STRUCTURAL INVESTIGATIONS OF NITROGEN-DOPED NIOBIUM FOR SUPERCONDUCTING RF CAVITIES*

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Abstract

Niobium is the standard material for superconducting radio frequency (SRF) cavities. Superconducting materials with higher critical temperature or higher critical magnetic field allow cavities to work at higher operating temperatures or higher accelerating fields, respectively. Enhancing the surface properties of the superconducting material in the range of the penetration depth is also beneficial. One direction of search for new materials with better properties is the modification of bulk niobium by nitrogen doping. In the Nb-N phase diagram the cubic δ-phase of NbN has the highest critical temperature (16 K). Already slight nitrogen doping of the α-Nb phase results in higher quality factors. Nb samples were processed in the refurbished UHV furnace at IKP Darmstadt. The first results on the structural investigations of the processed Nb samples at the Materials Research Department of TU Darmstadt are presented.

INTRODUCTION

Superconducting radio frequency (SRF) accelerator cavities are becoming the standard for particle energies beyond 1 GeV. The state-of-the art cavities are made of high quality bulk niobium. The cavities undergo multiple processing steps, including chemical etching, high pressure rinsing with ultrapure water and baking to reach their designed quality factor and accelerating gradient.

Grassellino *et al.* managed to increase the quality factor of Nb cavities by introducing an additional N-doping step to the then standard recipe [1]. Recently a new, milder way of nitrogen diffusion was introduced, the nitrogen infusion process [2]. In contrast to the short time, high temperature N-doping the introduction of nitrogen in the infusion is done at low temperature and for long time. The increase in quality factor is achieved without further electro-polishing, a necessary treatment after N-doping to remove the surface nitrides. Koufalis *et al.* emphasized the beneficial role of carbon and oxygen trace elements present in the nitrogen atmosphere [3], possibly causing the same effect in the long time annealing as the short N-doping at the end of the high temperature bake-out.

A different way of improving the performance for SRF cavities could be the enhancement of the critical temperature by transforming the surface region (in the depth of the penetration length) of the Nb-cavity to the cubic δ-phase of NbN [4].

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UHV FURNACE

The Darmstadt UHV furnace was originally built at the University of Wuppertal in 1983 [5] and relocated to Darmstadt in 2002. It was designed for temperatures of up to 1800 °C with vacuum pressures lower than 10^{-6} mbar. Since 2005 it has been used for hydrogen bake-out of several superconducting niobium cavities [6]. Due to technical constraints the temperature was limited to 850 °C. Recently the furnace has been upgraded and re-commissioned to operate again up to the designed value of 1800 °C [7]. Further modifications were made to allow nitrogen treatments of niobium samples and cavities [8]. As part of the recommissioning, Nb test samples were baked out in vacuum at different temperatures. The samples were characterized at the Materials Science Institute (MSI) of Technische Universität Darmstadt.

METHODS

Niobium samples (Fig. 1) were baked out in the high-temperature UHV furnace. The samples were characterized by x-ray diffraction (XRD) and secondary ion mass spectroscopy (SIMS).

High quality Nb sheets (RRR 300) were treated by buffered chemical polishing (BCP), then cut to 5x5 mm^2 squares by high pressure water at Research Instruments. The XRD measurements were done at the Advanced Thin Film group of the MSI. A Rigaku SmartLab diffractometer with rotating anode (λ=1.54 Å), parallel beam set-up and pyrolytic graphite analyser was used. The SIMS measurements were done at the Materials Analysis group of the MSI on a Cameca ims5f spectrometer with O^{2+} ions.

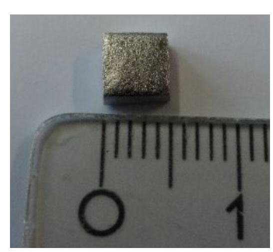

Figure 1: Niobium test samples cut from high quality Nb sheet. Typical sample size was 5x5x2.7 mm^3.

RESULTS

In Figure 2 the XRD scan of a virgin Nb sample is shown. After the bake-out process the intensity of the Bragg peaks changed (not shown). We attribute this change to uncertainties in the sample adjustment. The high quality Nb material is known to consist of huge crystallites. In such textured materials the intensity of the Bragg peaks depend on the presence of crystallites in reflection condition, in contrast to randomly distributed polycrystalline material, where crystallites of all orientations can be found. For highly textured Nb a small change in sample alignment can lead to huge changes in peak intensities, as was verified by taking rocking curves (ω-scans). No peaks related to other phases were seen.

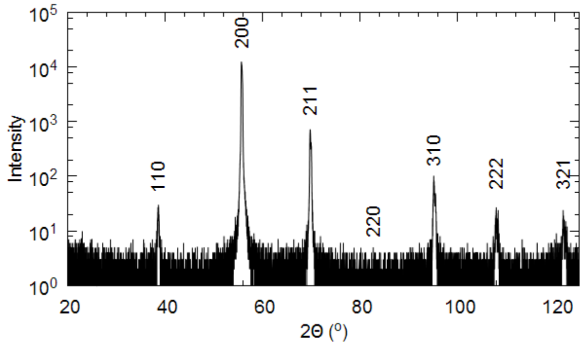

Figure 2: XRD pattern of a virgin Nb sample.

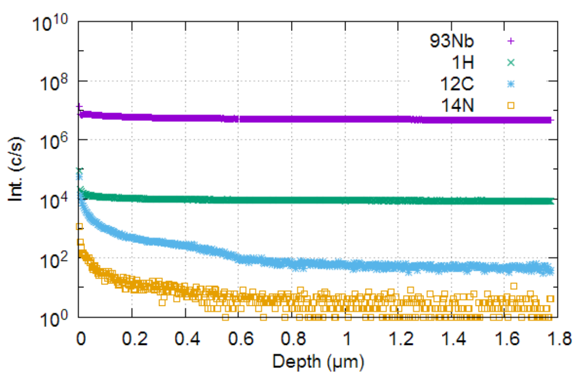

Figure 3: SIMS depth profile of the virgin Nb sample.

In Figure 3 the SIMS depth profiles of selected elements are plotted. Amongst niobium (matrix), the impurity elements hydrogen, carbon and nitrogen were measured. For the virgin sample the hydrogen concentration is constant for the first µm, while the amount of carbon and nitrogen decreases with increasing depth.

As part of the commissioning of the high-temperature UHV furnace two Nb samples were baked out in vacuum. The first sample was annealed at 850 °C for 4 h (maximal pressure: $6.8 \cdot 10^{-7}$ mbar). The second sample was annealed at 1027 °C for 4 h (maximal pressure: $5.0 \cdot 10^{-7}$ mbar). The SIMS profile scans of the virgin and treated samples are compared in Fig. 4. The hydrogen and carbon concentration decreased with the bake-out, while the nitrogen level remained approximately constant. This is in agreement with our expectations, as for the test procedure no nitrogen was introduced to the furnace.

The base pressure of the UHV furnace improved during the bake-out cycles, showing the cleaning effect of the repeated heating.

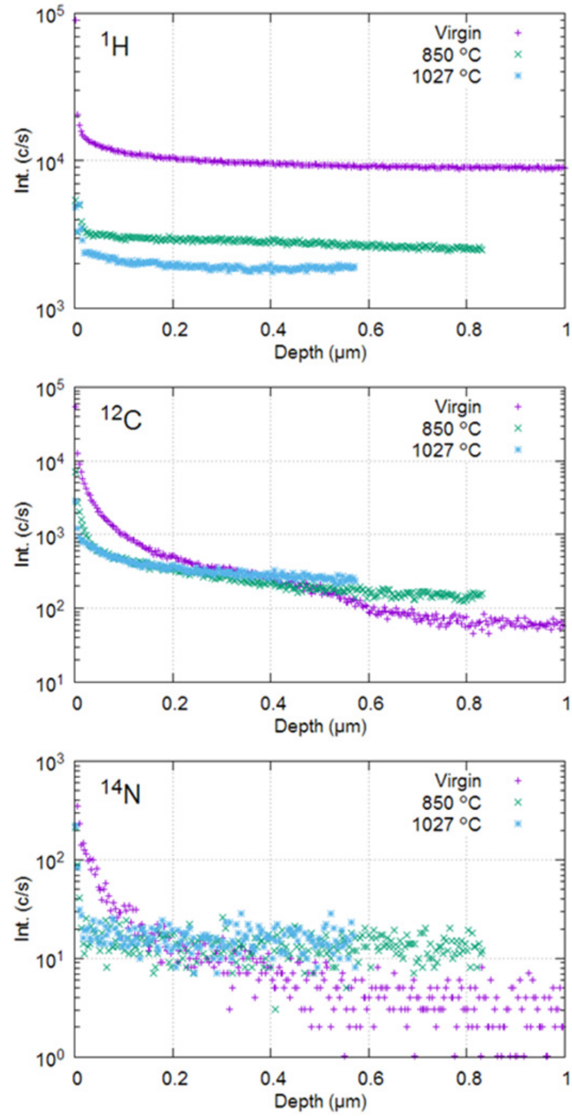

Figure 4: Comparison of the SIMS depth profiles for hydrogen (top), carbon (middle) and nitrogen (bottom) of the virgin and heat treated samples.

CONCLUSION AND OUTLOOK

Nitrogen doping has a beneficial effect on the quality factor of SRF cavities. Niobium samples were baked-out in vacuum in the high-temperature "Wuppertal" furnace as part of the recommissioning of the furnace. The Nb samples were characterized by XRD and SIMS. No new phases were found by XRD in agreement with the SIMS results, showing significant decrease of hydrogen trace concentration and no increase in nitrogen level, in accordance with the experimental conditions (nitrogen was not applied during the bake-out for this series).

The future plan is to continue the vacuum bake-out up to the maximal possible temperature, after which a new bake-out series is planned, now in nitrogen atmosphere. The treated samples will be analysed at the MSI, TU

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Darmstadt to optimize the doping conditions. Finally, single cell cavities should be N-doped according to the optimized recipe.

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