In-situ EXAFS investigations of Nitrogen doped Niobium

J. Kläs, R. Wagner, B. Bornmann, R. Frahm, D. Lützenkirchen-Hecht
[University of Wuppertal, Fakultät 4 Physik, Gaußstraße 20, 42119 Wuppertal, Germany]
mailto:klaes@uni-wuppertal.de

Introduction:

Heat treatments under nitrogen and argon gas atmospheres were successfully used recently to improve the RF-superconducting properties of niobium-based particle accelerator cavities in medium acceleration fields. A significant lowering of the surface resistance was reported, however, a full understanding of the structural changes which lead to these improvements is still missing. Thus, we have used EXAFS spectroscopy to probe the structural changes in-situ during the heat treatments of metallic niobium samples on an atomic level in a temperature range from 25 °C to ca. 900 °C.

Experimental methods:

For the in-situ preparation, a dedicated vacuum chamber (base pressure ca. 10^{-7} mbar) featuring a ceramic heating plate suited for temperatures of up to 1200 °C was constructed. Heating rates of more than 500 °C/minute were realized, while cooling took much more time. The preparation chamber is lightweight and sufficiently small to fit on a standard EXAFS beamline, enabling transmission as well as fluorescence and reflection mode EXAFS experiments. Processing of the Nb samples (foils of 25 µm thickness) included a treatment in vacuum for 1 h at 900 °C and a subsequent exposure to high-purity N_2-gas under a pressure of $3x10^{-3}$ mbar for varying times. EXAFS data were collected prior to any heat treatment as well as during the different process steps at elevated temperature, and after cooling to room temperature under vacuum. The experiments presented here were performed at the wiggler beamline 8 of the DELTA storage ring (Dortmund, Germany), at PETRA III beamline P64 (DESY, Hamburg, Germany) and the SuperXAS beamline at the Swiss Light Source (Villigen, Switzerland).

Results and Discussion:

The quantitative EXAFS data evaluation shows that all the data sets can be fitted using metallic Nb-Nb coordinations only, suggesting that no impurity phases were formed, in particular there was no evidence for Nb-nitride formation as proposed by previous studies. In contrast, the disorder parameters for the first few Nb-Nb shells substantially increase with process time, especially with N_2-exposure. This observation may be explained by the diffusion of N-atoms onto interstitial lattice sites, leading to a small local deformation in the Niobium bcc structure, which may be correlated with the improved superconducting properties of N_2-treated Niobium.

Conclusions:

According to the presented EXAFS experiments, a heat treatment of Nb foils in dilute N_2-atmospheres at elevated temperatures leads to an increasing disorder of the Nb-lattice with increasing exposure time, instead of a possible formation of Nb-nitrides. Obviously, N_2 is adsorbed and diffuses into the bulk Nb metal, where interstitial sites are increasingly occupied.

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