Low Temperature Doping of Niobium Cavities: What is Really Going on?

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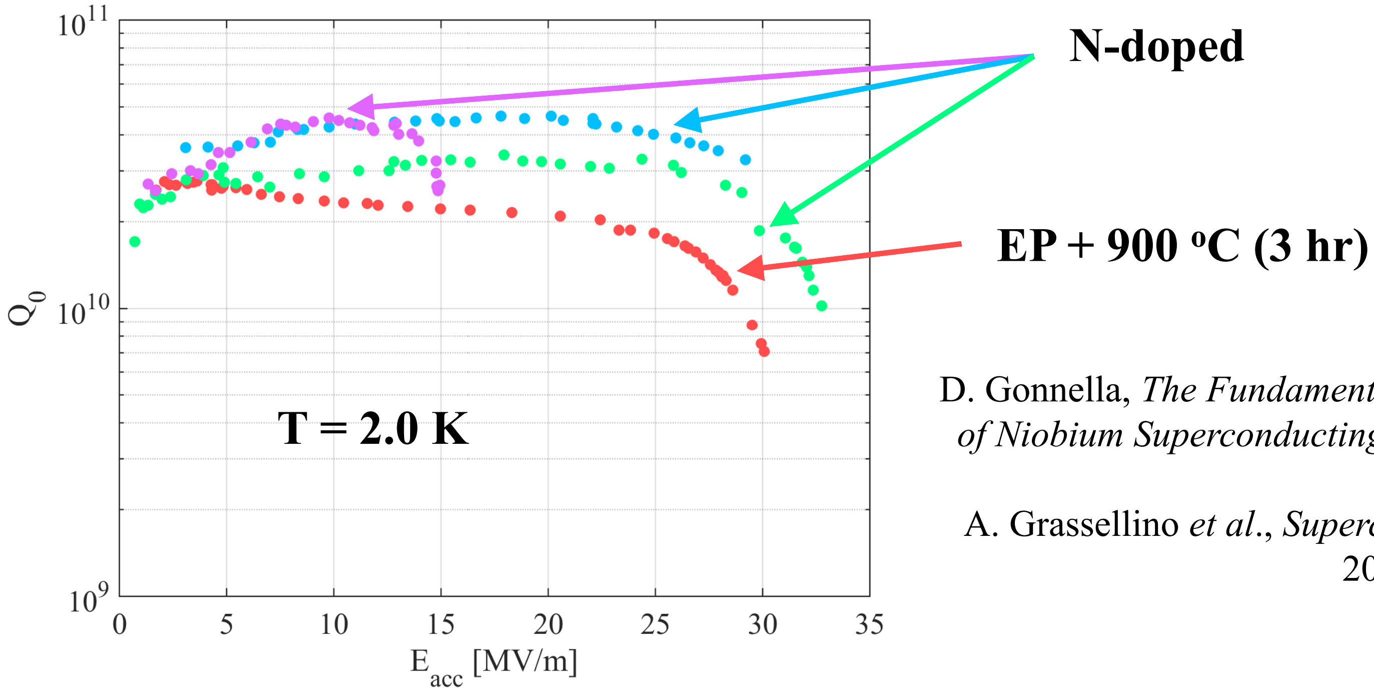
Introduction

- In high temperature nitrogen doping (**800 – 1000 ^{o}C**), nitrides form on cavity surface – nitrogen diffuses from nitride into niobium

- Diffuses **several microns** in **matter of minutes** reducing the electron **mean free path** in the RF layer (see TUYAA01 for explanation)

- Results in **anti-*Q*-slope** and **higher Q_0** values



D. Gonnella, *The Fundamental Science of Nitrogen Doping of Niobium Superconducting Cavities*. PhD Thesis. 2016.

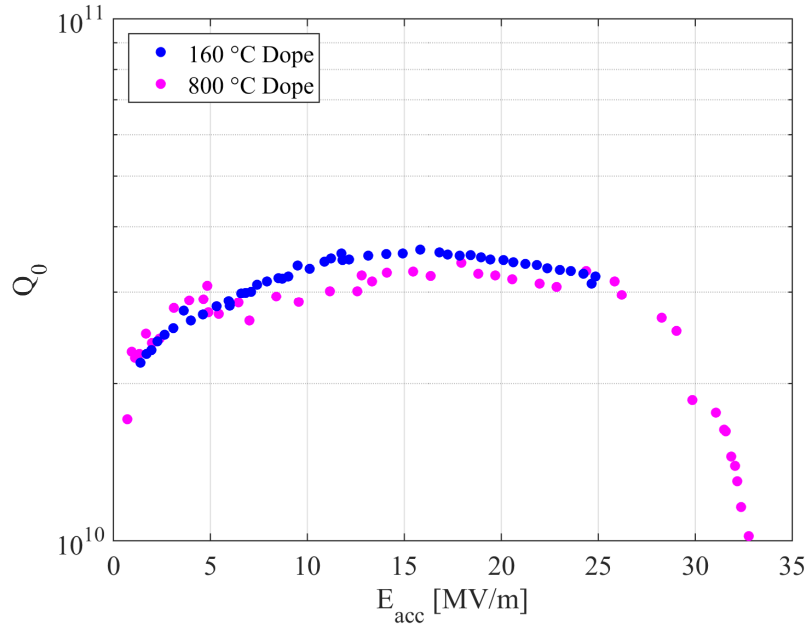
A. Grassellino *et al.*, *Supercond. Sci. Tech.*, **26**(102001), 2013.

Introduction

- Low temperature baking (**120 – 160 °C**) in a low pressure **nitrogen** atmosphere results in '***Q*-rise**' and **higher low-field Q_0 values**

  A . Grassellino *et al.*, *Unprecedented Quality Factors at Accelerating Gradients up to 45 MV/m in Niobium Superconducting Resonators via Low Temperature Nitrogen Infusion.* arXiv:1701.06077.

  P. N. Koufalis, D. L. Hall, M. Liepe, J. T. Maniscalco. *Effects of Interstitial Oxygen and Carbon on Niobium Superconducting Cavities.* arXiv:161208291.



- Nitrogen diffuses **only 2 – 5 nm** into niobium at these temperatures
- Observed that **carbon** and **oxygen diffuse significantly** into niobium at **160 °C**

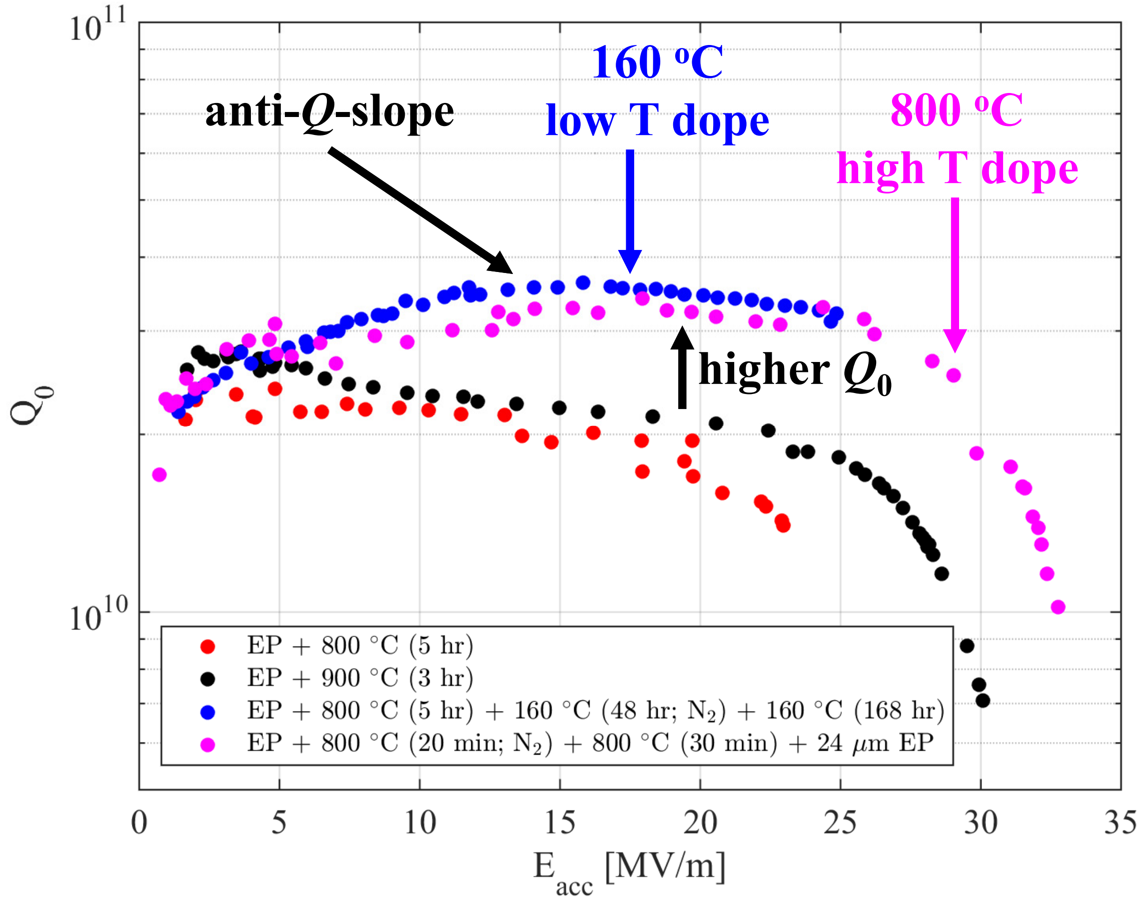
Motivation

1. Is **nitrogen** in the first **~2–5 nm** of the surface affecting cavity performance?
2. Could **carbon** and **oxygen** be responsible for the observed ***Q*-rise** and **high Q_0**?
3. How do **carbon**, **nitrogen**, and **oxygen** interact with each other in the lattice?
4. Can **other gases** be used for **low temperature doping**?
5. What are sources of **carbon**, **nitrogen**, and **oxygen**?

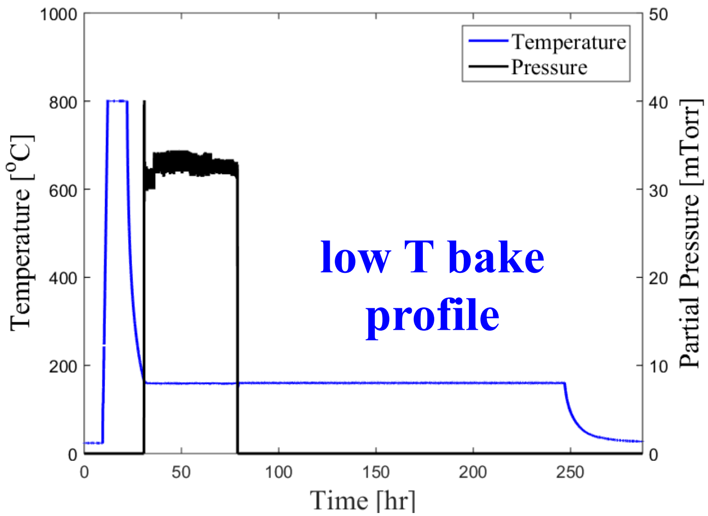
What is going on?

RF Performance

RF Performance



Continuously flowing N_2 atmosphere!



Max Quality Factor:

$Q_0 = 3.6 \times 10^{10}$ @ 16 MV/m

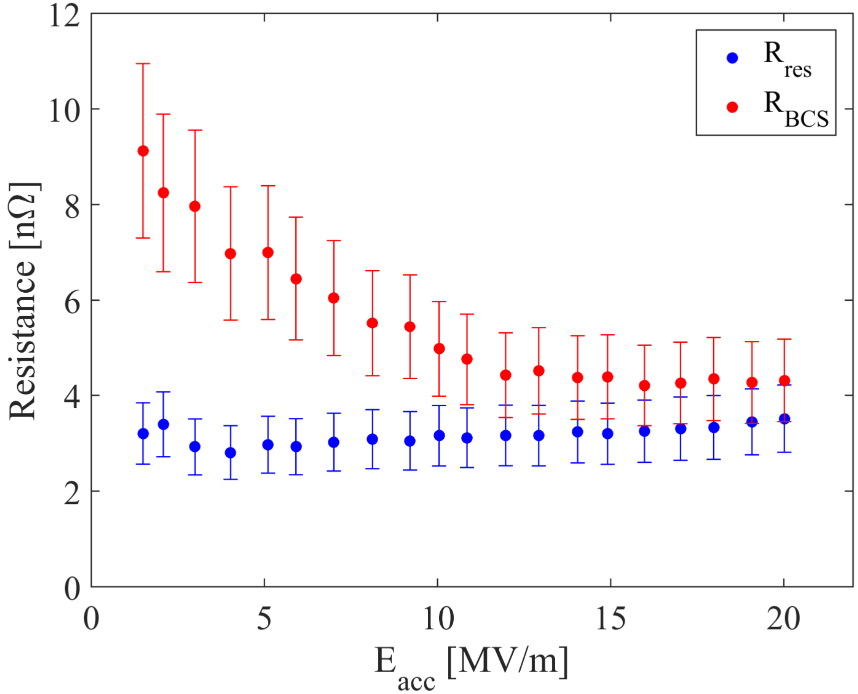
Thermal Quench:

25 MV/m (B_{pk} ~ 107 mT)

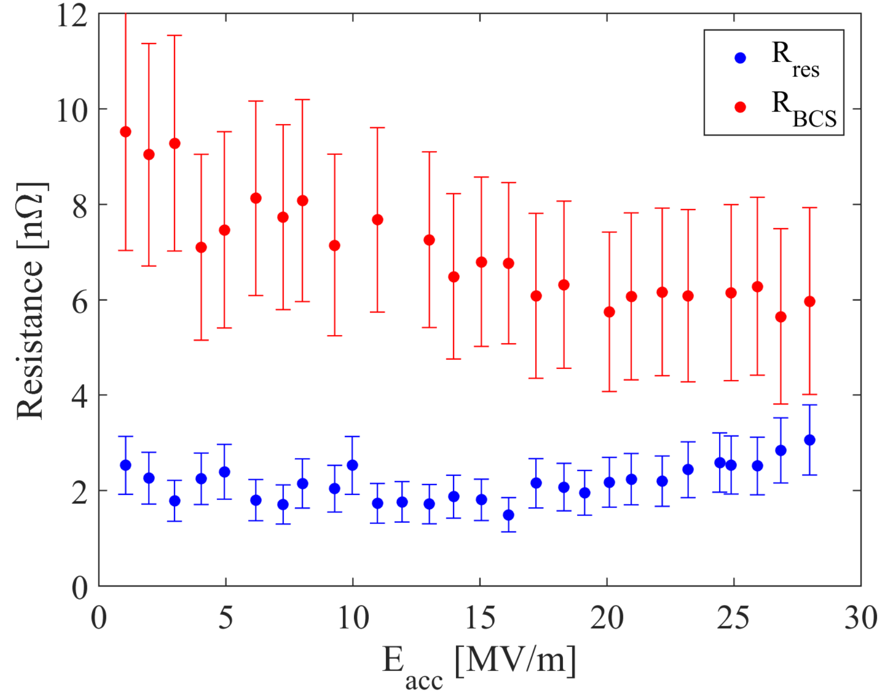
Field Dependence of R_S

- R_{BCS} **decreases** with increasing E_{acc}; **same effect** seen in **high-*T* nitrogen-doped** cavities (**caused by reduction of mean free path!**)

Low T Dope

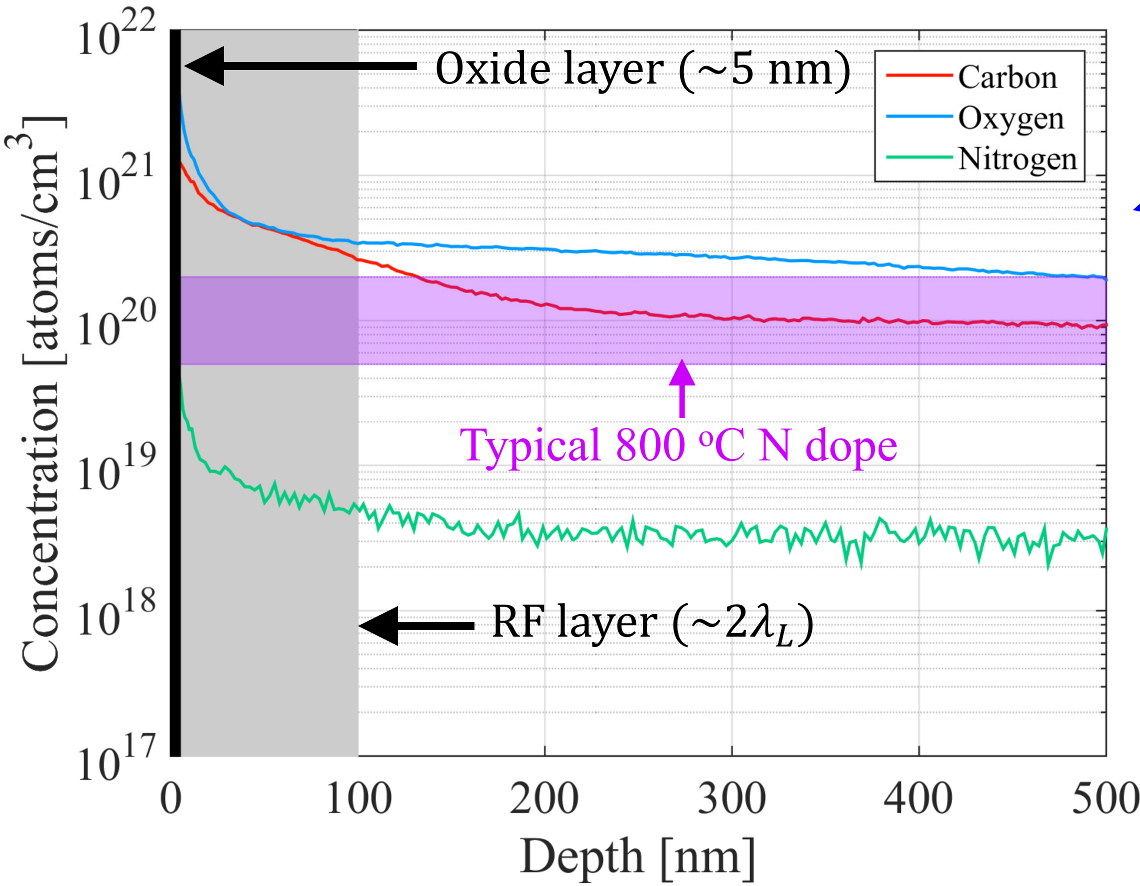


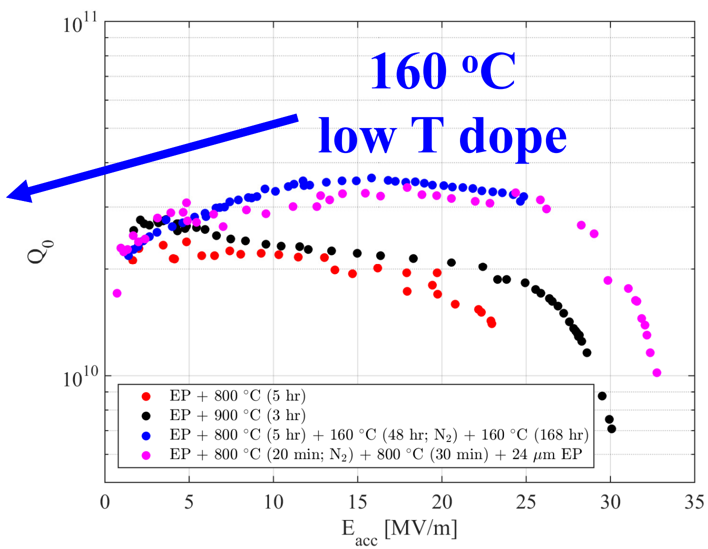
High T Dope



- Which **impurity** is **reducing** the **mean free path**?

SIMS Analysis





Carbon and **oxygen** concentrations **two orders of magnitude higher** than **nitrogen** in RF layer

Typical concentration between **$\sim 5 \times 10^{19} - 2 \times 10^{20}$ cm^{-3}** or (**0.09 – 0.36 at. % N**) for N-doped cavities

Estimated Mean Free Path

- Concentration at **depth of 50 nm** used to calculate **estimate for mean free path**

$$l = \frac{\sigma}{a \cdot c}$$

$$\sigma = 0.37 \times 10^{-15}\ \Omega \cdot \mathrm{m}^2\ ;\ a = \begin{cases} 4.3 \times 10^{-8}\ \Omega \cdot \mathrm{m}\ (\mathrm{C, O}) \\ 5.2 \times 10^{-8}\ \Omega \cdot \mathrm{m}\ (\mathrm{N}) \end{cases}\ ;\ c = \text{impurity concentration}$$

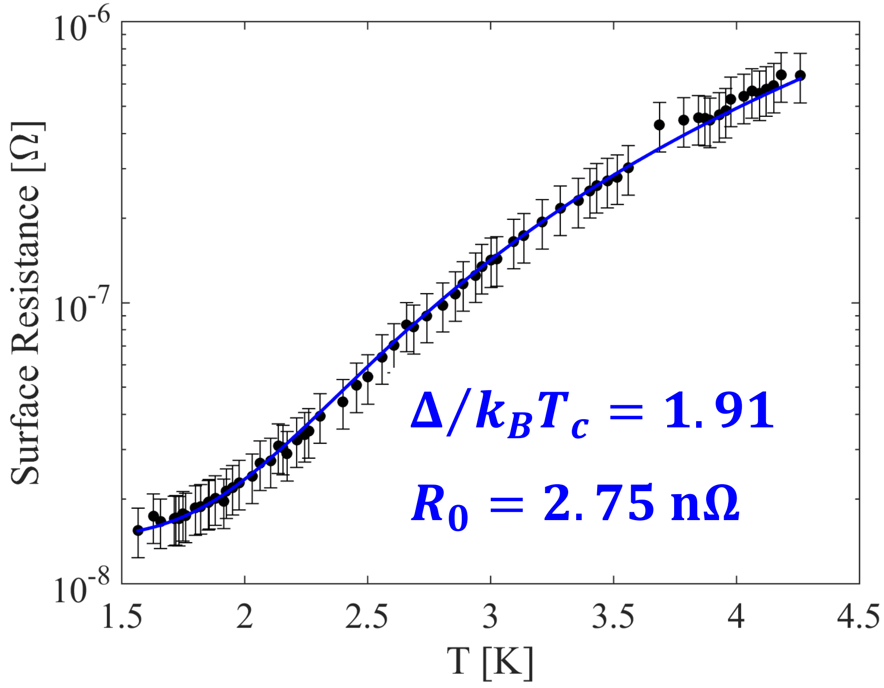
	MFP @ depth = 50 nm
C + O	5 nm
N	712 nm

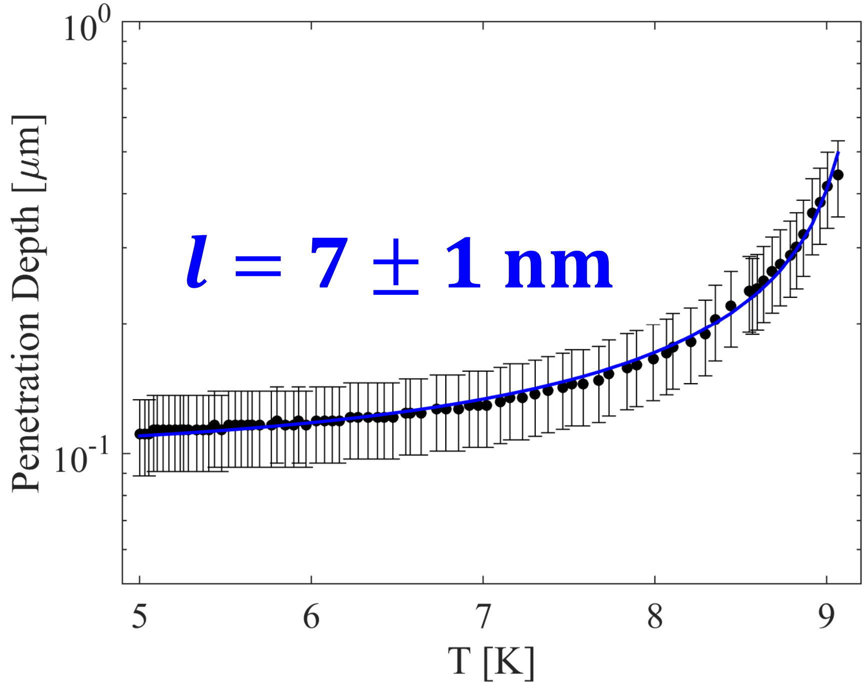
- Prediction of **mean free path** is **5 nm** based on the SIMS data – very short!

BCS Fitting

- RF measurements of **$\Delta\lambda$ vs. T** and **R_S vs. T** with BCS fit of **mean free path**, **energy gap**, and **residual resistance**
- Measured and estimated l in good agreement!

Low T Dope





Sample Analysis

Sample Studies

- **Secondary ion mass spectroscopy (SIMS)** was used to analyze **single-crystal** niobium samples prepared at **various baking temperatures** and **durations** to determine the impurity content in the first 500 nm
- All samples underwent a **~150 um EP** before being **de-gassed at 800 °C high vacuum for 5 hr**; low *T* treatment began immediately after de-gas!
- Samples were **wrapped in niobium foil** to prevent furnace contamination
- Recipes you will see throughout presentation shown in table

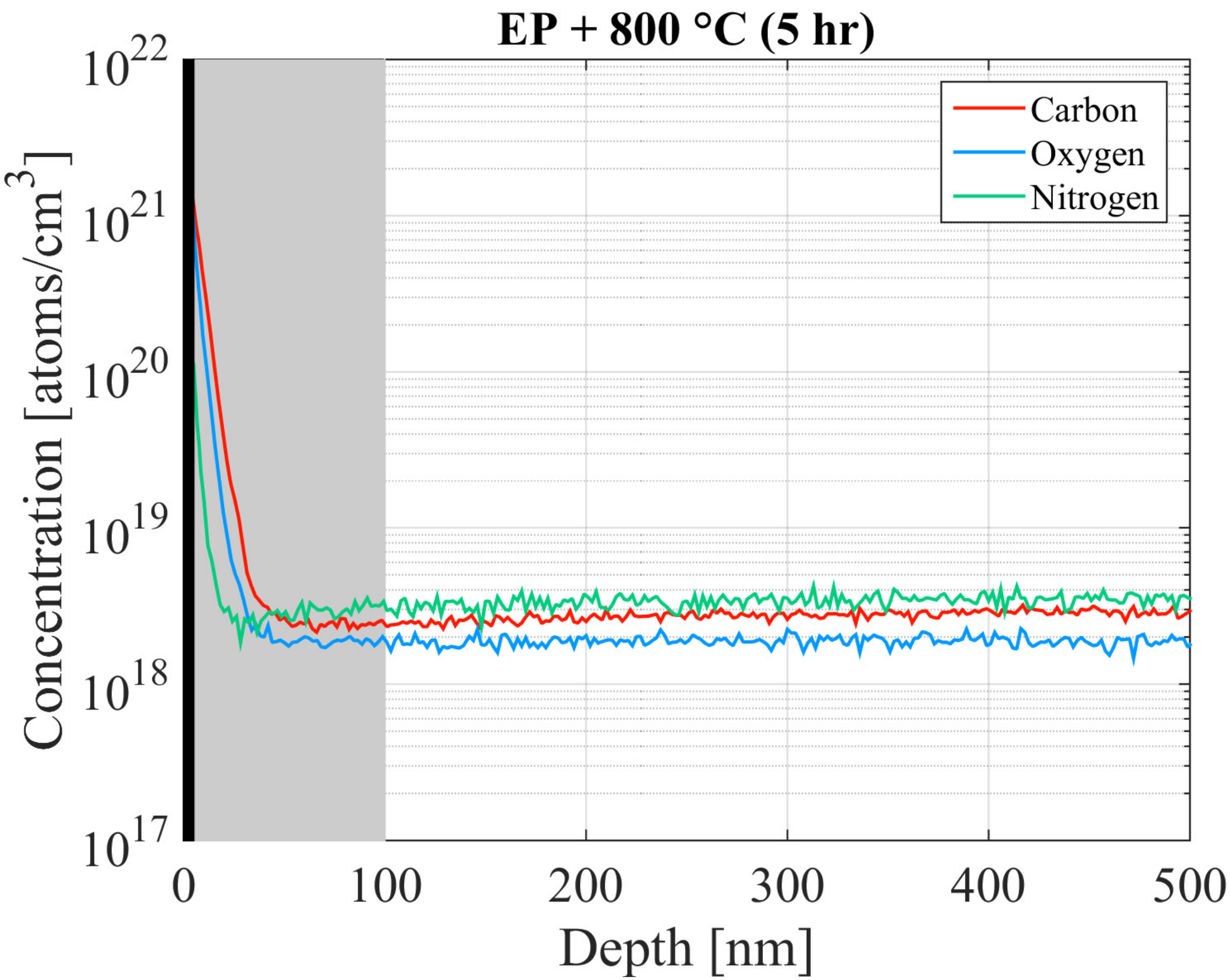
Sample #	Baking Recipe
1	800 °C (5 hr)
2	800 °C (5 hr) + 160 °C (48 hr)
3	800 °C (5 hr) + 160 °C (48 hr; N_2)
4	800 °C (5 hr) + 160 °C (96 hr; N_2)
5	800 °C (5 hr) + 120 °C (48 hr; N_2)
6	800 °C (5 hr) + 160 °C (48 hr; Ar) – **99.999 %**
7	800 °C (5 hr) + 160 °C (48 hr; Ar) – **99.9999 %**
8	800 °C (5 hr) + 160 °C (48 hr; Ar + CO_2) – 10 ppm CO_2

Motivation

- How does bake **temperature** and **duration** affect **concentration profiles** of **C**, **N**, and **O**?
- Can **other gases** be used for **low temperature doping**?
- Sources of **C**, **N**, and **O**?

Sample #	Baking Recipe
1	800 ^oC (5 hr)
2	800 ^oC (5 hr) + 160 ^oC (48 hr)
3	800 ^oC (5 hr) + 160 ^oC (48 hr; N_2)
4	800 ^oC (5 hr) + 160 ^oC (96 hr; N_2)
5	800 ^oC (5 hr) + 120 ^oC (48 hr; N_2)
6	800 ^oC (5 hr) + 160 ^oC (48 hr; Ar) – **99.999 %**
7	800 ^oC (5 hr) + 160 ^oC (48 hr; Ar) – **99.9999 %**
8	800 ^oC (5 hr) + 160 ^oC (48 hr; Ar + CO_2) – 10 ppm CO_2

Baseline



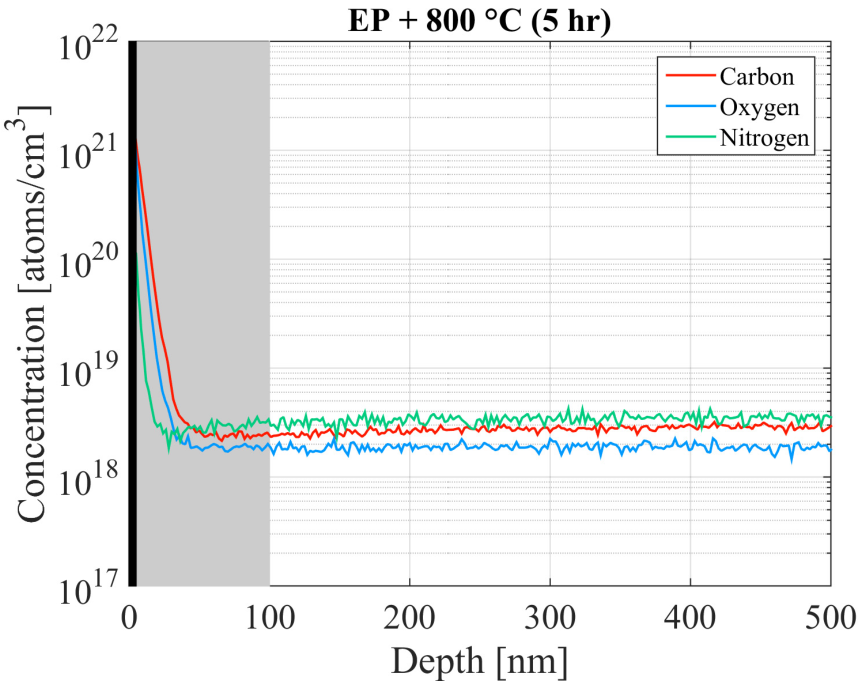
Degassing **800 °C vacuum bake** for 5 hr

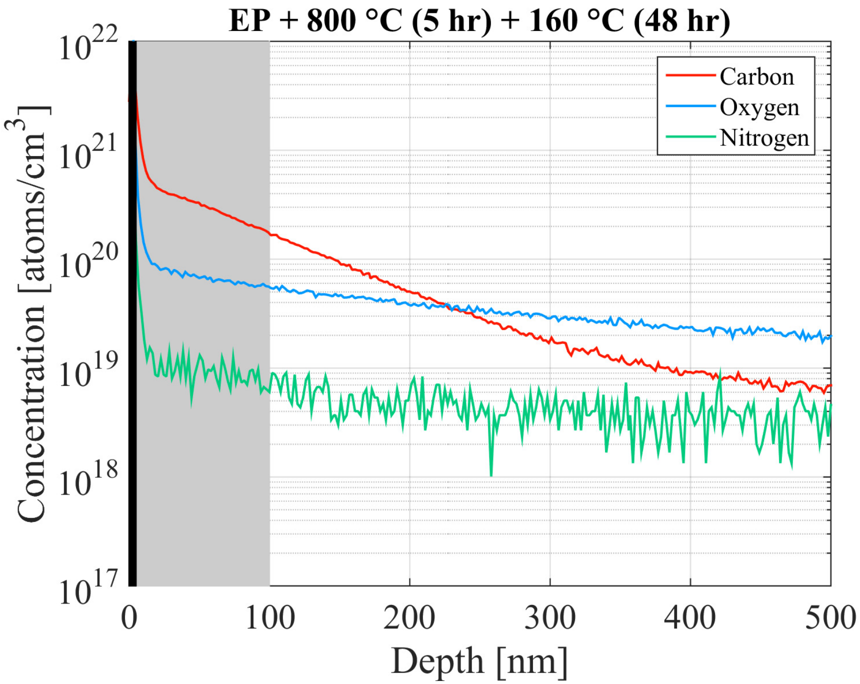
C, **N**, and **O** concentrations **drop off rapidly** to background levels

High mobility of all three impurities **at this temperature**

Background level ~ **1×10^{18} – 5×10^{18} cm^{-3}**

Nitrogen Baking

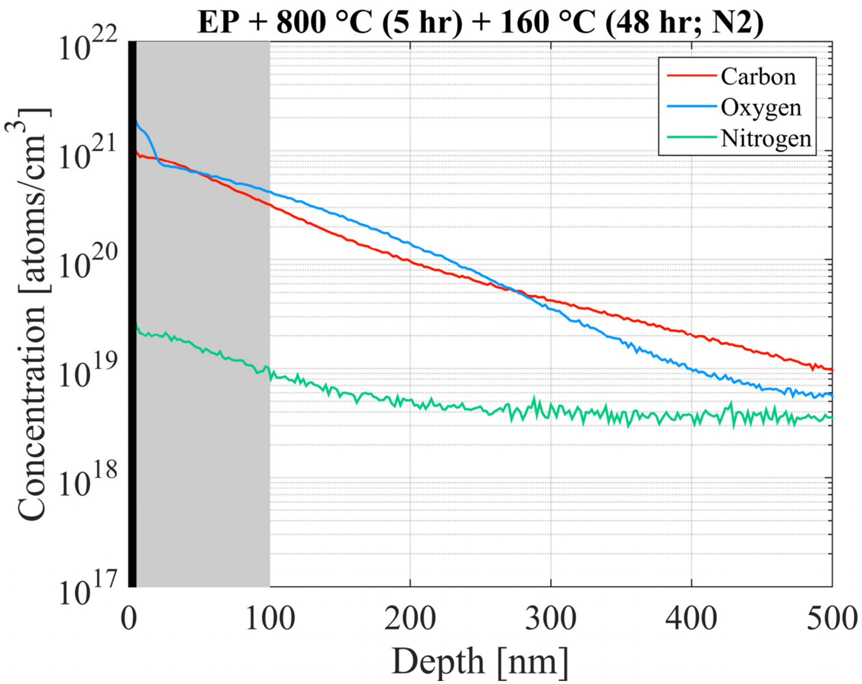


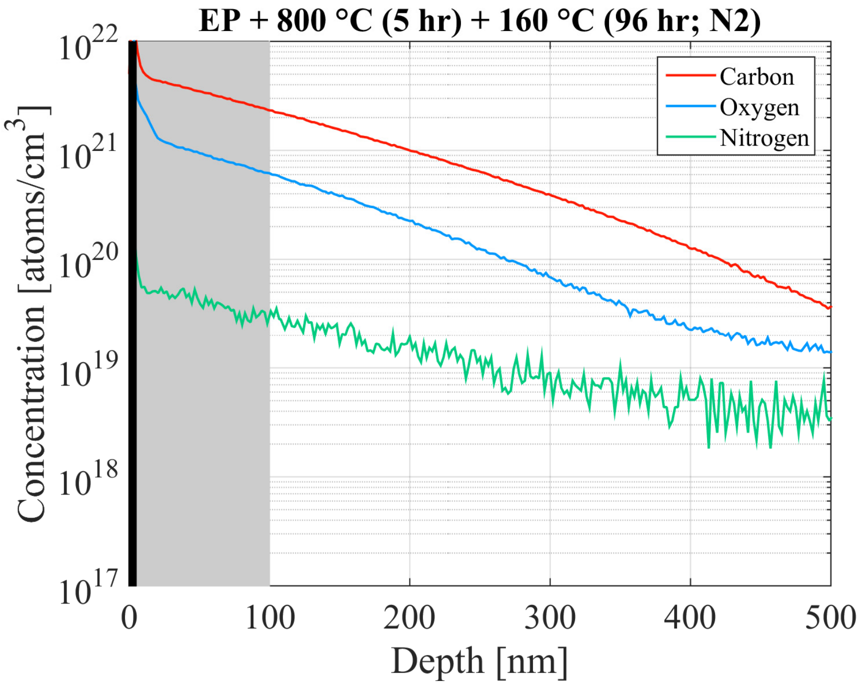


Baseline

+ 48 hr @ 160 °C

Niobium acts as a very **good getter in vacuum** – absorbs residual gases in furnace such as H_2O, O_2, CO, CO_2, N_2, etc.



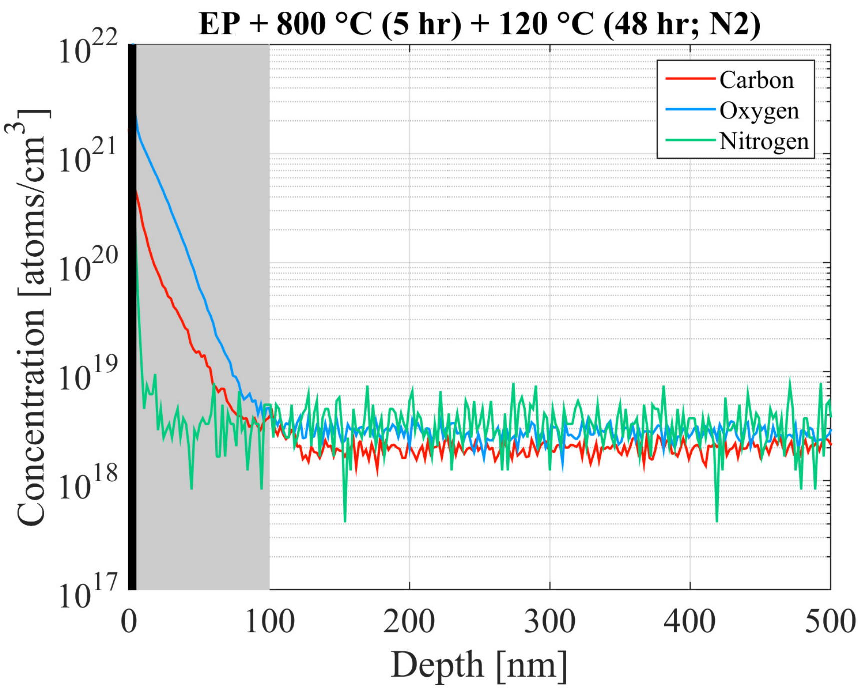


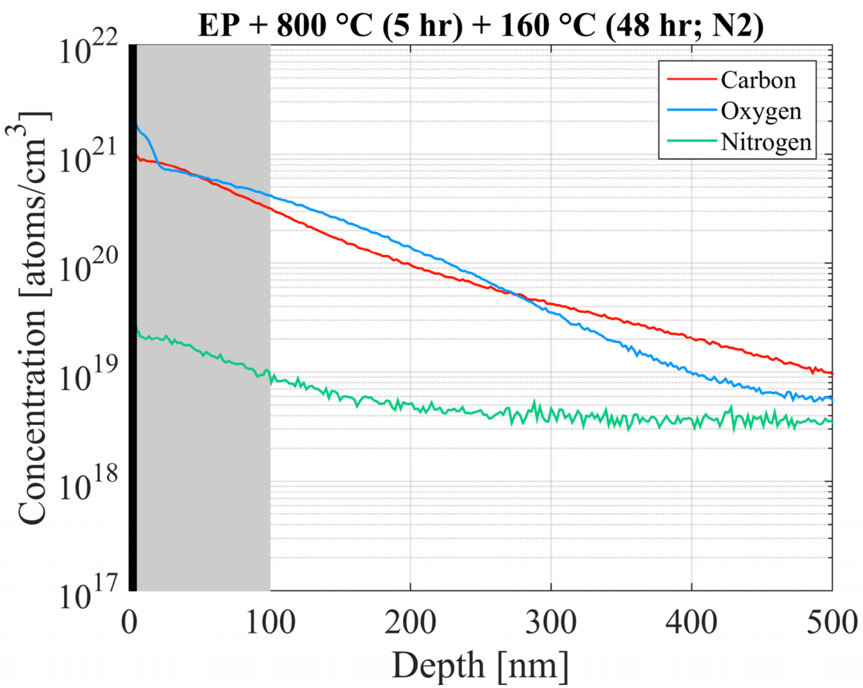
48 hr @ 160 ^{o}C

96 hr @ 160 ^{o}C

The diffusion length increases for carbon, nitrogen, and oxygen as when doping time is increased.

$$L_{\text{diff}} = \sqrt{Dt}$$





120 °C **160 °C**

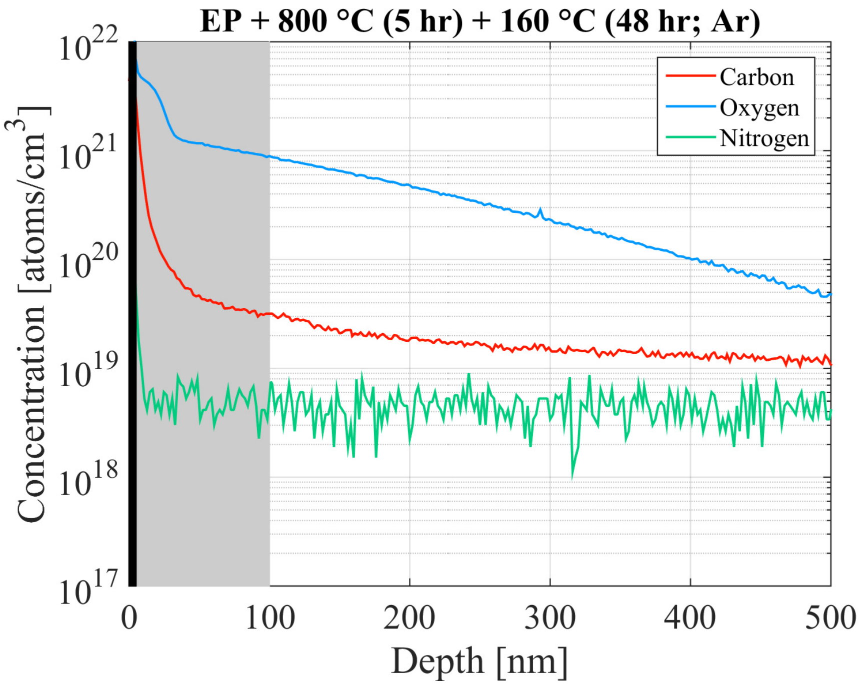
The diffusion length increases for carbon, nitrogen, and oxygen as when doping temperature increases.

$$L_{\text{diff}} = \sqrt{Dt}$$

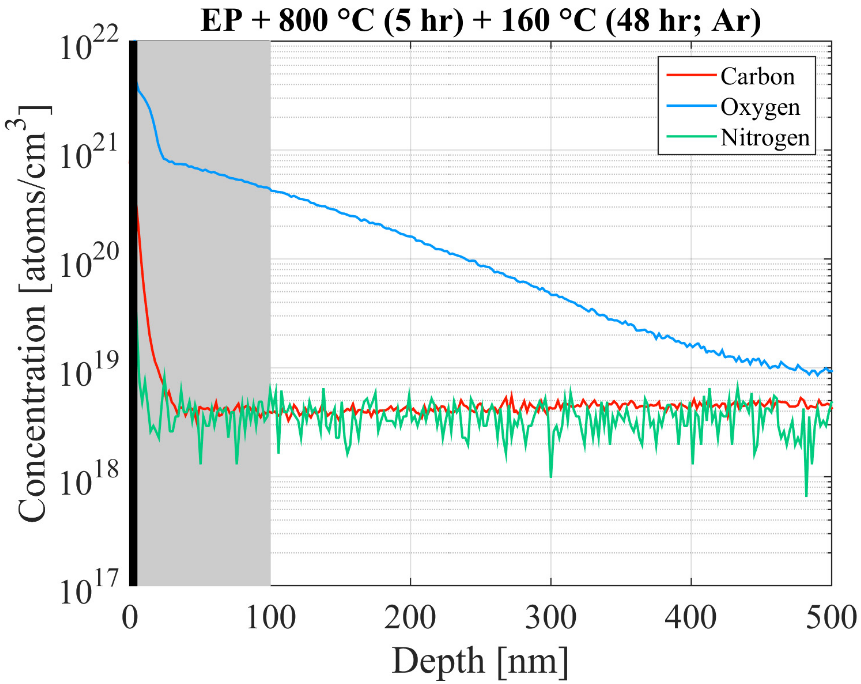
$$D = D(T)$$

Argon Baking

Gas Purity

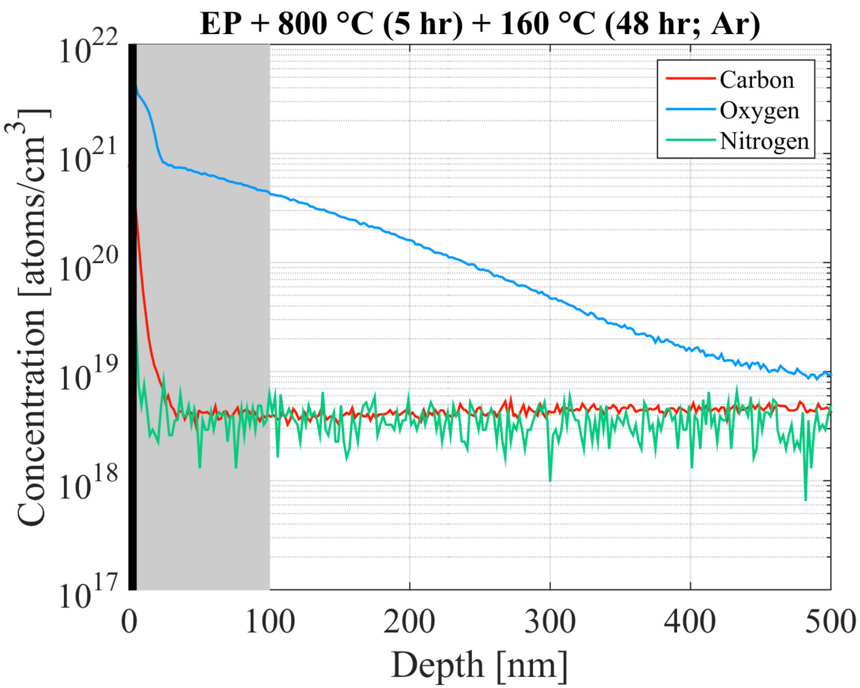


Ultra High Purity Argon
(99.999 %)



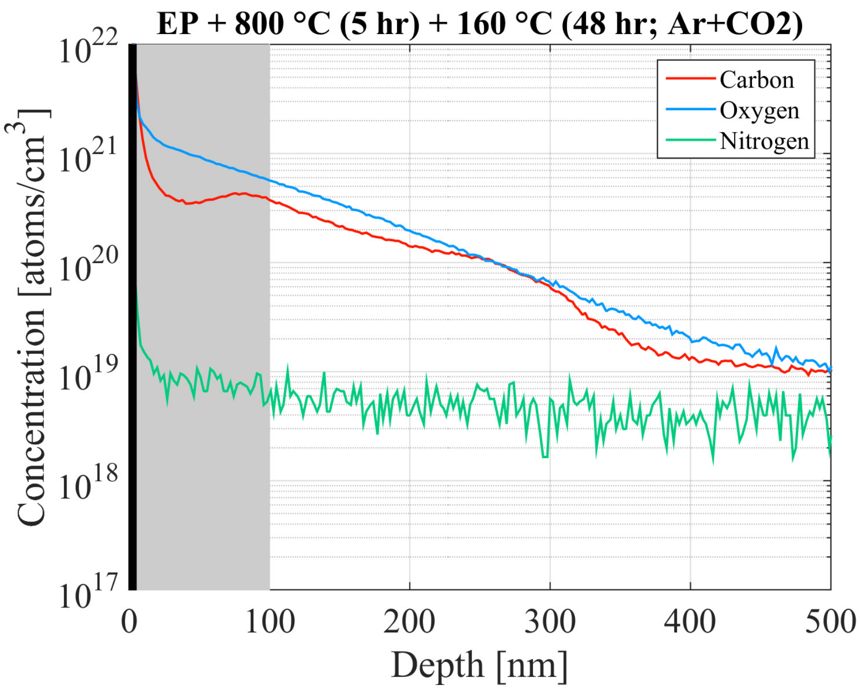
Research Plus Argon
(99.9999 %)

The purity of gas used has a high impact on the impurity concentration! The argon with higher trace impurities leads to a higher concentration in the RF layer.



Research Plus Argon

(99.9999 %)

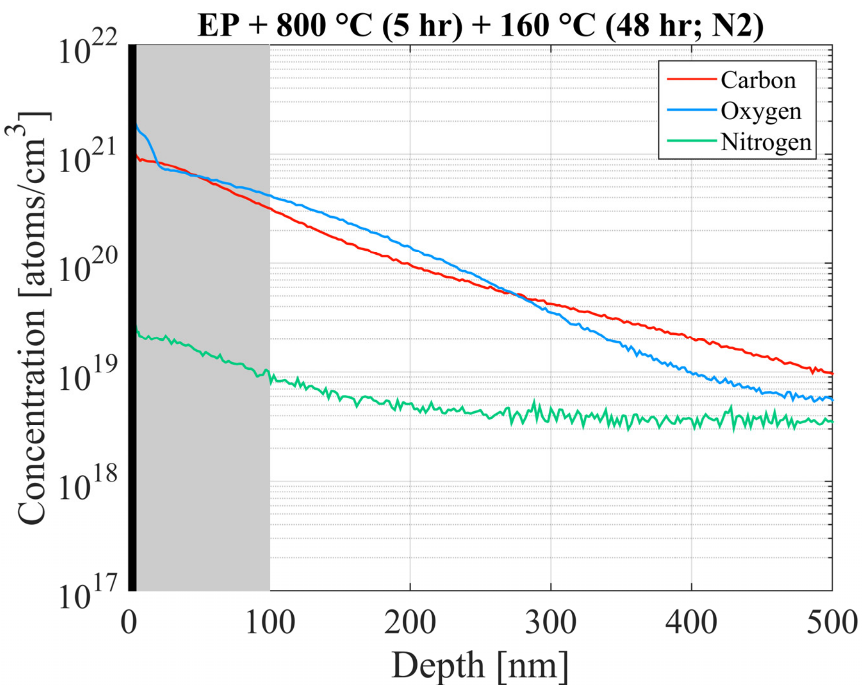


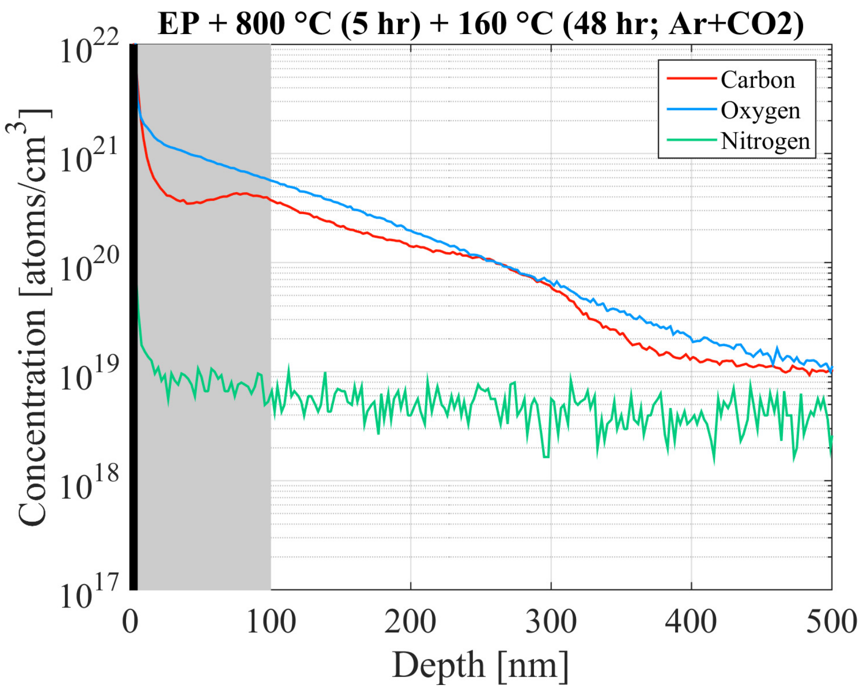
Research Plus Argon + 10 ppm CO_2

(99.9999 %)

The research plus argon mixed with 10 ppm CO_2 significantly increases the concentration of carbon in the niobium! Trace impurities in the gas are important.

Nitrogen vs. Argon





Nitrogen | **RP Argon + 10 ppm CO_2**

Argon gas mixture can be used to test whether or not nitrogen has an impact on performance.

Concluding Remarks

- **Anti-*Q*-slope** is correlated with **shorter mean free path** (l < 100 nm)
  - Shorter mean free path → stronger anti-*Q*-slope

J. T. Maniscalco, D. Gonnella, M. Liepe. *J. App. Phys.* **121**, 043910 (2017).

TUYAA01

- For low temperature baking (**120 – 160 °C**), **C** and **O** dominate in the reduction of the mean free path resulting in same effects observed in high temperature doping:
  - **anti-*Q*-slope** and **increased low-field Q_0** values
- Future work:
  - Cavity testing with various recipes used for sample baking
  - Further sample analysis
  - Sensitivity to trapped magnetic flux
  - Interactions between impurities?