

Nb₃Sn VAPOR DIFFUSION COATING SYSTEM AT SARI: DESIGN, CONSTRUCTION, AND COMMISSIONING

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Abstract

This paper describes the design of a coating system for the preparation of a superconducting radio-frequency cavity with Nb₃Sn thin films. The device consists of a coating chamber made of pure niobium, a vacuum furnace for heating the coating chamber, a superconducting cavity bracket and two crucible heaters. The chamber is vacuum isolated from the furnace body to protect the superconducting cavity from contamination during the coating process. The device has been built and commissioned, which could be used for Nb₃Sn coating of a 1.3 GHz single-cell superconducting cavity in future.

INTRODUCTION

The Nb₃Sn coating superconducting cavity offers significant efficiency advantages compared to a standard niobium cavity. Its high operating temperature of 4.2 K simplifies the cooling requirements, resulting in reduced cooling costs [1, 2]. These advantages make the Nb₃Sn cavity a hot of research field. Several preparation methods are being investigated, and the vapor physical deposition method has shown the most promising results. Several laboratories have successfully coated high-quality Nb₃Sn films on niobium base cavities using this technique. Remarkably, the 1.3 GHz single cell Nb₃Sn cavity coated by Fermilab shows excellent performance in vertical test at 4.2 K, the Q-value reaches 3×10^{10} at low accelerating field and the maximum accelerating field reaches 22.5 MV/m [3].

In order to develop Nb₃Sn superconducting cavity, we have designed and built a new coating system. The main body of the coating system is a horizontal vacuum heat treatment furnace for vapor phase physical deposition. In addition, the furnace body is equipped with vacuum module, electric control module and water-cooling module. The coating system will be used for the studies of coating 1.3GHz single-cell cavities.

REQUIREMENT FOR COATING SYSTEM

The design of this coating system is based on Fermilab's 9-cell coating system [1]. Despite the object being treated is a single-cell cavity with shorter length, the dual-tin source design is chosen for its advantage in achieving coating uniformity compared with a single tin source [4]. Accordingly, a horizontal furnace with two evaporation sources was selected. Figure 1 depicts the overall design of

the furnace, including the furnace body, the Nb coating chamber, and the bracket and crucible heater.

In physical vapor deposition processes, maintaining cleanliness is extremely important. Environmental impurities, including those originating from the furnace body, may adhere to the cavity during the coating process or react to form undesirable products [5]. In order to reduce the contamination, a dedicated coating chamber has been constructed using pure Nb, effectively isolating the furnace and minimizing the possibility of pollution.

The reaction between niobium and Sn can result in the formation of multiple compounds, which is dependent on the reaction temperature and the ratio of niobium to tin content in the system [6]. Among the various potential products, Nb₃Sn exhibits a high transition temperature, aligning with our expectations. Specifically, in the niobium-tin system above 930 °C, only Nb₃Sn will form when the Sn component content ranges between 17% and 25%. Any excess tin or niobium will exist in the formation of simple substance within the system [7].

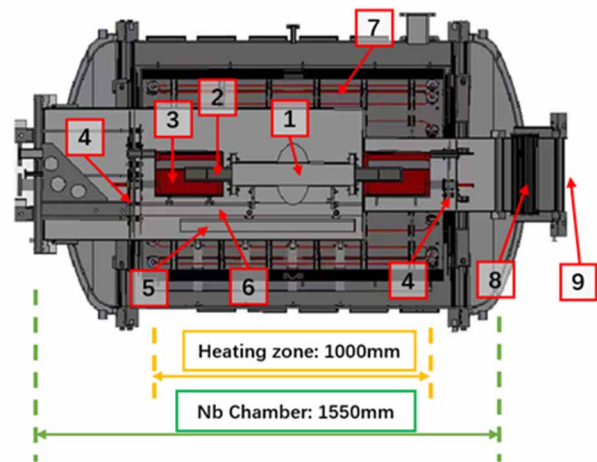


Figure 1: Overall design of the coating system at SARI: 1 Nb cavity, 2 Sn crucible, 3 Crucible heater, 4 Nb reflectors inside Nb chamber, 5 Bracket rails, 6 Cavity bracket, 7 Mo heater of furnace, 8 bellows, 9 Pumping port for Nb chamber.

DESIGN AND CONSTRUCTION

A photograph of the coating system's construction is depicted in Fig. 2. The photo provides a clear overview of the system's components, which have been built according to the description provided in the preceding section. The design parameters of the furnace are listed in Table 1.

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Table 1: Design Parameters of the Furnace for Coating System

| Parameters | Design values |
|------------------------|--|
| Maximum temperature | $\geq 1300\text{ }^{\circ}\text{C}$ |
| Effective heating zone | $\phi 600\text{ mm} \times 1000\text{ mm}$ |
| Temperature uniformity | $< \pm 4\text{ }^{\circ}\text{C}$ |
| Heating Rate | $\geq 720\text{ }^{\circ}\text{C/h}$ |
| Heater | Mo Heater |
| Target vacuum pressure | $< 8 \times 10^{-5}\text{ Pa}$ (25 $^{\circ}\text{C}$) $< 2 \times 10^{-3}\text{ Pa}$ (1100 $^{\circ}\text{C}$) |
| Molecular pump | 2500L/s (N ₂) |

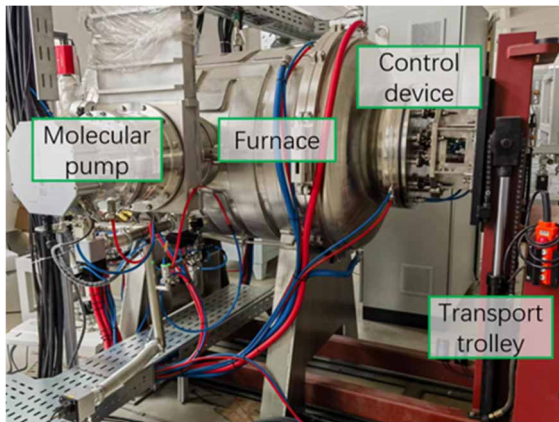


Figure 2: Nb₃Sn coating system at SARI.

The furnace's maximum operating temperature is 1300 $^{\circ}\text{C}$, despite the commonly used coating temperature of 1100 $^{\circ}\text{C}$. This higher temperature allows for potential temperature adjustments in future process improvements. Heating is achieved through evenly arranged Mo heaters on the inner wall of the furnace shell. Additionally, to rapidly increase the temperature from the nucleation stage at 500 $^{\circ}\text{C}$ to the desired coating temperature, the furnace body ramp-up rate is set at over 720 $^{\circ}\text{C/h}$. This high ramp-up rate helps minimize the formation of other phases at low temperatures. The furnace shell is constructed using stainless steel 304 L, and cooling water troughs are uniformly welded on its outer surface. During operation, the circulating water within these troughs ensures that the temperature difference between the outer furnace wall and the ambient room temperature remains within 20 $^{\circ}\text{C}$, even when the furnace reaches high temperatures.

To ensure the isolation of potential contamination from the furnace body, a dedicated coating chamber has been constructed. The parameters of this chamber are listed in Table 2. The chamber is composed of two pure niobium cylinders, each with a thickness of 4 mm, which have been welded together. The dimensions of these cylinders are $\phi 450\text{ mm} \times 1100\text{ mm}$ and $\phi 250\text{ mm} \times 450\text{ mm}$, respectively. The rear end of the niobium cylinder is connected to the molecular pump through a bellows and transition tube,

which are sealed by metal gaskets. The bellows serves to compensate for axial expansion, ensuring that the coating chamber is not subject to compression and deformation caused by thermal expansion during heating. The vacuum within the coating chamber is maintained by a molecular pump and a dry pump. The molecular pump operates continuously throughout the coating process to uphold the vacuum within the chamber and extract excess Sn vapor, thus reducing Sn residue. Additionally, the exterior of the coating chamber is also evacuated by another molecular pump to prevent collapse due to excessive differential pressure.

Table 2: Design Parameters of the Coating Chamber

| Parameters | Design values |
|------------------------|--|
| Material of chamber | Nb (RRR40) |
| Effective heating zone | $\phi 300\text{ mm} \times 600\text{ mm}$ |
| Inner size of chamber | $\phi 450\text{ mm} \times 1100\text{ mm}$ $+ \phi 250\text{ mm} \times 450\text{ mm}$ |
| Target vacuum pressure | $< 2 \times 10^{-5}\text{ Pa}$ (25 $^{\circ}\text{C}$) $< 3 \times 10^{-4}\text{ Pa}$ (1200 $^{\circ}\text{C}$) |
| Molecular pump | 2100 L/s (N ₂) |

The coating chamber is equipped with heat reflectors and crucible heaters on both the front and rear sides. The heat reflector, composed of multiple stacked niobium discs, serves to minimize heat dissipation and maintain temperature uniformity within the effective temperature zone. The size of the effective zone in the coating chamber is $\phi 300\text{ mm} \times 600\text{ mm}$, providing ample space for a 1.3 GHz single-cell cavity. To ensure optimal performance, the effective temperature zone requires an internal temperature difference of less than $\pm 4^{\circ}\text{C}$. Within the coating chamber, a niobium bracket supports the superconducting cavity. The bracket is positioned on rails that are welded on both sides of chamber. During the coating process, the crucible is suspended on both sides of the superconducting chamber and heated by crucible heaters. The specific parameters of the crucible heating are listed in Table 3.

Table 3: Design Parameters of the Sn Heater

| Parameters | Design values |
|---------------------|-------------------------|
| Heater | Wu & Mo heater |
| Outside material | Ceramic |
| Maximum temperature | 1400 $^{\circ}\text{C}$ |

The crucible heater in the coating system is designed with independent temperature control-mode, allowing for precise temperature adjustment up to 1400 $^{\circ}\text{C}$. The heater is housed within a ceramic shell and contains Wu heaters. Figure 3 shows the crucible and crucible heater. However, due to the brittle nature of Wu, the heating element has been prone to damage during installation. As a result, it was replaced with Mo, which is a harder metal. Both Wu and Mo have high melting points (3422 $^{\circ}\text{C}$ for Wu and 2602 $^{\circ}\text{C}$

for Mo) and exhibit low vapor pressure at coating temperatures, making them suitable materials to use within the coating system.

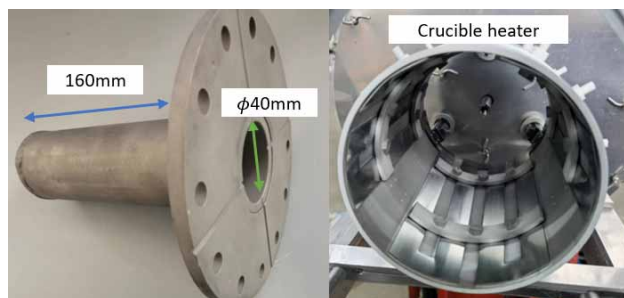


Figure 3: Sn crucible and crucible heater.

COMMISSIONING OF COATING SYSTEM

After completing the processing and assembly of all components, the coating system underwent a commissioning phase. During this phase, the system underwent a total of four heating tests. In the first two tests, the system was gradually heated up to 1200 °C at a rate of 600 °C/h and held at that temperature for 3 hours. These initial heating tests aimed to improve the ultimate vacuum level by allowing the furnace components to undergo a baking process for effectively removing impurities. Following these two heating tests, the vacuum level of both parts of the furnace reached the designated specification. The temperature and vacuum level during the 1200 °C baking process were monitored and recorded, as shown in Fig. 4.

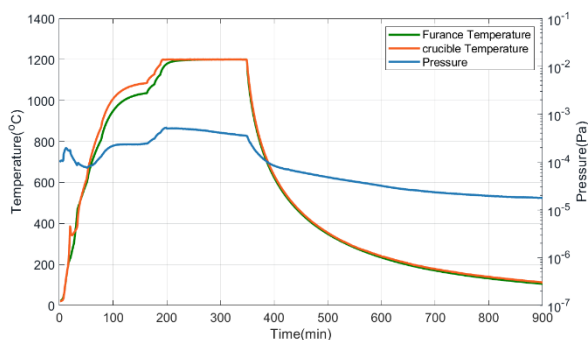


Figure 4: Temperature and pressure profile of 1200 °C baking test.

After the baking process, the experimental conditions were tested as well, and the temperature profile was set based on the classical coating profile [1]. The nucleation process lasted for 4 hours, during which the temperature of both the coating chamber and the crucible heater was maintained at 500 °C. Following the nucleation process, the coating chamber was heated to 1100 °C and held at that temperature for 3 hours. Simultaneously, the crucible heater was heated to 1300 °C for 1.5 hours and then cooled down to match the temperature of the coating chamber. The temperature and vacuum level during baking process were also recorded and shown in Fig. 5. At the end of the holding period, all heat sources were turned off, and the system naturally cooled down to room temperature. Throughout the

entire test, the system operated normally, and the vacuum level in the coating chamber remained below 1×10^{-3} Pa, indicating that the system passed the commissioning phase and was qualified for use in coating experiments.

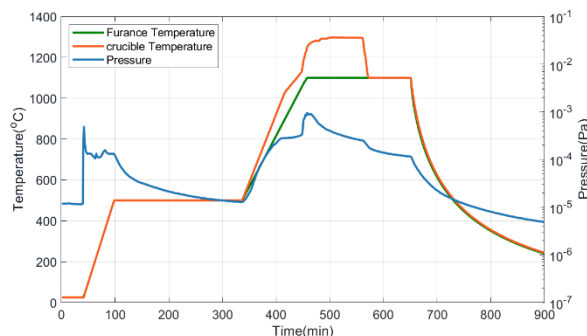


Figure 5: Temperature and pressure profile in the commissioning test.

SUMMARY

The Nb₃Sn coating system comprises a vacuum furnace along with corresponding vacuum, electric control, and water-cooling modules. After processing and assembly of these modules, the coating system has been commissioned and meets the design specifications. In the near future, the coating system will be put into operation to coat single-cell cavities.

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