In-situ EXAFS investigation of N_2 - treatment of Nb

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TESLA TECHNOLOGY TTC COLLABORATION

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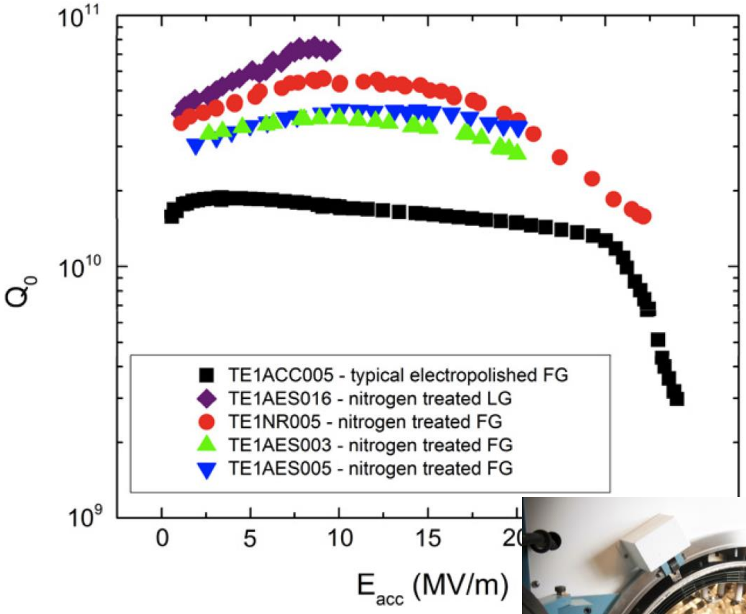
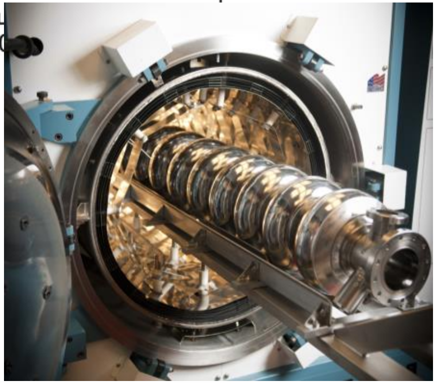
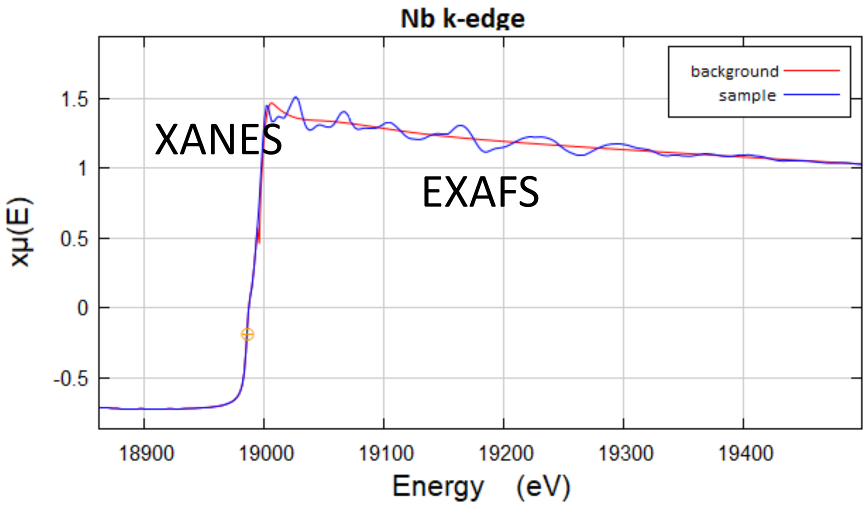
Outline

1) Motivation
2) Methodology
   - EXAFS spectroscopy
   - Experimental details
3) Results and discussion
4) Conclusions and outlook





- Improvements of Nb-cavity quality factors by N_2-treatments at elevated temperatures
  - ⇒ "nitrogen-doping"
- Structural details of N_2-doping still not understood in detail:
  - Position of nitrogen in the lattice?
  - Distortions of the lattice?
  - …
- Environment (N_2 - atmosphere, high temperatures, …
  - ⇒ difficult to investigate with standard techniques (XRD, XPS, SEM/TEM, …)
  - ⇒ alternative techniques!
- **Here:** in - situ investigations with EXAFS



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..

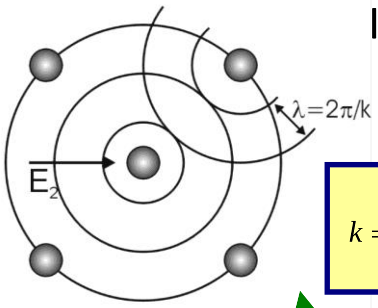
Low Concentration: concentrations down to 10 ppm for XANES, 100 ppm for EXAFS detectable!

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

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

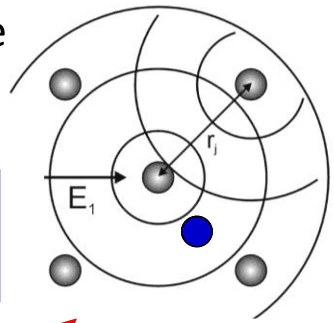
Continuous X-ray source: Synchrotron needed! **Here:** PETRA III / DESY, SLS / PSI (Ch), DELTA (Dortmund)

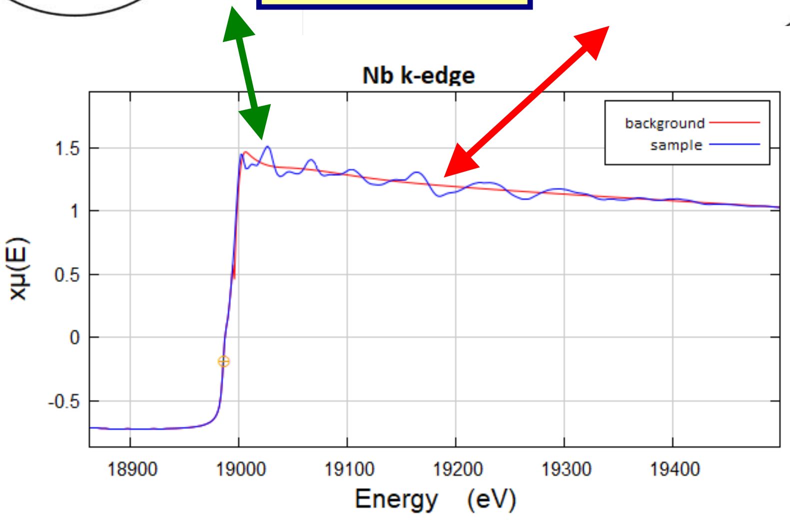
Constructive



Destructive Interference

$$k = \sqrt{\frac{2m_e(E - E_0)}{\hbar^2}}$$



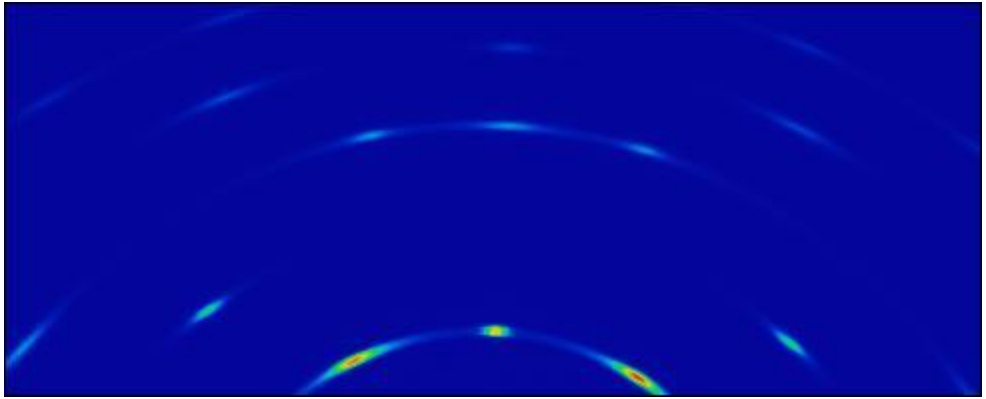


- Measurement of the energy-dependent X-ray absorption coefficient μ(E) of a core-level of a selected element (here: **Nb K-edge**)
- Good example of wave-particle dualism: Photoelectron acts like a wave ⇒ energy-dependent interference of outgoing and scattered waves ⇒ modulation of μ(E)
- Fourier-Transform of the oscillations gives bond length in R-space

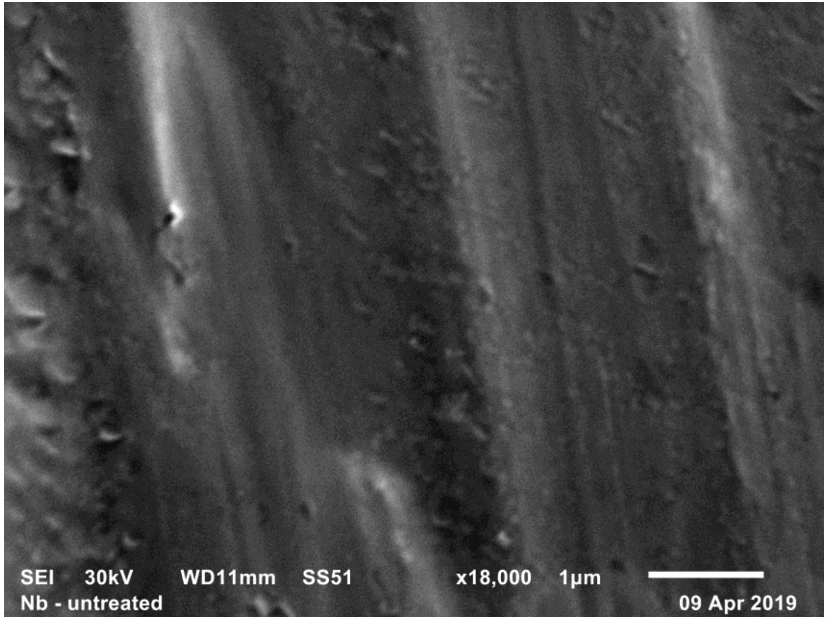
- **Main Idea:** Inserted N-atoms will slightly modify interference pattern of Nb

2. Nb-foil samples

High purity Nb-materials:
99.99% Nb foils of 6 – 25 µm thickness

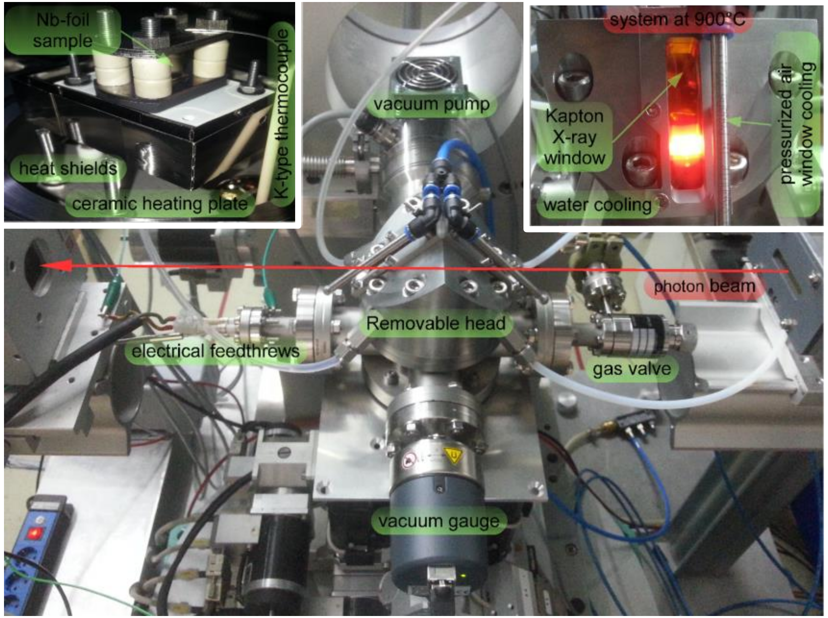


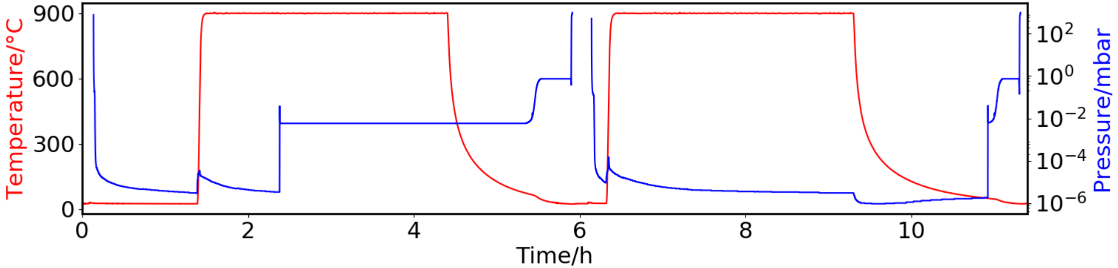
X-ray diffraction, E = 16 keV:
⇒ Polycrystalline, textured foils



SEM:
⇒ Smooth, wavy surfaces (rolling of the foils)

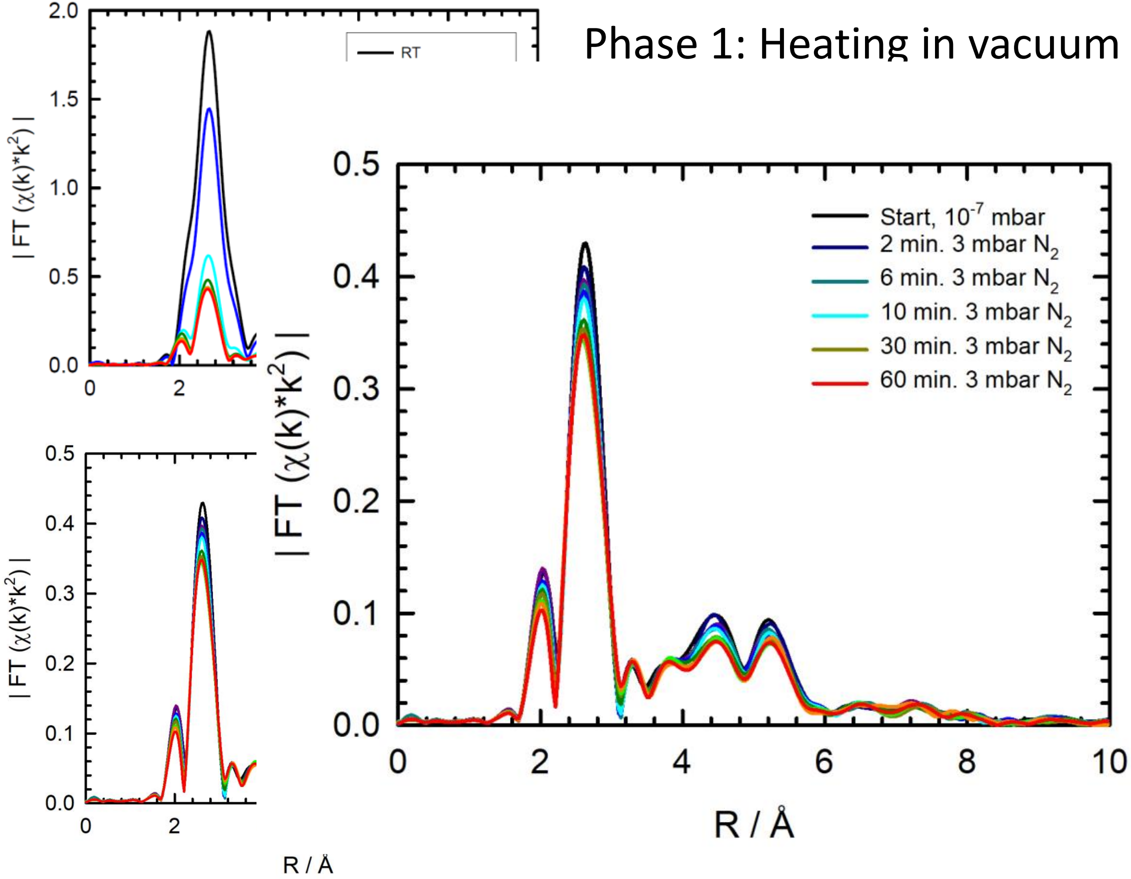
2. Experimental setup





- 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)
- Transmission X-ray measurements
- Treatment with up to 30 mbar N_2 (Kr, Ar, …) for minutes … to … several hours

3. In-situ X-ray measurements: Time dependence

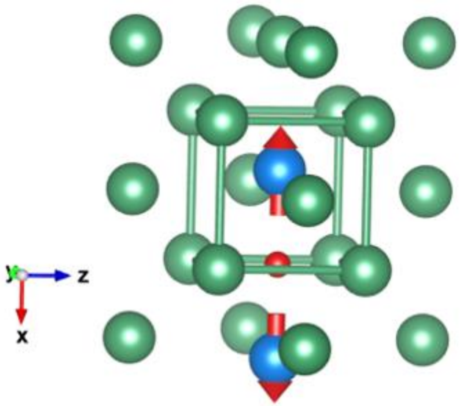


- Plot Fourier-Trafo in R-Space
- Reduction in NN-amplitude
  ⇒ caused by lattice vibrations
- Constant after first few minutes of baking
  ⇒ no effects of e.g. poor vacuum conditions etc.

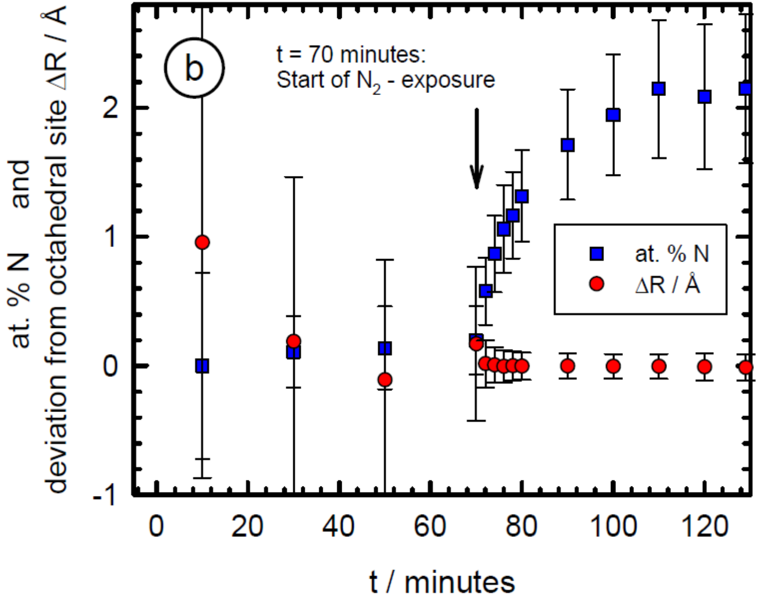
Phase 2: N_2 - exposure

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

3. Modelling and EXAFS data fitting with two phases

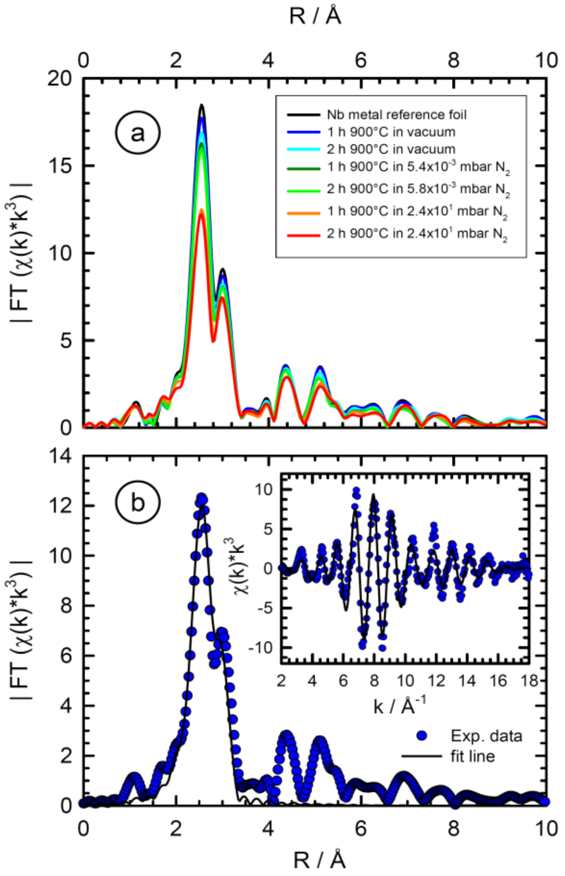


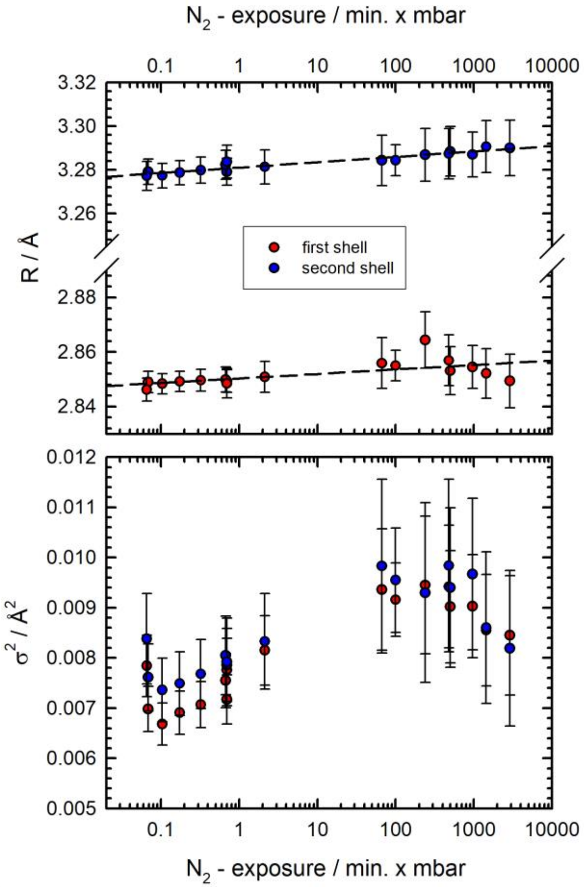
- Nb: bcc-crystal
- N_2-uptake (●) at octahedral inter-stitial sites
- Neighboring atoms (●) are (slightly) displaced



- Model of hard spheres
- Octahedral site competitive with ionic N-size of 70 pm
- Linear combination fits of unit cells with & without N-atoms
- bcc – lattice: 2 Nb atoms/unit- cell)
- up to ca. % of unit cells with N_2

3. Ex-situ measurements at 300 K: Exposure dependence

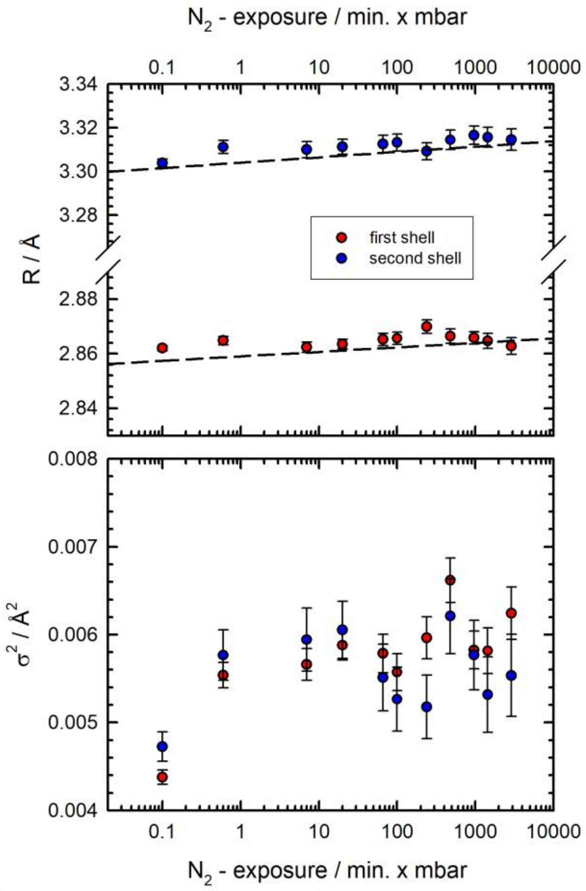




- Verification of in-situ measurements
- Fit of the first two coordination shells
- Investigation on effect of different exposure intensities on Nb-Nb bond distances (R_1, R_2) and mean squared displacement (σ_1^2, σ_2^2)

- Observed increase of R_1, R_2, σ_1, σ_2 agree qualitatively with model
- **But:** large uncertainties

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



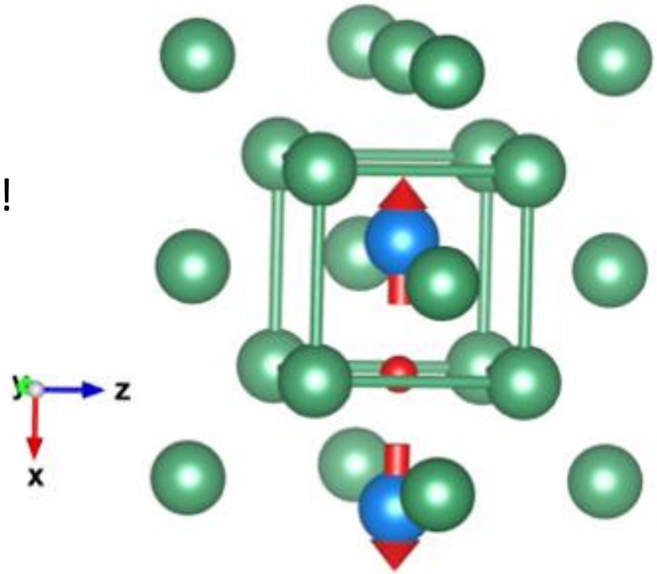
- Measurements at LN2-temperature:
  ⇒ Reduction of thermal lattice vibrations
  ⇒ larger EXAFS amplitudes, increased accuracy
  - Smaller uncertainties in R
  - Smaller σ^2

    But: static disorder increases with N_2- exposure!
    & is larger than bulk Nb reference

- Main results:
  Trends from RT measurements are fostered

4. Conclusions and Outlook

- N_2 -uptake in Nb on interstitial octahedral sites, increasing with time and pressure
- In- and ex-situ measurements agree with each other
- precision measurements foster trends (slightly increased average Nb-Nb-distance & disorder)
- Kr- and Ar-uptake: do not show any detectable effects!



Next steps:

- Improvement of cell: T-measurement, MS, …
- Measurements at 4 K – even more precise …
- Improvement of the 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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… and you for you attention!!!

EXAFS - Function $\chi(k)$:

K-edges, Gaussian pair-distribution function between neighboring atoms:

$$\chi(k) = \sum_i N_i \left| f_i(\pi,k) \right| \frac{1}{k r_i^2} e^{-2r_i/\lambda} e^{-2\sigma_i^2 k^2} \cdot \sin\left[2kr_i + \phi_i(k) \right]$$

Scattering power | damping | therm. + stat. disorder | oscillations

k	wave-number of the photo-electron: $k = \sqrt{\frac{2m_e(E-E_0)}{\hbar^2}}$
r	distance to neighboring atom
$\phi(k)$	phase-shift: absorber $\phi_A(k)$, scattering atom $\phi_B(k)$ (sensitive for the type of neighboring atom)
N	coordination number of neighboring atom
$\|f(\pi,k)\|$	scattering amplitude of neighboring atom (element - sensitive)
λ	mean free path of the photo-electron
σ^2	mean squared displacement (stat. & therm.!) of the neighbor