ISBN: 978-3-95450-184-7 doi:10.18429/JACoW-IPAC2018-THPAL126

NITROGEN BAKE-OUT PROCEDURES AT THE VERTICAL HIGH-TEMPERATURE UHV-FURNACE OF THE S-DALINAC *

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Abstract

As the performance limits of bulk Nb SRF cavities are reached, our research is focused on materials with superior SRF properties like Nb_3Sn and NbN. Research on NbN resulted in the "nitrogen-doping" process used for increasing the quality factors of SRF cavities for the LCLS-II project. This process leads to α-phase Nb-N, a phase with higher critical superconducting (sc) parameters than bulk Nb. This phase is formed at temperatures of 800°C in nitrogen atmosphere of 10^{-2} mbar. Other crystalline phases of NbN have even better sc parameters. We concentrate our research on applicability of δ-phase NbN for cavities. The δ-phase forms at temperatures of above 1300°C, which is more than most of the furnaces at accelerator facilites are capable of. Since 2005 the Institute for Nuclear Physics at the Technische Universität Darmstadt operates a high temperature vacuum furnace which has been upgraded to allow temperatures of up to 1750°C and bakeouts of niobium samples and cavities in nitrogen atmospheres. We will report on the current status of our research on nitrogen bake-out procedures on Nb samples. The samples have been analyzed at the Materials Science Departement with SIMS, REM and XRD.

INTRODUCTION

Today's state of the art SRF technology allows for high quality factors due to SRF cavities made of niobium. Prior to installation the cavities get a 800°C hydrogen bake-out with a subsequent nitrogen treatment [1], so called "nitrogen doping". At even higher temperatures in the range between 1300°C and 1700°C the δ-phase of NbN forms [2], as shown in the phase diagram in Fig. 1. The δ-phase is highly interesting for superconducting accelerator technology applications. Due to different nitrogen concentrations along the depth of the niobium, different phases of NbN form. In Fig. 2 the microstructure of NbN along the depth profile is shown.

UHV-FURNACE

In 1983 the University of Wuppertal built a high temperature UHV furnace [5]. The furnace was brought to the S-DALINAC [6] at TU Darmstadt in 2004 and has been used for hydrogen bake-out of the 20-cell SRF cavities several times with proven success [7]. In Fig. 3 a schematic cross section view of the UHV furnace is shown. The samples or cavities are heated in a niobium hot-pot, which has its own vacuum system to keep the samples and cavites clean from possible contaminations. It reaches temperatures of up to 1750°C at pressures $< 10^{-4}$ mbar. At a temperature of $\approx$ 1450°C the pressure is of the order 10^{-6} mbar. In 2016, the furnace has been upgraded to allow heat treatments of Nb samples and SRF cavities in a nitrogen atmosphere [8].

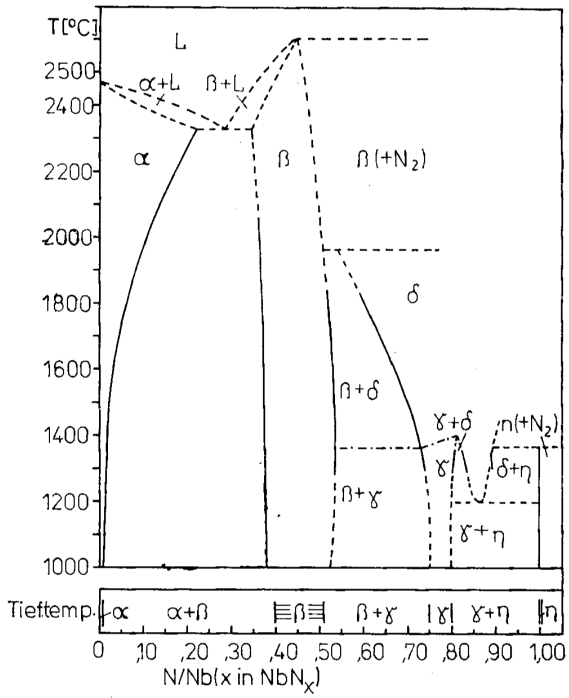

Figure 1: Phase diagram of NbN. The δ-phase of NbN forms at temperatures between 1300°C and 1700°C [3].

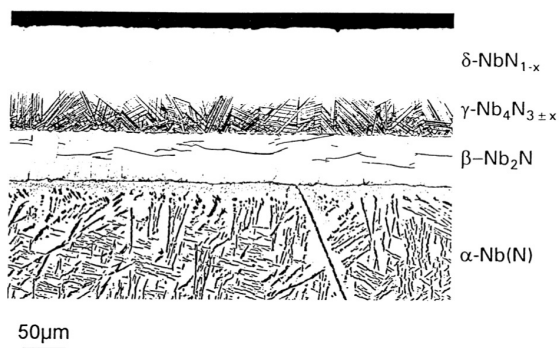

Figure 2: Cross section of the microstructure of NbN. The superconducting δ-phase forms at the highest nitrogen concentration at the top, followed by the normalconducting β-phase and the superconducting α-phase at the bottom. During cooldown the δ-phase transforms into the superconducting γ-phase [4].

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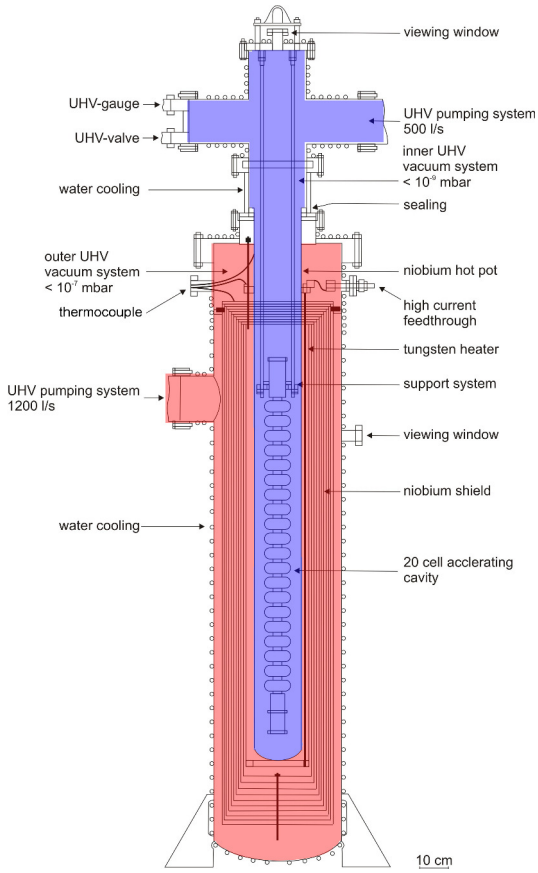

Figure 3: Cross section of the UHV-furnace. The inner hot-pot vacuum vessel is shown in blue, the insulating vacuum in red [9].

N_2 CONDITIONING

The vacuum and N_2 inlet schematic is shown in Fig.4. The inlet is used for inital vacuum pumping with a rotary pump and a turbomolecular pump, before an ion getter pump takes over. Due to the layout of the furnace, it is not possible to let N_2 in and, at the same time, pump for a flow N_2 through the hot-pot of the furnace. Instead, a scheme with multiple, subsequent N_2 inlets can be used, as shown in Fig. 5. Alternatively, the N_2 inlet can be kept open, as Nb is a very good getter material for N_2 at temperatures of 1400°C. In Fig. 6 a continuously N_2 flow with a resulting hot-pot pressure of $2 \cdot 10^{-2}$ mbar is shown. After treatment, the samples are analyzed at the Materials Science Department of TU Darmstadt. The N_2 depth profile is measured with SIMS (Secondary Ion Mass Spectrometry) using O_2^+ ions. For better comparison of the samples, a normalization has been done using a mean value of the ^{93}Nb measurement. The resulting N_2 depth profile is shown in Fig. 7 for a virgin sample, a sample treated with subsequent N_2 inlets and continuous N_2 flow. The difference in the N_2 content is evanescent. An explanation is the hot-pot made of Nb absorbing most of the N_2 before it reaches the Nb samples. A furnace upgrade introducing a pipe to the sample position is planned to reduce the risk of losing the N_2 to the hot-pot.

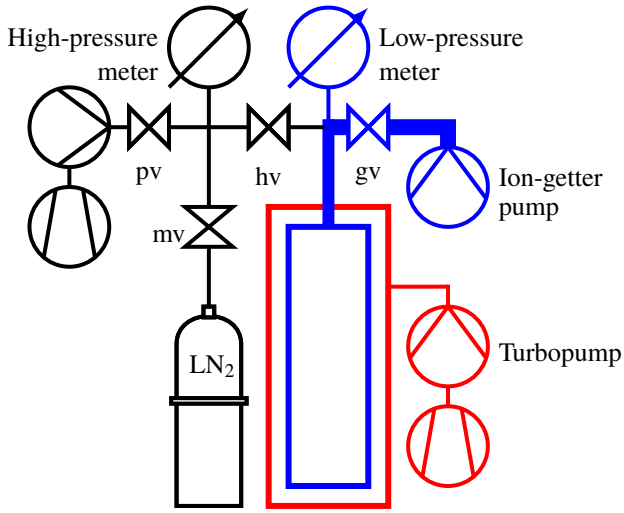

Figure 4: Schematic drawing of the vacuum system. The hot pot vacuum (blue), is pumped by a ion getter pump through a gate valve (*gv*), the insulating vacuum (red) by a turbomolecular pump (*tp*). For the initial pumping of the hot-pot, a second *tp* is used (black), seperated by a valve (*pv*) and an all-metal valve (*hv*). The N_2 inlet is controlled manually by a metering valve (*mv*).

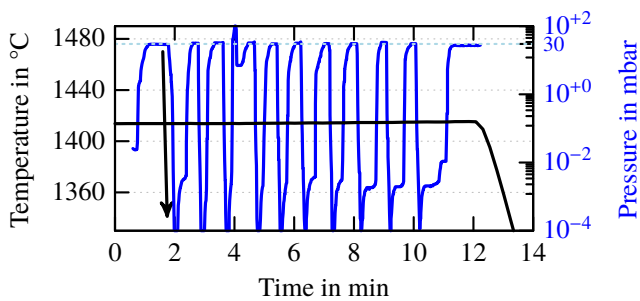

Figure 5: Furnace temperature (black) and pressure (blue) during subsequent N_2 inlets. *gv* is closed. The vacuum cross of the N_2 inlet (Fig. 4) is filled through the *mv* with N_2 until the desired pressure (here 30 mbar) is reached. *hv* is opened, and N_2 flows into the hot-pot (black arrow). The pressure drops below the lower limit of the pressure meter.

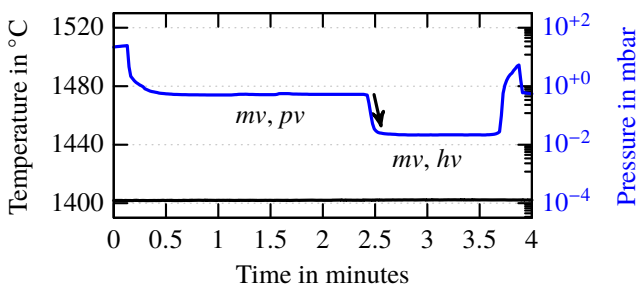

Figure 6: Furnace temperature (black) and pressure (blue) during continuously N_2 inlet. *gv* is closed. The N_2 flow is set indirectly by pressure (here $5 \cdot 10^{-1}$ mbar) reading with *mv*, *pv* opened. *pv* is closed and *hv* opened (black arrow) to allow flow into the furnace. The pressure reads $2 \cdot 10^{-2}$ mbar.

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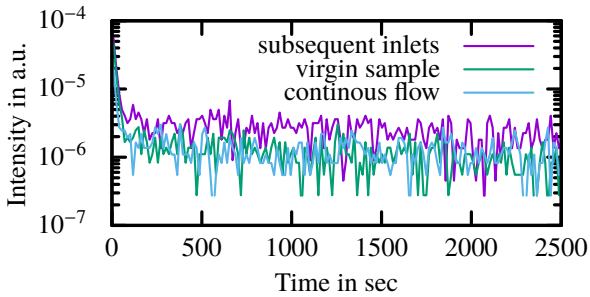

Figure 7: SIMS profile measurement of N_2. Two samples treated with different N_2 inlet schemes are compared to a virgin sample. The furnace temperature of (1400 ± 10)°C has been held for 4 h for all samples before treatment.

VERTICAL BATH CRYOSTAT UPGRADE

The samples are used to find a N_2 conditioning prodecure which leads to the growth of δ-phase NbN at the sample surface. This procedure is then applied to 3-GHz single-cell cavities made of niobium. The quality factor Q_0 and accelerating field E_{acc}, can then be measured using the vertical bath cryostat at the S-DALINAC [10]. The vertical bath cryostat is cooled down using liquid helium and allows measurements at temperatures between 2 K and 4 K. The vertical bath cryostat will be upgraded to reduce measurement uncertainties induced by a large coupling factor using a variable input coupler. This allows to match the external quality factor Q_{ext} of the input coupler to Q_0 of the cavity and thereby reduce the uncertainties. The input coupler design has been simulated using *CST Microwave Studio* [11] to maximize the transmission coefficient S_{31}. A field map of the cross section of the coupler is shown in Fig. 8, indicating simulation ports. Between the HN-Type vaccum feedthrough at *Port 1* and *Port 2* a geometric size matching between the vacuum feedthrough and the cut-off pipe of the cavitiy was optimized by maximizing the transmission coefficient to $S_{21} = -0.038$ dB (99.1 %). This leads to an overall transmission coefficient of $S_{31} = -0.242$ dB (94.6 %).

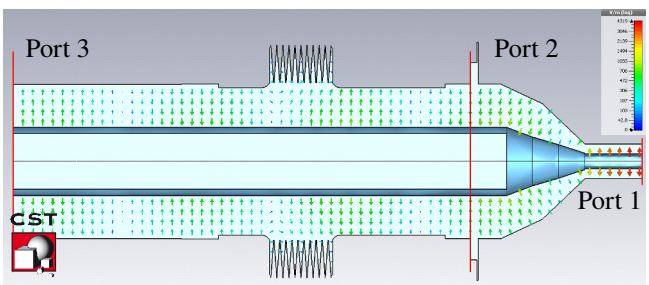

Figure 8: Cross section view of the CST MWS simulation of the variable input coupler for the vertical bath cryostat. The scattering matrix components have been calculated for different port combinations. The port locations are indicated by the red lines.

SUMMARY AND OUTLOOK

The upgraded furnace does work well with different N_2 conditioning schemes. So far we did not find any traces of N_2 in the treated samples. We will continue our work with higher N_2 pressures and longer conditioning times. Further upgrading the furnace will allow us to inject the N_2 at the position of the Nb samples and later single-cell cavities. For measurements of the figures of merit of the single-cell SRF cavities the vertical bath cryostat is enhanced by a variable input coupling, allowing low measurement uncertainties.

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