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INTRODUCING THE VERTICAL HIGH-TEMPERATURE UHV FURNACE OF THE S-DALINAC FOR FUTURE CAVITY MATERIAL STUDIES*

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Abstract

Since 2005 the Institute for Nuclear Physics in Darmstadt operates a high temperature UHV furnace for temperatures of up to 1750°C. It has been used several times for hydrogen bake-out of the SRF cavities of the S-DALINAC with proven success. In 2013, studies at FNAL have shown that cavities treated with nitrogen reached an up to four times higher q-factor. The cavities are exposed to N_2 at 850°C at the end of the H_2 bake-out. A thin layer of normal conducting (nc) hexagonal niobium nitride (NbN) forms at the surface which is removed by electropolishing while the higher quality factors are attributed to the N_2 diffusion into the bulk Nb. At temperatures from 1300°C to 1700°C a thin layer of the superconducting (sc) cubic phase of NbN can be observed, e.g. δ-phase NbN, which has a higher critical field and higher critical temperature and thus is very intereresting for applications for SRF cavities. The UHV furnace has been prepared for future treatments of Nb samples and cavities in an N_2 atmosphere at high temperatures for research on cubic NbN. The material properties of the samples will be analyzed at the ATFT group at the Department for Materials Science of TU Darmstadt.

INTRODUCTION

Research on doping of niobium cavities with nitrogen at temperatures of 850°C results in an up to four times higher quality factor compared to untreated cavities [1]. 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

The UHV-furnace at the S-DALINAC [5] was built at the University of Wuppertal in 1983 [6] and moved to Technische Universität Darmstadt in 2002. It was designed to reach temperatures of up to 1800°C with vacuum pressures lower then 10^{-4} mbar. Since 2005 it has been used for hydrogen bakeout of several superconducting niobium cavities at 850°C with proven success [7]. Due to technical constraints at TU Darmstadt the temperature was limited to 850°C. Beginning in 2015 the furnace has been upgraded

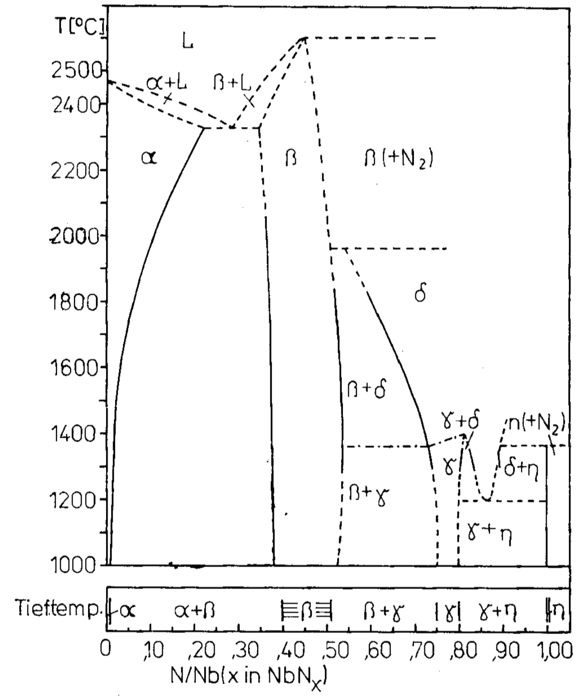

Figure 1: Phase diagram of NbN. The δ-phase of NbN forms at temperatures between 1300°C and 1700°C [3].

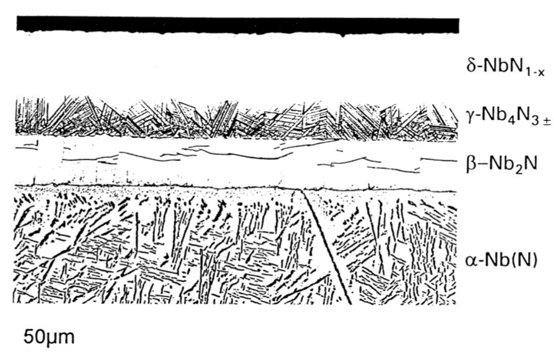

Figure 2: Cross section of the microstructure of NbN. The sc δ-phase forms at the highest nitrogen concentration at the top, followed by the nc β-phase and the sc α-phase at the bottom. During cooldown the δ-phase transforms into the sc γ-phase [4].

and recommissioned to operate at temperatures of up to 1800°C again [8]. The cross-section in Fig. 3 illustrates the main parts of the furnace [9]. The inner part of the UHV-furnace is a hot-pot made of niobium. The niobium samples

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or srf cavities are held by a niobium support system, which is mounted at the top of the furnace. Heat-shields made from ten layers of niobium sheets around the hot-pot minimize the radiation heat flow from inside the furnace to the outer water cooled casing.

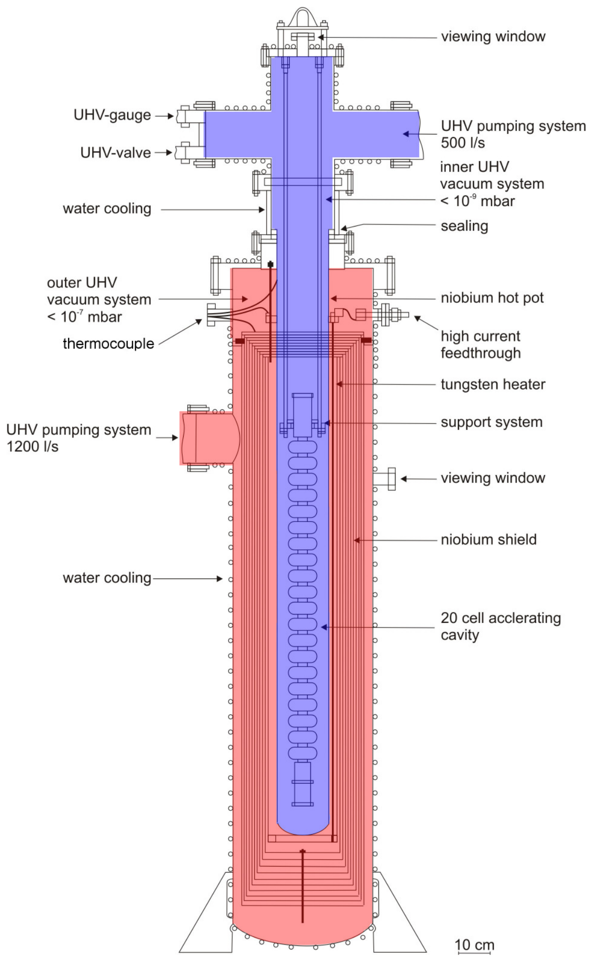

Figure 3: Schematic drawing of the UHV-furnace. The inner hot-pot vacuum vessel is shown in blue, the insulating vacuum in red.

The two vacuum systems, an outer insulating vacuum and a separate hot-pot vacuum to reduce contamination of niobium samples or cavities, are shown in Fig. 3. The hot-pot pressure is below 10^{-4} mbar. A turbomolecular pump and an ion-getter pump are used for the insulating and hot-pot vacuum, respectively.

The furnace is heated by three tungsten heaters. They are placed around the outside of the hot-pot. The current for the heaters is supplied by a power supply with an input power of up to 40 kW. All materials are carefully selected to meet the high operating temperature: The electrical insulating is made of Al_2O_3 with a maximum operating temperature of 1900°C. Other materials used are niobium with a melting point of 2477°C and tungsten with a melting point of 3422°C. The temperature is measured outside of the hot-pot with thermocouples of type C. For temperature correction a Pt100 RTD is located inside the vacuum feedthrough. Readings of pressure gauges, temperature sensors and current meters are recorded digitally. It is possible to attach a mass spectrometer to the hot-pot vacuum for residual gas analysis.

The temperature of the furnace is controlled indirectly by adjusting the electric voltage across the tungsten heaters, and thus changing the current through them. In Fig. 4 the vacuum and temperature trends are shown for a heat run. The furnace reached a temperature of 1750°C with a current of 320 A for each tungsten heater. The vacuum pressure in the hot-pot was below $2 \cdot 10^{-4}$ mbar.

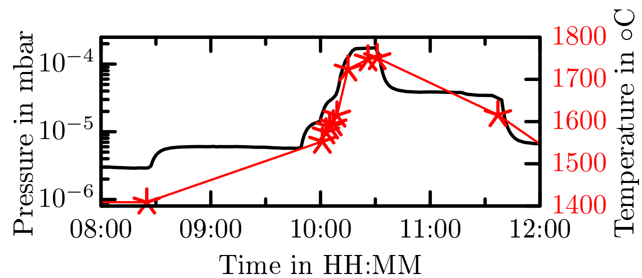

Figure 4: Relation between the temperature (red) near the hot-pot and the hot-pot pressure (black) over time of day.

SAMPLE PROCESSING

Niobium samples of the size 5×5 mm^2 with a thickness of 2 mm have been processed at different temperatures to obtain a baseline for comparison with nitrogen processed samples. Additionally it gives a hint to which impurities might be caused by the furnace. The Nb samples are put into the UHV-furnace on a niobium holder. The furnace temperature is raised slowly to keep vacuum pressures low (Fig. 5) until the required temperature is reached. This temperature is kept for 4 hours after wich the power is cut off. The furnace needs 40 minutes to cool down from 1400°C to 600°C.

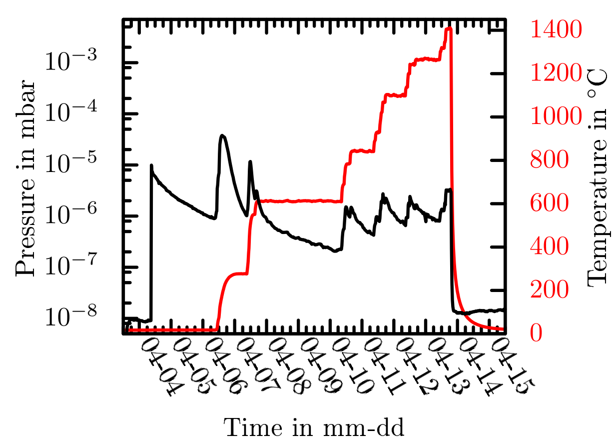

Figure 5: Example of the vacuum pressure and temperature trend for a sample processed at 1400°C.

Element depth profiles are obtained using SIMS (Secondary Ion Mass Spectrometry). In Fig. 6 the SIMS profiles for samples processed at different temperatures are shown.

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Further analysis comprises X-ray diffraction (XRD) and scanning electron microscopy (SEM).

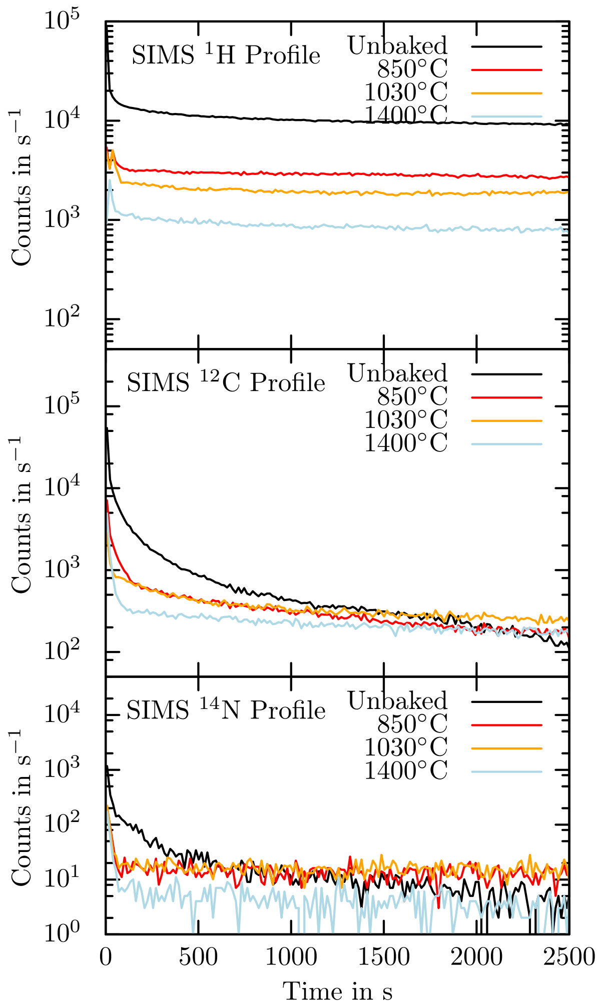

Figure 6: SIMS depth profiles of impurities for different temperatures. Compared to the unprocessed sample (black) the impurities get pushed out. Note that these measurements only allow a qualitative comparison.

To investigate the effect of nitrogen doping and the growth of δ-phase NbN a nitrogen inlet has been built with off-the-shelf components. Because of the general layout of the furnace with a long, tight hot-pot, it is difficult to reach the desired nitrogen atmosphere of 10^{-2} mbar with a steady flow of nitrogen. Instead, a known amount of nitrogen is supplied. In Fig. 7 the supply for nitrogen is shown. A Pirani-type pressure gauge has been selected for the nitrogen inlet with an accuracy of 10 %.

CONCLUSION AND OUTLOOK

The implemented nitrogen supply system is going to be tested. Niobium samples will be processed at different temperatures and nitrogen pressures. They will be investigated at the department of Materials Science of Technische Universität Darmstadt to find a good process for later treatments of single cell cavities. The quality factors of the single cell

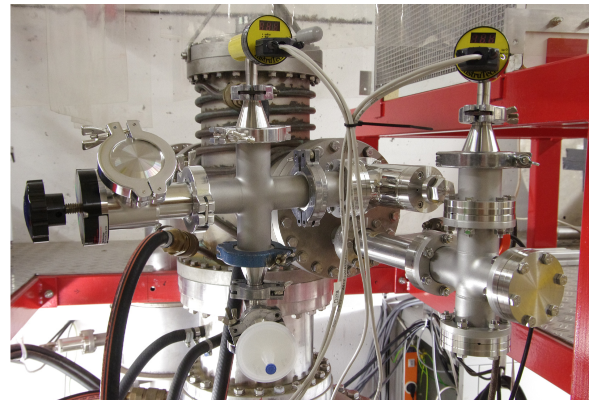

Figure 7: Nitrogen supply system at the top of the furnace. As only a small volume is required, it consists of a KF40 cross in the center with attached periphery: At the top a Pirani vacuum gauge, on the left a vacuum valve for a vacuum pump, at the bottom a nitrogen inlet with particle filter.

cavities can be measured at different temperatures at the vertical bath cryostat at the S-DALINAC.

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