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RF and Material Studies on Interstitial Impurities in Bulk Nb Cavities

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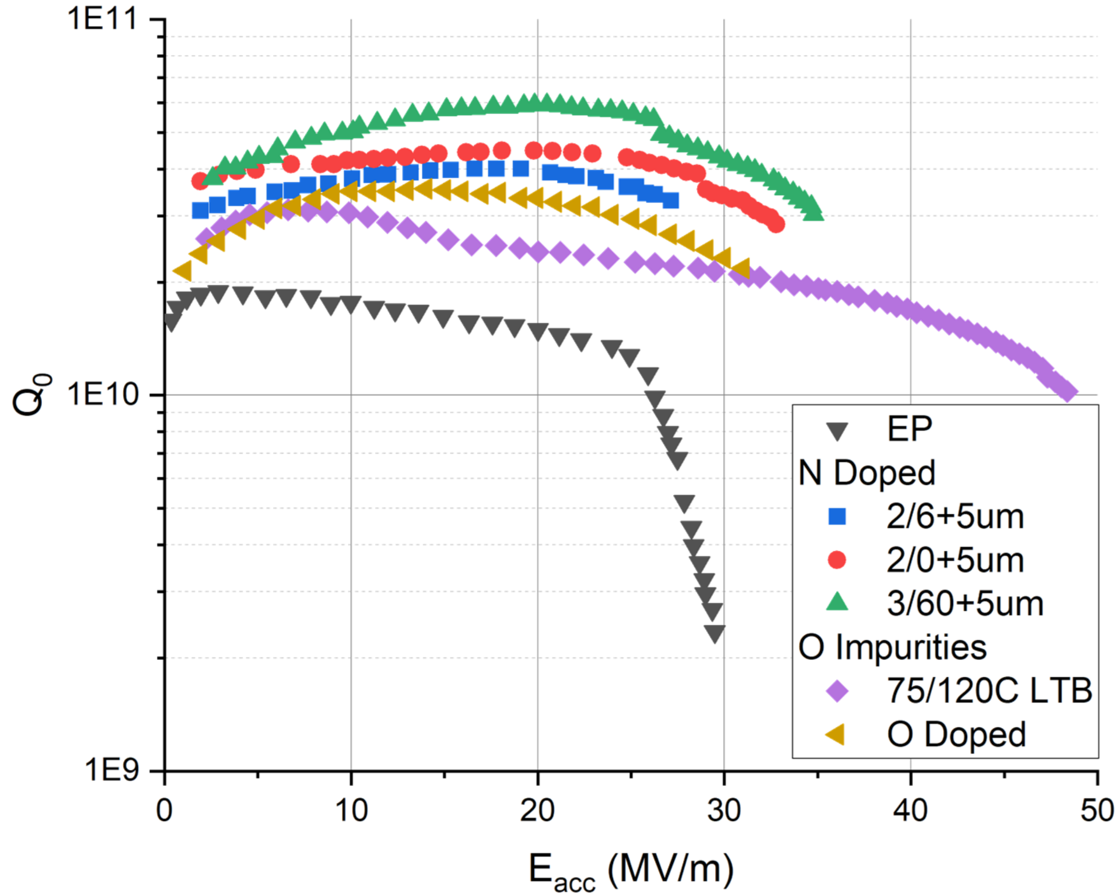
TESLA TECHNOLOGY COLLABORATION THE UNIVERSITY OF CHICAGO

Motivation

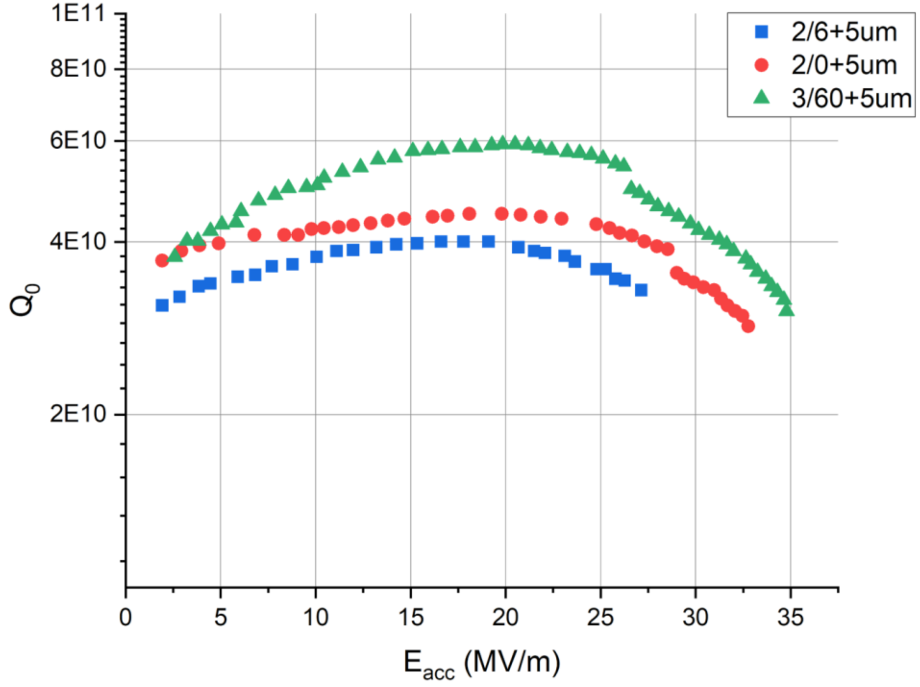
- O and N enable high Q or high E_{acc} performance
  - A. Grassellino *et al*., doi: 10.1008/0.953-2048/26/10/102001
  - D. Bafia *et al*., doi:10.18429/JACoW-SRF2021-THPTEV016
  - E. Lechner *et al*. arXiv:2106.06647
  - H. Ito *et al.*, doi:10.1093/ptep/ptab056
- How to achieve simultaneous high Q and high E_{acc}?
- Study the role of O and N impurities to fully understand their microscopic properties

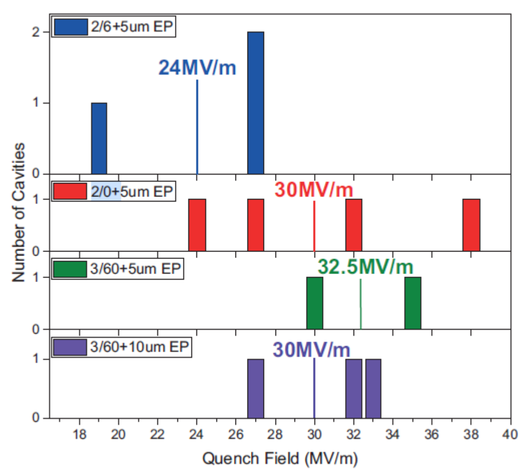


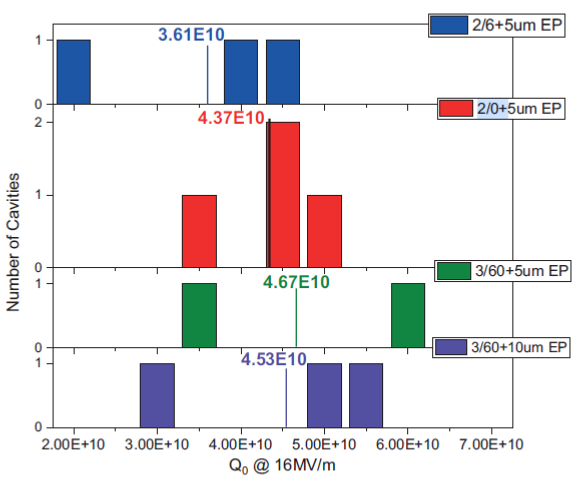
Corroborate differences in RF performance through material studies of samples subjected to N and O based treatments.

Nitrogen Doping

- Start with an in-depth analysis of nitrogen
  - LCLS-II HE: 2/0+5µm cold EP
  - LCLS-II: 2/6+5µm EP
  - 3/60+5µm EP
- Is observed improvement due to a change in N concentration?
- Can we correlate RF performance to material properties?







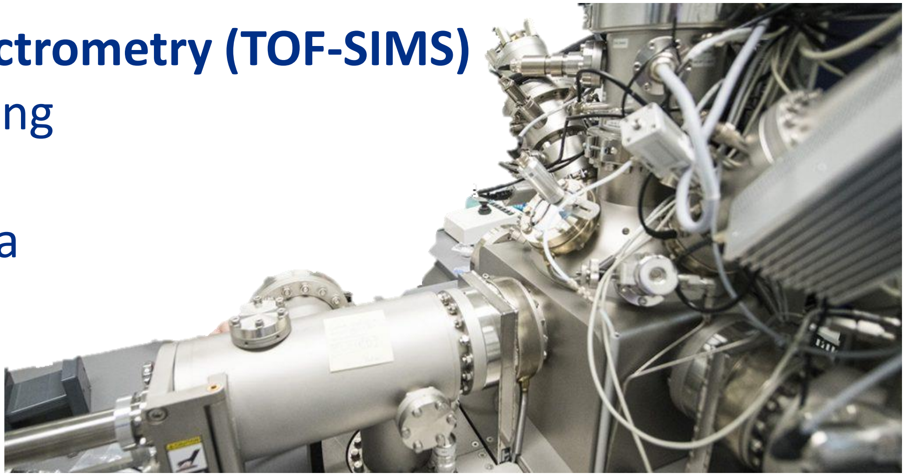
D. Bafia, PhD Dissertation, IIT, 2020.

Sample Prep and Measurement

2/6 Doping	800°Cx3hr UHV	800°Cx2min w/ 25mTorr N_2	800°Cx6min UHV	5µm EP removal
2/0 Doping	800°Cx3hr UHV	800°Cx2min w/ 25mTorr N_2	N/A	5µm EP removal
3/60 Doping	800°Cx3hr UHV	800°Cx3min w/ 25mTorr N_2	800°Cx60min UHV	5µm EP removal

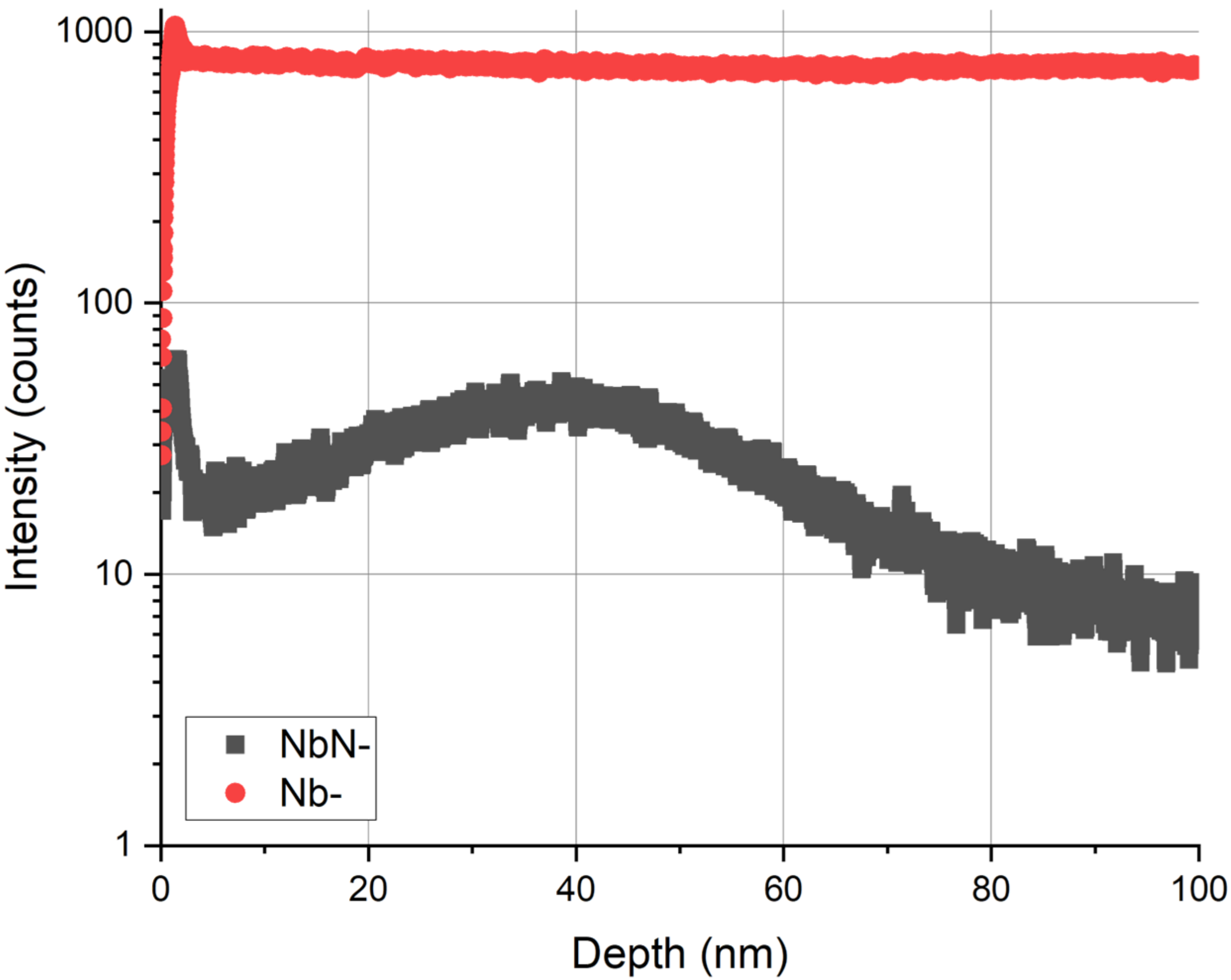
Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS)

- Depth profiling with Cs+ gun sputtering
- 3D imaging
- Removal of particle contaminant data
- Retroactive spectrum fitting
- In-situ baking up to 800°C
- Vacuum < 4 e-10 mbar



SIMS Absolute Concentration

O and N implanted standards for absolute concentration.



Nb implanted with dose: 6.37E14 of N at 100 keV

Concentration of N: C_N [ions/cm^3]

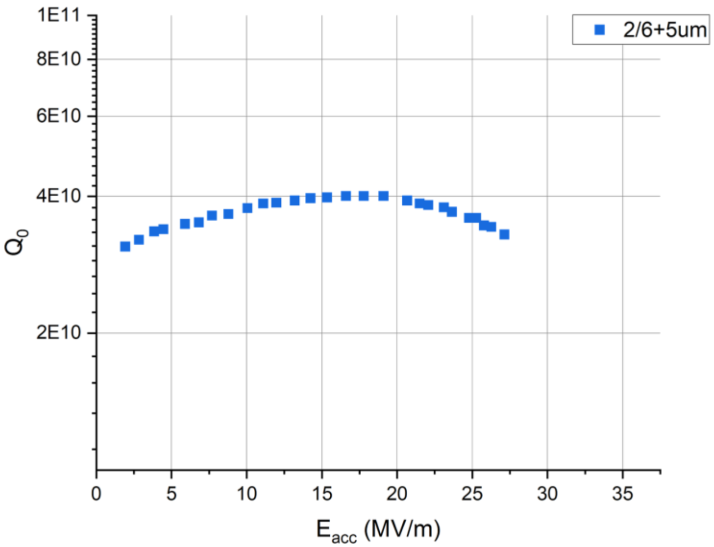
Secondary ion yield of NbN-: I_{NbN-} [ions/s]

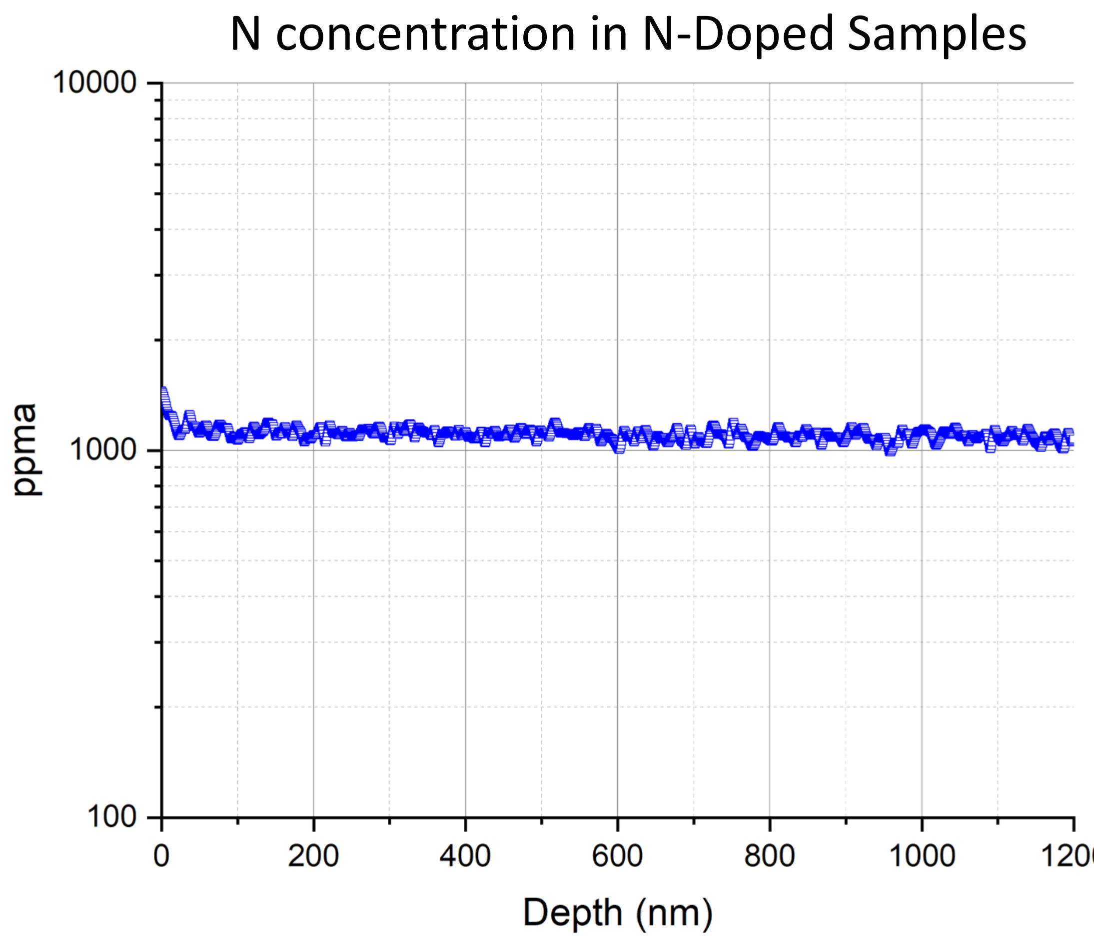
Secondary ion yield of Nb: I_{Nb-} [ions/s]

Relative Sensitivity Factor: RSF = $C_N \times I_{Nb-} / I_{NbN-}$ = 2.098E21[ions/cm^3]

N-Doped Nitrogen Concentration

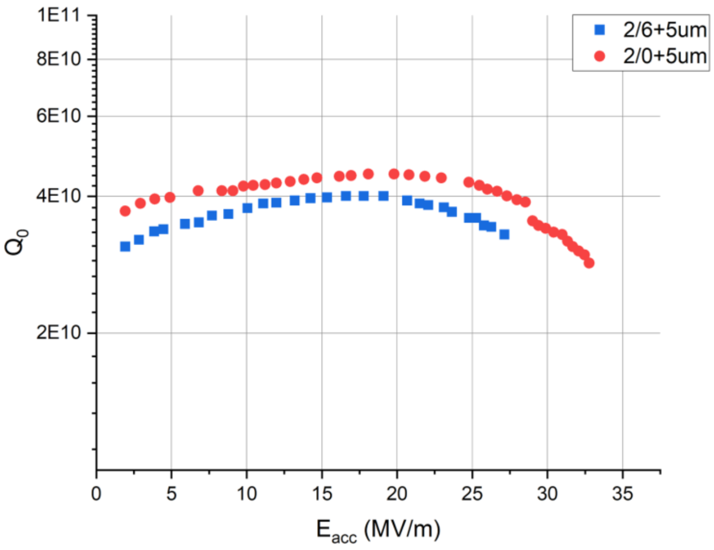
- 2/6+5μm EP

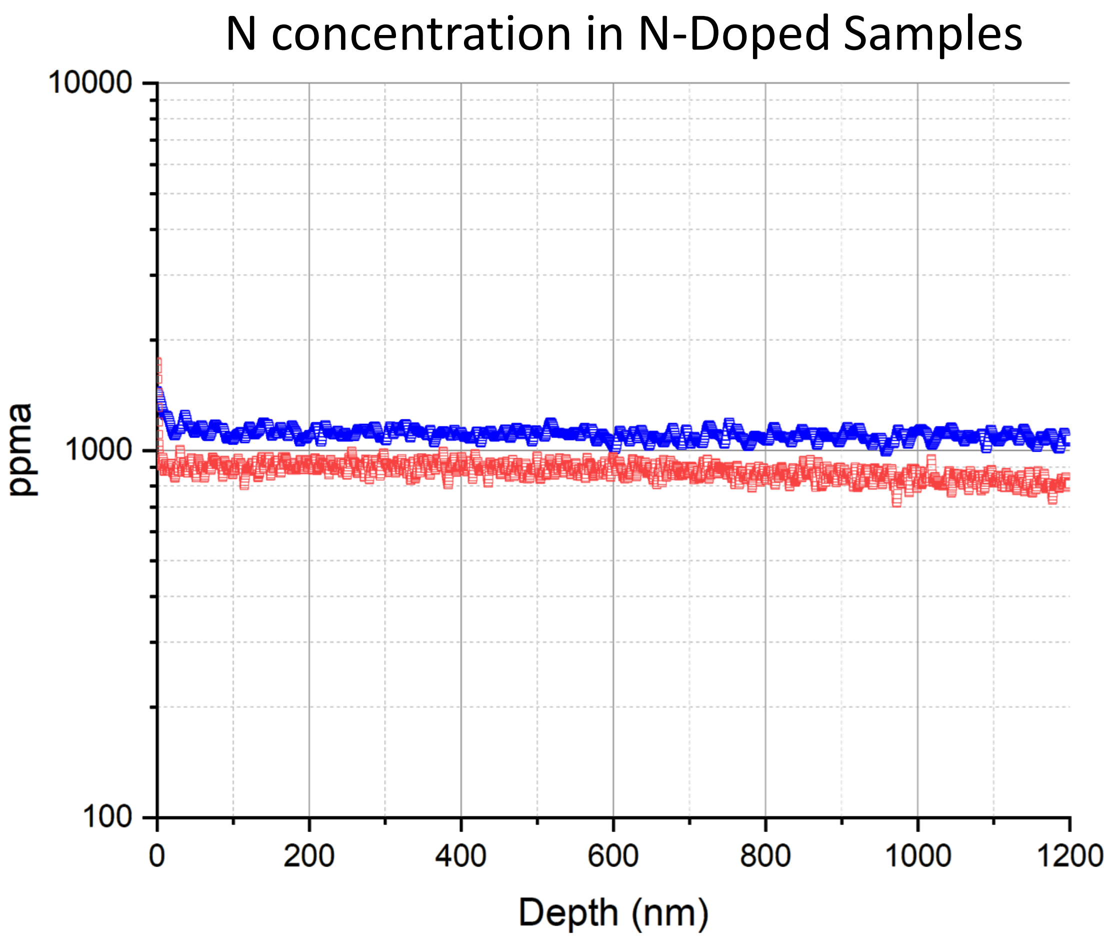




N-Doped Nitrogen Concentration

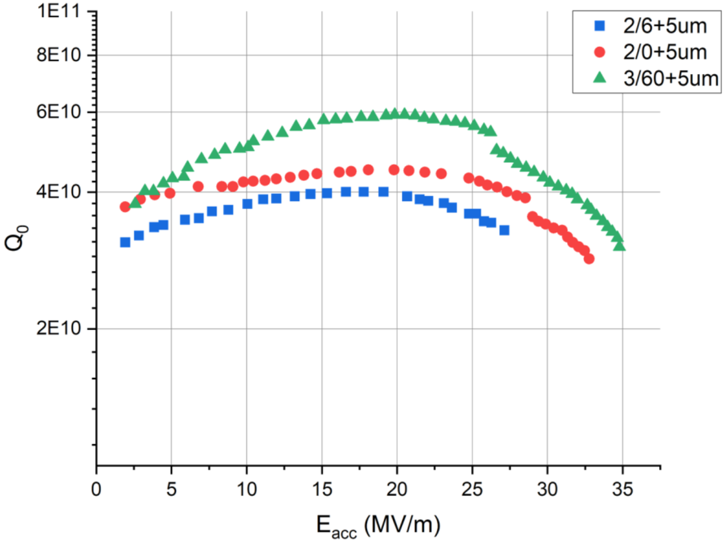
- 2/6+5µm EP
- 2/0+5µm EP

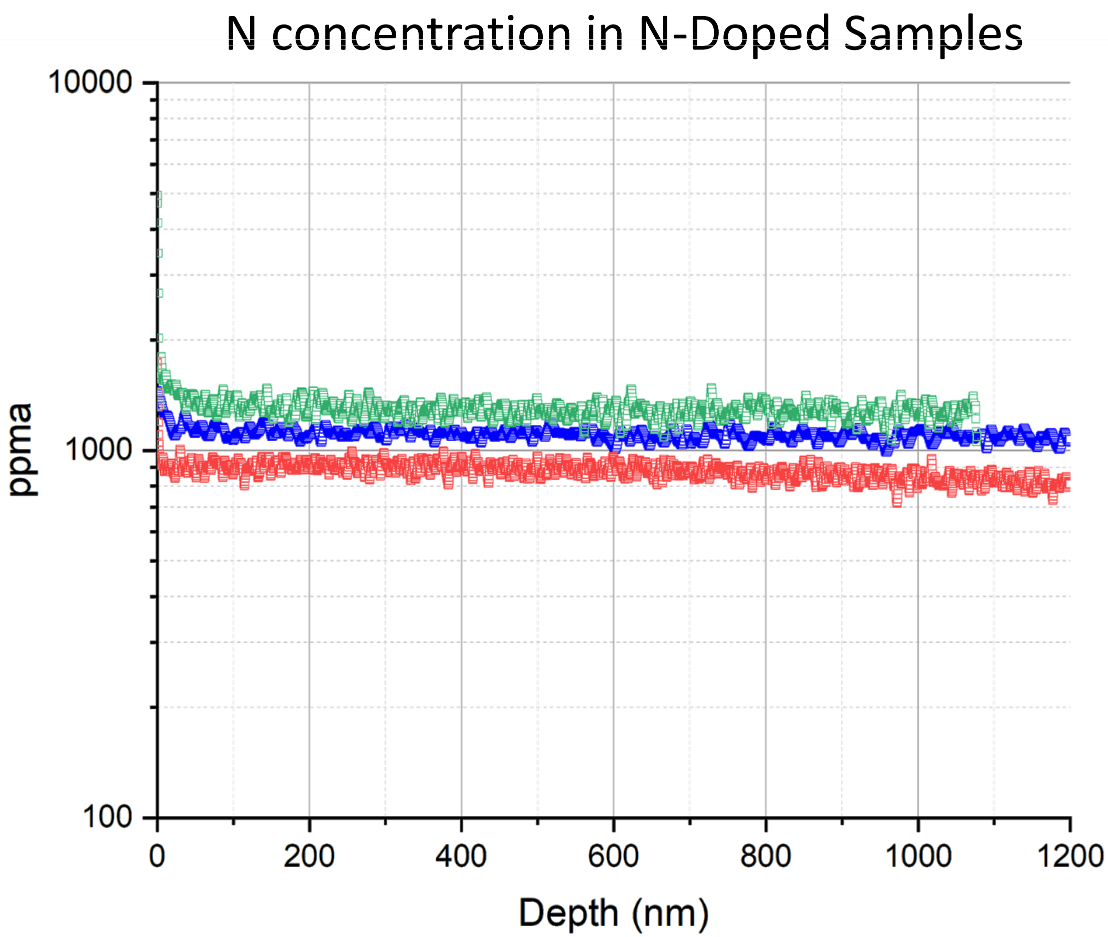




N-Doped Nitrogen Concentration

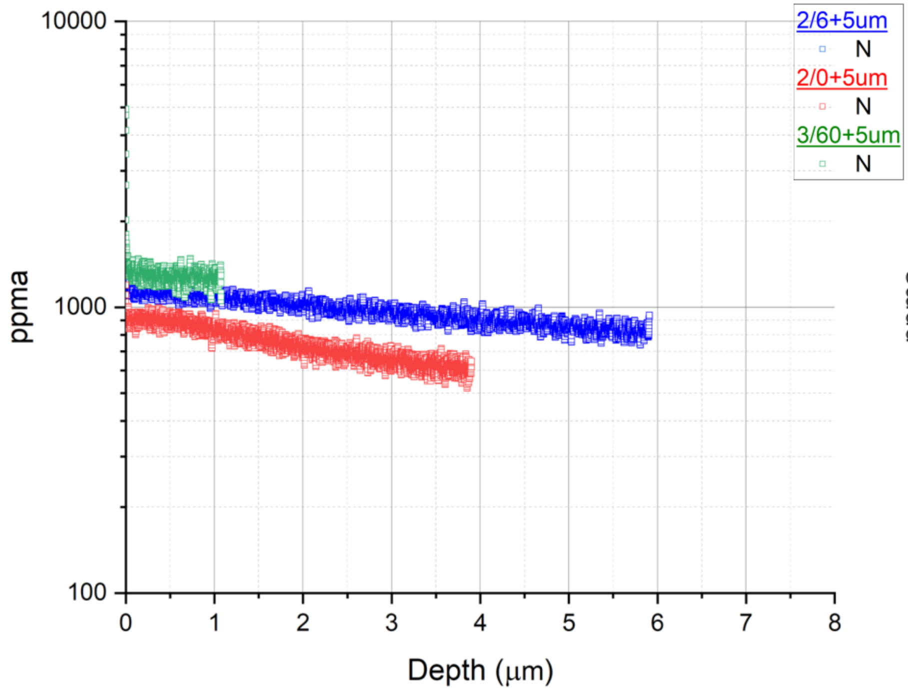
- 2/6+5µm EP
- 2/0+5µm EP
- 3/60+5µm EP



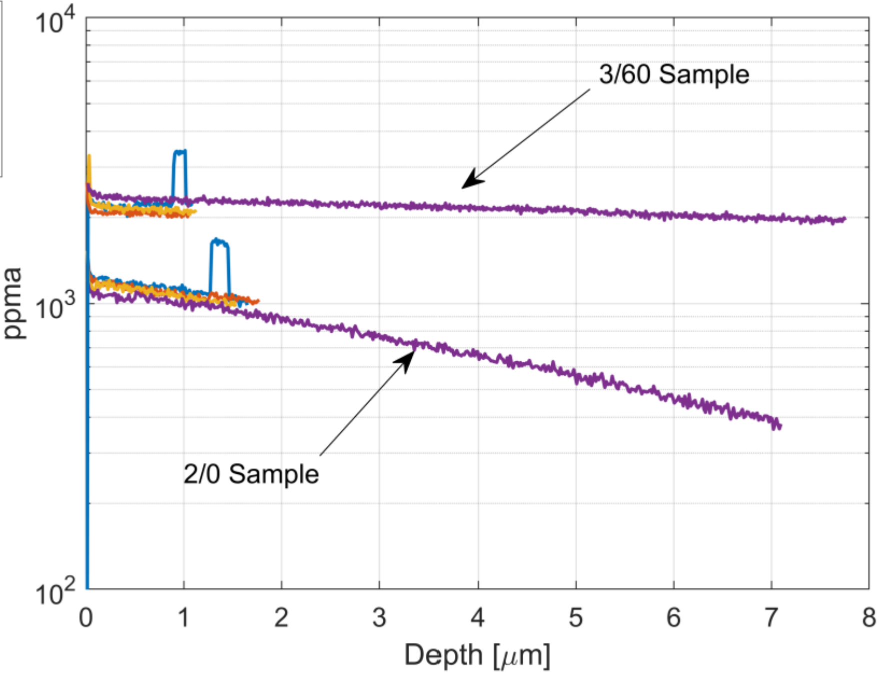


Comparison with Previous Work

N concentration in N-Doped Samples



D. Gonnella, SRF'19 doi:10.18429/JACoW-SRF2019-MOP045

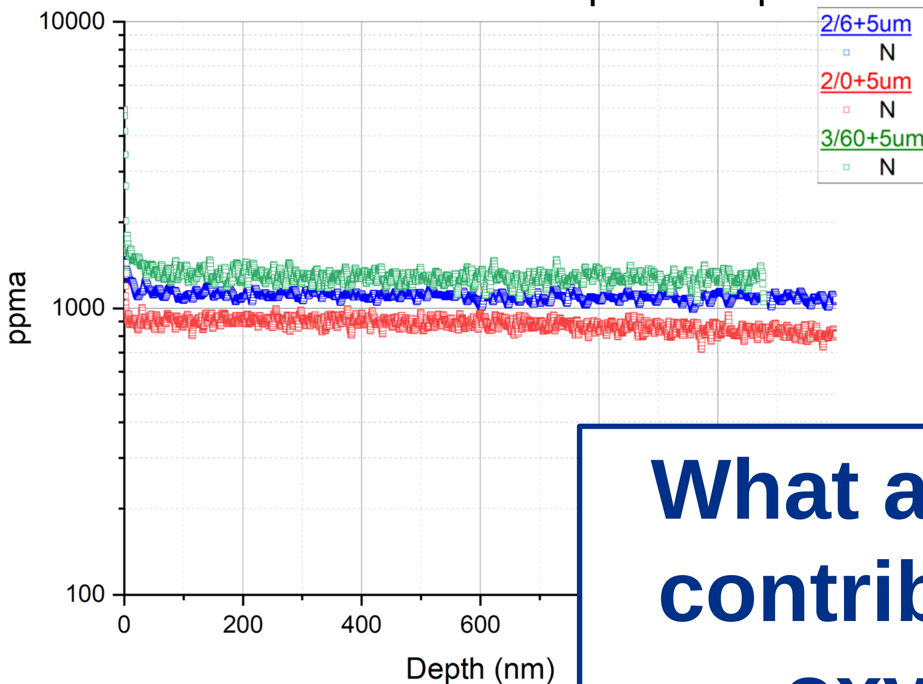


- Not as high 3/60 N concentration as previous work. Why?
- N concentration is inversely proportional to mean free path

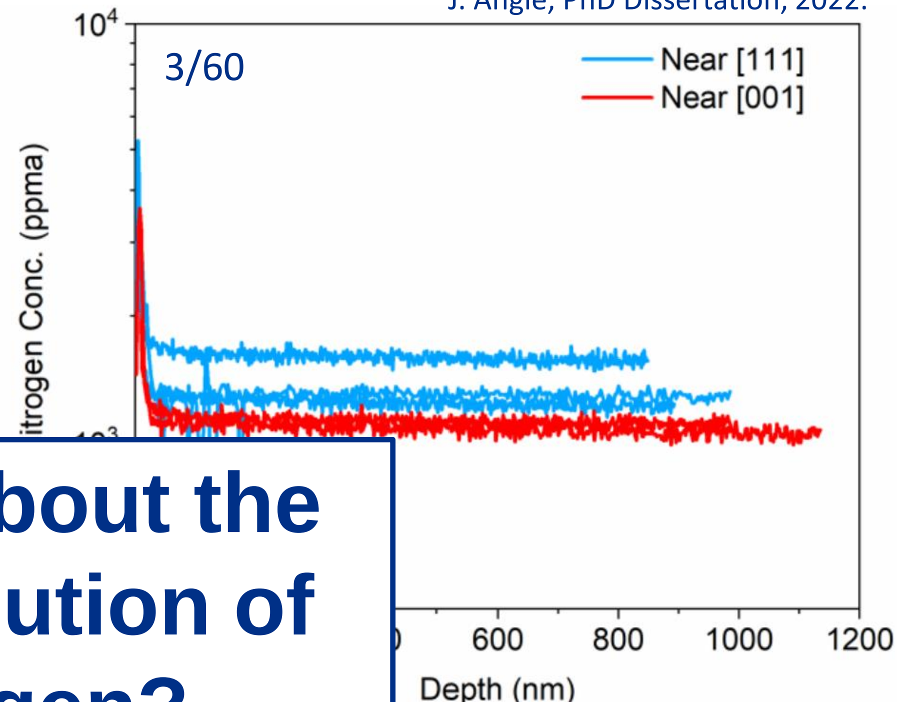
	Avg. N conc. in 1st 100 nm (ppma)	Mean free path from RF (nm)
2/0	900	125 ±37
3/60	1390	94 ± 7

MFP from D. Bafia, PhD Dissertation, IIT, 2020.

Comparison with Previous Work

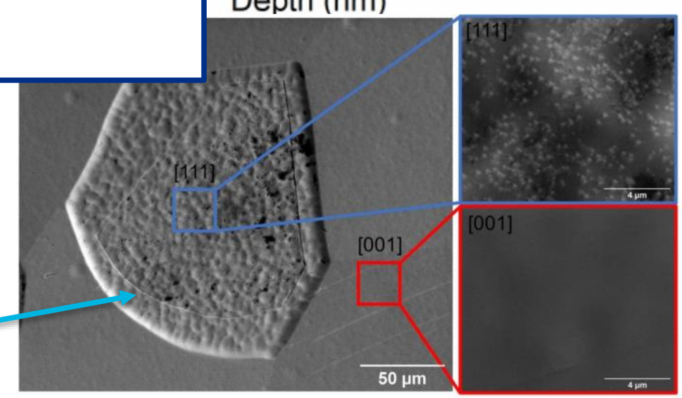


J. Angle, PhD Dissertation, 2022.

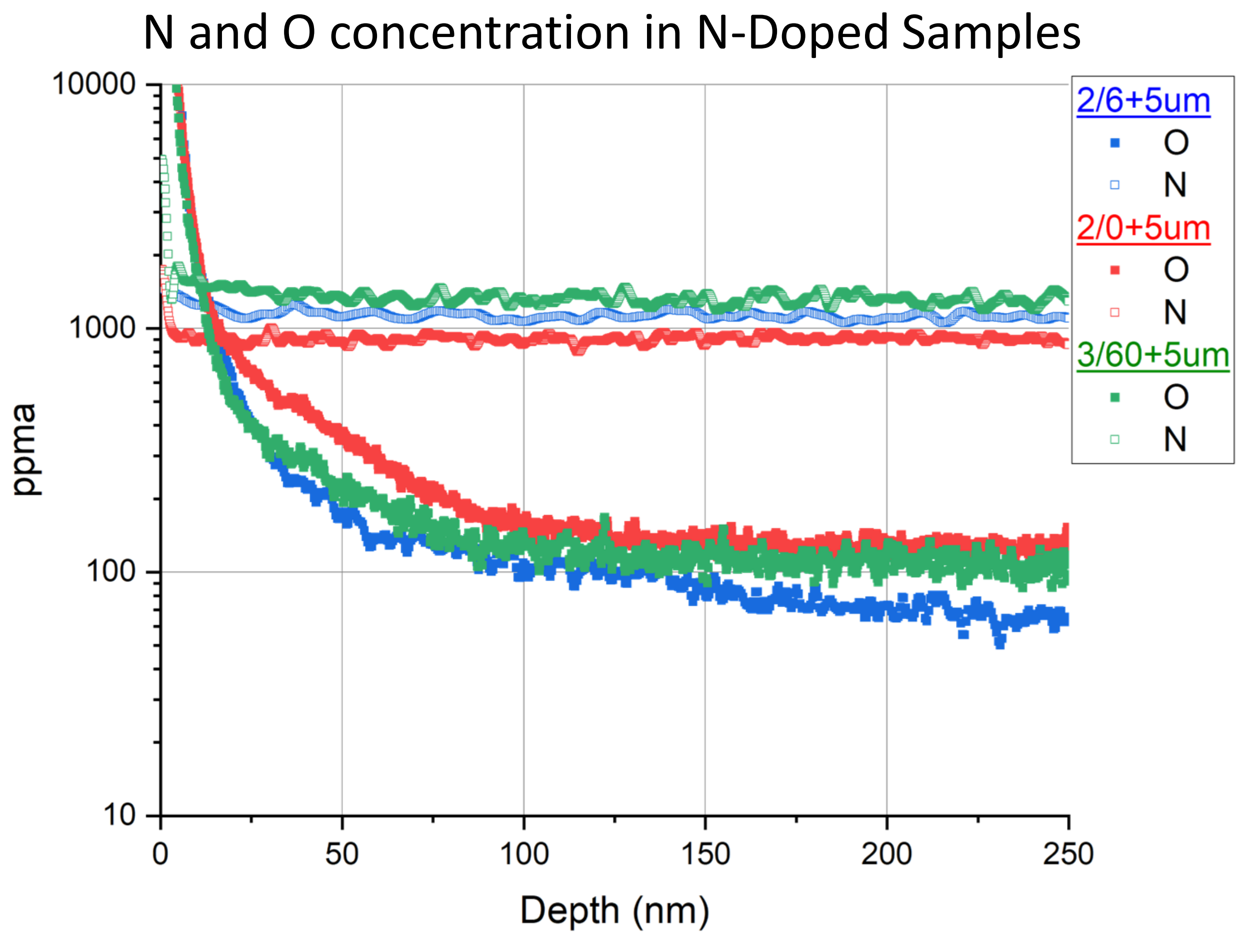


What about the contribution of oxygen?

- Not as high 3/60 N concentration as previous work. Why?
- Differences could be attributed to grain variation
- Nano-nitrides not observed



Oxygen in N-Doped Samples



- Much lower concentration of oxygen, perhaps O is playing a lesser role
- How does this oxygen compare to other cavity treatments?

Comparison with other treatments: Cavity Cutouts

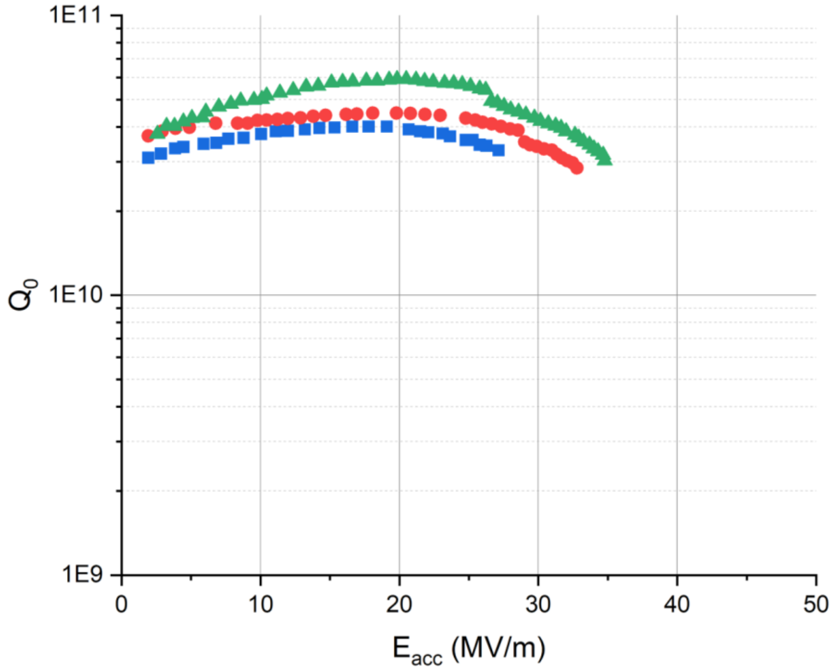
EP	Baseline		
75/120°C modified LTB	Baseline	75°Cx4hr *in-situ* bake	120°Cx48hr *in-situ* bake
O Doping	Baseline	205°Cx19hr *in-situ* bake	

Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS)

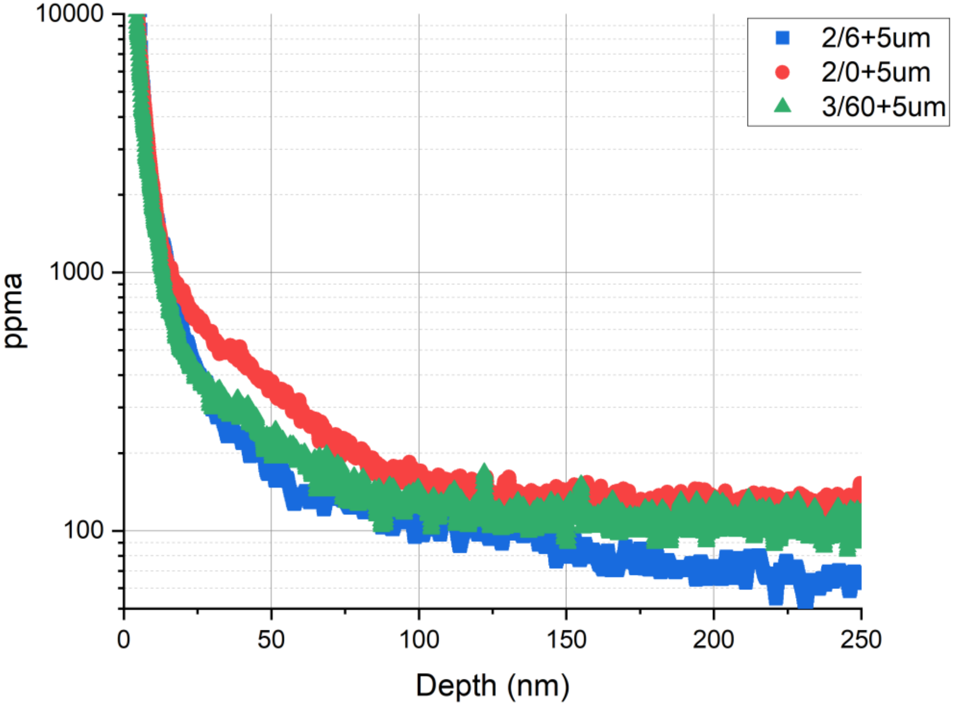
- Comparing oxygen concentration profiles of these cavity cutouts of treatments for which it is known that oxygen plays a key role with nitrogen doped samples

Comparing O Profiles: N-doped vs other treatments

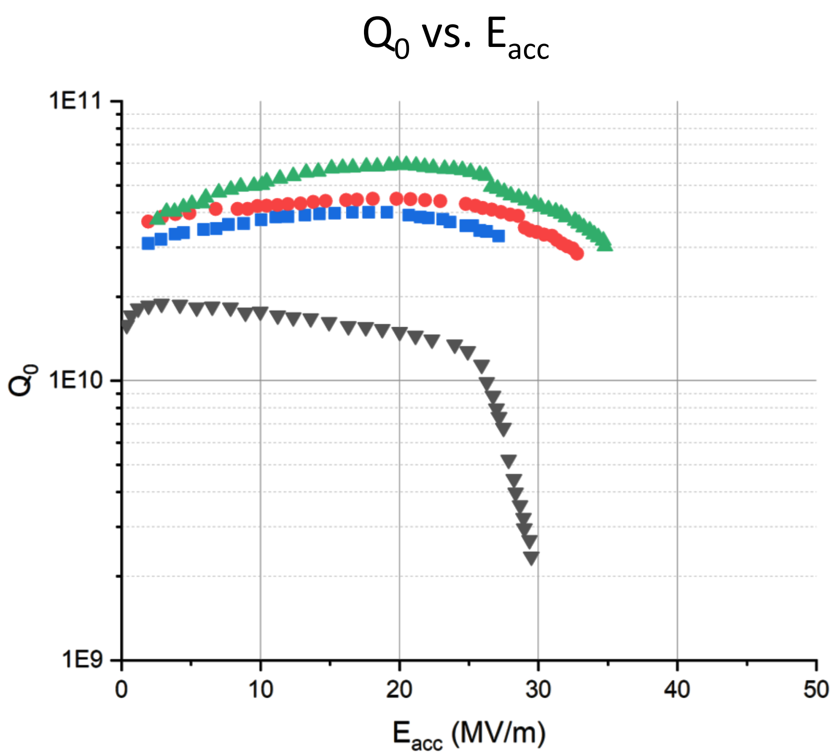
Q_0 vs. E_{acc}

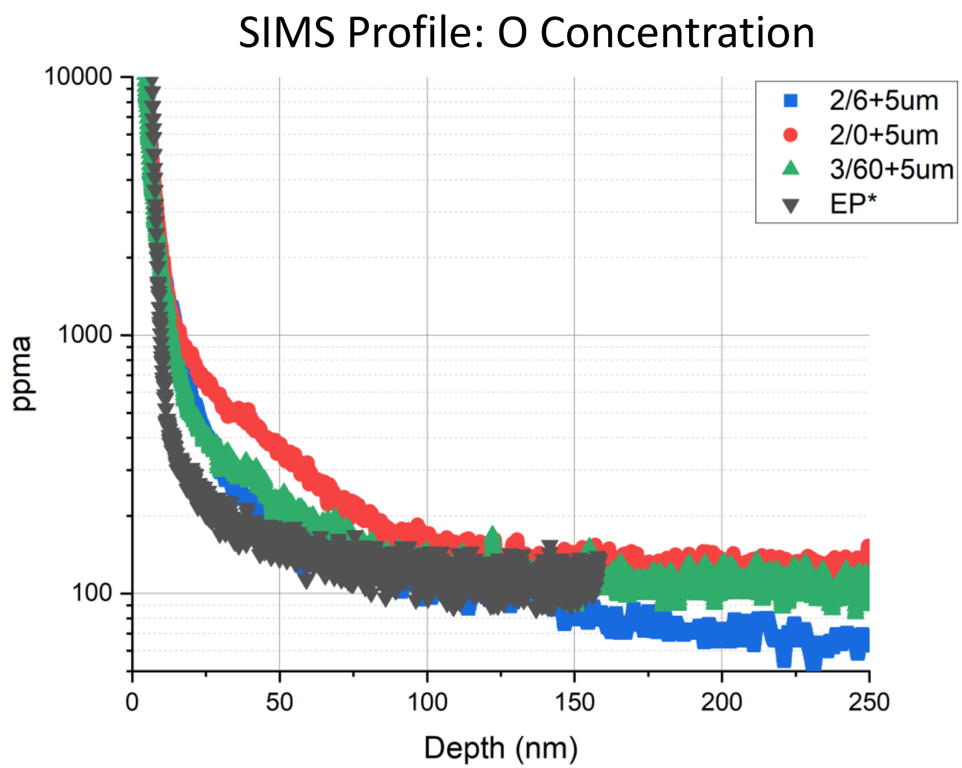


SIMS Profile: O Concentration



Comparing O Profiles: N-doped vs other treatments

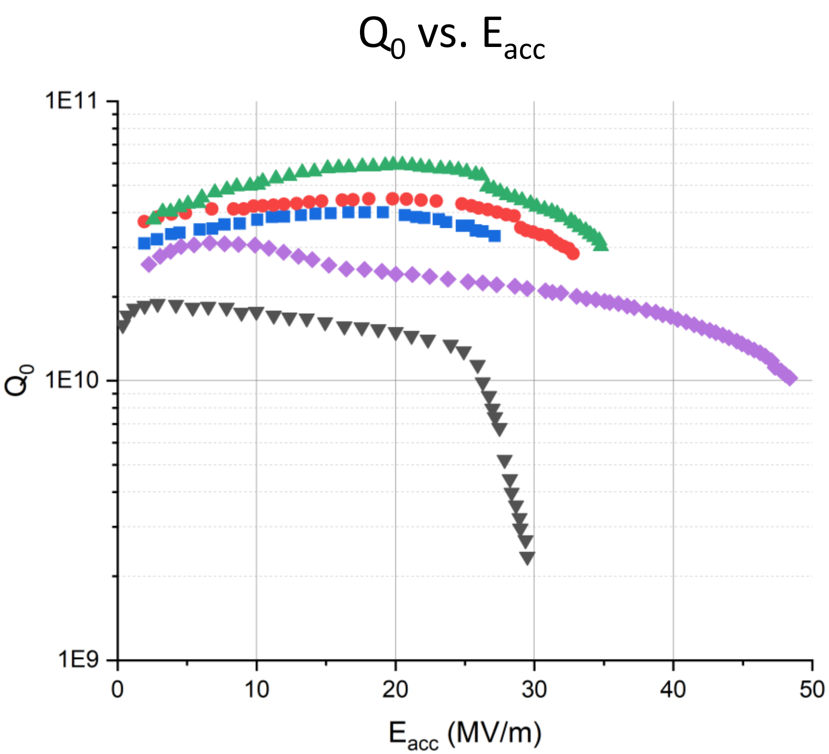


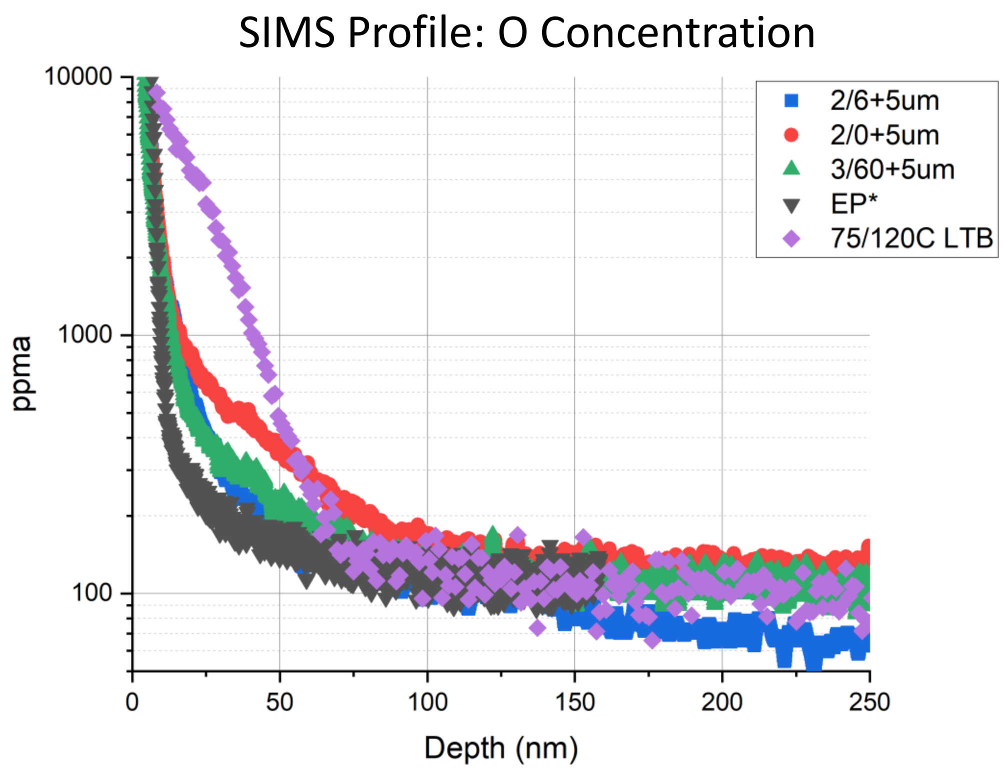


- EP: HFQS from breakdown of niobium hydrides from lack of interstitial impurities as confirmed with SIMS
- N-doped: Comparable but slightly elevated O concentration. Negligible effect?

*EP absolute concentration was not directly measured. Data is scaled relative to 75/120C modified LTB absolute concentration. Will verify will future measurements

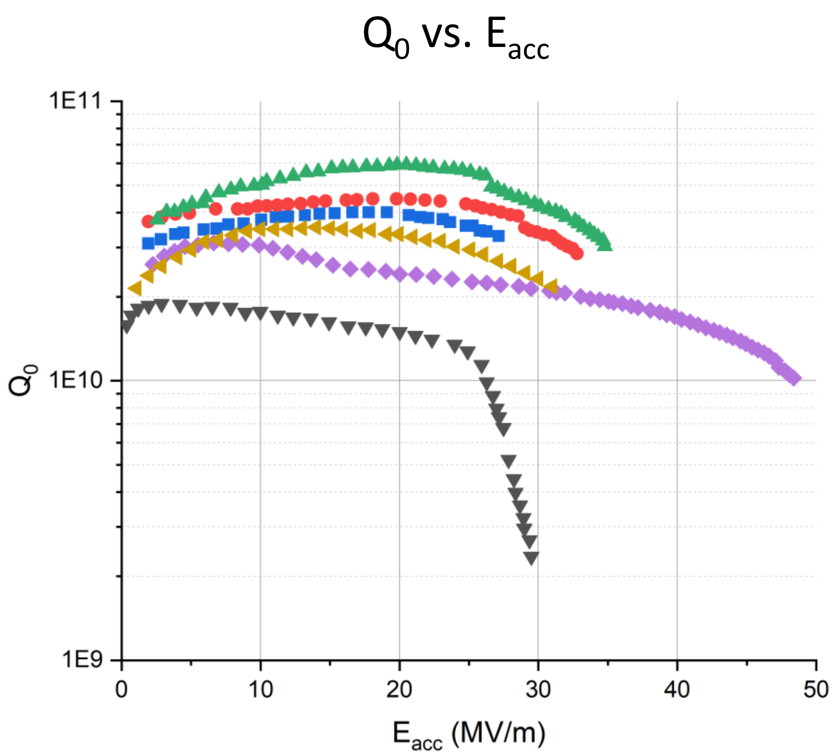
Comparing O Profiles: N-doped vs other treatments

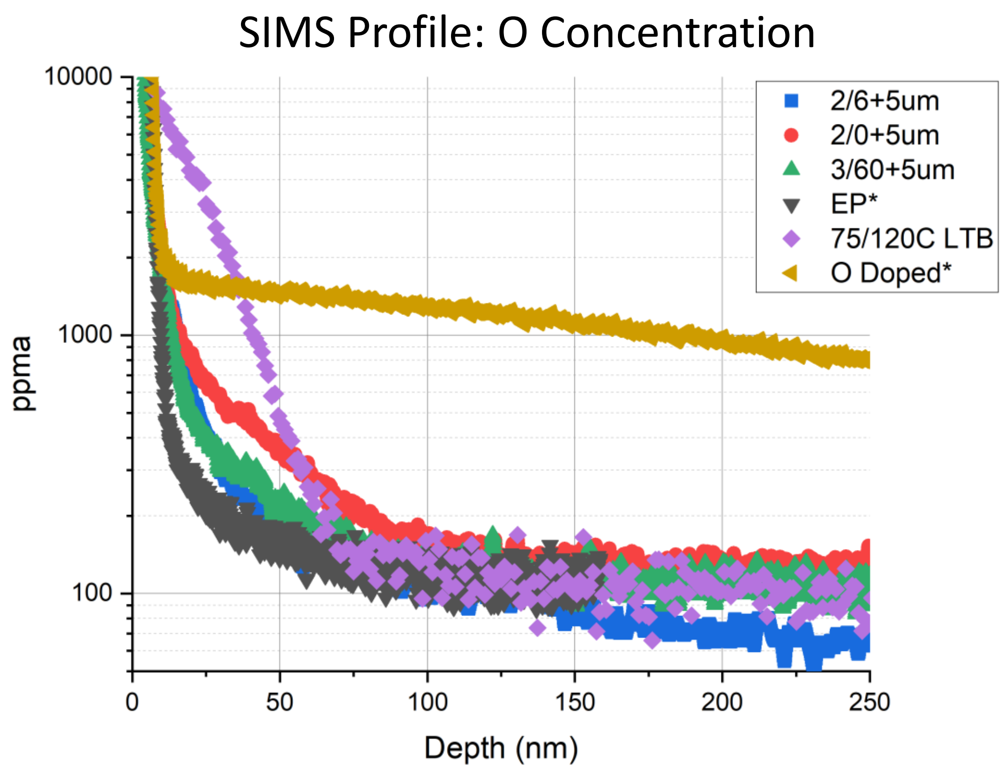




- 75/120C LTB: significant improvement in RF performance from roughly 2 – 3 times more oxygen in the first 100 nm of the surface than N-doped
- Dirty surface extends high gradient RF performance

Comparing O Profiles: N-doped vs other treatments

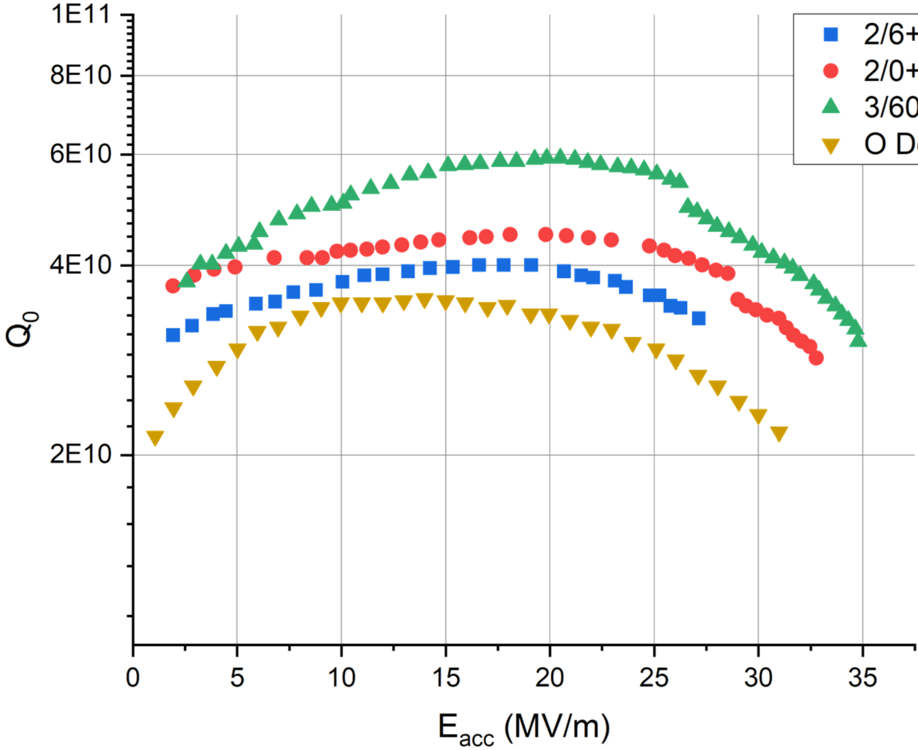


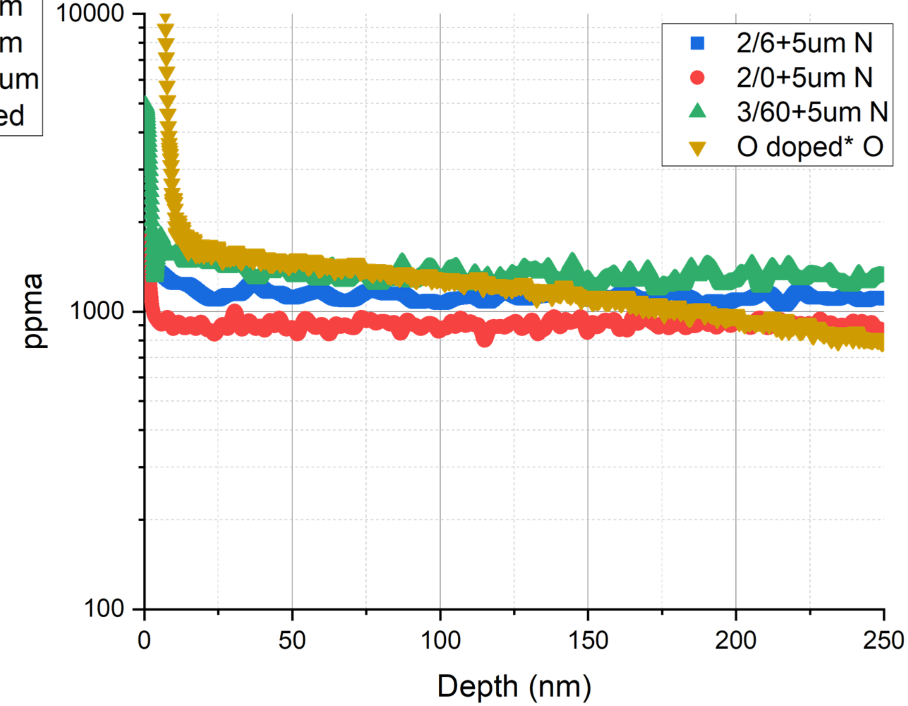


- O doped: Lower concentration at surface, but more uniform
- Uniform concentration gives anti-Q slope and high Q_0, similar to the effect of N in N-doped

*Absolute concentration was not directly measured. Data is scaled relative to 75/120C modified LTB absolute concentration.

Comparing O doped and N doped





- Similar RF cavity performance and impurity profile
- If we slightly increase O conc., will that increase Q_0 like with 2/x ⟶ 3/60 for N-doped?
- Since O-doped cavity is slightly lower in Q_0 but with similar concentration, does that mean O is slightly less effective than N at trapping H?
  - DFT study by D. Ford (2013) showed that the binding energy of -0.06 eV for H bonding with Nb-O instead of with Nb compared to -0.10 eV for Nb-N

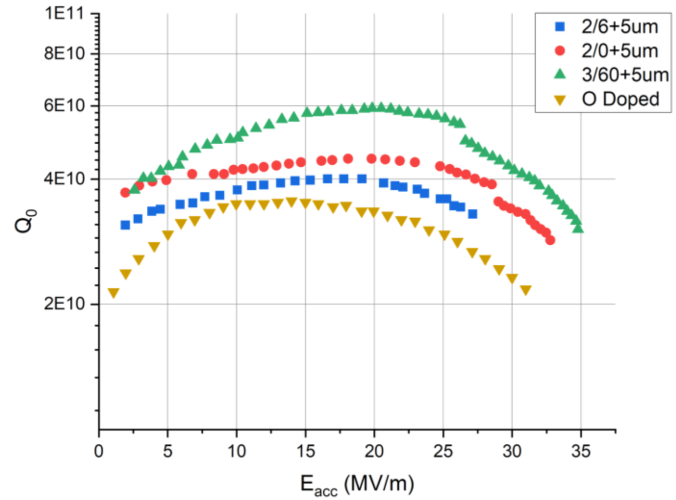
D. C. Ford *et al.*, doi:10.1088/09-2048/26/10/105003

Next Steps

- Get absolute concentration for EP, O doped treatments
- Alternative absolute concentration measurement techniques: Atom Probe Tomography (APT) at Northwestern University
- DFT calculations comparing O and N and their interactions with hydrogen (binding energies, bond lengths, lattice strain energy, etc.)
  - Extension of the work by D. Ford (2013), which focused on the interaction of oxygen and hydrogen, to nitrogen and hydrogen interaction as well
  - Looking for collaborators and hoping to discuss more on this topic

Conclusion

- Small differences in N concentration may be responsible for noticeable differences in N-doped RF performance
- SIMS results suggests minimal role of O in N-doped samples

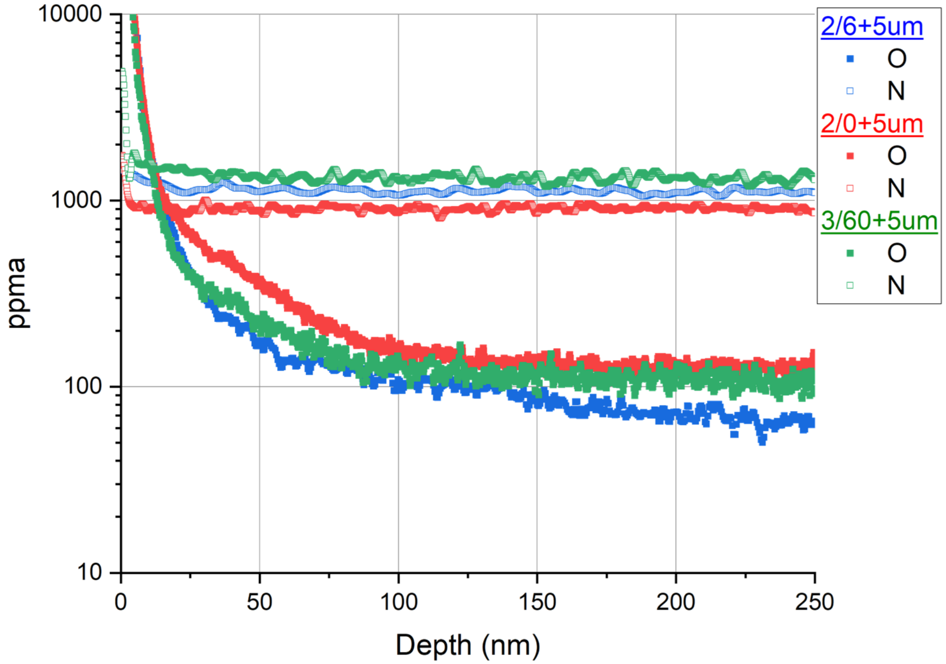


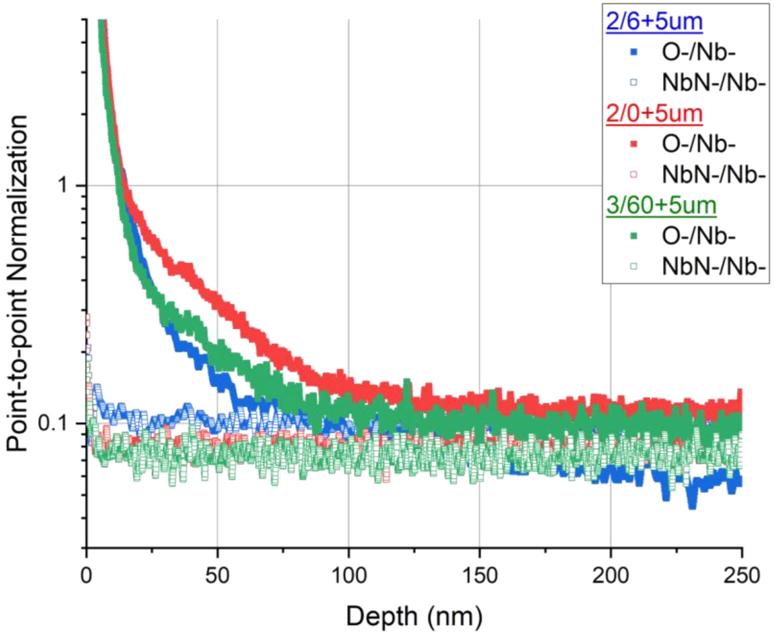
- N in N-doped and O in O-doped drive the same key performance features in RF results
- Roughly equivalent O in O-doped and N in N-doped absolute concentrations (to be verified) yielding different RF performance

1. To what degree do minor differences in impurity concentration drive differences in RF performance?
2. Is oxygen less effective than nitrogen at trapping hydrogen? → DFT studies required

Backup Slides

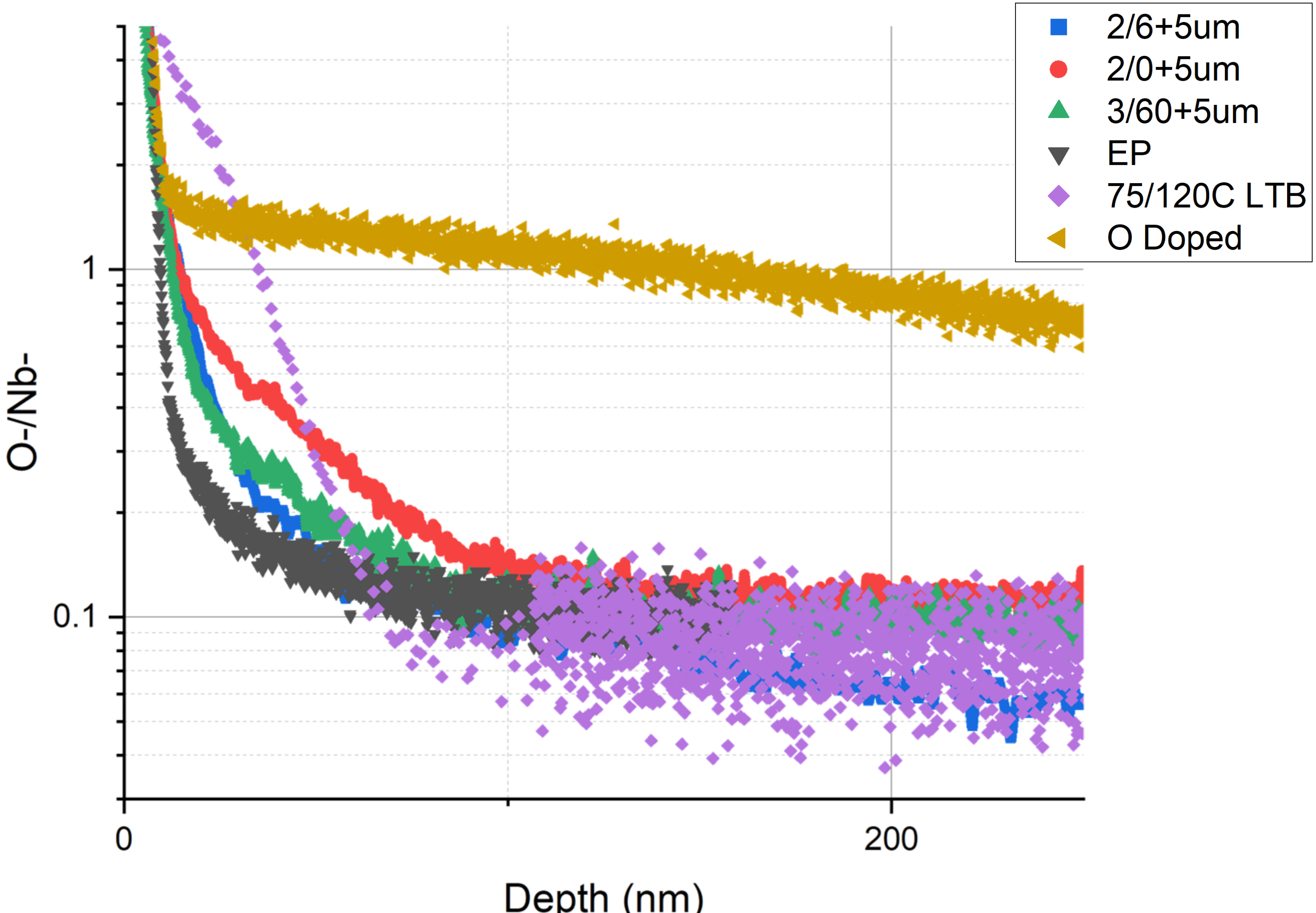
Note on absolute vs relative concentrations





- When normalized to Nb, N and O appears to be similar, but actually much more N in absolute concentration

Point-to-point normalizations



NbH-/Nb-

- NbH- as a measure of free hydrogen, lower NbH- indicates more trapped H