ISBN: 978-3-95450-211-0 doi:10.18429/JACoW-SRF2019-MOP028

MATERIALS SCIENCE INVESTIGATIONS OF NITROGEN-DOPED NIOBIUM FOR SRF CAVITIES

M. Major†[1], L. Alff, M. Arnold, J. Conrad, S. Flege, R. Grewe, N. Pietralla
Technische Universität Darmstadt, Darmstadt, Germany
[1]on leave from HAS Wigner Research Centre for Physics, RMKI, Budapest, Hungary

Abstract

Niobium is the standard material for superconducting RF (SRF) cavities for particle acceleration. Superconducting materials with higher critical temperature and higher critical magnetic field allow cavities to work at higher operating temperatures and 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. Niobium samples were annealed and N-doped in the high-temperature furnace at TU Darmstadt and investigated at its Materials Research Department with respect to structural modifications. Secondary ion mass spectrometry showed at which conditions N-diffusion takes place. X-ray diffraction (XRD) confirmed the formation of the δ-NbN and β-Nb_2N phases for the optimized doping process. XRD pole figures also showed grain growth during sample annealing.

INTRODUCTION

The technology of bulk niobium cavities is already close to the theoretical performance limit thanks to several decades of continuous research and technological innovation. Today industrialised cavity production with fields up to 45 MV/m and a Q-factors exceeding 10^{10} at 2 K operation is possible [1, 2], with a record of 49 MV/m achieved recently [3].

The research on new materials could lead to more compact and energy efficient accelerators. The recently published Conceptual Design Report of the Future Circular Collider [4] proposes the application of both niobium coated copper and bulk niobium cavities, as part of the accelerator structure of FCC-ee [5]. It builds on the long tradition of coated copper superconducting cavities at CERN. The high heat conductivity of copper would permit 4.5 K operation. Niobium coated Cu cavities are less prone to magnetic flux trapping compared to bulk Nb, but the steep Q-slope of the coated cavities must be mitigated to outperform the bulk niobium technology [5].

METHODS

High quality Nb sheets (RRR 300, approx. 2.8 mm thick) purchased from Research Instruments (RI) were treated by buffered chemical polishing (BCP), then cut to 5x5 mm^2 squares by high pressure water at RI. The cut samples were baked out in the high-temperature UHV furnace located at IKP, TU Darmstadt ("Wuppertal oven") [6]. The virgin and treated samples were characterized by X-ray diffraction (XRD), electron microscopy and secondary ion mass spectrometry (SIMS) at the ATFT group of TU Darmstadt.

The XRD measurements were done on a Rigaku SmartLab diffractometer with rotating copper anode (λ=1.54 Å), line focus mode and parallel beam set-up. Specular scans were taken in θ-θ geometry and pole figures at different Bragg-peak positions (constant detector angle).

The SIMS measurements were done on a Cameca ims5f spectrometer with O^{2+} ions.

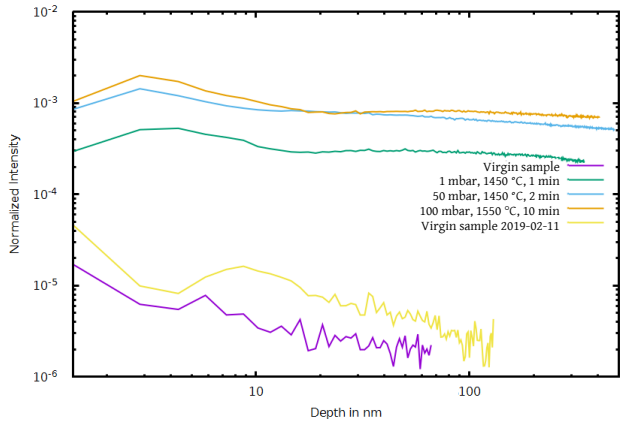

Figure 1: SIMS depth profiles of ^{14}N normalised to ^{93}Nb measured on the Nb samples with O^{2+} ions.

RESULTS AND DISCUSSION

Niobium samples were annealed and nitrided according different recipes. The process parameters were changed in a stepwise manner. Based on the SIMS feedback, the doping recipe was changed from single-shot nitrogen injection to a continuous N_2. With this enhanced doping protocol nitrogen diffusion was finally observed (Fig. 1). The SIMS measurements also showed the depletion of hydrogen and carbon in the annealed samples compared to the virgin ones (not shown) in accordance with previous runs [7].

X-ray diffraction showed the appearance of surface Nb-N phases. For annealing at 1450 °C for 2 min in 50 mbar nitrogen atmosphere the Bragg peaks could be described as a mixture of α-Nb and β-Nb_2N phases (Fig. 2, top). For the sample annealed at higher temperature, for longer time and with increased nitrogen pressure (1550 °C, 10 min at 100 mbar N_2 atmosphere) the appearance of the δ-NbN phase was found (Fig. 2, bottom). The Bragg peaks are consistent with a mixture of α-Nb, β-Nb_2N and δ-NbN phases.

† major@oxide.tu-darmstadt.de

ISBN: 978-3-95450-211-0 doi:10.18429/JACoW-SRF2019-MOP028

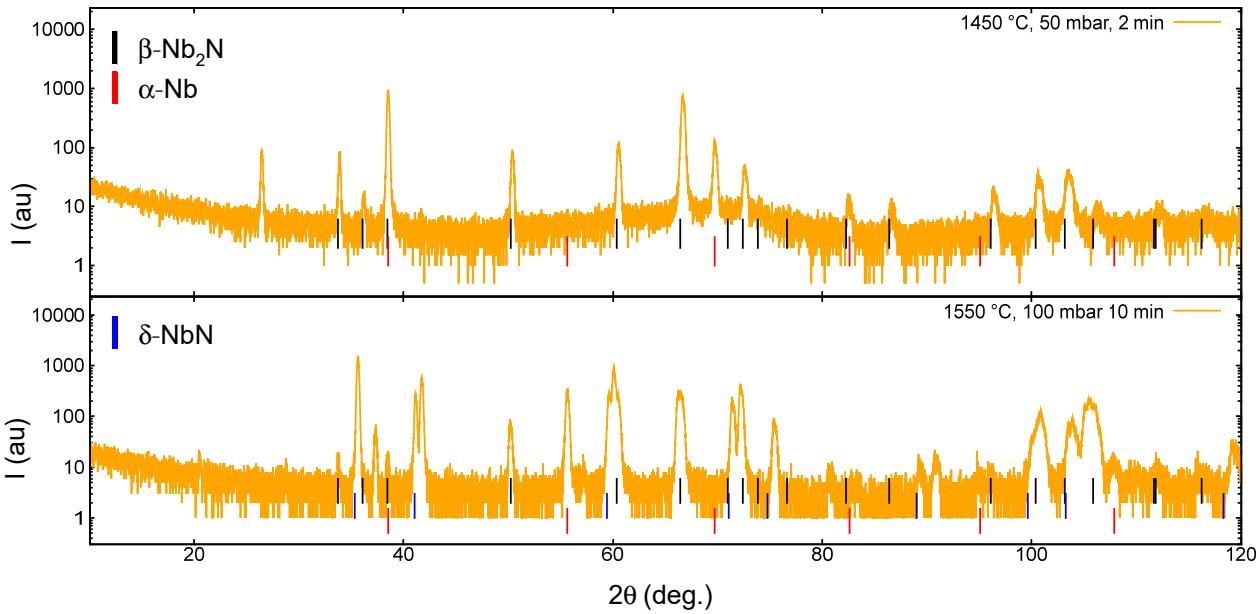

Figure 2: X-ray diffraction pattern of N-doped niobium samples. The nitridation was done in the "Wuppertal oven" at IKP, TU Darmstadt. The reflections of the phases are noted by tick marks: α-Nb (red), β-Nb_2N (black) and δ-NbN (blue).

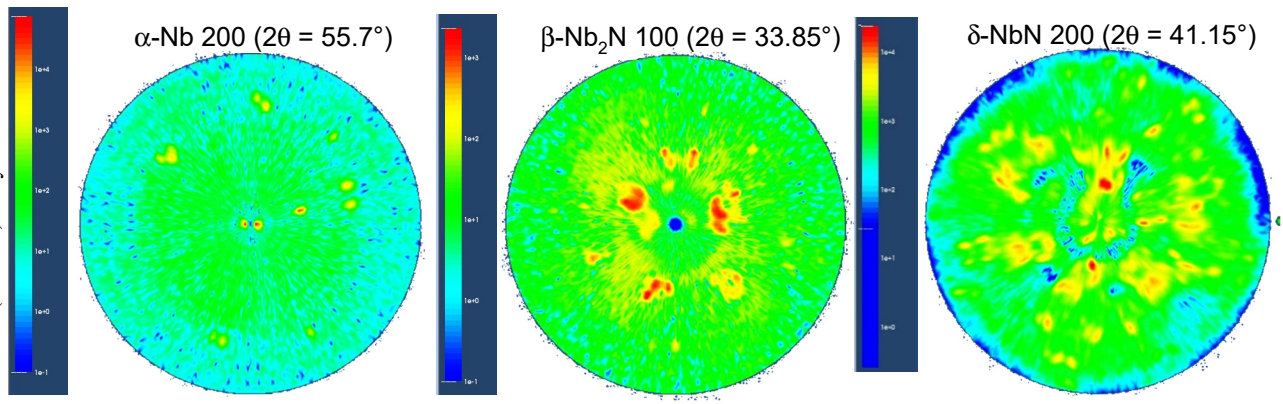

Figure 3: Pole figures of the different phases of a N-doped sample. The sample was annealed in the "Wuppertal oven" at 1550 °C in 100 mbar N_2 atmosphere for 10 minutes. Bragg peaks related to different phases were selected for the pole figures: α-Nb (left), β-Nb_2N (middle) and δ-NbN (right). The intensity is plotted on a logarithmic scale (colour bar).

As the materials is not in powder form and vertically layered (a strongly textured coating on an almost single crystal substrate) no quantitative phase analysis was possible. The possible transformation of the δ-phase to γ was also not investigated.

To check the crystallite size and texture of the newly formed surface phases, pole figures were measured (Fig. 3) in a similar way to previous studies [7]. This time different detector angles (2θ) were set for the three phases; 55.7°, 33.85° and 41.15° for the 200, 100 and 200 reflections of the α-Nb, β-Nb_2N and δ-NbN phases, respectively. The pole figure related to Nb (Fig. 3, left) shows a few very strong and narrow reflections, while the β- (middle) and δ- (right) phases are less textured with larger mosaicity (seen as larger number of broader peaks).

We interpret the broad distribution of the formed nitride phase as small crystallite grown on top the almost single crystalline Nb. The grain size of Nb is much larger, than of the nitrides due to the recrystallization seen before [7].

CONCLUSION

With the improved doping process Nb-N phases were first seen on samples doped in the "Wuppertal oven".

According to the pole figure measurements, different Nb-N phases showed different texture, and crystallite size. The δ-NbN phase showed the least texture, followed by β-Nb_2N, both sitting on top of the almost single crystalline α-Nb sample. The doping conditions (pressure, temperature and duration) will be further optimized to get a clean δ-NbN layer on top of the recrystallized niobium samples.

ISBN: 978-3-95450-211-0 doi:10.18429/JACoW-SRF2019-MOP028

ACKNOWLEDGEMENTS

This work is supported by BMBF through the projects 05H15RDRBA (part of "Superconducting Radio-Frequency Cavity Developments for Future Accelerators") and 05H18RDRB2 (part of "Key technologies for SRF accelerators") and the AccelencE Research Training Group (GRK 2128).

REFERENCES

[1] W. Singer *et al.*, "Production of superconducting 1.3-GHz cavities for the European X-ray Free Electron Laser", *Phys. Rev. Accel. Beams*, vol. 19, p. 092001, Sept 2016. doi:10.1103/PhysRevAccelBeams.19.092001

[2] N. Walker, D. Reschke, J. Schaffran, L. Steder, M. Wenskat, and L. Monaco, "Performance Analysis of the European XFEL SRF Cavities, From Vertical Test to Operation in Modules", in *Proc. LINAC'16*, East Lansing, MI, USA, Sep. 2016, pp. 657-662. doi:10.18429/JACoW-LINAC2016-WE1A04

[3] A. Grassellino *et al.*, "Accelerating fields up to 49 MV/m in TESLA-shape superconducting RF niobium cavities via 75°C vacuum bake", *arXiv*:1806.09824 [physics.acc-ph], 2018.

[4] Future Circular Collider Conceptual Design Report (FCC-CDR), https://fcc-cdr.web.cern.ch/

[5] A. Abada *et al.*, "FCC-ee: The Lepton Collider, Future Circular Collider Conceptual Design Report, Volume 2", *Eur. Phys. J. Special Topics*, vol. 228, p. 261, 2019.

[6] R. Grewe *et al.*, "Superconducting RF Cavity Materials Research at the S-DALINAC", presented at the 19th Int. Conf. RF Superconductivity (SRF'19), Dresden, Germany, Jun.-Jul. 2019, paper MOP022, this conference.

[7] M. Major *et al.*, "Structural Investigations of Nitrogen-Doped Niobium for Superconducting RF Cavities", in *Proc. 9th Int. Particle Accelerator Conf. (IPAC'18)*, Vancouver, Canada, Apr.-May 2018, pp. 3940-3942. doi:10.18429/JACoW-IPAC2018-THPAL127