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PREPARATION AND CHARACTERIZATION OF Nb FILMS DEPOSITED IN SRF CAVITY VIA HiPIMS*

H. C. Duan[1], Y. C. Yang, P. Zhang[1], P. He[1†], J. Dai, T. M. Xin[1], J. W. Kan[1], Y. S. Ma
Institute of High Energy Physics, Chinese Academy of Sciences, Beijing, China
[1]also at University of Chinese Academy of Sciences, Beijing, China

Abstract

The RF performance of the niobium superconducting cavity has been continuously improved in recent 50 years. Since the maximum acceleration field (E_{acc}) has approached its theoretical limit, developing a more efficient and low-cost SRF cavity is one of the key challenges of the next generation particle accelerators. Niobium coated copper cavities are promising solutions because the SRF phenomenon occur within several hundred nanometers under the cavity surface. In literatures, the Nb coated Cu cavity prepared by direct current magnetron sputtering has serious Q-slope problem, which may be related to the low energy deposition. High power impulse magnetron sputtering (HiPIMS) can produce a high peak power and high ionization rate which may improve the thin film quality. Therefore, we prepared Nb coated Cu samples via HiPIMS on the 1.3 GHz dummy cavity at IHEP.

INTRODUCTION

RF superconducting accelerating cavity has been widely applied in various particle accelerator in the world due to its advantages of low surface loss, high gradient (E_{acc}), high quality factor (Q), and continuous wave mode adaptability. As a key component of accelerators, improving the performance of RF superconducting cavities has been widely studied by scholars. The performance of conventional bulk niobium cavities has approached their theoretical limit over decades of development, and there is little room for further improvement. Developing a more efficient and cost reduction SRF cavity is a key challenge of the next generation particle accelerator. Since the penetration depth of the RF magnetic field on the surface of the cavity is less than 1 μm, one layer of superconducting material coated on cavity of materials with good thermal conductivity is a solution for further improving the cavity performance. The superconducting thin film cavity was first proposed by Benvenuti[1] at CERN in the 1980s, which was several microns of niobium deposited on copper cavity via magnetron sputtering. In recent years Nb/Cu thin film cavity has been successfully applied in the Large Electron Positron Collider (LEP) and Large Hadron Collider (LHC) at CERN, and the Superconducting Linear Accelerator (ALPI) at Italian National Institute of Nuclear Physics (INFN) [2-4].

The Nb/Cu thin film cavity deposited by direct current magnetron sputtering (DCMS) has a good low field quality factor Q, but the Q value decreases exponentially with RF injection power, which is called "Q-slope". This may due to the low energy deposition. HiPIMS is a high energy deposition technology, which has been applied in academia and industry in recent years. Because in HiPIMS the particles sputtered are ions, they have much higher deposition energy and can be further accelerated by substrate bias voltage. Compared to DCMS, HiPIMS has been proven to deposit Nb films with better microstructure, denser and better superconducting RF performance [5-8].

We have designed and constructed a deposition system at IHEP to conduct a series of research on preparing and characterizing 1.3 GHz Nb/Cu thin film cavity. Recently, this system has been used to deposit Nb films inside the 1.3 GHz Cu cavity via HiPIMS.

EXPERIMENTAL

Schematic diagram of the deposition system is shown in Figure 1. A 1.3 GHz dummy cavity made of stainless steel was mounted inside the cylindrical vacuum chamber. Copper plate samples treated by SUBU5 process[9] was fixed on the inner side of the dummy cavity at different positions. A cylindrical magnetron with a movable magnetic ring was mounted coaxial to the dummy cavity. The cylindrical Nb target was fixed outside the magnetron. The discharge was powered by a high pulse power supply (TruPlasma Highpulse 4002 G2). The dummy cavity was baked at 400 °C for 30 h by tantalum heater to obtain a base pressure of 8×10^{-7} Pa before the deposition. Krypton flow was set through a mass flow controller to obtain a working pressure of 0.6 Pa. The discharge was operated with a peak current of 110 A and a pulse voltage of 490 V. The pulse width was 30 μs with frequency of 800 Hz. The temperature of the samples was kept at 200 °C during deposition. The deposition duration was 6 h. The cathode target was a hollow cylindrical high-purity niobium target (RRR~300). The magnetic ring scans back and forth at a speed of 1 mm/s in the cavity.

SEM (HATACHI su8020) was used to investigate the surface morphology and cross-section. FIB was used to investigate the cross-section. The crystal structure and the size of crystallites are investigated by XRD (Bruker D8 Advance).

RESULTS AND DISCUSSIONS

The influence of the cavity shape on the morphology of Nb thin films was tested. SEM micrographs of the Nb thin films on copper plates at different positions of the dummy

* Work supported by High Energy Photon Source (HEPS).
† Email: heping@ihep.ac.cn

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cavity are shown in Fig. 2. It can be seen that the Nb films are smooth but ridged. This may due to the morphology and orientation of the Cu substrate [5].

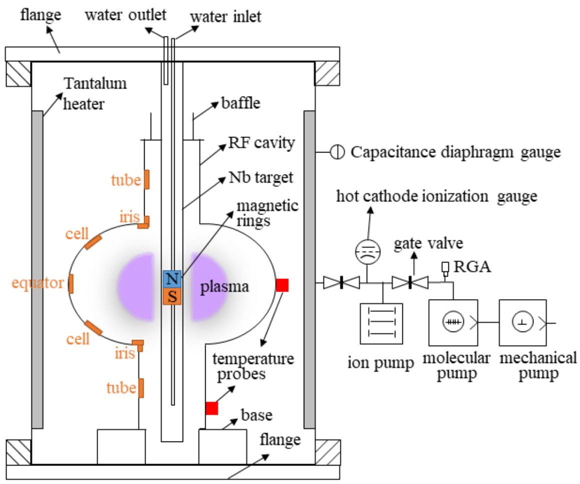

Figure 1: Schematic diagram of the deposition system.

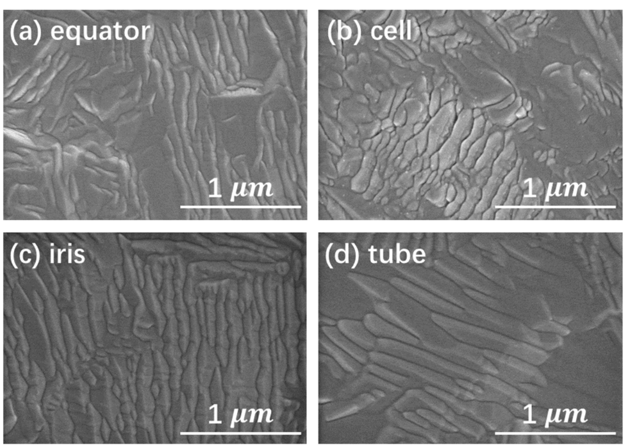

Figure 2: SEM micrographs of Nb thin films sputtered on copper plates at different positions of the dummy cavity.

Nb films doposited on Si wafer were used for observation of SEM cross sections and thickness, as shown in Fig. 3. It can be observed that the Nb films at equator are dense and closely attached to the Si wafer, but the Nb films at iris have nanoscale pores and are not fully attached to the Si wafer.

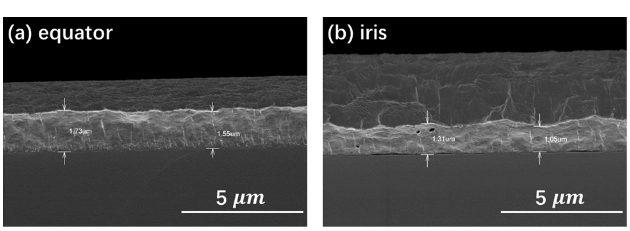

Figure 3: SEM cross sections of Nb thin films sputtered on silicon wafer at different positions of the dummy cavity.

FIB-SEM image of Nb films at cell is shown in Fig. 4. It shows that the Nb films are dense with no voids and obvious columnar growth.

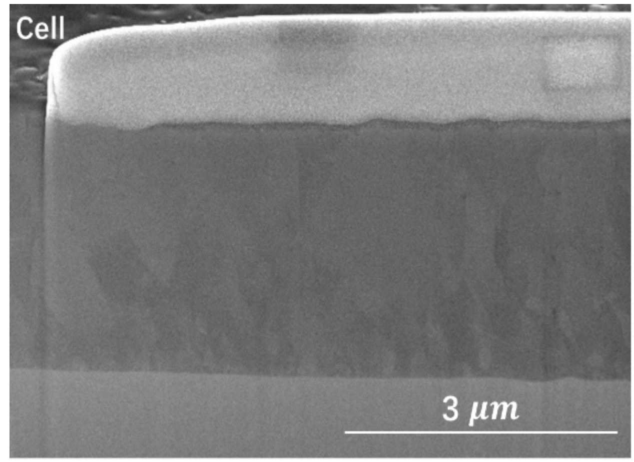

Figure 4: FIB cross section of Nb thin films sputtered on copper plates at the cell of the dummy cavity.

The chemical properties of the Nb films has been analyzed by grazing incidence angle XRD. The XRD spectra is shown in Fig. 5. Nb films at different positions on the cavity showed strong peak at Nb (110). The average grain size was calculated using the Scherrer equation and the FWHM of Nb (110), as shown in Table 1. The grain size values of all samples were in the range of 300 nm to 800 nm. The lattice constant varies between 3.29356 Å and 3.30977 Å with an average of 3.294 Å. The Nb (110) peaks of samples at different positions of the cavity were at 38.61 °. The above results indicate that there is little lattice distortion in the Nb film, and the lattice parameters are close to those of the bulk Nb materials.

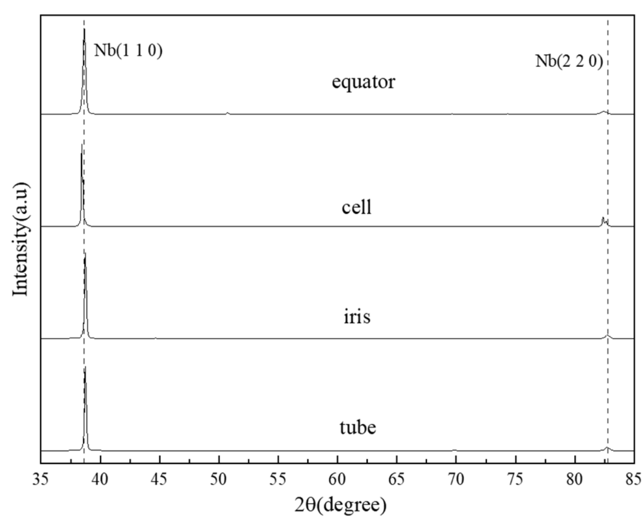

Figure 5: XRD spectrum of Nb films on different positions of the dummy cavity.

Table 1: Peak Position, Grain Size and Lattice Parameter Calculated from XRD Spectrum

Sample Position	(110) Position (°)	(110) Grain Size (nm)	Lattice Parameter (Å)
Equator	38.6	389	3.29356
Cell	38.4	882	3.30977
Iris	38.73	510	3.28564
Tube	38.73	540	3.28581

Figure 6 showed the critical temperature Tc and superconducting transition width ΔT for Nb films. They are important superconducting performance parameters. When Tc is high, and ΔT is small, superconducting niobium thin films usually have good crystalline quality. The Tc and ΔT values obtained from the superconducting transition curve are shown in Table 2. The Tc values at all positions are close to the theoretical Tc value of 9.2 K for bulk Nb. However, ΔT values are about 0.2 K, indicating few impurities in the Nb film.

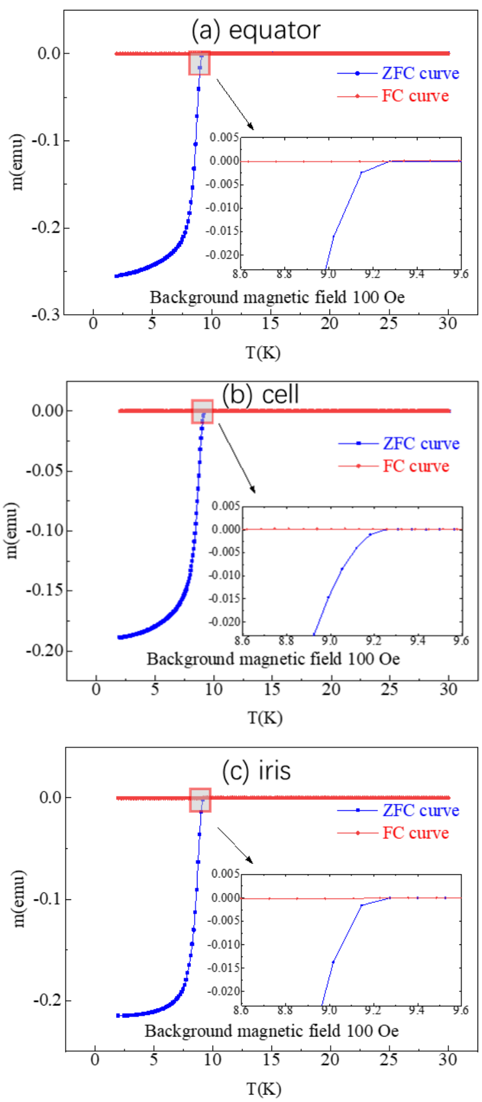

Figure 6: Tc measurements of Nb films at different positions on the cavity.

Table 2: Tc and ΔT Obtained from the Superconducting Transition Curve

Position	Tc (K)	Δ T (K)
Equator	9.27	0.25
Cell	9.24	0.19
Iris	9.27	0.26

RRR determines the average free path and purity of niobium films. The RRR value is calculated by the following equation:

$$RRR = R_{300K}/R_{10K} \tag{1}$$

where R_{300K} and R_{10K} are the resistances of Nb thin films at 300 K and 10 K, respectively. Figure 7 showed the R-T curve for Nb films deposited on Si at cell obtained by in-line four probe method. The Tc is 9.24 K with a ΔT of 0.11 K which is consistent with the VSM results, indicating excellent superconducting performance. In addition, the calculated RRR value is 33, which is higher than the 17 obtained by the previous DCMS method, indicating a good improvement in the quality of Nb thin films.

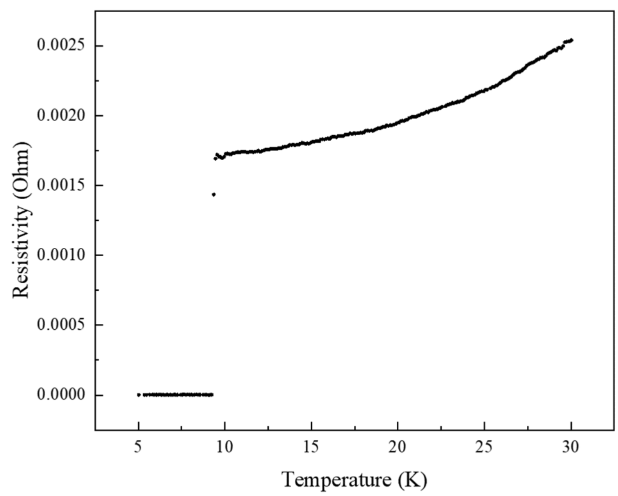

Figure 7: Resistivity of Nb films measured at temperatures from 5 K to 30 K.

CONCLUSIONS

We have prepared high quality Nb films on dummy cavity samples via HiPIMS. The Nb films showed good surface morphology continuity, dense cross-sectional structure, lattice parameters close to bulk Nb, and excellent superconductivity at different positions of the cavity, indicating that HiPIMS is highly promising in manufacturing high-quality Nb thin film cavities. In future, we will mount a cylindrical mesh as anode in between the Nb target and the RF cavity to deposit Nb films on dummy cavity via HiPIMS with bias voltage.

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REFERENCES

[1] C. Benvenuti, N. Circelli, and M. Hauer, "Niobium films for superconducting accelerating cavities", *Applied Physics Letters,* vol. 45, p. 583, 1984. doi:10.1063/1.95289

[2] S. Bauer *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. https://jacow.org/SRF99/papers/WEP016.pdf

[3] S. Stark et al., “Niobium Sputter-Coated QWRs”, in *Proc. SRF'97*, Padova, Italy, Oct. 1997, paper SRF97D37, pp. 1156-1163. https://jacow.org/SRF97/papers/SRF97D37.pdf

[4] W. Weingarten, "Progress in thin film techniques", in *Proc. SRF'95*, Gif-sur-Yvette, France, Oct, 1995, paper SRF95D02, pp. 129-142. https://jacow.org/SRF95/papers/srf95d02.pdf

[5] M. C. Burton, A. D. Palczewski, C. E. Reece, and A-M. Valente-Feliciano, “Progress with Nb Hipims Films on 1.3 GHz Cu Cavities”, in *Proc. SRF'19*, Dresden, Germany, Jun.-Jul. 2019, pp. 823-827. doi:10.18429/JACoW-SRF2019-THFUB2

[6] F. Avino, D. Fonnesu, T. Koettig, M. Bonura, and M. Taborelli, "Improved film density for coatings at grazing angle of incidence in high power impulse magnetron sputtering with positive pulse", *Thin Solid Films,* vol. 706, p. 138058, 2020. doi:10.1016/j.tsf.2020.138058

[7] M. Arzeo *et al.*, "Enhanced radio-frequency performance of niobium films on copper substrates deposited by high power impulse magnetron sputtering", *Supercond. Sci. Technol,* vol. 35, p. 054008, 2022. doi:10.1088/1361-6668/ac5646

[8] S. Leith *et al.*, "HiPIMS deposition of superconducting Nb thin films onto Cu substrates", *Vacuum,* vol. 212, p. 112041, 2023. doi:10.1016/j.vacuum.2023.112041

[9] F. Y. Yang, J. Dai, P. He, Z. Q. Li, Y. Ma, and P. Zhang, “Study of Surface Treatment of 1.3 GHz Single-cell Copper Cavity for Niobium Sputtering”, in *Proc. SRF'19*, Dresden, Germany, Jun.-Jul. 2019, pp. 605-609. doi:10.18429/JACoW-SRF2019-TUP068