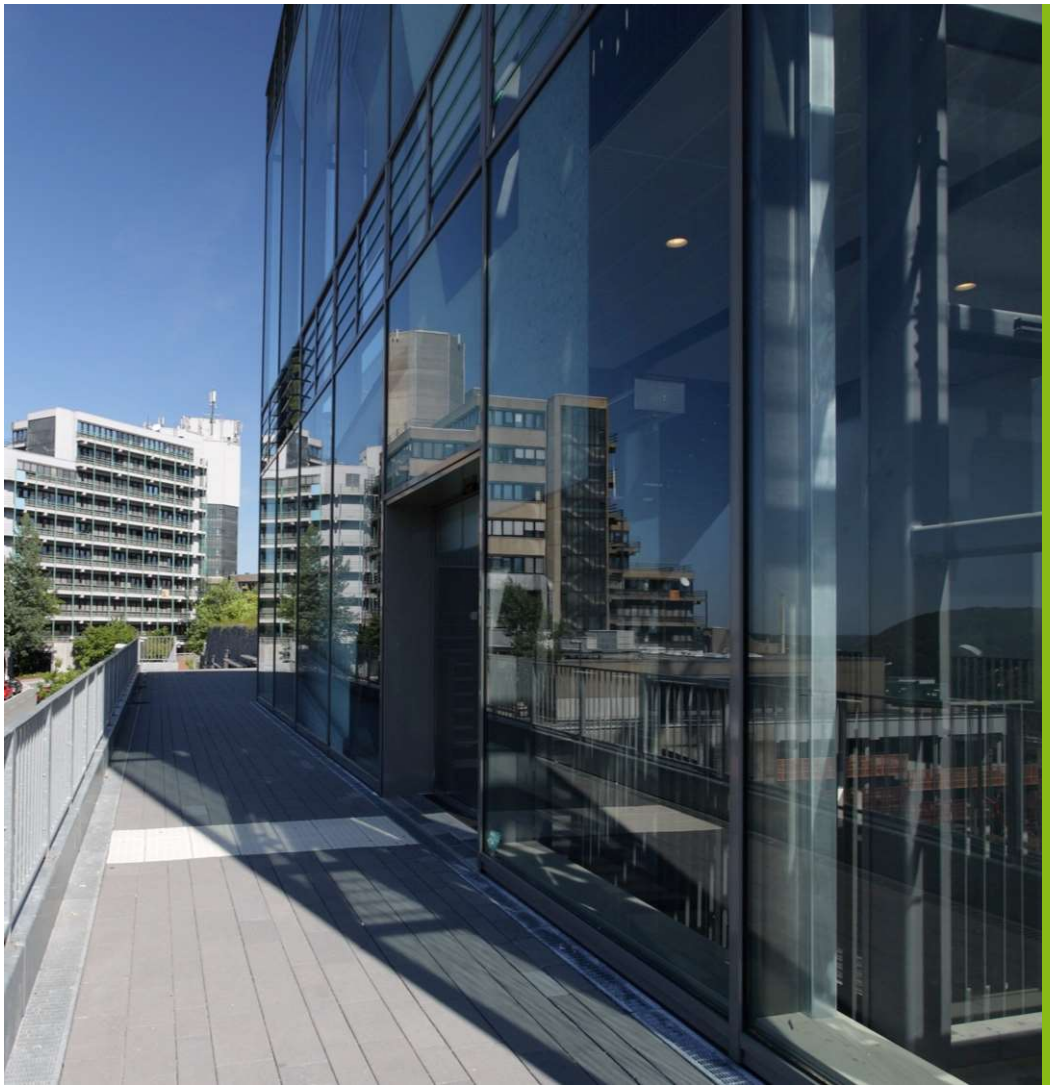

University of Wuppertal

In-Situ EXAFS Investigation of N_2 Treatment of Nb

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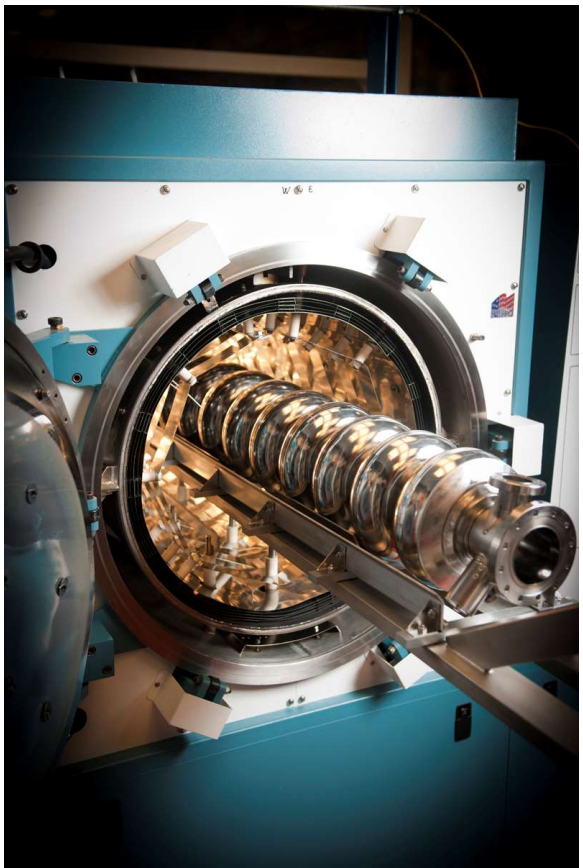
Motivation

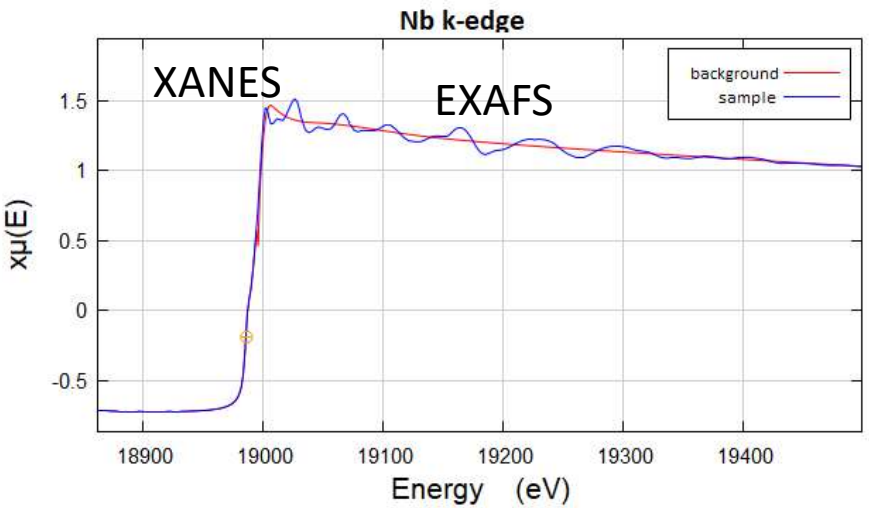


*Fermilab today

- Nowadays most Nb-cavities are treated with N_2 to improve Q-factor
  - "nitrogen-doping"
- Effects still not well understood: especially position of "doped" nitrogen in the Nb-lattice unclear
- Very low doses of N_2
  - Too low for e.g. XRD

$\Rightarrow$ in-situ measurements during doping procedure

X-Ray Absorption Spectroscopy



XANES = **X**-ray **A**bsorption **N**ear-**E**dge **S**pectroscopy
EXAFS = **E**xtended **X**-ray **A**bsorption **F**ine-**S**tructure

Element Specific: Absorption edges are element-specific

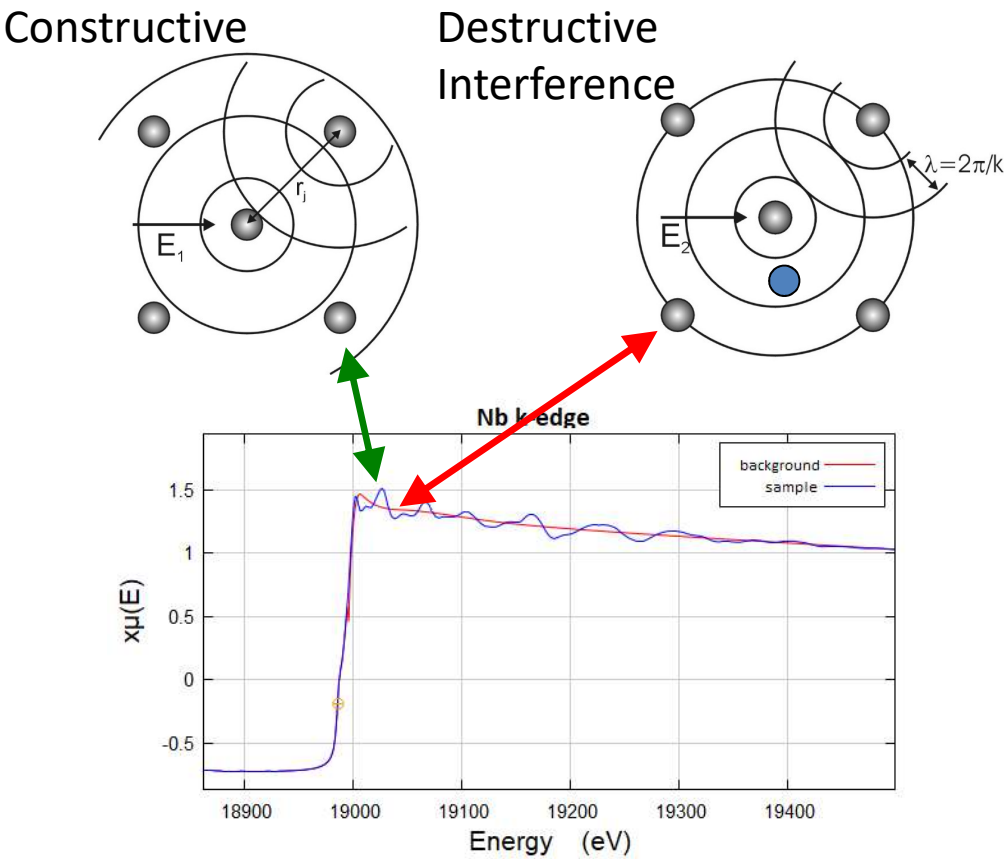
Valence Probe: XANES gives chemical state and formal valence of selected element.

Local Structure Probe: EXAFS gives atomic species, distance, and number of near-neighbor atoms around a selected element..

Low Concentration: concentrations down to 10 ppm for XANES, 100 ppm for EXAFS detectable!

Various Samples: samples can be solids, solutions, amorphous solids, soils, surfaces, etc.

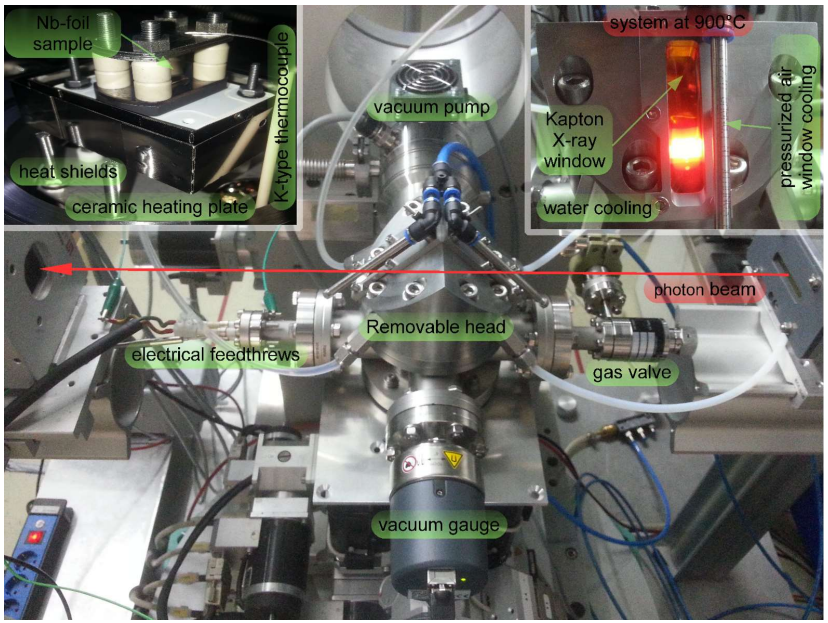
In-situ measurements: Suited for ***real in-situ studies*** with time resolution!

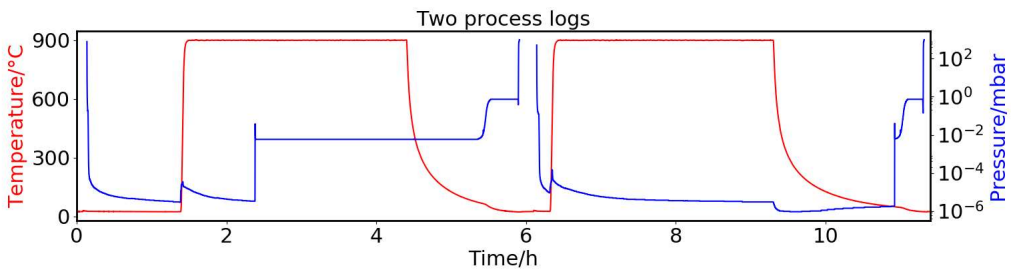


- Measure energy-dependence of X-ray absorption coefficient μ(E) of a core-level of selected element (i.e. ***Nb***)
- Good example of wave-particle dualism: Photoelectron acts like a wave!
- Interference effects on absorption coefficient
- Fourier-Transform of the oscillations gives bond length in R-space

- **Main Idea:** Inserted N-atoms will slightly modify interference pattern of Nb

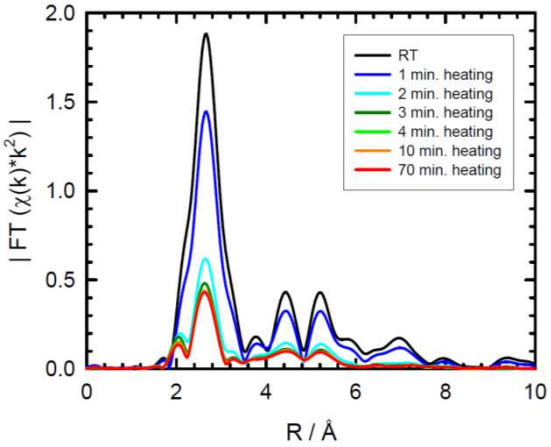
Experimental setup

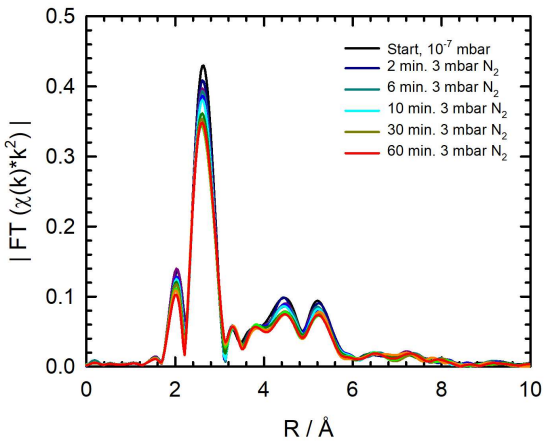




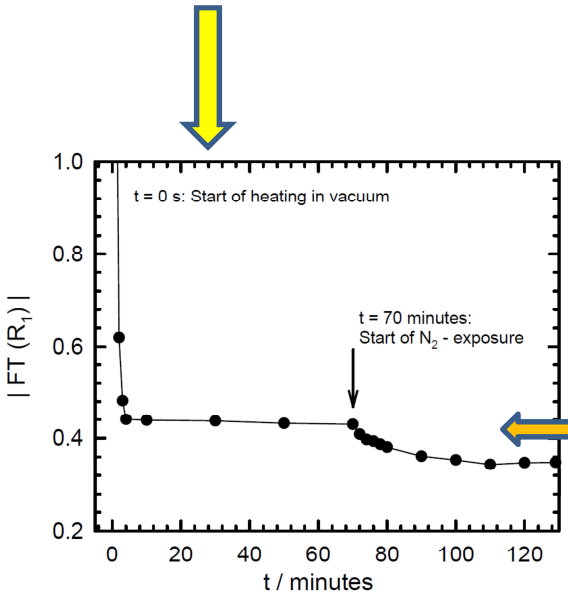
- In-situ heating chamber, fully remote controlled
- Kapton windows for X-ray measurements
- RT < T < 1200°C
- p < 10^{-6} mbar
  (at 900°C ca. 2x10^{-6} mbar)
- Nb foils of 6 – 25 µm thickness
- Measured in transmission
- Treatment up to 30 mbar N_2 (Kr, Ar,…) for minutes … to … several hours
- Slightly higher pressure and time (= exposure) because of very small effects

In-situ Measurements: Time dependence





Phase 1: Heating in vacuum

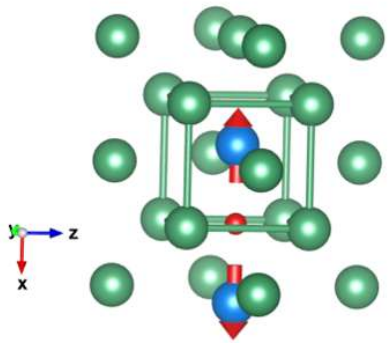


- Plot Fourier-Trafo in R-Space
- Reduction in NN-amplitude
  ⇒ caused by lattice vibrations
- Constant after first minutes of baking
  ⇒ no effects of e.g. poor vacuumconditions etc.

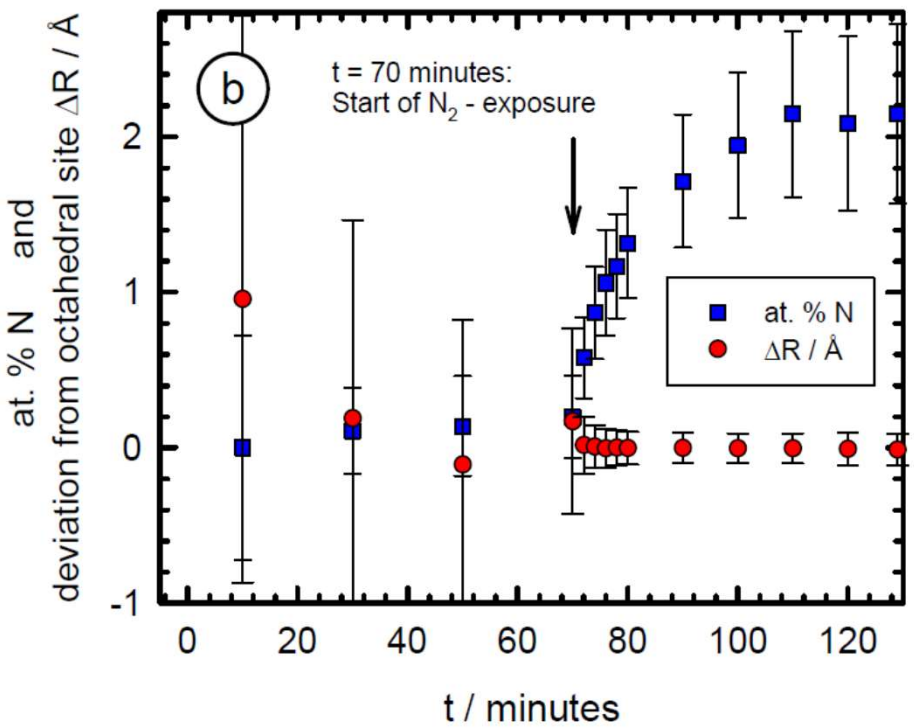
Phase 2: N_2 - exposure

- Further reduction in NN-amplitude caused by N-gas exposure
  ⇒ Effect of N_2 visible
- Uptake causes blurring

Used model and EXAFS data fitting with two phases

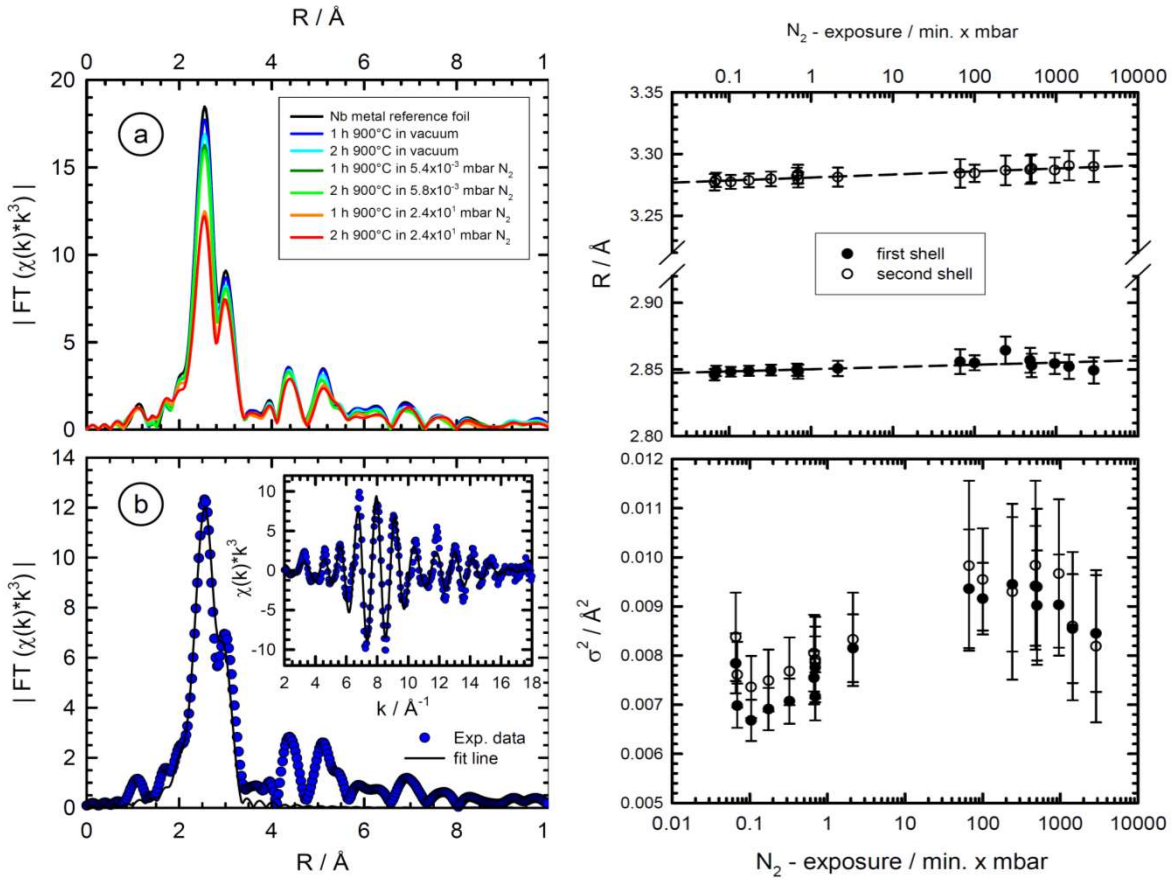


- Nb: bcc-crystal
- N_2-uptake at octahedral interstitial site
- Neighboring atoms are (slightly) displaced



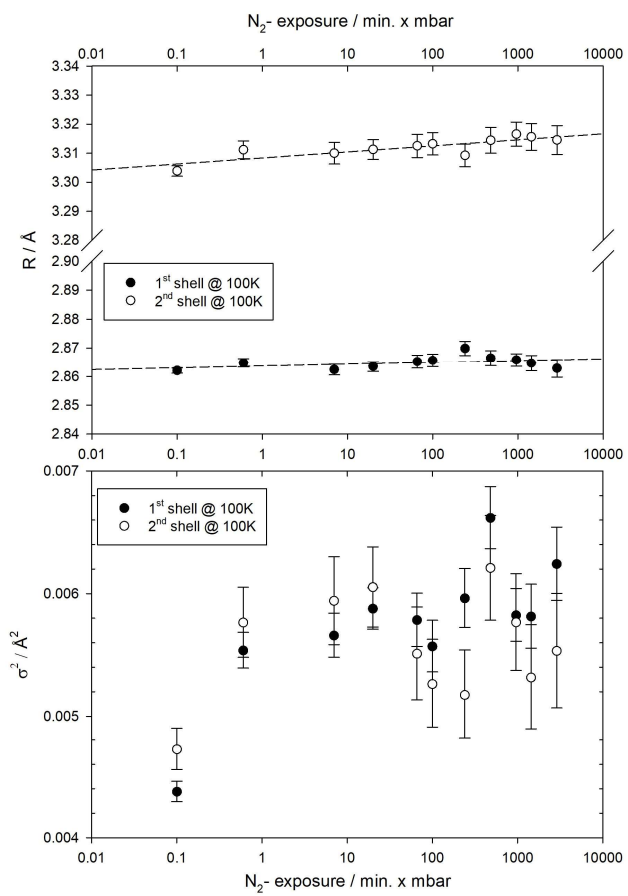
- Model of hard spheres
- Linear Fit of unit cells with and without N_2
- Up to 2% of unit cells with N_2
  - Bcc (2 Nb-Atoms/unit-cell): 1 atom%
- Octahedral site competitive with ionic N-size of 70 pm

Ex-situ measurements at Room-Temperature: Exposure dependence



- Verification of in-situ measurements
- Fit of the first two coordination shells
- Investigation on effect of different exposure intensities on Nb-Nb bond distances (R_1, R_2) and mean squared displacement (σ_1^2, σ_2^2)
- Increases agree qualitatively with model
- **<u>But:</u>** large uncertainties

High precision (ex-situ) EXAFS measurements at 100 K



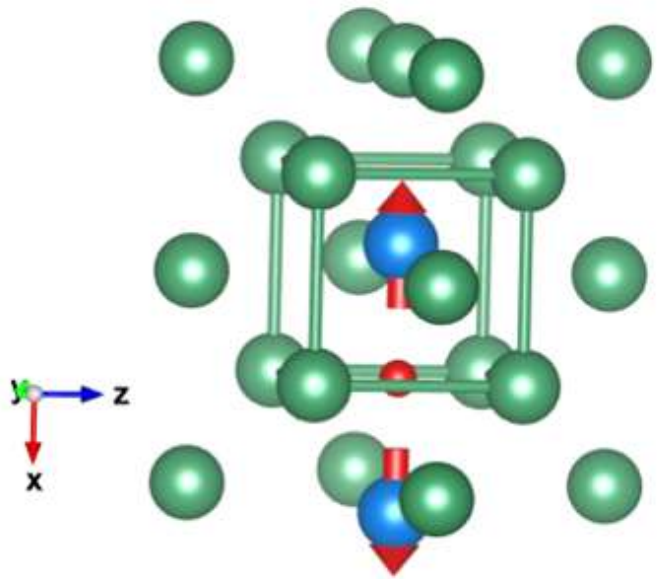
- High precision
  - Reduction of lattice vibrations
  - Smaller uncertainties in R
  - Smaller σ^2 (linear shift)

- Main result:
  Trends from RT measurements are fostered

- N_2 -uptake on interstitial octahedral sites
- Investigations of time- and pressure-dependence
- In- and ex-situ measurements agree with each other
- 100 K measurements show same trends
- Kr- and Ar-uptake: do not show any detectable effects

Next steps:

- Improvement of fit model
  - More shells / paths
- Measurements with samples from real cavities using surface sensitive EXAFS
  $\Rightarrow$ **samples from the community are welcome!**



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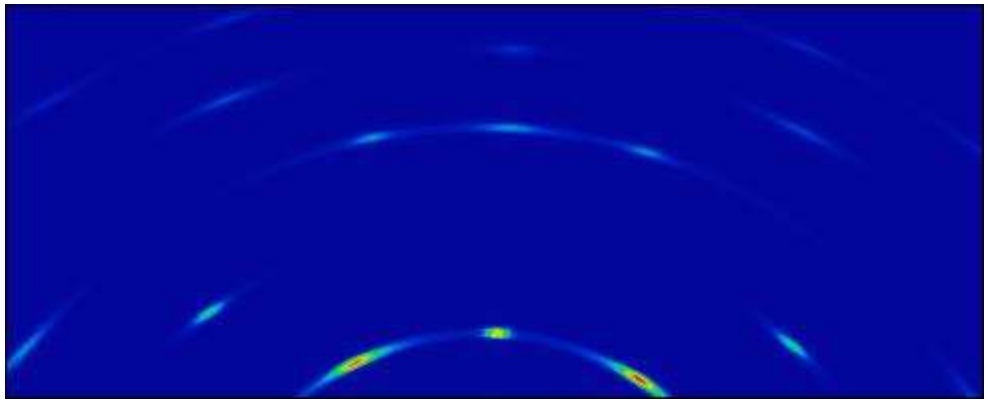
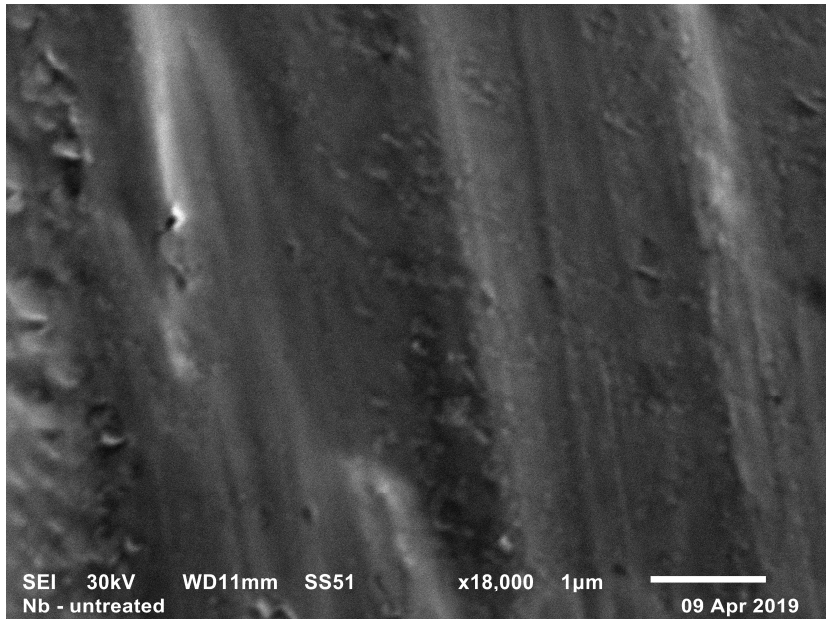
- MWF NRW: financial support

Addional material: Nb - foils



X-ray diffraction, E = 16 keV:

Polycrystalline, textured foils



SEM:

Smooth, wavy surfaces