



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

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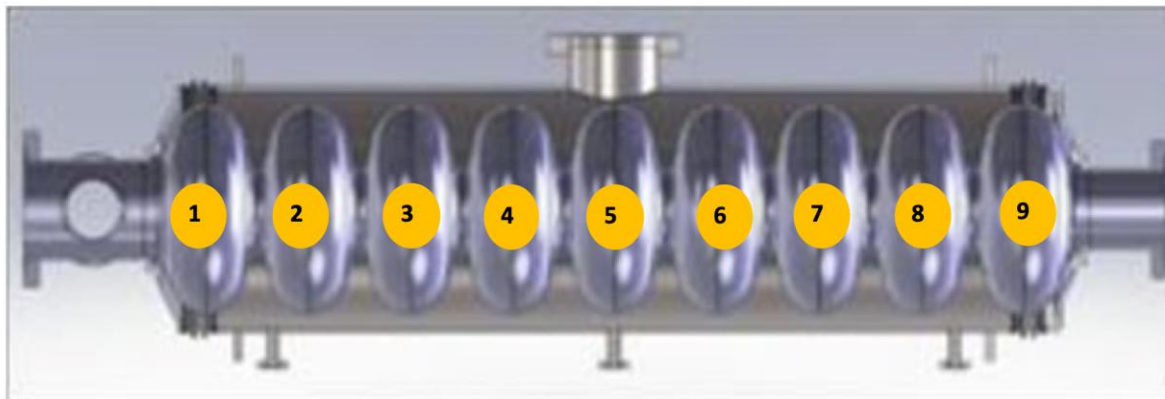
TTC meeting

04 Feb 2020

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

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

IUPAC Periodic Table of the Elements

| | | | | | | | | | | | | | | | | | | | |
|--|--------------------------------|---|----------------------------------|--------------------------------|---------------------------------|--|----------------------------------|-------------------------------|---|------------------------------|-------------------------------|--|----------------------------------|---|---|---|--|---|--|
| 1 H hydrogen 1.008 | | | | | | | | | | | | | | | | | 2 He helium 4.0026 | | |
| 3 Li lithium 6.94 (6.938, 6.977) | | 4 Be beryllium 9.0122 | | | | | | | | | | | | 13 B boron 10.81 (10.806, 10.821) | 14 C carbon 12.011 | 15 N nitrogen 14.007 | 16 O oxygen 15.999 | 17 F fluorine 18.998 | 18 Ne neon 20.180 |
| 11 Na sodium 22.990 | | 12 Mg magnesium 24.305 (24.304, 24.307) | | | | | | | | | | | | 13 Al aluminum 26.982 | 14 Si silicon 28.086 (28.084, 28.094) | 15 P phosphorus 30.974 (30.972, 30.976) | 16 S sulfur 32.06 (32.059, 32.074) | 17 Cl chlorine 35.45 (35.446, 35.457) | 18 Ar argon 39.95 (39.942, 39.963) |
| 19 K potassium 39.098 (40.0784) | 20 Ca calcium 40.0784 | 21 Sc scandium 44.956 | 22 Ti titanium 47.867 | 23 V vanadium 50.942 | 24 Cr chromium 51.996 | 25 Mn manganese 54.938 (54.9452) | 26 Fe iron 55.845 | 27 Co cobalt 58.933 | 28 Ni nickel 58.693 (63.5463) | 29 Cu copper 63.546 | 30 Zn zinc 65.38 | 31 Ga gallium 69.723 | 32 Ge germanium 72.6308 | 33 As arsenic 74.922 | 34 Se selenium 78.9718 | 35 Br bromine 79.904 | 36 Kr krypton 83.7982 | | |
| 37 Rb rubidium 85.468 | 38 Sr strontium 87.62 | 39 Y yttrium 88.906 | 40 Zr zirconium 91.2242 | 41 Nb niobium 92.906 | 42 Mo molybdenum 95.96 | 43 Tc technetium 98.9062 | 44 Ru ruthenium 101.072 | 45 Rh rhodium 102.91 | 46 Pd palladium 106.42 | 47 Ag silver 107.87 | 48 Cd cadmium 112.41 | 49 In indium 114.82 | 50 Sn tin 118.71 | 51 Sb antimony 121.76 | 52 Te tellurium 127.603 | 53 I iodine 126.90 | 54 Xe xenon 131.29 | | |
| 55 Cs caesium 132.91 | 56 Ba barium 137.33 | 57-71 lanthanoids | 72 Hf hafnium 178.49 | 73 Ta tantalum 180.95 | 74 W tungsten 183.84 | 75 Re rhenium 186.21 | 76 Os osmium 190.23 | 77 Ir iridium 192.22 | 78 Pt platinum 195.08 | 79 Au gold 196.97 | 80 Hg mercury 200.59 | 81 Tl thallium 204.38 (204.38, 204.38) | 82 Pb lead 207.2 | 83 Bi bismuth 208.98 | 84 Po polonium | 85 At astatine | 86 Rn radon | | |
| 87 Fr francium | 88 Ra radium | 89-103 actinoids | 104 Rf rutherfordium | 105 Db dubnium | 106 Sg seaborgium | 107 Bh bohrium | 108 Hs hassium | 109 Mt meitnerium | 110 Ds darmstadtium | 111 Rg roentgenium | 112 Cn copernicium | 113 Nh nihonium | 114 Fl flerovium | 115 Mc moscovium | 116 Lv livermorium | 117 Ts tennessine | 118 Og oganesson | | |



| | | | | | | | | | | | | | | |
|---------------------------------|-------------------------------|------------------------------------|---------------------------------|----------------------------------|--------------------------------|--------------------------------|----------------------------------|-------------------------------|----------------------------------|-------------------------------|------------------------------|-------------------------------|---------------------------------|--------------------------------|
| 57 La lanthanum 138.91 | 58 Ce cerium 140.12 | 59 Pr praseodymium 140.91 | 60 Nd neodymium 144.24 | 61 Pm promethium 144.91 | 62 Sm samarium 150.36 | 63 Eu europium 151.96 | 64 Gd gadolinium 157.25 | 65 Tb terbium 158.93 | 66 Dy dysprosium 162.50 | 67 Ho holmium 164.93 | 68 Er erbium 167.26 | 69 Tm thulium 168.93 | 70 Yb ytterbium 173.05 | 71 Lu lutetium 174.97 |
| 89 Ac actinium | 90 Th thorium 232.04 | 91 Pa protactinium 231.04 | 92 U uranium 238.03 | 93 Np neptunium | 94 Pu plutonium | 95 Am americium | 96 Cm curium | 97 Bk berkelium | 98 Cf californium | 99 Es einsteinium | 100 Fm fermium | 101 Md mendelevium | 102 No nobelium | 103 Lr lawrencium |

For notes and updates to this table, see www.iupac.org. This version is dated 1 December 2018.
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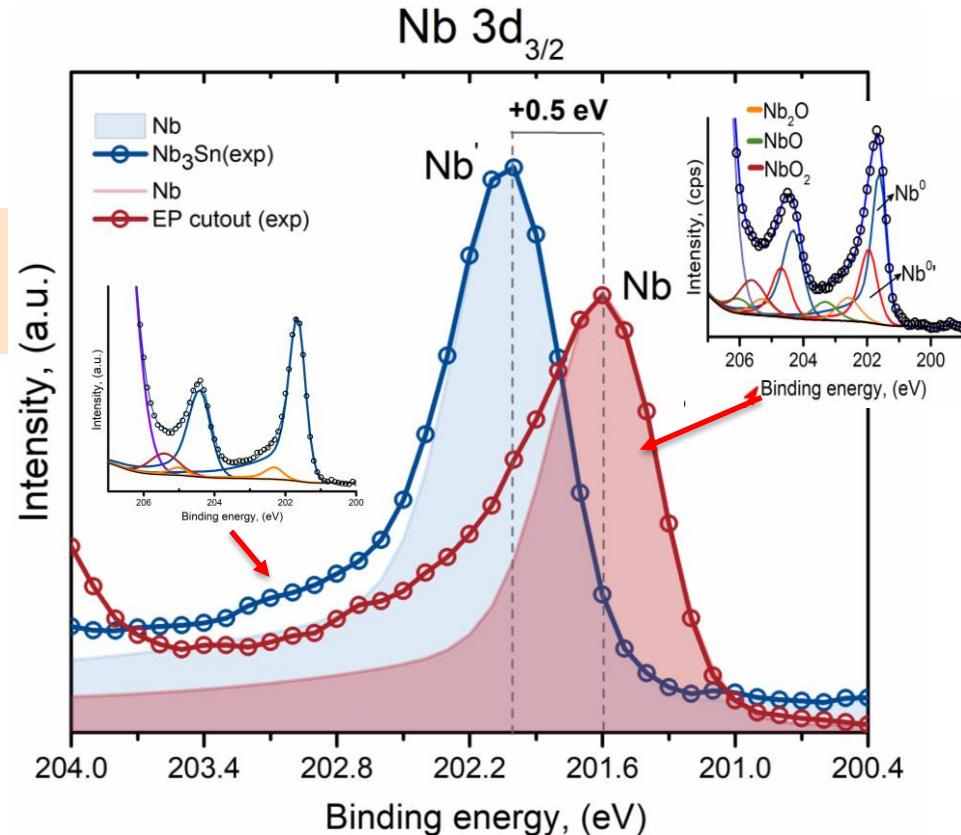
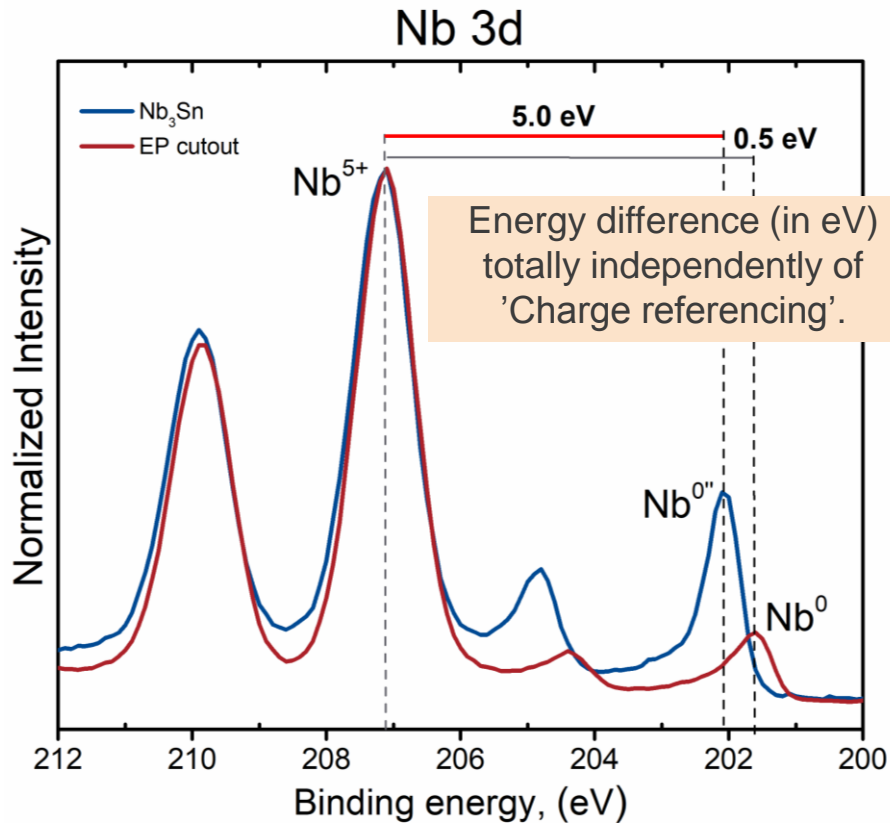
Nb shows a high reactivity for the sorption and reaction with **Hydrogen**, **Nitrogen** and **Oxygen**. These three elements can be found dissolved in the near surface region and, in the bulk and forming precipitates under certain conditions.

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



On the Nb surface reactivity: Nb in Nb₃Sn as reference

Chemical shift found at the Nb 3d spectral low-BE region for Nb in Nb₃Sn



In Nb₃Sn, 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

Motivation

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

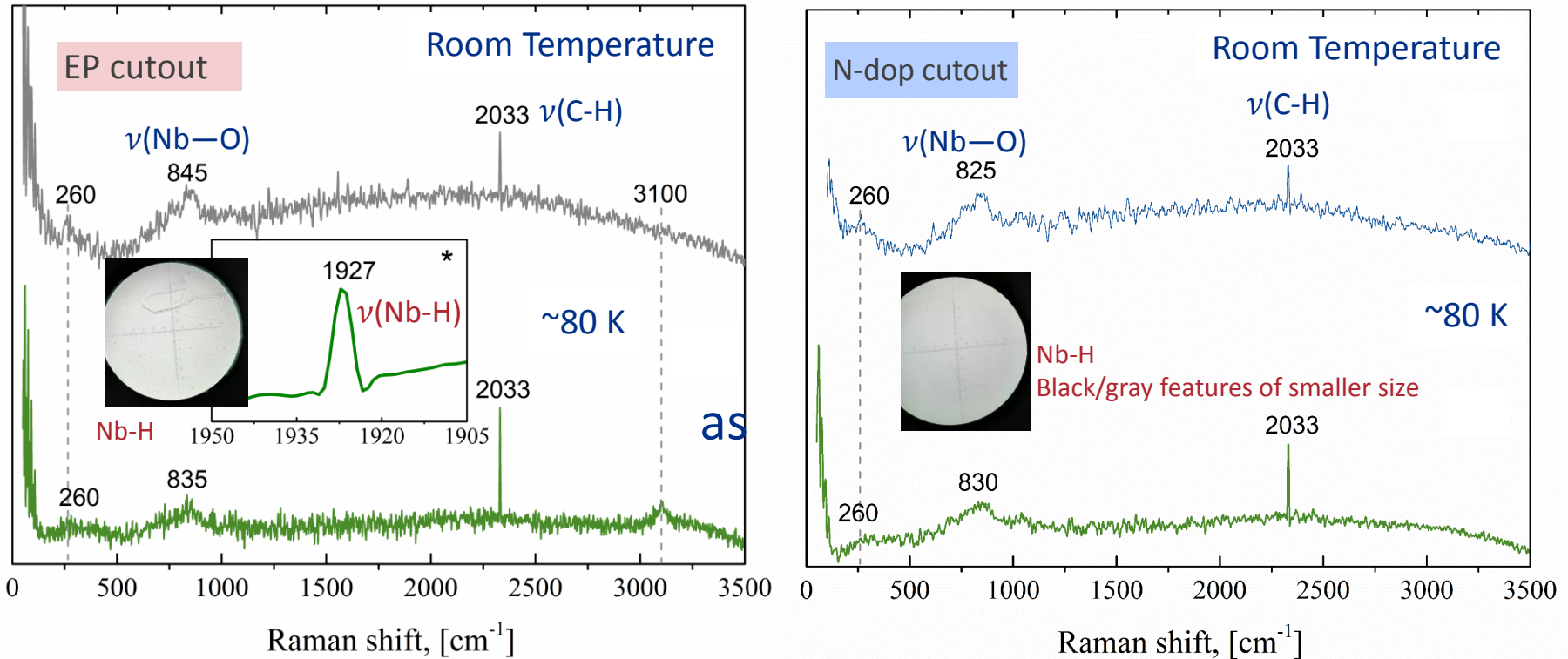
We try to understand the nature of such effect, from an atomic point of view, considering a possible interaction between Nb and N atoms, and how that interaction could modulate the physical and functional properties of Nb metal in the SRF cavity.

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



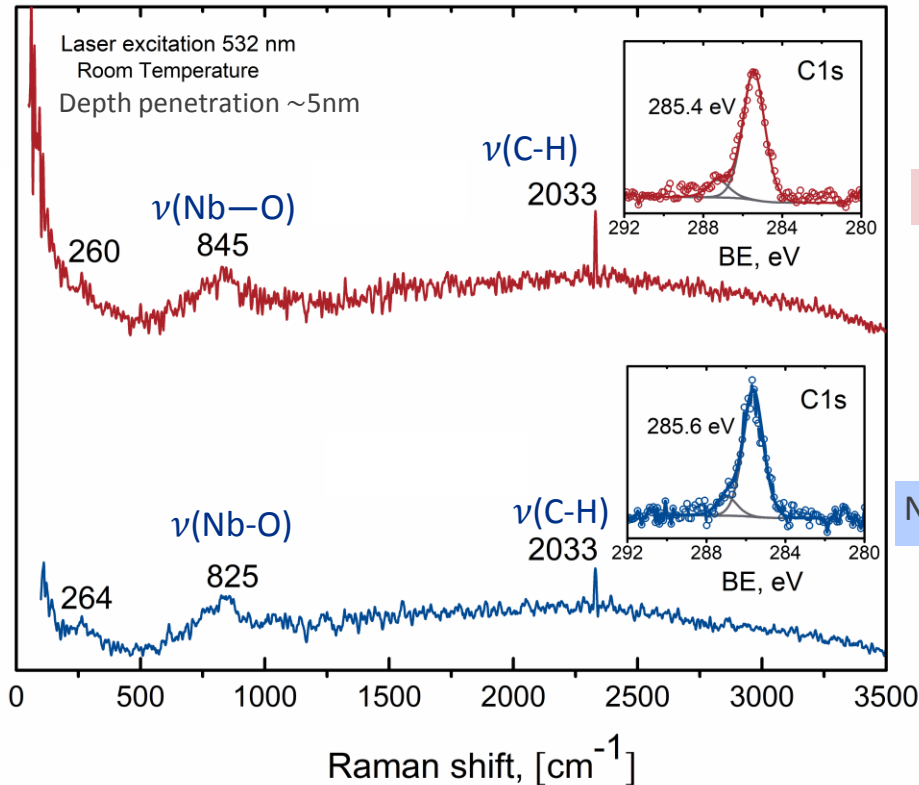
Raman spectra recorded under RT and cryogenic conditions ($\sim 77\text{K}$)

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

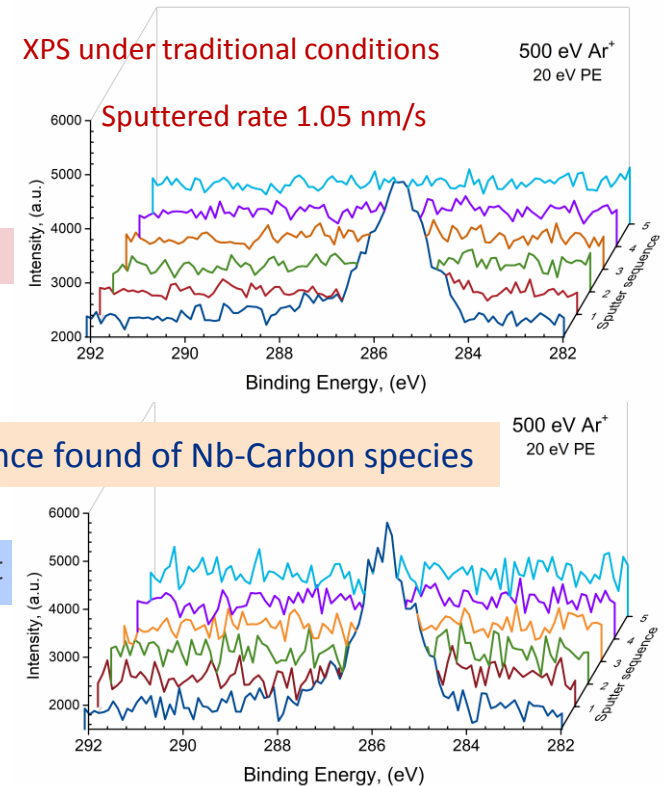
Nb, at its surface, is able to absorb species from the environment, e.g. carbon containing compounds, etc.



EP cutout

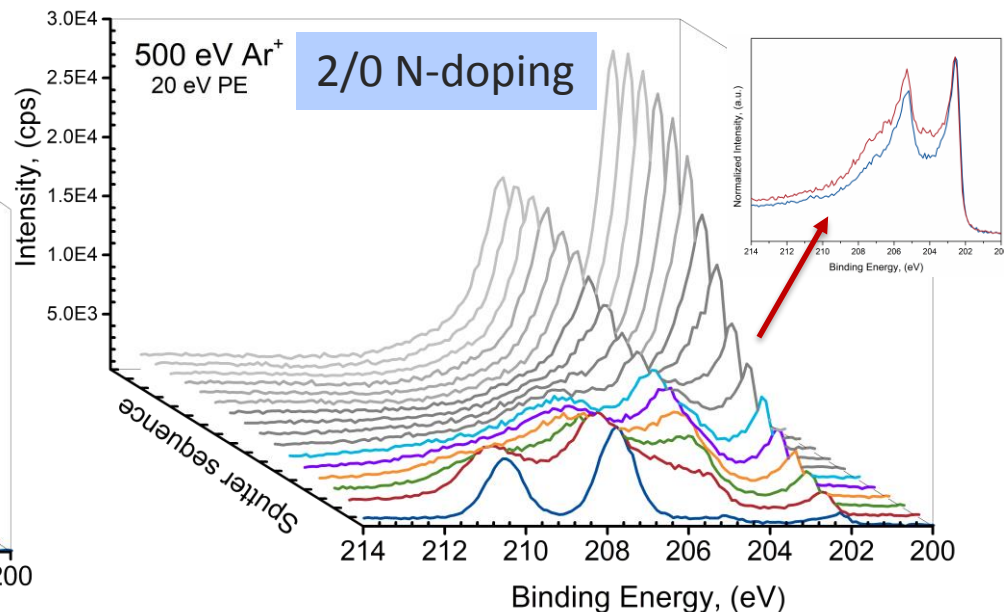
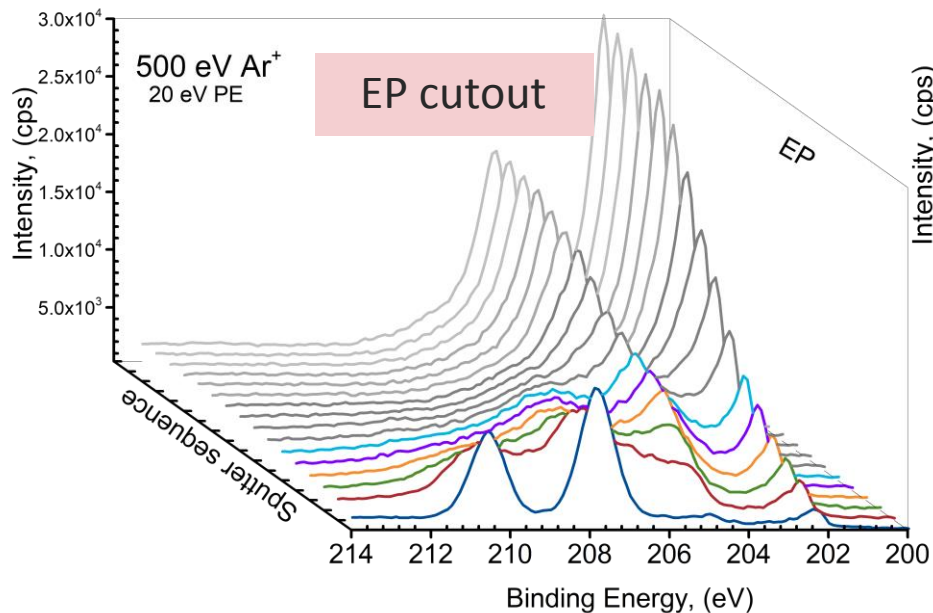
No evidence found of Nb-Carbon species

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).

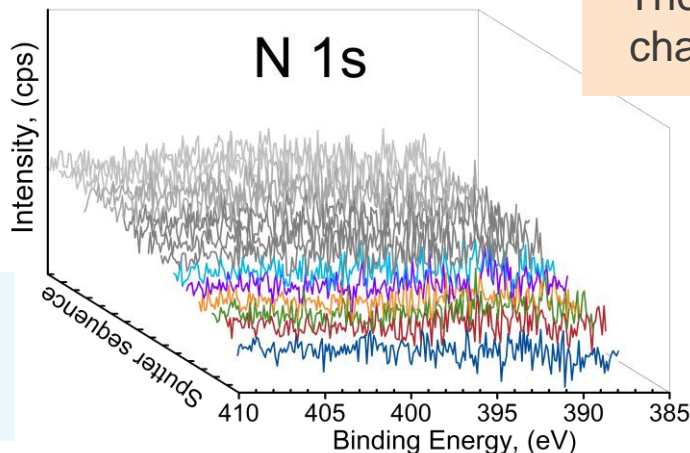
Sputtering profile of Nb 3d



XPS under traditional conditions

Sputtered rate 1.05 nm/s

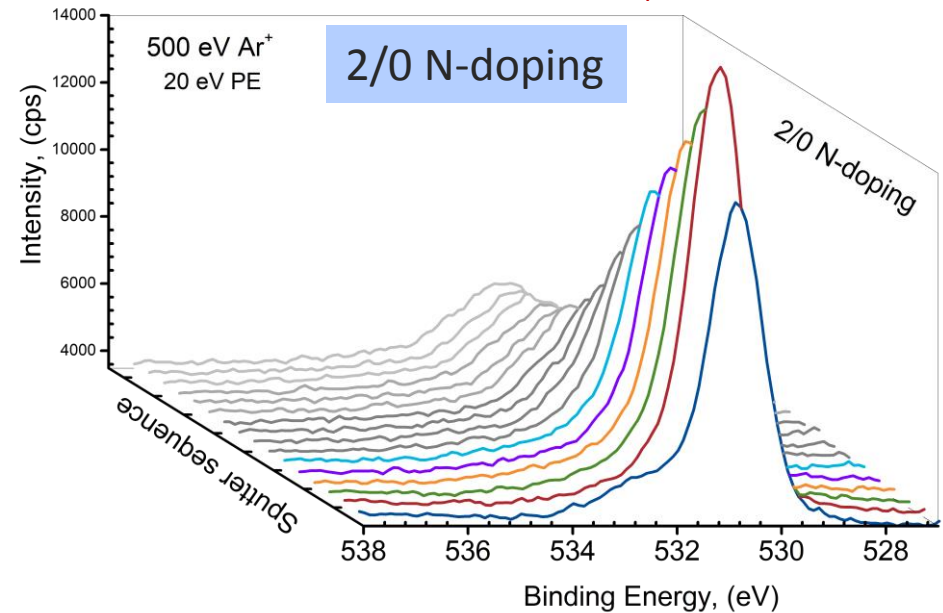
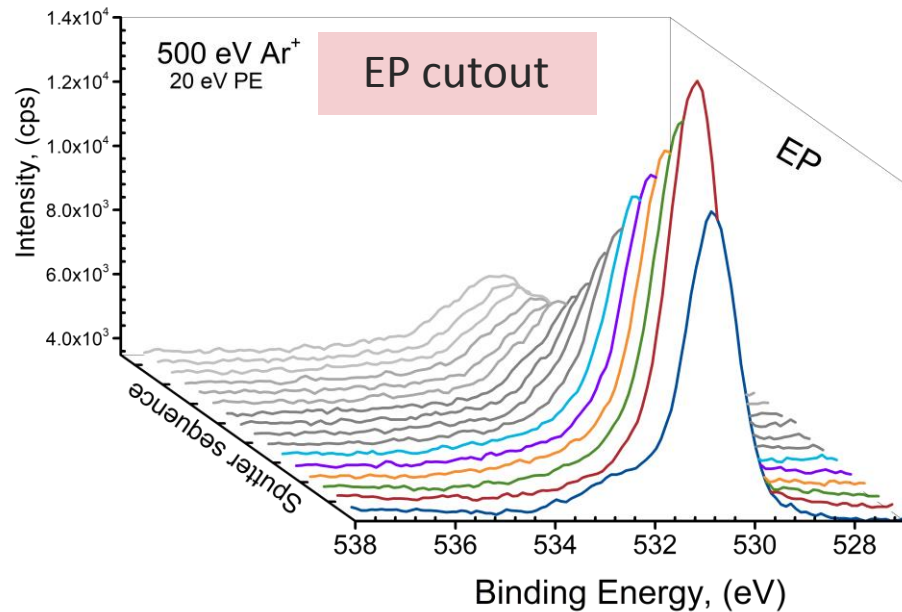
No evidence on formation of N containing precipitates



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

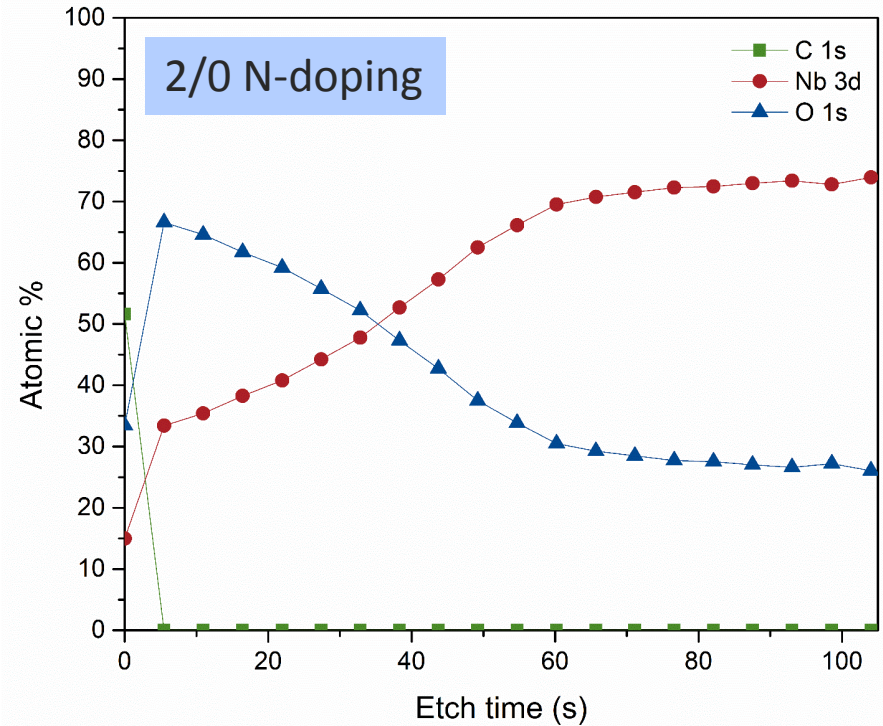
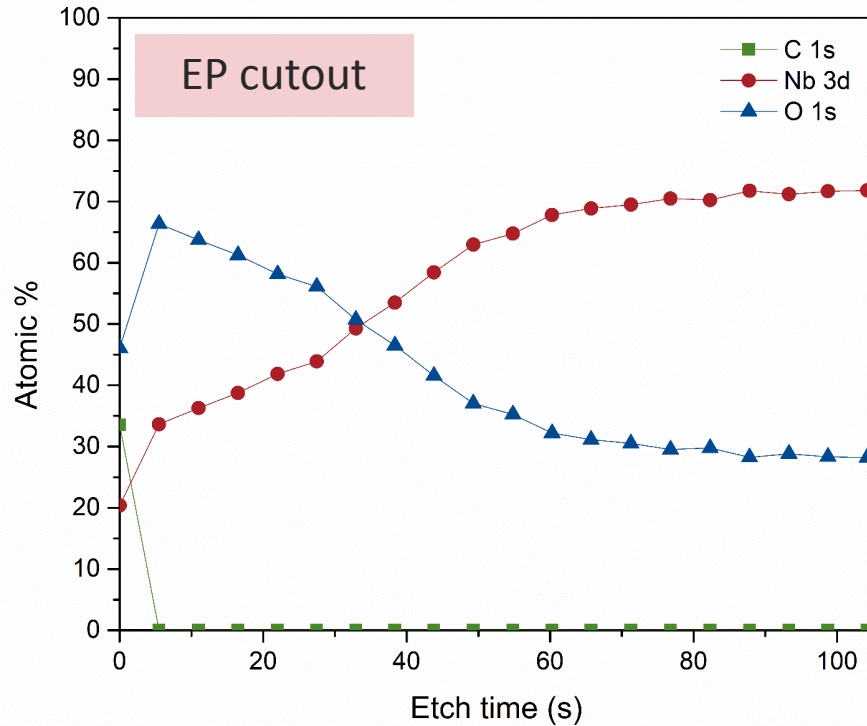
Sputtering profile of O 1s

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.

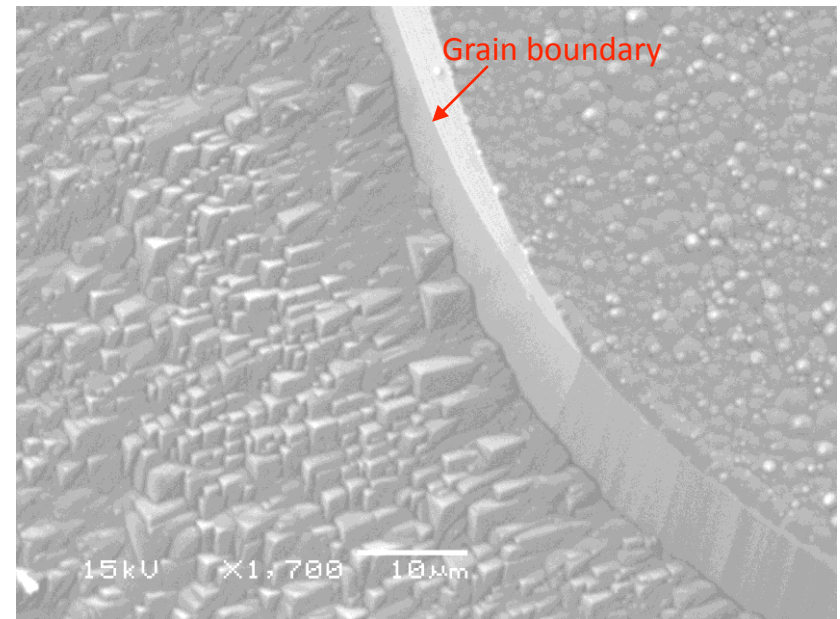
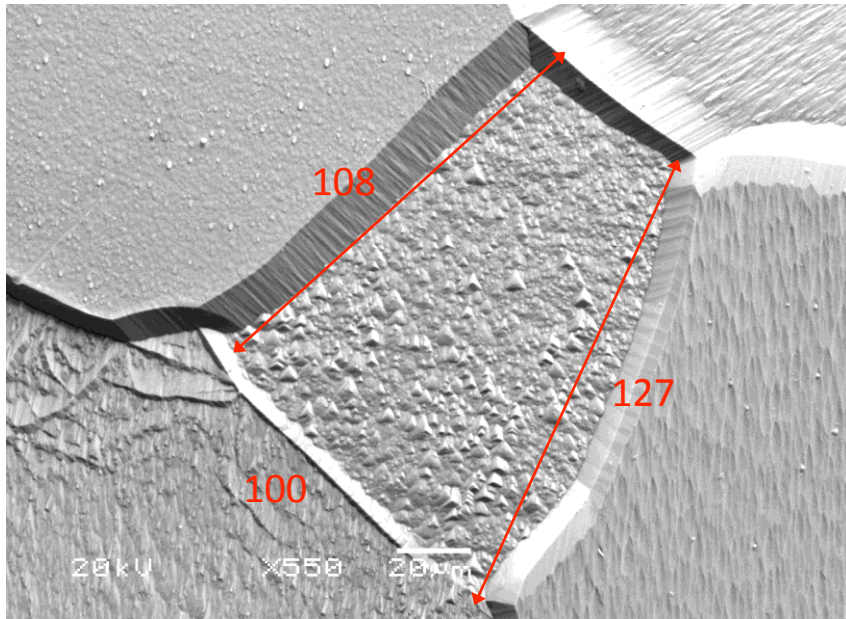
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Morphologies observed for some Nb SRF cutouts

At this point, it is convenient to 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 atoms could result in a material with such features. The presence of N dissolved in the structure could favor such a 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

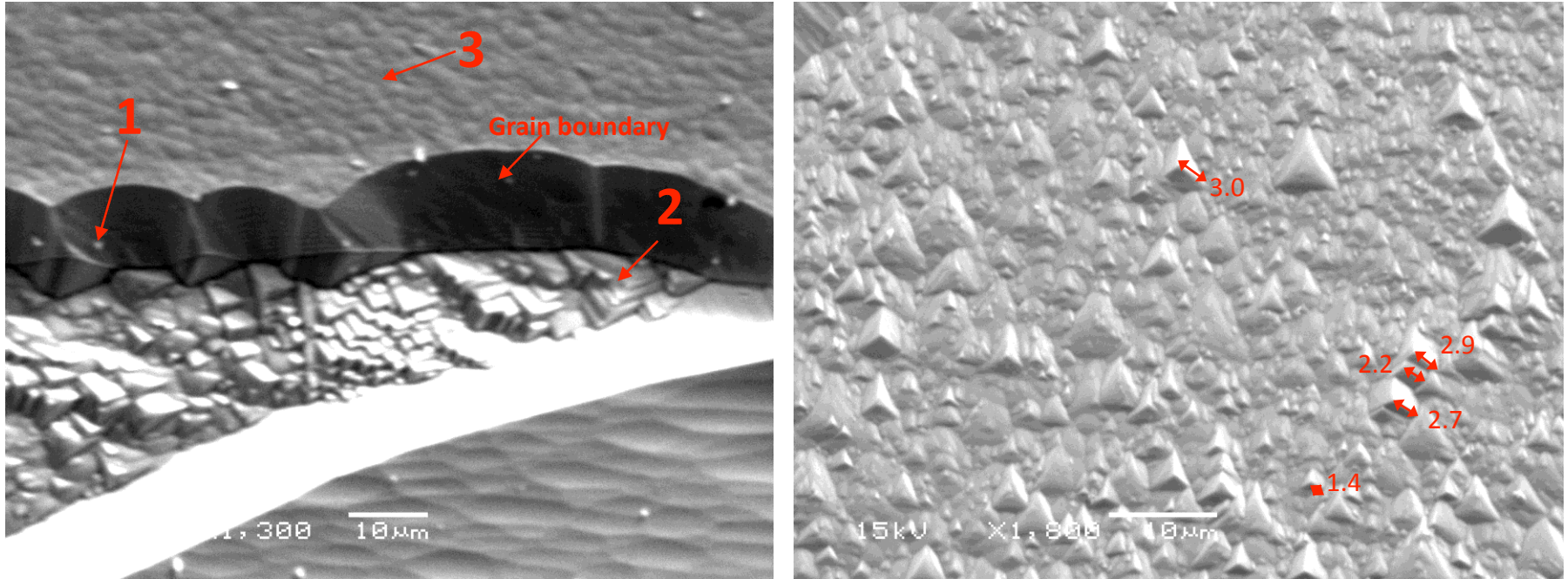
A cutout from cell 1 from the 2/0 N-doping structure shown grains with prominent roughness.

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

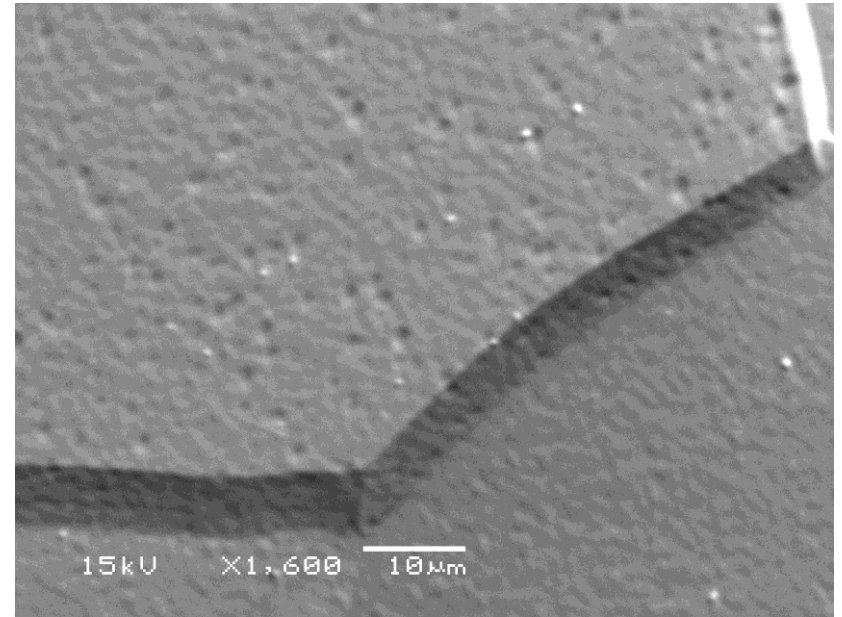
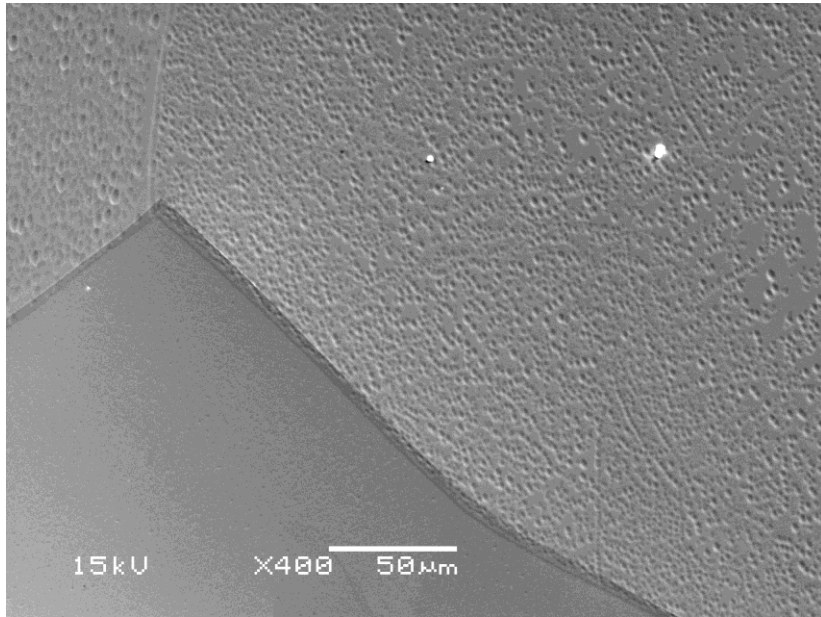


Electron images recorded from (2/0 N-doping) cell 1, which was affected by premature quench

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



NORTHWESTERN
UNIVERSITY

Thanks for your attention!!