# UPGRADE OF A UHV FURNACE FOR 1700 C HEAT TREATMENT AND PROCESSING OF NIOBIUM SAMPLES *

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## *Abstract*

In 2005 a high temperature vacuum furnace was put into operation at the Institute for Nuclear Physics at the Technische Universität Darmstadt. It has been designed for firing pure Niobium at temperatures of up to 1870 °C. Until now several Nb cavities have been heat treated at 850 °C with a proven record of success [1]. The current focus of research in improving the superconductive characteristics of accelerator cavities is on new materials such as $Nb_3Sn$ or NbN or on the doping of Nb surfaces with nitrogen, so called $N_2$-Doping [2]. The surface preparations generally take place at temperatures of not more than 1000 °C. To study phenomena that occur at higher temperatures, like the formation of δ-phase NbN at 1300 to 1700 °C, we refurbished the UHV furnace and equipped it with state-of-the-art infrastructure. The vacuum system was updated as well as a new power interlock was applied due to a failure of the previous system. We designed a new annealing pot and planned its construction; the dimensioning of an appropriate sample holder is in progress.

## INTRODUCTION

The Darmstadt UHV furnace was originally built at the University of Wuppertal in 1983 [3] and relocated to Darmstadt in 2002. The device consists of four main assembly groups: the vacuum system, the heat shield, the heating system and the object mounting (see Fig. 1). Due to the design temperature of 1870 °C, only high temperature materials can be used in the hot zone of the oven. So the heating rods are made of tungsten with a melting point of 3422 °C, the insulating rings are made of alumina ($Al_2O_3$, melting point 2072 °C) with an application temperature of up to 1900 °C. Pure niobium (melting point 2477 °C) is used for the heat shield as well as for a special hot pot, which is applied to avoid contamination of the probe with unwanted metal oxides. The infrastructure of the furnace consists of a power supply for the heating rods, several vacuum pumps, a water cooling system, a safety interlock including a video surveillance and an extensive measuring system including a data acquisition. From the relocation in 2002 until now all of the equipment has been replaced and updated. More details will be reported in the next sections.

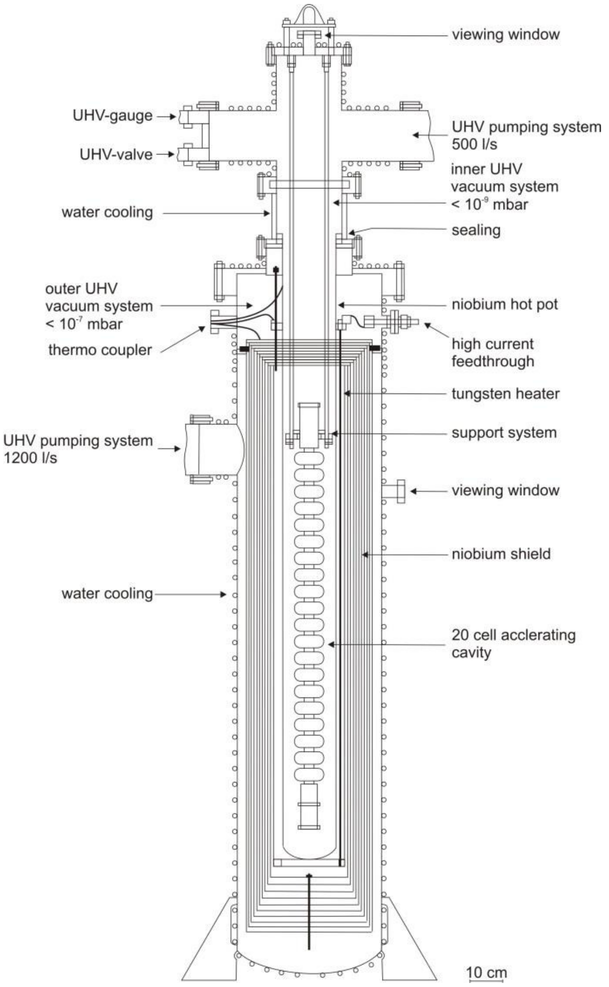

Figure 1: 2-D cross-section of the UHV furnace.

## VACUUM SYSTEM

There are two separate vacuum sections inside the furnace: the insulation vacuum and the hot pot vacuum. The insulation vacuum contains the hot pot, the heat shield, the heating rods, the insulating rings and all transitions to the outside like the current feedthroughs. It is pumped by a double stage oil sealed rotary vane backing pump and a water cooled turbo molecular pump down to $10^{-6}$ mbar. The hot pot vacuum section contains only the support system for cavities or samples, all parts in the hot zone of the pot are made of pure niobium. This vacuum system is built up of a mobile pumping unit (rotary vane backing pump, turbo molecular pump) as a booster pump stage

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and a titan sublimation pump connected in series to an ion getter pump for a high vacuum of up to $10^{-8}$ mbar (see Fig. 2). All assemblies within the hot pot vacuum area need to be carried out in clean room conditions in order not to contaminate any parts of the cavities or samples.

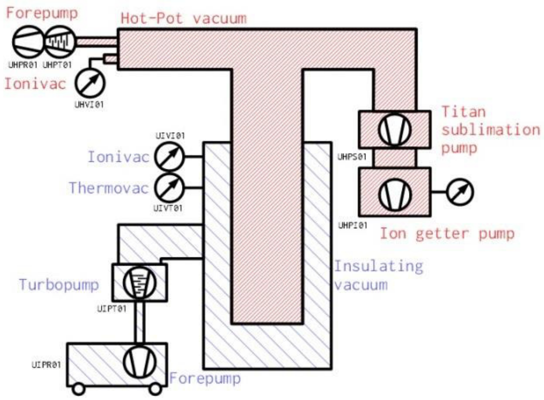

Figure 2: Schematic set up of the vacuum system.

## HEAT SHIELD

To reduce the power consumption and also to reduce the thermal radiation on the casing, it is essential to use an efficient heat shield. The furnace contains 10 concentric interlaced cylindrical niobium foils with a thickness of 0.2 mm. The cover plate foils at the bottom are spot-welded to the cylinders, at the top they are mounted to the shell cover flange. The flange as well as the top foils are equipped with a centered hole and a window on the outside to allow a line of sight into the hot pot. Alumina spacers with a height of 6.5 mm ensure the accurate distance between each separate foil (see Fig. 3).

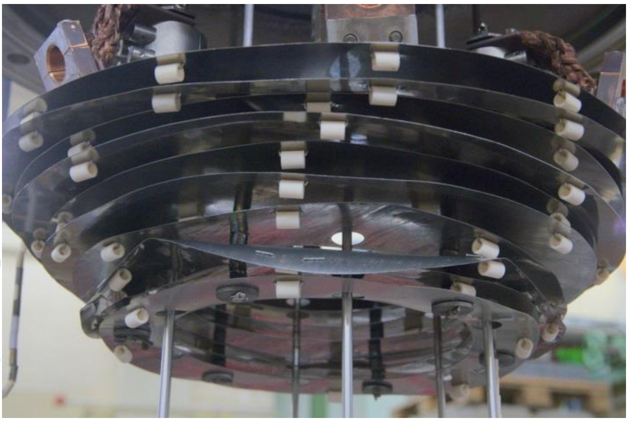

Figure 3: Top cover foil with $Al_2O_3$ spacers.

## HEATING SYSTEM

Three separate U-shaped heating rods were chosen as a resistance heating. They are made of tungsten with a diameter of 5 mm and a length of 3500 mm and placed between the heat shield and the hot pot. For supporting, stabilization and electrical insulation of the rods there are two alumina insulation rings mounted on top and bottom. The upper ring is outside the hot zone, the lower ring hangs at the end of the heating rods inside the hot zone. Additional niobium foils are attached between the ring and the hot pot to minimize the pollution of the hot zone with outgassed metal oxides.

### *Power Supply System*

As a power source a galvanically isolated 3-phase power transformer is plugged. The star-connected device provides the heating system with a current of up to 340 ampere each phase at a power of 40 kilowatts total. The original thyristor controller was replaced by a variable ratio transformer with a voltage range from 0 to 400 volts and a maximum current of 63 ampere.

### *Power Interlock*

The primarily used method for short circuit detection was an insulation resistance test. With higher temperatures the monitor showed a decreasing resistance between phase and electrical grounding. At a temperature of approximately 900 °C the value of resistance reached the threshold and the interlock interrupted the power supply. An inspection of the inside of the furnace showed no signs of a short circuit. Further investigations suggested that cooling water is the reason for decreasing insulation resistance. Due to the losses on the top cover caused by thermal radiation and heat conduction, the lead-in wires, which are made of copper, need to be water-cooled (see Fig. 4).

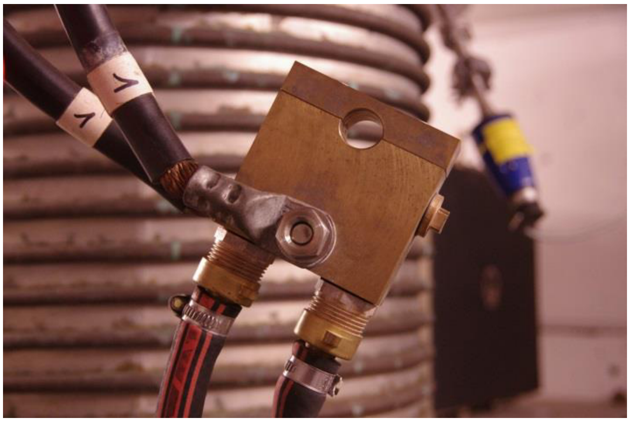

Figure 4: Current plug with connected cooling water hoses.

The electric conductivity of water rises with increasing temperature, in our case the cooling water temperature rises from 15 °C to 60 °C, as one can see in Fig. 5, measured at the current feedthroughs. This implies an eightfold value of the conductivity of the cooling water [4], what suggests this as the reason for the decreasing resistance between phase and ground. With this installation we were unable to reach furnace temperatures above 900 °C. However this variation of resistance means not a short circuit

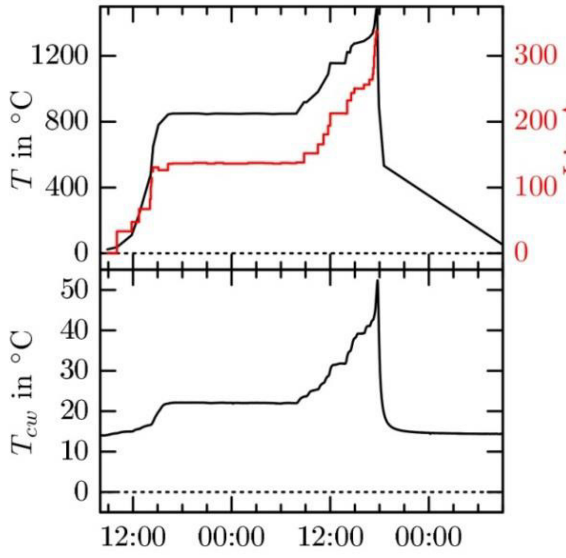

Figure 5: The upper chart shows the temperature inside the furnace and the current through the heating rods, the lower chart shows the temperature of the cooling water.

but a problem with the interlock. To solve this problem we chose another method of short circuit detection. Instead of the insulation resistance test we monitor the current of each phase in the current supply cable from the transformer to the feedthrough with three separate clamp-on ammeters (see Fig. 6). Every change in value which is not effected by the operator leads to a shutdown of the power transformer. Since then no power failure occurred even at the maximum power output of 340 ampere each phase.

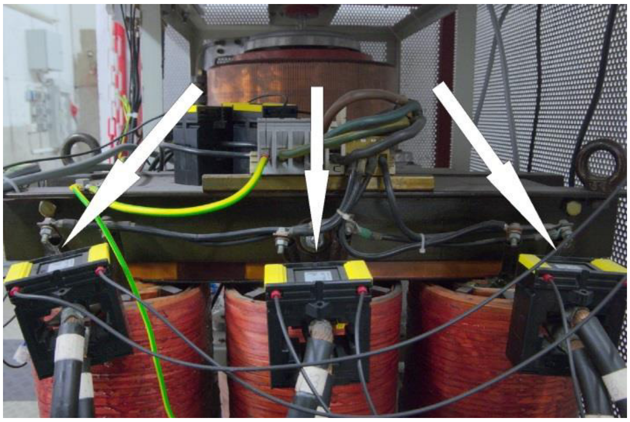

Figure 6: Power transformer with clamp-on ammeters.

## OBJECT MOUNTING

The object mounting is housed in the niobium hot pot which has an original length of 1875 mm and a diameter of 160 mm. It is designed for the heat treatment of 20 cell niobium accelerator cavities with a length of 1280 mm. Our focus however is on firing and preparation of small niobium samples, so a shorter sized annealing pot was designed (see Fig. 7), its construction is scheduled for the immediate future. The layout of the niobium sample holder for up to three samples is still in progress and will be finished within the next weeks.

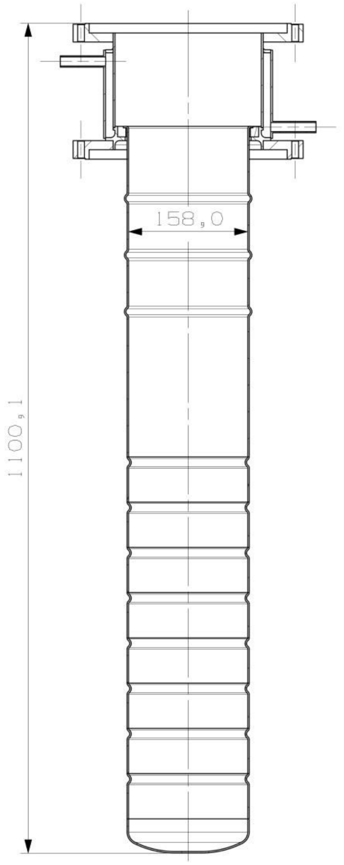

Figure 7: Newly designed, shortened hot pot.

## CONCLUSION

We equipped our UHV furnace with a state-of-the-art infrastructure, like a new plc-based interlock or an up-to-date vacuum system, we designed a new annealing pot and a sample holder for firing or preparation of niobium or niobium alloy samples. A nitrogen doping process is planned, so we will be able to study processes at a temperature range not investigated so far.

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