Structural Investigations of Nitrogen-Doped Niobium for Superconducting RF Cavities

MOP028

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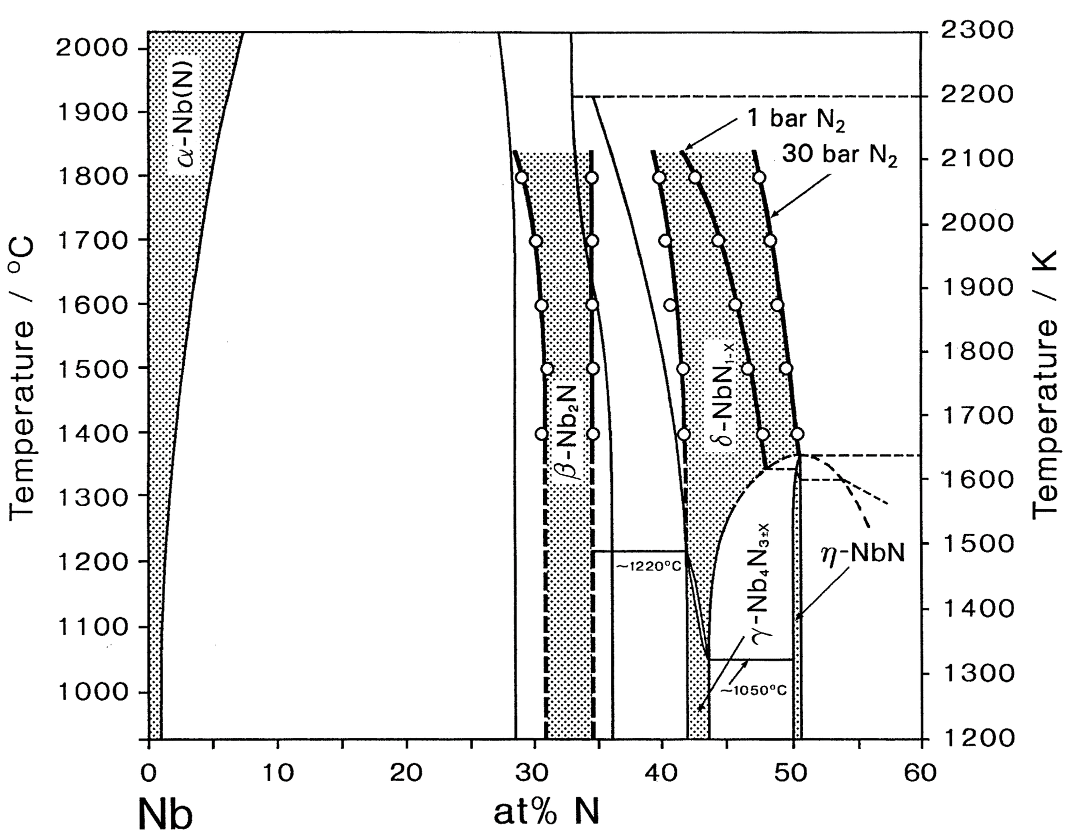
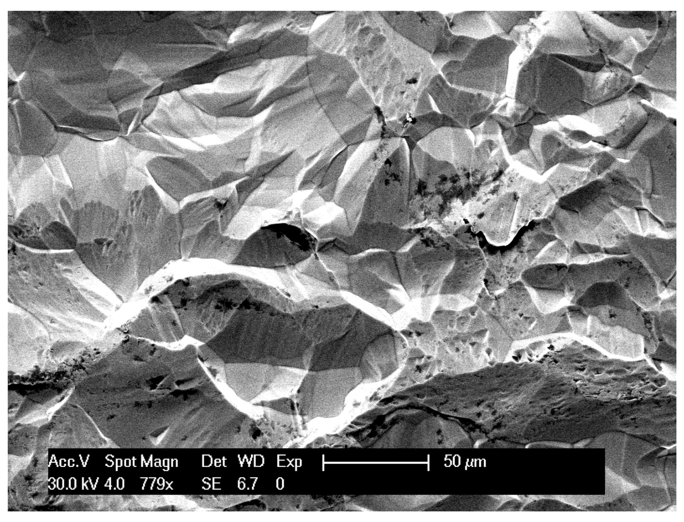
Introduction

Superconducting Radio Frequency (SRF) accelerating cavities are the *de facto* standard for high energy particle acceleration. The very high achievable quality factor (Q), low losses, robustness and reliability are all in favour of SRF cavities compared to traditional copper cavities. SRF technology could be even more attractive with reduced cooling costs. The possible increase of operating temperature from 2 K to 4.5 K by the implementation of high-T_c materials would reduce the size and running cost of cooling plants.

SRF cavities are made of niobium, which has a critical temperature $T_c = 9.2$ K, the highest amongst the elements. Nb based materials, like NbN ($T_c = 17.3$ K) or Nb_3Sn ($T_c = 18$ K) are potential candidates for high-Q cavities to be operated at higher temperatures. Costs reduction could be also achieved by using copper cavities coated by superconducting films (Nb or Nb_3Sn), a direction followed by CERN.

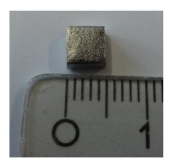


The low-N region of the Nb-N phase diagram [3].



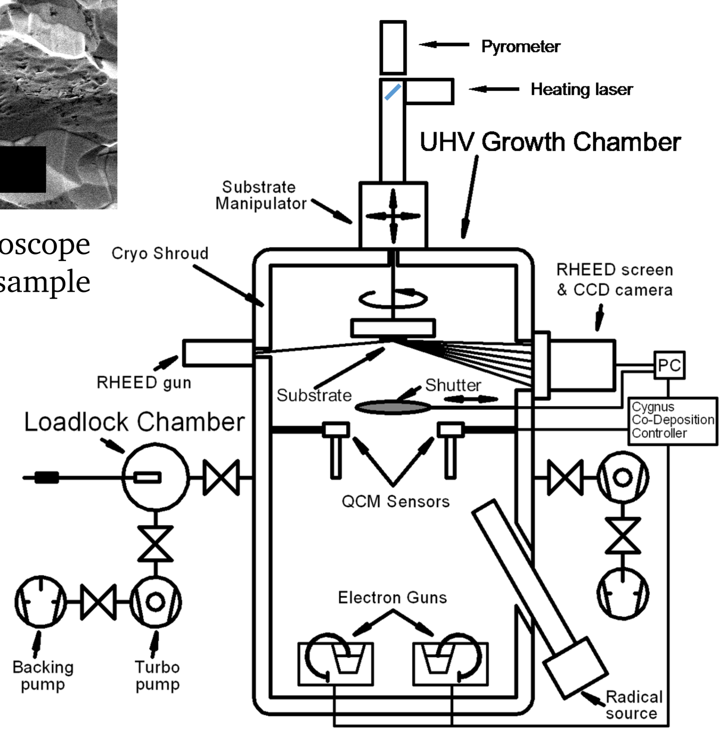
High resolution scanning electron microscope image of a typical virgin niobium sample showing the relatively high roughness.

Nb samples



High RRR Nb sample 5x5x2.7 mm³

ADOMBE @ ATFT



The ADvanced Oxide Molecular Beam Epitaxy (ADOMBE @ ATFT) system [3] (**right**) is made for complex material depositions. The custom built system contains the latest generation of MBE equipment and is specifically designed for multi-cation materials. It is also equipped with RF **atom** source for strong oxidation (atomic oxygen), or other gases (**N**, Ar).

N-doping of Nb samples (@ ADOMBE)

As a reference to later work, **virgin** Nb samples were baked out in the ADOMBE chamber [2] @ ATFT. The first reference sample was annealed at **850 °C** for 4 h, maximal chamber pressure: $6.8 \cdot 10^{-7}$ mbar. The second sample was annealed at **1027 °C** for 4 h, maximal chamber pressure $5.0 \cdot 10^{-7}$ mbar. The **N-doped** reference sample was annealed at 965 °C for 80 min in $2 \cdot 10^{-6}$ mbar nitrogen atmosphere, with an *atomic nitrogen* RF-source pointing at the sample surface.

The SIMS measurements (Fig. 1) showed less hydrogen and carbon in the annealed samples, compared to the virgin one. The level of nitrogen also increased in the **N-doped** reference sample.

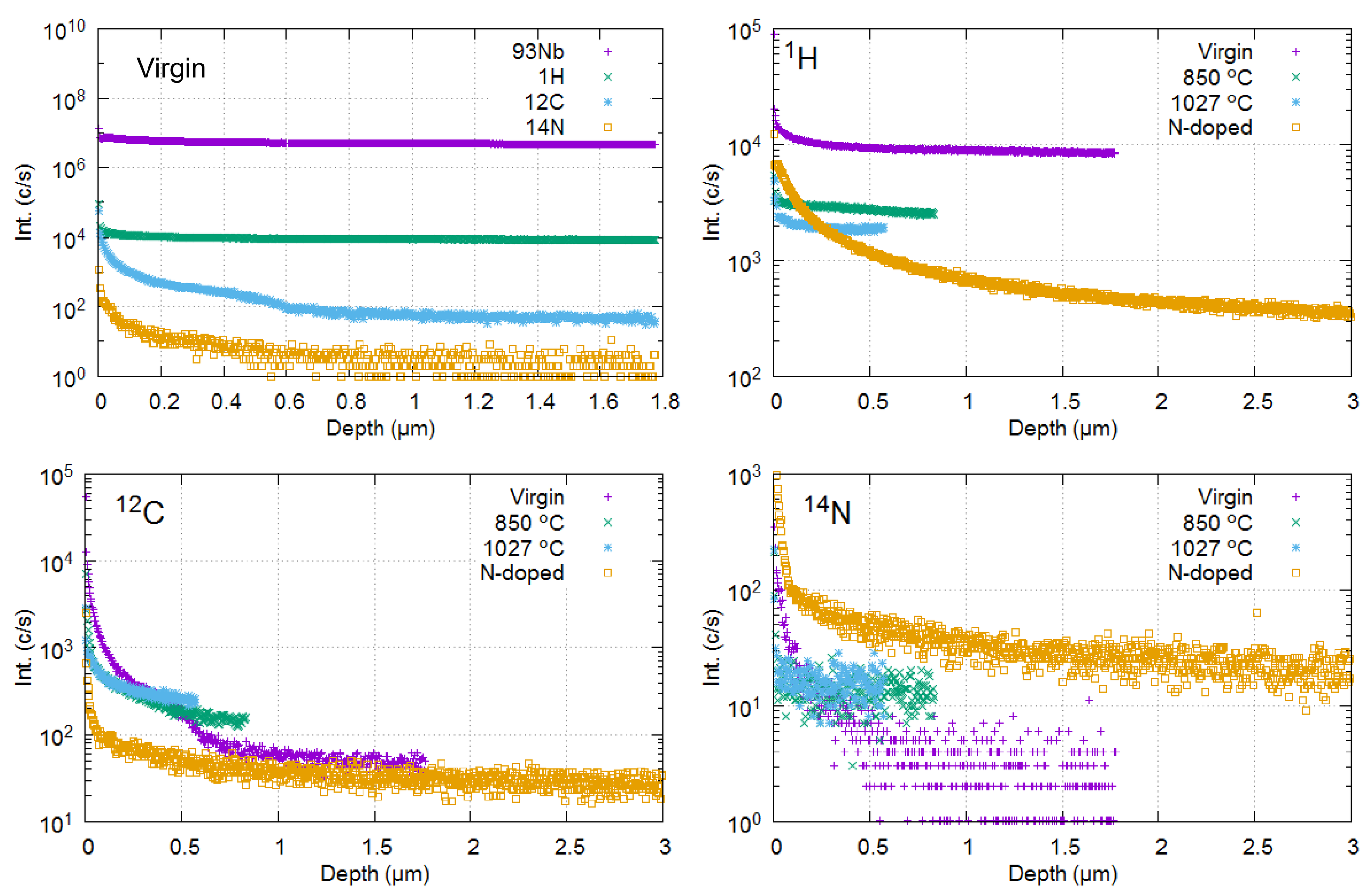


Figure 1. Secondary Ion Mass Spectrometry elemental depth profiles.

XRD

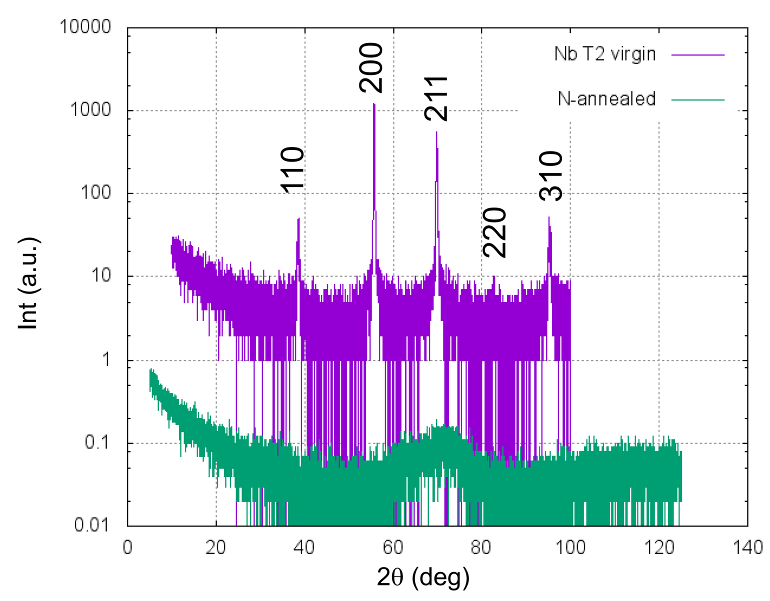


Figure 3. X-ray diffraction (XRD) pattern of the reference Nb sample before (**top**) and after N-doping (**bottom**) in the ADOMBE chamber. The curves are shifted vertically for clarity.

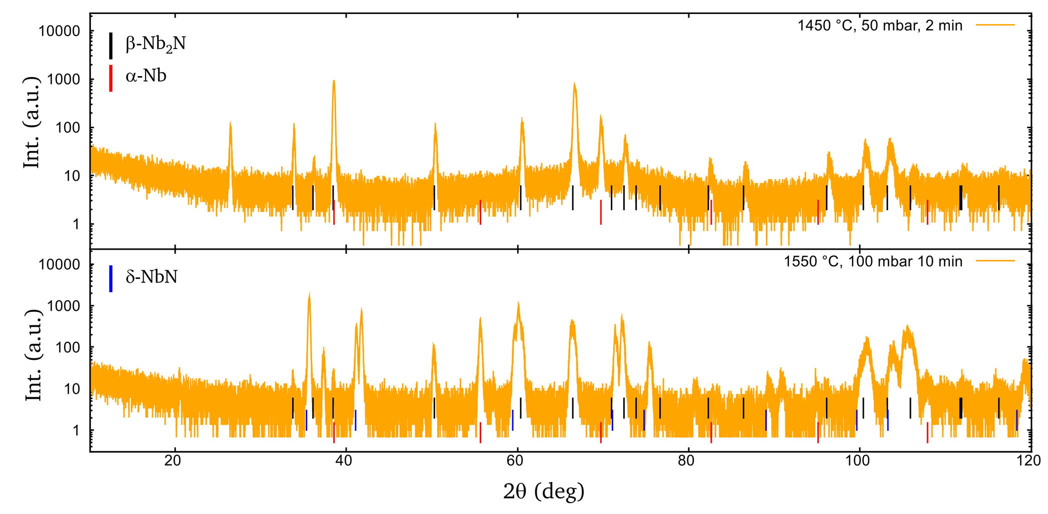


Figure 4. X-ray diffraction pattern of nitrogen doped Nb samples. The nitridation took place in the Wuppertal oven @ IKP. The Bragg peaks related to different crystal phases are noted by coloured tick marks.

Pole figures

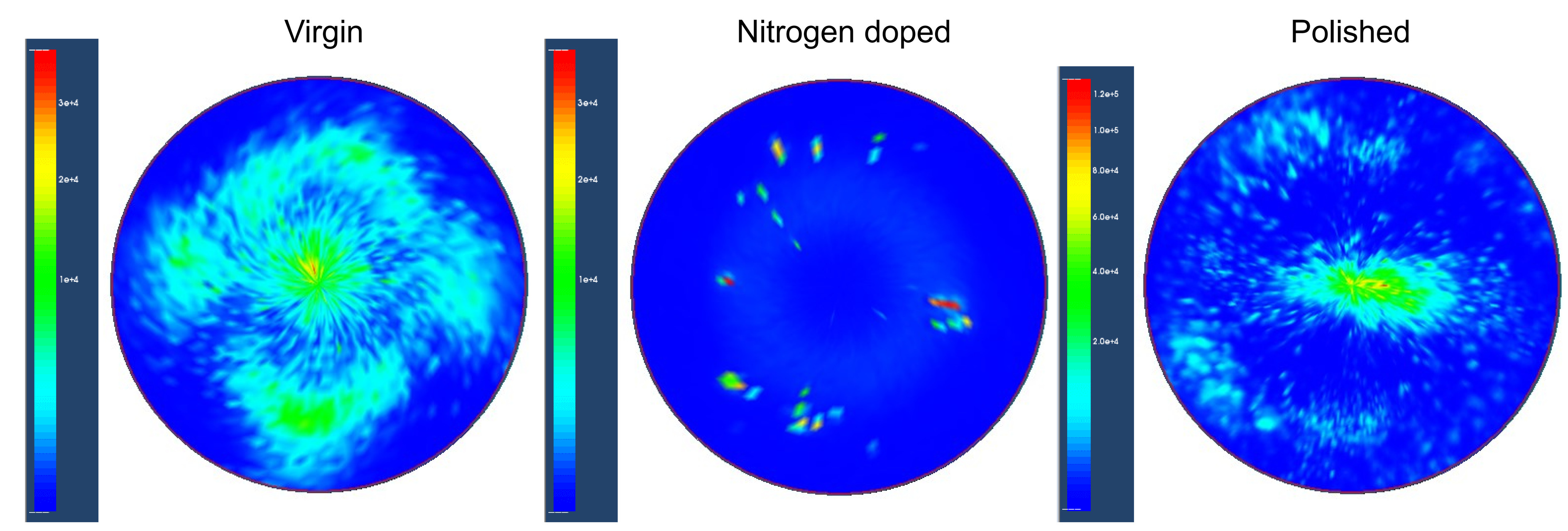


Figure 5. Pole figure of the Nb 200 reflection (2θ = 55.7°) of a Nb sample before (**left**) and after nitrogen doping (**middle**) at 1200 °C @ ADOMBE. On the **right** the pole figure of a polished sample is shown. The intensity is represented on square root scale (colour bar).

N-doping of Nb samples (@ Wuppertal-oven)

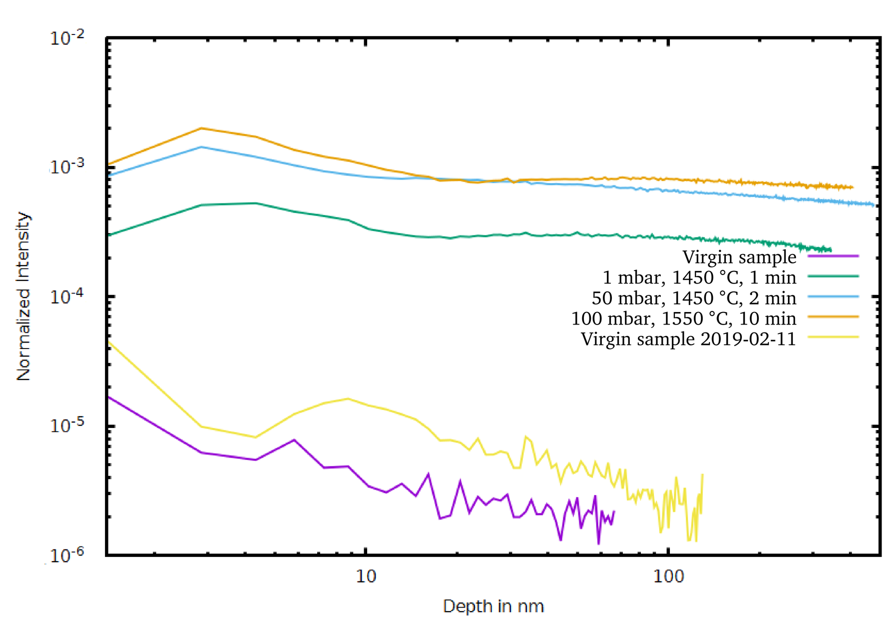


Figure 2. SIMS depth profiles of ^{14}N normalised to ^{93}Nb measured on the Nb samples with O^{2+} ions.

The **virgin** Nb samples were baked out in the *high-temperature UHV furnace* (Wuppertal oven @ IKP) [1]. Samples were annealed in vacuum and in nitrogen atmosphere up to 100 mbar, 1550 °C, 10 min (Fig 2). The nitrogen pressure was kept constant by adjusting the entrance valve of the hot-pot (the niobium inner walls of the sample chamber adsorbed nitrogen at high temperatures).

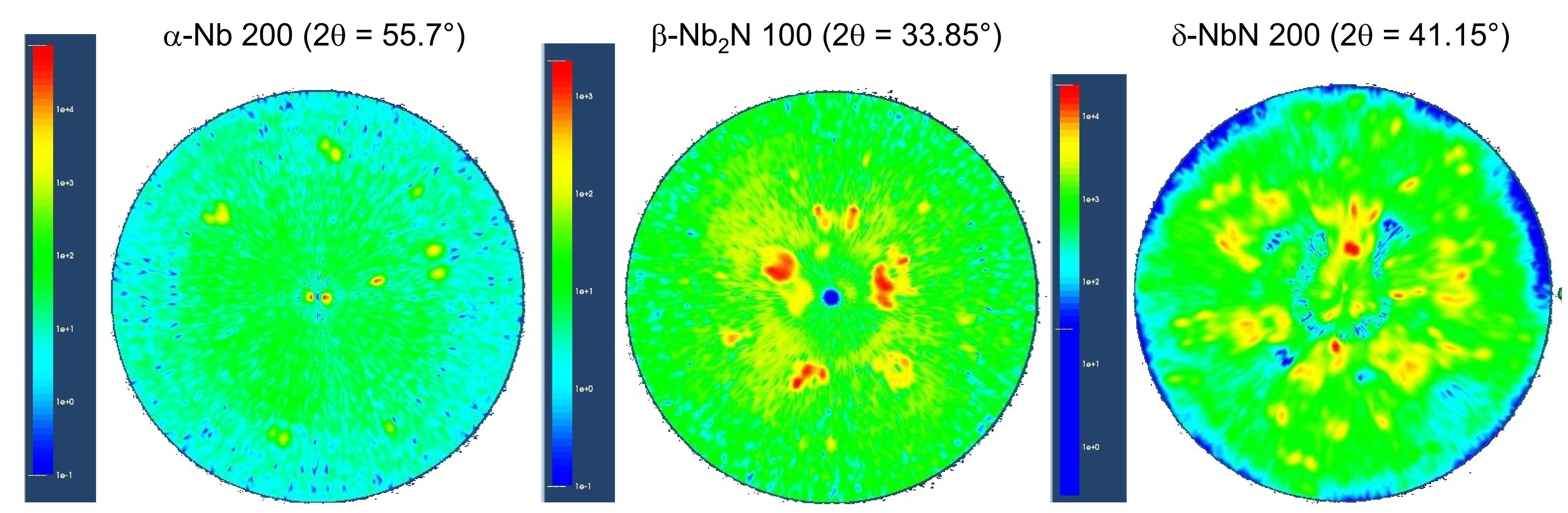
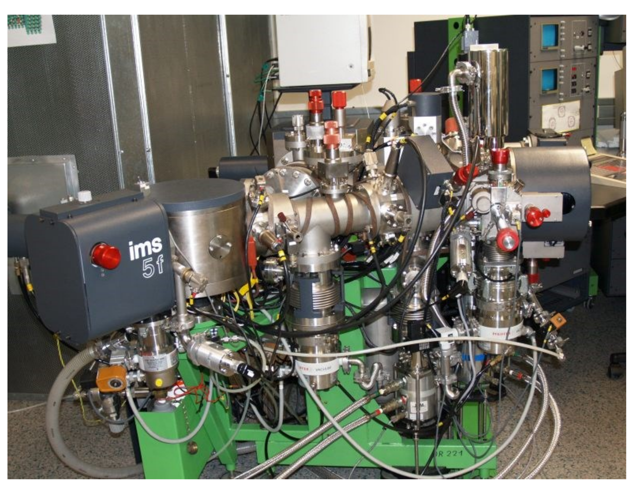


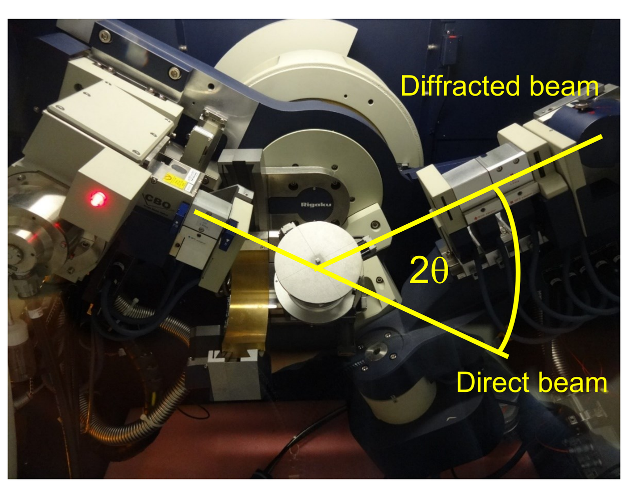
Figure 6. Pole figures of the different phases of a nitrogen doped sample. The sample was annealed in the Wuppertal oven @ IKP [1] at 1550 °C in 100 mbar N_2 atmosphere for 10 minutes [4]. Bragg peaks related to different phases were selected: α-Nb (**left**), β-Nb_2N (**middle**) and δ-NbN (**right**). The intensity is plotted on a logarithmic scale (colour bar).

SIMS (Cameca ims5f)



Secondary Ion Mass Spectrometer (SIMS)
Charged atomic and molecular species are ejected from the uppermost layers of a surface under ion bombardment. These secondary ions can be mass separated and detected.
Primary ions: oxygen, argon or cesium.
Detection limit: down to ppb.

Texture measurement



For texture measurement a pole figure is taken. Here the detector angle (2θ) is fixed while the sample is rotated and tilted. The resulting graph, the pole figure, shows the 3D orientation and distribution of a given lattice plane. The plane (distance in reciprocal space) is selected trough the 2θ detector angle and Bragg's law.

Results

Nb samples were annealed and N-doped in high-temperature UHV ovens (ADOMBE and Wuppertal). With the updated doping protocol N-diffusion was observed (Fig. 2). The SIMS measurements also showed less hydrogen and carbon doping in the annealed samples (Fig. 1). The annealed Nb samples showed changes in the microstructure (Figs. 5-6). According to the pole figures the grains of the initially already textured Nb surfaces have grown into a few larger ones, changing drastically the pole figure. Simple polishing also changed the texture. Different NbN phases showed different texture, a hint on different crystallite size. The change in microstructure could have effect on the cavities physical performance.

Acknowledgement

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