PREPARATION FOR CAVITY MATERIAL STUDIES AT THE VERTICAL HIGH-TEMPERATURE UHV-FURNACE OF THE S-DALINAC*

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Abstract

Since 2005 the Institute for Nuclear Physics at the Technische Universität Darmstadt operates a high temperature vacuum furnace. It is designed to reach temperatures of up to 1800°C. It has been used for baking out several niobium superconducting rf cavities at 850°C with proven success. Current research for improving the performance of superconducting rf cavities is focused on nitrogen treatment of such cavities. Nitrogen doping of srf cavtities results in an up to four times higher quality-factor as compared to untreated cavities. At higher temperatures between 1300°C and 1700°C the δ-phase of NbN forms, which is highly interesting for applications to superconducting accelerator technology. The UHV-furnace at the S-DALINAC offers the possibility to treat niobium samples at considerably higher temperatures than what has been done up to now in order to study the effect of delta-phase NbN and N-doping on superconducting properties. The furnace has been refurbished and recommissioned to realize research on nitrogen treatment of niobium samples.

INTRODUCTION

The UHV-Furnace at the S-DALINAC [1] was built at the University of Wuppertal in 1983 [2] and moved to Technische Universität Darmstadt in the year 2002. It was designed for temperatures of up to 1800°C with vacuum pressures lower then 10^{-6} mbar. Since 2005 it has been used for hydrogen bakeout of several superconducting niobium cavities at 850°C with proven success [3]. Due to technical constraints the temperature was limited to 850°C. Beginning in 2015 the furnace has been upgraded and recommissioned to operate at temperatures of up to 1800°C again [4].

Research on doping of niobium cavities with nitrogen at temperatures of 850°C results in a up to 4 times higher quality factor compared to untreated cavities [5]. At even higher temperatures in the range between 1300°C and 1700°C the δ-phase of NbN forms [6]. The δ-phase is highly interesting for superconducting accelerator technology applications.

In 2016 the furnace was further upgraded to allow nitrogen treatments of niobium samples and cavities at temperatures of up to 1800°C. The following sections introduce the furnace and the upgrades made.

UHV-FURNACE

The cross-section in Fig. 1 illustrates the main parts of the furnace [7]. The inner part of the uhv-furnace is a hot-pot made of niobium. The niobium samples 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.

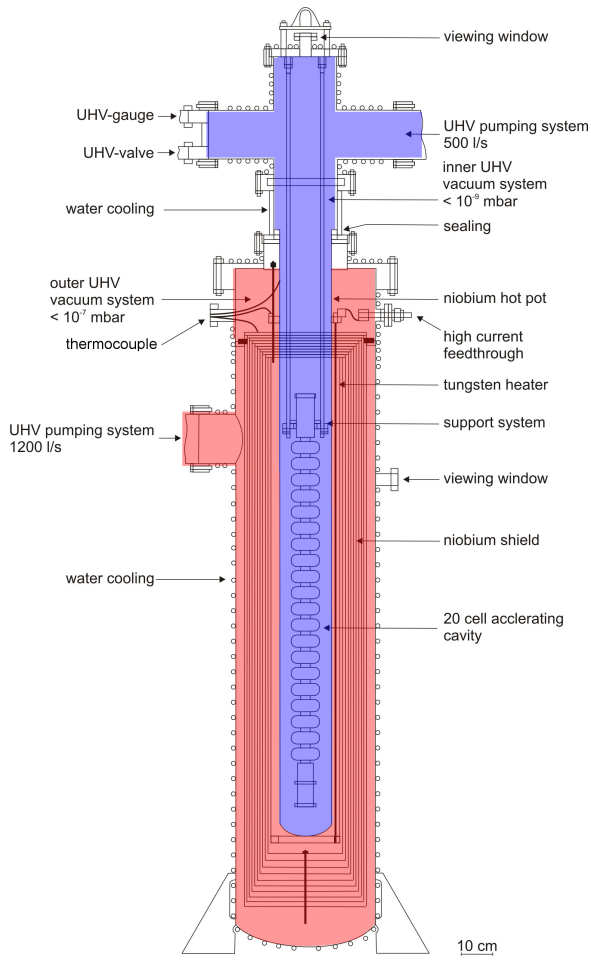

Figure 1: Schematic drawing of the UHV-Furnace. The inner hot-pot vacuum vessel is shown in blue, the insulating vacuum in red.

The furnace has two vacuum systems, an outer insulating vacuum and a separate hot-pot vacuum to reduce contamination of niobium samples or cavities, as indicated in Fig. 1 with pressures below 10^{-6} mbar. A turbomolecular pump

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and an ion-getter pump is 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 logged digitally. It is possible to attach a mass spectrometer to the hot-pot vacuum for residual gas analysis.

Furnace Operation

The temperature of the furnace is controlled indirectly by adjusting the electric current of the tungsten heaters. In Fig. 2 the current 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 progress of the vacuum pressure inside the hot-pot is displayed in Fig. 3. The higher vacuum pressure of 10^{-4} mbar compared to 10^{-6} mbar might be caused by a not fully regenerated ion-getter pump.

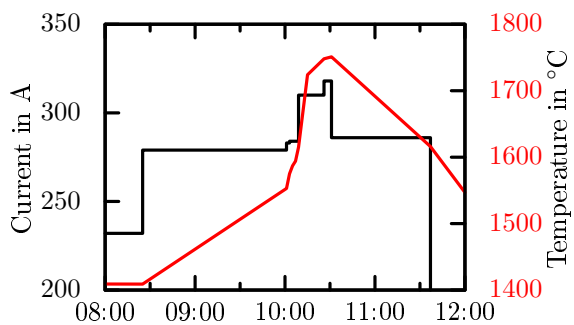

Figure 2: Relation between the heater current (black) and the temperature (red) near the hot-pot over time of day.

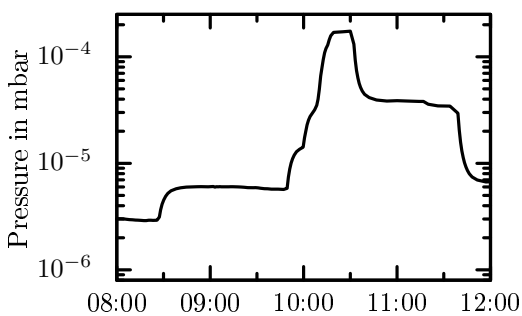

Figure 3: Hot-pot vacuum pressure in the same timeframe as in Fig. 2.

New Hot-Pot Design

To prevent contamination of the existing hot-pot with niobium-nitride, a new hot-pot design, based on the existing hot-pot, has been developed. In Fig. 4 the newly designed hot-pot is shown with the furnace head at the top and a sample holder inside. For treatments of niobium samples or single-cell cavities, the hot-pot can be shorter, the hot zone is approximately half as large as in the original design. Only the bottom 700 mm of the hot-pot are surrounded by the tungsten heaters. In this area the samples or cavities are located during treatments.

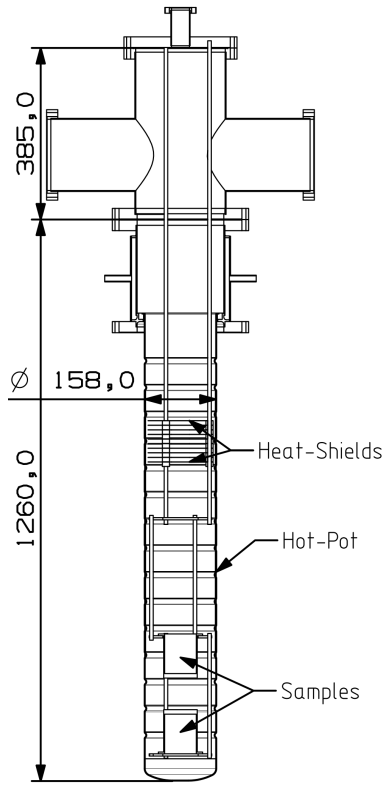

Figure 4: Cross-section of the new hot-pot design with a shorter overall length. The position of the samples is shown as well.

Vacuum Simulation

For better understanding of the vacuum inside the hot-pot including heat-shields and samples as seen in Fig. 4, the vacuum system has been simulated with Molflow+ [8]. The simulation parameters have been set to meet the experimental obtainted temperature and pressure values. As the real outgassing rates of the different materials are unkown only a qualitative conclusion will be given. The result of the simulation is shown in Fig. 5. The cross-section of the hot-pot is reduced by the heat-shield, the sample-holder and the samples resulting in a decreased pump-performance. The pressure rises by approximately two orders of magnitude between pumping port and sample surface. To reduce possible effects of different nitrogen pressures at the critical surface

of the samples, the sample surfaces point against each other.

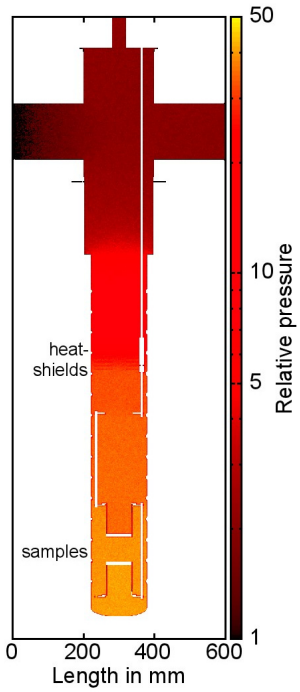

Figure 5: Result of the Molflow+ [8] simulation, showing the pressure at different positions relative to the pressure at the pumping port at the top left of the assembly.

Nitrogen Inlet

To investigate the effect of nitrogen doping and the growth of δ-phase NbN a nitrogen inlet has been designed. For simplicity, it has been designed 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. 6 the supply for nitrogen is shown.

To reach a pressure of 10^{-2} mbar inside the hot-pot with a volume of around 46.4 l, the pressure of the nitrogen inlet with a volume of 0.5 l has to be around 0.2 mbar. 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 will be tested at different temperatures and pressures. It is expected that at the beginning most of the nitrogen will be trapped in the niobium hot-pot until it is saturated. First samples for nitrogen treatment will be in the size of 1 cm, cut from niobium sheets. They will be investigated by Advanced Thin Film Technology group (ATFT), at the material sciences institute of Technische Universität Darmstadt to find a good process for later treatment of more ambitious samples [9].

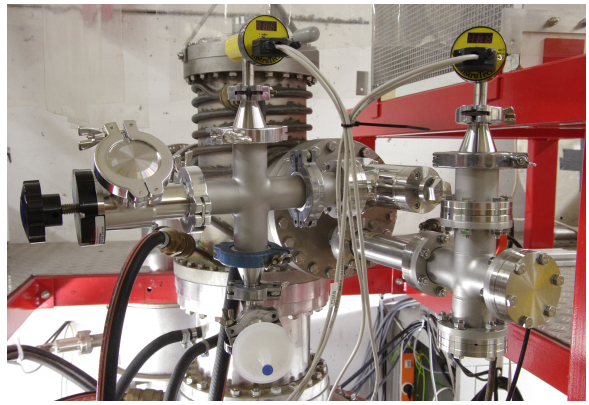

Figure 6: Nitrogen supply system at the top of the furnace. As only a small volume is required, it consists of a KF40 cross in the center with attached periphery: At the top a Pirani vacuum gauge, on the left a vacuum valve for a vacuum pump, at the bottom a nitrogen inlet with particle filter. On the top right is a additional Pirani vacuum gauge to monitor the pressure inside the hot-pot.

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