The FWHMs and binding energies of the Nb compounds obtained by fitting (PE 1200 eV)

Thermal treatment of Nb Samples

- large-grain Nb (Heraeus) + BCP

Three recipes:
- 800°C vacuum anneal during 3h (10^-6 mbar)
- nitrogen infusion (800°C + 120°C/3h/10^-6 mbar of N2)
- infusion but without N2 supply (800°C + 120°C/3h/10^-6 mbar of N2)

After the treatment, the samples were subjected to ambient atmosphere for 4 hours and transferred to XPS set up for further investigation.

Survey Synchrotron Radiation XPS Spectra (PETRA III)

- Nb 3d
- Ti 2p
- NbOx (and adsorbates, O 1s)
- NbNx - ? (FWHM > 5 eV => not resolved)

950°C anneal, HV, no air exposure (XPS, BESSY II)
- large-grain Nb (Heraeus) + BCP
- 955°C/11h, p=2·10^-10 – 2.5·10^-9 mbar
- no air exposure before XPS!

Residual gas analyzer registered:
- at 300°C (p=1.3·10^-8 mbar):
  - CO: 6.7·10^-10 mbar, CO2: 1.0·10^-9 mbar, H2O 1.4·10^-9 mbar
- at 885°C (p=9.1·10^-10 mbar):
  - N2: 1.6·10^-7 mbar, CO: 1.6·10^-10 mbar, CO2: 1.6·10^-10 mbar, H2O 5.6·10^-10 mbar
- at 950°C (p=2.5·10^-10 mbar, the end of anneal): H2O (3.1·10^-9 mbar) and CO (2.7·10^-9 mbar)

Formation of negligible amount of niobium carbide and nitride phases (along with Nb2O5 and Nb3O8)

Main Results

800°C-anneal, Infusion, and Infusion w/o N2 after Air Exposure:
- No carbide or nitride phases formation was detected upon 800°C anneal, infusion and infusion without nitrogen gas by XPS, RRD, SEM and Raman Spectroscopy.

Synchrotron radiation XPS revealed a trace amount of TiO2 at the surface of the infused samples at 955°C/11h, while no effect of N2 addition was detected onto Nb phase formation in our experiments.

Small changes in sub-oxides and the interstitial content among the samples were observed between 955°C- and infused samples, while minimal for the infused w/o N2 sample. Infused Nb+4 is this increase due to Nitrogen interstitials?