In-situ X-ray studies of heat treatments/nitrogen treatments of Nb

Niobium surface investigations for RF cavity applications.

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International Workshop on Future Linear Colliders
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HELMHOLTZ RESEARCH FOR GRAND CHALLENGES

DESY.

The project

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"Performance improvement of SRF cavities by surface treatment including mechanical polishing and nitrogen thermal treatment"

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HELMHOLTZ
RESEARCH FOR GRAND CHALLENGES

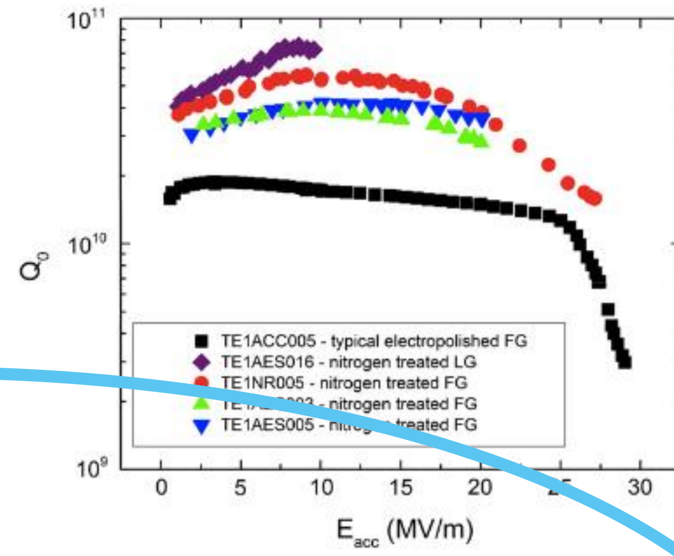
Bundesministerium
für Bildung
und Forschung

Motivation

Surface preparations of SRF cavities involving N_2

Nitrogen doping:

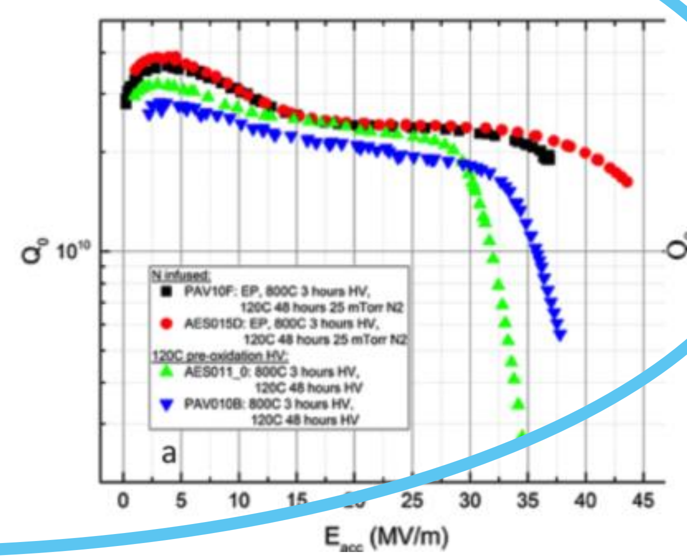
- 800°C - 1000°C in high vacuum (2-3 hours)
- Addition of ~2.5×10^{-2} mbar N_2 (min - hours)
- Material removal: <u>5-80 µm</u>
- High Q_0 - E_{acc} ~ 25 MV/m



Nitrogen infusion:

- 800°C in high vacuum (2-3 hours)
- Cooldown to 120°C
- Addition of ~3×10^{-2} mbar N_2 for 48 hours
- No material removal

Slightly higher Q_0 - E_{acc} > 25 MV/m



A Grassellino et al 2013 Supercond. Sci. Technol.26 102001, A Grassellino et al 2017 Supercond. Sci. Technol.30 094004

Motivation

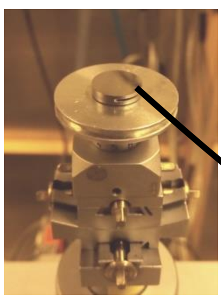
Key questions:

- What is the surface state before the treatments?
- How is the surface affected by annealing in high-vacuum?
- Do we promote the formation of different phases (nitrides, oxides) at the surface?
- What happens to possible interstitial atoms (nitrogen, oxygen) during such treatments?
- What happens when the surface is re-exposed to air?

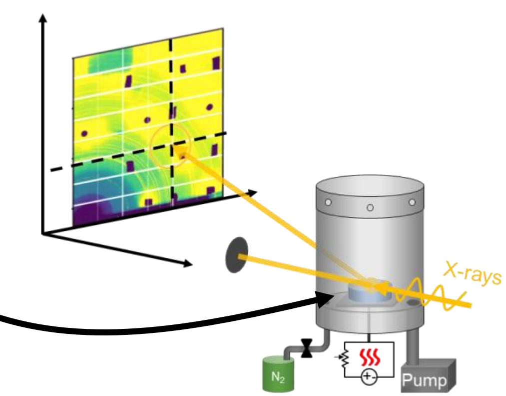
Our approach:

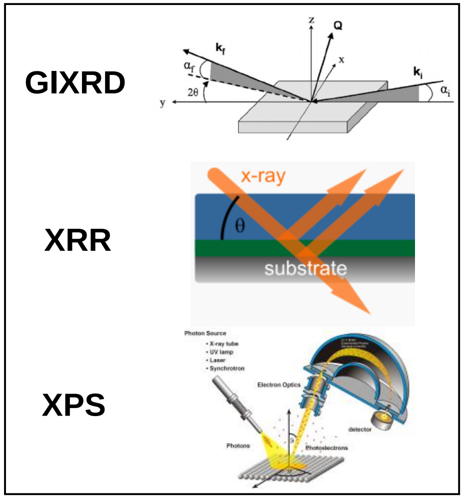
- X-ray based methods to study the near-surface region of Nb before, during and after thermal treatments applied to real Nb SRF cavities

Nb samples



Model system

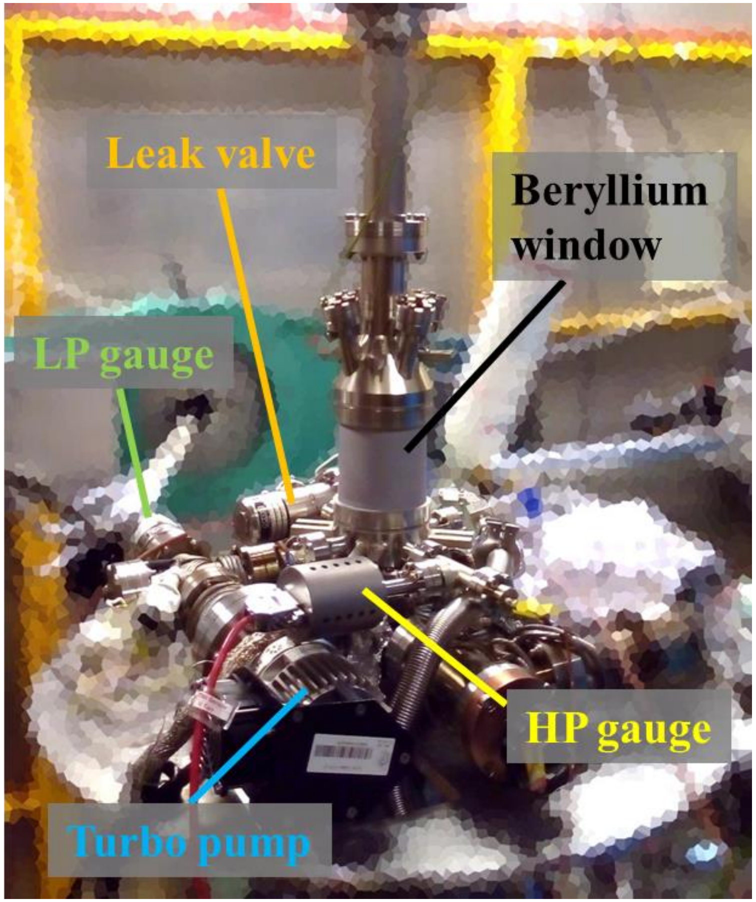




Wang et al. Phys. Rev. B 74, 235304, mpip-mainz.mpg.de/62518/Scattering_methods, jacobs.physik.uni-saarland.de

Experimental setup

In-situ X-ray reflectivity and diffraction



- Mobile
- Allows for direct measurements during treatments
- Different gas-conditions
- RT - 1200°C
- Base pressure ~ 10^{-8} mbar
- UHV prep. ~ 10^{-10} mbar

Near-surface region of Nb under annealing

UHV and N_2 atmosphere - Ultraclean sample

ESRF ID31; E = 70 keV

RT

↓

120°C

↓

200°C

↓

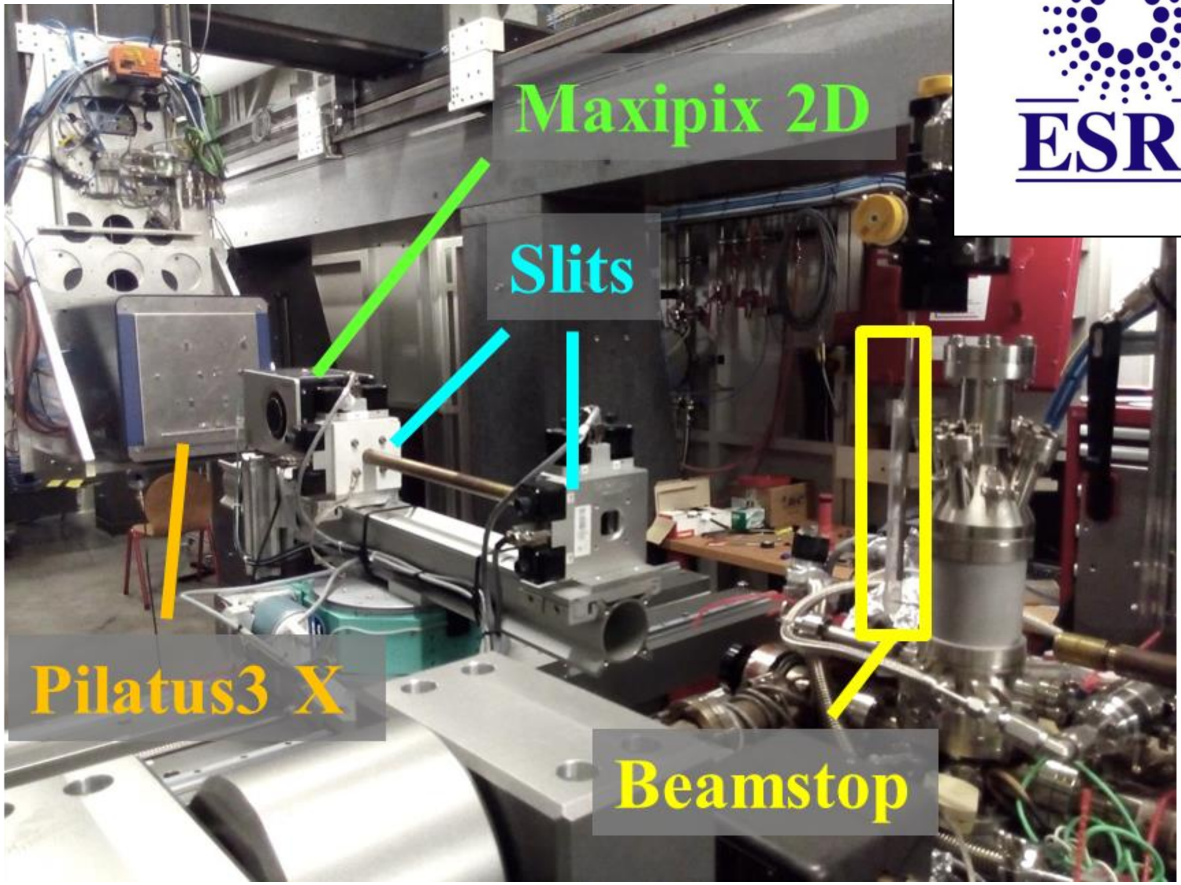
250°C

↓

250°C + N_2

↓

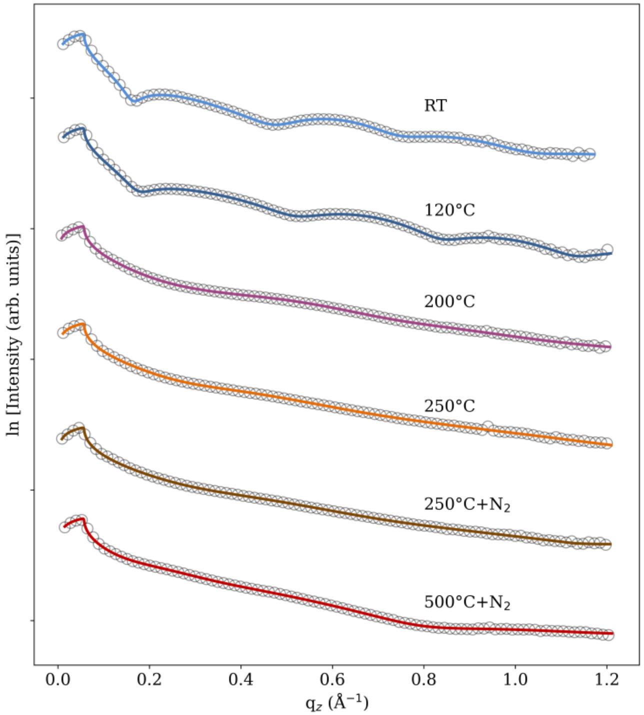
500°C

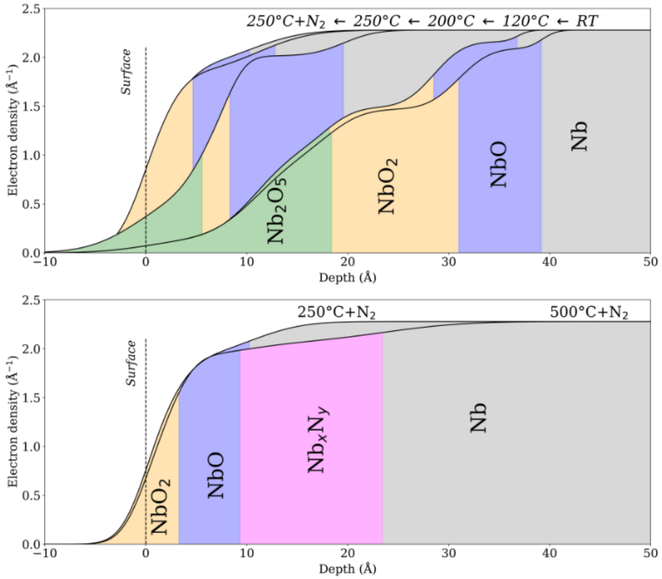


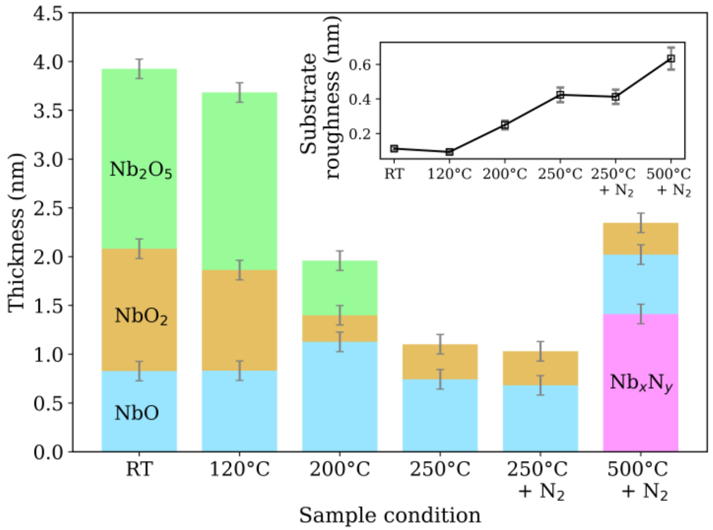
- Base pressure ~ 10^{-8} mbar
- N_2 pressure = 3.3×10^{-2} mbar (Infusion)
- Each step ~10-12 hours

Near-surface region of Nb under annealing

X-ray Reflectivity: natural oxide consumption







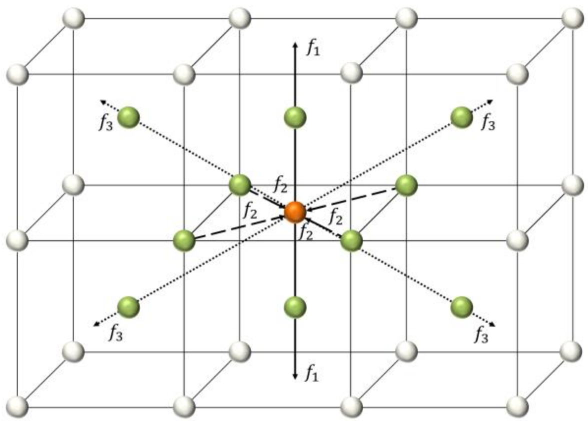
- Initial stage: Nb_2O_5 - NbO_2 - NbO
- Progressive consumption of Nb_2O_5 and NbO_2
- No nitrogen-rich layer detected at 250°C + N_2
- Nb_xN_y layer detected underneath the natural oxides at 500°C + N_2
- Substrate roughness increases with temperature

G. Semione *et al. accepted for publication at PRAB - arXiv: https://arxiv.org/abs/1906.11530*

Near-surface region of Nb under annealing

Interstitial diffuse X-ray scattering

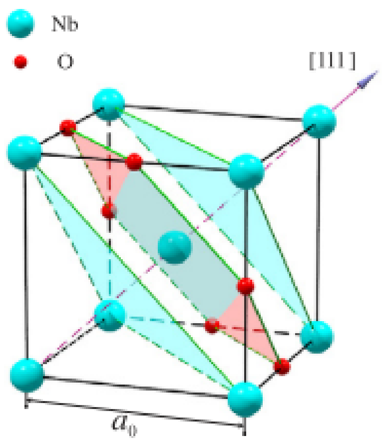
- How to measure the concentration of interstitials in Nb with X-ray diffraction?

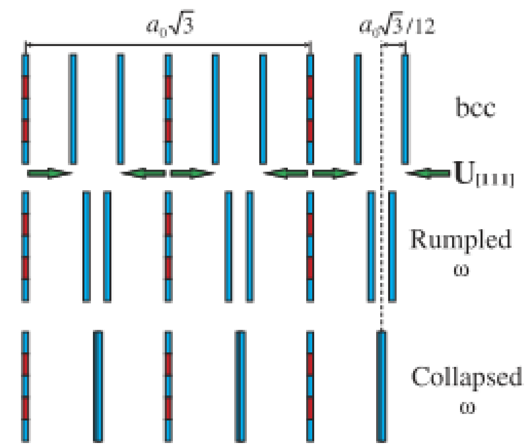


- Oxygen/Nitrogen in octahedral site
- Nearest neighbors Nb atoms affected
- bcc Nb → soft phonon in [111]

Each interstitial is a point-defect: induces ω phase

transition bcc → hexagonal

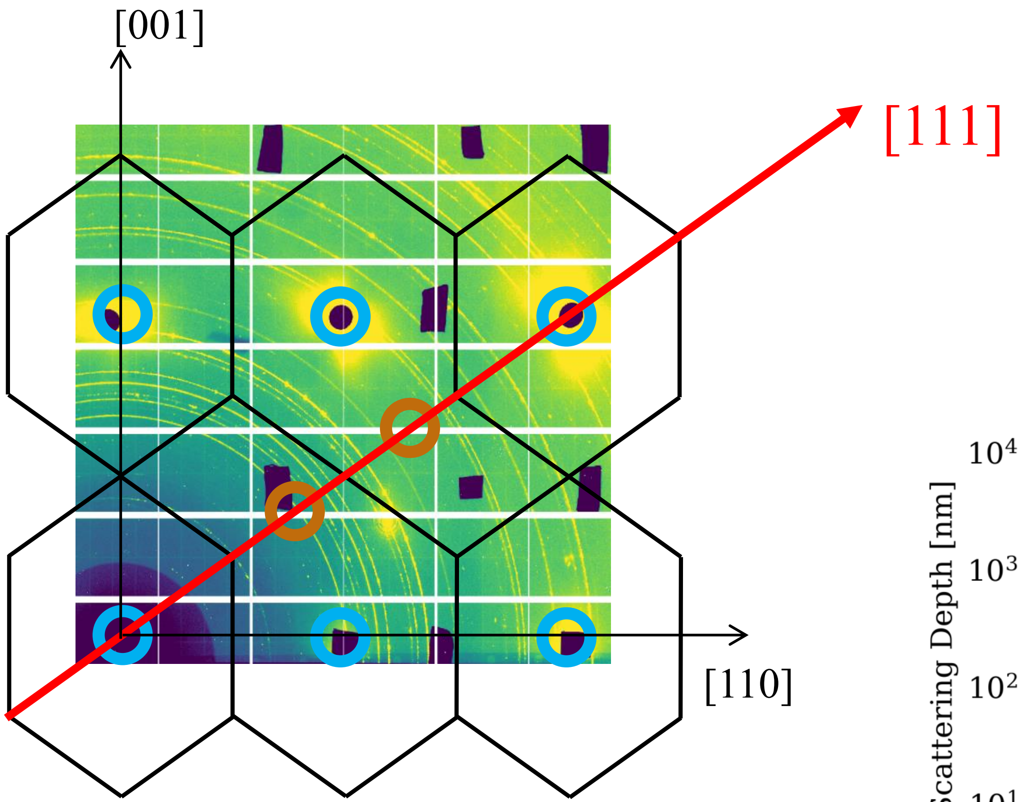




R. P. Kurta *et al.* J. Phys. Condens. Matter 20, 275206 (2008)

Near-surface region of Nb under annealing

Interstitial diffuse X-ray scattering

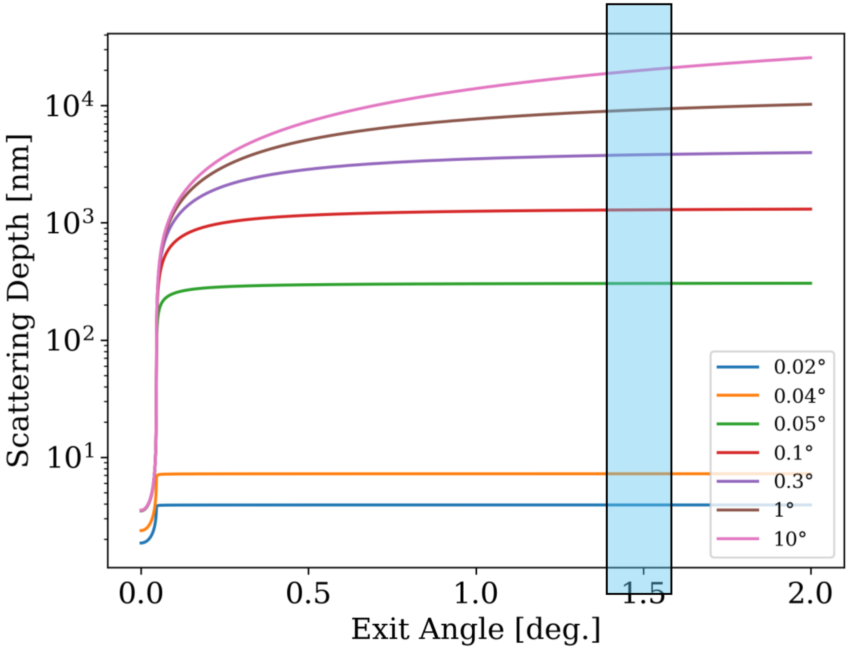


$$c(z) = ae^{(-z-z_0)/\tau} + d\,, \qquad for\ z < z_0$$

$$I \propto c(1-c)S_D(\boldsymbol{Q})$$

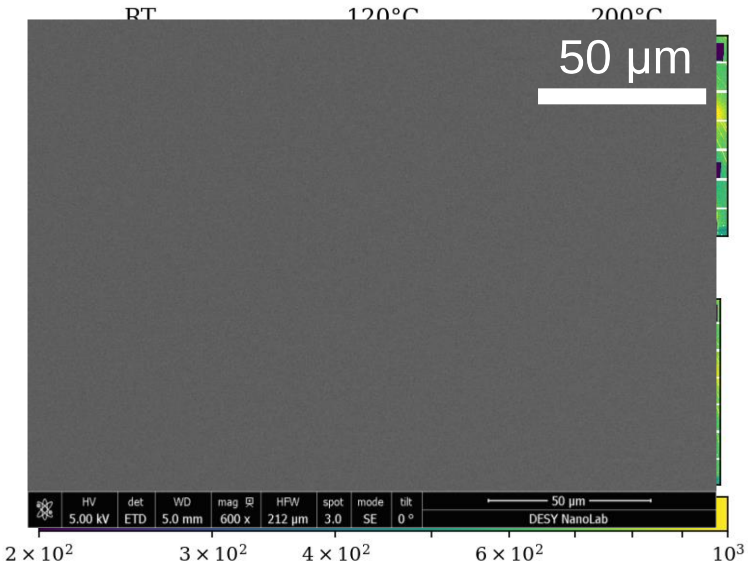
Depth-resolved:

- Tuning incident angle of X-rays

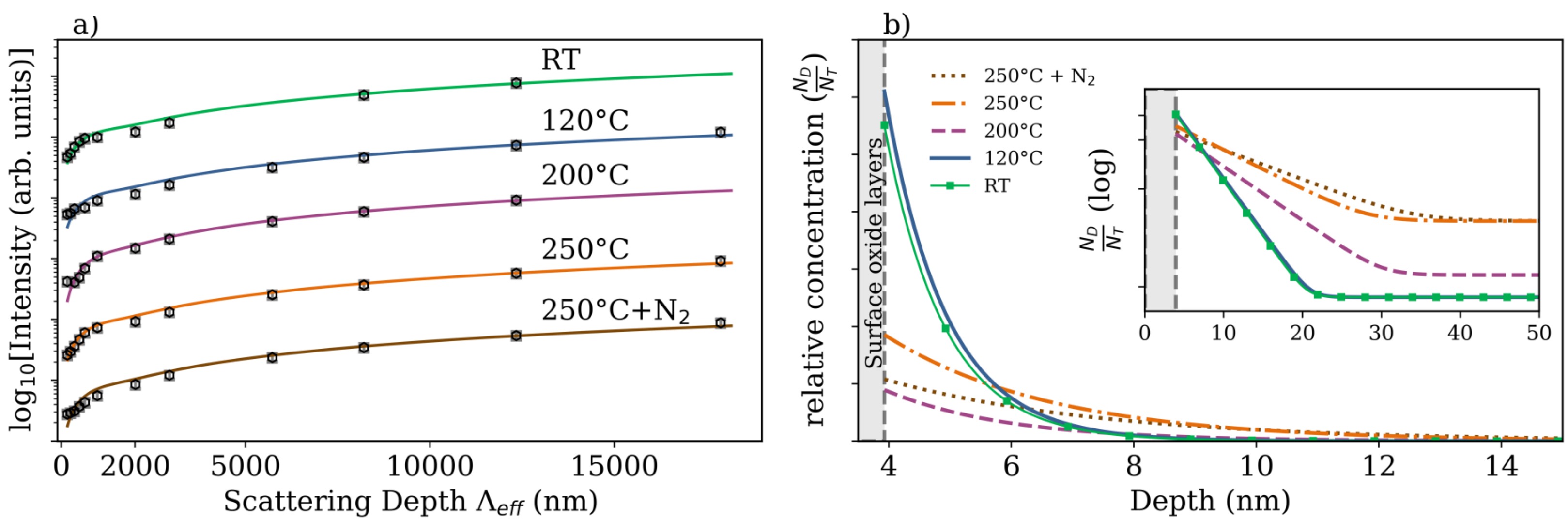


Near-surface region of Nb under annealing

Interstitial diffuse X-ray scattering

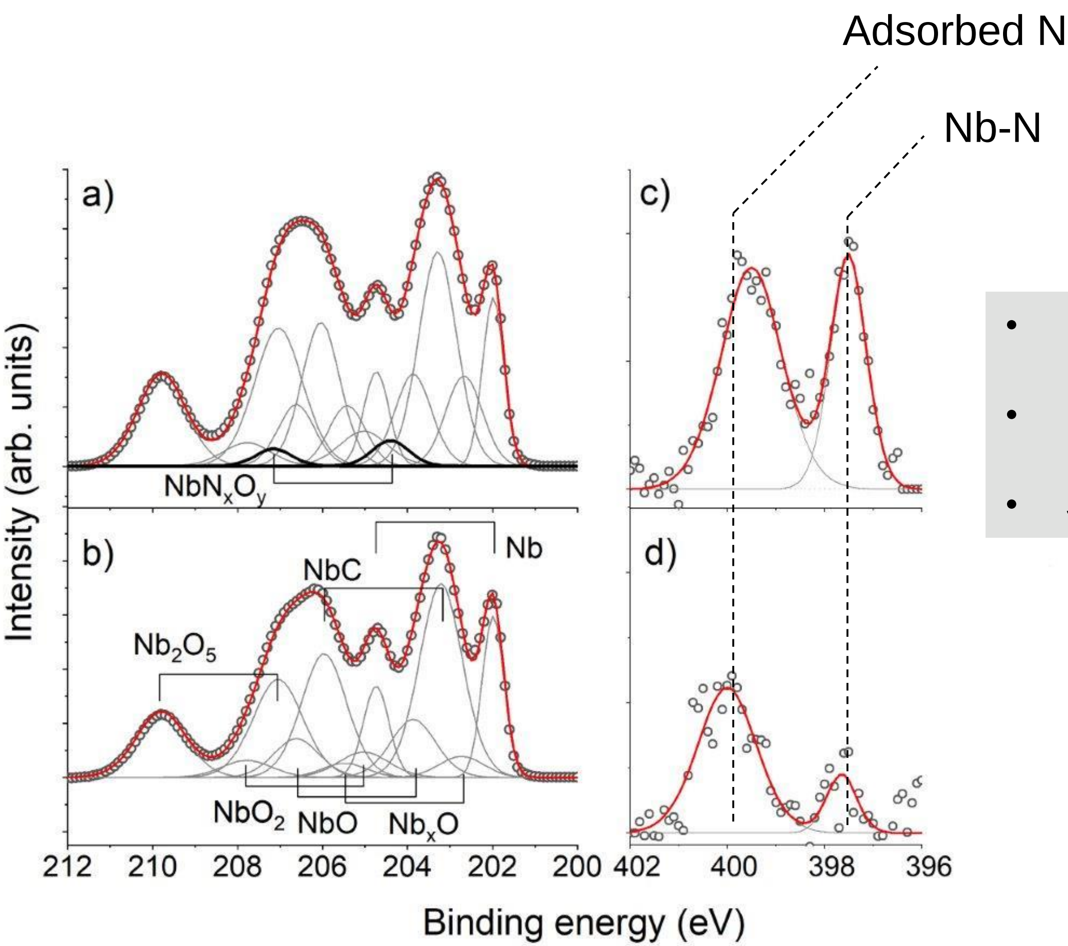


- At 120°C interstitial oxygen is mostly present in the first 10 nm
- Temperature increase leads to further diffusion of oxygen species liberated from the oxide layer
- No evidence of interstitial nitrogen at 250°C



Near-surface region of Nb under annealing

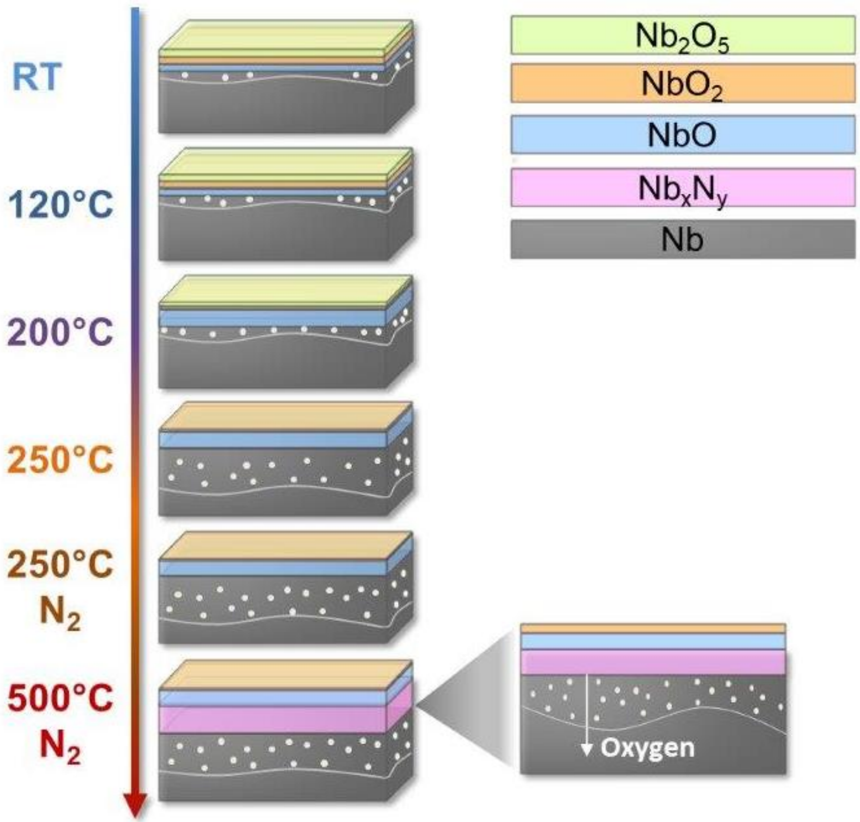
XPS after air exposure



- N 1s: Nb-N and adsorbed N
- Nb 3d: NbN_xO_y with normal exit angle
- Strong NbC presence

Conclusions

- Stepwise annealing in UHV:
  - Progressive consumption of natural oxides
  - Enrichment of the subsurface layer with oxygen interstitials
  - No evidence of N-rich layer or interstitial N at 250°C
  - Nb_xN_y layer formed underneath the oxides at 500°C
  - No precipitates observed



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Thank You.

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