Development and characterization of Nb$_3$Sn/Al$_2$O$_3$ superconducting multilayers for particle accelerators

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Superconducting radio-frequency (SRF) resonator cavities provide extremely high quality factors $> 10^{10}$ at 1–2 GHz and 2 K in large linear accelerators of high-energy particles. The maximum accelerating field of SRF cavities is limited by penetration of vortices into the superconductor. Present state-of-the-art Nb cavities can withstand up to 50 MV/m accelerating gradients and magnetic fields of 200–240 mT which destroy the low-dissipative Meissner state. Achieving higher accelerating gradients requires superconductors with higher thermodynamic critical fields, of which Nb$_3$Sn has emerged as a leading material for the next generation accelerators. To overcome the problem of low vortex penetration field in Nb$_3$Sn, it has been proposed to coat Nb cavities with thin film Nb$_3$Sn multilayers with dielectric interlayers. Here, we report the growth and multi-technique characterization of stoichiometric Nb$_3$Sn/Al$_2$O$_3$ multilayers with good superconducting and RF properties. We developed an adsorption-controlled growth process by co-sputtering Nb and Sn at high temperatures with a high overpressure of Sn. The cross-sectional scanning electron transmission microscope images show no interdiffusion between Al$_2$O$_3$ and Nb$_3$Sn. Low-field RF measurements suggest that our multilayers have quality factor comparable with cavity-grade Nb at 4.2 K. These results provide a materials platform for the development and optimization of high-performance SIS multilayers which could overcome the intrinsic limits of the Nb cavity technology.
screens at $B < B_{c1}$ where $B_{c1}$ is strongly enhanced in films with $d < 2^{8-10}$. Because the inner surface of the Nb cavity is partially screened by multilayers, both $Q(H)$ and the breakdown field can be increased due to lower surface resistance $R_s$ and higher $H_c$ of the layer material. The multilayer coating, which opens up a principal opportunity to break the Nb monopoly in SRF cavities, has been tested by several groups using MgB$_2$, Nb$_3$Sn, NbN, NbTiN, and dirty Nb as coating materials. These experiments have shown an increase of the dc field onset of penetration of vortices on Nb surfaces coated with different SIS structures, although such key SRF characteristics as the surface resistance and quality factors of SIS multilayers under high-amplitude RF fields have been investigated to a much lesser extent. The first results on low-field $Q$ measurements on NbN/MgO multilayers have shown that SIS multilayers can have lower $R_s$ than bulk Nb. However, the SRF performance of Nb$_3$Sn, the current material of choice for the next generation coating material, has not yet been investigated in SIS structures. The development of SIS structures requires overcoming many materials science and technological challenges to achieve good superconducting properties and SRF performance while providing optimal stoichiometry and morphology of the layers and the interfaces and transparency of grain boundaries to extremely high RF current densities. In this work we report results on growth and characterizations of Nb$_3$Sn/Al$_2$O$_3$ multilayers which exhibit good superconducting properties and low-field SRF performance on par with the cavity-grade Nb.

Results and discussion

Multilayer growth. We developed a technique of high-temperature confocal sputtering of Nb and Sn from elemental targets to grow stoichiometric Nb$_3$Sn multilayers with Al$_2$O$_3$ interlayers. Details are given in the Supplemental Information. Thin films and multilayers of different thicknesses were grown on different sapphire single crystal substrates for the subsequent characterizations. For instance, 60 nm thick Nb$_3$Sn films were grown on 10 × 10 mm sapphire substrates for transport, scanning tunneling spectroscopy and electron microscopy characterizations. For RF tests, we grew Nb$_3$Sn/Al$_2$O$_3$ multilayers on 2″ diameter sapphire wafers (R-plane, 300 μm thick). These multilayers had up to three 60 nm Nb$_3$Sn layers separated by 6 nm Al$_2$O$_3$. The thickness of the Nb$_3$Sn layers was chosen to be smaller than the London penetration depth. Films were grown by confocal sputtering on the backside of the wafers to prevent leakage of RF field during cavity measurements. The geometry of multilayer samples used in our RF measurements of quality factors is shown in Fig. 1.

The Nb-Sn phase diagram contains several line compounds. For instance, Nb$_6$Sn$_5$ and Nb$_3$Sn$_3$ coexist in the region marked in Fig. 2a. Here a low-$T_c$ Nb$_6$Sn$_5$ phase is clearly undesirable in these films. Within the Nb$_3$Sn phase region extending from 17 to 25% Sn, the critical temperature $T_c$ degrades steeply as stoichiometry moves away from a 3:1 ratio. These two conditions demand that Nb$_3$Sn films should contain 25% of Sn. This was accomplished by providing processing conditions reflecting the field in the upper right of the phase diagram in Fig. 2a, a two-phase region containing only stoichiometric Nb$_3$Sn and liquid Sn. Films were grown by confocal sputtering of Nb and Sn from elemental targets. By providing a large over-pressure of Sn at high growth temperatures, it has been found that the ratio of Nb:Sn can be pinned at 3:1. The abundance of Sn drives the
material into the two-phase region, where excess Sn re-evaporates from the film, avoiding the formation of Sn precipitates\(^{23,24}\). To achieve the high temperatures (\(> 930^\circ\text{C}\)) required for this growth, sapphire substrates were heated from behind with a SiC radiative heater. Radiation passed through the substrate and heated the depositing metal directly. Growth temperature was measured by pyrometer. Details of the film growth are given in the Supplemental Information.

A series of films was grown with fixed Nb flux (0.7 Å/s) and varying Sn flux (0.4–2.5 Å/s), and low-temperature resistance measurements were carried out to find the window for this self-regulating adsorption-controlled process. Shown in Fig. 2b are the dependencies of the critical temperature \(T_c\) and transition width \(\Delta T_c\) on the deposition rate of Sn which clearly saturate at \(~ 1\) Å/s. Given the dependence of \(T_c\) on Sn content in Nb\(_3\)Sn, this growth rate roughly corresponds to the boundary between two processing regimes. At lower flux, Sn evaporates from the film faster than it can be incorporated, resulting in a Sn-deficient film. At higher flux, sufficient Sn is provided to react with all available Nb, and only excess Sn re-evaporates.

The dielectric Al\(_2\)O\(_3\) interlayers were grown after allowing Nb\(_3\)Sn to cool down to \(< 400^\circ\text{C}\), using a single stoichiometric target with RF power at a rate of 1.8 nm/min without any further heating applied to the substrate. Depositing under these conditions protects the SiC heater element from oxygen evolved during the sputtering process and prevents undesired reactions with the Nb\(_3\)Sn surface. This Nb\(_3\)Sn/Al\(_2\)O\(_3\) stack was then heated again to above 900 \(^\circ\text{C}\), which allows the Al\(_2\)O\(_3\) to crystallize, and the process was repeated to grow heterostructures of up to three Nb\(_3\)Sn layers. The chamber setup and growth steps are depicted in Fig. 3.

**Structural characterization.** A SIS sample with three Nb\(_3\)Sn layers was prepared for analysis by cross-sectional scanning transmission electron microscopy (STEM). A low-magnification image (Fig. 4a) represents the morphology and nanostructure of the stack. Each Nb\(_3\)Sn layer is polycrystalline with irregular interfaces and grain size is 20–100 nm along the film surface direction. The Al\(_2\)O\(_3\) layers conform closely to the layer below but are discontinuous along the Nb\(_3\)Sn/Al\(_2\)O\(_3\) interface. Despite the repeated thermal cycling during stacking, it appears that the lower layers have not degraded in comparison to the top layer.

The chemical stability of these films is further confirmed by compositional mapping with energy dispersive spectroscopy (EDS) (Fig. 4b). Intensity of the Al Ka and O Ka peaks are mapped from the region shown on the left. Although the Al\(_2\)O\(_3\) layers are not continuous, Al and O are confined to the Al\(_2\)O\(_3\) layers, and do not mix with the Nb\(_3\)Sn layers. The exception to this is the presence of O at the interface of the topmost Nb\(_3\)Sn film with the atmosphere, where ambient conditions are sufficient to cause a reaction. A capping Al\(_2\)O\(_3\) layer could be deposited...
to prevent this oxidation, but we did not use it for the multilayer samples described in this work. Note that the slight O signal in the Nb₃Sn layers in Fig. 4b are due to the slight oxidation of the TEM specimen surface. As our RF cavity measurements show, these Al₂O₃ layers do not contribute significantly to surface resistance at low fields.

A higher-magnification image of the S–I interface is shown in Fig. 4c. The atomic structure of Nb₂Sn is well-preserved at the interface, suggesting that there is almost no diffusion or intermixing from the Al₂O₃. The lower Nb₂Sn grain orients the [023] direction normal to the film surface, and this direction is also preserved in the upper Nb₂Sn grain. This can occur when the upper Nb₂Sn layer deposits with the same epitaxial relationship to the underlying Al₂O₃ as the lower layer has with the Al₂O₃ substrate. This structure can also form when a Nb₂Sn...
grain nucleates on top of a Nb$_3$Sn surface exposed by breaks in the discontinuous Al$_2$O$_3$ layer. X-ray diffractometry indicates that Nb$_3$Sn grains in the second layer have more random crystallographic orientation compared to the first layer (see the Supplemental material).

**Superconducting properties.** Our dc transport measurements have shown that the Nb$_3$Sn films capped with Al$_2$O$_3$ and annealed with no further deposition exhibit good superconducting properties. For instance, the superconducting resistive transitions of a bare Nb$_3$Sn film and a Nb$_3$Sn/Al$_2$O$_3$ structure annealed at 900 °C for 10 min are shown in Fig. 5a. Here the critical temperature of the annealed sample is about 0.25 K higher than $T_c$ of the unannealed sample, and residual resistivity ratio (RRR), an indicator of crystalline and metallic quality, is improved from 3.5 to 4.26. On the other hand, Nb$_3$Sn films annealed without the Al$_2$O$_3$ cap, even under high Sn flux to prevent evaporative loss, have degraded superconducting properties compared to an un-annealed film.

The superconducting properties essential for the RF performance were characterized by scanning tunneling spectroscopy (STS) which measures the differential tunneling conductance $dI/dV$ proportional to the quasi-particle density of states (DOS), $N(E)$. Shown in Fig. 5b is a representative tunneling spectrum measured in the center of a Nb$_3$Sn grain at 4 K. The DOS curves, which clearly show the superconducting gap $\Delta$, were fit using the conventional Dynes model:

$$N(E) = N_0 \Re \left[ \frac{E - i\Gamma}{\sqrt{(E-i\Gamma)^2 - \Delta^2}} \right]$$

where the phenomenological parameter $\Gamma$ accounts for the broadening of the DOS peaks due to a finite lifetime of quasiparticles, and $N_0$ is the DOS in the normal state. The fit was done with $\Gamma = 0.4$ meV and $\Delta \approx 3.1$ meV, consistent with the conventional gap value for a stoichiometric Nb$_3$Sn. The ratio $\Gamma/\Delta \approx 13\%$ in our samples turns out to be about 2–3 times larger than the values observed by tunneling spectroscopy on 1–2 μm thick


Nb₃Sn films for rf applications and Nb coupons. The deviations of the STM data from the Dynes model at low energies $E < \Delta$ may indicate the effects of local non-stoichiometry, gap anisotropy and strain, scattering of quasiparticles on magnetic impurities, and a thin layer with deteriorated superconducting properties at the surface. In turn, the subgap quasiparticles states which appear at $|E| < \Delta$ due to a finite $\Gamma$ contribute to a temperature-independent residual surface resistance $R_i$ at $k_BT < < \Delta$. Here $\mu_0$ is the permeability of free space, $\rho_n$ is the normal-state resistivity, $\lambda$ is the magnetic penetration depth, and $\omega = 2\pi f$ is the circular RF frequency. For $\lambda = 120 \text{ nm}, \rho_n = 3.0 \times 10^{-7} \Omega m$, and the fit parameters $\Delta = 3.1 \text{ meV}$ and $\Gamma = 0.4 \text{ meV}$, we obtain $R_i \approx 5.0 \text{ n}\Omega$ at $f = 1.3 \text{ GHz}$. This estimate is of the order of $R_i \approx 5-10 \text{ n}\Omega$ for large-grain Nb cavities. Below a few nm thick surface layer but well within the rf penetration depth $\lambda = 120 \text{ nm}$, the gap peaks in the DOS are likely much sharper. There are other essential contributions to $R_i$, most notably due to non-stoichiometric regions in the bulk, grain boundaries and trapped vortices.

**Low-field RF characterization.** Multilayer samples grown on 2” sapphire wafers were tested in a hemispherical Nb-coated cavity at SLAC National Accelerator Laboratory. The experimental setup was described previously. A rendering of this cavity is shown in Fig. 6a. The cavity operates in a TE₀₃₂-like mode at 11.4 GHz and the surface RF field of the order of 30 μT, with a pocket on the flat face for mounting 2”-diameter samples (shown in purple). The overall cavity quality factor is measured, and the properties of the wafer can be deduced by comparison with known samples. The geometry of the cavity is engineered such that the magnetic field is strongest at the sample surface, limiting the contribution of the cavity material to the overall cavity loss. Accord-
ing to simulations, the participation factor is 0.33 for the 2”-diameter sample. Crucially, the magnetic field at the sample is in the radial direction and parallel to the sample surface, making it possible to measure RF properties of the sample without interference from the perpendicular component of the field. Low-field measurements of $Q(T)$ of a Nb$_3$Sn film in an uncoated Cu cavity are presented in the Supplementary Information.

The SRF performance of two Nb$_3$Sn samples were compared in this system to a cavity-grade bulk Nb coupon. A 500 nm (~4k) Nb$_3$Sn film intended to completely screen out the RF magnetic field, and a 3 × 60 nm Nb$_3$Sn/Al$_2$O$_3$ trilayer were tested under the RF field. Both samples were coated with a 200 nm Nb film on the backside of the wafer to prevent leakage of magnetic field as shown in Fig. 1. The quality factor of the cavity with each sample, measured at low power with a network analyzer, is plotted in Fig. 6b. The abrupt increase in Q at about 15 K corresponds to the superconducting transition of Nb$_3$Sn, followed by an increase of Q(T) at $T_c = 9$ K of the Nb-coated host cavity.

As shown in Fig. 6b, the thick Nb$_3$Sn film and the trilayer have nearly identical Q at T < 9 K, indicating that Al$_2$O$_3$ dielectric layers and interfaces do not contribute significantly to the RF dissipation. We would expect the thick Nb$_3$Sn film to have a higher Q, as magnetic field is more fully screened before reaching the substrate and backside, so this result suggests that the maximum Q of these films and multilayers is limited by the quality of the Nb$_3$Sn material rather than by the interfaces with Al$_2$O$_3$. The quality factors of both the film and the trilayer samples exceed Q(T) of Nb at T > 6 K due to the higher $T_c$ of Nb$_3$Sn and is about 2 times smaller than Q of Nb at 4 K.

Discussion. The results of this work show that, despite the obvious non-stoichiometry and inhomogeneity of superconducting properties, grain boundaries, Nb inclusions, and incomplete Al$_2$O$_3$ layers, our multilayers with three 60 nm Nb$_3$Sn layers separated by 6 nm Al$_2$O$_3$, and both had Q approximately 2 times lower than a backside, so this result suggests that the maximum Q of these films and multilayers is limited by the quality of the Nb$_3$Sn material rather than by the interfaces with Al$_2$O$_3$. The quality factors of both the film and the trilayer samples exceed Q(T) of Nb at T > 6 K due to the higher $T_c$ of Nb$_3$Sn and is about 2 times smaller than Q of Nb at 4 K.

The slopes of Q(T) for both the Nb$_3$Sn film and multilayer shown in Fig. 5 tend to level off at 4–5 K and are clearly smaller than the slope of Q(T) for Nb. This indicates that Q(T) of the Nb$_3$Sn samples at T = 4–5 K is not limited by the BCS surface resistance for which the slope of Q(T) $\propto e^{-\Delta/4k_BT}$ for Nb$_3$Sn would be larger than Nb because $\Delta_{Nb3Sn} \approx 2\Delta_{Nb}$. The behavior of Q(T) of the Nb$_3$Sn samples at 4–5 K is thus indicative of a significant residual surface resistance caused by the multiphase structure of the films and multilayers and trapped vortices. Yet $Q_0 \approx 10^4$ observed on our Nb$_3$Sn multilayers at 11.4 GHz and 4 K suggests values of $Q_0 \sim 10^7$ at 4 K and 1 GHz given the frequency dependence $Q \propto \omega^{-2}$ which comes from the BCS surface resistance, ohmic losses in metallic precipitates smaller than the RF skin depth and perhaps Josephson vortices trapped on grain boundaries.

SRF performance at high RF fields and breakdown fields of Nb$_3$Sn/Al$_2$O$_3$ multilayers are yet to be explored. Generally, the effects of nonstoichiometry, proximity-coupled normal precipitates and weakly-coupled grain boundaries become more pronounced at higher RF fields. For instance, nonstoichiometric grain boundaries in Nb$_3$Sn have been identified as prime pinning centers for vortices in Nb$_3$Sn wires for high-field dc magnets. However, weakly-coupled grain boundaries in Nb$_3$Sn coating layers would block RF currents and cause dissipative penetration of Josephson vortices at fields well below the superconducting transition $T_c$ and sub-stoichiometric regions in Nb$_3$Sn-coated Nb cavities are suspected to play an important role in RF cavity quench. At the same time, meandering and breaks in Al$_2$O$_3$ layers shown in Fig. 4 may not be detrimental for SRF performance as the layers can still provide their main role of intercepting and pinning small vortex loops originating at surface structural defects since the pinholes sizes 10–50 nm in the Al$_2$O$_3$ layers are smaller than magnetic size of the vortex $\lambda \approx 100–200$ nm of Nb$_3$Sn. The misaligned breaks with lateral sizes smaller than the Nb$_3$Sn layer thickness in neighboring dielectric Al$_2$O$_3$ layers are not expected to strongly deteriorate the SRF performance of multilayers. Such imperfect dielectric layers still produce effective pinning barriers against penetration of parallel vortices and arresting vortex semi-loops originating on surface materials defects, which is instrumental in the multilayer approach. At the same time, the roughness and breaks in Al$_2$O$_3$ interlayers, as well as the variable thickness of Nb$_3$Sn layers can pin short perpendicular vortices and do not let them propagate along the layers under RF current, which is also beneficial for the SRF performance of multilayers. Though Al$_2$O$_3$ layers do not fully separate Nb$_3$Sn layers, we found that a 500 nm thick Nb$_3$Sn film had a quality factor identical to a multilayer with three 60 nm Nb$_3$Sn layers separated by 6 nm Al$_2$O$_3$, and both had Q approximately 2 times lower than a cavity-grade Nb reference. This indicates that losses in the thin Al$_2$O$_3$ and the oxide-metallic interfaces do not contribute much to the surface resistance of our multilayer samples.

Conclusions. In summary, we have developed a self-regulating, adsorption-controlled process for growth of Nb$_3$Sn films and Nb$_3$Sn/Al$_2$O$_3$ multilayers. We have produced and characterized multiple multilayer samples with up to four superconducting layers. Despite the detrimental effects of nonstoichiometry, grain boundaries and breaks in the meandering Al$_2$O$_3$ interlayers, the SRF performance of our multilayers turned out to be on par with that of Nb films. The growth technique reported in this work provides a platform for further optimizations of the SRF properties of SIS high-performance multilayers for superconducting resonator applications.
Methods

Film deposition was carried out in a vacuum chamber pumped down to 3.0 × 10⁻⁸ Torr before being backfilled with Ar. Nb₃Sn films were sputtered from elemental Nb (99.95%) and Sn (99.99%) targets in 3 mTorr of Ar to maximize deposition rate, at a distance of 15.5 cm from the substrate to improve flux uniformity. DC power to the sputter guns was current-controlled, and deposition rate was measured with an in situ quartz crystal monitor.

Superconducting transitions were measured in a closed-loop He cooler using 4-point van der Pauw geometry on 10 × 10 mm samples. The critical temperature \( T_c \) is defined as the temperature at which the sheet resistance falls below 1% of its normal state value at 18 K. The transition width \( \Delta T \) is defined as a difference between \( T_c \) and the point at which the lines drawn through the normal-state resistance and transition region intersect.

Low-temperature scanning tunneling microscopy/spectroscopy (STM/S) measurements were carried out in a STM system (USM1300, UNISOKU) at 4 K using polycrystalline PtIr tips. The dI/dV spectra were acquired using standard lock-in technique by applying a bias modulation of 0.2 mV (r.m.s.) at 732 Hz.

Data availability

The data that supports the findings of the work are in the manuscripts main text and Supplementary Information. Additional data are available from the corresponding author upon reasonable request.

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Author contributions
C.S. developed multilayer growth process and fabricated samples. J.M. participated in RF characterization. Y.S., F.K., and L.X. performed cross-sectional TEM characterization. H.Z. and L.L. performed low-temperature STM characterization. P.B.W. performed low-temperature RF surface resistance characterization. C.B.E and A.G. initiated and directed the project. C.S and A.G. wrote the manuscript with contributions from all authors.

Competing interests
The authors declare no competing interests.

Additional information
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