Nb$_3$Sn multicell cavity coating system at Jefferson Lab

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ABSTRACT

Superconducting radio frequency niobium cavities are the building blocks of modern accelerators for scientific applications. Lower surface resistance, higher fields, and high operating temperatures advance the reach of the future accelerators for scientific discovery as well as potentially enabling cost-effective industrial solutions. We describe the design and performance of an Nb$_3$Sn coating system that converts the inner surface of niobium cavities to an Nb$_3$Sn film. The niobium surface, heated by radiation from the niobium retort, is exposed to Sn and SnCl$_2$ vapor during the heat cycle, which results in about 2 $\mu$m Nb$_3$Sn film on the niobium surface. Film composition and structure as well as radio frequency properties with 1-cell R&D cavities and 5-cell practical accelerator cavities are presented.

I. INTRODUCTION

Superconducting radio frequency (SRF) cavities are building blocks of modern particle accelerators. State-of-the-art SRF cavities accelerate particle beams in the Continuous Electron Beam Accelerator Facility (CEBAF), Spallation Neutron Source (SNS), European X-ray Free-Electron Laser facility (European XFEL), and other accelerators.\textsuperscript{1–4} The energy reach and operational efficiency are defined by the properties of the top-most surface layer of SRF cavity surfaces, where the surface (within about 100 nm) RF current shields the superconductor Meissner state. The quality of the niobium surface has been steadily improved over the years by growth in the understanding of the relationship between surface treatments and RF properties of the underlying superconductor.\textsuperscript{5,6} While the work to improve the niobium surface, which is the superconductor of choice so far, continues, interest is growing toward superconductors with a higher critical temperature. In particular, Nb$_3$Sn has been revisited and could be a solution for near-term compact scientific and industrial accelerators due to its potential to sustain a factor of two higher magnetic fields and two orders of magnitude higher quality factor than that of niobium. Various techniques have been developed to grow the Nb$_3$Sn superconductor. Bronze routes, sputtering techniques, tin dip, etc., have been studied (see Refs. 7–10 and references therein), but require further development due to the exceptional quality of the RF surface necessary to sustain high RF fields. The best Nb$_3$Sn-coated SRF cavities so far have been produced with the so-called vapor diffusion process.\textsuperscript{11} The process is attributed to Saur and Wurm\textsuperscript{12} and comprises exposure of the niobium surface to tin vapors at temperature above about 900 $^\circ$C. This process has been and is being typically used to grow several $\mu$m-thick Nb$_3$Sn layers on the inside of niobium SRF cavities.\textsuperscript{13–17} This contribution describes the developed Nb$_3$Sn-coating system and coating process for coating R&D as well as accelerator SRF cavities.
and a high-vacuum coating chamber built at Jefferson Lab that contains coated samples and process vapors. The system was designed to separate process vapor in the coating chamber from the furnace heating environment in order to avoid cross-contamination and to increase the flexibility and longevity of the system.

B. High-vacuum furnace

The top-loaded vertical furnace was procured from T-M Vacuum Products, Inc. The furnace was specified to reach \(1315 \degree C\) with the vacuum in the \(10^{-7}\) Torr range empty and was built with three hot zones. Each hot zone comprises two 2 in.-wide low-resistance molybdenum flat elements powered by a 20 kW power supply. Each hot zone is independently controlled by a calibrated molybdenum-sheathed type R thermocouple. Three hot zones create an effective cylindrical heating space of about 50 cm long and 40 cm diameter, where the temperature is uniform to within 3\(^\circ\) and controlled to within 1\(^\circ\) by an Allen-Bradley 5/05 Programmable Logic Controller (PLC). Three molybdenum and two stainless steel (SS) sheets outside the heating elements serve as radiation shields to isolate the hot zone from the electropolished vacuum vessel, which is water-cooled by 6 gpm room temperature water. The interface door on the top of the furnace was modified to increase the process volume. A custom water-cooled retort door was built by Lesker Company. A new extension to the existing heat shield with six molybdenum sheets was built in-house to extend the hot zone length to about 80 cm.

Prior to the process initiation, the chamber is evacuated by a Leybold SC 30 scroll pump. When the pressure drops to below 0.1 mbar, the roughing pump is isolated and shut off. The gate valve to a Sumitomo Marathon 250 cryopump is opened to establish high vacuum in the furnace. Vacuum levels are monitored with thermocouple vacuum gauge tubes and a single cold cathode sensor. The standard base vacuum prior to start of the heating process is below \(10^{-8}\) Torr. A typical furnace vacuum level during process runs rises into the \(10^{-7}\) Torr range, sometimes reaching lower \(10^{-9}\) Torr for about 10 min–20 min. Residual gas analysis, using a differential pumping setup, indicates that hydrogen is the dominating species at high temperatures.

C. Niobium reaction chamber

The coating chamber was built out of 4 mm niobium sheets into a 17 in. OD by 40 in. long cylinder via rolling half cylinders and their electron beam welding. Furnace heating elements on the outside irradiate the cylinder, heating it to the desired temperature up to \(1300 \degree C\). The cylinder irradiates the inside, where the coating setup with Nb-substrates and Sn-sources is located, heating the reaction chamber to the desired process temperature. The niobium cylinder is closed from one end with a 4 mm niobium blank, which was deep-drawn into dome shape and electron beam welded to the cylinder shell. The other end of the cylinder shell was welded to a 21 in. OD titanium (Ti6Al4V) flange with a half-dovetail 1/4 in. o-ring groove for Viton o-ring seals. On one side of the flange, the seal provides vacuum insulation via the o-ring to the new furnace door; on the other side, the o-ring seals against a zero-length water-cooled reducer, which reduces the opening from 21 in. OD to 14 in. copper gasket seal. This zero-length reducer allows us to use instrumentation and pumping on a smaller multiport spool top plate (Fig. 1).

The reaction chamber is initially evacuated with either Edwards nXDS 15i pump or Edwards TIC pumping cart to below 10 Torr. Once the pressure reaches 10 Torr, the Pfeiffer HiPace 300C turbo pump is turned on to bring the pressure to below \(10^{-5}\) Torr before the process is started. Vacuum levels are monitored with Edwards active Pirani vacuum gauges (APG100) for soft vacuum and Edwards Active Inverted Magnetron (AIM) Gauge for high vacuum. A Residual Gas Analyzer (RGA) SRS300 from the Stanford Research system with differential pumping is sometimes used to record the residual gas composition during the process. A sample RGA scan at room temperature and at \(1200 \degree C\) is shown in Fig. 2.
Three 0.5 in. OD niobium rods extend into the hot zone space from the top plate of the multiport spool piece. Six heat shields made out of niobium are attached to the rods to reduce radiative heat loss from the hot zone. A sample chamber or SRF cavities are suspended from a 4 mm niobium plate, and its covers, which form the reaction space, is used as the total surface area for the mass calculations. Niobium covers, 3 mm–4 mm thick, are used to cover the open sample chamber or cavity ports and restrict the gas flow from the inside of the cavity, where Sn and SnCl$_2$ are placed, during the process. Witness samples are hung inside the cavity by attaching them to the top cover using niobium wires. The crucible as well as all other niobium covers are assembled to a sample or RF cavity with niobium brackets and molybdenum hardware inside the cleanroom.

III. COATING DESCRIPTION

Sn shots (99.999% purity from Sigma-Aldrich) and SnCl$_2$ powder (99.999% purity from Sigma-Aldrich) packaged inside niobium foils are placed inside the niobium crucible. About 3 mg/cm$^2$ of Sn and a similar amount of SnCl$_2$ are used for each Nb$_3$Sn coating, where the surface area of the cavity and its covers, which form the reaction space, is used as the total surface area for the mass calculations. Niobium covers, 3 mm–4 mm thick, are used to cover the open sample chamber or cavity ports and restrict the gas flow from the inside of the cavity, where Sn and SnCl$_2$ are placed, during the process. Witness samples are hung inside the cavity by attaching them to the top cover using niobium wires. The crucible as well as all other niobium covers are assembled to a sample or RF cavity with molybdenum hardware in the cleanroom and double bagged before being transferred to the thin film lab. Typically, molybdenum hardware that is used to fasten niobium covers to open ports is lightly tightened to about 1 ft-lb. In the thin film lab, the cavity is attached to the deposition chamber and installed in the Nb$_3$Sn deposition chamber. After the furnace and the reaction chamber are evacuated to ultra-high vacuum (UHV) and high vacuum (HV), respectively, the heating cycle is started. The standard temperature profile has two temperature ramps: the first ramp to 500°C at 6°C/min and the second ramp to 1200°C at 12°C/min (Fig. 3). The standard temperature profile has two plateau regions, at 500°C for 1 h and 1200°C–1250°C for 3 h–24 h. During the first temperature plateau at 500°C, only SnCl$_2$ evaporates because of the higher vapor pressure ($\approx 100$ Torr) compared to that of Sn ($\approx 10^{-10}$ Torr), which reacts with the niobium surface, and Sn nucleation sites are formed on the surface. The nucleation produces Sn droplets on the surface from a few tens of nanometers up to about half a micrometer size on the surface. By the time the second plateau is reached at 1200°C, the Sn vapor deposits on the surface around the nucleation sites, and Nb$_3$Sn grains begin to crystallize on the surface. The growth proceeds in the presence of the Sn vapor resulting in micrometer-sized grains after 3 h. The temperature profile shows the temperatures measured with the three furnace thermocouples located inside the furnace, but outside the coating chamber. The thermocouples are separated vertically by 10 cm, and typically, they are within ±1°C of the target temperature during soaks. Inside the chamber, niobium- or tantalum-sheathed type C thermocouples are used to monitor the temperature of the coated samples. In the case of large structures, such as 5-cell CEBAF, the cavity temperature measured on the cavity is about 50°C lower than the temperature indicated by the control thermocouples outside, cf. orange and gold lines in Fig. 3. The vacuum curves show the vacuum in the furnace, inside and outside the coating chamber (Fig. 3). The vacuum inside the coating chamber is typically about $10^{-3}$ Torr and increases during the coating run up to about $10^{-2}$ Torr due to Sn and SnCl$_2$ vapors.

Single-cell R&D cavities are coated using both one-cavity and two-cavity setups. The best results are achieved using the two-cavity setup. In this setup, two cavities are high pressure water rinsed separately and then assembled together at one of the flanges using niobium brackets and molybdenum hardware inside the cleanroom. Two cavities then form the reaction chamber. They are covered with a niobium cover on the top and niobium crucible on the bottom, temperature profile has two plateau regions, at 500°C for 1 h and 1200°C–1250°C for 3 h–24 h. During the first temperature plateau at 500°C, only SnCl$_2$ evaporates because of the higher vapor pressure ($\approx 100$ Torr) compared to that of Sn ($\approx 10^{-10}$ Torr), which reacts with the niobium surface, and Sn nucleation sites are formed on the surface. The nucleation produces Sn droplets on the surface from a few tens of nanometers up to about half a micrometer size on the surface. By the time the second plateau is reached at 1200°C, the Sn vapor deposits on the surface around the nucleation sites, and Nb$_3$Sn grains begin to crystallize on the surface. The growth proceeds in the presence of the Sn vapor resulting in micrometer-sized grains after 3 h. The temperature profile shows the temperatures measured with the three furnace thermocouples located inside the furnace, but outside the coating chamber. The thermocouples are separated vertically by 10 cm, and typically, they are within ±1°C of the target temperature during soaks. Inside the chamber, niobium- or tantalum-sheathed type C thermocouples are used to monitor the temperature of the coated samples. In the case of large structures, such as 5-cell CEBAF, the cavity temperature measured on the cavity is about 50°C lower than the temperature indicated by the control thermocouples outside, cf. orange and gold lines in Fig. 3. The vacuum curves show the vacuum in the furnace, inside and outside the coating chamber (Fig. 3). The vacuum inside the coating chamber is typically about $10^{-3}$ Torr and increases during the coating run up to about $10^{-2}$ Torr due to Sn and SnCl$_2$ vapors.

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which hosts 6 g of Sn and 3 g of SnCl₂ and is covered with molybdenum or niobium diffuser. The best results are achieved when another small tin source is added at the top, and a temperature gradient of about 80 °C, as measured by the thermocouples attached to the cavities, from the top to the bottom is used.

In the case of 5-cell cavities, the bottom crucible is loaded with 10 g of Sn and 3 g of SnCl₂. The best results are achieved when additional Sn sources are used. In such cases, one or two crucibles are added, which are positioned at the top and at the center of the cavity. They are typically loaded with 1 g–2 g of Sn, and the amount of Sn in the bottom crucible is lowered accordingly. The nucleation step in 5-cell cavity coatings is extended to 5 h, and the coating step is typically extended beyond 3 h. It was 1250° for 6 h for the cavity coatings reported below.

IV. SRF FILM PERFORMANCE

A. Analysis of small samples

10 × 10 cm² coupons are cut from 3 mm thick high purity [Residual Resistivity Ratio (RRR) ≈300] niobium sheet material by Electrical Discharge Machine (EDM) cutting. Each sample receives ≈100 μm Buffered Chemical Polishing (BCP) removal using a solution of 49% HF, 70% HNO₃, and 85% H₃PO₄ in the ratio of 1:1:1 by volume. These samples are either coated in a sample coating chamber or as witness samples with RF cavities using the Nb₃Sn coating procedure described above.

The surface topography is measured with a Digital Instruments Nanoscope IV Atomic Force Microscope (AFM) in tapping mode using silicon tips with diameters less than 10 nm. Each sample was scanned in three different regions with a scan size of 5 μm by 5 μm. AFM images revealed depressions on Nb₃Sn grains formed by curved facets (Fig. 4). These depressions were also visible in Secondary Electron Microscope (SEM) images as a dark spot in many grains; see Fig. 4. The typical average rms roughness was found to be close to 70 nm in each coated sample for 5 μm by 5 μm scans.²⁰

The electron backscatter diffraction (EBSD) map of the coated sample cross section is shown in Fig. 5. The map shows that the grains, apparent in the SEM image, are single crystals. There is no clear preferential grain orientation or correlation to the adjacent grains and underlying niobium. Neither significant crystal orientation variation inside the grains, nor evidence of lateral or depth compositional variation in the coated Nb₃Sn is observed.²¹ The EBSD data are consistent with energy-dispersive x-ray spectroscopy (EDS) measurement, which shows that the compositional variation in our Nb₃Sn is within the instrument sensitivity and reproducibility (24.3 ± 2) at. %. We have not found significant variations in the Nb₃Sn composition outside this range. Secondary ion mass spectrometry (SIMS) data were collected on some samples with a CAMECA IMS-7f magnetic sector instrument. The data show that Nb₃Sn extends to the depth of about 1 μm with little variation in Nb and Sn concentrations. SIMS results were further corroborated by x-ray photoelectron spectroscopy (XPS) data, which were collected on a ULVAC-PHI “Quantera SXM.” High resolution XPS scan of Nb and Sn peaks shows that Nb₃Sn is covered with Nb₂O₃ and SnO₂.

The transition temperature of one of the coated samples was measured via the 4-probe resistive measurement technique. The transition temperature was found to be 18 K, which is consistent with the transition temperature from cavity measurements.

B. Single-cell cavity results

Single-cell cavities RDT7 and RDT10 were made from a high purity (RRR ≈300) fine-grain niobium sheet by stamping and electron beam welding. Each cavity has received the standard surface preparation after manufacturing: over 100 μm surface material removal and several heat treatments at 800 °C for 2 h or 3 h.²² In the baseline test before Nb₃Sn coating, the cavities reached the accelerating gradient (Eacc) of about 30 MV/m with the low-field Q₀ of about 1.6 · 10¹⁰ at 2 K.
Cavities were coated using the two-cavity setup.22 A temperature gradient of about 85 °C between the top (cold) and bottom (hot) of the paired cavity setup was used. Almost Q-slope free results were achieved as shown in Fig. 6. The measured value of RDT7 low-field $Q_0$ was 3 $\cdot$ 10^10 at 4 K and 10^{11} at 2 K without a significant Q-slope. The cavity maintained a $Q_0$ of about 2 $\cdot$ 10^{11} at 4 K and above 4 $\cdot$ 10^{11} at 2 K before quench at above 15 MV/m. Post-coating inspection showed uniform coating inside the cavity. Tin consumption was very similar between the two coatings. Examination of witness samples showed uniform coating without any tin residue or patches. EDS examination showed usual NbSn composition. RF test results from RDT10 were similar to RDT7 except for the quench field, which was lower in RDT10. Coated cavities at 4 K exhibited a quality factor of about 3 $\cdot$ 10^{10}, which is almost two orders of magnitude higher than that of niobium cavities. The field-dependence of surface resistance caused quality factor degradation to 2 $\cdot$ 10^{10} at $E_{acc} \approx 15$ MV/m, equivalent to the peak surface magnetic field of about 60 mT. Field reach was limited by a thermal breakdown, which has been a common limitation of the NbSn cavity coated by vapor diffusion. At lower temperature of 2 K, the low field quality factor reached 10^{11} corresponding to about 2 mΩ~3 mΩ of surface resistance similar to the lowest observed in SRF NbSn-coated cavities. The quality factor exhibited a non-trivial field-dependence quickly reducing to about 7 $\cdot$ 10^{8}, corresponding to about 4 mΩ at $E_{acc} = 2$ MV/m (8 mT), and reaching 4.5 $\cdot$ 10^{8} (60Ω) at about $E_{acc} \approx 16$ MV/m (70 mT). The surface resistance of these cavities is one of the best measured at a lower temperature of 2 K, the low field quality factor reached 10^{11}. (HOM) couplers, which complicates application of uniform surface and addition of auxiliary components, such as High Order Mode (HOM) couplers, which complicates application of uniform surface treatment. Similar observations have been made with Nb3Sn coatings of multicell cavities with additional challenge of adequate tin supply throughout the volume of the cavity.23–25 The coating system was designed to coat CEBAF 5-cell cavities. Two 5-cell cavities, 5C75-RI-NbSn1 and 5C75-RI-NbSn2, were built by RI Research Instrument, GmbH according to Jefferson Lab’s specifications. The cavities were built to C75 shape with HOM and Fundamental Power Couplers (FPCs), which allows these cavities to be integrated into a CEBAF cryomodule.26 The cavities were electropolished for 120 μm removal, annealed in vacuum at 800 °C for 2 h, and again electropolished for 25 μm as the final material removal step. The baseline test of each cavity was performed at 2 K. In the test, 5C75-RI-NbSn1 reached $E_{acc} \approx 29$ MV/m with the low-field $Q_0$ of about 2 $\cdot$ 10^{10}. 5C75-RI-NbSn2 was measured up to $E_{acc} \approx 22$ MV/m with the low-field $Q_0$ of about 2 $\cdot$ 10^{10}.

Both cavities were coated using the 5-cell cavity setup described above. After NbSn coating, cavities were ultrasonically cleaned, rinsed with high pressure (80 bars) ultra pure water, assembled, evacuated, and tested. Both cavities were tested in a liquid helium bath at 4.4 K and 2.0 K. Non-uniformity was observed in the coating of 5C75-RI-NbSn1 similar to the earlier observations,27 which was linked to low tin pressure in the early stages of the coating.28 To improve the tin availability, three tin sources were used to coat 5C75-RI-NbSn2 in the configuration described above. The low-field quality factor was about 1.2 $\cdot$ 10^{10} corresponding to about 20 nΩ of surface resistance at 4.4 K. At 2 K, the quality factor improved to about 2 $\cdot$ 10^{10} at low fields. The field-dependence of the surface resistance caused the reduction in the quality factor with field, but, because x rays were observed in the measurements, it was not clear whether the field-dependence was inherent to the film or was caused by field emission loading. The cavity was then shipped to FNAL, where it was partially disassembled, rinsed with high pressure ultra pure water, assembled, evacuated, and tested. The test at FNAL was carried out about two months after the test at JLab. The low-field...
quality factor was measured almost exactly the same as those measured at JLab at both 4.4 K and 2 K (Fig. 7). The field-dependence of the surface resistance was still present and caused the reduction in the quality factor to about \(3 \times 10^6\) (90 Ω) at about 15 MV/m (60 mT) at 4.4 K. No x rays were observed in the latest measurement.

V. CONCLUSION

We have designed and commissioned a coating system, comprising a commercial furnace and custom niobium reaction chamber, for coating Nb$_3$Sn films on the inner surface of SRF cavities. The system is capable of coating multicell accelerator cavities inside the niobium reaction chamber with the vacuum system separated from the furnace vacuum. 2 μm thick uniform Nb$_3$Sn films with the transition temperature of 18 K are grown on small samples and multicell cavities.

Coated single cell cavities exhibit quality factors close to \(3 \cdot 10^{10}\) at 4 K, about two orders of magnitude improvement over typical quality factor, and close to \(5 \cdot 10^{10}\) at 2 K, a factor of three improvement of baseline tests of these cavities. Multicell cavities exhibit quality factors in excess of \(10^{10}\) and reach above 10 MV/m, suitable for accelerator applications.

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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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