ISBN: 978-3-95450-208-0 doi:10.18429/JACoW-IPAC2019-WEPRB110

RECENT RESULTS FROM Nb_3Sn COATED SINGLE-CELL CAVITIES COMBINED WITH SAMPLE STUDIES AT JEFFERSON LAB*

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Abstract

The critical temperature (~ 18 K) and superheating field (~ 425 mT) of Nb_3Sn are almost twice that of niobium, thereby promising a higher quality factor and accelerating gradient at any given temperature compared to traditional SRF cavities made of niobium. It can enable higher temperature for cavity operation (4 K vs. 2 K), resulting in significant reduction in both capital and operating costs for the cryoplant. Several single-cell cavities along with witness samples were coated with Nb_3Sn to explore, understand, and improve the coating process towards improving the cavity performance. RF measurements of coated cavities combined with material characterization of witness samples were employed to update the coating process. Following some modifications to the existing coating process, we were able to produce Nb_3Sn cavity with quality factor $\geq 2\times10^{10}$ at an accelerating gradient up to 15 MV/m at 4 K, without any significant Q-slope. In this article, we will discuss recent results from several Nb_3Sn coated single-cell cavities combined with material studies of witness samples.

INTRODUCTION

Superconducting radio-frequency cavities (SRF) are the leading technology for modern particle accelerators. Niobium (T_c ~ 9.2 K, H_{sh} ~ 210 mT and Δ ~1.45 meV) is the material of choice so far to build them. After several decades of research and development, niobium cavities now operate near fundamental limit of the material [1]. Often they need to operate at ~ 2 K for optimal performance. This demands complicated cryogenic facilities, and it is the one cost driver for SRF accelerators. The intermetallic compound Nb_3Sn (T_c ~ 18.3 K, H_{sh} ~ 425 mT and Δ ~3.1 meV) is a potential alternate material [2] that would allow SRF cavities operation at 4.2 K for similar performance of Nb at 2 K. Nb_3Sn cavity could operate with a simplified small cryogenic facility and potentially enhance the performance. R&D efforts are underway in several labs to produce high performance Nb_3Sn cavities aiming at applications for accelerators [3, 4, 5].

Vapor diffusion process, preferred technique so far to fabricate promising Nb_3Sn coated Nb cavities has been adopted at JLab to coat coupon samples, single-cell cavities, double-cell cavities, and recently multi-cell cavities. Initially, coated cavities had quality factors (Q) as high as ~1×10^{10} at 4 K, but suffered strong Q-slope, limiting the attainable maximum gradient. The Q-slope, very similar to the one seen in early cavities coated at Wuppertal University was consistently seen in several cavities [6]. Following the coating system upgrade in 2017, an almost Q-slope free Nb_3Sn cavity was produced for the first time with $Q < 1\times10^{10}$ at 2 K [7]. Since then we have prepared and tested several cavity coatings. Witness samples were studied as well for continual modifications in the coating process. In this contribution, we will present RF results from two single cell cavities, which had been used repeatedly in several coating experiments. Analysis of witness samples coated with cavities is also discussed.

Nb_3Sn COATING

RDT7 and RDT10 were single-cell cavities made from high purity (RRR~300) fine grain Nb. Both cavities, with low field Q ~ 1.6×10^{10} were limited to ~ 30 MV/m with high field Q slope during the baseline test at 2 K. Each cavity normally received BCP or EP for 15-25 µm removal followed by HPR before the coating.

In the first set of experiments, each cavity was coated individually according to a typical Nb_3Sn coating process at JLab [6]. 3 g of Sn (99.999% purity from Sigma Aldrich) loaded in a crucible and 3 g of $SnCl_2$ (99.99% purity from Sigma Aldrich) packaged inside two pieces of Nb foils were placed inside the cavity at the bottom flange. Both sides of the cavity were closed with Nb covers before installation into the furnace. A witness sample was also hung inside the cavity by attaching it to the top cover using a Nb wire. The heat profile included nucleation at ~ 500 °C for an hour and three hours of deposition at ~ 1200 °C. The temperature was monitored with sheathed type C thermocouples attached to the cavity at different locations. There was a temperature gradient of ~20 °C between the top and bottom of the cavity.

In a second set of experiments, RDT7 or RDT10 was coupled with another single-cell cavity, RDT2. RDT2 had many macroscopic pits inside the cavity, and was used as a dummy cavity on top of RDT7 or RDT10 during the coating. About 3.4 g of tin and 3 g of $SnCl_2$ were placed at the bottom alike the first set of experiments. The coating setup also comprised of a secondary tin crucible, which was loaded with 1.4 g of Sn. It was attached to the top cover with a Nb rod, and hung inside the bottom beam pipe of RDT2. A witness sample was always suspended to secondary Sn crucible with Nb wire. The heat profile was similar to the first set of experiments except there was a

*Partially authored by Jefferson Science Associates under contract no. DE-AC0506OR23177. This material is based upon work supported by the U.S. Department of Energy, Office of Science, Office of Nuclear Physics. This work is supported by Office of High Energy Physics under grants DE-SC-0014475 to the College of William and DE-SC-0018918 to Virginia Tech.

ISBN: 978-3-95450-208-0 doi:10.18429/JACoW-IPAC2019-WEPRB110

temperature gradient of ~ 85 °C between the top and bottom of the whole setup. The bottom tin container was mostly covered with a diffuser, consisting of a molybdenum disk with holes.

RESULTS AND DISCUSSION

RDT7 and RDT10 were coated individually in similar setup and conditions, described above as the first set of experiments. Figure 1 [right] show post-coating pictures from RDT10. Visual inspection indicated uniform coatings inside both cavities.

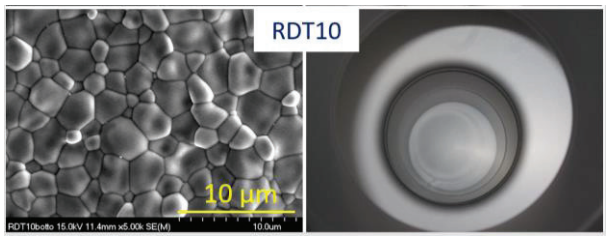

Figure 1: SEM image captured from RDT10 witness sample is shown on the left. Right image is the appearance RDT10 after coating [right].

Witness samples obtained from each cavity coating were examined with secondary electron microscope (SEM) and energy dispersive X-ray spectroscopy. SEM images captured from witness samples showed uniform coating in both cavity coating. SEM image from RDT10 coating is shown in Fig. 1 [left]. EDS analysis of each sample revealed (24.5±0.5) at. % Sn, close to the nominal composition of Nb_3Sn. However, witness samples from both coatings revealed residues on the surface. These features were few tens of nanometer in diameter as shown in Fig. 2 [left]. Probing those residues was not possible with EDS resolution, but some of the larger residues showed ~ 30 at. % Sn compared to neighboring area. It indicated these residues were Sn-rich particles. These residues were mostly removed after one-hour soak in 5 % HCl at room temperature (see Fig. 2) or 10-minute soak in 22% HNO_3 at 110 °C with a stirrer. RF test both at 4 K and 2 K showed precipitous Q-slope in each cavity as shown in Fig. 3. Low field Q at 4 K was $\geq 1\times10^{10}$ with quench field of ~ 10 MV/m. The coating experiments were repeated a couple more times on RDT10 with some variation in coating temperature, which consistently produced similar performance. Since Sn-residues appeared in the witness sample from each coating, they were considered as a potential causative of observed Q-slope.

Several potential solutions were proposed to produce a residue free cavity. The first approach was to remove Sn residues. Acid treatments (described above) or annealing were considered. We only discuss HCl treatment of coated cavity here. RDT10 was given 30 minutes of HNO_3 soak at room temperature to remove possible indium contamination after disassembly from the first test. It was then soaked in 5 % HCl for an hour, similar to witness sample, HPRed and tested again at 4 K and 2 K. Comparison of RDT10 performance before and after acid soak, see Fig. 4, did not show any improvement. We suspected that acid soak might not have removed residues completely, which may not appear in SEM images. It is also not clear yet how such acid soak affects Nb_3Sn surface.

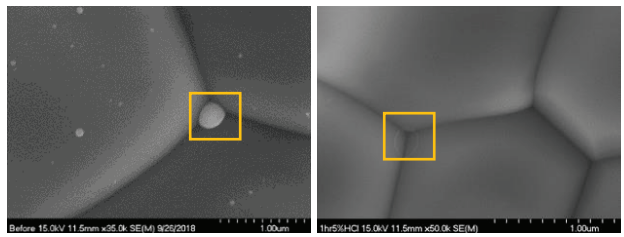

Figure 2: SEM images from RDT7 witness sample before [left] and after [right] 5 % HCl soak for one hour. Bright features are Sn-rich residues.

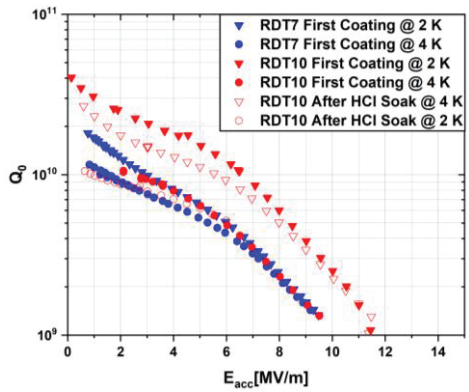

Figure 3: RF test results from RDT7 and RDT10 after first Nb_3Sn coating. Note that RF results following 5 % HCl soak are also shown.

Another approach was to avoid residue formation during the coating. We speculated that the Sn-rich residues were the result of Sn condensation from Sn vapor, which might have been present inside the cavity at the end of the coating. In an attempt to reduce Sn condensation in the cavity of interest, RDT2, was added on top as a dummy cavity, and maintained its temperature lower than RDT7/RDT10 at the bottom, so that tin vapor more likely condenses into RDT2. Coating temperature was set in such a way that there existed a ~ 85 °C gradient between the top and bottom of the paired structure. RDT7 and RDT2 were paired first and coated as described in the previous section. The coating appeared uniform during visual inspection. SEM examination of witness sample exhibited Sn-residues again. Note that the witness sample here was inside RDT2. Since it was suspended from the secondary Sn-crucible, which was attached with Nb rod to the top cover, it could have lower temperature than the cavity, and more likely to have Sn-residues. Since there was no witness sample inside the cavity at the bottom, it was not clear if tin residue was eliminated from RDT7 or not. RF test results obtained from RDT7 are shown in Fig. 4. The measured value of low field Q_0 was 3×10^{10} at 4 K and 1×10^{11} at 2 K without any significant Q-slope. The cavity maintained a Q_0 of ~ 2×10^{10} at 2 K and > 3×10^{10} at 2 K before quench at > 15 MV/m. The cavity performance of RDT7 showed noteworthy improvement over recurrent Q-slope, compared to data previously reported from Wuppertal and Jefferson Lab.

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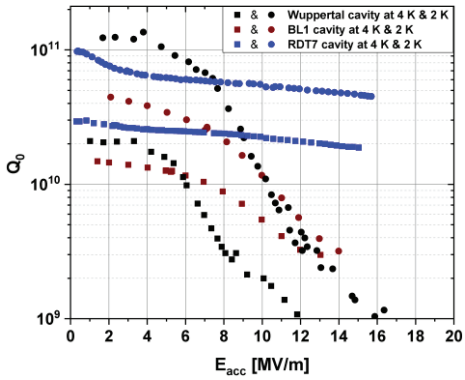

Figure 4: Comparison of RF results from RDT7 with previous data with Q-slopes.

Next, RDT10 was coated together with RDT2 with similar setup and parameters used for RDT7. Another witness sample was installed next to RDT10, which more likely represents the cavity coating. Some non-uniformity was visible first time in the bottom half-cell of the cavity, see Fig. 5 [right]. SEM images from witness sample, next to RDT10 revealed some patchy regions, known to have thinner coating as shown in Fig. 5 [left], which is known to be harmful for RF performance. The RF test result of this cavity (not shown here) was very similar to the previous test result of RDT10, shown inn Fig. 3. Despite having a similar temperature profile compared to previous coating of RDT7, it is found that the consumed amount of tin was almost half (1.7 g vs 3.3 g) during RDT10 coating. It is known that low flux of tin produces defective Nb_3Sn coating with non-uniformity [4]. Reasons behind the lower tin evaporation is not understood completely. We speculate reduction of effective surface area of tin for evaporation. Note that the Mo diffuser, replaced for RDT10 coating, had smaller holes compared to that used in RDT7 coating. It was also suspected to have displaced from its original position during the installation into the furnace. Another attempt was made to coat RDT10 again without diffuser to allow maximum tin evaporation. The cavity was coated uniformly this time, but it was found that Sn was splattered and carried over to the cavity. SEM/EDS analysis of witness sample confirmed the splattering of tin.

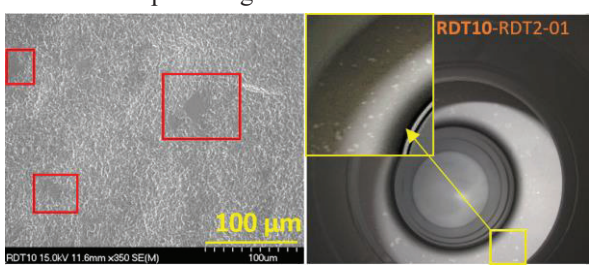

Figure 5: RDT10-RDT2 coating. SEM image [left] shows patchy regions observed in witness sample. Non uniformity in the coating can be seen in the right picture. Note that the top half cell had less non uniformity than the bottom.

The diffuser made from Nb foil, which almost replicated molybdenum diffuser used in RDT7 coating was used in the third attempt to coat RDT10. Supplied tin was reduced based on RDT7 coating experiment, to limit tin vapor at the end of the process. Post-coating inspection showed uniform coating inside the cavity. The Sn consumption was very similar to RDT7 coating. Examination of witness samples, one from the bottom and another next to the bottom beam pipe of RDT2 showed uniform coating without any tin residue or patches, shown in Fig. 6. EDS examination showed usual Nb_3Sn composition. RF test results from RDT10 now appeared similar to RDT7 except the quench field, which was lower in RDT10. The latest test results from RDT10 and RDT7 are compared to the ones after their first coating in Fig. 7.

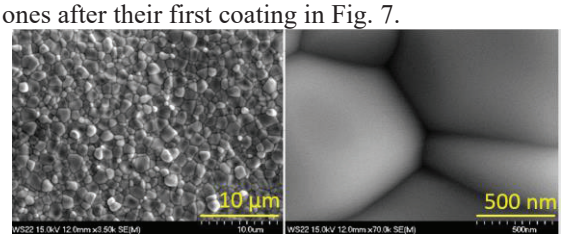

Figure 6: SEM images from RDT10-RDT2 coating. Note that there is Sn residue in the surface [right].

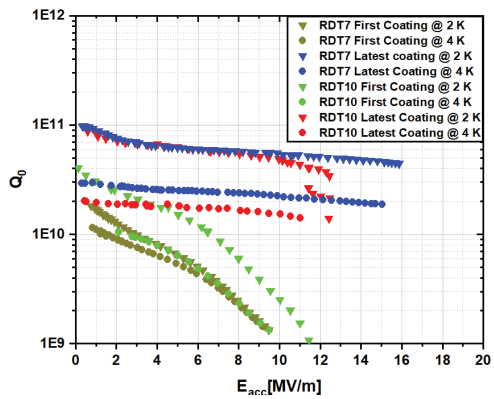

Figure 7: Comparison of latest RF test results from RDT10 and RDT7 with those after their first coating. RDT10 is expected to have higher Q_0 at 4 K than presented here as we expect losses on the flanges because of shorter beam pipes.

SUMMARY AND OUTLOOK

Several single cell cavities coated during the first years of R&D at Jefferson Lab resulted in a strong Q-slope. Analysis of the corresponding witness sample revealed a distribution of Sn-rich residues on the surface. These features mostly disappeared following HCl/HNO_3 acid soak in SEM images, but similar treatment did not improve the cavity performance. In an attempt to reduce Sn residues formation during the coating process, changes were made in the coating process, which resulted in nearly Q-slope free cavities. The best coated cavity had a low field $Q_0 \sim 10^{11}$ at 2 K. $Q_0 \geq 2\times10^{10}$ was measured for accelerating gradient up to 15 MV/m at 4 K. Q-slope free results were reproduced in both experimental cavities. Evaporation, consumption and distribution of Sn during the coating is shown to affect the cavity performance remarkably. Further analysis of witness samples is in progress to understand the origin of the recurrent Q-slope. More experiments with recently added new cavities are in place to refine the coating process.

ISBN: 978-3-95450-208-0 doi:10.18429/JACoW-IPAC2019-WEPRB110

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