

SPUTTER COATING OF Nb₃Sn INTO SRF CAVITY USING STOICHIOMETRIC TARGET*

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Abstract

Nb₃Sn has emerged as a leading alternative material due to its higher superconducting critical temperature ($T_c \sim 18.3$ K) and superheating field ($H_{sh} \sim 400$ mT), promising a viable solution to the intrinsic performance limit currently faced by Nb superconducting radiofrequency (SRF) cavities. We sputter-coated Nb₃Sn inside Nb SRF cavity using a stoichiometric Nb₃Sn tube target in a DC cylindrical magnetron sputter coater. The target was fabricated by growing an estimated >20 μm thick Nb₃Sn layer on a Nb tube via Sn vapor diffusion using Jefferson Lab's coating system. Nb-Sn films of thickness about 150 nm were sputter-deposited onto flat Nb samples at positions representing the beam tubes and equator of a 2.6 GHz Nb cavity. Post-deposition annealing at 950 °C for 3 h resulted in the formation of Nb₃Sn. Microstructural analysis of the annealed films was carried out to investigate the morphology and structure of the Nb₃Sn films. Later, a 2.6 GHz Nb SRF cavity was coated with a ~ 1.2 μm thick sputtered Nb-Sn film using a stoichiometric Nb₃Sn target, followed by annealing. Cryogenic RF testing of the annealed cavity demonstrated a T_c of 17.8 K, indicating the formation of Nb₃Sn. After a light recoating treatment, the cavity achieved a quality factor (Q_0) of 6.7E+08 at lower field at 2.0 K.

INTRODUCTION

Nb₃Sn is a promising candidate for next-generation SRF cavities due to its favourable superconducting properties, enabling efficient operation at higher temperatures compared to bulk Nb cavities, which is approaching its theoretical performance limit [1-3]. Nb₃Sn coated cavities can operate efficiently at 4.2 K while maintaining high quality factors, potentially replacing Nb cavities operated at 2 K and significantly reducing cryogenic operating costs [4-6]. The most widely used method for coating Nb₃Sn onto Nb SRF cavities is the Sn vapor diffusion process [7-9]. However, magnetron sputtering has the potential to provide a viable alternative to deposit Nb₃Sn at lower temperatures on different substrates, offering improved control over film composition, uniformity, and deposition rate. [10, 11].

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Previous works have demonstrated Nb₃Sn coating into SRF cavities using a DC cylindrical magnetron sputtering system via multilayer sequential and co-sputtering methods [12, 13]. In this work, we extend the sputtering approach by coating a 2.6 GHz Nb cavity using a stoichiometric Nb₃Sn tube target, custom-fabricated for use in a cylindrical magnetron sputter coater. The sputtering parameters were optimized, and Nb₃Sn coating was first deposited on flat Nb substrates inside a mock cavity, and later into a 2.6 GHz Nb cavity. This method enables direct deposition of Nb-Sn films from the stoichiometric Nb₃Sn target surface, hence offering more precise control over the coating composition onto the inner surface of the Nb SRF cavity.

STOICHIOMETRIC TARGET PREPARATION AND PLASMA DISCHARGE

A stoichiometric Nb₃Sn tube target was prepared by growing a thick Nb₃Sn layer on a high-purity Nb tube (99.95% purity). First, the Nb tube (OD 0.9" × ID 0.8" × length 4.5", purchased from Matsurf Inc.) underwent buffered chemical polishing (BCP) using a 1:1:1 mixture of HF (49%), HNO₃ (70%), and H₃PO₄ (85%), removing approximately ~ 25 μm from the surface.

Later, Nb₃Sn layer was grown on the surface of the Nb tube by Sn vapor diffusion method using the Nb₃Sn coating system at Jefferson Lab [14]. The Nb tube was suspended vertically inside a Nb chamber using a Nb wire during the coating. High-purity Sn (99.9999%, Alpha Aesar), loaded into a crucible, and SnCl₂ (99.99%, Sigma Aldrich) powder, packaged into Nb foil pieces, were placed on a Nb cover plate at the chamber's base. The chamber was closed at both ends using Nb covers with molybdenum fasteners inside an ISO4 cleanroom and later transferred to the Nb₃Sn coating furnace. The temperature profile for Nb₃Sn growth included a nucleation step at (540 ± 10) °C for 90 min, preceded by a ramp at 6 °C/min from room temperature. This was followed by a ramp at 12 °C/min to the coating temperature of (1195 ± 10) °C. The coating temperature was cumulatively kept constant for 148 h to grow a Nb₃Sn coating of a thickness of about 20 μm on the Nb tube target surface. The temperature runs were interrupted after ~ 60 hours to prevent Sn exhaustion and to allow refilling of the Sn source.

As shown in Fig. 1(a), the stoichiometric Nb₃Sn target was installed on the bottom magnetron of the cylindrical sputter coater, which has two magnetrons that can move axially in the vertical direction. The cylindrical magnetrons

and control software were developed by Plasmionique Inc. The software regulates both the discharge and the movement of the magnetron inside the cavity. A 1.75" long Nb target was mounted on the bottom magnetron, followed by the 4.5" long stoichiometric Nb₃Sn target. An aluminium spacer coated with ~1 µm of sputtered Nb layer was used to secure the Nb₃Sn target on the magnetron. The chamber was evacuated down to 1.8×10^{-7} Torr, and the plasma discharge was initiated at 11 mTorr with 50 sccm background Ar (99.99%) flow. Figure 1(b) shows the plasma discharge when the magnetron is powered using 30 W DC power, showing the plasma rings produced around the stoichiometric Nb₃Sn target surface across the magnets inside the magnetrons, while Fig. 1(c) shows marks on the target surface after use for the sputtering discharge [15].

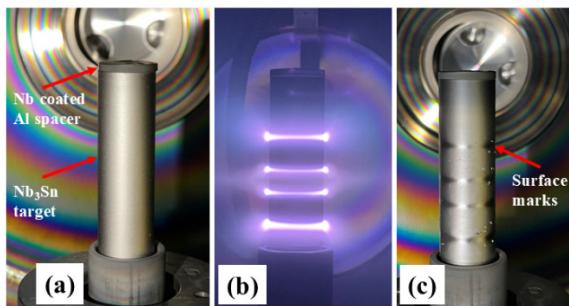


Figure 1: (a) Stoichiometric Nb₃Sn target mounted on the cylindrical magnetron, (b) plasma discharge during sputtering at 11 mTorr Ar pressure, (c) the target surface showing ring marks due to depletion after use.

NIOBIUM TIN COATING ON FLAT SUBSTRATES AND MATERIAL ANALYSIS

Using an aluminium-made mock cavity and a custom-made sample holder, flat Nb substrates were mounted on three locations representing the top beam tube, equator, and bottom beam tube of a 2.6 GHz Nb SRF cavity. Flat Nb substrates (10 × 10 × 3 mm) were made from high-purity Nb sheets (RRR ~300, obtained from Tokyo Denkai Co., Japan) using electrical discharge machining, followed by BCP for ~100 µm material removal. The Nb substrates were wiped with ethanol, then with acetone, and dried with air before using for deposition. With chamber base pressure of 2.4×10^{-7} Torr and a deposition pressure of 11 mTorr with an Ar background of 50 sccm, the magnetron was powered using 30 W DC power in constant power mode. The magnetron current during the plasma discharge was within 96–107 mA while the magnetron travelled inside the mock cavity. The bottom magnetron completed five passes across the mock cavity during the deposition, with moving speeds of 0.94, 0.13, and 1.02 mm/s across the top beam tube, equator, and the bottom beam tube region of the mock cavity, respectively. With similar deposition conditions, another deposition was conducted on sapphire substrates (12 × 12 mm, 430 µm thick, double-sided polished, C-M plane, obtained from University Wafer) mounted on the beam tubes and equator location of the mock cavity. The thickness of the sputtered Nb-Sn films on sapphire was

measured with a surface profiler (Bruker Dektak XT) to be about 149, 170, and 153 nm, for the top beam tube sample, equator sample, and bottom beam tube sample, respectively. Later, the sputtered Nb-Sn samples, both on Nb and sapphire substrates, underwent annealing treatment at 950 °C for 3 h in a high vacuum furnace at Jefferson Lab [14].

The Sn content in the as-deposited Nb-Sn films on sapphire substrates was measured to be within 25–26 at.%, which was consistent with the composition of the stoichiometric Nb₃Sn target surface. After annealing, the Sn content in the beam tube location samples was measured within 19–20 at.%. The reduction in Sn is attributed to Sn evaporation during the annealing process, which has been observed in earlier works on Nb₃Sn fabrication via sputtering [10]. However, the annealed equator sample exhibited significantly lower Sn content of about 3.5 at.%.

SEM analysis of the samples on Nb substrates shows that the as-deposited film from the top beam tube exhibits tiny grain-like structures on the surface, visible as bright features in Fig. 2(a). Annealed samples on Nb substrates exhibited well-defined Nb₃Sn grains of similar size (about 87 nm in diameter) in the top beam tube and bottom beam tube samples, as shown in Fig. 2(b) and 2(d), respectively. The annealed equator sample exhibited an average grain size of about 75 nm, as shown in Fig. 2(c). However, localized regions of non-uniformity with voids are observed on the annealed equator sample surface, with plausibly reduced film thickness in those areas. Such regions may turn into deeper voids or porous channels as film thickness increases. These areas are likely to promote increased Sn evaporation during annealing, resulting in the lower Sn content observed by EDS in the equator sample.

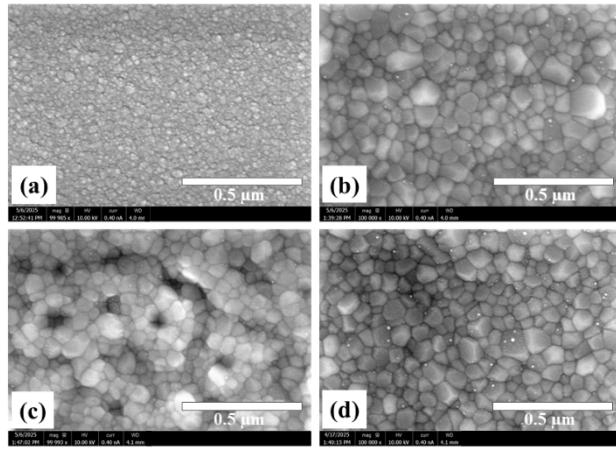


Figure 2: Surface of films sputtered on Nb using the stoichiometric Nb₃Sn target: (a) as-deposited top beam tube sample, and annealed samples at 950 °C for 3 h, prepared at (b) top beam tube, (c) equator, and (d) bottom beam tube.

XRD patterns of the annealed samples from all three locations, top beam tube, equator, and bottom beam tube, showed characteristic Nb₃Sn diffraction peaks of (200), (210), (211), (222), (320), (321), (400), (420), (421), and (332), as shown in Fig. 3. Additionally, Nb peaks (200), (211), and (310), contributed by the substrates, were

observed in all three samples. The Nb (200) and (310) peaks were more intense in the annealed equator sample, suggesting increased X-ray exposure to the underlying substrate due to the regions consisting of surface voids, which aligns with the observation from SEM.

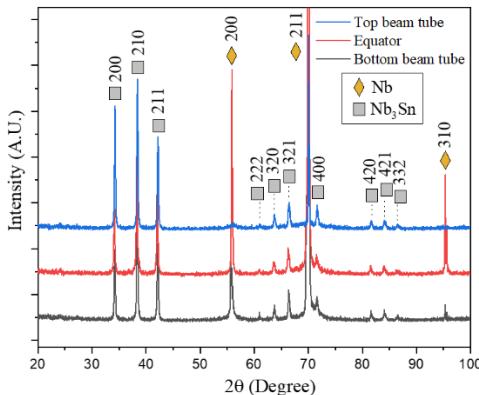


Figure 3: X-ray diffraction patterns of annealed Nb-Sn samples prepared on Nb substrates using a stoichiometric Nb₃Sn target. The samples correspond to the beam tube and equator locations of a mock cavity replicating the geometry of a 2.6 GHz Nb SRF cavity.

CAVITY COATING AND RF PERFORMANCE

A 2.6 GHz Nb SRF cavity was coated via sputtering deposition using stoichiometric Nb₃Sn target, following the similar deposition conditions as used for coating on flat Nb substrates. During the deposition, the bottom magnetron completed 36 passes across the SRF cavity to deposit a ~1.2 μm thick Nb-Sn coating throughout the cavity. Post-deposition, the cavity underwent high-pressure rinsing (HPR) with ultra pure water at 50 bar. Subsequently, the cavity was annealed at 950 °C for 3 h in an ultrahigh vacuum furnace at Fermilab [16].

Nb-Sn film coated cavity surface after HPR, exhibits a uniform coating with consistent coloration and no visible peeling or particulates, as shown in Fig. 4(a). The cavity surface after annealing, as shown in Fig. 4(b), shows a slight grayish coloration is observed, likely due to increased roughness, caused by larger grain size following the annealing. Following the annealing process, the cavity underwent HPR and drying, then was assembled in an ISO4 cleanroom for RF testing in the vertical test stand system at Fermilab [17]. The temperature dependence of the loaded quality factor, Q_L of the annealed cavity demonstrated a transition (T_c) at about 17.8 K, attributed to the superconducting transition temperature of the Nb₃Sn layer. A second transition was observed at about 7.9 K, which is attributed to potential low T_c phases of Nb-Sn other than Nb₃Sn or contribution from the substrate Nb. Later, after the RF test, the cavity underwent a light recoating treatment via Sn vapor diffusion process in the Nb₃Sn coating furnace in Fermilab [18].

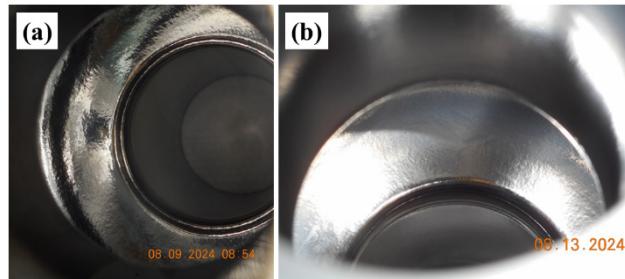


Figure 4: Photographs of Nb-Sn film coated cavity surface: (a) as-deposited, followed by HPR, (b) after annealing at 950 °C for 3 h.

The cavity performance was evaluated after annealing and again following a subsequent light Sn vapor diffusion recoating. After annealing, the cavity was limited to 5 MV/m with a low-field quality factor around 10⁹ at 2 K, as shown in Fig. 5. The light recoating improved the quality factor above 1.5 MV/m; however, the maximum accelerating gradient decreased to approximately 3.6 MV/m. In both cases, the performance was likely limited by poor coating quality near the equator, where non-uniformity was observed, which contributed to excessive Sn evaporation and degraded superconducting properties.

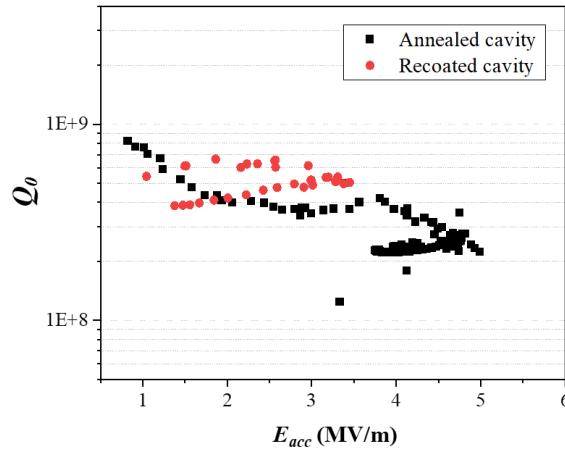


Figure 5: Q₀ versus E_{acc} performance of the cavity at 2 K after annealing and with additional recoating treatment.

CONCLUSION

A stoichiometric Nb₃Sn target was fabricated and used for sputter coating a 2.6 GHz Nb SRF cavity. Nb-Sn films on Nb coupons representing the beam tubes and equator locations of the cavity and annealed at 950 °C for 3 h, exhibited Nb₃Sn phase formation without the presence of any other Nb-Sn phases. A ~1.2 μm thick film was deposited inside the 2.6 GHz Nb cavity, and RF testing confirmed formation of Nb₃Sn coating inside cavity showing a superconducting transition at about 17.8 K. After light recoating treatment, the cavity demonstrated Q₀ of 6.7E+8 at 2.0 K. The coating non-uniformity at the equator due to the distance from the target causes the current performance limitation.

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