

Co-sputter deposition of Nb₃Sn layer into SRF cavity using Nb-Sn composite target*

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Abstract

Nb₃Sn, with its superior superconducting critical temperature ($T_c \sim 18.3$ K) and superheating magnetic field ($H_{sh} \sim 400$ mT), is considered a promising material for superconducting radiofrequency (SRF) cavities, offering enhanced cryogenic performance compared to bulk Nb cavities. A Nb₃Sn coating technique has been developed for SRF cavities using co-sputtering of Nb-Sn composite target in a DC cylindrical magnetron sputtering system. The composite target configuration and discharge conditions for co-sputtering were optimized to deposit Nb-Sn films on flat Nb substrates, followed by annealing to form Nb₃Sn. Methods have been explored to improve the surface homogeneity of the Nb₃Sn coating, including optimizing a two-step annealing process, and a light Sn recoating process. A 1.5 μm Nb-Sn co-sputtered film was deposited on the interior of a 2.6 GHz Nb SRF cavity and annealed at 600 °C for 6 h, followed by 950 °C for 1 h. Cryogenic RF testing of the annealed cavity demonstrated a T_c of 17.8 K, confirming the formation of Nb₃Sn. Then, the annealed cavity underwent a light Sn recoating treatment and attained a quality factor (Q_0) of 8.5E+08 at 2.0 K.

INTRODUCTION

SRF cavities made from high-purity Nb have played a central role in modern particle accelerators due to their excellent superconducting properties and ease of fabrication into complex structures [1-5]. Significant progress in cavity performance has been achieved through advancing surface preparation techniques such as electropolishing, doping, and heat treatments to improve the Nb cavity surface quality [6-9]. However, bulk Nb cavities are now approaching their fundamental performance limits in terms of accelerating gradient and surface resistance [10,11]. Nb₃Sn, an A15 compound, offers several advantages over Nb, including a higher T_c and H_{sh} , nearly double those of Nb, can improve SRF cavity efficiency and enable operation at elevated temperatures, thereby reducing cryogenic load and operating costs [12-14]. The most widely used method for coating Nb₃Sn onto Nb cavities is Sn vapor

diffusion [15-17]. Recently, a cryomodule based on vapor diffused Nb₃Sn coated Nb cavities has demonstrated an accelerating gradient of about 10 MV/m with low cryogenic loss at 4.4 K [18]. While effective, this process presents challenges such as need for stringent control on Sn evaporation sources, non-uniform patchy regions and Sn-deficient areas on the coating surface, and the requirement for high processing temperatures [16,19]. Magnetron sputtering is an alternative method offering improved control over film stoichiometry, faster deposition process, and lower substrate temperatures [20-22]. We discuss the deposition of Nb₃Sn onto a 2.6 GHz Nb SRF cavity using a composite Nb-Sn target inside a custom-designed DC cylindrical magnetron sputtering system at Old Dominion University [23]. The process development includes optimization of the co-sputtering target configuration and plasma discharge conditions for achieving suitable stoichiometry, followed by optimizing post-deposition annealing strategies and a surface treatment using a light Sn recoating step. Lastly, cryogenic RF test results of the co-sputtered Nb₃Sn coated cavity are presented.

CO-SPUTTERING DEPOSITION OF NB-SN ON FLAT SUBSTRATES

To identify optimum composite Nb-Sn target configuration and discharge condition, several combinations of Nb and Sn ring targets (OD 0.9", ID 0.8") were tested using the bottom magnetron of the cylindrical sputter coater. The lengths of the Nb and Sn targets in the composite configuration were adjusted to achieve the desired Nb-Sn stoichiometry in the sputtered film. The target configuration shown in Fig. 1(a) was identified optimal. Fig. 1(b) shows the plasma discharge, for the target configuration in Fig. 1(a), at 11 mTorr Ar pressure for 53 mA discharge current. One strong plasma glow ring appears around the central Nb target, while three weaker glow rings are observed around the Sn targets. These glow rings correspond to the magnetic field distribution across the magnetron surface [23]. Using this target configuration, Nb-Sn films were deposited on flat Nb substrates (10 × 10 × 3 mm) mounted at three representative locations of a replica 2.6 GHz Nb SRF cavity: at top beam tube, equator, and bottom beam tube. Before deposition, the chamber's base pressure reached 2×10^{-7} Torr. The sputtering discharge was carried out at 11 mTorr Ar deposition pressure using a fixed magnetron discharge current of 53 mA. The magnetron speed was adjusted to ensure uniform coating thickness, and $\sim 0.5 \mu\text{m}$

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thick Nb-Sn films were deposited on Nb samples at the beam tubes and equator.

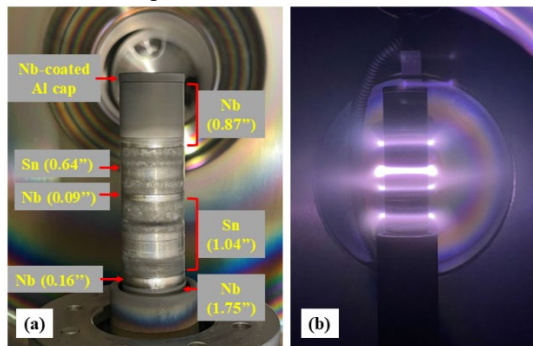


Figure 1: (a) Composite Nb-Sn target configuration used for co-sputter deposition, (b) Plasma discharge during co-sputtering at 11 mTorr Ar pressure using the composite Nb-Sn target.

ANNEALING FOR IMPROVED SURFACE HOMOGENITY

The sputtered films on Nb substrates were annealed in a high-vacuum furnace at Jefferson Lab to facilitate Nb₃Sn phase formation by reacting Nb and Sn in the as-deposited Nb-Sn layer [17]. The annealing process began once the base pressure of the Nb insert accommodating the samples inside the furnace chamber was pumped down to the low 10⁻⁵ Torr. The furnace temperature ramped at 12 °C/min from room temperature to 950 ± 10 °C, followed by a 3 h hold at that temperature.

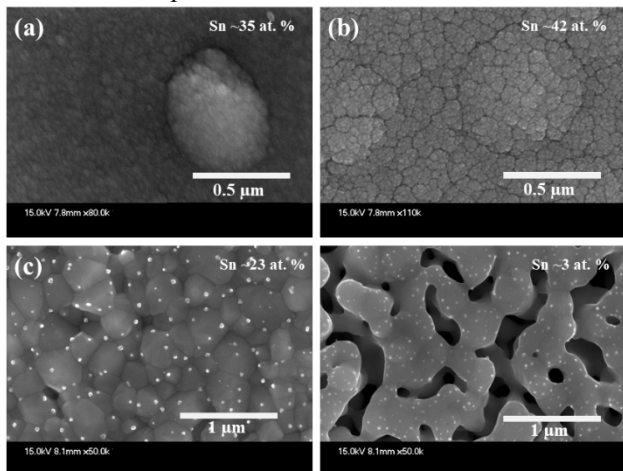


Figure 2: Surface morphology of co-sputtered films using the composite Nb-Sn target: as-deposited (a) top beam tube and (b) equator sample; 950 °C - 3 h annealed (c) top beam tube and (d) equator sample.

Energy-dispersive X-ray spectroscopy (EDS) analysis showed that the as-deposited samples contained ~32–42 at. % of Sn. Scanning electron microscopy (SEM) images revealed that the as-deposited samples did not exhibit well-formed grains, rather showed randomly oriented, nanoscale particle-like features, as shown in Fig. 2(a) and Fig. 2(b). After annealing at 950 °C for 3 h, X-ray diffraction (XRD) patterns confirmed the formation of Nb₃Sn in all annealed samples, each exhibiting polycrystalline

Nb₃Sn phase, without any diffraction peak corresponding to other Nb-Sn phases. EDS measurements of the annealed top beam tube and bottom beam tube samples indicated ~23 at. % Sn content. However, the annealed equator sample showed a significantly lower Sn content of ~3 at. %. SEM analysis demonstrated that the surfaces of the annealed beam tube samples consisted of homogeneous and uniformly distributed Nb₃Sn grains with clearly defined boundaries and an average grain size of ~260 nm, as shown in Fig. 2(c). However, the annealed equator sample exhibited elongated Nb₃Sn structures with poorly defined grain boundaries and large porous regions across the surface, as shown in Fig. 2(d).

This disparity is likely due to the equator sample being located ~40 mm from the sputter target surface, compared to the beam tube surfaces those were only ~8 mm away, allowing more magnetron plasma interaction during deposition. Also, the surface cracks observed in the as-deposited equator sample (Fig. 2 (b)) may have provided pathways for rapid Sn evaporation during annealing, leading to significant Sn loss. Small particle-like structures ranging from 17 to 36 nm in size were observed on the surfaces of annealed samples.

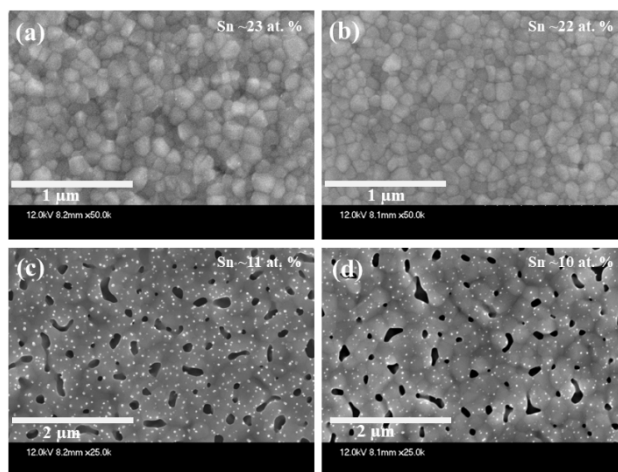


Figure 3: SEM images of top beam tube samples annealed at (a) 600 °C for 1 h, followed by 950 °C for 3 h and (b) 600 °C for 6 h, followed by 950 °C for 1 h. Corresponding SEM images of annealed equator samples are shown in (c) and (d), which underwent similar annealing steps as (a) and (b), respectively.

SEM images of top beam tube samples annealed at 600 °C for 1 h, followed by 950 °C for 3 h and annealed at 600 °C for 6 h, followed by 950 °C for 1 h are shown in Fig. 3(a) and 3(b), respectively. In both annealing conditions, the top beam tube sample demonstrated well-formed Nb₃Sn grains without presence of voids.

To reduce Sn evaporation and void density from the equator sample as a result of annealing, multiple annealing approaches were investigated. A preliminary annealing step at 600 °C was introduced and the optimum duration for this step was determined, prior to annealing at 950 °C. Additionally, the annealing duration at 950 °C was reduced from 3 h to 1 h to minimize Sn loss. It was observed that

equator sample which underwent annealing at 600 °C for 1 h, followed by 950 °C for 3 h, demonstrated surface void coverage area of about 16 %, shown in Fig. 3(c). Another equator sample which underwent annealing at 600 °C for 6 h, followed by 950 °C for 1 h, exhibited surface void coverage area of about 9 %, as shown in Fig. 3(d). The Sn content in both annealed equator samples increased to 10–11 at. %.

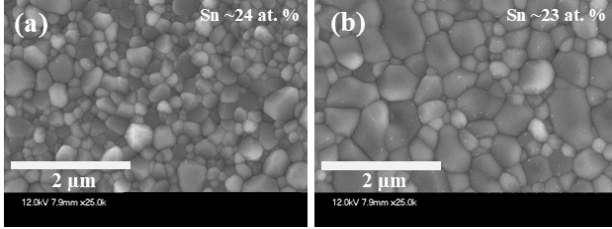


Figure 4: SEM images of (a) top beam tube and (b) equator samples after light Sn recoating treatment. Prior to recoating, both co-sputtered Nb-Sn films were annealed at 600 °C for 6 h, followed by 950 °C for 1 h.

We explored an additional surface treatment involving light Sn recoating using vapor diffusion at Fermilab, which has previously been applied on Nb₃Sn coated 1.3 GHz Nb cavities to heal cracks in the Nb₃Sn layer [24]. In our study, light Sn recoating was applied to eliminate voids and enhance surface homogeneity in co-sputtered samples after annealing. The top beam tube and equator samples, annealed at 600 °C for 6 h followed by 950 °C for 1 h, underwent the light Sn recoating process. The treatment successfully eliminated surface voids and promoted the formation of Nb₃Sn grains. It is plausible that during the Sn recoating process, Sn diffuses into the Nb substrate through void areas, enabling Nb₃Sn formation and effectively eliminating the voids. Hence after recoating process, the equator sample exhibited larger Nb₃Sn grains, with grain sizes reaching up to 975 nm in diameter as shown in Fig. 4(b), compared to the top beam tube sample as shown in Fig. 4(a).

CO-SPUTTERING DEPOSITION OF Nb-Sn INTO SRF CAVITY

An anodized single-cell 2.6 GHz Nb SRF cavity, fabricated from high-purity Nb, was installed in the deposition chamber for Nb-Sn coating using the composite target configuration shown in Fig. 1(a). The base pressure reached 1.9×10^{-7} Torr, and magnetron discharge was carried out at 47 mA. During deposition, the discharge power was varied between 16.7 to 17.6 W while the magnetron moved inside the SRF cavity, resulting in the deposition of about 1.5 μm of co-sputtered Nb-Sn film. The coated cavity underwent high-pressure rinsing at 50 bar using ultra-pure deionized water. The as-deposited surface appeared visually uniform, with a consistent appearance across the cavity and no visible signs of film peeling or particle adhesion. Next, the cavity was placed inside a Nb box and transferred to a high-vacuum furnace at Fermilab for annealing. Once the furnace reached a base pressure of about 8.8×10^{-7} Torr, a degassing step was performed at 200 °C for 60 h. The

annealing procedure began with a temperature ramp of 3 °C/min from room temperature to 600 °C, where it was held for 6 h. This was followed by a ramp of 12 °C/min to 950 °C, with a 1-h hold at that temperature. After annealing, the cavity surface displayed a uniform, slightly whitish coloration across its entire surface. The cavity then underwent a light Sn recoating treatment using vapor diffusion, following the procedure described in [24].

The annealed cavity was removed from the furnace and prepared in a cleanroom for cryogenic RF testing in the vertical test stand system at Fermilab. The temperature dependence of the loaded quality factor (Q_L) exhibited two distinct transitions: one at about 17.8 K, corresponding to the T_c of the Nb₃Sn layer, and another at about 7.4 K, attributed to possible Sn-deficient Nb₃Sn in the equator region of the cavity. The Q_0 versus E_{acc} performance of the Sn-recoated 2.6 GHz Nb cavity was measured during cryogenic RF testing. As shown in Fig. 5, At T_{bath} of 4.2 K, the cavity achieved quality factor Q_0 of 1.5×10^8 at low fields, with the accelerating gradient E_{acc} limited to 4 MV/m. At $T_{bath} = 2.0$ K, Q_0 increased to 8.5×10^8 at low fields, with E_{acc} constrained to 6.1 MV/m. The T_c of the Sn-recoated cavity was measured to be about 17.6 K, attributed to the Nb₃Sn layer.

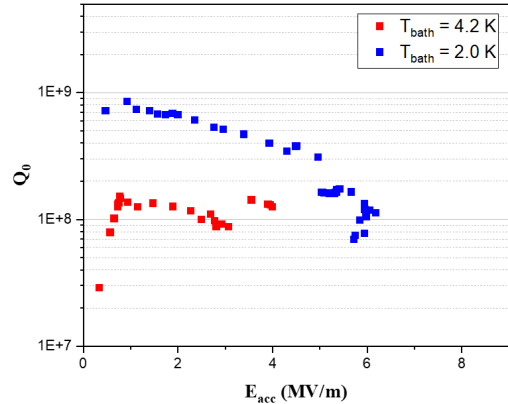


Figure 5: Q_0 versus E_{acc} performance after light Sn recoating of the 2.6 GHz Nb cavity. Prior to recoating, approximately 1.5 μm of Nb₃Sn was deposited inside the cavity via co-sputtering, followed by annealing at 600 °C for 6 h and then 950 °C for 1 h.

CONCLUSION

Nb₃Sn coating was deposited by co-sputtered Nb-Sn films on flat Nb substrates. Annealing strategies were optimized to reduce surface voids and improve Sn retention in the annealed films. A ~1.5 μm thick co-sputtered Nb-Sn layer was deposited inside a 2.6 GHz Nb SRF cavity. The cavity was annealed at 600 °C for 6 h, followed by 950 °C for 1 h. During cryogenic RF testing, the annealed cavity exhibited a T_c of about 17.8 K. After undergoing a light Sn recoating treatment, the cavity demonstrated a Q_0 of 8.5×10^8 at low fields, with E_{acc} limited to 6.1 MV/m at 2.0 K.

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