CUTOUT STUDY OF A Nb_3Sn CAVITY *

S. Posen†, A. Romanenko, Y. Trenikhina, O. Melnychuk, and D. A. Sergatskov
Fermi National Accelerator Laboratory, Batavia, IL 60510, USA
D. L. Hall‡, M. Liepe
Cornell Laboratory for Accelerator-Based Sciences and Education, Ithaca, NY 14853, USA

Abstract

The first 1.3 GHz single cell Nb_3Sn cavity coated at Cornell was shown in RF measurements at Cornell and FNAL to have poor RF performance. Though subsequent cavities showed much higher quality factors, this cavity exhibited Q_0 on the order of 10^9 caused by strong heating concentrated in one of the half cells. This paper presents an investigation into the source of this excess heating, for the purpose of process improvement, so that similar degradation can be avoided in future coatings. Through the use of temperature mapping both at Cornell and at FNAL, locations with high and low surface resistance were located, cut out from the cavity, and studied with microscopic tools. We present the RF measurements and temperature maps as well as the microscopic analyses, then conclude with plans for continued studies.

BACKGROUND

Nb_3Sn is a promising alternative material for SRF cavities, with the potential to increase both accelerating gradients and quality factors at a given temperature compared to niobium, reducing the cost of large accelerators and making feasible new applications for small industrial accelerators. Pioneering work was performed on this SRF material in the 1970s to 1990s at various labs [1–7], and, recently, additional development at Cornell led to 1.3 GHz Nb_3Sn cavities with Q_0 on the order of 10^{10} at accelerating gradients above 15 MV/m [8, 9].

The development effort of Nb_3Sn continues at Cornell and Fermilab, and in this paper, we present a collaborative effort between these two labs to understand a limitation that was observed in a Nb_3Sn cavity, ERL1-5. This was a 1.3 GHz single cell cavity, the first coated under Cornell's program, and it showed considerably poorer performance than cavities coated afterwards. After the first coating, it showed an unusual appearance: looking into the surfaces of the cavity visible from the beamtubes, one half-cell appeared a matte gray as expected, while the other appeared shiny, as shown in Fig. 1.

After coating, the cavity was treated only with high pressure water rinse (HPR), followed by testing, where it showed a Q_0 of 10^9 at low field, with significant Q-slope above 5 MV/m, with the accelerating gradient eventually being RF power-limited, as shown in Fig. 2. The cavity was then

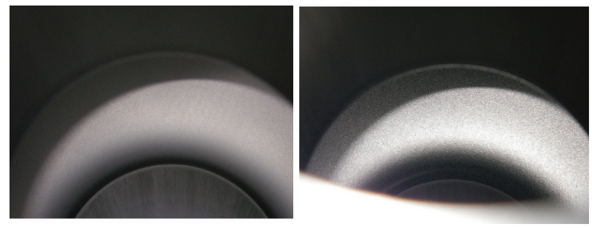

Figure 1: After coating, one half cell showed expected matte gray appearance (left) and the other showed unusually shiny appearance (right).

oxypolished and tested again, with poorer performance. Following this test, the coating was removed with BCP, and the cavity was recoated in the same orientation. Again, the same half cell appeared shiny, so a method was devised to coat the cavity in the flipped orientation, and the coating cycle was run again in this manner. Though the same half cell again appeared shiny, the cavity was tested again, and showed similar performance. The cavity was finally given two cycles of HF rinse and retested, with similar results.

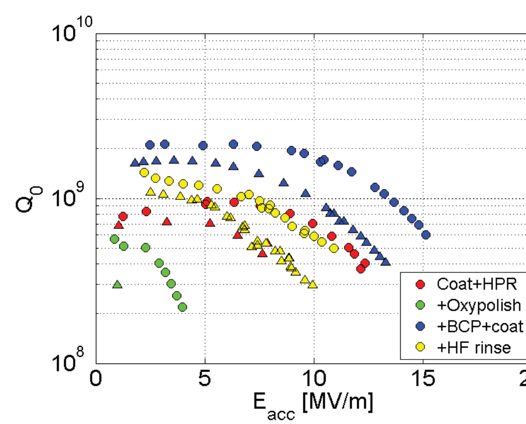

Figure 2: Q vs E curves for the cavity with unusual appearance. Curves were measured at each stage of treatment at 4.2 K (triangles) and 2 K (circles).

OPTICAL INSPECTION

An optical inspection was performed on the cavity before the BCP to remove the first coating. After optical inspection, the cavity was anodized to attempt to distinguish between niobium and tin during optical inspection. The procedure from [4] was followed, which is expected to cause niobium to turn blue and Nb_3Sn to turn purple. However, the procedure specifies that the process should be current limited

* This work was supported by the US Department of Energy and the National Science Foundation
† sposen@fnal.gov
‡ dlh269@cornell.edu

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at a specific current density and eventually reach 75 V, but the process was instead voltage limited below the specified current density. One possible explanation is that the procedure specified use of 50% concentrated NH_4OH whereas only 10% was available. The relatively small surface area of the aluminum cathode stalk compared to the cavity area may have contributed as well. The colors observed after this procedure should not be used as positive identification of materials, but rather as an indication of local surface differences that should be studied with more precise methods.

Fig re3 shows a panoramic assembly of images from optical inspection after anodization, acquired moving the mirror along the cavity axially across the equator. Interestingly, the color appears to correlate strongly with the niobium grain structure. In addition, the color seems to be strongly purple on one side of the heat affected zone of the equator weld, and more of a mix of purple and blue on the other. The half cell that showed blue areas was the one that showed a shiny appearance after coating. The beamtube welded to the blue half-cell turned purple after anodization, and the beamtube welded to the purple half-cell turned blue.

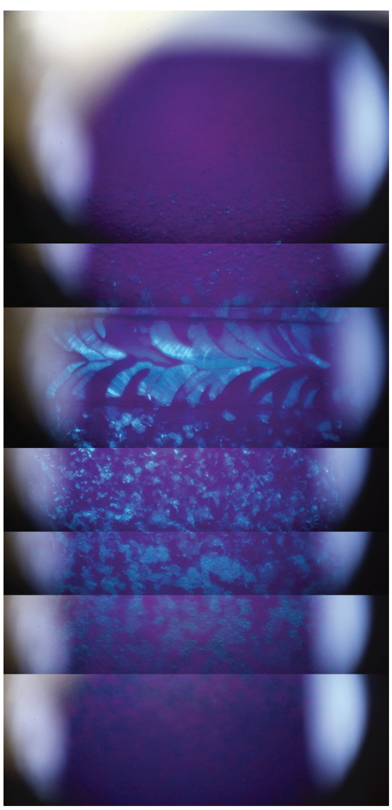

Figure 3: Stitched-together images from optical inspection of the cavity after anodization. Note the dependence of color on niobium grain structure. Note also the dominance of purple regions above the heat affected zone of the equator weld, compared to the bluer regions below.

TEMPERATURE MAPPING

The differences between the two half-cells were further highlighted by temperature maps measured during RF test. Fig. 4a) shows a T-map measured at 9 MV/m at 4.2 K. The T-map data indicate that the half-cell with the unusually shiny appearance has large regions with far higher surface resistance than is observed in the other half cell, showing that the problems with this part of the cavity are more than aesthetic.

The cavity was put aside for some time while other cavities were studied. So far, none have shown similar problems. Recently, the cavity was taken out again, for further investigation of the link between the low Q_0 and the half cell with shiny appearance. The cavity was retested both at Cornell and at Fermilab, each time with temperature mapping, receiving only HPR before each test. The resulting temperature maps are shown in Fig. 4 b) and c). The intention of these tests was to map out well the surface resistance of the cavity, so that the "hot" and "cold" regions could be identified with a high degree of confidence. The figure shows that there were several regions that showed very high levels of heating on multiple T-maps. Coupons located at these "hot" spots were subsequently cut out from the cavity. In addition, several "cold" spots and "medium" spots were cut, as well as some spots at the equator.

RESULTS

Preliminary microscopic analysis has been ongoing for these coupons. So far, scanning electron microscopy (SEM) with energy dispersive X-ray (EDX) analysis has been performed, and several images have been recorded with transmission electron microscopy (TEM). SEM-EDX images of hot spots are shown in Figs. 5 and 6. The EDX maps are presented in terms of atomic percentage, where the ideal expected ratio for Nb_3Sn is approximately 25%.

SEM images show regions with the micron-sized grains observed in high quality Nb_3Sn and regions with more clump-like composition with less pronounced grain boundaries (as previously observed elsewhere, e.g. [10]). EDX analysis of these clump-like regions reveals significantly reduced tin content below the surface. This suggests that the shiny appearance is caused by lustrous niobium with relatively poor coverage of Nb-Sn alloy. In contrast, cutouts from cold spots on the other half-cell show composition consistently close to 25%. Typical microscopy results are shown in Fig. 7.

TEM cross section measurements are still in early stages, but preliminary results agree well with SEM-EDX. A cutout from a hot spot is shown in Fig. 8. It shows a very thin Nb_3Sn region, only hundreds of nm thick, compared to the micons-thick films observed in high quality Nb_3Sn [11].

DISCUSSION

The shiny appearance of the poorly performing half-cell suggested the presence of uncovered niobium, as did the blue regions observed after anodization. Microscopy revealed

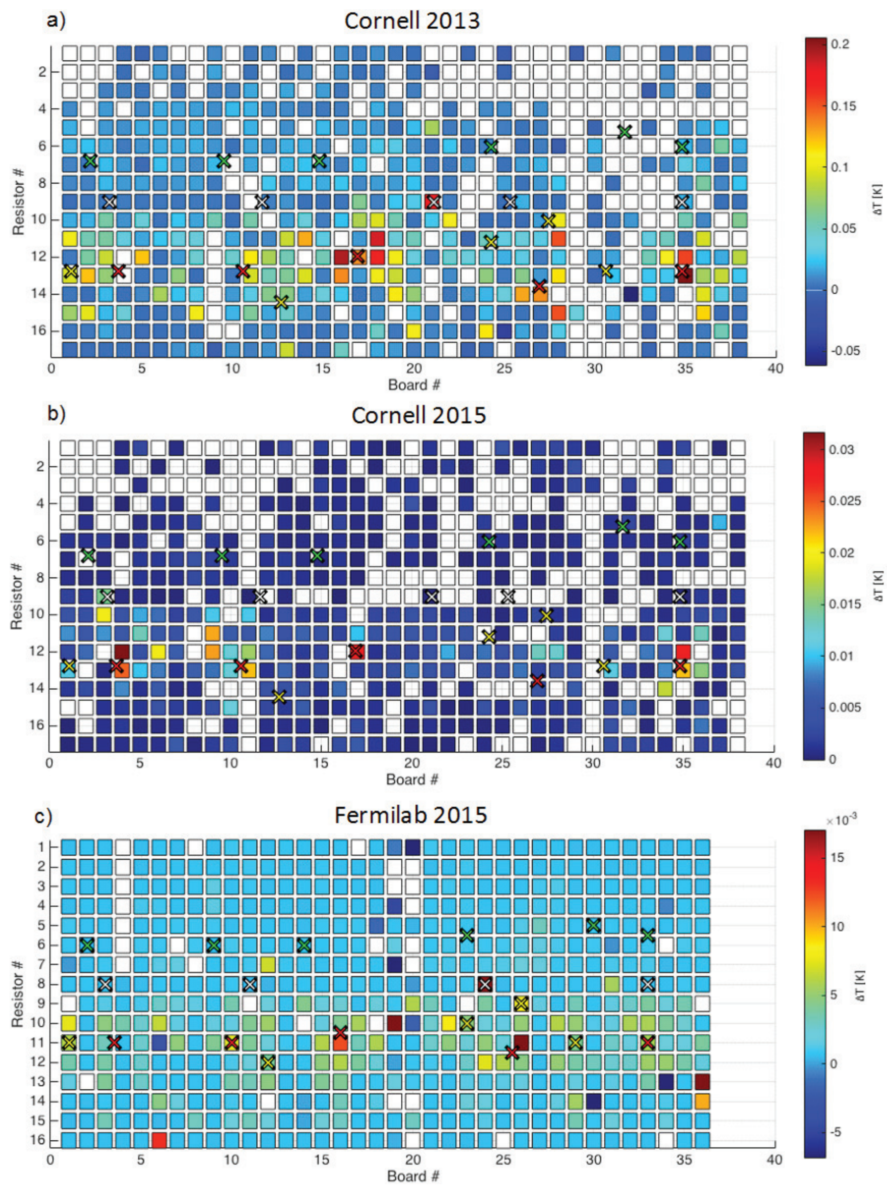

Figure 4: Temperature maps showing excessive heating in one half cell (regions below resistor 9 on the Cornell T-map and below resistor 8 on the Fermilab T-map). First measurements were taken at Cornell in 2013 (a) then new studies were performed in 2015 at Cornell (b) and FNAL (c). Locations from which cutouts were extracted are marked with x's.

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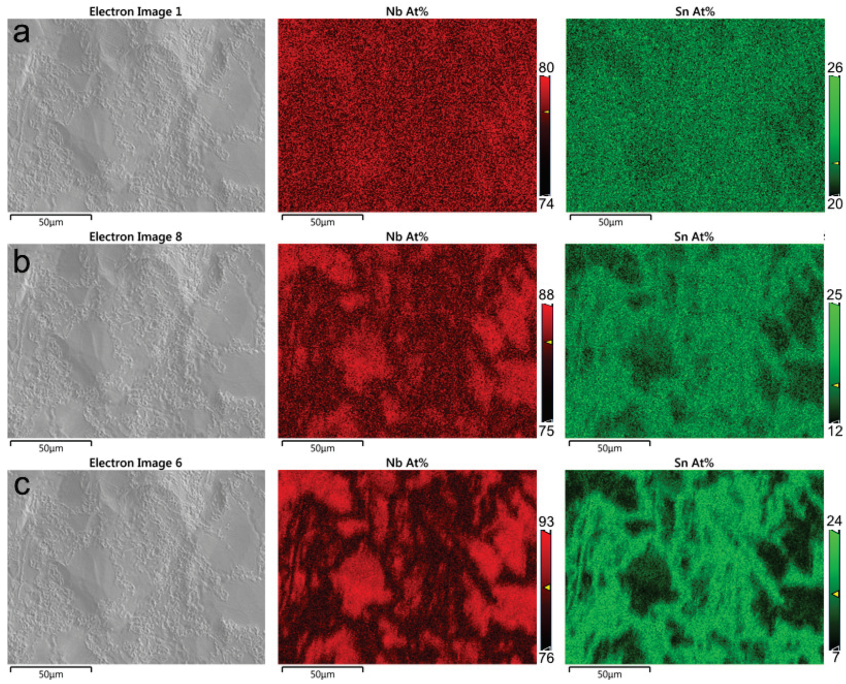

Figure 5: SEM-EDX images of a hot spot, showing some areas with micron-sized grains with tin content close to 25 atomic % and others with less pronounced grain boundaries and lower than ideal tin content. By increasing the energy of the input electrons, their penetration can be increased. Three energies are shown: a) 10 keV, b) 15 keV, c) 20 keV. At smaller penetration levels, the composition appears significantly more uniform than 1-2 microns below the surface. Note that due a large sampling volume and limited collection time, the uncertainty for this measurement is relatively large. A scale bar is added for clarity, but no absolute values should be extracted from the plot (as indicated by the variation with energy).

regions with very thin layers of Nb_3Sn, with regions that are possibly so thins as to be optically transparent, in agreement with visual observations. With penetration depth on the order of 100 nm at $T \ll T_c$ for high quality Nb_3Sn [11], the layer in these regions would only be a few penetration depths thick, not enough to fully attenuate the RF fields, such that the field would be significant on low tin content Nb-Sn alloys. These regions are the likely cause for the relatively high surface resistance values observed at 2 K.

Previous experimenters studying the vapor diffusion process for SRF applications have studied the link between Nb_3Sn coverage and grain orientation using large grain niobium substrates [12]. They observed that some niobium crystal orientations lead to a higher fraction of surface left uncoated if no nucleation step is performed. Considering the strong correlation between color and niobium grain structure observed after anodization, grain orientation may play a role.

OUTLOOK

The study will continue with additional microscopic measurements, including TEM, and analysis of grain orientation. The goal will be to try to understand why very thin Nb-Sn layers formed on one half cell, and how the coating process can be improved to avoid their formation in the future. If there is something problematic for some substrates, avoiding these will be especially critical for multi-cell cavities made from many sheets.

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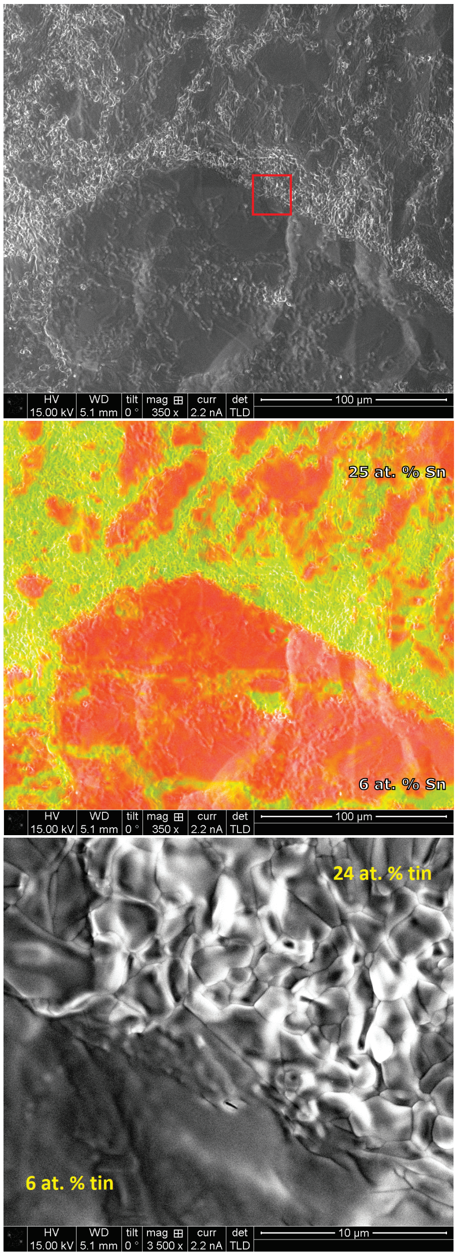

Figure 6: SEM image of a different hot spot, measured in a different microscope, showing similar results. The electron energy used here is 15 keV. Shown here are an SEM image (top), EDX map of the same area (middle), and a zoom-in SEM image of the red box from the first image (bottom).

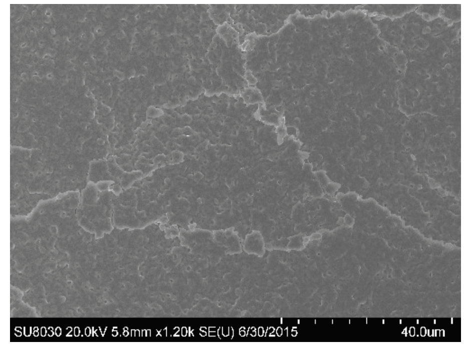

Figure 7: SEM image of a cold spot. EDX shows approximately uniform composition, close to ideal tin content at 20 keV energy.

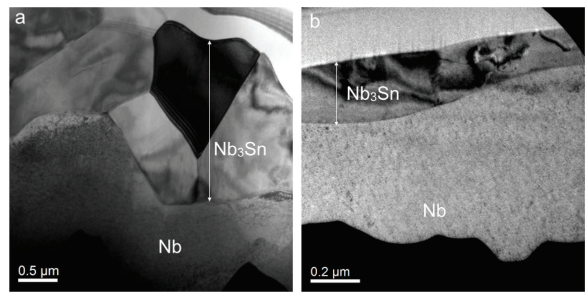

Figure 8: TEM cross-section of a cold spot (left) and hot spot (right). The hot spot shows a relatively thin layer of Nb_3Sn, consistent with SEM-EDX results. The thin layer is not expected to be sufficient to screen RF fields with small surface resistance.

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