# Study of the Niobium Oxide Structure and Microscopic Effect of Plasma Processing on the Nb Surface

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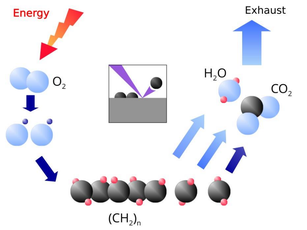
## 1. Abstract

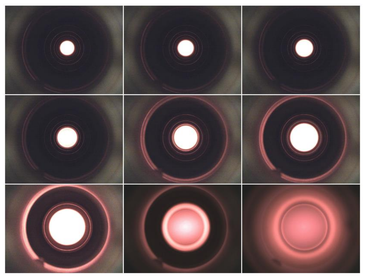
A study of the niobium oxide structure is presented here, focusing on the niobium suboxides. Multiple steps of argon sputtering and XPS measurements were carried out until the metal surface was exposed. Subsequently, the sample was exposed to air for different time intervals and the oxide regrowth was studied. In addition, three Nb samples prepared with different surface treatments were studied before and after being subjected to plasma processing. The scope is investigating the microscopic effect that the reactive oxygen contained in the glow discharge may have on the niobium surface. This study suggests that the $Nb_2O_5$ thickness may increase. Nevertheless, since the $Nb_2O_5$ is dielectric, its thickening would not negatively affect the cavity performance.

## 2. Plasma processing to mitigate FE

Reducing FE through $C_xH_y$ removal from cavity Nb surface

Increasing the niobium work function by 10% results in 15% increase in $E_{acc}$ $O_2 + C_xH_y \rightarrow CO + CO_2 + H_2O$

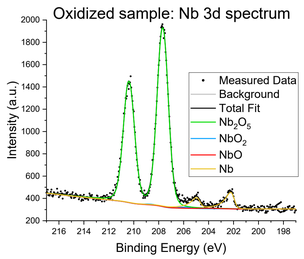


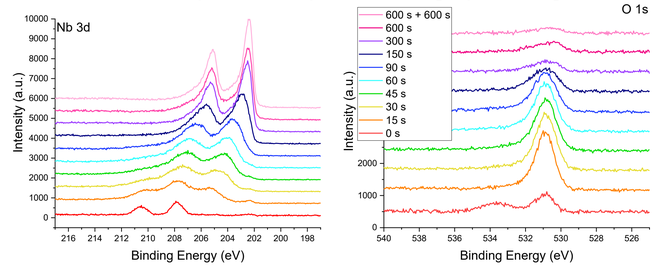
- M. Doleans *et al,* NIMA 812 (2016)
- P. Berrutti, B. Giaccone *et al.*, J. Appl. Phys. 126, 023302 (2019)
- B. Giaccone *et al.*, Phys. Rev. Accel. Beams 24, 022002

## 3. Argon ions sputtering on oxidized Nb sample

- Sample preparation: 800 ˚C x 3h + 20 µm EP + 5 min HF rinse
- After surface treatment: 1 week to fully oxidize prior to XPS measurement

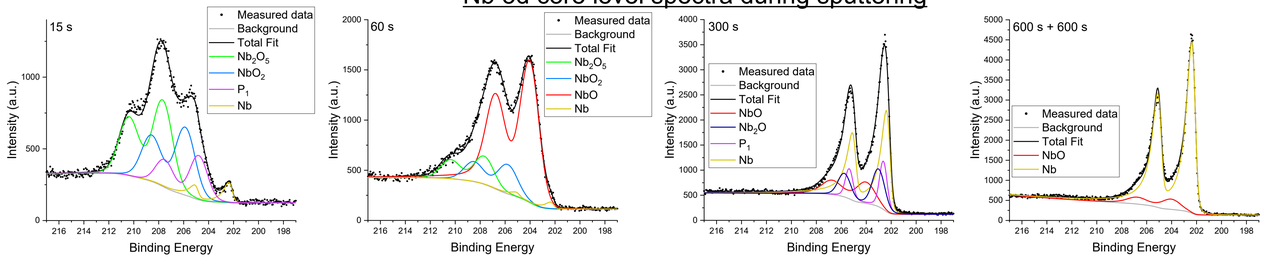



Spectra acquired during sputtering




- Nb 3d: shift towards lower energy values as the oxidation state decreases
- O 1s: initially three peaks are detected: niobium oxide, C-O, C=O. The niobium oxide peak is due to $Nb_2O_5$ plus the suboxides

Nb 3d core level spectra during sputtering



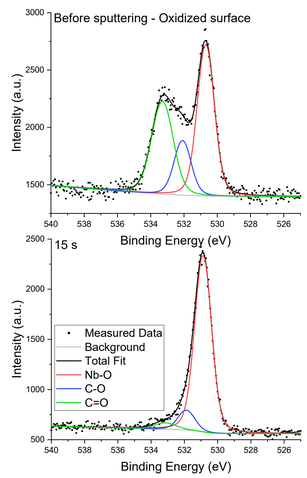

- Nb peak: 202.2 eV
- $P_1$: $NbO_{1+x}$ peak: 204.8 eV
- $NbO_2$ peak: 205.9 eV
- $Nb_2O_5$ peak: 207.7 eV

- Nb peak: 202.2 eV
- NbO peak: 203.8 eV
- $NbO_2$ peak: 205.8 eV
- $Nb_2O_5$ peak: 207.8 eV

- Nb peak: 202.2 eV
- $P_1$: $Nb_{2+x}O$: 202.6 eV
- $Nb_2O$ peak: 203 eV
- NbO peak: 203.8 eV

- Nb peak: 202.2 eV
- NbO peak: 203.8 eV

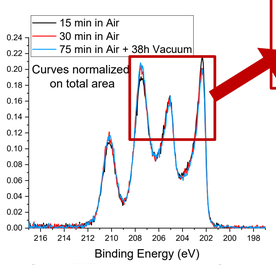
O1s core level spectra

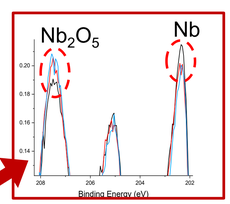


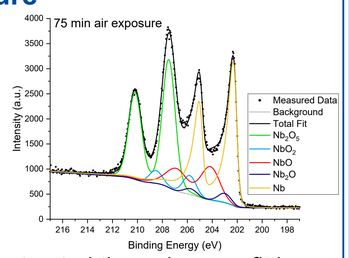

- $P_1$ here identifies a nonstoichiometric oxide peak whose energy decreases at each sputtering step as its chemical state is modified by the Ar ions.
- After 600 s of sputtering: sample kept under vacuum for 12 h, followed by additional 600 s of sputtering and final measurement prior to oxide regrowth in air.

## 4. Oxide growth through air exposure

Air exposure steps:
- 15 min
- Additional 15 min
- Additional 45 min




Ratio of the areas extracted through curve fitting

| Air exposure time | $Nb_2O_5$/Nb | $NbO_2$/Nb | NbO/Nb | $Nb_2O$/Nb |
|---|---|---|---|---|
| 15 min | 0.87 | 0.12 | 0.36 | 0.08 |
| 30 min | 0.99 | 0.14 | 0.41 | 0.11 |
| 75 min | 1.00 | 0.15 | 0.40 | 0.07 |

After 75 minutes of exposure to air: surface has not reached full oxidization.

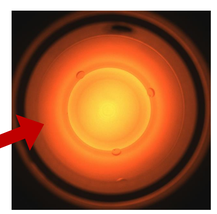
Multiple suboxides were identified through curve fitting: NbO, $NbO_2$, $Nb_2O$

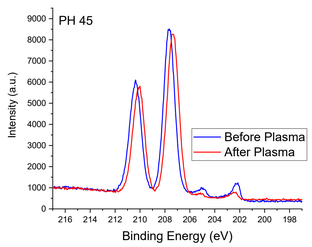
The initial measurements on the oxidized sample showed possible traces of NbO and $NbO_2$, but no $Nb_2O$ was detected.

## 5. XPS analysis on plasma processed Nb samples

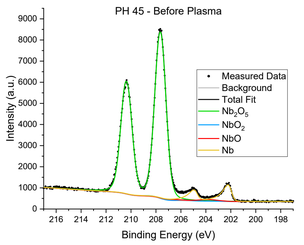
Sample preparation:
- PH 45: 800 ˚C x 3h + 20 µm EP
- PH 35: 2/6 N doping + 5 µm EP + 5 min HF rinse
- PH 40: 2/0 N doping + 7 µm cold EP

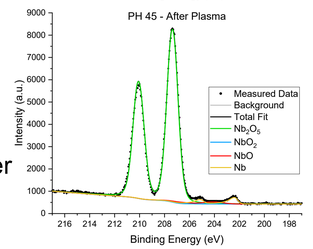
Samples were positioned on cavity iris and plasma processed for 6h.






- Relative intensity of Nb and $Nb_2O_5$ is modified: ratio of $Nb_2O_5$ area over Nb area consistently increases after plasma processing
- ΔE between Nb and $Nb_2O_5$ may be decreased after plasma processing. Further analyses are needed to understand if it is caused by plasma cleaning.




- Similar results obtained on all three samples, independently of the surface treatment
- No particular change was observed in the suboxide spectra

## 6. Conclusions

- Sputtering and oxide regrowth: studied niobium oxide structure, identified multiple suboxides (NbO, $NbO_2$, $Nb_2O$) and extracted parameters for data analysis
- Plasma processed samples:
  - Consistent change in $Nb_2O_5$/Nb area ratio suggesting **oxide thickening**
  - **Possible reduction in ΔE between $Nb_2O_5$ and Nb peaks.** If caused by plasma processing, it may be due to the introduction of oxygen vacancies in $Nb_2O_5$ lattice.
  - However, $Nb_2O_5$ is dielectric: thickness increase would not affect cavity performance

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