New insights on the SRF Nb cavities performance from spectroscopic data.

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Outline

- Modulation of the Nb atoms reactivity by their neighbors (Nb 3d core levels binding energy for Nb in Nb3Sn (XPS)

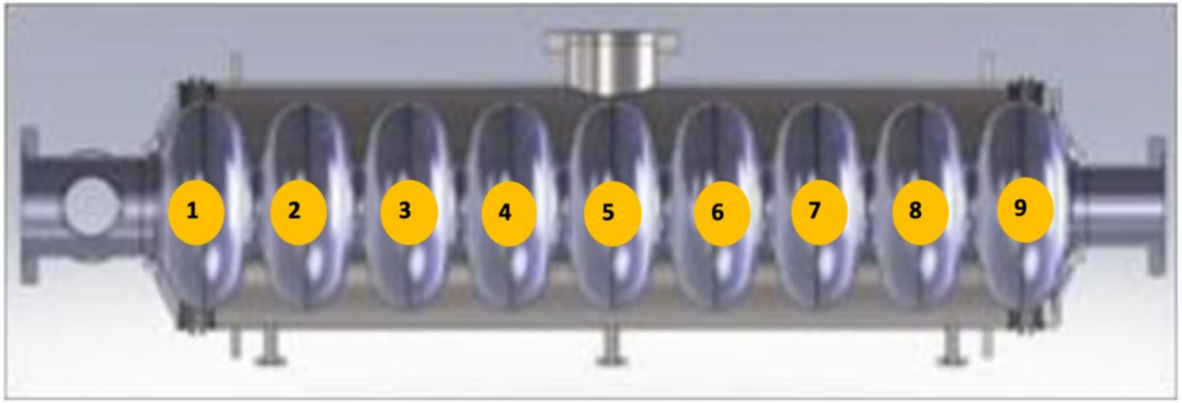
- Evidence on the role of N doping on the modulation of Nb atoms reactivity in Nb SRF cavity from the hydride formation (Raman spectra at low temperatures).

- XPS Depth profile spectra as source of information on the reactivity modulation of Nb in N-doped SRF cavity cutouts

- Morphologies observed in N-doped Nb SRF cavity (SEM)

- Summary

Analyzed samples



All the samples herein analyzed were cutouts:

-EP treated single cavity: random election

-2/0 N-doping 9-Cell structure: Cell 1 (identified quench spot) and cell 5

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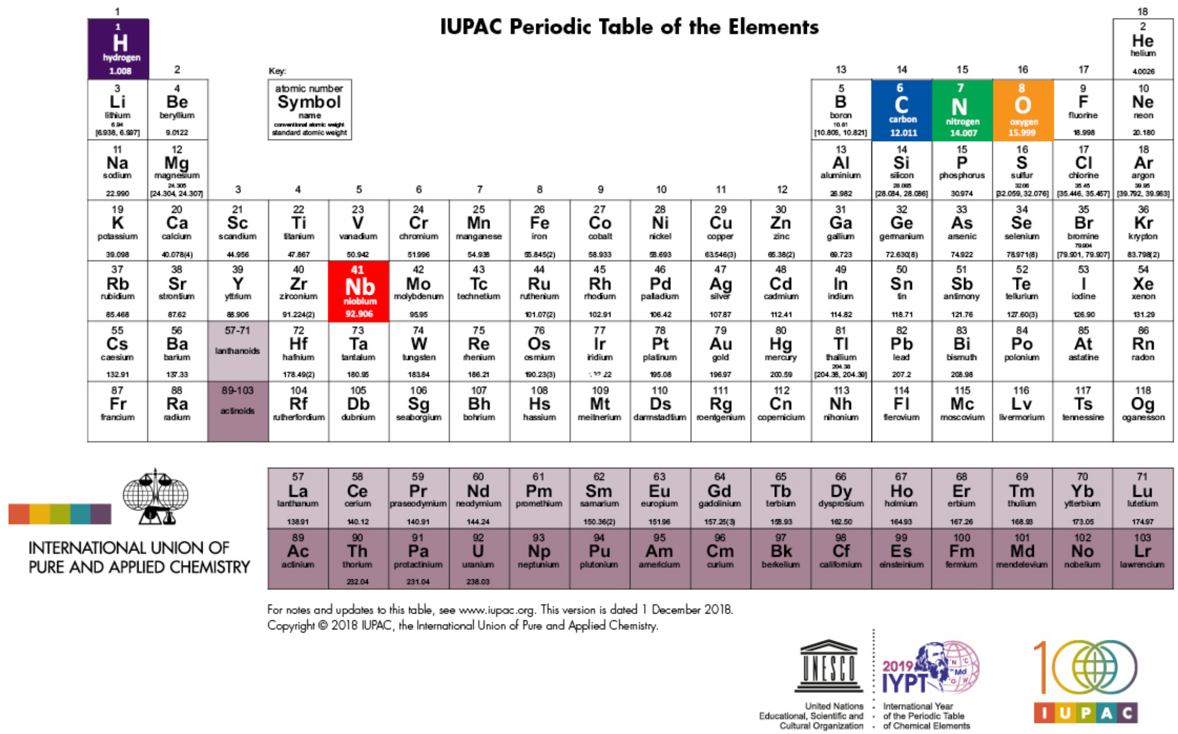
On the Nb reactivity and its impact on the near-surface physics and chemistry



Nb shows a high reactivity for the sorption and reaction with **Hydrogen**, **Nitrogen** and **Oxygen.** These three elements can be found dissolved in the near surface region and, in the bulk and forming precipitates under certain conditions.

Nitrogen is known to have a positive effect on the cavity performance!!

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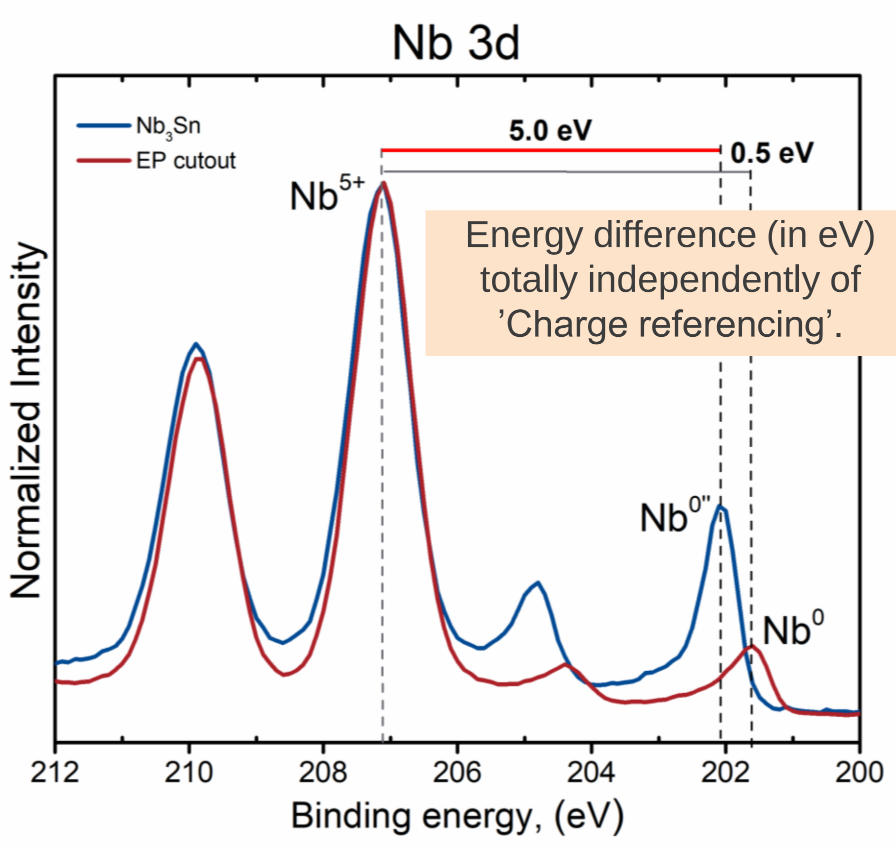
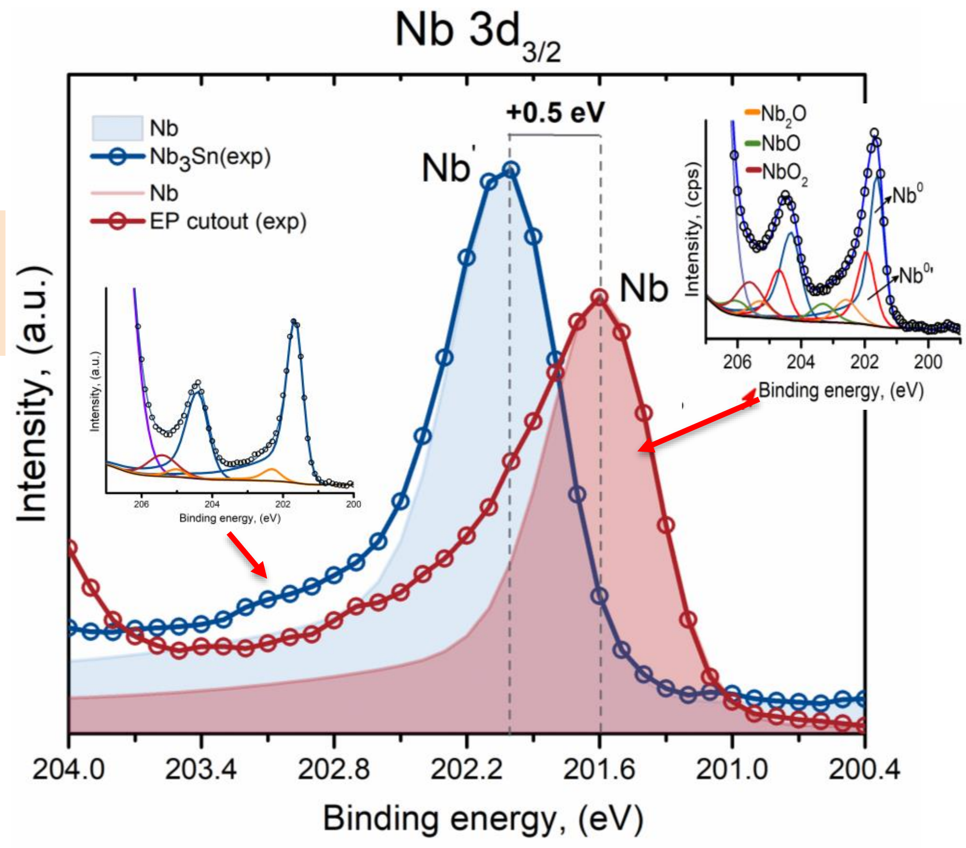
On the Nb surface reactivity: Nb in Nb_3Sn as reference

Chemical shift found at the Nb 3d spectral low-BE region for Nb in Nb_3Sn





In Nb_3Sn, Nb atoms have a positive character (compared to metallic Nb); the Sn atoms are subtracting electron density from their Nb neighbors
∴ the interaction of Nb ⟷ H, N and O is weaker

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Motivation

It is well-known the positive effect that the N-doping has on the Nb SRF cavity performance.

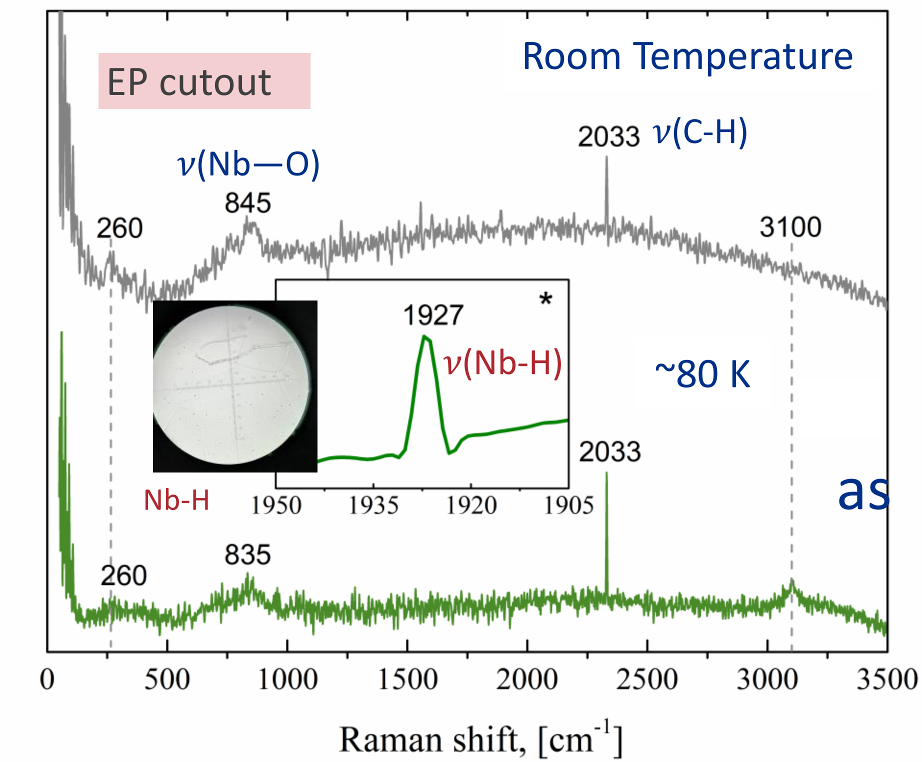
We try to understand the nature of such effect, from an atomic point of view, considering a possible interaction between Nb and N atoms, and how that interaction could modulate the physical and functional properties of Nb metal in the SRF cavity.

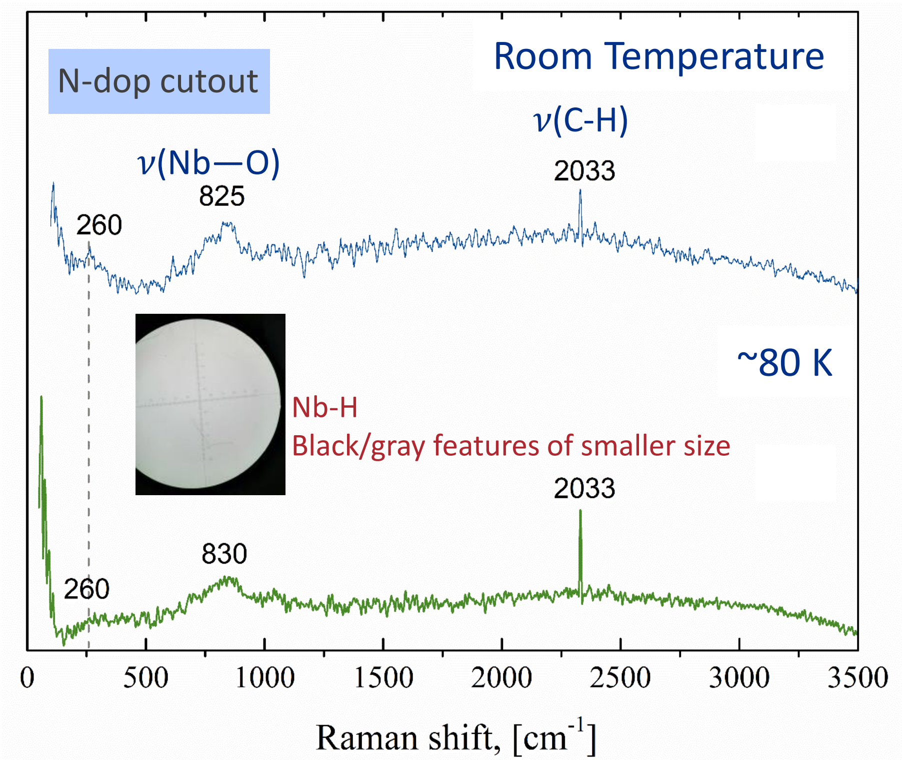
Nitrogen occupies an intermediate position, within C, N and O for their reactivity with Nb.

Could the presence of N in the Nb metal network be modifying its reactivity and related physical properties?

In this study, we support such hypothesis from experimental evidence and propose new experiments to shed light on that possible mechanism to explain the nature of the N positive effect on the Nb SRF cavity performance.

Formation of Nb-hydrides





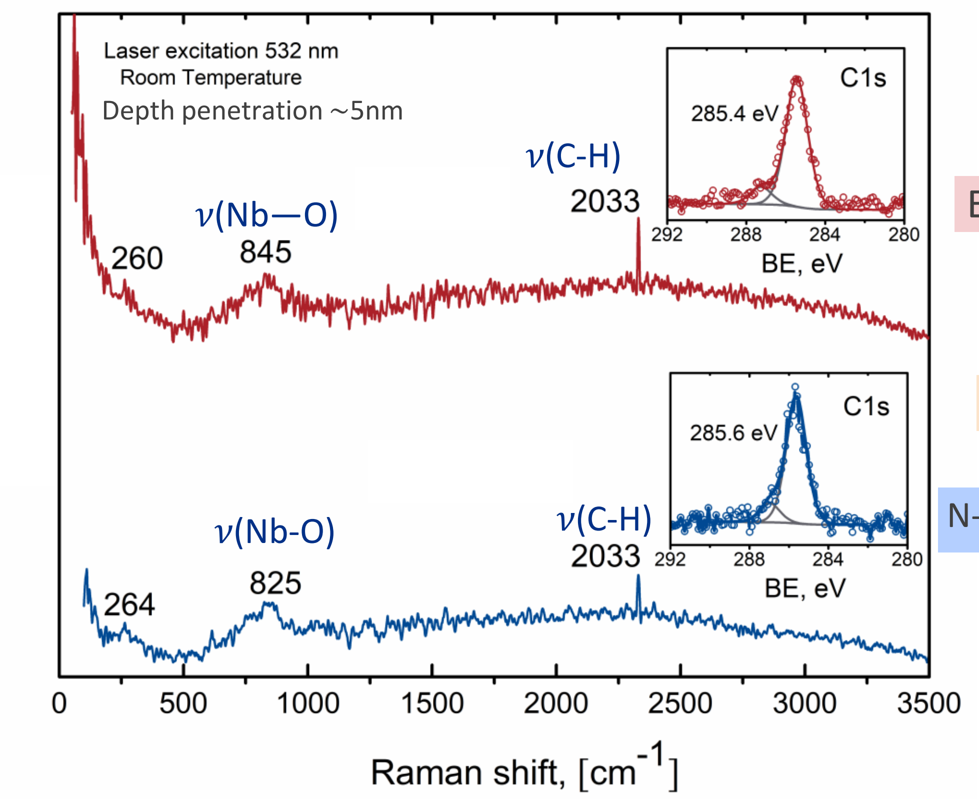
Raman spectra recorded under RT and cryogenic conditions (~77K)

The optical images and Raman spectra under low T, shown a evidence that the cavity treatment with N reduces the material ability to form hydrides and the formed ones are of small size.

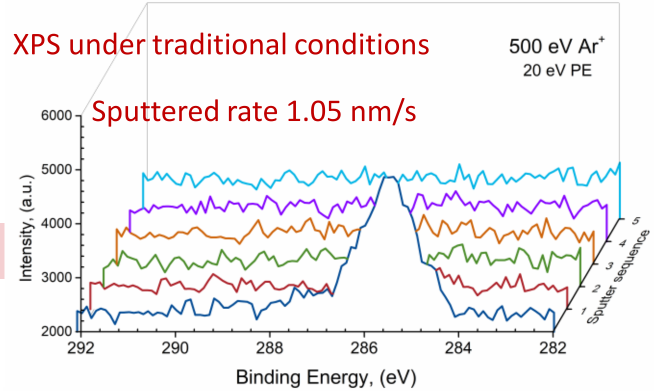
This contributes to support the hypothesis regarding to a lower material (Nb) reactivity in the presence of N.

Sorption of species at the Nb surface

Nb, at its surface, is able to absorb species from the environment, e.g. carbon containing compounds, etc.

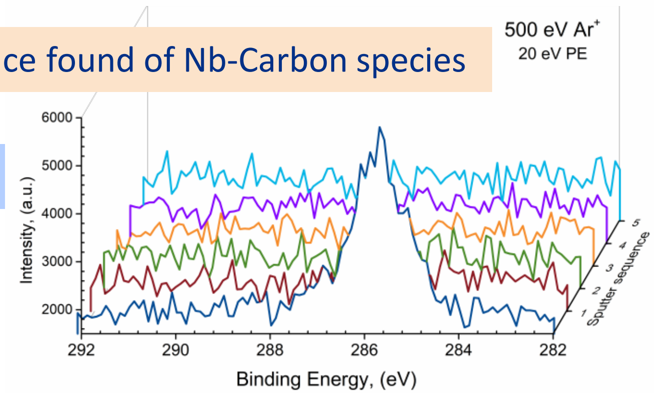


EP cutout



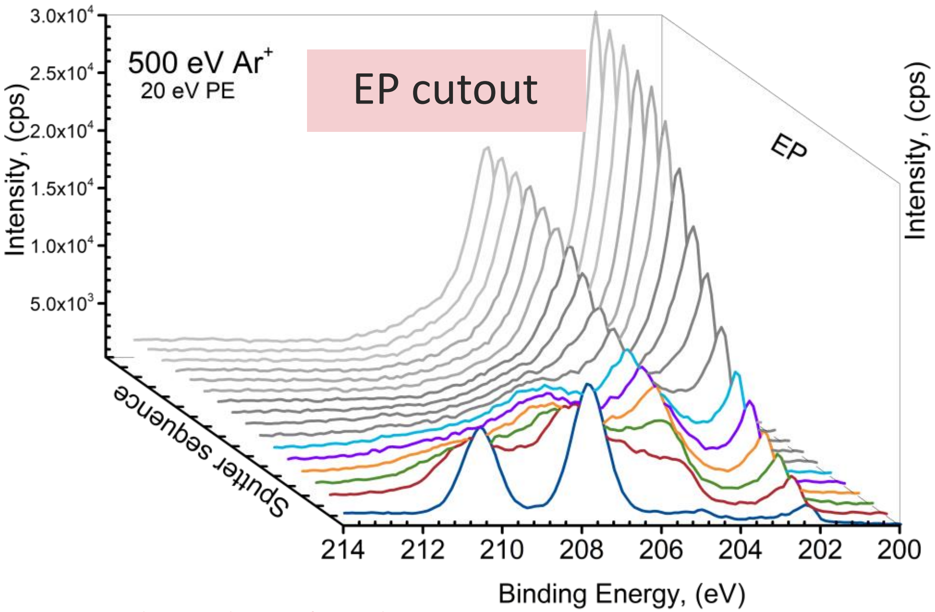
No evidence found of Nb-Carbon species

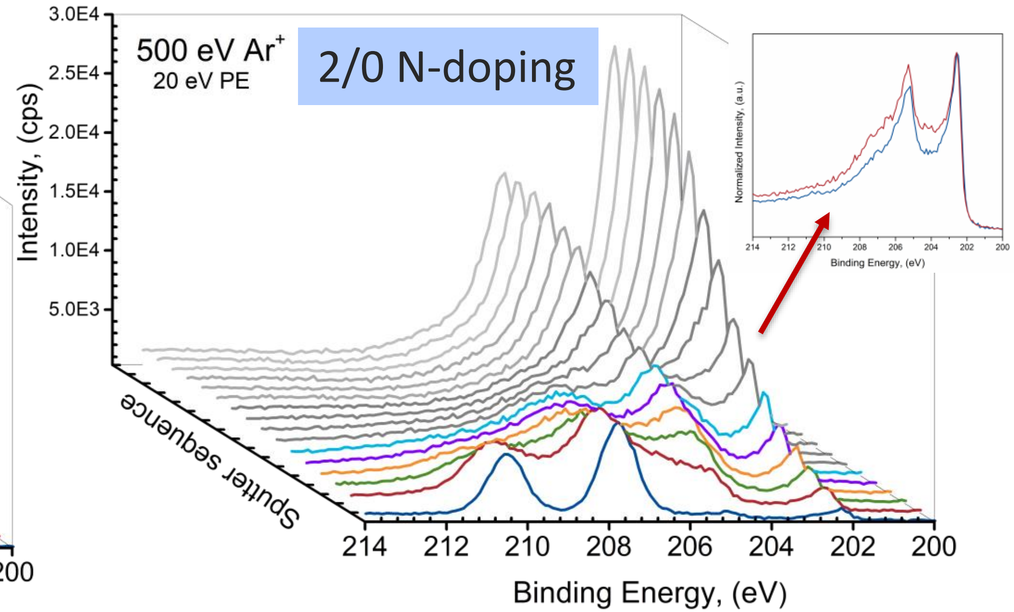
N-dop cutout



The sorption of the carbon species varies with the chemical composition at the surface layers (Raman spectra) and its presence is limited to the top surface layer (XPS).

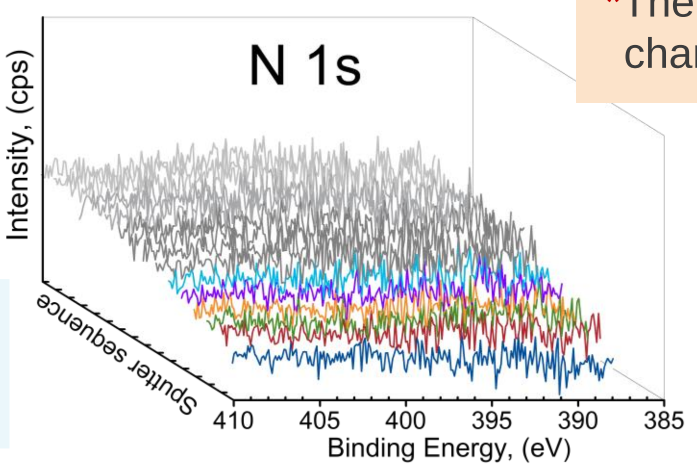
Sputtering profile of Nb 3d





XPS under traditional conditions

Sputtered rate 1.05 nm/s

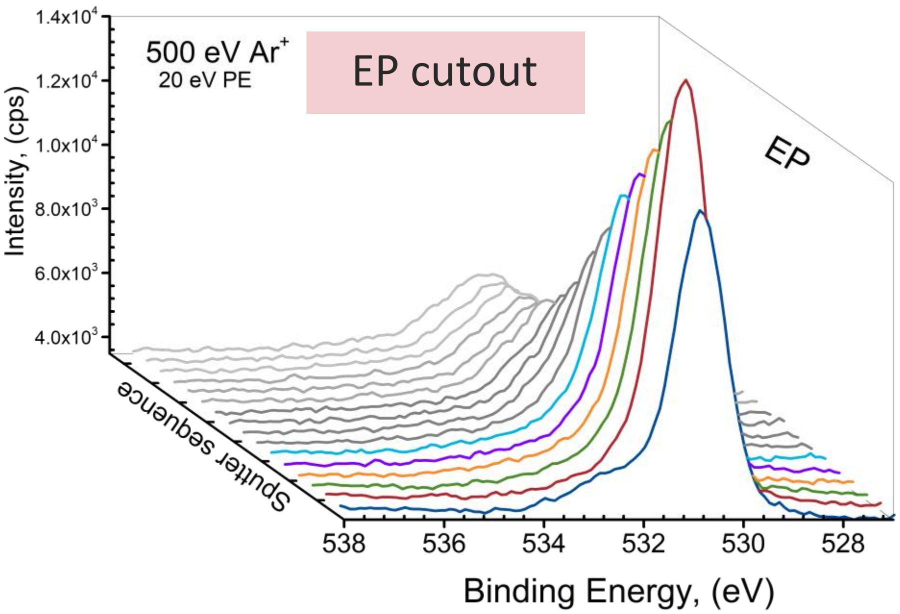


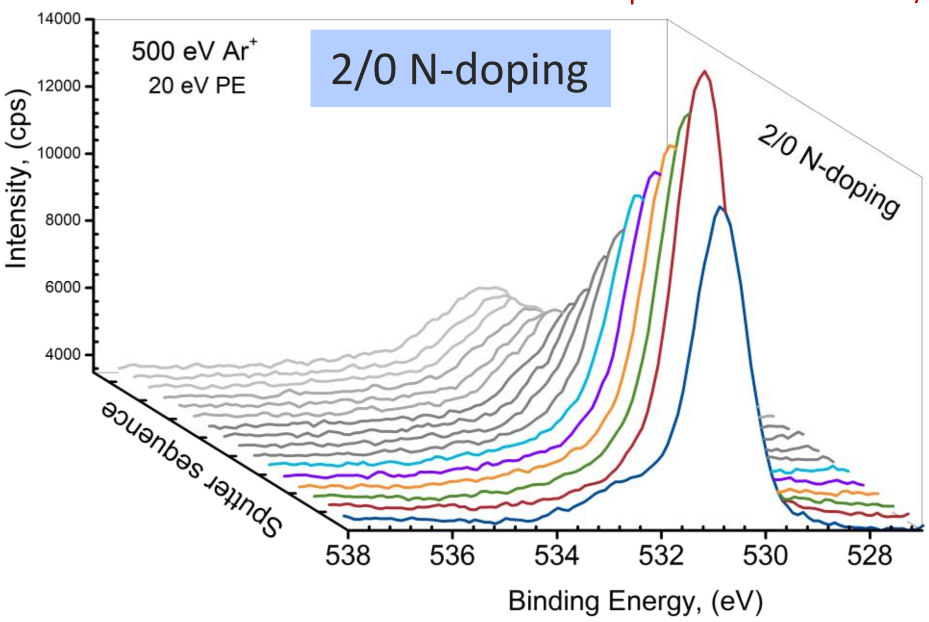
*The presence of N could be sensed by changes on the Nb 3d spectral region

No evidence on formation of N containing precipitates

Sputtering profile of O 1s

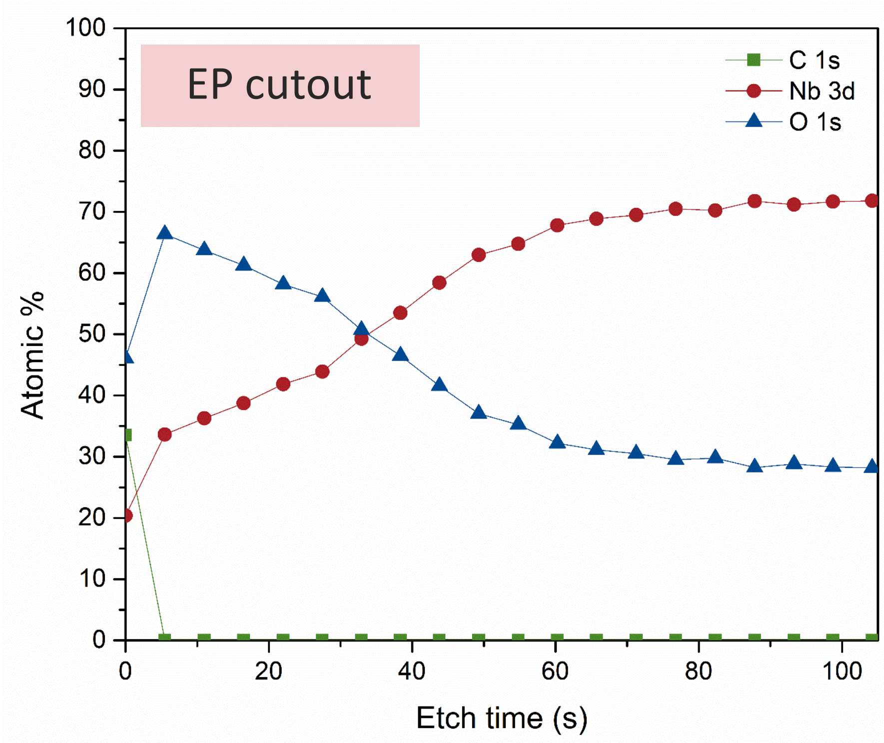
XPS under traditional conditions
Sputtered rate 1.05 nm/s

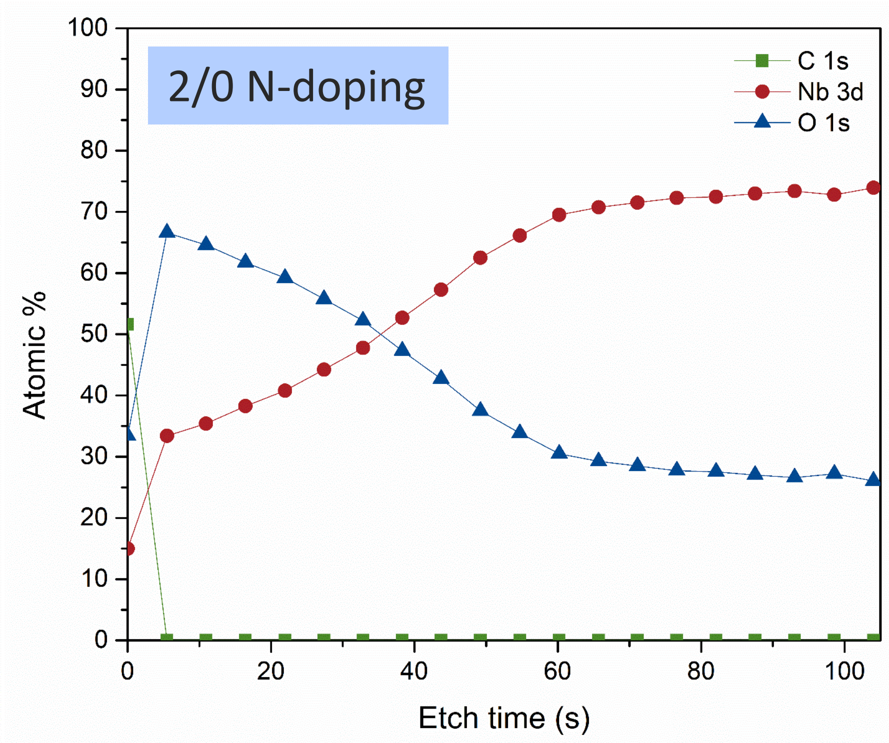




On the removal of surface layers, the concentration of oxygen atoms in the near-surface region decreases, with slight changes in the O 1s spectral profile in the first two sputtering circle. From sputtering circle 3, the O 1s spectral profile for both samples not shown differences.

XPS Depth profile





The presence of N atoms could be sensed by the preferential etching in Nb 3d spectral profile favoring the reduction of Nb in Electropolish compared to N-doping cutout cavity.
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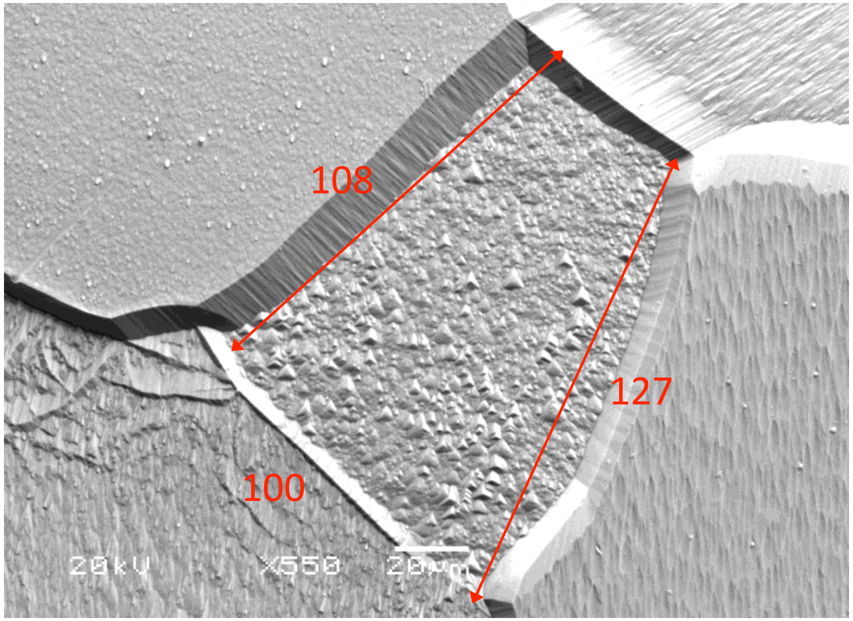
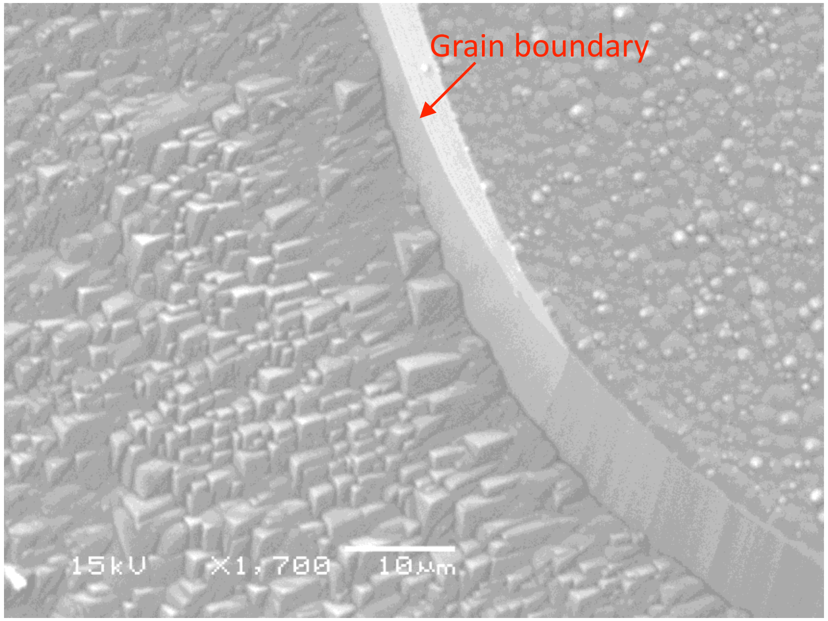
Morphologies observed for some Nb SRF cutous

At this point, it is convenient examine the morphology on Nb cutouts. The solid morphology could have relevant information on the crystal nucleation and growth habits and on their control during the recrystallization process. When these processes are controlled, a more homogeneous and compact structure could be formed, with a low density of structural defects.

A decreased reactivity for Nb atom could result in a material with such features. The presence of N dissolved in the structure could favor such positive effect.

Microscopy inspection of spot causing quench





Electron images recorded from identified spot to cause quench in a 2/0 N-doping 9 cell

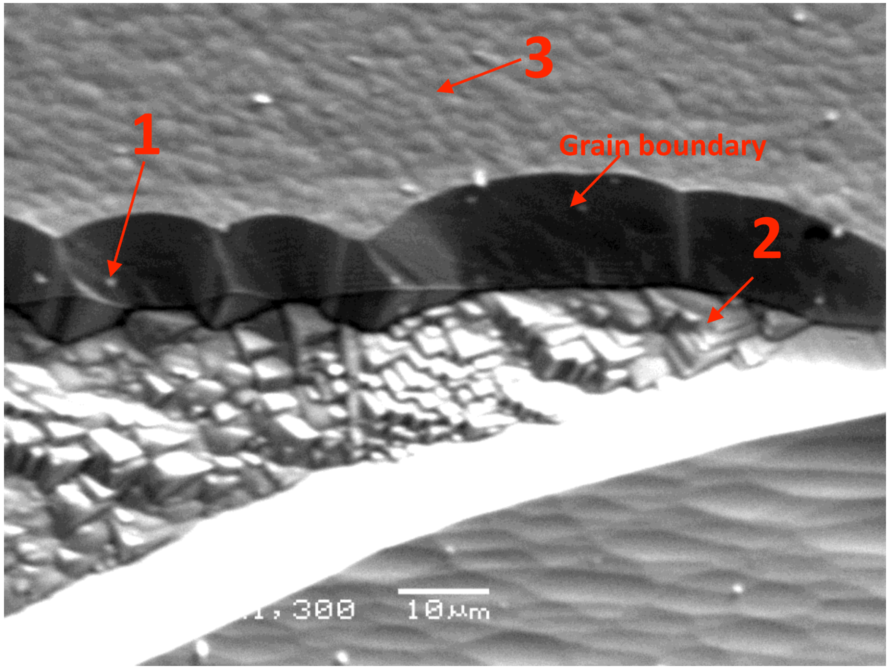
A cutout from cell 1 from the 2/0 N-doping structure shown grains with prominent roughness.

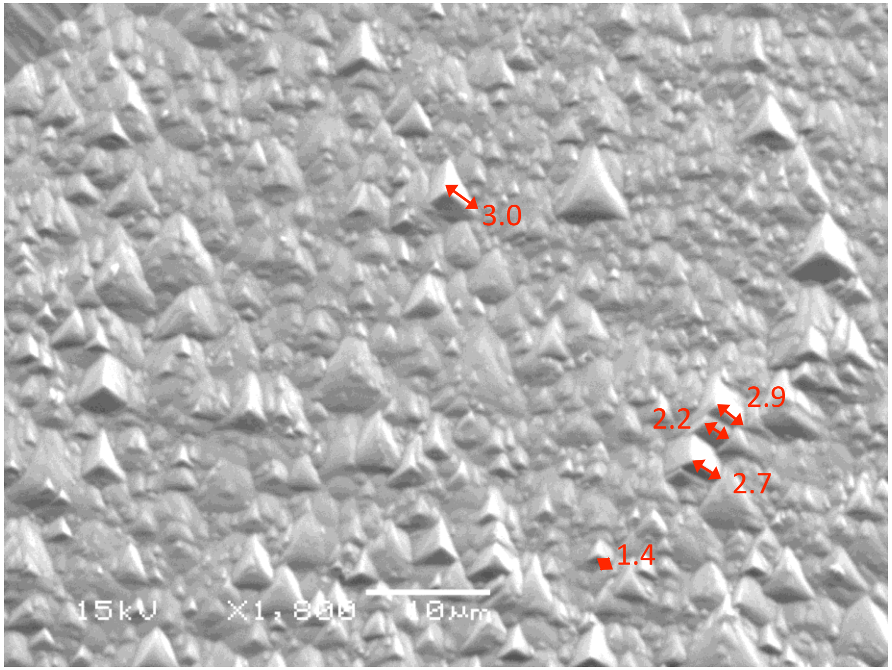
The impurities, in this case N atom, could be conditioning the growth and morphology of the crystals.

XPS do not show presence of N or any other N-containing compounds.

Microscopy inspection of spot causing quench

The three morphologies are compatible with the cubic (bcc) structure of metallic Nb



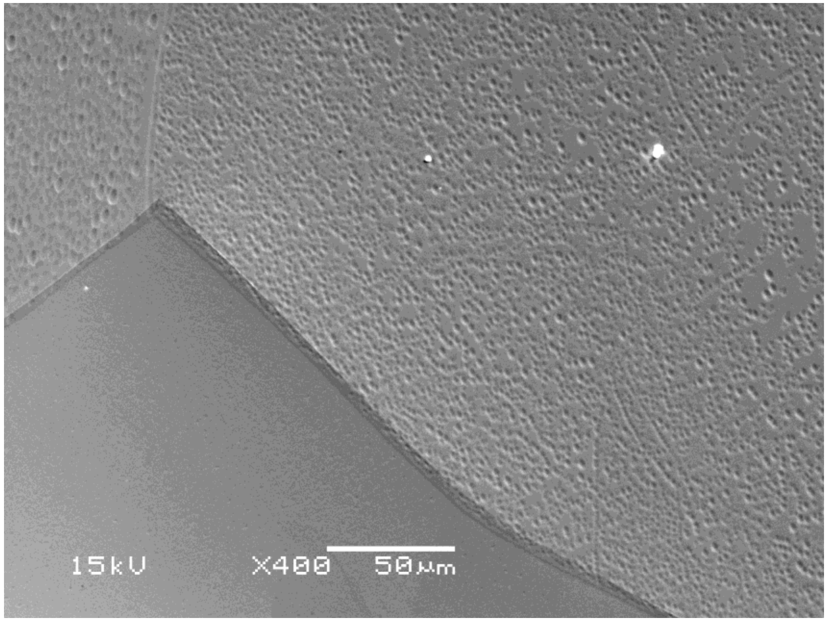


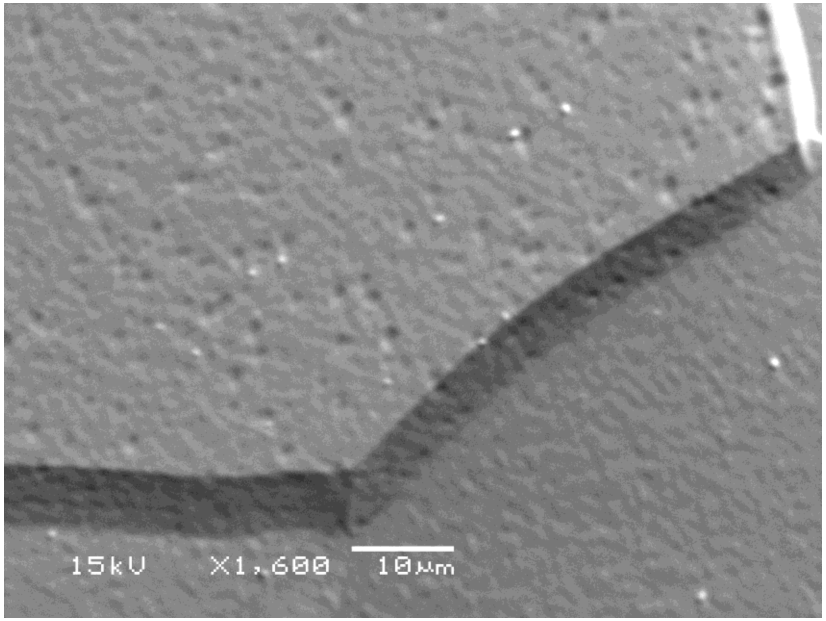
Electron images recorded from (2/0 N-doping) cell 1, which was affected by premature quench

Three types of micro morphologies were observed in specific grains:

1) Cubes with well-defined faces and edges
2) Stacked layer with characteristic relief forming cavities of ellipsoidal shape along their transversal section
3) Crystallites of irregular shape, probably related to a chemical attack of the edges of small cubes during the EP process.

Electron images from 2/0 N-doping 9Cell





The presence of a large amount of small pores at surface of this cutouts from cell1 suggests that the near-surface region was initially occupied by aggregates of particles of a different nature from the matrix.

Such surface porosity, could also be related to a differential attack during the cavity's EP treatment.

In Summary

The high reactivity of Nb can be modulated by its interaction, at atomic level, with atoms capable of subtract electron density from Nb atoms; also with N ?

On the sample cooling, at ~80 K, the precipitation of hydrides were detected from microscopic inspection and from Raman spectra. The hydrides population shows certain dependence on the sample previous treatment, including with N.

From depth profile XPS spectra, N was not detected; but its presence could be revealed by slight changes on Nb 3d spectral region (as chemical shift or preferential etching)

Within a same cavity, a wide variety of morphologies and defects concentration were observed, with regions free of structural defects and a compact structure. The presence of such regions suggests that the crystal nucleation and habits growth occurred under controlled conditions.

New studies are required to support or discard the hypothesis herein considered regarding a mechanism based on the controlled reactivity to explain the positive role of N on the Nb SRF cavity performance.

Collaboration

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Thanks for your attention!!