RECENT FININGS ON NITROGEN TREATED NIOBIUM

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Abstract

Based on recent findings at Fermilab, Cornell investigated the role of Nitrogen being present during the cavity hydrogen degassing process. We treated several samples at different temperatures being exposed to nitrogen between 10 minutes and 3 hours at pressures around 15 mbar. This contribution will summarize our findings from surface analysis and Tc measurements addressing the question, if such a process can form Niobium-Nitride.

INTRODUCTION

During many decades, materials other than Niobium have been investigated for RF applications [1,2]. Due to the progress of the pure Niobium technology, many activities stopped during 1990s. However, as todays Niobium cavities are reaching closely their limits, alternative material with higher theoretical potential become interesting again [3]. Among them are Nb_3Sn, which Cornell started to work on several years ago (and which now lead to a great success), MgB_2 which is currently investigated in Los Alamos and NbN with several institutes involved [4].

While Nb_3Sn has turned into a success story [5], the situation with NbN is still unclear. From the theoretical standpoint, a NbN cavity should reach fields as high as a Nb cavity but with quality factors of above 10^{11} (for a 1.3 GHz cavity at 2 K) [6]. Nonetheless, the performance of different NbN cavities produced so far was rather poor and there is a great doubt that current coating techniques are suited.

Based on this, a process was investigated which was applied by the Fermilab group. There, a Niobium cavity was fired in a vacuum furnace to 1000 C while nitrogen (~15 mbar) was introduced for some minutes. This resulted in a remarkable high Q cavity after a 70 µm BCP removal- while the cavity performed poorly prior to the BCP [7]. We closely followed this heating procedure and addressed the question, if Niobium Nitride could be formed under these circumstances. We especially addressed the question, if the Niobium Nitride could have been formed under the surface layer.

PREPARATION OF THE SAMPLES

All Niobium samples were baked in a vacuum furnace for 50 hours at 1000 C to ensure a proper Hydrogen degasing. Then the samples individually were heated up again to 1000 C with a rate of 50 C/min under vacuum conditions. Once the temperature was reached, ultrapure Nitrogen was bleed in, maintaining a pressure of 15 mbar. The exposure time was varied from sample to sample, ranging from 10 min up to 3 hours. The exposure time was followed by a quick cool-down being technically limited by the oven. Down to 600 C, the rate was 30-40 C/min.

SET-UP TO MEASURE THE CRITICAL TEMPERATURE

To characterize the samples treated as described above, its fundamental superconducting property was measured, namely the critical temperature (Tc). The idea behind this approach was the following: as there is the suspicion that NbN was formed during the process, the critical temperature of the Nitrogen treated Niobium sample should raise from 9.2 K to around 16 K. This layer might be buried under the surface, so we choose to measure Tc with and induction set-up. This would allow the detection of an NbN layer even if it is not located on the surface. However, the limitations are clear: if there is no closed layer, the method would fail.

Our set-up consists of two coils located on either side of the sample under test. Driving one coil with an AC of 200 Hz, a voltage should be induced in the pic-up coil if the magnetic field of the two solenoids couple to each other. This is the case, if the sample placed in-between is normal conducting; if it is superconducting, the magnetic field of the exited coil would be shielded due to the Meisner effect and the pic-up coil would not experience any induced currents.

In practice, the electric shielding effect generates eddy currents in the normal conducting state, too, so a carful experimental set-up has to be chosen. We used a log-in amplifier and shielded current leads to minimize the noise. However, choosing the right parameters was still

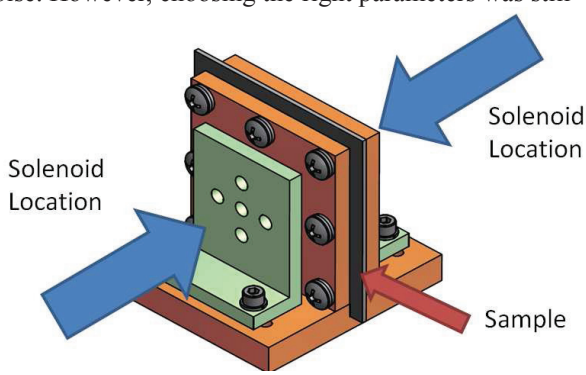

Figure 1: Sketch of the inductive Tc measurement apparatus. The sample under test is placed between two coils decoupling their magnetic field if the sample is in the superconducting Meisner state.

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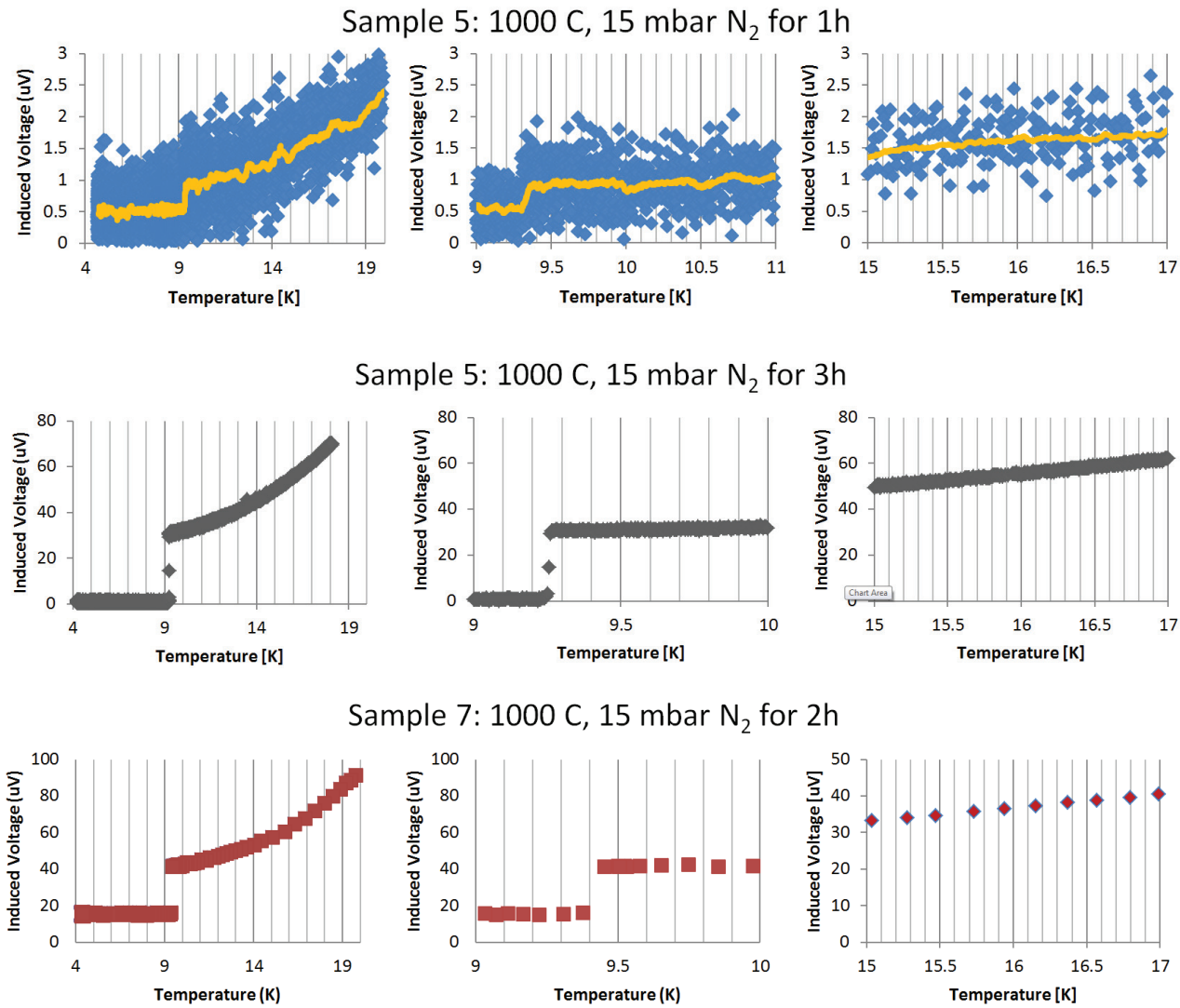

Figure 2: Induced induction voltage in the pic-up coil (measured with a log-in amplifier) as a function of temperature for the different samples. Sample 5 was measured with a non-optimal set of parameters, but averaging the data clearly indicates the transition temperature. The left plots show the full data sets, the plots in the middle magnify the 9.5 K region where Niobium becomes superconducting, the 16 K regions are zoomed into on the right hand indicating, where signatures of a Niobium Nitride transition should be found.

difficult, as can be seen from the first data we took, shown in fig 2, upper plots.

TC MEASURMENTS

So far, three of the samples treated as described above were measured against their critical temperature: Sample 1 was exposed 3 h to the nitrogen atmosphere, sample 7 for 2 h and sample 5 for 1 h.

These samples – one by one – were mounted between the solenoids (see fig. 1 for reference), enclosed by a copper block with two cernox resistors attached to it to ensure an accurate temperature calibration. After immersing the block into a small vertical dewar it was cooled down with liquid helium until it was fully covered.

As soon as the liquid was evaporated, the copper block warmed up slowly by itself due to the omic heating of the sending solenoid. During this phase, the induced voltage on the receiving coil was monitored. Initially, as the sample was superconducting no voltage could be detected which changed during the warm-up and the onset was interpreted as the critical temperature. The data taken is shown in fig. 2. Going from 4 K to 20 K typically took 20 minutes.

For all three samples, no indication of a closed superconducting layer inside the niobium substrate with a higher critical temperature than niobium could be observed.

SURFACE ANALYSIS

In addition to measuring the critical temperature we analyzed the surface by methods of spectroscopy. As the inductive method suggested that there is no closed layer of Niobium Nitride we addressed the question if islands.

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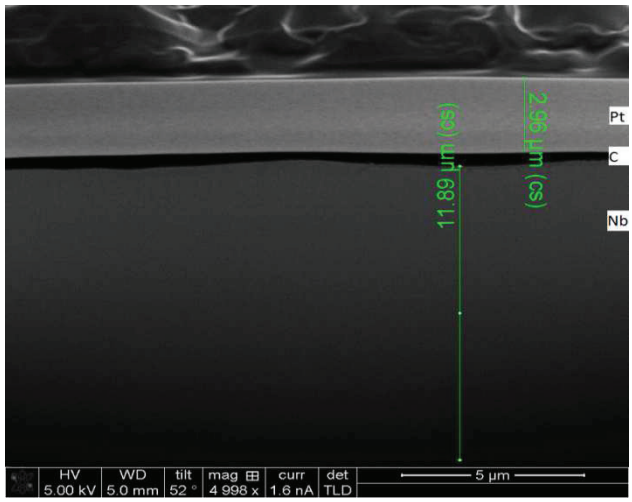

Figure 3: Focused Ion Beam Microscopy (FIB) image of the cross section of sample #7 (120 min Nitrogen exposure).

of NbN may exist or if there are other indications of Nitrogen, diffused into the Niobium forming low Nitrogen Niobium-Nitrogen phases.

Therefore, sample #7 was scanned with a Focused Ion Beam Microscopy (FIB), see fig. 3. Below the Platinum and Carbon layer (which was coated onto the sample to increase the contrast) a uniform Niobium layer was detected. Figure 4 gives a deeper cut into the material showing no indication of a Niobium Nitride phase.

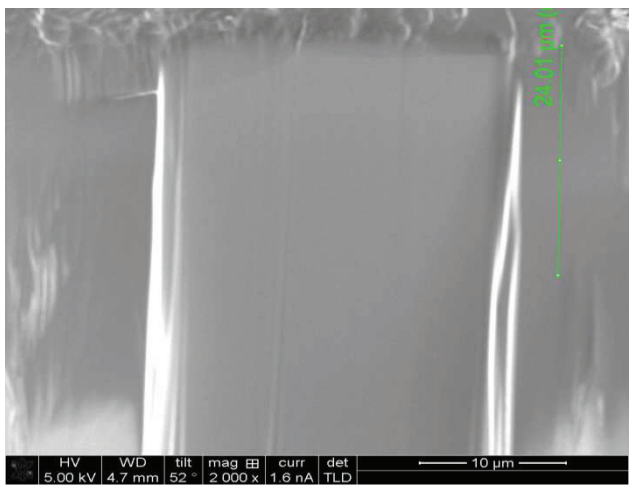

Figure 4: FIB image of sample #7 with a depth greater than 24 µm. Again, no nitride layer could be seen.

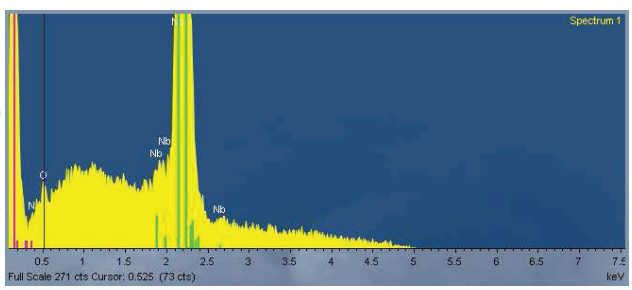

Figure 5: EDX spectrum of sample #7 showing unpopulated Nitrogen lines.

Table1: Atomic Decomposition Analysis from the EDX

Element	Weight%	ΔWeight%
C	18.78	0.61
N	-2.19	0.58
O	3.05	0.23
Si	0.49	0.04
Nb	79.87	0.77
Totals	100	

For further analysis, the sample was analysed with an EDX spectrometer. To increase the sensitivity for Nitrogen, the electron energy was kept as low as 5 keV. The spectrum taken is shown in fig. 5, tab. 1 summarizes the atomic composition analysis.

CONCLUSION

We addressed the question, if Niobium at 1000 C, exposed to a 15 mbar atmosphere of Nitrogen can react thermally to δ-NbN. Our inductive measurement of the critical temperature suggests that there is no closed layer of NbN inside Niobium samples treated this way. Surface analysis by means of FIB and EDS showed no signature of Nitrogen inside the Niobium which let us conclude that no Niobium Nitrite was formed or remained during the cool-down process.

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