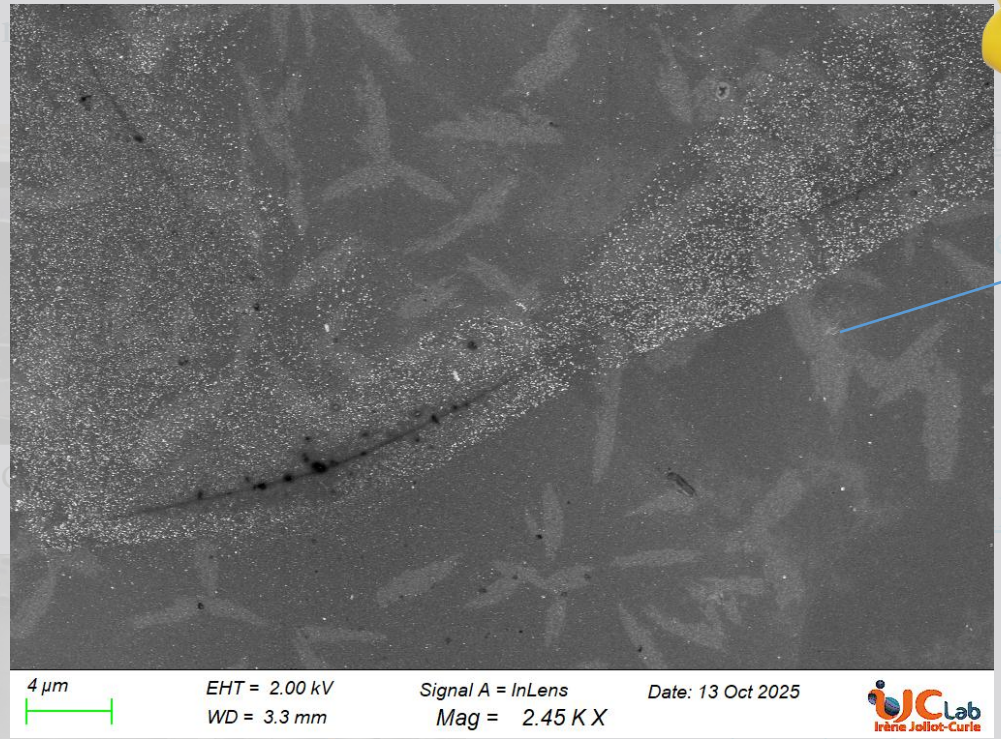
- Cavity and samples were annealed at 300°C 3h with :

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

Contamination study of mid–T Bake with KEK furnace



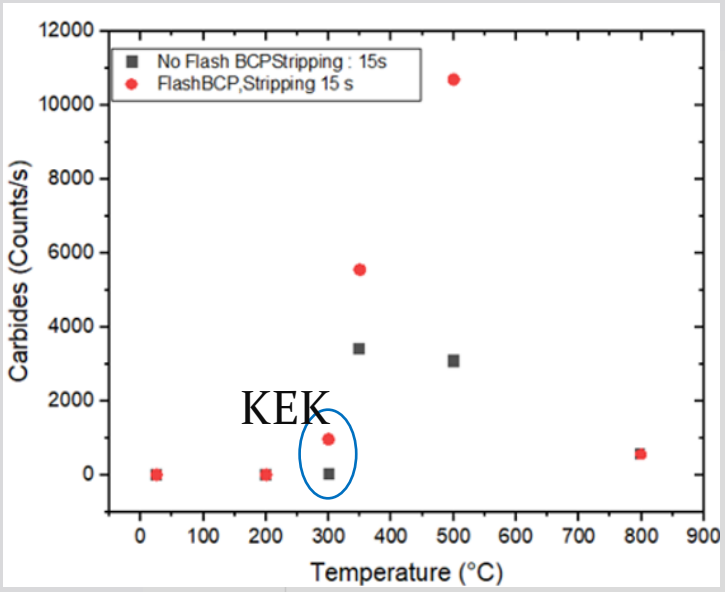


White point

- SEM shows **no carbide (Stars)** formation on the surface.
- SEM shows **white marks** across the entire surface !

@Vide et surface Plateform

Contamination study for mid–T Bake with KEK furnace

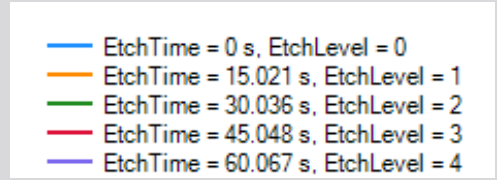


Quantification of carbides (No flash BCP vs flash BCP) by XPS as a function of temperature for 15s of ion etching.

- More carbides are observed with flash BCP (KEK furnace)
- Confirms the results with TRIUMF baking.

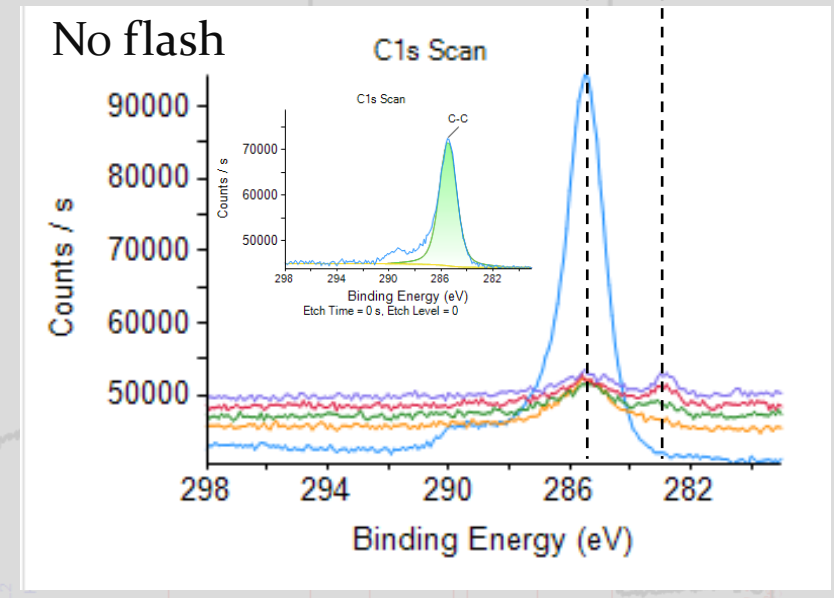
XPS survey of Nb sample at IJCLab :

- Presence of : C 1s (No flash) , C 1s (Flash)



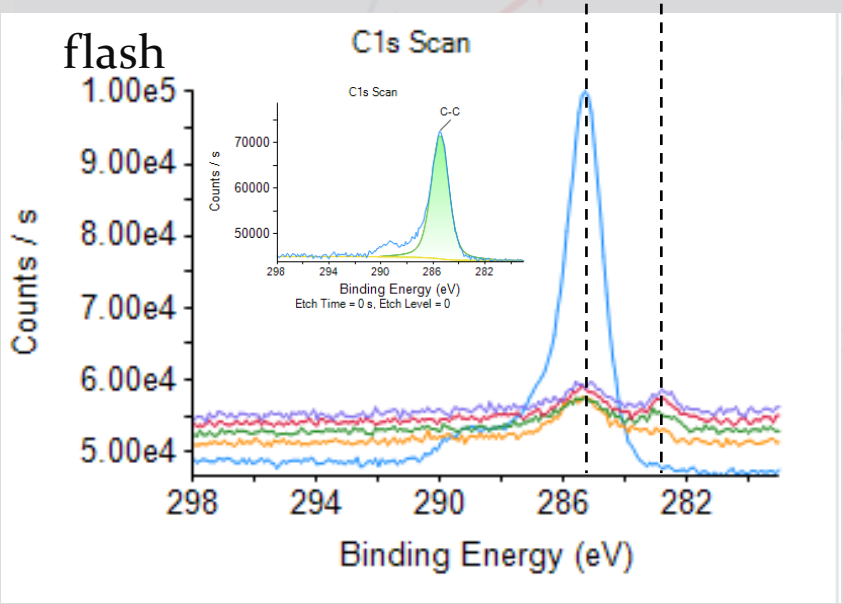
0.9nm =15s

C-C C-Nb



- Low presence of carbides after abrasion up to 2 nm for the sample without flash BCP.

C-C C-Nb



- Low presence of carbides after abrasion up to 1 nm for the sample with flash BCP.

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Summary and future outlook

Summary

=> The aim of the present work is to investigate the consequences of thermal treatments in the range 30 – 800°C on the surface chemical composition of niobium by **XPS, SEM and SIMS**.

⇒ The formation of carbides begins at 250 °C and increases progressively up to 500 °C. Beyond this temperature, their formation decreases until it completely disappears around 800 °C.

⇒ Does this dynamic depend solely on the sample or also on the furnace environment ??

⇒ Results different for 800°C (Samples) between TRIUMF and Supratech Furnace.

Outlook

⇒ ***In-situ* XPS study of Nb surface during mid-T bake with plasma treatment (TTC SRF 2026)**

Thank you for your attention

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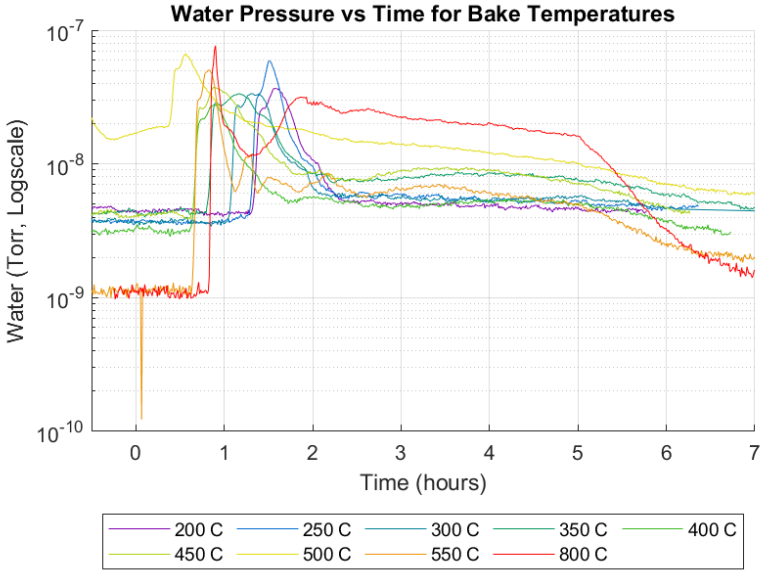
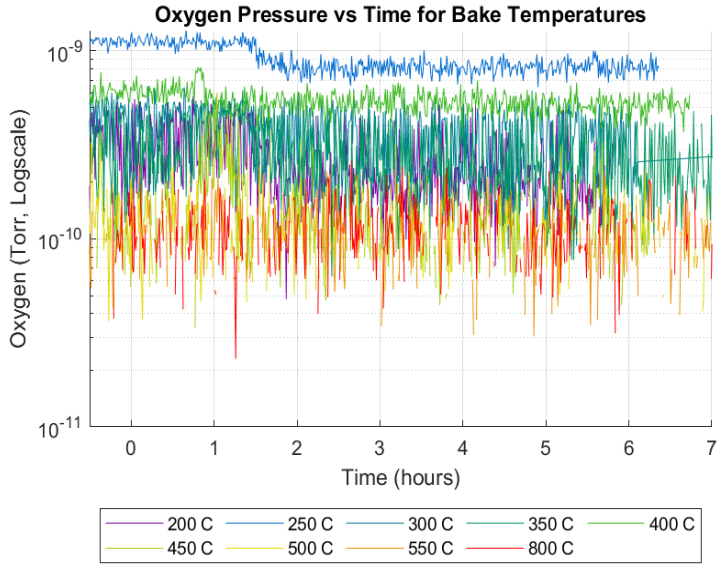
Thanks to :

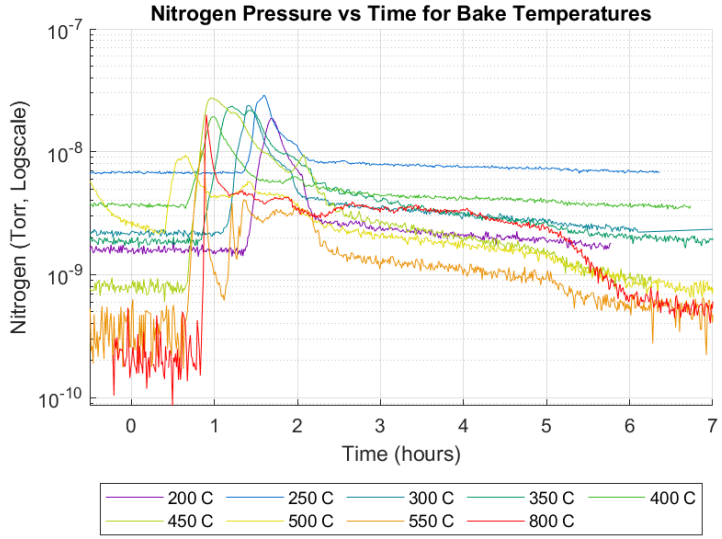
- All the department, groups and people that help with this experiment.

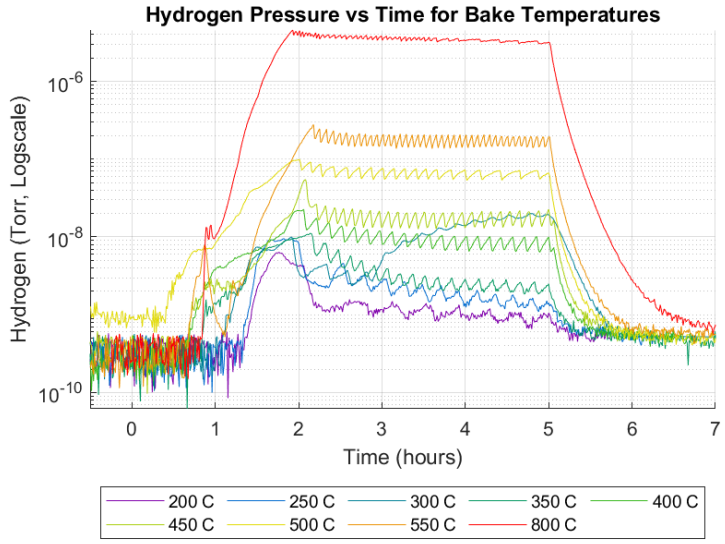
- MAVERICS Team, IJCLAB
- SRF team, TRIUMF
- Vide et Surface plateform, IJCLAB
- International Research Laboratory (IRL) beetween TRIUMF and CNRS
- ANR-21-ESRE-0049

BACK SLIDES

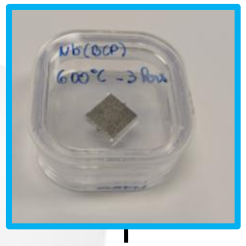

Water Pressure vs Time for Bake Temperatures
Water (Torr, Logscale)
Time (hours)
200 C
250 C
300 C
350 C
400 C
450 C
500 C
550 C
800 C


Oxygen Pressure vs Time for Bake Temperatures
Oxygen (Torr, Logscale)
Time (hours)
200 C
250 C
300 C
350 C
400 C
450 C
500 C
550 C
800 C

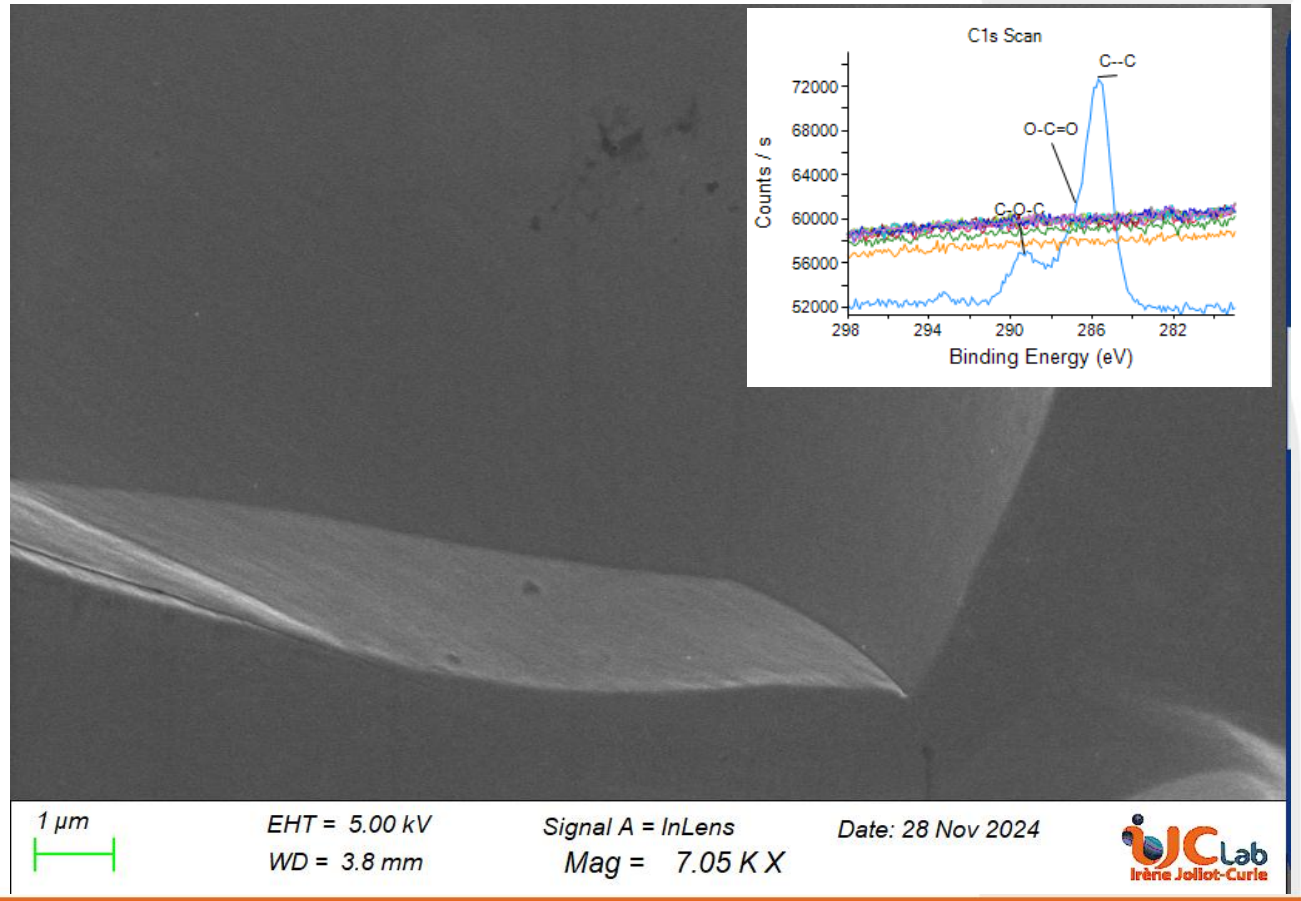

Nitrogen Pressure vs Time for Bake Temperatures
Nitrogen (Torr, Logscale)
Time (hours)
200 C
250 C
300 C
350 C
400 C
450 C
500 C
550 C
800 C


Hydrogen Pressure vs Time for Bake Temperatures
Hydrogen (Torr, Logscale)
Time (hours)
200 C
250 C
300 C
350 C
400 C
450 C
500 C
550 C
800 C

Same sample (1 year ago Vs Now)

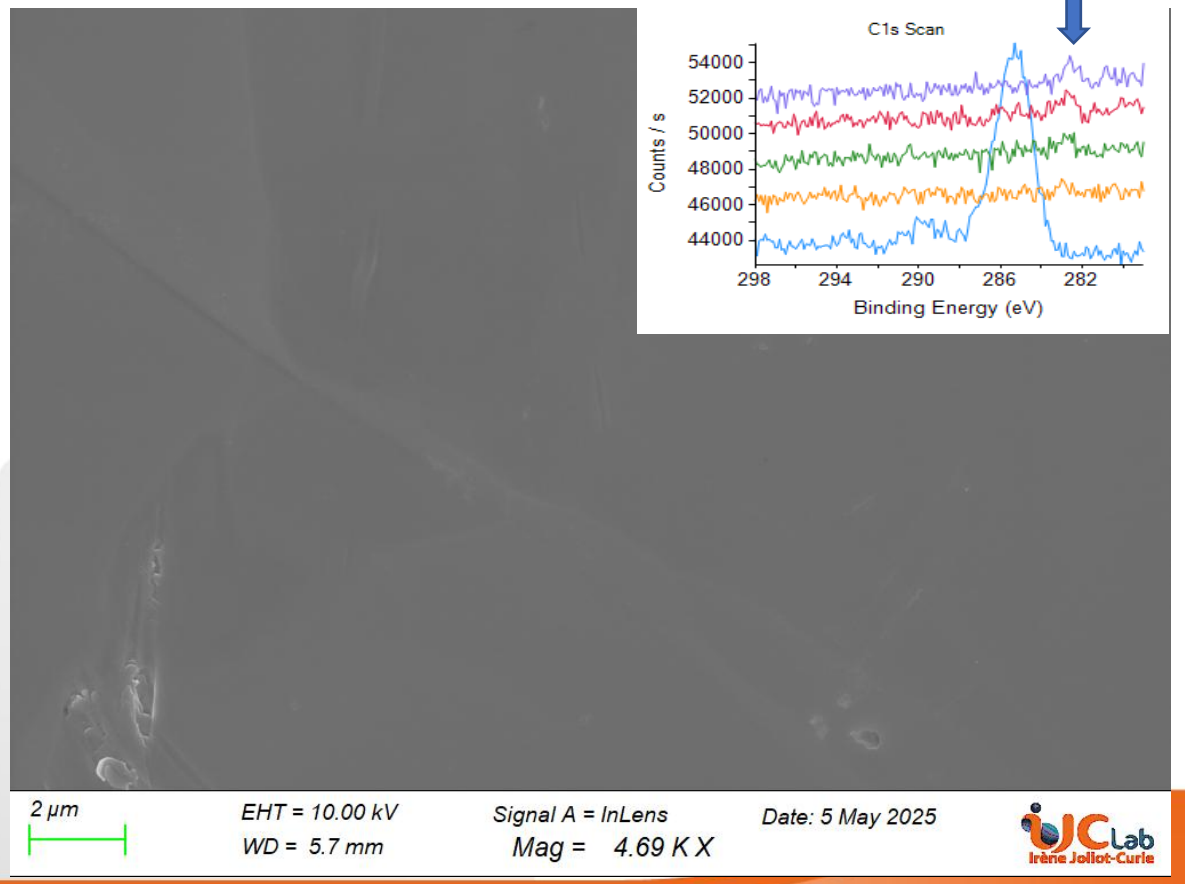


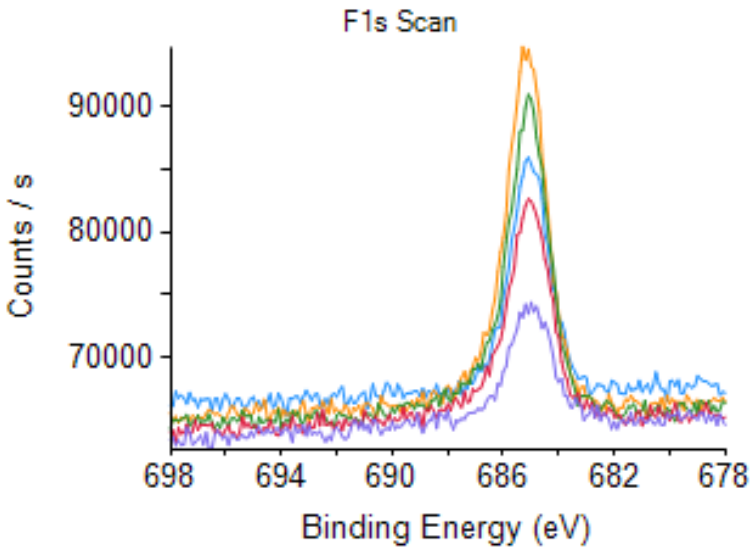
Nb-RT-BCP-2024



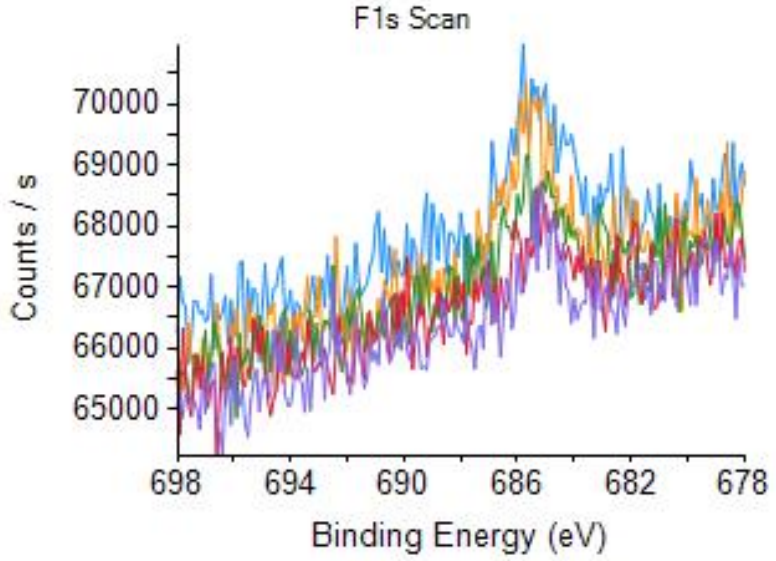
Nb-RT-BCP-2025

Nb-C

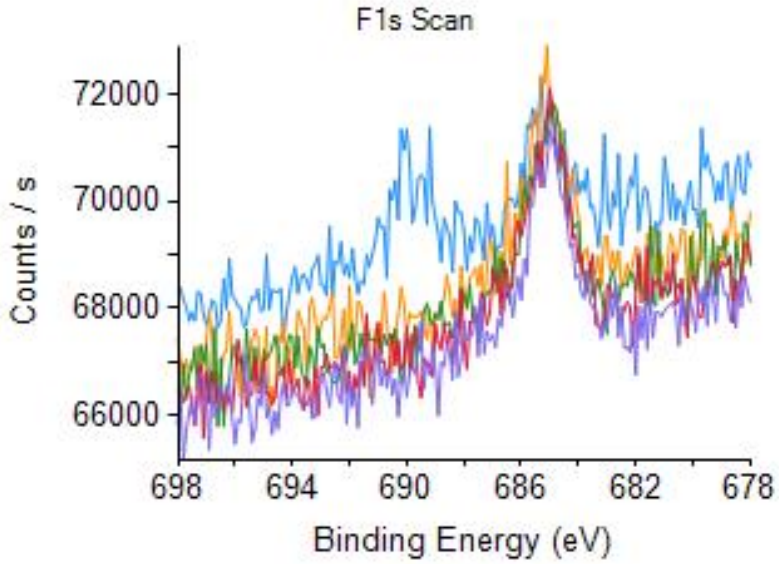




RT – flash

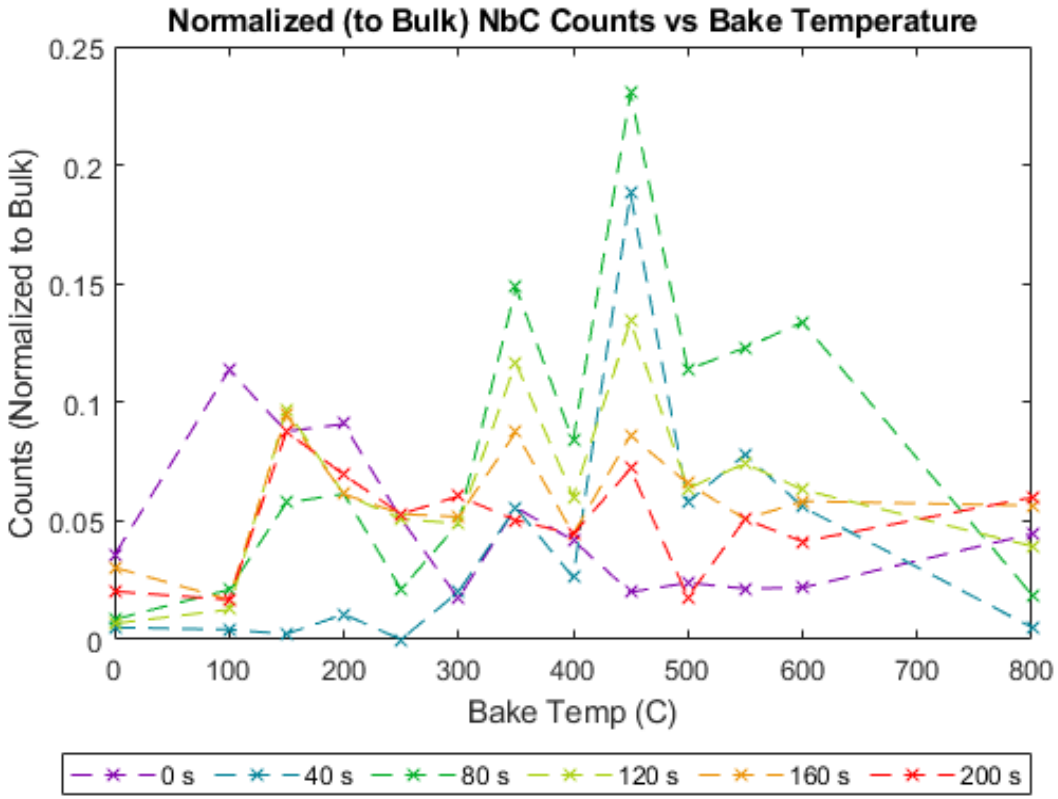


300°C-no flash-KEK



300°C-flash-KEK

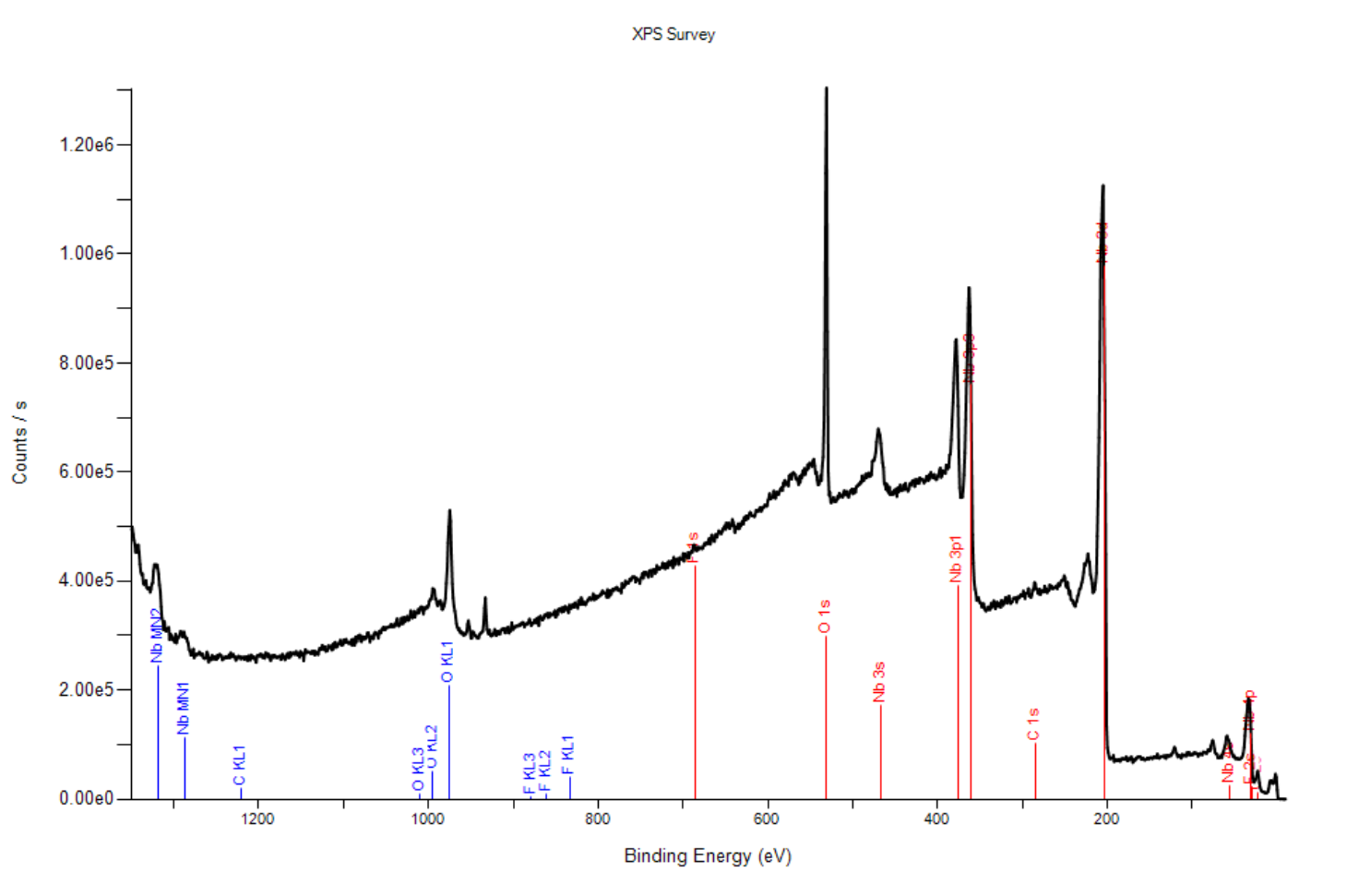
Please use this plot in your experiments

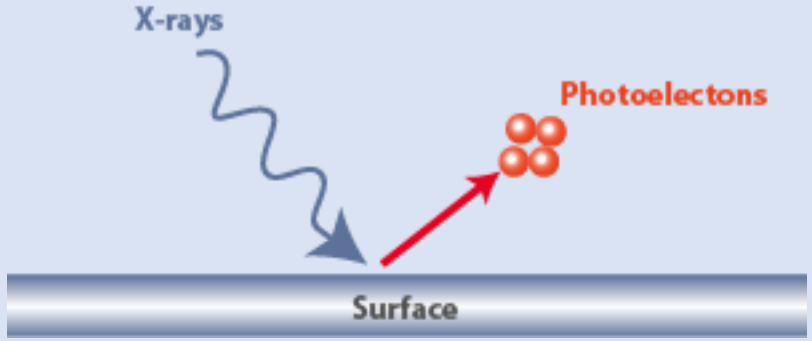


Contamination study of mid–T Bake with KEK furnace

KEK

Analysis at IJCLab by XPS : X-Ray spectroscopies





XPS survey of Nb sample at IJCLab :

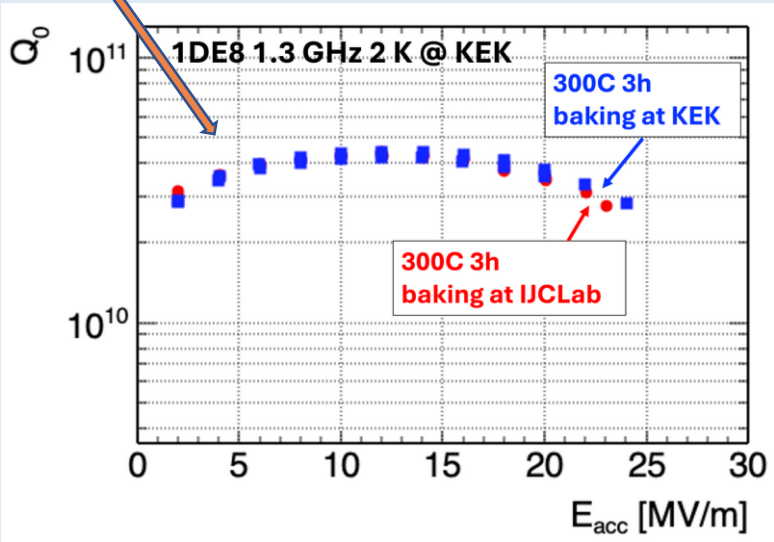
- Presence of : Nb, C , O and F

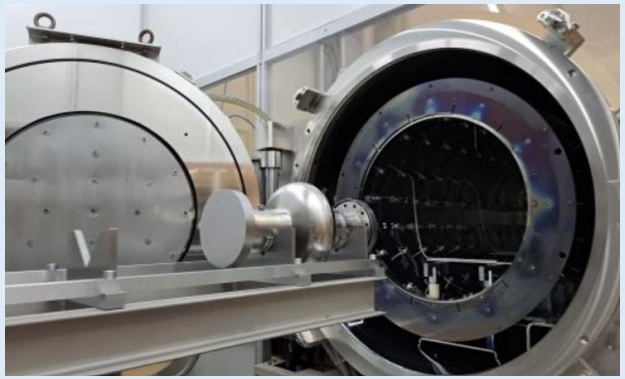
@Vide et surface Plateform

Contamination study of mid–T Bake with KEK furnace

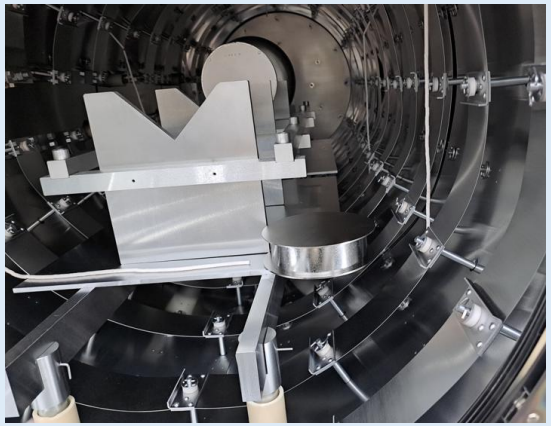
Aims at KEK :
Perform heat treatments with KEK furnace
(300°C-3h) for elliptical cavities (1.3 GHz)+ Nb sample => Analysis at IJCLab (XPS and SEM)

300°C-3h





KEK furnace + Cavity + Nb sample



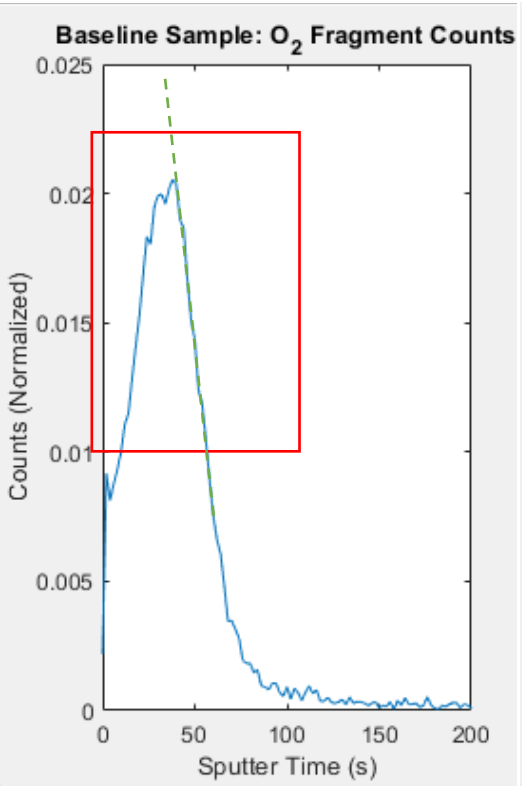
Cavity preparation :
1) bulk EP at CEA
2) 600C annealing at IJCLab
3) flash EP at CEA
4) Test at CEA (2024)
5) 300C 3h baking at IJCLab
6) Test at KEK in 2024
7) 800C annealing to reset mid-T without flash EP at KEK
8) Test at KEK in 2025
9) 300C 3h baking at KEK
10) Test at KEK in 2025 (same results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

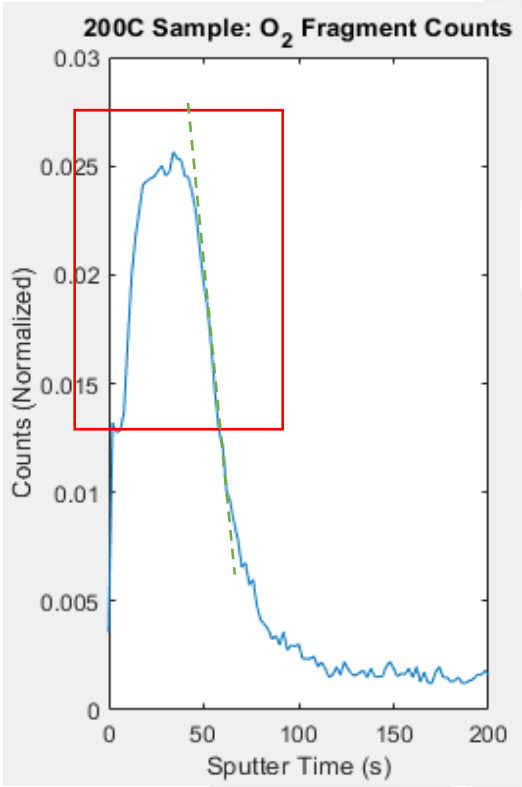
Mid-T bake conditions at KEK :
- Cavity and samples were annealed at 300°C 3h with :

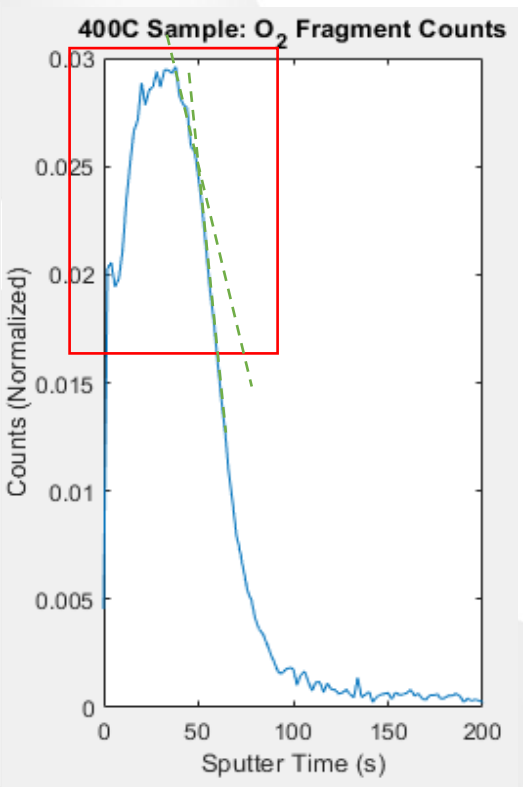
Before (26°C): <3.2e-6 Pa = <3.2e-8 mbar
During (300°C): 8.2e-5 Pa
After (35°C): 2.5e-6 Pa

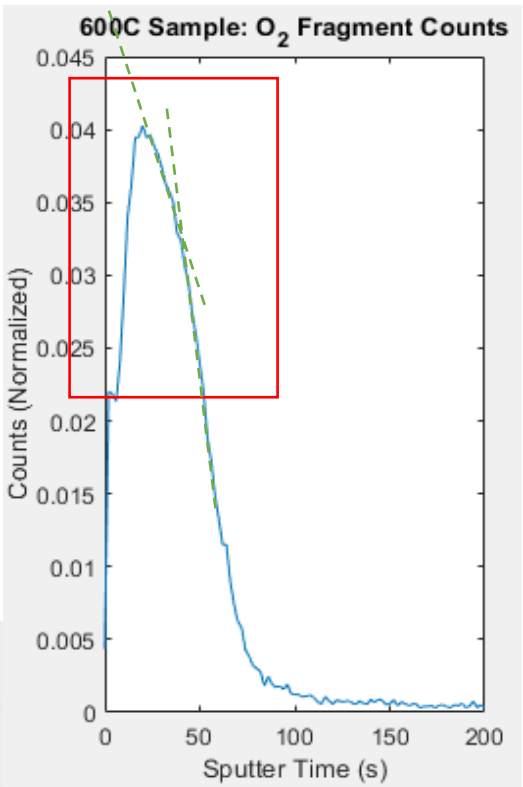
- Purge with Argon at 50°C

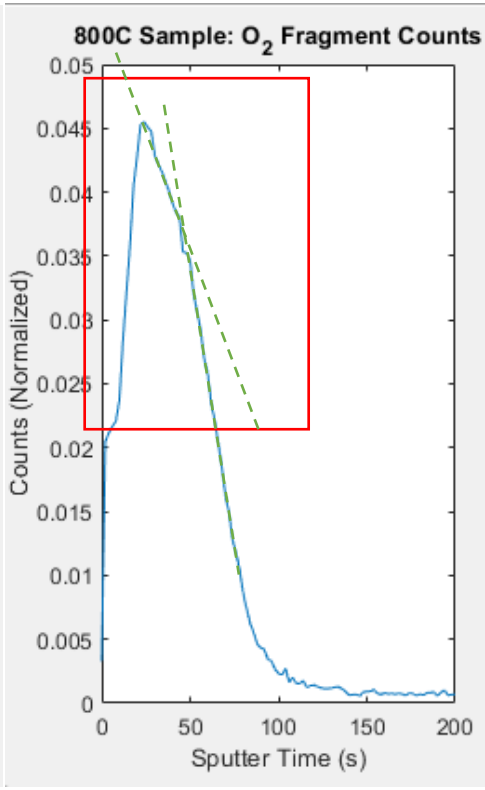
Oxygen Diffusion Behavior





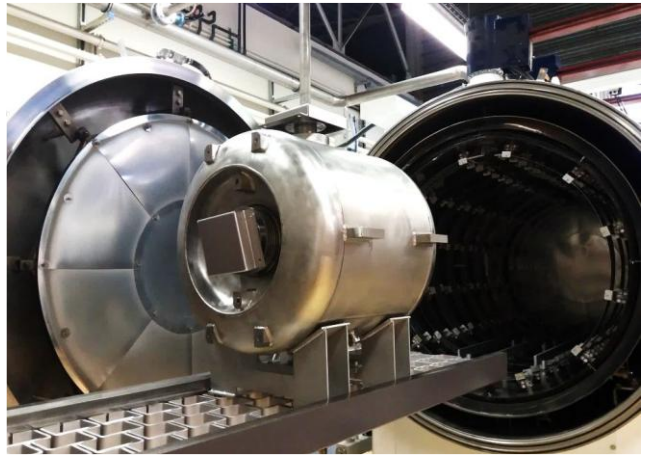




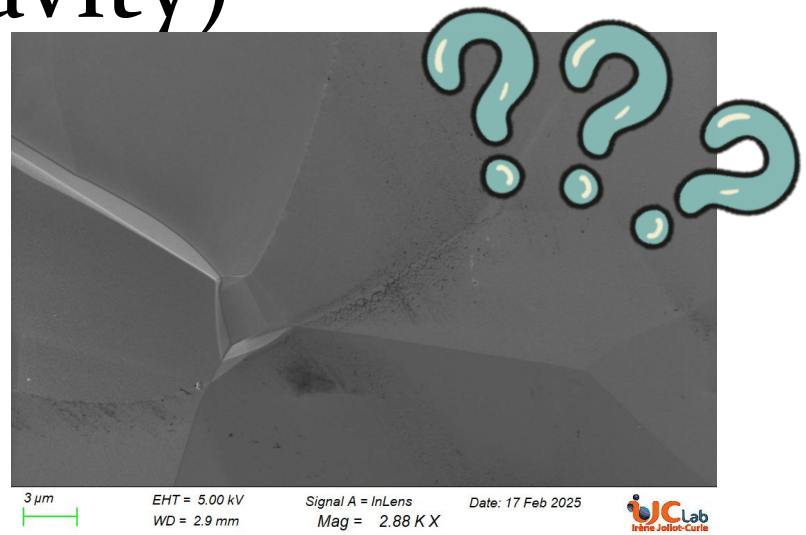


- Profiles don't seem to exhibit classic diffusion profiles straightforwardly
- Qualitatively: as bake temp goes up, O2- profile as we move past peak morphs from linear to two-sloped
- Could be evidence of diffusion of oxygen deeper into samples? Unsure

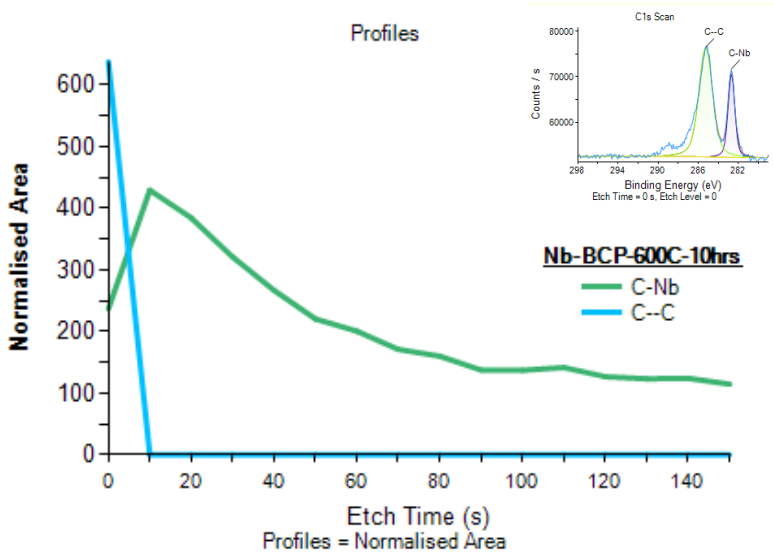
600°C for 10h with supratech furnace (Nb sample + cavity)



600°C for 10h with Supratech furnace



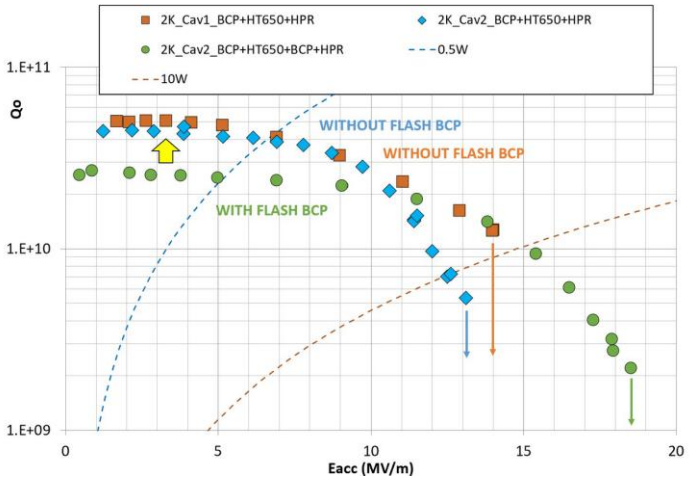
SEM pictures of Nb samples at 600°C during 10h.



Carbides depth profile for 600°C.

Nb sample

MYRRHA Simple Spoke Cavity



D.Longuvergne,JT-RTV-2025

Discussion:

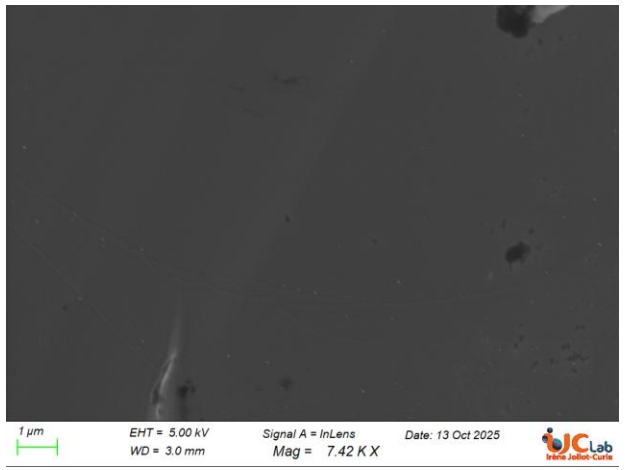
- **Best results at IJClab for 650°C 10hrs.**

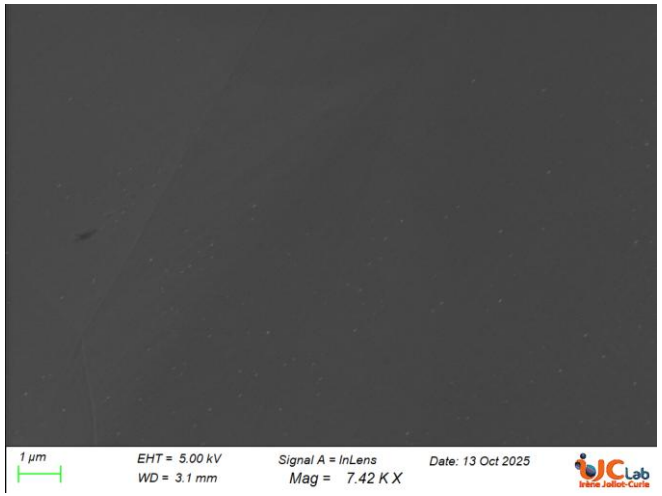
Questions :

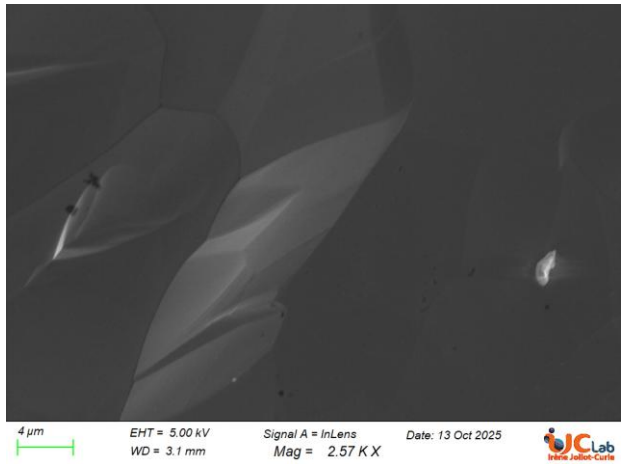
- **SEM results confirm good Qo test, but we observe Nb-C by XPS !**
- **Issues only with the structure of carbides?**

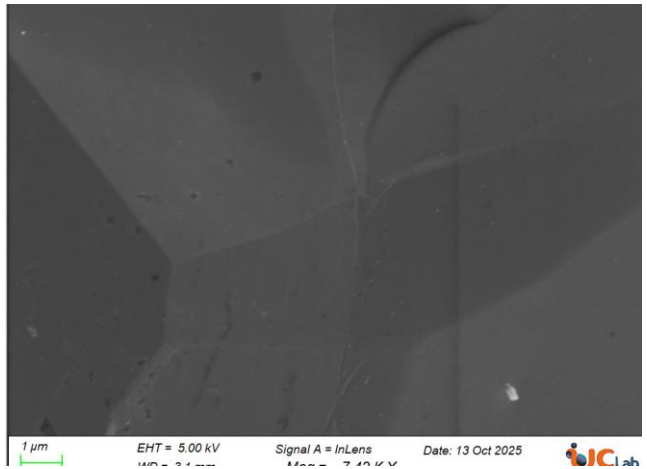
C. Boutelaa, D.longuergne (IJCLab), G.Sattonnay (IJCLab)

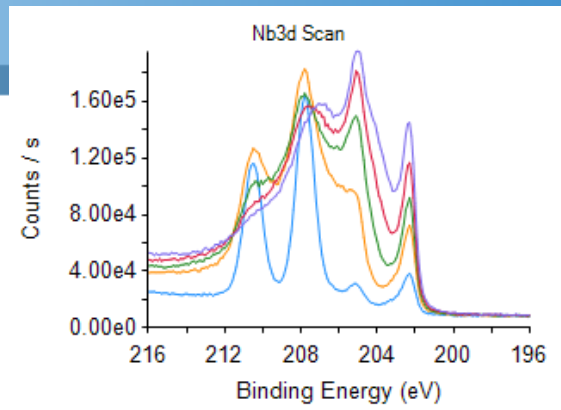
RT – no flash

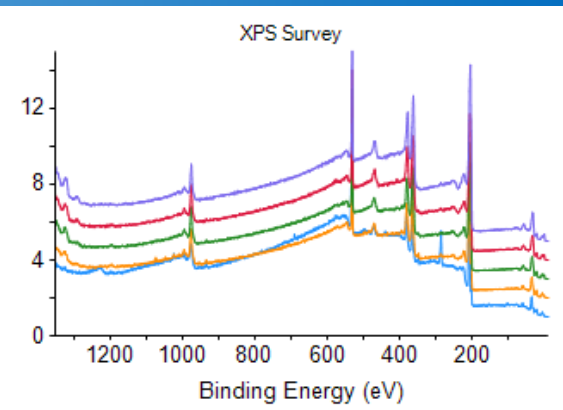

1 µm
EHT = 5.00 kV
WD = 3.0 mm
Signal A = InLens
Mag = 7.42 K X
Date: 13 Oct 2025

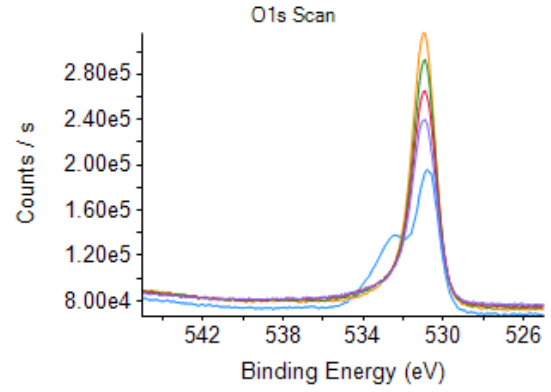

1 µm
EHT = 5.00 kV
WD = 3.1 mm
Signal A = InLens
Mag = 7.42 K X
Date: 13 Oct 2025

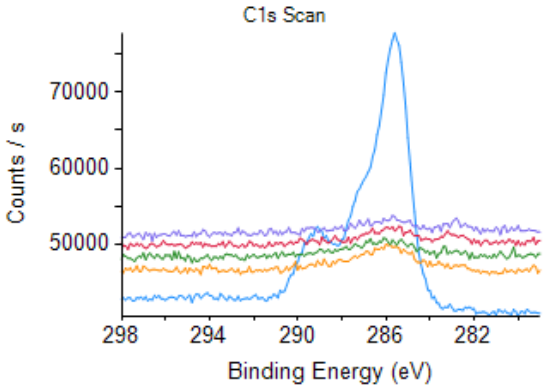

4 µm
EHT = 5.00 kV
WD = 3.1 mm
Signal A = InLens
Mag = 2.57 K X
Date: 13 Oct 2025

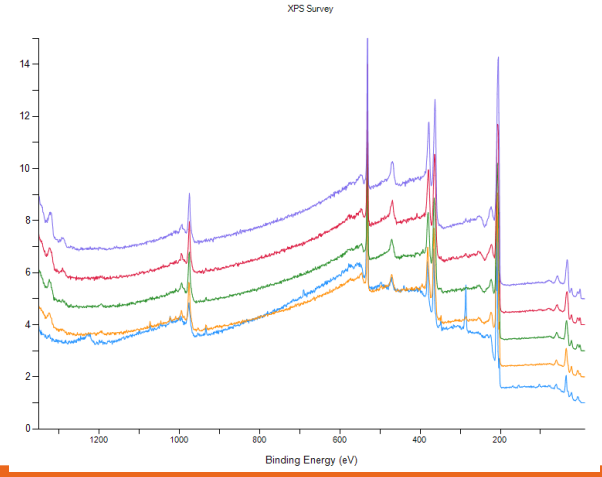

1 µm
EHT = 5.00 kV
Signal A = InLens
Date: 13 Oct 2025

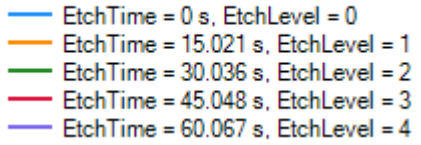

Nb3d Scan
Counts / s
1.60e5
1.20e5
8.00e4
4.00e4
0.00e0
216
212
208
204
200
196
Binding Energy (eV)


XPS Survey
12
8
4
0
1200
1000
800
600
400
200
Binding Energy (eV)


O1s Scan
Counts / s
2.80e5
2.40e5
2.00e5
1.60e5
1.20e5
8.00e4
542
538
534
530
526
Binding Energy (eV)


C1s Scan
Counts / s
70000
60000
50000
298
294
290
286
282
Binding Energy (eV)


XPS Survey
14
12
10
8
6
4
2
0
1200
1000
800
600
400
200
Binding Energy (eV)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EtchTime = 15.021 s, EtchLevel = 1
EtchTime = 30.036 s, EtchLevel = 2
EtchTime = 45.048 s, EtchLevel = 3
EtchTime = 60.067 s, EtchLevel = 4