REFURBISHMENT AND REACTIVATION OF A NIOBIUM RETORT FURNACE AT DESY*

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Abstract

For research in the field of heat treatments of superconducting cavities, a niobium ultra-high vacuum furnace built in 1992 – originally used for the titanization of 1.3 GHz nine-cell cavities – and later shut down has recently been refurbished and reactivated. A significant upgrade is the ability to run the furnace in partial pressure mode with nitrogen. The furnace is connected directly to the ISO4 area of the clean room for cavity handling. At room temperature vacuum values of around 3×10^{-8} mbar are achieved. The revision included the replacement of the complete control system and a partial renewal of the pump technology. The internal mounting structures are optimized for single-cell operation including tandem operation (two single-cell cavities at once) and corresponding accessories such as witness-samples and caps for the cavities. The installation of additional thermocouples for a detailed monitoring of the temperature curves is also possible at the mounting structure. Due to the furnace design, its location and the strict routines in handling, very high purity levels are achieved in comparison to similar set-ups and hence provide a mighty tool for SRF cavity R&D at DESY.

THE FURNACE ARCHITECTURE

The furnace, designed for a vertical loading of 1.3 GHz nine-cell cavities, was originally built in 1992 for titanization purposes. After about 20 years of inactivity the furnace was reactivated in the context of research of new cavity temperature treatments.

The inner cylindrical recipient provides a diameter of 350 mm and is made entirely out of niobium. Also the removable furnace frame including the upper thermal shield consists completely of niobium. Loading and unloading takes place from above using a hand-operated crane. The recipient is surrounded by a second vessel which provides an additional vacuum for the heaters and the thermal shield. The structure is shown in Fig. 1.

Heating System

The heating capacity is dimensioned in such a way that temperatures of up to 1400 °C can be reached. The two tungsten heating coils, separated in upper and lower section, are located in the heater vacuum along with the thermal shielding.

Pump Infrastructure

The furnace has three different pump systems.

- Support vacuum
  Volume: 1000 l
  Target pressure: $p < 1\times10^{-6}$ mbar
  Tubo pump: Leybold Turbovac 1000C
  Scroll pump: Edwards nXDS10i
- Recipient vacuum
  Volume: 330 l
  Target pressure: depends on recipe
  Cryo pumps: 2x Leybold COOLVAC 2000
  Compressor: Leybold COOLPAK 6000H
  Tubo pump: Edwards NEXT85D
  Roots pump: Edwards EH250EU
  Scroll pump: Edwards XDS35i
- Intermediate suction (lid)
  Target pressure: $p < 2$ mbar
  Membrane pump: Welch Ilmvac MP 201 T

Cooling System

The cooling system consists mainly of two separate water-cooling circuits.

- Jacket cooling
  Net cooling capacity: 90 kW
  Work pressure: 4 – 6 bar
  Flow rate: 125 l/min
- Cryo chiller
  Net cooling capacity: 0.97 kW
  Work pressure: 1.5 – 2.5 bar
  Flow rate: 20-30 l/min

The outer surface of the furnace and components that are particularly exposed to thermal loads, such as the flanges and electrical connections of the heaters, are connected to a circumferential pipe system for active water cooling. The system consists of a closed circuit that transfers the thermal energy to the nearby cooling water circuit via a heat exchanger. The system can be filled via a bypass to the well water network and can be fed directly from the well network in case of emergency.

The compressor of the cryo pump system possesses a dedicated cooling circuit connected to the well water network. The chiller itself works with an air-cooled cooling medium. It is located in the neighbouring hall section. The output water temperature is set to 16 – 17 °C to avoid condensation.

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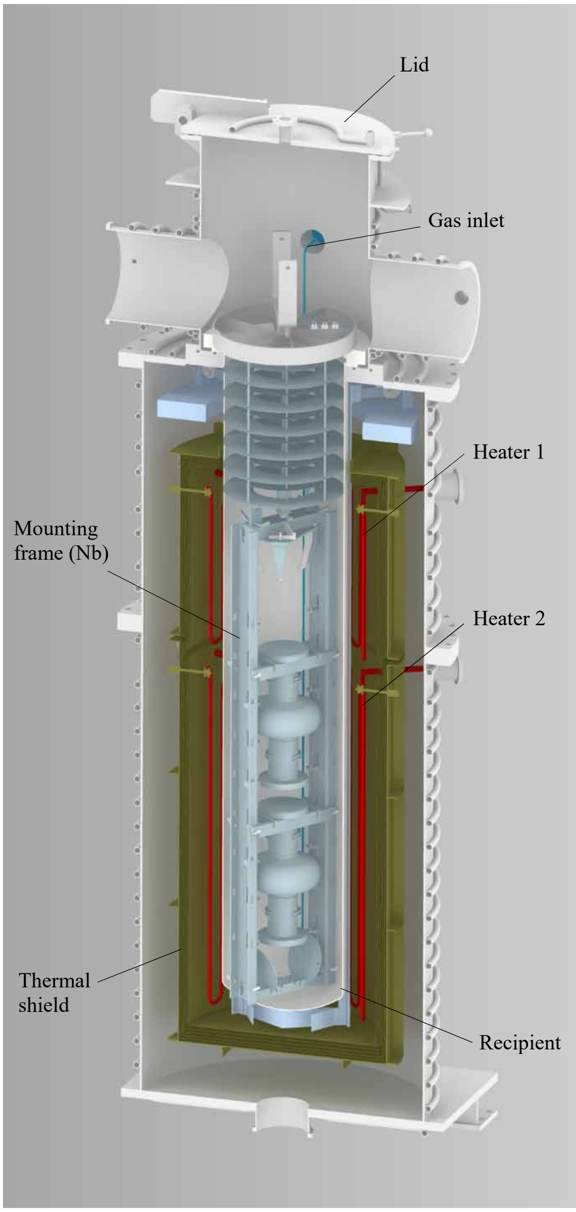

Figure 1: Sectional view of Nb-furnace (furnace frame and thermal shielding of the retort shown in light blue, heaters shown in red, thermal shielding inside the support vacuum shown in yellow and surrounding vessel with water-cooling shown in light grey).

CONTROLS & ELECTRONICS

As part of the revamp of the furnace, the entire control system, including the electrical wiring and control cabinets, was renewed. Only selected components, such as the control unit of the turbo pump for the support vacuum, were integrated into the new system.

The new system includes a new operating concept with the possibility of modular recipe creation and a redesigned user interface.

The measured values of all digital sensors are now recorded in the higher-level data server.

OPERATING CONDITIONS

The furnace-control works with different operating states of the furnace, each of which represents a building block which can be used to create individual recipes. The following list only considers the essential step chains for the recipient. These blocks are named as follows.

- Pre-vacuum
- High vacuum throttled
- High vacuum
- Flood
- Pre-vacuum partial pressure
- High vacuum throttled partial pressure

With the preselection of the operating mode, it is determined which pumps are actively connected to the recipient.

"Pre-vacuum" means the two Edwards backing pumps are active. The turbo pump is disabled and the cryo pumps are disconnected. At room temperature pressures down to 7×10^{-3} mbar are achieved in this mode.

"High vacuum throttled" in addition activates the turbomolecular pump. In this mode pressures of around 2×10^{-7} mbar can be reached at room temperature. It is useful for the initial pumping or the partial pressure operation at a pressure below 1×10^{-2} mbar.

"High vacuum" deactivates the turbo pump and switches on the two cryo pumps, thus opening the full potential of the system. Pressures now reaches down to 3×10^{-8} mbar at room temperature.

The supplement "partial pressure" refers to the flow controller of the system, which, depending on the setting, introduces a defined nitrogen volume flow into the chamber.

PARTIAL PRESSURE MODE

The possibility of introducing nitrogen directly into the recipient is a new feature. The furnace is then operated in partial pressure mode. For this purpose, a niobium tube was routed to the bottom of the recipient via a flange in the upper area just below the hatch. The volume flow is controlled by an mks mass flow controller, which is secured with an additional diaphragm valve. The setting range reaches from 1.69×10^{-6} mbar l/s – 824 mbar l/s. The nitrogen is extracted from an existing ring line that is fed by a liquid tank outside the building.

The backing pumps as well as the turbo pump is connected to the recipient via a butterfly valve, which is 100% open while running in ultra-high vacuum mode to prevent a virtual leak (turbo pump is disabled in this mode). Switching to partial pressure mode the butterfly valve adjusts the overall pressure inside the chamber by reducing or increasing the pump speed of the connected and activated pumps. The chamber pressure is therefore a function of the present nitrogen flow on the mass flow controller and the valve position of the butterfly valve, considering the pump capacity provided. This control loop is visualized in Fig. 2.

A mass flow of 3.38×10^{-6} mbar l/s is currently being used in "pre-vacuum partial pressure" mode. In this case, at a given target pressure of $3.3e\times10^{-2}$ mbar the butterfly valve is about 60% open and thus allows stable control. The "high vacuum throttled partial pressure" mode is currently not used, as it can only reliably maintain very small mass flows due to the low pumping capacity of the mounted turbo pump.

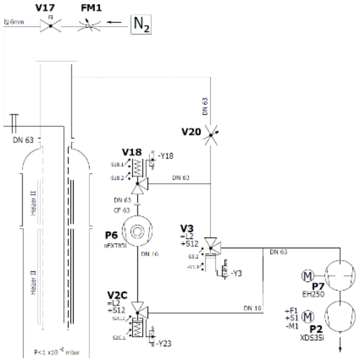

Figure 2 Flow chart detail (gas inlet).

Nitrogen Purity

The nitrogen is routed through a particle filter directly behind the extraction point from the supply line before it is routed to the flow controller. It has purity class 5.0 (99,999%) and is delivered by Linde. According to the data sheet [1], the mixture can contain the following impurities:

- H_2O ≤ 2 ppm
- O_2 ≤ 2 ppm

The batches are randomly checked by the DESY gas group with regard to their purity. In addition, the purity of the system is planned to be validated annually using the built-in mks Microvision 2 mass spectrometer. With this method a greater accuracy is achieved and also leaks in the inlet can be identified.

RECIPES

Mid-T recipes in the temperature range 250 – 300° and holding times from 3 – 20 h were tested (recipe no. 1 – 4 in Table 1). In addition to the soft reset recipe for R&D (no. 5) one infusion recipe (no. 6) was tested as well. In contrast to all other recipes listed, which work with the best possible vacuum (usually better than 5×10^{-8} mbar), the system is adjusted to 3.3×10^{-2} mbar in partial pressure mode during the 120 °C step of the infusion recipe. The last recipe on the list is a bake out recipe.

Details on the measurement results of the subsequent tests can be found in the associated paper [2].

Table 1: Tested Recipes

No.	Recipe	Runs
1	20h@250 °C	2
2	20h@300 °C	1
3	3h@250 °C	2
4	3h@300 °C	4
5	3h@800 °C	2
6	3h@800 °C + 48h@120 °C	3
7	5h@1150 °C	4

Two exemplary temperature and pressure curves can be seen in Fig. 3 and Fig. 4.

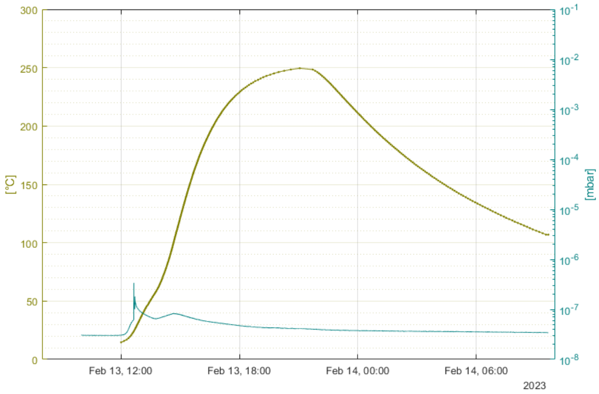

Figure 3: Temperature and pressure curve recipe 3h@250 °C (cavity 1DE12, 16.02.23).

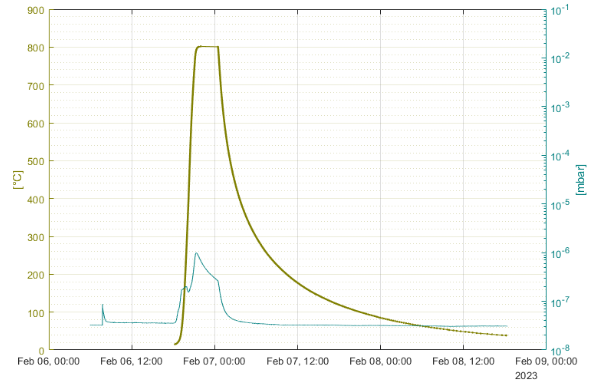

Figure 4: Temperature and pressure curve recipe 3h@800 °C (cavity 1AC02, 08.02.23).

OPERATING ROUTINE

Each cavity and associated samples that will be treated in the niobium furnace undergo an advanced procedure described in Table 2.

Table 2: Cavity Pre- and Post-Treatment in the Clean Room

No.	Work Step	ISO
1	1x Rinsing with ultrapure water (min. 16 MΩ) + high pressure rinsing	6
2	Drying for 48 hours (vertical cavity axis, rotated 180° after 24 hours to avoid liquid pockets)	4
3	Furnace run	4
4	1x Rinsing with ultrapure water (min. 16 MΩ) + high pressure rinsing	6
5	Mounting of flanges	4
6	6x High pressure rinsing	6
7	Mounting of antenna (single-cell) / mounting of pump flange (nine-cell)	4

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Touched supporting elements of the frame as well as temporary built-in small parts such as ceramic screws for the caps are blown off with the ionizing gun before installation. The registered particles are noted with the help of the particle counter. After closing the lid, the furnace is pumped via the corresponding step chain. If the pressure is lower than 1×10^{-5} mbar bar, the valve to the mass spectrometer is opened and the device is started. During the test runs, a seven-day cycle for the furnace operation was established. To ensure appropriate pre- and post-treatment of the cavities in the clean room, the cavity is usually installed or removed on Thursdays. Pumping takes three days and is typically performed over the weekend. The run starts on Monday morning after a check with the mass spectrum. After the run is finished an additional output spectrum is written. Figure 5 shows a comparison of in- and output spectrum as an example.

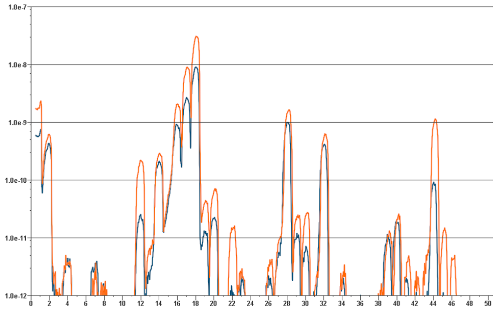

Figure 5: Comparison orange input (orange curve) & output mass spectrum (blue curve), recipe 3h@250 °C (cavity 1DE12, 16.02.23).

Afterwards the mass spectrometer is deactivated, the corresponding valve is closed and below 50 °C cavity-temperature the recipient can be flooded with nitrogen. This step is activated manually shortly before the planned (weekly) installation/ removal of the cavity. Whenever possible, the retort is kept under vacuum for reasons of cleanliness.

MOUNTING STRUCTURE

The frame is based on the nine-cell cavity design and has been optimized for single-cell use. The entire structure, with the exception of the sensors and the upper support ring, is made of niobium to avoid contamination during treatment. Due to the ambient conditions, every mechanical connection is designed to be form-fitting. The frame was completely freed from the titanium sheets (originally needed for titanization) and then BCP'ed. A continuous recess was added to the suspension and shielding to create space for the optional nitrogen inlet.

Tandem Loading

For single-cell cavity runs, the loading capacity has been doubled by installing an additional slot. This setup can be seen in Fig. 6. The cavities are held by corresponding niobium profiles on the upper flange. These have recesses to accommodate additional samples. Mounting is performed manually from top to bottom to keep the particle contamination low.

Temperature Monitoring

Three type S thermocouples are used to monitor the temperature inside the recipient. These sensors are attached to the frame. The measuring tips are each inserted in niobium blocks. The idea is to map the temperature profile inside the cavity material. Apart from that handling of the thermocouples showed that they quickly become brittle and fail. This is due to the temperature changes in connection with bending changes due to manual handling. For this reason, a temporary connection to the cavity was avoided. Instead, the sensors are fixed to the frame permanently. The connectors are firmly connected to the upper support ring with the help of a holding device. All of these measures significantly increased the service life of these sensors and the operational safety of the furnace. The sensor holders are arranged in corresponding recesses so that they can be adjusted over the length of the frame

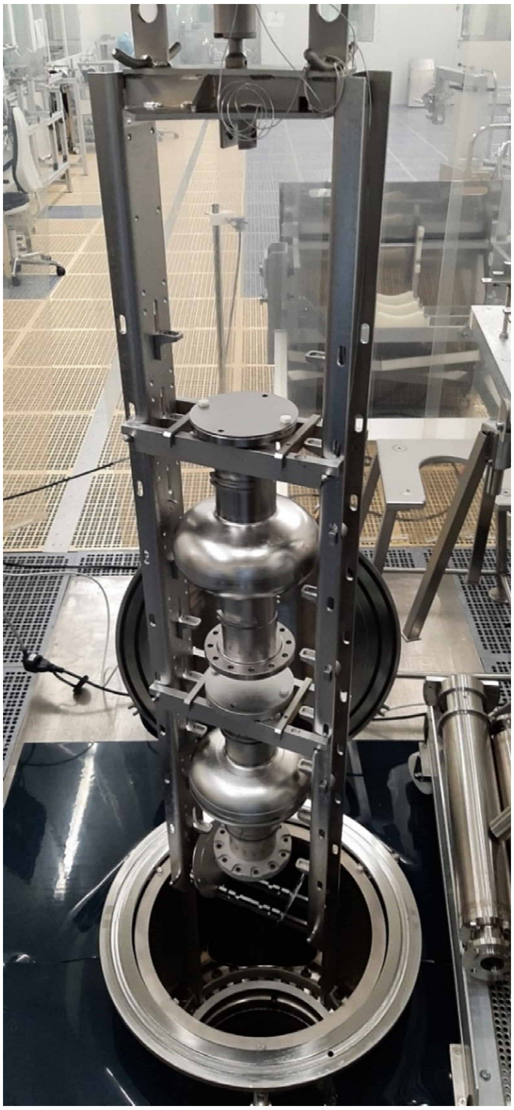

Figure 6: Furnace frame equipped with two since-cell cavities.

The temperature values are recorded, but they are not part of the temperature control loop of the heaters. The heaters are controlled by two additional temperature sensors within the support vacuum. The attempt to control the temperature with the sensors inside the retort did not result in stable control due to the thermal inertia of the system. The sensors inside the retort are distributed over the length of the frame in such a way that one sensor is approximately at the height of the equators (distance approx. 50 mm between sensor and equator of the single-cell cavity) and the third sensor is mounted in the middle between the two loading slots.

During the test runs it was shown that there is a temperature gradient between top and bottom sensor (distance of 450 mm) of approx. 10 K (applies to the target temperature of 800 °C). This fact is particularly relevant when a tandem or nine-cell treatment is planned. In that case the cavity cells would undergo different temperature treatments depending on their position in the furnace. This circumstance is essentially due to the construction of the furnace. The mechanical connection of the frame to the structure of the furnace on top causes an outflow of thermal energy via conduction. The compensation of these differences required an adjustment of the controller and the program. Depending on the target temperature and the load, the upper heating circuit now has a significantly higher target value. For 800 °C operation, the difference can be reduced to approx. 1 K. The additional parameters were added to the program when creating the recipes for this type of runs.

Furthermore, with this furnace it should be noted that the offset between the heating circuit in the support vacuum and the actual temperature at the cavity depends on the target temperature. This connection can be seen in Fig. 7.

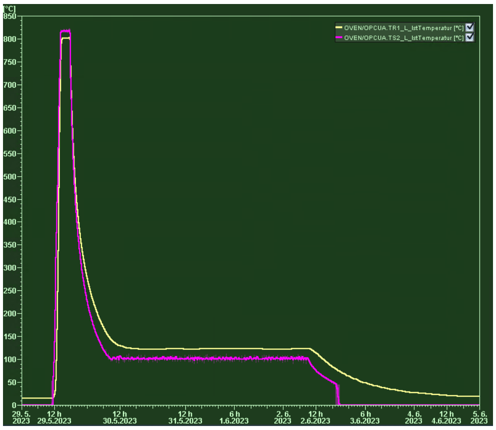

Figure 7: Plot of infusion recipe with the regulated temperature of the heaters (pink curve) in the supporting vacuum and the resulting temperature (yellow curve) on the furnace frame (cavity 1DE1, 06.04.23).

Caps

In order to avoid potential particle contamination of the interior of the cavity during installation in the furnace frame and during craning of the entire frame, the cavity is fitted with niobium caps. These are 2.8 mm thick niobium plates, which have the contour and hole pattern of the cavity flanges. The caps are laid on the top of the cavity flange before each run. Optionally, caps with a concentric hole can also be mounted on the lower flange of the cavity. The caps are BCP'ed at regular intervals and remain in a holder in the lower area of the furnace frame even when not in use.

SUMMARY

The niobium furnace was refurbished both mechanically and electrically to create a solid basis for testing new temperature treatments for single- and nine-cell cavities. In addition to the conventional temperature treatments up to 1200 °C at pressures of up to 3×10^{-8} mbar, the revised infrastructure now also allows infusion recipes to be run with a high level of purity.

CONCLUSION & OUTLOOK

The refurbishment was carried out successfully and thus equips the DESY SRF team with a flexible and high-purity furnace as a solid basis for research in the field of heat treatments of cavities. Due to the sluggish temperature control behavior, especially at low temperatures, there is still potential for improving the curve shape and accuracy of the temperature curves. Furthermore, preparations for the nine-cell cavity treatment are currently taking place. In order to increase the flexibility in the infusion treatments, a gas bypass is to be installed in order to be able to connect conventional gas bottles. In addition, new sample holders are being planned and in order to establish an alternative temperature measurement a pyrometer will be included.

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