

DESIGN AND CONSTRUCTION OF Nb₃Sn VAPOR DIFFUSION COATING SYSTEM AT KEK

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Abstract

The vapor diffusion Nb₃Sn coating system was developed at KEK. At most, it can be used to coat a 1.3 GHz 3-cell cavity. The coating system comprises a coating chamber made of Nb, vacuum furnace for heating the Nb chamber, and Sn heating device in the crucible. The Nb chamber vacuum and furnace vacuum are isolated to prevent contamination from the furnace. Heating device for increasing the Sn vapor pressure was installed for the coating system. This paper reports the details of the Nb₃Sn coating system at KEK.

INTRODUCTION

Nb₃Sn cavities have a smaller heat load at approximately 4.2 K, and they have a higher efficiency than Nb cavities [1]. Therefore, Nb₃Sn cavities are anticipated to be operated at 4.2 K by using a small refrigerator with a cryocooler. Studies have carried out cooling tests and cavity performance tests by a small refrigerator [2–4].

Several methods have been investigated for Nb₃Sn coating. Among them, coating via the vapor diffusion method has exhibited the best cavity performance [5]. The performance of Nb₃Sn coating cavities by vapor diffusion was evaluated in many laboratories. In FNAL, a cavity performance test of a 1.3 GHz single-cell cavity was carried out at 4.2 K. The results were as follows: the Q-value in the low accelerating field was 3×10^{10} and the maximum accelerating field reached 22.5 MV/m. [6].

KEK has undertaken Nb₃Sn cavity R&D, and its final goal is to realize Nb₃Sn cryomodule, which is cooled by small refrigerators.

A Nb₃Sn film coating system using the vapor diffusion method was constructed for developing Nb₃Sn coating cavities. The coating system comprises a heating vacuum furnace, coating chamber, Sn crucible, and heater for Sn crucible. This system is designed for Nb₃Sn coating on up to a 1.3 GHz 3-cell cavity.

REQUIREMENT FOR Nb₃Sn COATING SYSTEM

The Nb₃Sn vapor diffusion coating system at KEK was constructed based on the system at Cornell University [7]. Figure 1 shows a schematic of the Nb₃Sn coating system at KEK. The coating system comprises a furnace for heating, coating chamber made of Nb, Sn and SnCl₂ crucibles, and Sn crucible heater.

The conditions of the coating temperature and coating time for Nb₃Sn coating are described below. Nb₃Sn is

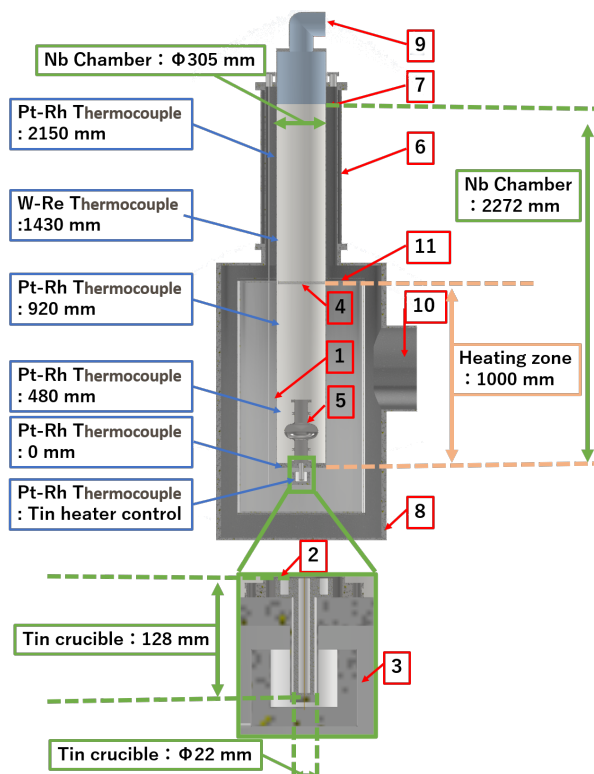


Figure 1: Design of the coating system at KEK: 1 Nb chamber, 2 Sn crucible, 3 Heater for Sn, 4 Mo reflectors inside Nb chamber, 5 Nb cavity, 6 SUS extension tube, 7 Nb-SUS conversion flange, 8 Vacuum furnace, 9 Pumping port for Nb chamber, 10 Pumping port for vacuum furnace, 11 Mo reflectors in vacuum furnace.

coated at approximately 1100°C to avoid the growth of Nb-Sn compounds, other than Nb₃Sn, which have a lower superconducting transition temperature. Nb₃Sn is formed when the Sn composition is between 17 and 25 at%, and the temperature is above 930 °C in the Nb-Sn reaction. Nb-Sn compounds with a low superconducting transition temperature are formed depending on the Sn composition of the reaction below 930 °C [8]. In general, Nb₃Sn is coated at 1100 °C for approximately 3 h to maintain sufficient thickness [1].

Next, the clean requirements are described below. Impurities that contaminate the Nb₃Sn film during the coating process can degrade the cavity performance. Therefore, impurities need to be prevented from contaminating the coating chamber during Nb₃Sn coating. The coating chamber and furnace are possible sources of impurities. The material of the chamber should be Nb to avoid any impurities from the coating chamber. A double vacuum structure with an

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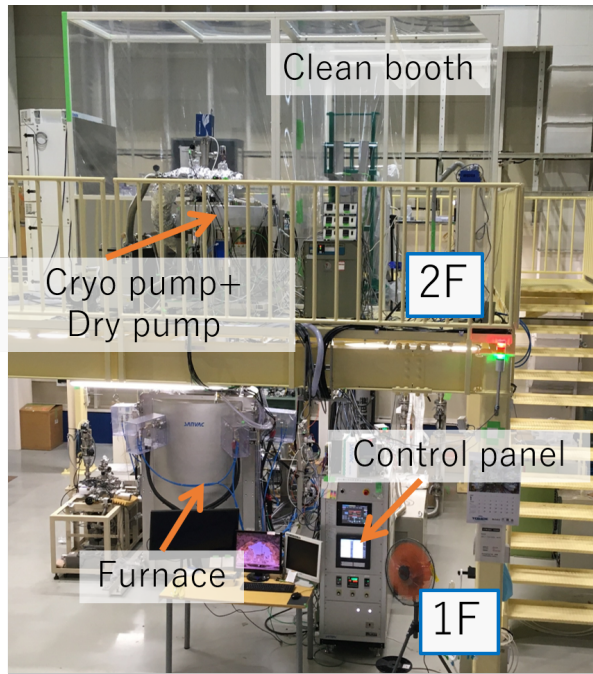


Figure 2: KEK coating system.

Nb coating chamber is required to avoid impurities from the furnace during Nb_3Sn coating. In addition, impurities from outside the coating system need to be prevented when a cavity is inserted into the coating system.

Finally, the conditions for uniform coating are described. The mean free path of the Sn vapor needs to be shorter than the cavity iris diameter for a uniform Nb_3Sn coating [1]. In addition, nucleation by evaporation of $SnCl_2$ is necessary to achieve a uniform Nb_3Sn coating. The Sn crucible and $SnCl_2$ crucible were used for Nb_3Sn coating. The Sn crucible heater evaporates Sn. The heater's maximum temperature should be more than $1500^\circ C$ to realize a shorter Sn mean free path than the cavity iris diameter.

DESIGN OF Nb_3Sn COATING SYSTEM

The furnace for heating, coating chamber, Sn crucible, and Sn crucible heater for the Nb_3Sn coating were designed and constructed to realize the requirement described in the previous section. Figure 2 shows a photograph of the entire coating system at KEK.

Table 1 lists the design parameters of the furnace.

Nb-Sn compounds with a low superconducting transition temperature are formed below $930^\circ C$, as described in the previous section. Therefore, the furnace temperature need to increase from $500^\circ C$, which is the nucleation process temperature, to $1100^\circ C$, which is the coating process temperature, as fast as possible. Therefore, the heating rate is chosen to be $600^\circ C/h$. In addition, the maximum operating temperature of the furnace was chosen to be $1200^\circ C$ to enable the change in the coating temperature conditions. The effective heating zone was determined to be 1 m, which sufficient for coating a 1.3 GHz 3-cell cavity.

Table 1: Design Parameters of the Furnace for Coating System

Parameters	Design values
Effective heating zone	$\phi 400 \text{ mm} \times 1000 \text{ mm}$
Heating Rate	$600^\circ C/h$
Operation temperature	$100^\circ C \sim 1200^\circ C (\pm 10^\circ C)$
Maximum temperature	$1400^\circ C$
Heater	Mo heater $\times 3$ lines
Cryopump (CRYO-U20H)	$170 \text{ L/min (N}_2)$
Drypump (CR 300B)	5000 L/min
	RT: $< 1 \times 10^{-4} \text{ Pa}$
Target vacuum pressure	$600^\circ C: < 1 \times 10^{-2} \text{ Pa}$
	$1200^\circ C: < 1 \times 10^{-1} \text{ Pa}$

Table 2 lists the design parameters of the coating chamber and the pumping system for the coating chamber.

Table 2: Design Parameters of Coating Chamber

Parameters	Design values
Material of chamber	Nb (ASTM commercial grade)
Flange material and size	TF480, ICF356
	$\phi 305 \text{ mm} \times 2272 \text{ mm}$
Inner size of chamber	$+ \phi 30 \text{ mm} \times 80 \text{ mm}$
	(for Sn crucible)
Size of coating zone	$\phi 305 \text{ mm} \times 1000 \text{ mm}$
Cryopump(CRYO-U20H)	$170 \text{ L/min (N}_2)$
Drypump(CR 300B)	5000 L/min
Target vacuum pressure	RT(after baking): $< 1 \times 10^{-5} \text{ Pa}$
	Coating Process: $< 1 \times 10^{-4} \text{ Pa}$

The coating chamber is made of Nb to prevent impurities from the chamber during coating. For vacuum sealing, titanium (TF480) flange is used. The Nb coating chamber and titanium flange were welded together by TIG welding. When the vacuum pressure is high during Nb_3Sn coating, the impurities from the coating chamber might contaminate the Nb_3Sn film. Therefore, a cryopump is used to evacuate the chamber to achieve low vacuum pressure in the coating chamber. The cryopump was selected to maintain the vacuum pressure under $1 \times 10^{-4} \text{ Pa}$ during coating. In the coating system at KEK, the Sn crucible is heated by the Sn heater which is separate from the furnace. Therefore, the coating chamber has a Sn crucible installation point on the bottom. The inner diameter of the coating chamber is 305 mm. It is sufficiently large to install the 1.3 GHz cavity, which diameter is approximately 210 mm. The coating chamber and evacuation port are vacuum-sealed with a copper gasket. The operating temperature of the copper gasket is under $300^\circ C$. Therefore, the coating chamber has a cooling section for cooling the titanium flange. The total length of the chamber is 2272 mm to keep the temperature of the flange below $300^\circ C$. Table 3 lists the design parameters of the Sn heater for the Sn crucible.

Table 3: Design Parameters of the Sn Heater

Parameters	Design values
Heater	Mo heater
Outside material	Ceramic, Ta
Maximum temperature	1500 °C

The heating part of the Sn heater is made of Mo. The heater's maximum temperature was chosen to be 1500 °C. Under this condition, the Sn crucible was heated to approximately 1400 °C during Nb₃Sn coating. The outside of the heater was covered with ceramic.

The top of Fig. 3 shows the Sn heater installed in the coating system, and the bottom of Fig.3 shows the Sn crucible and SnCl₂ crucible used for coating. Both the crucibles are made of W.



Figure 3: Top: Sn heater. Bottom: Sn crucible and SnCl₂ crucible. Both crucible are made of W.

Figure 4 shows the Sn crucible combined with the sample chamber. A disk-shaped jig made of Mo is used to combine the Sn crucible and the sample chamber. When a cavity is coated, the same jig is used to combine the Sn crucible and the cavity. For reference, the melting points of W and Mo are 3422 °C and 2602 °C. The vapor pressure of W is approximately 1.2×10^{-3} Pa at 2602 °C, and the vapor pressure of Mo is approximately 4.4×10^{-3} Pa at 1868 °C [9, 10]. Therefore, W and Mo can be used in the coating system. Figure 5 shows a photograph of the inside of the furnace. The Sn heater shown in Fig. 3 is installed in the lower part of the Nb coating chamber, as shown in Fig. 5. The Mo

heaters in the furnace are divided into three parts. Two of the heaters are shown in Fig. 5.



Figure 4: Sample chamber with Sn crucible.

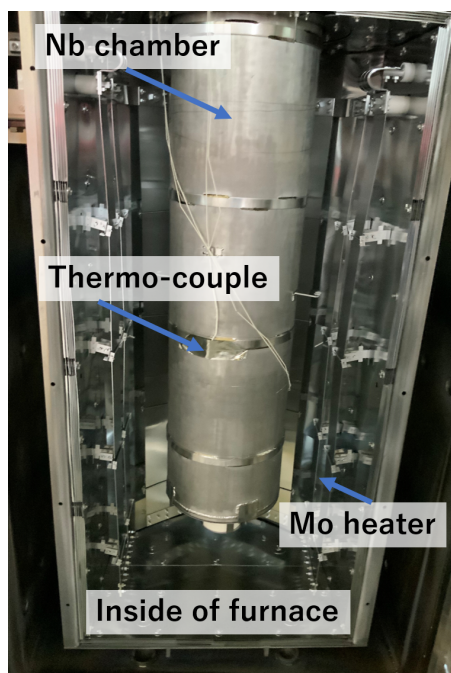


Figure 5: Inside of the furnace

A clean booth was built in the cavity insertion area to prevent any impurities from entering the chamber. The clean booth can realize the cleanliness of ISO-class 3. The size

of the clean booth was approximately 2 m in height, 1 m in width, and 4 m in length.

COMMISSIONING OF COATING SYSTEM

After constructing the coating system, the stand-alone test was performed. In the test, the temperature profile of the furnace was set as follows: 500 °C for 4.5 h in the nucleation process, and 1100 °C for 3.0 h in the coating process. The temperature profile of the Sn heater was 1300 °C for 1.5 h in the coating process. The heating rate from 500 °C to 1100 °C was 600 °C/h. The temperature profile and partial pressure of each mass in the stand-alone test are shown in Fig. 6. As shown in Fig. 6, the Nb₃Sn coating pattern was realized in the constructed coating system. The vacuum pressure in the coating chamber was less than the design value of 1×10^{-4} Pa. Thus, it was confirmed that the constructed coating system can coat Nb₃Sn films.

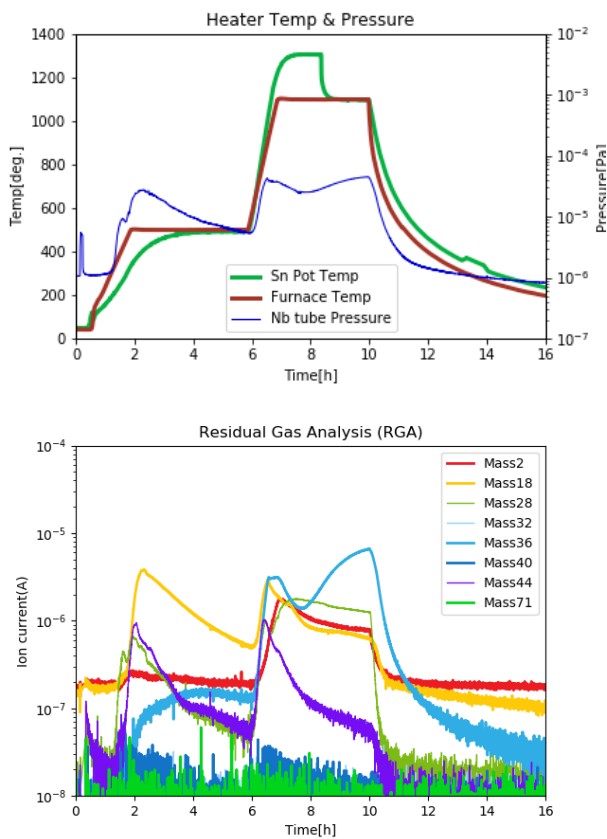


Figure 6: Top: Temperature and pressure profile in the commissioning test. Bottom: RGA profile in commissioning test.

SUMMARY

The Nb₃Sn coating system was constructed at KEK. The coating system comprises the furnace, Nb coating chamber, and the Sn crucible heater. After constructing the coating system, the stand-alone test was performed. The test showed that the Nb₃Sn coating system can coat Nb₃Sn films. In the future, we will study Nb₃Sn coating for a high-efficiency Nb₃Sn cavity using the coating system.

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