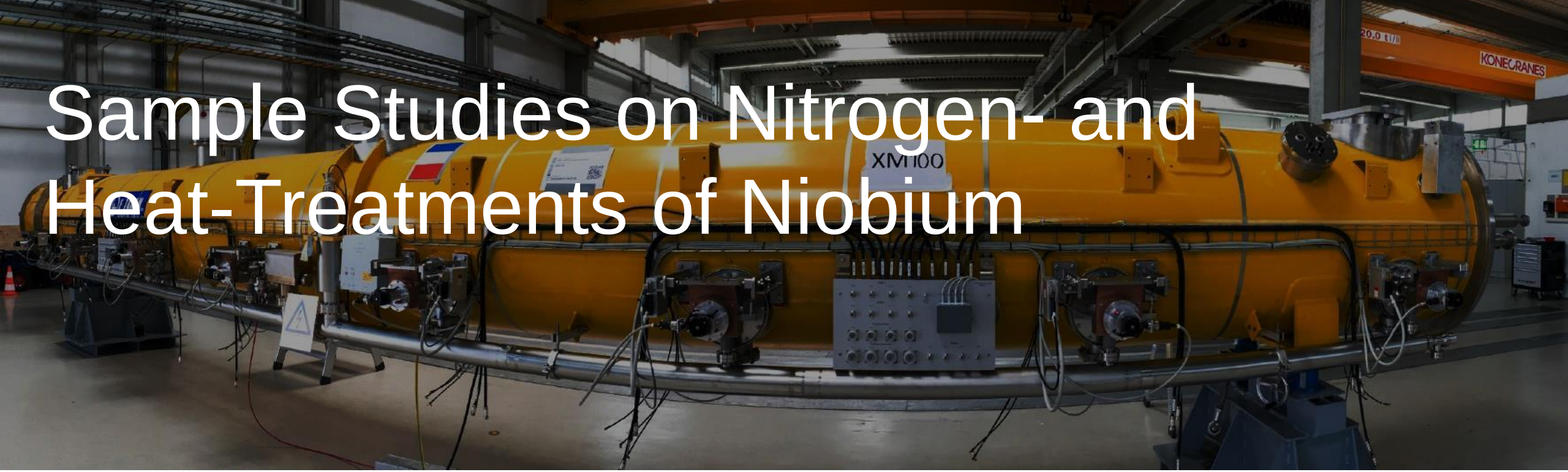


Sample Studies on Nitrogen- and Heat-Treatments of Niobium

M. Wenskat
on behalf of the SRF R&D Team

TTC Collaboration Meeting – February 2020 @ CERN

Agenda:

- Cavity Cut-Outs
- FG RRR30 Nb Samples
- Ultrapure Single-Crystal Samples

HELMHOLTZ
RESEARCH FOR GRAND CHALLENGES

GEFÖRDERT VOM
Bundesministerium für Bildung und Forschung

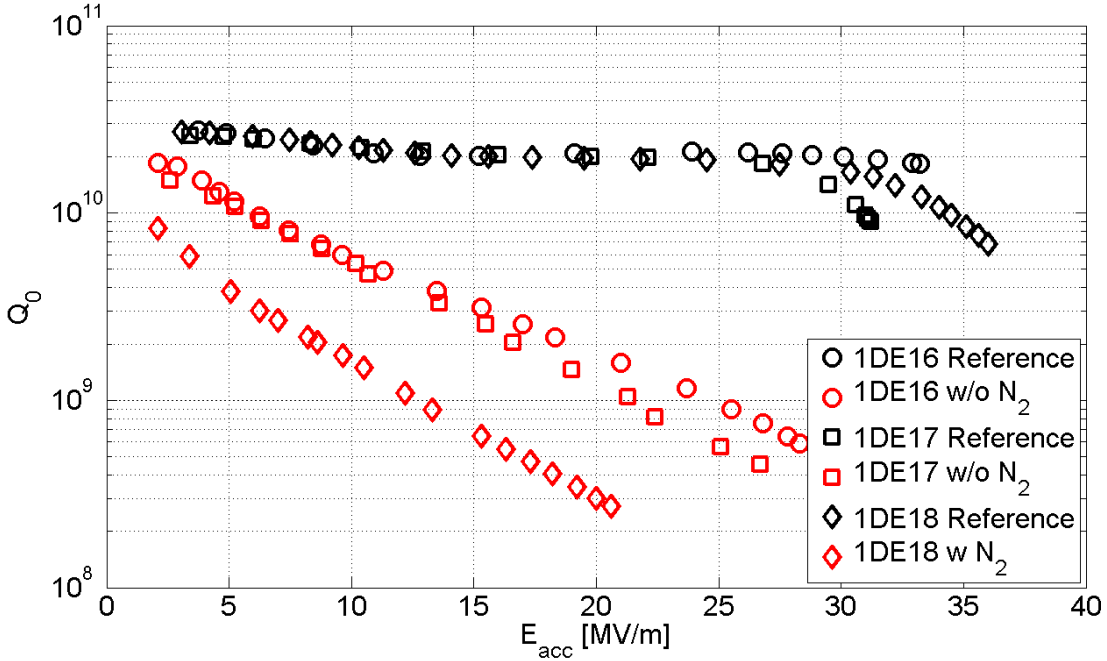
MML
From Matter to Materials and Life

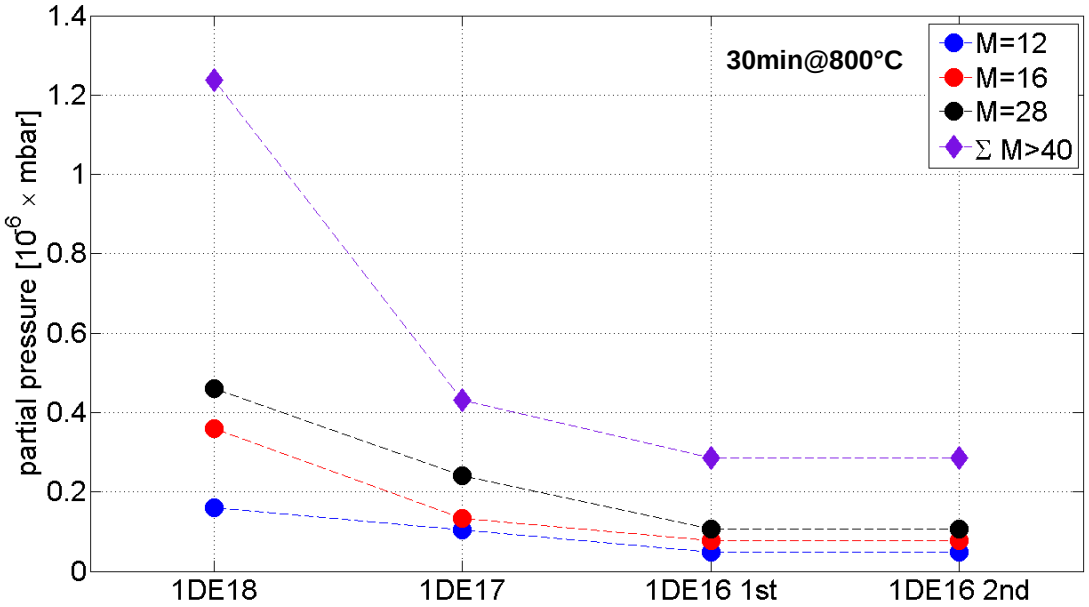
MT ARD
MATTER AND TECHNOLOGIES
ACCELERATOR RESEARCH AND DEVELOPMENT

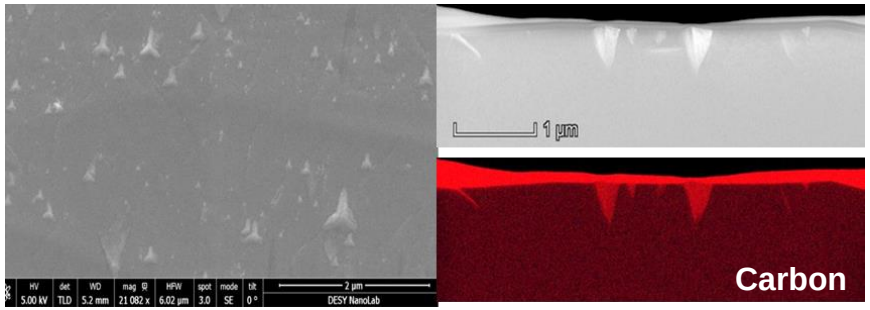
DESY.

Infusion study

„Forensic“ studies





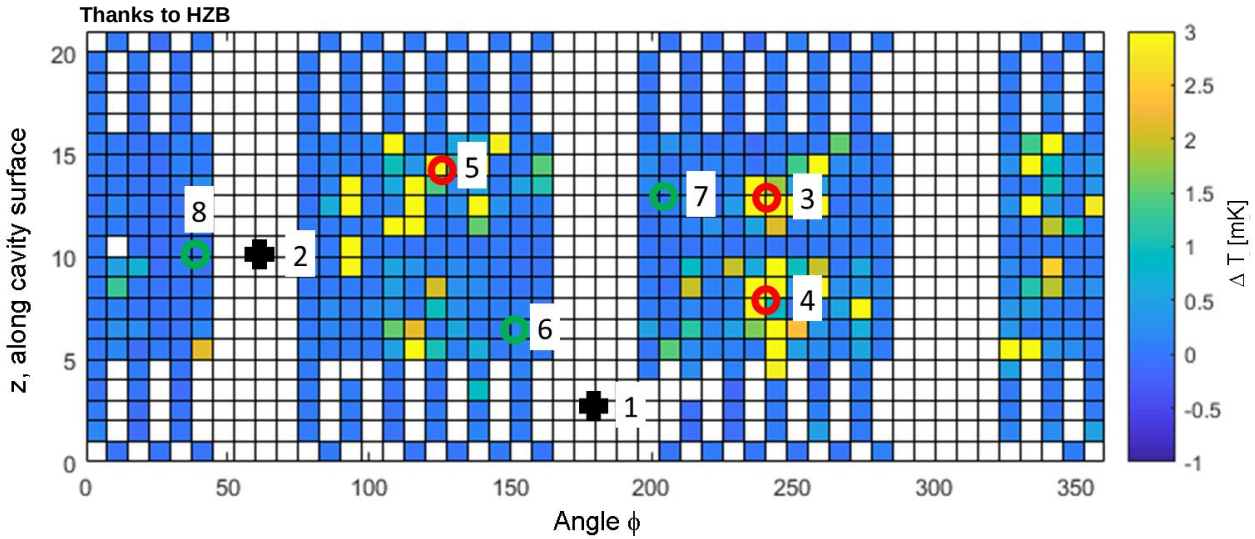


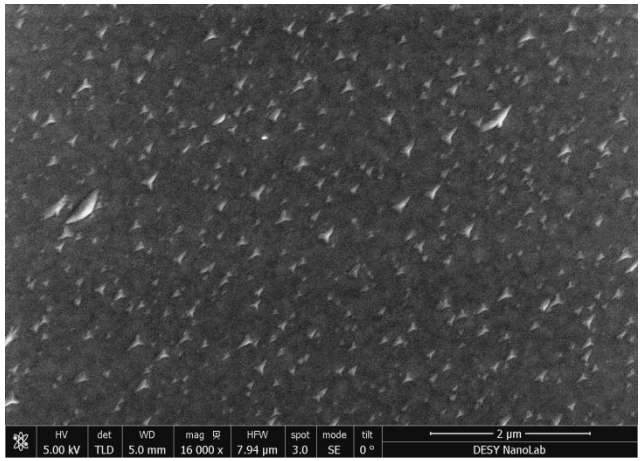
- „Varying“ cavity performance
- Pressure improved over runs but „saturated“ & hydrocarbon contributions
- Nb_2C seen on all samples inside Nb box
- Regardless of rf performance
- Sample studies showed that material purity influence carbide formation

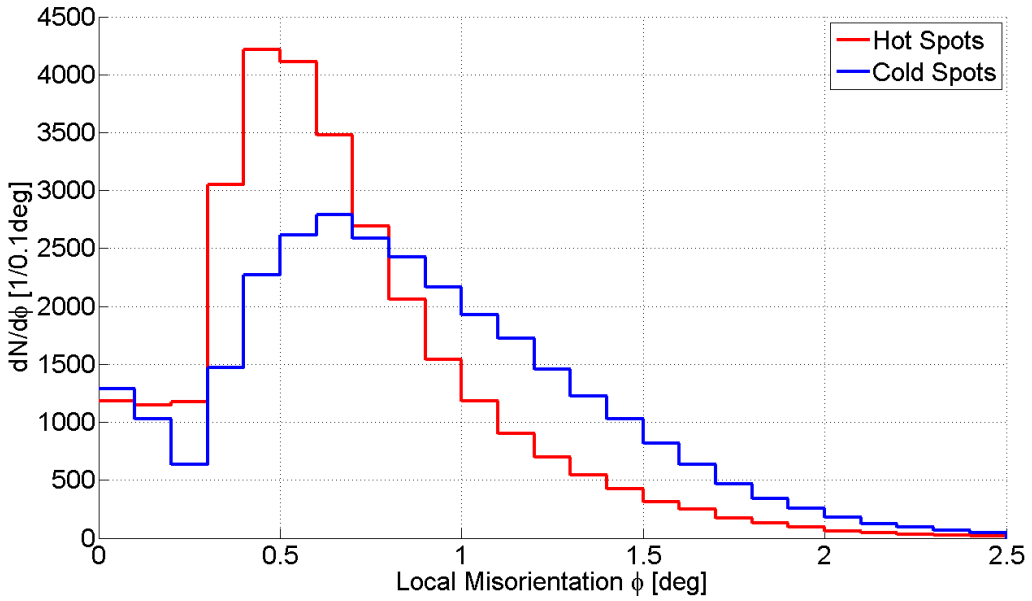
Are carbides the origin of the deterioration?
Do they form inside a cavity at all?

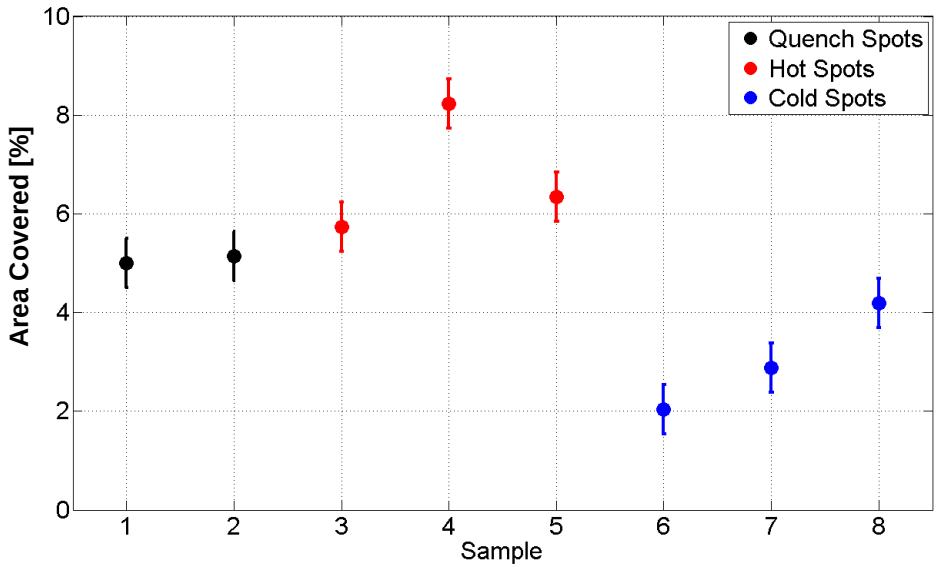
Do Niobium-Carbides form inside the Cavity?

Cut a cavity and look

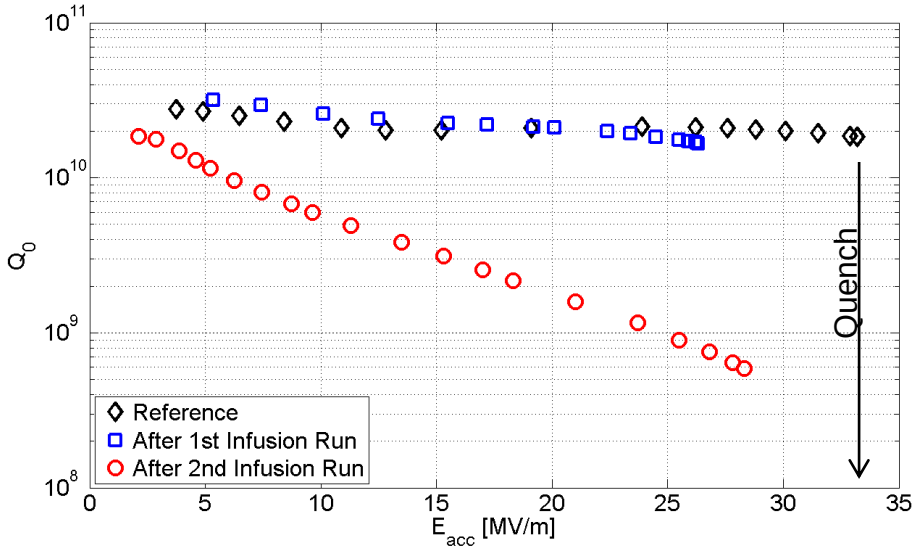


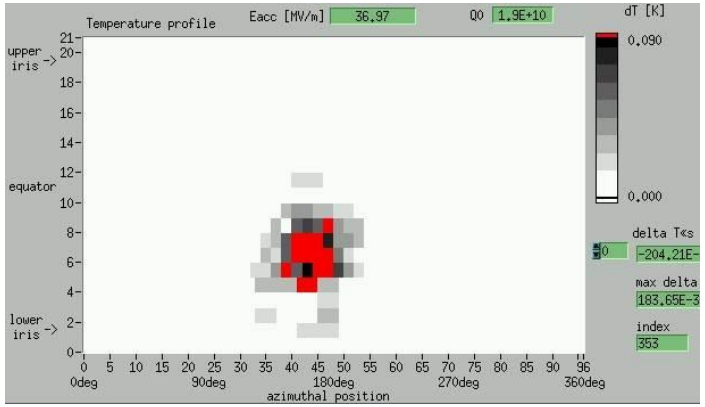


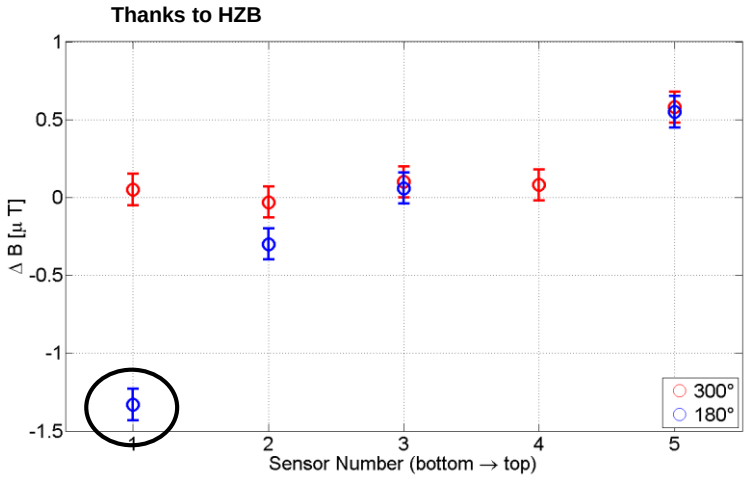


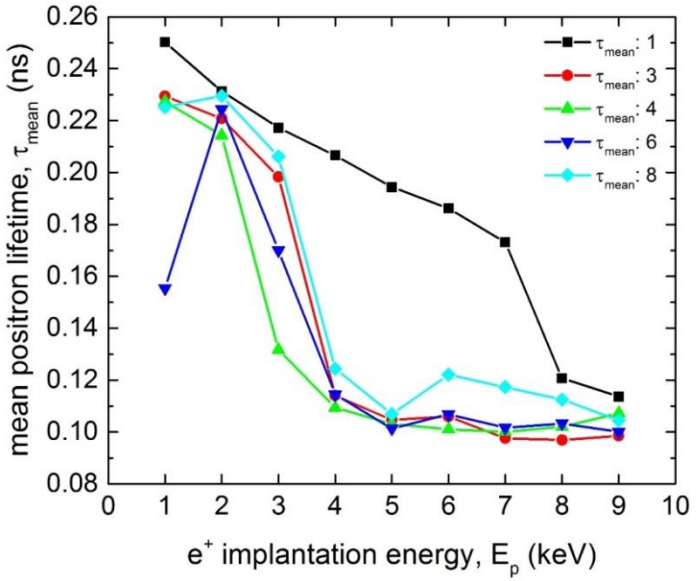


Vacancy-Clusters and Flux-Trapping









- Cavity quenched before 1st infusion run
- H-Map shows significant amount of flux-trapping (1.4 µT)) at quench spot cut-out
- Optical inspection showed no features

- High density of near-surface vacancy-clusters in the first 150nm observed in quench spot cut-out

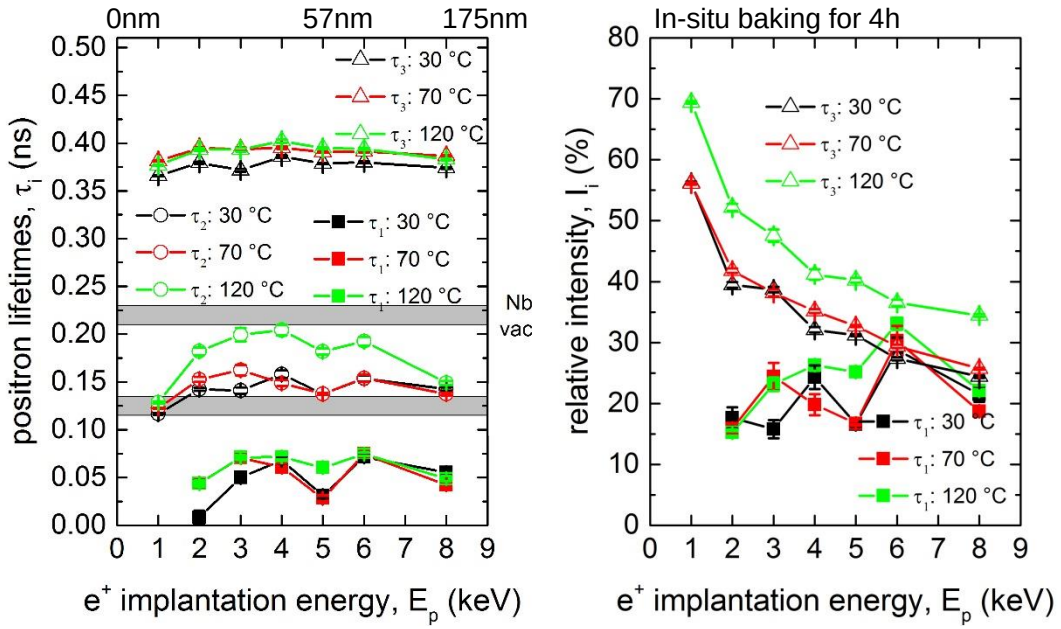
- Flux-trapping efficiency of vacancy-clusters known

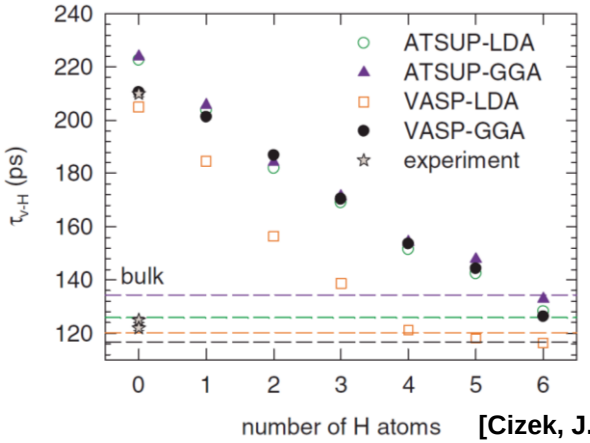
[Antoine, C., Phys Rev AB 22(2019): 034801]

Low Temperature Bakings

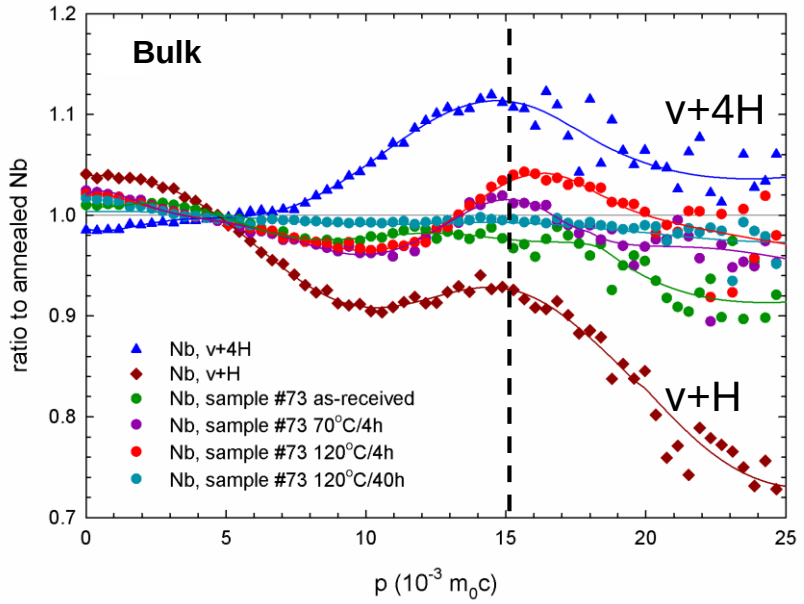
Hydrogen-Vacancy Interaction in Niobium

[Wenskat, M., et al. Submitted to Scientific Reports]





[Cizek, J., et al. Phys Rev B 79 (2009): 054108]

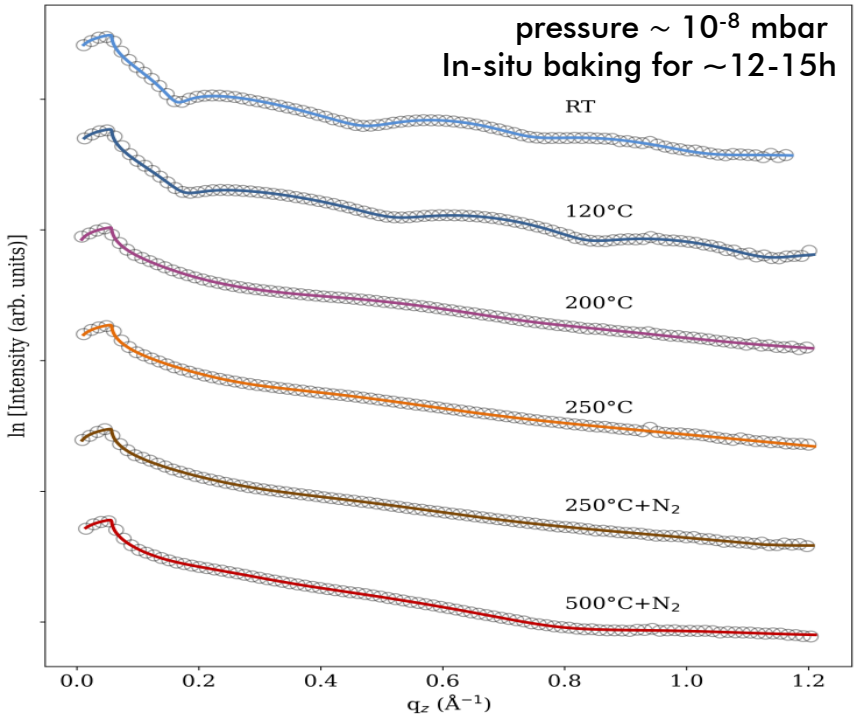


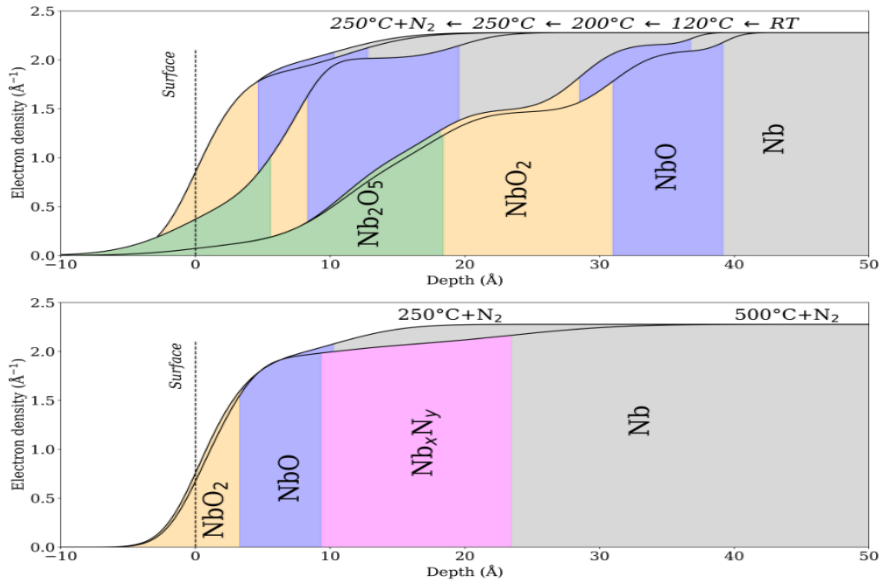
- Clear indication of dissociation of v+nH complexes during 120°C bake for 48h
- Disagrees with nanohydride model
  [Romanenko, A. et al., Supercond. Sci. Technol. 26(2013): 035003]

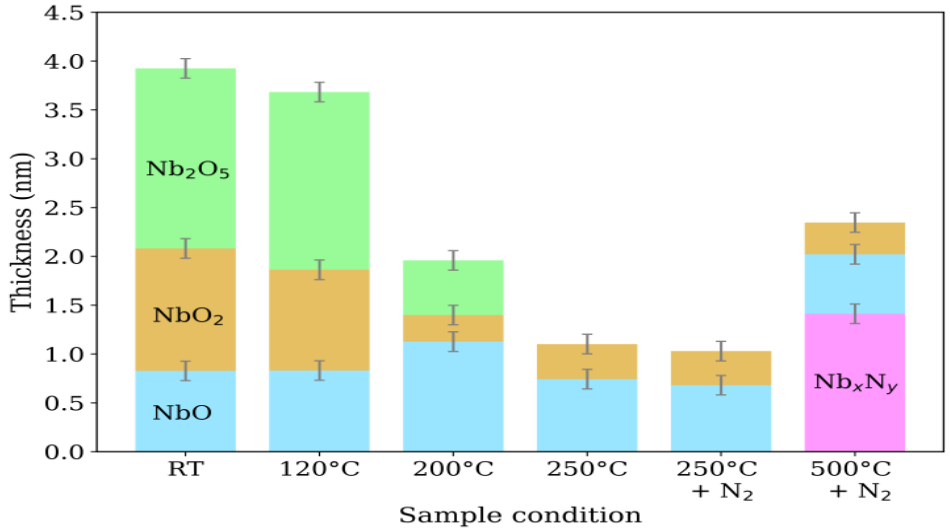
Oxide layer during baking

Combinatorial study with XRR and diffuse X-ray scattering

[Semione, G. D. L., et al. PRAB 22.10 (2019): 103102]





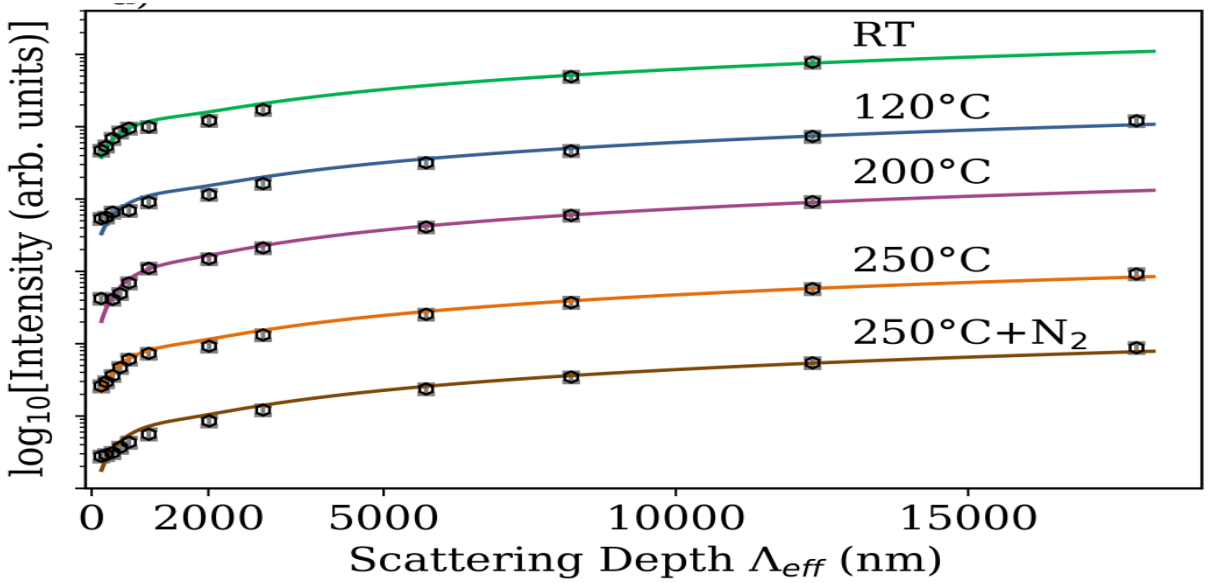


- Initial stage: Nb_2O_5 - NbO_2 - NbO
- Progressive dissolution of Nb_2O_5 and NbO_2
- Nb_xN_y layer detected underneath natural oxides at 500°C + N_2

Oxygen interstitial while baking

Retrieving interstitial concentration profiles by X-ray diffuse scattering

[Semione, G. D. L., et al. PRAB 22.10 (2019): 103102]

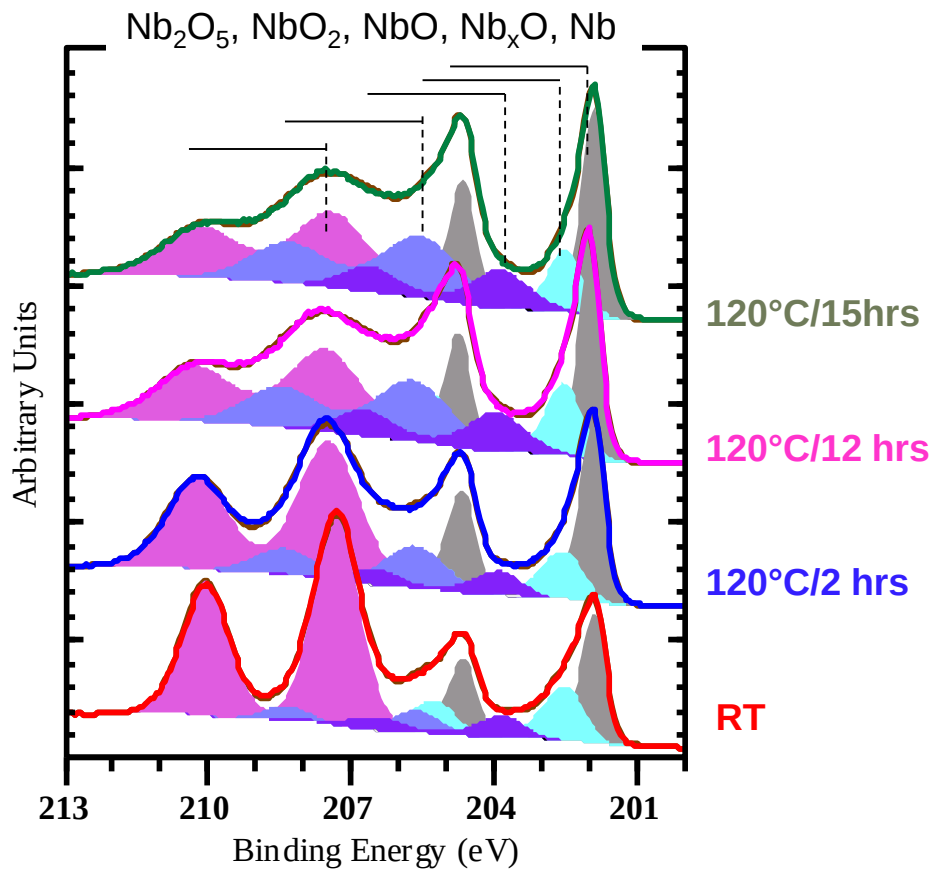


- At 120°C interstitial oxygen is mostly present within the first 10 nm
- Temperature increase leads to further diffusion of oxygen species liberated from the oxide layer
- T- dependency of O-concentration at O-Nb interface agrees with oxygen diffusion model
  [G. Ciovati, Appl.Phys.Lett. 89(2006): 022507]
- Effect of grain-boundaries to be clarified in future experiments

Surface evolution during thermal treatments: *in-situ* XPS

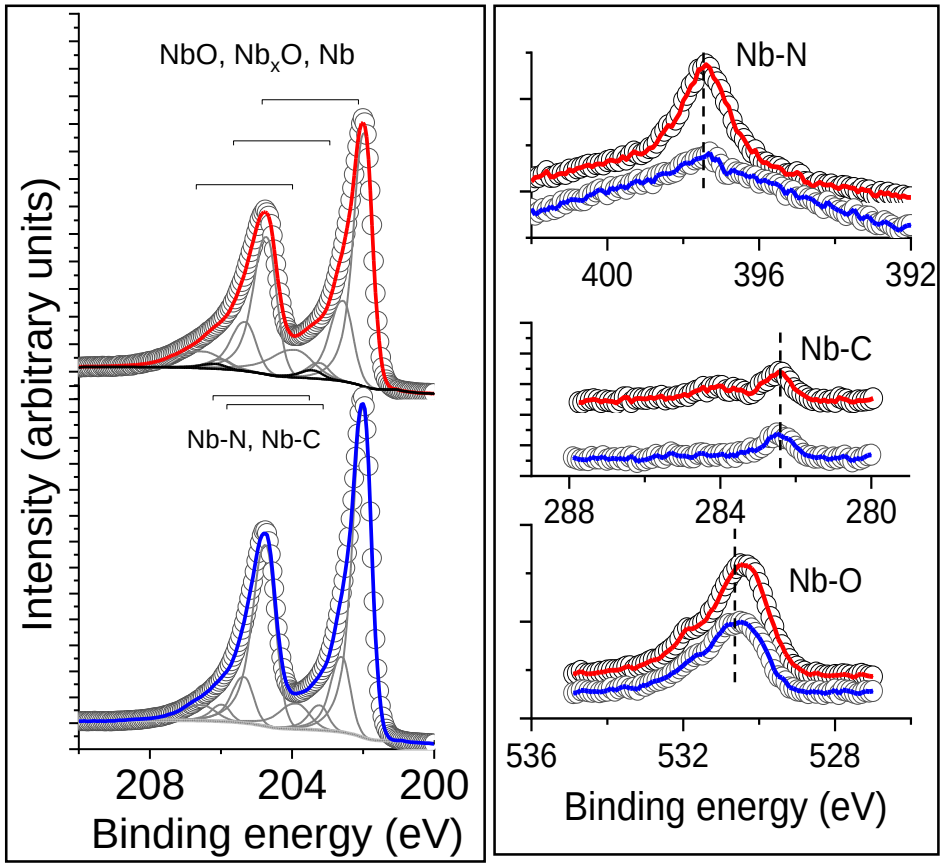
Single crystal Nb (100) model system studied in UHV

I. Bake at 120 °C:



- Stable surface after 12 hours baking,
- Increased thickness of NbO_2 > NbO
- Dissolution of Nb_2O_5

II. N-infusion in UHV: 800 °C + 120°C/12 hrs in UHV (no N_2) + 120 °C in N_2 (0.004 mTorr)/13 hrs



- Nb-N bond formed, slightly thicker NbO and Nb_xO
- Reproduction of results on cavity-grade Nb as well

Summary

- Broad spectrum of sample R&D results
  - Nb_2C are the origin of our failed runs
  - Correlation of intra-grain local misorientation with Nb_2C formation
  - Correlation of higher density of near-surface vacancy-clusters and flux trapping in quench spot
  - Dynamics of vacancy-hydrogen complexes & oxygen diffusion studies during baking procedures
  - A NbN phase forming during 120°C infusion even at lower pressure

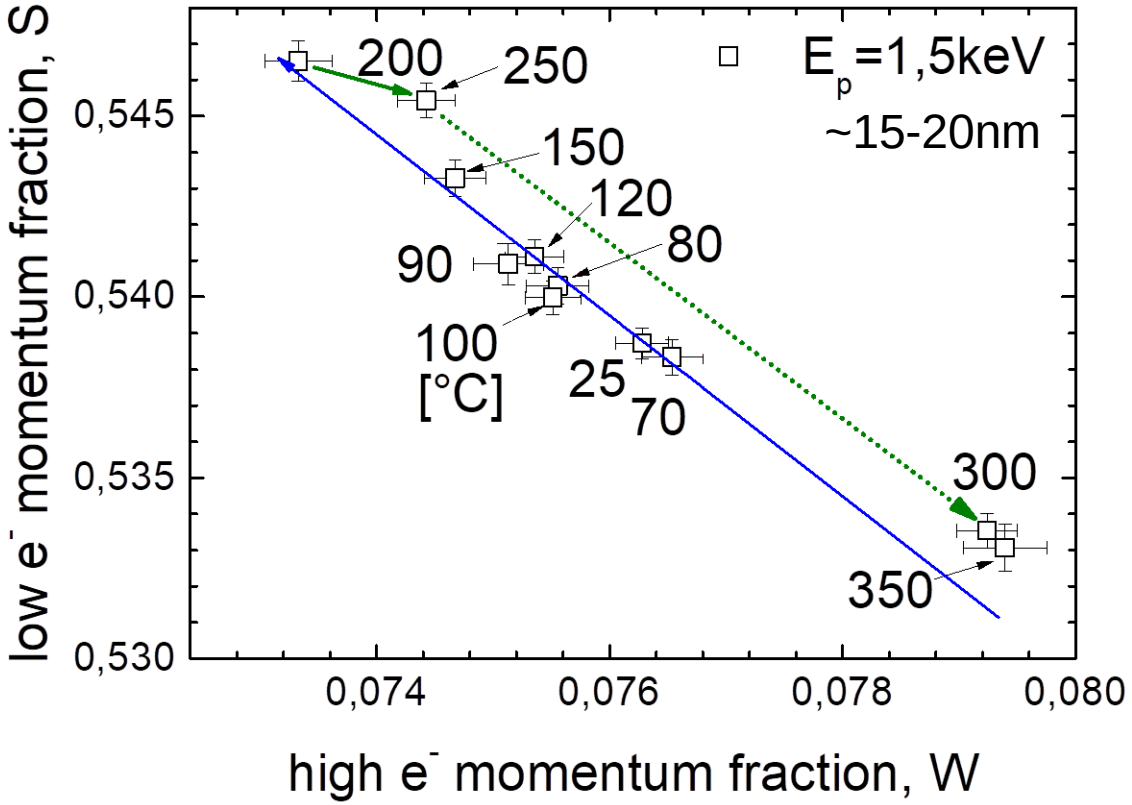
  → All results tend to say: Do not bring H into Nb (e.g. cold EP) but if it is there, try to distribute it controlled (v+nH) or trap it (O, N) to prevent formation of Nb-hydrides

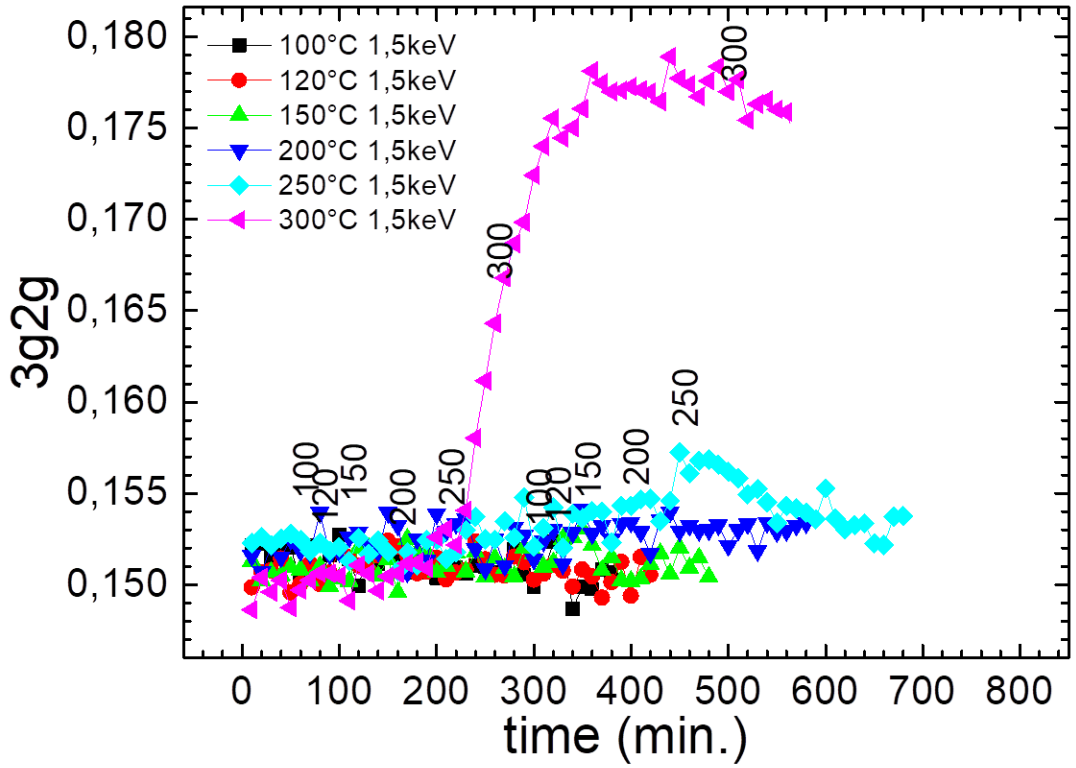
- Furnace upgrade(s) will improve our situation for N-treatment studies of cavities

Mid-T baking

Vacancy-dynamics and interstitial diffusion

Preliminary Results





- Near-surface defects are getting annealed / cluster (or better: process starts) → different S_0

- "Surface cleaning" takes place
  - increased release of elements from vacancies into bulk → higher probability to form para-Ps which decays into 3 γ