ISBN: 978-3-95450-211-0 doi:10.18429/JACoW-SRF2019-TUP072

THE DEVELOPMENT OF NIOBIUM SUPTTERING ON COPPER CAVITIES AT IHEP*

J. Dai†, P. He, Z.Q. Li, Y.S. Ma, F.Y. Yang, P. Zhang, Institue of High Energy Physics, Chinese Academy of Science, Beijing 100049, China

Abstract

A R&D program focusing on niobium sputtering on copper cavities started at IHEP in 2017. Single-cell 1.3 GHz elliptical cavity shape has been initially chosen as sputtering substrate. A magnetron sputtering system have been developed in 2018. In addition, a surface treatment facility to polish the copper substrate before sputtering has been developed and commissioned. This paper will present the Nb/Cu coating activities at IHEP.

INTRODUCTION

In recent years, radio frequency superconducting technology has developed rapidly in China. IHEP-related projects include Accelerator Driven Sub-critical System (ADS) project [1], Platform of Advanced Photon Source technology (PAPS) project, and High Energy Photon Source (HEPS) project [2] and so on. At present, the mainstream technology is pure niobium superconducting cavity, but the superconducting performance of niobium cavity has almost approached the superconducting limit of niobium in recent years [3], so the niobium sputtering copper cavity was officially launched at IHEP in 2017 as an alternative technical reserve.

Niobium sputtering copper cavity is a technology has been developed for many years, first proposed by CERN in 1980s [4]. It has been successfully applied to LEP2 [5] and LHC [6] accelerators of CERN and ALPI heavy ion accelerators [7] of INFN. It has been running steadily for many years. The niobium-sputtered layer has not been found to fall off and the performance of superconducting cavity has not been reduced due to the change of niobium-sputtered layer [8].

IHEP adopts SUBU agent of CERN as the basic formula of copper cavity chemical polishing, and already built a chemical polishing facility to prepare smooth and clean substrate for niobium sputtering. A magnetron sputtering system was also built in Mid-2018. At the end of 2018, four copper cavities, two stainless steel sample holder cavities and niobium cathodes were completed. Glow discharge debugging was realized in April 2019, and the first experimental coating was completed in May 2019.

SURFACE TREATMENT

After the half-cell of copper cavity is finished, 600 mesh sandpaper is used for mechanical polishing, then dilute sulfuric acid is used to clean the cavity equator area for the next step electron beam welding. After welding, the SUBU formula [9] of CERN is used for chemical etching. A chemical polishing system for copper cavity was built, as shown in Fig. 1.

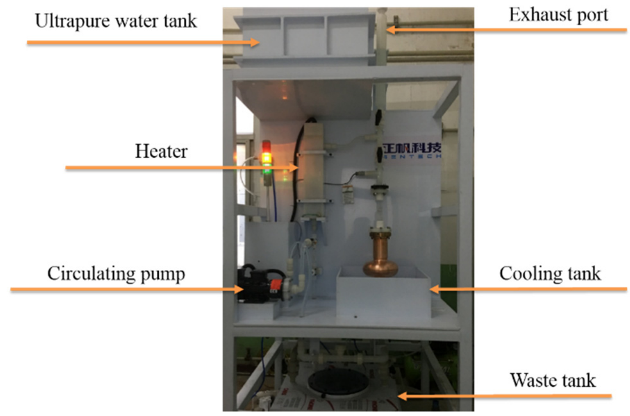

Figure 1: Chemical polishing system for copper cavity.

As long as the copper cavity is mounted on the system, SUBU acid is pumped into the circulating pipeline through a pneumatic pump from the acid container, and the heater and circulating pump open simultaneously. The acid is continuously heated to 72 degrees Celsius during circulation in the polypropylene pipeline. An exhaust port is reserved at the top of the circulating pipeline. The acid flow will guide the corroded gas and related bubbles generated on the cavity surface to the exhaust port and then enter an exhaust gas treatment system. This bubble removal would help obtaining a smoother cavity surface. After chemical etching, cold water is used to spray the outer surface of the copper cavity for cooling, as shown the cooling tank in Fig. 1. It usually takes about 15 minutes for the temperature of copper cavity to decrease from 72 degrees Celsius to room temperature.

The cooled acid is then discharged into the waste acid tank under the circulation system. Then the passivation solution is pumped into the circulating system by a pneumatic pump. The inner surface of the copper cavity is passivated for 1-2 minutes. Then the passivation solution is discharged into the waste acid tank. Then open the pure water valve above and pump the pure water into the circulating system to clean the acid remaining on the surface of the copper cavity. After about four cycles of cleaning, the acid-laden wastewater is discharged into the waste acid tank.

After the whole chemical treatment finish, the copper chamber is dismantled. Then, the inner surface of the copper cavity is purged with high purity nitrogen, so as to the residual pure water on the surface is washed out to prevent oxidation of the cavity surface. Then, the copper cavity is vacuum-preserved. Figure 2 shows the cavity inner surface after chemical treatment.

*Work supported by Platform of Advanced Photon Source (PAPS) project
†daijin@ihep.ac.cn

Content from this work may be used under the terms of the CC BY 3.0 licence (© 2019). Any distribution of this work must maintain attribution to the author(s), title of the work, publisher, and DOI.

ISBN: 978-3-95450-211-0 doi:10.18429/JACoW-SRF2019-TUP072

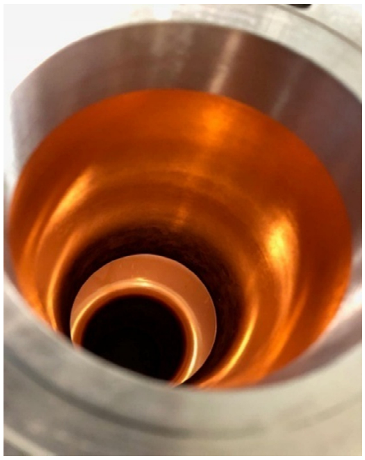

Figure 2: Copper cavity surface after chemical treatment.

This polishing system was put into use in early 2019. So far, a copper cavity has been treated once, and the surface roughness after treatment reaches 0.2 µm. At present, the system is limited by the capacity of acid storage vessel (6L), so the surface treatment of copper cavity can only reach 6 µm. New larger capacity (60L) acid storage vessels and larger power heaters are being machining. The expanded system has a design value of heating 60L acid solution to 72 degrees Celsius in 10 minutes, which can etch the surface of copper cavity about 50 µm.

NIOBIUM SPUTTERING

Magnetron sputtering device was completed in 2018. It consists of two parts, one is the niobium-sputtering chamber and the other is the vacuum system, as shown in Fig. 3.

Figure 3: Magnetron sputtering device.

The film sputtering vacuum chamber is placed in a class 100 clean room. The installation and disassembly of niobium cathode and copper cavity is inside the clean room to ensure the cleanliness of the sputtering system. The diameter of the sputtering chamber is 400 mm, and the copper cavity is placed on the base of the vacuum chamber. The base is sealed with the vacuum chamber through a copper O-ring. The vacuum system is placed outside the clean room. It consists of a primary pump, two molecular pumps and an ion pump. After about 72 hours of baking in May, the vacuum of sputtering chamber reaches 3×10^{-9} mbar.

The niobium cathode inside the chamber contains a permanent magnet with a diameter of 32 mm, a width of 20 mm, and a magnetic field strength of 5000 Gauss. The copper cavity is divided into 5 evaporation areas, and the evaporation time of each area is determined by the relative distance between the magnet and the evaporation position. In the process of coating, the magnet is moved from bottom to top, and different sputtering time is set at different positions of the cavity to achieve more uniform coating at the copper cavity surface. Since the whole system was integrated and debugged in April 2019, there is no time for sputtering on the real copper cavity, but a niobium sputtering on a sample cavity has been completed.

The sample cavity is made of stainless steel, and the inner wall morphology is exactly the same as that of the copper cavity used in the project, as shown in Fig. 4. Lot of circular holes were drilled in the stainless steel sample cavity for vacuum pumping. Some of the holes are screwed with oxygen-free copper samples, which is placed in different positions of the cavity tube, iris area and equator area to ensure that the evaporation quality of each position on the cavity can be known after one sputtering.

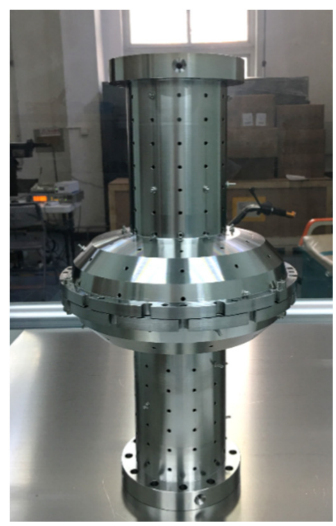

Figure 4: Stainless steel sample cavity.

In May 2019, the 1st sample cavity sputtering was completed. After the vacuum reaches 3×10^{-9} mbar, the ion pump is closed and krypton gas is filled into the vacuum chamber. The gas pressure is 5×10^{-2} mbar, the cathode current is set as 1 A. The sputtering time of iris and cavity tube is 2 hours. The position of equator area is about twice as far as that of cavity tube, so set the sputtering time as 4 hours. After the experiment, the sample cavity is removed from the sputtering chamber, and a layer of niobium film was deposited on the sample cavity surface and all copper sample sheets, as shown in Fig. 5. The copper sheets is then disassembled from cavity and analysed by SEM. It is found

Content from this work may be used under the terms of the CC BY 3.0 licence (© 2019). Any distribution of this work must maintain attribution to the author(s), title of the work, publisher, and DOI.

ISBN: 978-3-95450-211-0 doi:10.18429/JACoW-SRF2019-TUP072

that the thickness of niobium film at the cavity tube position was about 6 μm and the equatorial position was about 1 μm.

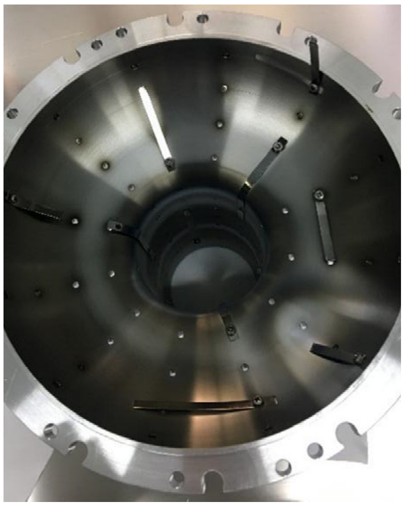

Figure 5: The first sputtering result.

CONCLUSION

By mid-2019, IHEP has completed chemical polishing equipment for niobium sputtering copper cavity and a set of magnetron sputtering equipment. The chemical polishing of a copper cavity was completed in January 2019, and the preliminary commissioning of the magnetron sputtering equipment was completed in April 2019. The first film preparation on the sample cavity was realized. The key parameters of evaporation, including discharge current, magnetic field arrangement and vacuum, still need to be optimized to achieve more uniform and stable niobium film in the future.

ACKNOWLEDGEMENT

This work has been supported in part by Platform of Advanced Photon Source technology (PAPS) project. The author also would like to thank all the IHEP colleagues who participate in this program and thank the colleagues in Ningxia Orient Superconducting Technology Co., Ltd (OSTEC) for providing us post-processing support.

REFERENCES

[1] Z. Li, *et al*, "Physics Design of an Accelerator for an Accelerator-driven Subcritical System", *Phys. Rev. Spec. Top. Accel. Beams* 16, 080101 (2013).

[2] Y. Jiao *et al.*, "Progress of the Lattice Design and Physics Studies on the High Energy Photon Source", in *Proc. IPAC'17*, Copenhagen, Denmark, May 2017, pp. 2697-2699. doi:10.18429/JACoW-IPAC2017-WEPAB052.

[3] AM. Valentefeliciano, "Superconductivity RF Materials Other than Bulk Niobium: a Review." *Superconduct. Sci. Tech.* 29.11(2016).

[4] C. Benvenuti, N. Circelli, M. Hauer, "Niobium Films for Superconducting Accelerating Cavities", *Appl. Phys. Lett*. 1984, 45(5): 583

[5] W. Weingarten and E. Chiaveri, "Industrial Production of Superconducting Niobium Sputter Coated Copper Cavities for LEP", in *Proc. SRF'93*, Newport News, VA, USA, Oct. 1993, paper SRF93I22

[6] H. Vogel *et al.*, "Production of Nb/Cu Sputtered Superconducting Cavities for LHC", in *Proc. SRF'99*, Santa Fe, NM, USA, Nov. 1999, paper WEP016, pp. 437-439.

[7] A. M. Porcellato *et al.*, "Operation Experience with ALPI Resonators", in *Proc. SRF'01*, Tsukuba, Japan, Sep. 2001, paper SA006

[8] V. Arbet-Engels *et al.*, "Superconducting Niobium Cavities, a Case for the Film Technology", *Nuc. Instr. Meth. Phys. Res. Sect. A: Accel., Spectrom., Detect. Assoc. Equip*. 2001, 463(1-2): pp. 1-8

[9] G. Lanza *et al.*, "The HIE-ISOLDE Superconducting Cavities: Surface Treatment and Niobium Thin Film Coating", in *Proc. 14th Int. Conf. RF Superconductivity (SRF'09)*, Berlin, Germany, Sep. 2009, paper THPPO075, pp. 801-805.

Content from this work may be used under the terms of the CC BY 3.0 licence (© 2019). Any distribution of this work must maintain attribution to the author(s), title of the work, publisher, and DOI.