Kinetically induced low-temperature synthesis of Nb₃Sn thin films

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ABSTRACT

Nb₃Sn thin films are promising candidates for future application in superconducting radio frequency cavities due to their low surface resistivity, high critical temperature, and critical field, as compared to bulk niobium, which is the current state of the art. In this paper, we report the deposition of Nb₃Sn thin films by magnetron co-sputtering at the extremely low temperature of 435 °C. These thin films show a critical temperature of 16.3 K, a high critical current density of 1.60×10^5 A/cm², and a strong shielding effect. The key to achieving low-temperature growth is the independent kinetic control of Nb and Sn species in the sputtering process. From a technological viewpoint, the low-temperature approach paves the way for the use of Nb₃Sn as a coating in cryogenic efficient copper based cavities, thereby avoiding the detrimental interdiffusion of Cu.

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I. INTRODUCTION

The majority of modern particle accelerators are equipped with superconducting radio frequency (SRF) cavities which are typically fabricated from deep drawn, welded niobium sheets. To maintain its superconducting phase at high magnetic fields, niobium cavities require supercooled helium at 2 K for heat removal. Therefore, even increasing the operating temperature by only 2.2 K to above 4.2 K would considerably reduce the cost of operation. Among different candidates like Nb and Nb–Ti alloys, Nb₃Sn is most promising due to its high critical temperature and low surface resistivity. Nb₃Sn is an intermetallic compound and a type-II superconductor. The bulk critical temperature, T_c , at 0 T magnetic field is about 18 K, and the upper critical field B_{c2} is about 29 T at the lowest temperature.¹ Thus, utilizing Nb₃Sn coatings for SRF cavities could increase tremendously the cryogenic efficiency as compared to existing technologies. Large scale Nb₃Sn coatings could be utilized in combination with Cu cavities providing superior heat conductivity as compared to existing Nb-based cavities, thus, considerably improving cooling efficiency. Among the different intermetallic phases of the Nb–Sn system, only the A15-type Nb₃Sn is superconducting above 10 K. However, thin film synthesis of high quality Nb₃Sn is still a

huge challenge.² Among the reported thin film deposition methods are tin evaporation,^{3–5} chemical vapor deposition,⁶ sputtering,^{7–11} electron beam evaporation,¹² bronze processing,¹³ and electrodeposition.¹⁴ However, the tin diffusion process is the only process used so far for the production of cavities. A key challenge is to control the small stoichiometry window (24%–26% tin content¹⁵) needed for best performance. The huge difference of the equilibrium evaporation pressures of Nb and Sn complicates evaporation based physical vapor deposition methods. Additionally, Sn gradients are known to be the reason for quality factor variations (Q-slope) and early quenching.¹⁶ Until today, most processes relied on combinations of deposition steps and annealing methods utilizing multi-layer approaches. These require high annealing temperatures and long annealing times to form the desired Nb₃Sn-phase. While niobium needs elevated temperatures for moderate diffusivity, these elevated temperatures result in surface segregation and loss of tin. Therefore, the difference in diffusivity of niobium and tin results in concentration gradients after the annealing step. For most annealing approaches, phase pure Nb₃Sn is only formed above 930 °C.¹⁴ For electrochemical approaches in combination with annealing, the lowest formation temperature of superconducting Nb₃Sn was

reported to be 630 °C, requiring additional Cu as a flux medium.¹⁷ However, Cu interdiffusion at this temperature results in a severe decrease of performance, and only a considerable further reduction of growth temperature (as reported here) would allow the combination of Nb₃Sn with Cu. Previously reported approaches potentially lead to inhomogeneous or graded distributions of Nb and Sn and, furthermore, to the formation of non-superconducting phases limiting the desired performance since they all require annealing.^{4,17,18} Tin evaporation is the first and most advanced process for actual production of cavities. Sputtering approaches are significant as an alternative route of coating copper cavities. So far, for the deposition of Nb₃Sn, there are several sputtering approaches reported in the literature. Sputtering is performed either sequentially from two targets forming a multilayer structure of Nb and Sn, which, subsequently, is annealed at high temperatures to form Nb₃Sn or from a single target. The latter approach has to deal with the vastly different equilibrium vapor pressures of both elements. In our case, two separate targets are used in a co-sputtering setup, which allows tuning of the kinetic energies of both elements independently. This leads to phase formation already at substrate temperatures much lower as compared to conditions where the thermal energy is the main driving source of compound forming. The described direct growth of the desired phase without additional annealing steps technologically enables direct coating of Cu cavities.

II. EXPERIMENTAL

Thin films of Nb₃Sn have been deposited on fused silica substrates (5 × 5 mm²) by magnetron co-sputtering. Thereby, co-deposition of Nb and Sn has been carried out at a pressure of 1.0 × 10⁻² mbar, utilizing an Ar flow of 0.58 standard cubic cm per minute. The thin films were synthesized in a high-vacuum chamber with a base pressure between 10⁻⁹ mbar and 10⁻⁸ mbar, with three sputtering guns from Lesker (TORUS 2C). The substrate temperature was controlled by a projection lamp. The individual sputter rate was controlled by the power ratio supplied to the niobium and tin gun. Sputter rates were observed to be most reliable for Nb and Sn guns run with DC and RF power sources, respectively. The distance between target and substrate is set to 12.3 cm. The sputtering time is 25 min, with the goal of about 1 μm total film thickness. X-ray diffraction (XRD) was performed in a grazing incidence setup with $\theta_{\text{source}} = 5^\circ$. Morphology and chemical composition were analyzed using a scanning electron microscopy (SEM) in combination with an energy dispersive x-ray spectroscopy (EDX) analyzer from EDAX. DC resistivity measurements were carried out in a helium supercooled cryostat from OXFORD Instruments. Magnetic measurements were conducted in a Magnetic Property Measurement System from Quantum Design. $T_{C,90}$ is determined by the temperature where the resistivity falls below 90% of its value at 20 K. Kinetic energy distributions were calculated using SIMTRA. RRR-values were determined using the resistivity at 300 K and 20 K.

III. RESULTS AND DISCUSSION

In this work, the stoichiometry, crystalline, and superconducting properties of Nb₃Sn thin films grown at low temperatures are studied. In the growth study, three different ways to tailor the film

properties by co-deposition utilizing magnetron sputtering are discussed and combined to achieve phase pure Nb₃Sn films with high T_C at low deposition temperatures. First, the effect of the ratio of the individual sputtering input powers on film stoichiometry and T_C is explored. Second, the effect of deposition temperature on crystal quality, size, and T_C is evaluated. Third, the effect of the integrated sputtering powers on the performance and morphology is investigated. By combining these three different approaches, the structural and superconducting properties of the films have been fine-tuned, resulting in high- T_C and high critical current density (J_C) films at unprecedented low deposition temperatures.

A. Thin film growth

1. Effect of ratio of individual sputtering powers

First, to obtain the desired tin stoichiometry of 24%–26%, a series of varying power input ratios (defining the stoichiometry) of the respective sputter sources (Sn and Nb, $P_{\text{Nb}}/P_{\text{Sn}}$) has been carried out. To minimize temperature effects on the film stoichiometry, the layers have been deposited at room temperature, followed by a stoichiometry evaluation by EDX. The results are depicted in Fig. 1, showing a hyperbolic dependence of the tin content on the applied power ratio. As can be seen, a broad range of the tin content can be accessed due to the proportionality of the sputtering rate to the input power.¹⁹ To achieve the desired Sn concentration, which has been correlated with the formation of high- T_C Nb₃Sn,¹⁵ a power ratio of 5.25 was utilized.

2. Effect of substrate temperature

To investigate the effect of substrate temperature on the thin film composition, a series with varying temperature conditions at the optimal constant power ratio of 5.25 has been grown.

FIG. 1. Tin content measured by EDX plotted as a function of Nb dc magnetron sputtering power input at room temperature. The stoichiometric tin content in Nb₃Sn of 25% is marked by the line.

The results of the stoichiometry evaluation of this study are shown in Fig. 2. For thin films grown in the temperature range from 260 °C to 435 °C, a constant Sn concentration within the desired window from 24% to 26% Sn was achieved, indicating the independence of film stoichiometry and deposition temperature in this range. This offers a wide temperature range for further thin film optimization. The most important result is that, as opposed to other deposition methods, thin films by magnetron sputtering offer the desired stoichiometry at the targeted low temperatures.

To obtain insight into the impact of deposition temperature on the crystalline quality, the films were characterized by XRD, as illustrated in Fig. 3 (values summarized in Table I). All detected diffraction peaks were correlated to the desired Nb₃Sn-phase (space group Pm $\bar{3}$ n), indicating a high phase purity. No additional diffraction peaks of other phases (see Fig. 3 bottom) are present, further indicating the sole formation of the desired phase. The high intensity peaks at $2\theta = 34.11^\circ$, 38.28° , and 42.10° can be indexed as Nb₃Sn 200, 210, and 211 diffraction peaks, respectively. Note the uncommon peak intensity ratio between the 210, 211, and 200 reflections, which indicates the textured growth along the (100) orientation. Pole figure measurements (see Figs. 1 and 2 in the supplementary material) verified this texture. With decreasing deposition temperature, a systematic and proportional peak shift of all reflexes to lower diffraction angles was observed, resulting from an increase in the lattice constant. Additionally, an increase of the full width at half maximum (FWHM) was observed, indicating a decrease of the crystallite size with decreasing deposition temperatures. In general, a smaller crystallite size results in a higher density of grain boundaries.²⁰ As expected, higher deposition temperatures are beneficial for the formation of larger grains and thus, reduced grain boundary density.

This study aims to achieve a high transition temperature close to the bulk value of 18.3 K, despite the low-temperature growth.¹ Note that besides Nb₃Sn, only Nb₆Sn₅ and NbSn₂ are known superconducting phases, however, with transition temperatures below that of pure Nb (9.2 K).²¹ Resistivity vs temperatures curves are summarized in Fig. 4 as well as in Table I. As can be seen, all films become superconducting, except the film grown at 260 °C. All samples grown at deposition temperatures higher than 280 °C show transitions above 9 K and hence, confirm the formation of Nb₃Sn, for the first time, at deposition temperatures as low as 320 °C.

FIG. 2. Tin content measured by EDX plotted as a function of substrate temperature at a fixed power ratio of 5.25. The stoichiometric tin content in Nb₃Sn of 25% is marked by the dashed line.

On the other hand, all samples grown above 280 °C show a second step in their transition. One scenario could be that Sn deficient Nb₃Sn regions (with a tin content below 25%¹⁵) form. In this scenario, the two steps would indicate the formation of two or more regions with distinct but different Sn concentrations. However, it is unlikely that a second superconducting composition with distinct transition temperature forms. More likely, the step-like feature is due to thermally activated phase slippage (TAPS) across

FIG. 3. XRD patterns of Nb₃Sn thin films grown on fused silica substrates. The substrate temperature is decreased from 435 °C to 260 °C, while the power input is fixed to 105 W Nb and 20 W Sn (power ratio of 5.25). Calculated Nb₃Sn Bragg peaks are marked by tip down triangles in their respective intensity. For comparison, patterns of Nb, Sn, Nb₆Sn₅, and NbSn₂ are given.

TABLE I. Sample overview of Nb₃Sn grown on fused silica by co-sputtering. The substrate temperature was decreased from 435 °C to 260 °C at a constant power ratio of 5.25 and 20 W Sn/105 W Nb. The tin content, FWHM of the 210 Bragg peaks and critical temperature are listed.

T_{sub} (°C)	Sn (at. %)	FWHM (°)	$T_{C0.9}$ (K)
435	25.33 ± 0.15	0.367	13.93
410	25.32 ± 0.16	0.387	15.06
360	24.12 ± 0.58	0.381	14.67
320	23.03 ± 0.36	0.414	11.54
280	24.49 ± 0.39	0.493	7.31
260	22.76 ± 0.38	0.497	...

weakly coupled regions.²² This foot structure could be observed for grain boundaries.²³ In this scenario, the formation of weak links is likely to occur across grain boundaries. This issue could be solved by further increasing the deposition temperatures⁹ as has been demonstrated for different deposition techniques facing loss of Sn. However, as the intention of this work is to keep the growth temperature as low as possible, other solutions are investigated.

3. Role of integrated sputtering power

To further impact film composition, morphology, and superconducting properties in magnetron sputtering at low temperatures, the variation of total or integrated sputtering power has been studied. By increasing the sputtering power, additional kinetic energy is transferred to the system that is associated with a higher mobility of species. Because of the closest peak position to bulk patterns in XRD at 435 °C, a second series of samples has been grown at the highest

FIG. 4. Resistivity normalized to the resistivity at 20 K of sputtered Nb₃Sn thin films vs temperature. The substrate temperature is decreased from 435 °C to 260 °C, while the power input is fixed to 20 W Sn/105 W Nb at a power ratio of 5.25. T_C is determined at 90% of the resistivity at 20 K. Substrate temperatures above 280 °C result in superconducting phases. With increasing substrate temperature, the primary transition temperature tends to increase except for the highest substrate temperature of 435 °C.

FIG. 5. XRD patterns of Nb₃Sn thin films grown on fused silica substrates. The sputtering power was increased from 15 W Sn/79 W Nb to 30 W Sn/158 W Nb, while the substrate temperature was fixed to 435 °C at a power ratio of 5.25. All detected reflections are correlated to Nb₃Sn (Pm3̄n). For comparison, patterns of Nb, Sn, Nb₆Sn₅, and NbSn₂ are given.

temperature of 435 ° used in the previous study, now only varying the overall sputtering power, while keeping the power ratio of both sources constant at 5.25. The XRD measurements of the samples from this series are shown in Fig. 5 and summarized in Table II.

TABLE II. Sample overview of Nb₃Sn grown on fused silica by co-sputtering. The sputtering power was increased from 15 W Sn/79 W Nb to 30 W Sn/158 W Nb at a constant power ratio of 5.25 and 435 °C substrate temperature. Tin content, FWHM of the 210 Bragg peak, and critical temperature are listed.

P_{Sn}/P_{Nb} (W)	Sn (at. %)	FWHM (°)	$T_{C0.9}$ (K)
15/79	17.69 ± 0.34	0.367	13.80
20/105	25.33 ± 0.15	0.387	15.05
25/131	24.07 ± 0.14	0.368	14.82
30/158	25.66 ± 0.11	0.337	16.31

The XRD patterns reveal a preferentially textured growth showing only diffraction peaks of the Nb_3Sn -phase. Note that the diffraction peak positions do not change as a function of sputtering power. The FWHM is the lowest for the sample grown at the

highest sputtering power (see Table II). We have performed additional SEM investigations comparing the films grown using the lowest and highest sputtering power. The results are shown in Figs. 6(a) and 6(b), respectively. As can be seen in Fig. 6(a), a low

FIG. 6. Secondary electron images [(a) and (b)] vs NbL [(c) and (d)] and SnL [(e) and (f)] emission of the same area by EDX of sputtered Nb_3Sn thin films at a fixed substrate temperature of 435°C and a power ratio of 5.25, while the power input is increased from 15 W Sn/79 W Nb (a) to 30 W Sn/158 W Nb (b).

sputtering power leads to the formation of rather small grains. For the highest utilized sputtering power (30 W Sn/158 W) on the other hand, large grains are observed, consistent with the reduced FWHM observed by x-ray diffraction. The grain size is one order of magnitude lower than the film thickness (see Table 2 in the [supplementary material](#)), and therefore, the FWHM has no direct relation to the film thickness. Utilizing EDX mappings [see Figs. 6(c)–6(f)], no segregation of other phases within the resolution limit are detectable. In combination with the obtained XRD results, where no Sn or Nb segregations can be identified, a good homogeneity of the produced films can be assumed.

We show the resistivity behavior of the sample series with increasing total sputtering power in Fig. 7. All samples have a transition temperature above 9.2 K, indicating the formation of Nb₃Sn. Most important, the critical temperature tends to increase with increasing sputtering power from 13.8 K up to 16.3 K. Additionally, the foot structure becomes less relevant. Equally important is the fact that the TAPS foot structures vanish in favor of sharp superconducting transitions in agreement with the observation of increased crystallite sizes at higher sputtering powers. $I(V)$ characteristics and calculated kinetic energy statistic values are listed in Tables 3 and 4 in the [supplementary material](#). Obviously, the increased kinetic energy of the sputtered species and their increased individual fluxes due to the increased overall input power has a beneficial effect on the homogeneous phase formation of Nb₃Sn. The enhanced kinetic energies enable surface diffusion processes across atomic steps and edges, homogenizing the resulting thin film.

Since the coherence length of Nb₃Sn is only a few nanometers, grain boundaries easily act as weak links. The reduction of grain boundary density and the reduction of local stoichiometry fluctuations enriching at grain boundaries suppresses the observed TAPS, leading to sharp superconducting transitions.

FIG. 7. Resistivity normalized to the resistivity at 20 K of sputtered Nb₃Sn thin films vs temperature. The power input is increased from 15 W Sn/79 W Nb to 30 W Sn/158 W Nb, while the substrate temperature is fixed to 435 °C. T_C is determined at 90% of the resistivity at 20 K.

B. Magnetic properties of the superconducting state

In this section, magnetization vs temperature is measured in a field of 2 mT after the zero field (ZFC) and field cooling (FC) for a Nb₃Sn sample sputtered at 435 °C and 30 W Sn/158 W Nb. In Fig. 8, we have plotted the magnetization of the sample measured with a field of 2 mT applied perpendicular to the film plane after the zero field and field cooling. The zero field cooling curve shows a strong shielding effect due to strong superconducting surface currents. The onset temperature of the shielding effect is 15.5 K, which corresponds to the temperature where the resistivity (see Fig. 7) drops to zero and macroscopic superconducting shielding currents can flow. The transition width is similar to experimental results in the literature of Nb₃Sn and other materials. The field cooled measurement shows only a tiny feature at the superconducting transition temperature, indicating strong flux pinning in the sample. This, in turn, implies a high critical current density.

For further characterization, the magnetization vs external magnetic field (hysteresis) was measured as shown in Fig. 9 at 5 K and 10 K and between −6 T and 6 T. At high fields, typical superconducting hysteresis curves are observed. At low fields, flux jumps lead to the shown fluctuation behavior.^{24,25} The onset fields for the flux jumps depend on the force exerted on the flux lines in their pinning centers, which is proportional to the superconducting current density which, in turn, depends on the temperature. As flux jumps result in electrical resistivity, they should be suppressed for applications, e.g., shunting by the cavity material.

From the magnetic hysteresis curves, the superconducting current density can be estimated from the difference between the upper and lower magnetization (ΔM in emu/cm³) at a specific external field strength divided by the sample dimension in the percolation plane (given here by the sample width d which was

FIG. 8. Magnetization vs temperature measured in a field of 2 mT after the zero field (ZFC) and field cooling (FC). Sample was Nb₃Sn sputtered at 435 °C and 30 W Sn/158 W Nb.

FIG. 9. Magnetization of sputtered Nb₃Sn thin films vs external magnetic field measured at 5 K and 10 K.

determined to be 0.5 cm), using the formula,²⁶

$$J_C = 30 \frac{\Delta M}{d}.$$

Using this relation, the critical current density is determined to be 1.6×10^5 A/cm² at 3 T and 5 K.

IV. CONCLUSION

In this work, the direct growth of high quality Nb₃Sn thin films by magnetron co-sputtering at low-temperatures was explored. It has been shown that by tuning the respective deposition conditions, stoichiometry and superconducting properties close to bulk values can be reproducibly obtained. The maximal process temperature is as low as 435 °C, which is of great technological importance for deposition onto a material like copper with much superior heat conductivity as compared to niobium. Our result could be an important step toward the next generation of SRF cavities opening the way to increased acceleration fields and low cooling power consumption at a fraction of production costs of current Nb-based technology.

SUPPLEMENTARY MATERIAL

See the [supplementary material](#) for texture measurements and detailed process parameters of the presented samples.

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DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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